NATURAL FIBRE REINFORCED CEMENT COMPOSITES

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YONG NI

DEPARTMENT OF MECHANICAL ENGINEERING
VICTORIA UNIVERSITY OF TECHNOLOGY
AUSTRALIA

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Abstract

The health problems associated with asbestos and its related products necessitated in finding alternative resource of fibres. Over the last two decades natural fibre (mainly wood pulp fibre) has emerged as the most acceptable alternative reinforcement for fibre cement products. The first three chapters of this study describe in some depth the preparation and properties of natural fibres, the methods of incorporating such fibres into cements and mortars, the theoretical principles of fibre reinforcement, the properties obtained from these natural fibre (mainly wood pulp fibre) reinforced cement composites and their applications as commercial products, especially as the main alternatives to asbestos reinforced cement materials. Chapter four and five discuss fabrication and performance characterisation of the resulting composites. Whereas, chapters six, seven, eight and nine incorporate results and conclusions. The detailed experimental procedures and methods are described in Appendix A and B.

Bamboo pulp fibre was investigated as reinforcement for incorporation into cements and mortars. The results show that bamboo fibre is a satisfactory fibre for incorporation into a cement matrix. The composites so formed have acceptable flexural strength, but lack fracture toughness due to the short fibre length and the high fines content of the bamboo pulp used in this study. Experimentation was conducted in an attempt to improve the fracture toughness properties of bamboo fibre reinforced cement composites. Blending bamboo fibre with varying proportions of softwood fibre led to a range of materials with improved performance, especially with respect to the property of fracture toughness.

There is a need to be able to specify the properties of natural cellulose fibres, in particular wood pulp fibres, when they are to be used as reinforcement in fibre cement products. Preliminary studies have attempted to isolate specific fibre properties such as fibre length, fibre strength and fibre lignin content. Then these parameters were used to relate the mechanical performance of fibre cement composites. This study has shown that the fibre length plays a major role in the development of flexural strength and fracture toughness of a fibre reinforced composite. As the length increases both properties improve over a range of fibre contents. There is some concern however, over the best manner of specifying this parameter - average fibre lengths or fibre length distributions. Fibre strength was found to have less effect on composite flexural strength than on fracture toughness values, for both air-cured and autoclaved products. It was somewhat unexpected that weak fibres could provide relatively strong, but; as might be expected, brittle materials. Fibre lignin content was shown to have a considerable effect upon the formation of autoclaved products. There is difficulty, however, to examine fibre lignin content as an isolated parameter, due to interaction from fibre strength, stiffness and fibre-matrix interface bonding.

Overall, this study has provided better understanding of the complex behaviour of natural fibre reinforced cement composites.

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Chapter One:

Introduction

1.1 Natural Plant Fibre Reinforced Cement Composites (NFRC)

Since the end of the 19th century, asbestos cement has had a wide range of applications, such as building / cladding sheets, corrugated roofing elements, pipes and tiles.

Because of the well known health risks associated with the use of asbestos fibres, together with a possible future shortage of asbestos, there has in recent years, been considerable research into the development of new high-performance reinforcing fibres. A variety of fibres such as glass, steel, synthetic polymer and natural cellulose fibres have been evaluated in both laboratory and pilot plant equipment. Among these fibres, natural cellulose fibres (Kraft pulped soft wood fibres) demonstrate both cost effectiveness and suitable performance to act as a replacement for asbestos fibre. James Hardie Industries in Australia marketed non-asbestos wood fibre reinforced cement (WFRC) boards in 1981 (Anon, 1981).

1.1.1 History and development of (NFRC) composites

Although patents from the last century refer to the use of natural plant fibre as a component of building materials made from cements and plasters (US Patent, 1884,1899,1900), interest in natural fibre (mainly wood fibre) as a reinforcement for fibre cement has mainly taken place in the last 10 - 20 years.

Unfortunately the large fibre cement manufacturing companies are the real custodians of the history of the fibre cement development and, from the obvious gap in the literature, they have released very little information about the use of natural fibres in cement.

James Hardie and Coy Pty Ltd started manufacturing asbestos cement products in Australia in 1917 (James Hardie Industries, 1984). James Hardie Industries took an active interest in the use of cellulose, as an economic asbestos substitute, in fibre reinforced cement in the early to mid-1940s. This work was intensified during the post-World War II years when there was a worldwide shortage of asbestos fibre. An investigation was conducted by Heath and Hackworthy to discover whether paper pulp could be used to replace asbestos completely or partially in asbestos cement sheets (James Hardie and Co. Pty Ltd., 1947). Fibres studied included bagasse, groundwood, wheat straw, cement bags and brown paper. The experimental autoclaved sheets showed brown paper (Kraft) was the best of the pulp sources, giving greatest strength to the composite material. However, when asbestos supply was reinstated, this work was discontinued.

Renewed interest in wood fibres began almost inadvertently in 1960. In those days, the asbestos fibre board, containing 15% asbestos, was made between steel interleaves. James Hardie's was believed to be the only group in the world which at that time was steam-curing its sheets. To make a cheap board as an alternative interleaf, boards were made up with half the asbestos replaced by wood fibres. This board became the first generation "Hardiflex", and full production started in 1964. From the 1960s onwards their products have contained no more than 8% asbestos, which was about half the amount used by the rest of the industry.

Attempts to further reduce the asbestos content by adding more wood fibre were unsuccessful due to the ineffectiveness of these fibres, compared to asbestos, in trapping the cement particles during formation of the sheet.

James Hardie entered into collaborative research with CSIRO in 1978 to study, among other things the refining of cellulose fibres in an attempt to overcome the difficulties of retaining the cement in the wood fibre reinforced cement sheet (Anon, 1981). By May 1981 the new generation of asbestos-free cement products - Hardiflex II - was being commercially manufactured. This autoclaved product was totally reinforced by refined Kraft wood fibres (Coutts, 1982a; Aus Patent, 1981).

In Europe during 1975 - 77, Cape Industries had made boards reinforced with 5% cellulose and high levels of mineral fillers called "Supalux", "Monolux" and "Vermiculux", mainly for fire-resistant use. "Masterboard" and "Masterclad" were more dense and stronger and used for external cladding (Harper,1982). In 1976 Sweden banned asbestos cement products, and all asbestos cement production was closed down. Other Scandinavian countries were forced to use alternatives, and A /S Norcem in Norway and OY Partek AB in Finland decided to form a joint development under the name of NOPA (Pedersen, 1980). The material produced was called "Cellcem", and contained cellulose fibres mixed with other fibres. Manufacture of the products "Internit" and "Pernit" started in Norway in 1977. In 1979 Finland started to produce "Minerit".

In was stated in 1985 that the UK manufacturers had replaced asbestos in about 50% of the fibre cement sheeting products (Crabtree, 1986). James Hardie Industries by this time had

totally replaced asbestos fibre from its range of building products, which included flat sheet, corrugated roofing and moulded products.

As well as flat sheet products, James Hardie Industries had become a world leader in injection moulded fibre cement products and non-pressure fibre cement pipes, all based on wood fibre as the reinforcement material. The first experimental production of WFRC pipe was undertaken at the Brooklyn factory in September 1980. Commercial production began in Western Australia at the Welshpool factory in July 1984. The last asbestos pipes made by James Hardie were manufactured in March 1987.

At the present time there is considerable activity in the patent literature concerning the use of natural plant fibres (mainly wood fibres) or mixtures of natural plant fibres (mainly wood fibres) with other synthetic fibres. This is taking place throughout Europe and Japan, and companies such as Dansk Eternit-Fabrik A /S, Cape Boards and Panels Ltd, Partek of Finland, Asano of Japan and others are involved.

1.1.2 Fabrication processes

The manufacture of asbestos fibre cement products is a mature industry. Hodgson (1985) suggested that over 1100 sheet machines and 500 pipe machines were in production around the globe, with total capital investment in excess of A$ 4.6 billion. The Hatschek process (or wet process) is the most widely used method of production (see Figure 1.1). The manufacturing techniques are closely related to conventional heavy paper and board making processes. An aqueous slurry of asbestos and cement matrix, about 7-10% solids by weight, is supplied to a holding tank which contains a number of rotating screen cylinders. The cylinders pick up the solid matter removing some of the water in the

operation. An endless felt band travels over the top surfaces of the cylinders and picks up a thin layer of formulation from each cylinder. The " built-up " laminated ply then travels over vacuum de-watering devices which remove most of the water. The formulation is then wound up on a steel calender, or assimilation roll, until a product of desire thickness is formed. The material is further compressed by pressure rolls which are in contact with the assimilation rolls.

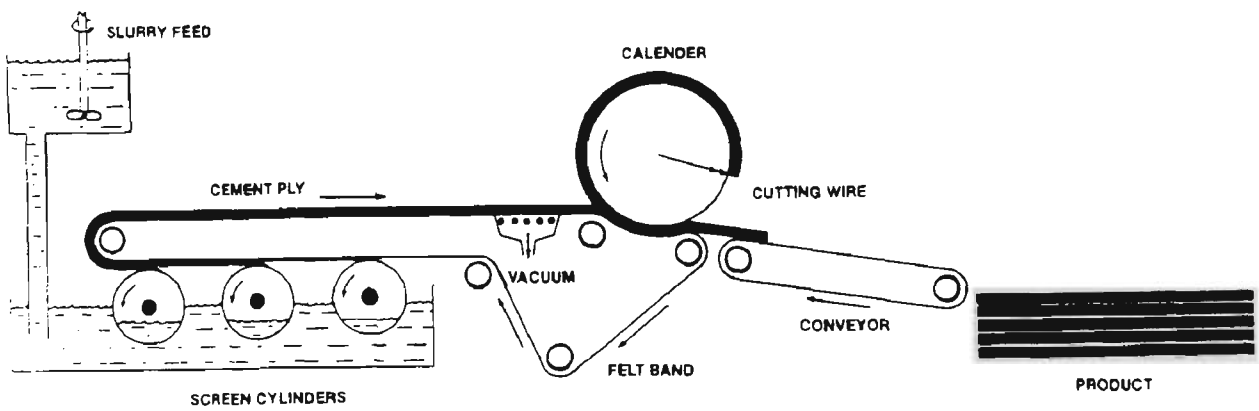

Fig. 1.1. The Hatschek process.

For sheet production the layer built up on the assimilation roll is automatically cut off and drops onto a conveyor to be transferred to a stack for curing. If corrugated roofing is to be made, the flat sheet is taken off to a corrugating station where the sheets are deposited onto oiled steel moulds for shaping. Pipe machines are similar to the Hatschek process but usually have only one or two vats in series. The pressure imposed on the mandrel by the press rolls is much greater than for sheet production so as to form a dense product. The machine may be stopped while the mandrel carrying the pipe is set to one side for pipe withdrawal. The process is often referred to as the Mazza process.

The Magnani (or semi-dry) process can be used to prepare pipes and corrugated sheet. This process has the advantage that it can provide a greater thickness of material at the peaks and troughs of the corrugations and so increase the bending strength. The thick slurry (about 50% solids) of this process can flow uniformly and directly onto a felt conveyor which passes over numerous vacuum boxes to dewater the formulation. In the case of corrugated roofing the felt is compressed over a corrugated former by a shaped roller. Pipe formation is similar to the Mazza process.

Injection moulding is now tending to replace the hand moulding of green sheets (from the Hatschek process) for the manufacture of special fittings. A slurry of 40-50% solids is pumped into a permeable mould and then subjected to pressures, in excess of 20 atmospheres, in a hydraulic press via a rubber diaphragm. The mix is dewatered by this process of pressure filtration, and then has sufficient green strength for the product to be demoulded by means of a suction lifting pad, and transferred to a pallet for curing. The operation is very fast.

The formulation of the matrix, and hence the cure of the product, has varied from country to country and between companies within a country. The formulations remain confidential to the company or its licensees and only general details will be discussed here. The autoclaved curing process has always been favoured in Australia and the USA, and in some European countries. In the autoclave process, the matrix is usually a mixture of ordinary Portland cement (OPC) and finely ground sand (silica), or lime and silica. The product, after an initial pre-cure period in air, is cured in an autoclave in a steam environment, say 8 hours at 170-180 ^{0}C. The cured sheets are virtually at full strength after autoclaving and can be dispatched from the factory in a short time. By contrast the

more traditional air-cured products require 14-28 days of air-curing before they can be dispatched, this involves considerable stock inventory. The air-curing process is lower in capital outlay, as no high pressure autoclaves and steam raising plant are required; however, cement is more expensive than silica, and therefore material costs are higher.

1.1.3 Property requirements for asbestos alternatives

After reviewing generally the processes used to manufacture asbestos fibre cement, a number of requirements of replacement fibres can be noted, if the existing capital intensive equipment is to be used. For the Hatschek process a replacement fibre must be water dispersable in a relatively dilute slurry and able to form a film on the screens. At the same time the fibre must be able to resist chemical attack due to the high alkalinity (~ pH 13) of the matrix. If the product is to be autoclaved, resistance to temperatures above 170 ^{0}C is also required. The basic essentials of cost, availability and mechanical performance of the fibre are obvious.

As has been reported glass, steel, carbon, synthetic organic as well as natural plant fibres (mainly wood pulp fibre) have been under examination for use in cement systems. We will look at a comparison of the properties of these fibres as possible asbestos replacements in existing processes in Table 1.1.

For countries committed to autoclaved products the combination of high alkalinity and high temperature eliminates most fibres apart from natural plant fibres (mainly wood pulp fibre), steel, carbon and aramid fibres. The cost of the latter two is almost a factor of twenty times higher than natural plant fibres (eg. wood pulp fibre) and so look unattractive. Steel fibres have processing problems. If one considers air-curing fibre

cement, to eliminate the temperature problems, there are still processing limitations. The inorganic fibres such as steel or glass, tend to be too stiff or dense to perform well during film-forming from dilute slurries; while the organic fibres lack a surface suitable for bonding to the matrix and / or introduce drainage problems. Mixtures of organic fibres (mainly PVA) and natural plant fibres (mainly wood pulp fibre) fibres were successfully used to produce air-cured products in Europe (Studinka, 1989).

Table 1.1 Comparison of properties of fibres for possible asbestos alternatives (Coutts, 1988).

Fibre	Alkalia. resist.	Temp. resist.	Process ability	Strength	Toughness	Price
Wood pulp (chem)	1	1	1	1	1	3
Wood pulp (mech)	2	2	2	2	3	3
Polypropylene	1	3	3	3	3	2
PVA	1	3	3	1	1	2
Kevlar	1	1	2	1	1	1
Steel	1	1	3	3	3	2
Glass	3	1	3	3	3	2
Mineral fibre	3	1	3	3	3	3
Carbon	1	1	3	1	1	1

1. High, 2. Medium, 3. Low.

1.1.4 Mechanical and physical properties of NFRC

As stated above, natural plant fibre (mainly wood fibre) is the most cost / performance asbestos alternative. Natural plant fibre contains cellulose, hemicellulose and lignin. The cellulose fibre is the main reinforcing elements. Most natural plant fibres contain more than 45% cellulose and some fibres even yield great than 75% cellulose (see section 3.3).

Natural fibre has been successfully employed as an asbestos fibre alternative either by itself or as mixture with other synthetic fibre for 10 - 15 years. James Hardie Industries in Australia manufactured a varies of autoclaved fibre cement sheets and pipes reinforced with about 8%-10% beaten softwood (*P. radiata*) Kraft pulp. The Eternit Group in

Switzerland promoted various types of cellulose fibre reinforced autoclave composites in South Africa and some air-cured cement products in Europe and Latin America reinforced with synthetic fibres and natural cellulose fibres.

The amount of data available on natural fibre reinforced cement (NFRC) products, in the scientific literatures, has been limited due to the fact that manufacturing interests had been responsible for much of the preliminary work and for commercial reasons had retained the knowledge in house or locked away in patent literature. Unfortunately, due to the difficulty in handing theoretical treatments involving natural fibres, there was less interest from the academic fraternity than in say cement materials containing glass, steel or synthetic organic fibres which form the basis of a voluminous scientific literature.

This chapter will only address some selected results, relating to products containing natural plant fibres (mainly wood fibres) as the sole reinforcement for cement matrices, in order to give an appreciation of various effects.

1.1.4.1 Strength and fracture toughness of NFRC

During last two decades a number of papers have appeared in which wood fibres are the sole source of fibre reinforcement. These studies have included chemical and mechanical pulps of softwoods and hardwoods in air-cured and autoclaved matrices (Coutts, 1985,1986,1987a).

It will be seen that refined wood fibres can afford a strong, tough and durable fibre cement, when produced commercially by traditional slurry/dewatered systems followed by autoclaving. Such WFRC formulations can be used for the production of flat sheeting,

corrugated roofing, moulded products and low-pressure pipes which traditionally have used asbestos fibre. Sometimes the laboratory experiments are misleading with respect to the manufacturing processes and care must be taken in extrapolating the laboratory results into production.

Although natural cellulose fibre (eg. wood pulp fibre) is cheap and readily processed it has the disadvantage of being hygroscopic. The composite properties are altered by absorption of water and, for this reason, extensive testing when both wet and dry is required. WFRC products are generally loaded in bending and so flexural strength has more meaning than tensile strength in the characterisation of these materials.

At CSIRO Australia, there was an interest in using high yield pulps [(thermomechanical pulp (TMP) and chemithermomechanical pulp (CTMP)], as an alternative to chemical pulps, for reinforcing fibre cements. Such pulps make less demand on the forest resources for a given quantity of pulp (yields twice that of chemical pulps), less problems with effluent treatment, chemical requirements are much lower and processing plants are economical at a smaller scale. Coutts (1986) reported that mechanical pulps of *P.radiata* in general were unacceptable as cement reinforcement when autoclaved (with MOR less than the matrix) but when air-cured had flexural strengths greater than 18 MPa at 8-10% by mass of fibre. This compares poorly when one notes flexural strengths of WFRC's containing chemical pulps of *P. radiata* are in excess of 20 MPa when autoclaved and 30 MPa when air-cured. When autoclaving mechanical pulps the high temperature and alkalinity virtually "chemically pulps" the fibres releasing extractives of polysaccharides and wood acids, which "poison" the matrix near the fibre causing poor interfacial bonds.

Air-curing is less drastic with respect to chemical attack, hence better properties are evident in the final composites as shown in Figure 1.2.

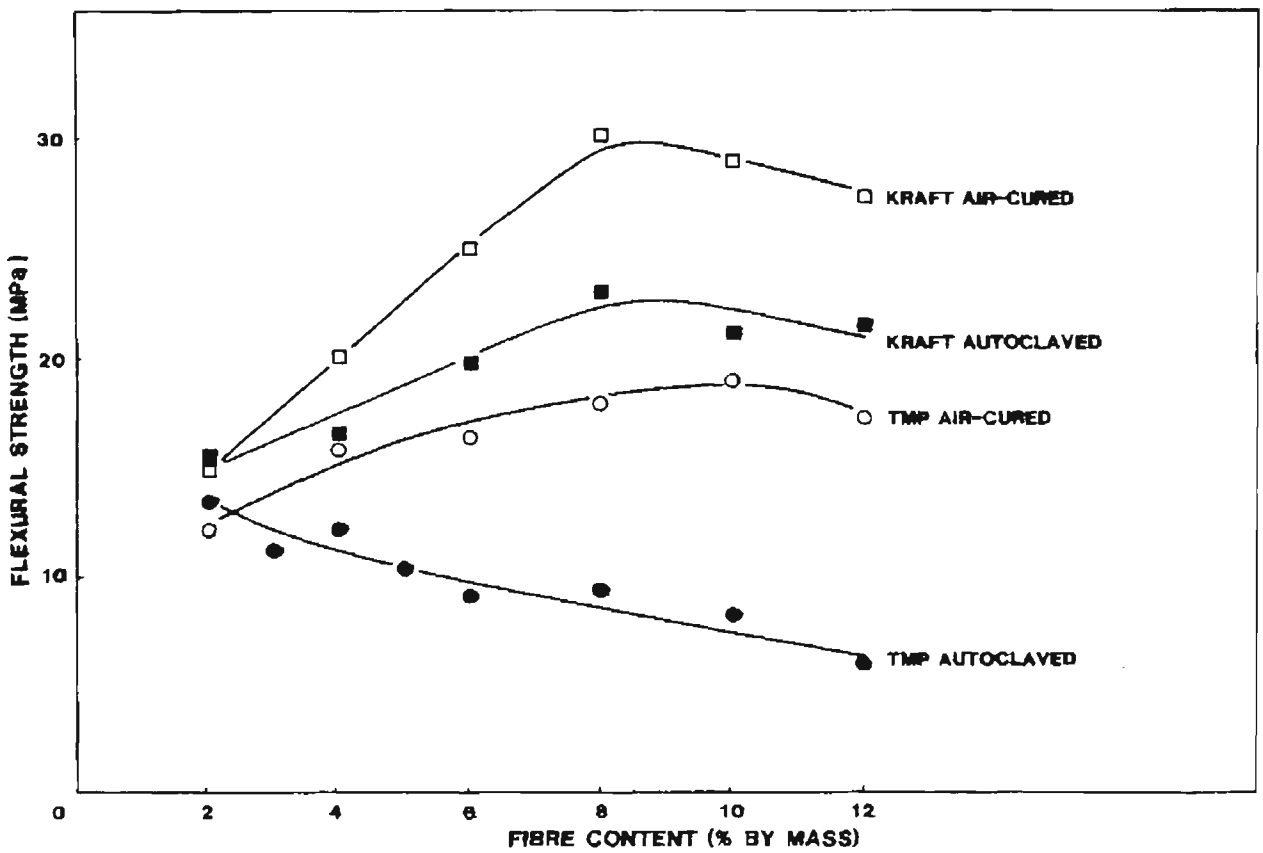

Fig. 1.2. Graph of flexural strength v. fibre content for various WFRC products (Coutts, 1988).

Although it was documented from work in the laboratory that Kraft wood fibre was effective as a reinforcement in a cement matrix (Coutts, 1979a), the pulp performed poorly on a pilot-plant Hatschek machine because the fibres were unable to form a web capable of retaining cement and silica particles. The open nature of the web permitted rapid drainage, with loss of matrix, resulting in low product strength. A collaborative project, between CSIRO and James Hardie Industries, starting in 1978 resulted in laboratory data which demonstrated the benefit of refining wood fibres for use in WFRC materials (Coutts, 1982a, 1986; Aus Patent, 1981). The breakthrough that made commercial production possible came about from the work of the Hardie's team in adapting the fibre refining step to suit the Hatschek machine. Before launching the product in 1981 over

50,000 sheets had been prepared and tested on the pilot-plant, and about $10 million invested in installing refining equipment in the factories.

The effect of moisture on the strength properties of WFRC composites is of importance, and early on in the research variations in test conditions produced variations in test results. It was evident that standard conditions must be adopted. The flexural strength of WFRC's can be reduced to as low as 50% of dry strength values in laboratory samples, in the case of commercial products the reduction is considerably less but is taken into consideration for product application.

Fördös and Tram (1986) reported WFRC's containing micro silica with excellent strength values ranging between 25-55 MPa. A very high stack compression pressure, approximately 20 MPa, was used. Whereas most results usually report pressures of approximately 2-3 MPa. Coutts and Warden (1990a) demonstrated the effect on compaction on the properties of air-cured WFRC and showed that flexural strength increased with casting pressure without resulting in a reduction in fracture toughness. Few workers have reported the elastic modulus of WFRC materials. Andonian et.al.(1979) have shown that both tensile and bending moduli are reduced from approximately 13 GPa to 9 GPa as the fibre content increases from 2 to 10% fibre by mass.

Fracture toughness is perhaps the most important property for a building material. Although strength and stiffness are important, the ability of a material to absorb impact during handling can decide whether it will find an application in the market place. Fibre type, pulping method, refining conditions and test conditions all have an effect on the fracture toughness results of a given formulation. As the fibre content increases up to

about 8-10% by mass the fracture toughness increases rapidly then starts to taper off. Values of fracture toughness in excess of 60 times matrix values can be obtained with 10% fibre by mass. Fracture toughness increases even further when tested wet. This effect will be discussed when we consider bonding and microstructure. TMP pulps when compared with chemical pulps of the same species tend to have fracture toughness values less than half that of the chemical pulp reinforced composite. This can be partly explained in terms of fibre number and fibre morphology (Coutts, 1986). The variation of fracture toughness values between softwoods and hardwoods can be attributed to fibre length and fibre morphology, which we will see is so important for fibre pull-out which takes place during failure under load.

Beside softwood fibre, considerable research has been conducted on some hardwood fibres, non-wood natural plant fibres and waste paper in order to search for cheaper and more naturally available fibre resources and to better understand the natural fibre reinforced cement based composite materials. The mechanical and physical properties of commercial WFRC and some laboratory fabricated NFRC are listed in Table 1.2 and Table 1.3, respectively.

It can be seen from Table 1.2 and 1.3 , when natural fibre are prepared by the chemical pulping method, they can produce acceptable fibre cement products, when either air-cured or autoclaved, with flexural strength properties comparable to those of softwood. However, their fracture toughness values tend to vary and most of them less than WFRC. Such phenomena might be explained by different fibre parameters such as fibre length {eg. hardwood (Coutts,1987a), bamboo (Coutts,1994a), waste paper (Coutts, 1984a)}, fibre

content {eg. TMP (Coutts, 1986)}, fibre strength {eg. NZ flax (Coutts,1983c,1994b)} and helical angle {eg. banana (Coutts,1990b)}, while the other phenomena remain uncertain.

Table 1.2 Mechanical and physical properties of commercial WFRC materials based on James Hardie's products (Coutts, 1988).

	Flex. Strength (MPa)		Mod. of Elast.(GPa)		Frac. Toughness(kJ/m^2)		Density	W.Abs.
Product	RH	Wet	RH	Wet	RH	Wet	(g/cm^3)	(%)
Villaboard II	19.2(L)	12.9(L)	9.2(L)	6.1(L)	6.7(L)	9.0(L)	1.31	32.1
(6 mm)	13.5(T)	8.6(T)	8.5(T)	5.2(T)	3.0(T)	5.6(T)	"	"
	16.3(av)	10.7(av)	8.9(av)	5.7(av)	4.9(av)	7.3(av)	"	"
Hardiflex II	24(L)	/	/	/	/	/	1.40	30.5
	12(T)	/	/	/	/	/	"	"
	18(av)	/	/	/	/	/	"	"
Compressed	29.4(L)	21.0(L)	16.6(L)	12.7(L)	4.2(L)	19.5(L)	1.62	18.6
sheet II	22.1(T)	15.9(T)	15.3(T)	12.3(T)	2.4(T)	8.1(T)	"	"
(12 mm)	25.7(av)	18.4(av)	15.9(av)	12.5(av)	3.3(av)	13.8(av)	"	"

*L: longitudinal direction of manufacture
*T: transverse direction

Table 1.3 Mechanical and physical properties of laboratory fabricated NFRC

Pulp	Fibre	Curing	Strength(MPa)	Toughness(kJ/m^2)	Density(g/cm^3)	W.Abs.(%)	Reference
Kraft	softwood	air	30.3	1.93	1.55	21.1	Coutts85
"	"	auto	23.1	1.86	1.31	33.9	Coutts84a
"	hardwood	air	20.3	1.37	1.45	25.8	Coutts87a
"	abaca	air	27.3	2.08	1.55	21.9	Coutts87b
"	NZ flax	auto	23.2	0.84	1.31	35.0	Coutts83
"	banana	air	20.0	0.83	15.4	22.9	Coutts90b
"	"	auto	18.5	0.55	/	/	Coutts90b
"	sisal	auto	18.3	2.49	1.37	27.6	Coutts92a
"	bamboo	air	17.0	0.34	1.59	16.7	Coutts94b
"	"	auto	15.5	0.29	1.41	30.7	Coutts94a
TMP	softwood	air	12.3	0.57	1.47	17.8	Coutts86
"	"	auto	9.5	0.89	1.29	30.5	Coutts86

*All composites containing 8% fibre (by mass)
*TMP denotes thermomechanical pulp

1.1.4.2 Physical properties of NFRC

The physical properties of NFRC materials can have a considerable influence on their acceptability for use in the construction industry. If a product is strong and tough and has low density it will be preferred by the workers, who handle such products on the building

site, compared to similar materials that are dense. At the same time due consideration must be given to water absorption, for as the density is lowered, the void volume increases with an associated potential increase in water absorption. Thus a load on a structure may be considerably increased should the material become wet, with more than 30% increase in weight occurring in some laboratory cases. High temperature mechanical pulps are very stiff, compared to chemical or low temperature mechanical pulps, and cause poor packing as the fibre content increases. As void volume increases with poor packing so the density decreases and water absorption increases. Matrix material also affects the density and water absorption. Air-cured NFRC materials are more dense than autoclaved materials.

1.1.5 Durability of NFRC

Considerable doubt has been cast on the ability of natural fibres to resist deterioration in cement matrices, yet no evidence has been put forward to support the claims. When poor mechanical performance of the composite has been offered as confirmation of fibre failure, due consideration should be given to the potential of the fibre strength loss and to "poisoning" of the matrix surrounding the fibre, resulting in weak interfacial bonding. An extensive review by Gram (1983) reflects this uncertainty.

Lola (1986) reported the failure of natural fibre cement products after only a few years of service. In many cases the reinforcement fibre used was aggregates of fibres and the high alkalinity (matrix), coupled with cycles of wetting and drying, "pulped" the fibre bundles resulting in loss of fibre strength, "poisoned" cement and weak interfacial bonds, and thus low durability. On the other hand there are many claims which suggest that natural fibre reinforced cement products are durable after 30 years of service.

Sharman and Vautier (1986) have done some excellent work on the durability of autoclaved WFRC products at the Building Research Association of New Zealand. They discussed the possible ageing mechanisms of corrosion, carbonation, moisture stressing and microbiological attack.

Akers and co-workers (1986) have published a series of papers which discuss the ageing behaviour of cellulose fibres both autoclaved and air-cured, in normal environments and accelerated conditions. Testing had taken place which showed exposure of WFRC composites to natural weathering led to an overall increase in flexural strength and elastic modulus after 5 years. The same workers found that air-cured WFRC products when aged, either normally or by accelerated means, showed a marked reduction in fracture toughness. The ageing of autoclaved materials did not result in mineralisation of the fibres and maintained good levels of fracture toughness. The need to use synthetic fibres in air-cured products was apparent, however, with aging there appears to be an increase in the interfacial bond which leads to a greater occurence of fibre failure rather than pull-out and thus leads to higher strengths but lower fracture toughness. A general picture is emerging as more studies are conducted that the autoclaved WFRC products are more durable than the air-cured hybrid composites which contain mixtures of cellulose and synthetic organic fibres.

1.2 Natural Plant Fibre Resources

The use of natural plant fibre as reinforcement in building products has been known since man made mud bricks using straw or sailed the seas in boats made from reeds and sealed with resins or bitumen. The main use of such fibres, in developing countries, has been to provide cheap, relatively low performance materials. The tendency in developed countries

was to neglect the research of natural plant fibres for use in cement composites, that is until the "explosion of interest", as evidenced by the scientific and patent literature, which occurred in the mid 1980's and is expanding to the present time.

Natural plant fibres exist in large quantities all over the world including wood fibre and nonwood plant fibre. Great amounts of non-wood natural plant fibre are available and produced in most developing countries. For example, in India alone, some 6.0 million hectares of land is occupied with banana plantations and it was stated that 3 million tons of banana fibre are available (Coutts, 1990b). Bamboo is another readily available fibre source, there are altogether 62 genera and over 1000 species of bamboo in the world, of which 37 genera and about 700 species grow in Asia. China has the greatest number of bamboo species and the area of bamboo plantation in China is 3.2 million hectares, which is one fifth of the total bamboo grove coverage in the world (Zhao, 1990).

An important factor in the availability of any plant for fibre is its collectable yield per unit land area. Such estimated yields are given for a number of non-wood plants in Table 1.4, first as collectable raw material, then as the estimated equivalent in bleached pulp.

Based on the total world-wide production of agricultural crops and the land area planted in each crop, it is possible to make reasonably accurate estimates of the total amount of each agricultural residue, useful as fibre, which might be collected in each country. Similar estimates can be made for crops grown specifically for their fibre content. However, for natural growing species, e.g. reeds and bamboo, such estimates are far more difficult, and accurate data are not available.

Table 1.4 Annual collectable yields of various non-wood plant fibrous raw materials (estimated) (Atchison, 1983).

Fibrous raw material	Collectable as raw material BD metric tons per hectare year	Equivalent in bleached pulp BD metric tons per hectare year
Sugar cane bagasse	5.0 - 12.4	1.7 - 4.2
Wheat straw	2.2 - 3.0	0.7 - 1.0
Rice straw	1.4 - 2.0	0.4 - 0.6
Barley straw	1.4 - 1.5	0.4 - 0.5
Oat straw	2.2 - 3.0	0.4 - 0.5
Rye straw	1.4 - 2.0	0.8 - 1.0
Bamboo, natural growth	1.5 - 2.0	0.6 - 0.8
Bamboo, cultivated	2.5 - 5.0	1.0 - 2.1
Reeds in the USSR	5.0 - 9.9	2.0 - 4.0
Kenaf-total stem weight	7.4- 24.7	3.0 - 9.9
Kenaf bast fibre	1.5 - 6.2	0.7 - 3.2
Crotalaria bast fibre	1.5 - 5.0	0.7 - 2.5
Papyrus in Upper Sudan	20.0 - 24.7	5.9 - 7.4
Abaca (Manila hemp)	0.7 - 1.5	0.4 - 0.7
Seed flax straw	1.0 - 1.5	0.18 - 0.27
Cotton staple fibre	0.3 - 0.9	0.25 - 0.86
Corn stalks	5.5 - 7.0	1.55 - 1.95
Sorghum stalks	5.5 - 7.0	1.55 - 1.95
Cotton stalks	1.5 - 2.0	0.60 - 0.80

Table 1.5 presents estimates giving a reasonably good indication of the tremendous quantities of these non-wood plant fibres which can become available if economic necessity requires their use as papermaking and reinforcing raw materials.

Table 1.5 Availability of various non-wood plant fibrous raw materials, 1982 (estimated) (Atchison, 1983)

Raw material	Potential world-wide availability BD tons
Straw (wheat, rice, oat, barley, rye, seed flax, grass seed)	1,145,000,000
Sugar cane bagasse	75,000,000
Bast fibres (jute, kenaf, roselle, true hemp)	2,900,000
Core material from jute, kenaf, hemp	8,000,000
Leaf fibres (sisal, abaca, henequen)	480,000
Reeds	30,000,000
Bamboo	30,000,000
Papyrus	5,000,000
Corn stalks and sorghum stalks	900,000,000
Cotton stalks	70,000,000

An estimate of world-wide pulp production from nonwood plants, by type of fibre, is given in Table 1.6. These figures include production for dissolving pulp. These fibres are

mainly used in paper and paperboard products. However, these fibres also provide ready reinforcement source for composite materials, such as fibre reinforced cement products.

Table 1.6 Total production of various non-wood plant fibre pulps in 1982 (estimated) (Atchison, 1983)

Type of no-wood plant pulp	World production (air-dry tons)
Cereal straw-mainly wheat and rye	1,390,000
Rice straw	750,000
Bamboo	960,000
Bagasse	1,600,000
Reeds	1,400,000
Cotton linters (paper grade and dissolving pulp)	360,000*
Esparto and sabai grass	120,000
Rags, abaca, flax, seed straw, hemp, sisal and other plant fibres	1,420,000

* Includes about 60,000 metric tons for paper grade pulp, remainder for dissolving pulp & nonwovens.

1.3 Scope of the Present Work

1.3.1 Bamboo fibre and bamboo-wood hybrid fibre reinforced cement composites

The fibre cement industry has moved towards autoclaved natural plant fibre (wood) reinforced cement mortars as the most commercially viable product to replace asbestos cement products. In those countries without adequate forest resources but which have a great amount of other natural plant fibre resources, then manufacturing fibre cement products with these non-wood plant fibre would be of great advantage.

Bamboo is a rapid grown natural plant, which has good fibre qualities and is widely used in the paper industry throughout the Asia region. Although bamboo has been used in various forms in the construction industry, there is limited information in the scientific literature concerning the use of bamboo pulp fibre. One objective of the current work is to evaluate bamboo pulp fibre reinforced cement composites properties and study bamboo fibre combined with wood fibre in order to improve the composites performance. The work is covered under the following three topics:

1. Air-cured bamboo pulp fibre reinforced cement composites;

2. Autoclaved bamboo pulp fibre reinforced cement composites;

3. Hybrid bamboo and wood pulp fibre reinforced cement composites.

1.3.2 Influence of fibre properties on composite performance

Much has been written about the enhancement of engineering properties of cement based products by the inclusion of fibres as reinforcement. Excellent texts and reviews on the basic concepts of fibre reinforcement of brittle matrices are available, which deal with synthetic fibres such as steel, polypropylene, glass and more recently kevlar and carbon. Such fibres are homogeneous with respect to chemical composition and anatomical structure, they have uniform cross-section area and can be obtained at a constant length, and their tensile strength and modulus are constant or could be easily modified. Unfortunately, natural pulp fibres are not homogeneous in chemical composition nor uniform in shape or size even from one given species. At the same time the tensile strength and modulus varies from fibre to fibre within a given pulp source.

The development of asbestos free fibre cement industry has made it most desirable to obtain definite information on the relationship between the nature of the natural plant fibres and their composites properties. It has been generally recognized that the fibre length and strength are two of the most important factors but, because various features of fibre morphology and chemical composition can influence composites properties, it has been difficult to obtain a clear picture of the effect of any one property.

Further work is aimed at developing a fibre model which identifies those properties of wood pulp fibre that are most significant for the production of fibre cement products. This

model may assist in identifying alternative cellulose fibre resources suitable for reinforcement and to better understand natural fibre reinforced cement based composites. The experiment work undertaking will be directed at better understanding of the following:

1. Influence of fibre length on composite properties;

2. Influence of fibre strength on composite properties;

3. Influence of fibre lignin content on composite properties.

Chapter Two:

Theoretical Principles of Fibre Reinforcement

If the maximum benefit of composite materials as engineering materials is to be achieved, it is necessary to understand their potential for bearing loads. Failure in a fibre composite emanates from defects in the material. These may be broken fibres, flaws in the matrix and debonded fibre / matrix interfaces. Figure 2.1 shows a schematic representation of a cross-section through a fibre reinforced matrix. The diagram shows several possible local failure events occurring before fracture of the composite. At some distance ahead of the crack, which has started to travel through the section, the fibres are intact. In the high-stress region near the crack tip, fibres may debond from the matrix (eg. fibre 1). This rupture of chemical bonds at the interface uses up energy from the stressed system. Sufficient stress may be transferred to a fibre (eg. fibre 2) to enable the fibre to be ultimately fractured (as in fibre 4). When total debonding occurs, the strain energy in the debonded length of the fibre is lost to the material and is dissipated as heat. A totally debonded fibre can then be pulled out from the matrix and considerable energy lost from the system in the form of frictional energy (eg. fibre 3). It is also possible for a fibre to be left intact as the crack propagates. The process is called crack bridging.

From this simplistic approach we are immediately made aware of the importance of fibre to matrix bond strength, frictional stress opposing pull out, tensile strength of the fibre, fibre length and fibre content.

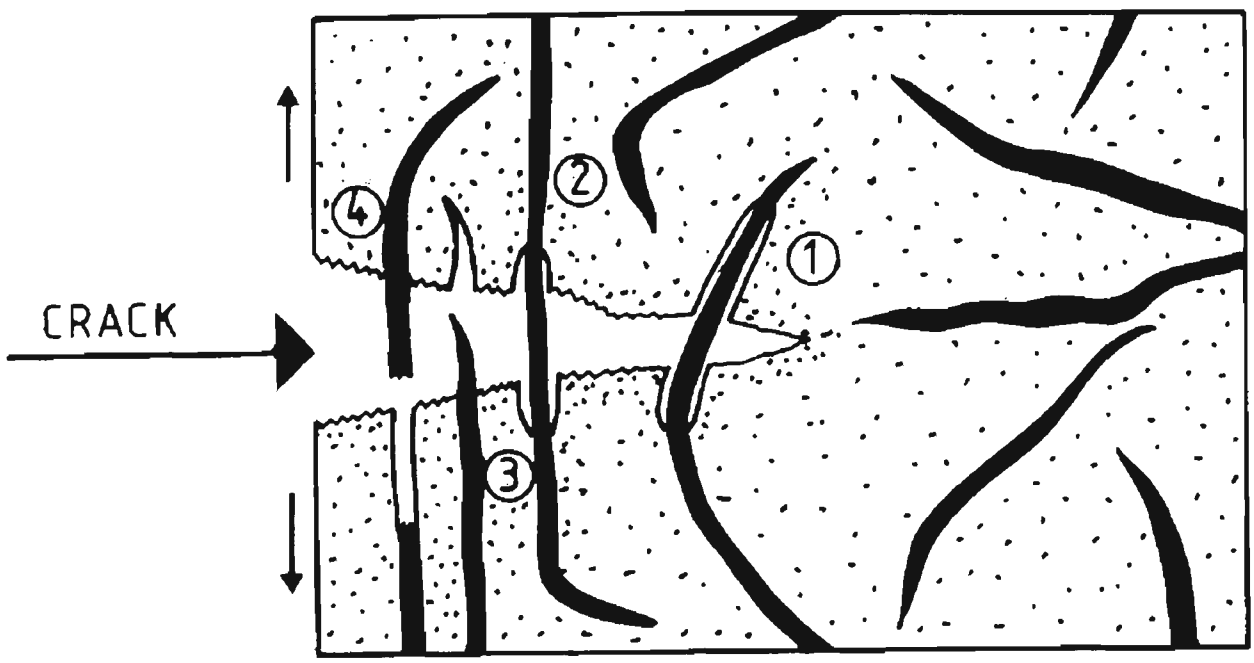

Fig. 2.1. Schematic representation of crack travelling through a fibre reinforced matrix

2.1 Strength and Toughness

2.1.1 Mixture rule for strength

The law of mixtures for aligned continuous fibrous composites may be modified to predict the strength of natural fibre reinforced cements (Mai, 1978). It will be assumed that at failure the fibres are not broken but are pulled out of the cement matrix. Thus the stress in the fibre (σ_f) is given by:

$$\sigma_f = 2\tau (l/d) \qquad (2.1)$$

where τ is the fibre-matrix interfacial bond strength, l and d are the length and diameter of the fibre. To account for the random distribution of the discontinuous short fibres, Romualdi and Mandel (1964) suggest that the effective fibre volume fraction is 41% of the nominal volume fraction. It is therefore possible to rewrite the ultimate tensile strength (σ_t) equation for the natural fibre reinforced cement as:

$$\sigma_t = \sigma_m v_m + 0.41 \sigma_f v_f \qquad (2.2)$$

Equation (2.2) is reduced to equation (2.3) by substituting equation (2.1) for σ_f:

$$\sigma_c = \sigma_m v_m + 0.82\tau v_f(l/d) \qquad (2.3)$$

In equation (2.3), σ_m is the tensile strength of the un-reinforced cement mortar matrix and v_m, v_f are the volume fractions of the matrix and the fibre respectively. Equation (2.3) may be extended to predict the modulus of rupture (σ_b) of fibre cement since in general we have $\sigma_b = \alpha\sigma_c$ and $\sigma_{mb} = \beta\sigma_m$, where α, β are constants which can be determined from experiments and σ_{mb} is the modulus of rupture of the cement mortar matrix in bending. Thus,

$$\sigma_b = [\alpha/\beta]\ \sigma_{mb}v_m + 0.82(\alpha\tau)v_f(l/d) \qquad (2.4)$$

$\alpha\tau$ may be regarded as the fibre-matrix interfacial bond stress in flexure.

Equations (2.3) and (2.4) are first given by Swamy and Mangat (1974) for steel fibre reinforced concrete. Although they have not experimentally proven the validity of the ultimate tensile strength as predicated by equation (2.3) they have however shown that equation (2.4) is valid for the prediction of ultimate flexural strength of concrete reinforced with randomly distributed short discontinuous steel fibres.

The values of α, β, l/d, σ_m, σ_{mb} and τ are suggested to be 2.96, 2.81, 135, 9.71 MPa, 27.27 MPa and 0.35 ~ 0.45 MPa, respectively. Andonian and Mai (1979) attempted the first theoretical analysis of the strength properties of WFRC composite using the mixture rule for random fibre-cement using equations 2.3 and 2.4. The calculations gave close predictions for the experimental results they obtained. While the theory predicts a continuous increase in bending strength with increasing fibre mass fraction, the experimental results show no strength improvement beyond 8% fibre mass fraction. This

is probably a consequence of the relatively large void fractions at larger fibre mass fraction, which cause further reductions in the interfacial bond strength and matrix strength due to poor compaction.

2.1.2 The ACK theory

In the case of fibres reinforced cement and gypsum plaster, however, the matrix is brittle and fails at a strain very much lower than the failure strain of the fibres, and it is widely accepted that the tensile strength depends on the fibre contribution alone and equation (2.3) is given simply by:

$$\sigma_t = 0.82\tau v_f(l/d) \qquad (2.5)$$

While there is considerable experimental support for mixture rule predicting the tensile strength and flexural strength of fibre reinforced brittle matrices although it is difficult to see a mechanism that would allow a matrix contribution to the tensile strength once the matrix has failed.

Aveston *et al.* (1971) have defined the salient points on the tensile stress / strain curve for composites such as glass reinforced cement where the fibre is more extensive than the cement, and there are sufficient fibres to support the extra load when the matrix cracks (The "ACK theory"). Tensile stress / strain curves predicted by the ACK model are shown in Fig. 2.2 (full lines). For an " ideal " composite there is an initial elastic response after which the matrix cracks and continues to crack at constant stress and increasing composite strain. The multiple cracking process continues until the distance between cracks is too small to allow transfer of sufficient load from fibre to matrix to crack it further. Thereafter, further increase in load is taken by the fibres alone, and they extend and slip

relative to the matrix until they break or pull-out. Provided the volume traction is sufficient to allow this multiple cracking process to occur, the strength of the composite depends on the fibres alone and Equation 2.5 applies.

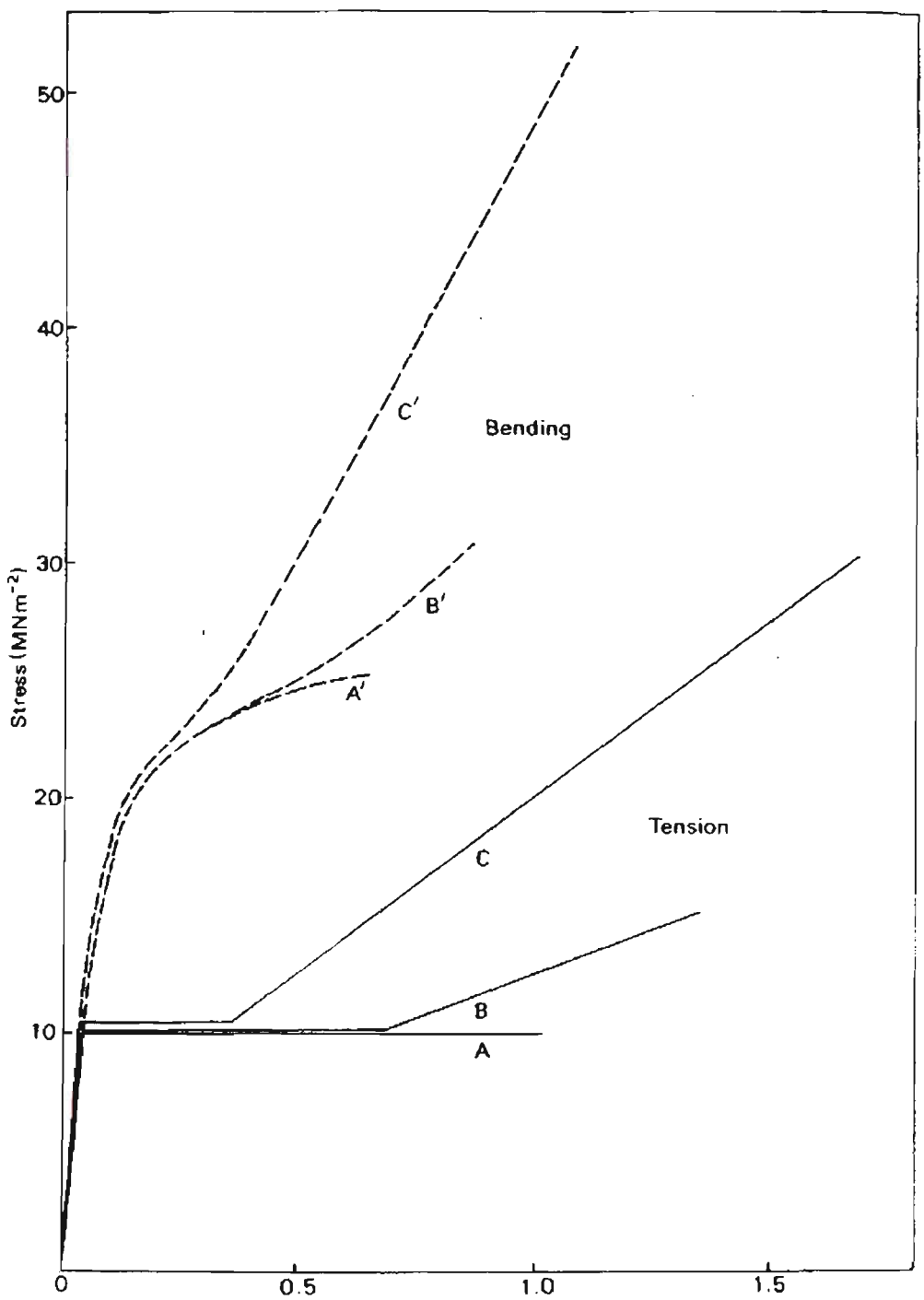

Fig. 2.2. Tensile stress strain curves for fibre reinforced brittle matrices predicted by the ACK theory (full line), and the bending response calculated from them (broken lines) (Laws, 1983)

In bending, while the beam behaves in a linear elastic manner the neutral axis is in the centre of the beam and the nominal stress given by simple beam theory is equal to the

actual stress in the beam. When the tensile stress in the surface of the beam exceeds the elastic limit it can no longer increase linearly with increasing strain but will follow the tensile stress / strain response of the material; and the nominal stress calculated from simple beam theory is no longer equal to the actual stress in the beam (Fig. 2.3).

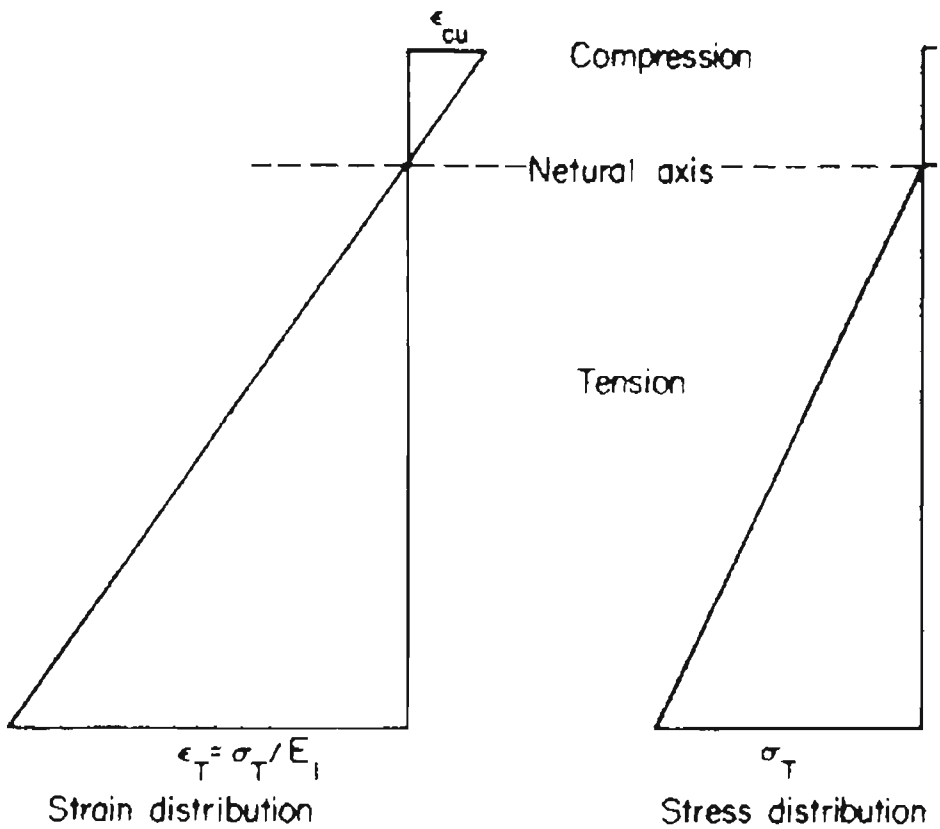

Fig. 2.3. Theoretical model applicable to low modulus fibre-reinforced cement composite at flexural failure (Swift, 1979)

The bending curves calculated from the tensile curves in Fig. 2.2, are also shown in Fig. 2.2 (broken lines). At the critical volume fibre fraction (v_{min}) the fibres support the stress after the matrix has cracked in tension, and there is a long multiple cracking region (curve A). Over this region the bending moment continues to rise (curve A') and the ratio of nominal bending strength or " modulus of rupture" (MOR) to ultimate tensile strength (UTS) is high. As the volume fibre fraction increases, the composite becomes more stiff and the MOR / UTS ratio decreases. At high volume fractions the tensile curve

approaches that of the fibres alone and the MOR / UTS ratio approaches unity. Thus a relationship between bending strength and fibre volume fraction having the appearance of a mixture rule can arise although the tensile strength / volume fibre fraction relationship depends on the fibre contribution alone (Laws, 1983).

Swift and Smith (1979) suggest that direct tensile strength cannot be significantly improved by low modulus fibres within the limits of strain acceptable in a tensile member, whereas for flexural strength it is theoretically possible to obtain a large increase resulting from the inclusion of low modulus fibres in the composite. They have theoretically explained and demonstrated empirically the improvement of flexural strength for sisal slivers reinforced cement.

2.1.3 Basics of fracture mechanics

When the tensile strength of a brittle material is reached in a structure, cracking will occur. The study of the conditions around and in front of a crack tip is called fracture mechanics. Fracture mechanics was first studied for brittle materials such as glass. The first applications to concrete appear to have been made by Neville (1959).

The application of fracture mechanics to concrete structures has provided new ways of understanding and modeling phenomena which could be treated empirically before. Fracture mechanics refers to the analysis of the fracture of materials by the rapid growth of pre-existing flaws or cracks. Such rapid (or even catastrophic) crack growth may occur when a system requires sufficient stored energy that, during crack extension, the system releases more energy than it absorbs. Fracture of this type (often referred to as fast

fracture) can be predicted in terms of energy criterion (Romualdi, 1963; Bazant, 1985; Bentur, 1990).

If we consider an elastic system containing a crack and subjected to external loads, the total energy in the system, U, is

$$U = (-W_i + U_E) + U_S \tag{2.6}$$

Where, $-W_i$, U_E and U_S are the work due to the applied loads, strain energy stored in the system and surface energy absorbed for the creation of new crack surfaces, respectively.

A crack will propagate when $dU/dc < 0$, where dc is the increase in the crack length. Using this theory, one can derive the Griffith equation, which gives the theoretical fracture strength for brittle, linearly elastic materials,

$$\sigma_{1st} = (2E\gamma_S/\pi c)^{1/2} \tag{2.7}$$

Where, σ_{1st}, c and γ_S are the stress at first crack strain, one half of crack length and the surface energy of the material. This is the basic equation of linear elastic fracture mechanics (LEFM).

If we define a parameter $G_C = 2\gamma_S$ = critical strain energy release rate, then the equation (2.7) becomes,

$$\sigma_{1st}(\pi c)^{1/2} = (E\, G_C)^{1/2} \tag{2.8}$$

That is, fracture will occur when, in a stressed material, the crack reaches a critical size (or when in a material containing a crack of some given size, the stress reaches a critical value).

Alternatively, we may define a parameter $K_C = \sigma_{lsf}(\pi c)^{1/2}$ = critical stress intensity factor.

$$K_C^2 = EG_C \tag{2.9}$$

K_C has the units of $N/m^{3/2}$, and is often referred to as the fracture toughness (not to be confused with the term "toughness", which is used to refer to the area under the load-deflection or stress-strain curve).

The LEFM parameters, G_C and K_C, are one-parameter descriptions of the stress and displacement fields in the vicinity of a crack tip. In much of the early work on the applications of fracture mechanics to cement and concrete, it was assumed that they provided an adequate failure criterion. However, later research showed that even for those relatively brittle materials. LEFM could only be applied to extremely large sections (*eg.*, mass concrete structures, such as large gams). For more ordinary cross-sectional dimensions, non-linear fracture mechanics parameters provide a much better description of the fracture process.

Fibres enhance the strength and, more particularly, the toughness of brittle matrices by providing a crack arrest mechanism (see also section 2.1.6). Therefore, fracture mechanics concepts have also been applied to model fibre reinforced cement composites. Mindess (19) has reviewed the difficulties in modelling cement composites based on the fracture mechanics approach. LEFM might be adequate to predict the effects of the fibres on first cracking. However, to account for the post-cracking behaviour (which is responsible for the enhanced toughness of fibre-cement composites), it is essential to resort to elastic-plastic or non-linear fracture mechanics. A measure of toughness (*ie.*, the energy absorbed during fracture) can be obtained from the area under the stress-strain curve in tension. The fracture mechanics concepts which could provide a more precise measure of toughness of

fibre reinforced cement composites include the crack mouth opening displacement (CMOD), R-curve analysis, the fictitious crack model (FCM), and various other treatments, all of which model (either implicitly or explicitly) a zone of discontinuous cracking, or process zone, ahead of the advancing crack. These approaches provide fracture parameters which are, at least, dependent on the fibre content, whereas the LEFM parameters (G_C or K_C) are most often insensitive to fibre content. It might be added here that, while the J integral has often been used to describe these systems, theoretically it cannot be applied to composite systems such as fibre reinforced concrete, where there is substantial stress relaxation in microcracked region in the vicinity of the crack tip.

In the investigation of the fibre-crack interactions using fracture mechanics concepts, the crack suppression, stabilisation and fibre-matrix de-bonding, three distinct issues must be considered.

2.1.4 Fibre critical fracture length

The fibre critical fracture length is defined as twice the length of fibre embedment which will cause fibre failure during pull-out. The fibre critical fracture length, l_c can be calculated from equation (2.9), assuming fibre strength (σ_{fu}), fibre diameter (d) and the shear stress (τ_s) developed at the interface are all uniform.

$$l_c = \sigma_{fu}\, d / 2\tau_s \qquad (2.9)$$

Andonian *et al.* (1979) calculated the critical fracture length of *P.radiata* fibres in WFRC composites to be between 18 and 23 mm, and as the measured length of the fibre is about 3.5 mm, fibre fracture was not possible. Davies (1981) and Coutts (1982b) had observed

fibre fracture during WFRC composite failure and concluded that the "apparent fibre critical fracture length" must be less than 3.5 mm.

The conflicting reports on the predominance of fracture or pull-out of wood fibres from a cement matrix prompted by Morrissey and Coutts (1985) to study a model system consisting of sisal slivers embedded in cement and protruding from one end of the cement matrix. About 200 such samples were tested under tension. It was found that the slivers did not behave in the manner predicted for uniform cylindrical fibres. After a break of the elastic bond between the fibre and the matrix, the fibre started to pull out. The force resisting pull-out was not proportional to the length of embedment but was dominated by the highest local resistances present due to the fibre morphology. As pull-out proceeded, anchor spots developed and the force required rose or fell in an apparently random manner. When the anchorage was too strong to be dislodged by the maximum force the fibre could carry, tensile fracture of the fibre occurred (Fig. 2.4).

There was a "critical fracture length" of embedment, which for the sisal slivers was approximately 30 mm. When the embedment was shorter, fibre tended to be pulled out, (Fig. 2.4) and when it was longer they tended to break. This "critical fracture length " is not the length for which a uniformly distributed frictional stress reaches its critical values under the maximum sustainable tensile load, as is commonly assumed (Equation 2.9), but it corresponds to the length around which the probability of a local strong anchorage becomes high.

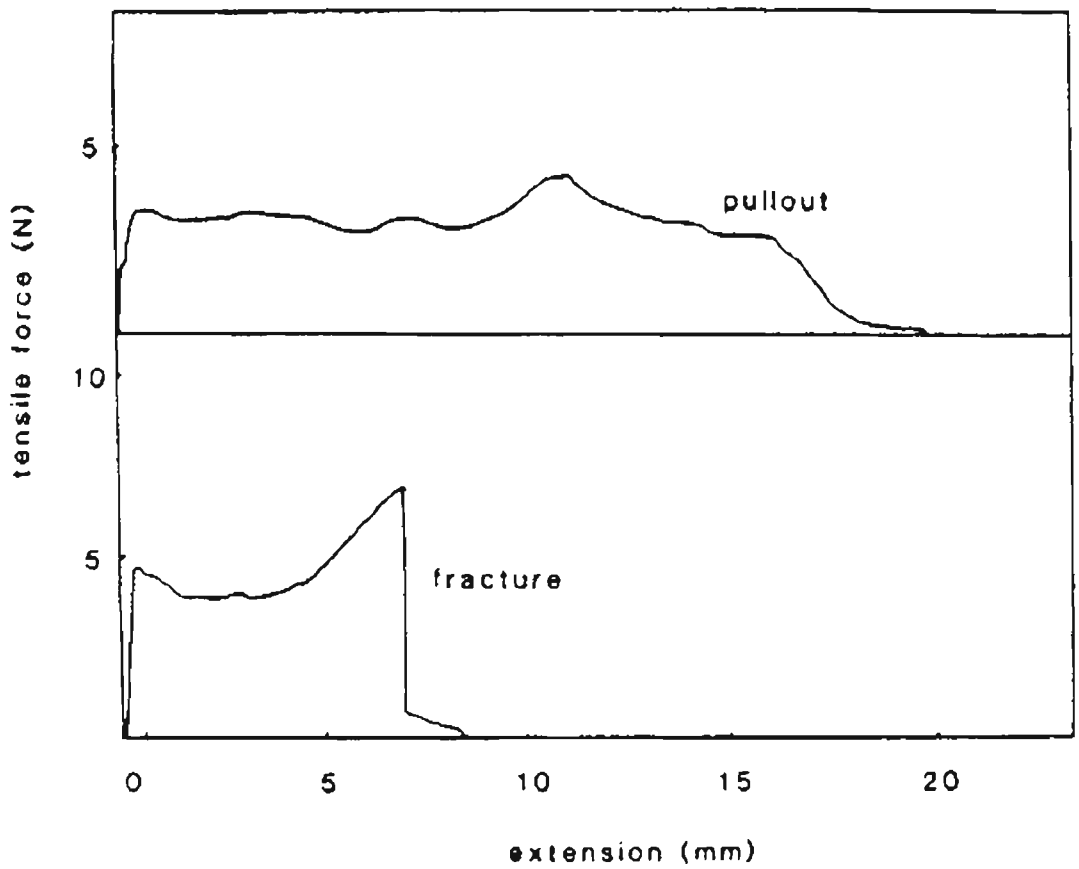

Fig. 2.4. Tensile load-extension curves for different failure modes of sisal silvers embedded in cement (Morrissey, 1985).

The "critical fracture length" of 30 mm for sisal slivers corresponds to an aspect ratio of 110 ± 50, which is comparable with the aspect ratio of *P. radiata* fibres. This would support the observation that *P. radiata* wood pulp fibre cement composites frequently experience fibre fracture during failure.

2.1.5 Fibre aspect ratio

Equation 2.4 and numerous studies of steel fibre and glass fibre reinforced cements and concretes have indicated that the major factors affecting flexural strength are the fibre volume and aspect ratio of the fibre (l / d). Higher values of either lead to higher values of flexural strength (Hannant, 1978).

Without considering the theoretical equations leading to the above conclusions, it is interesting to note that in cellulose fibre reinforced cement composites an increase of fibre volume (not surprisingly) leads to increased flexural strength up to about 8% by mass of fibre, at which stage efficient packing of the fibres becomes difficult and strength starts to drop (Table 2.1).

Table 2.1 Effect of aspect ratio and fibre content (Coutts, 1983c, 84a, 85, 87a)

	Flexural strength (MPa)				Fracture toughness (kJ/m^2)			
	P. radiata		*NZ flax*		*P. radiata*		*NZ flax*	
Fibre % by mass	RH†	Wet	RH	Wet	RH	Wet	RH	Wet
2*	15.6	11.5	17.4	12.8	0.31	0.37	0.19	0.24
4*	16.6	11.7	20.5	16.9	0.57	0.88	0.34	0.71
6*	19.9	12.4	21.4	16.6	1.15	1.81	0.48	0.94
8*	23.1	14.8	23.2	18.0	1.86	3.15	0.84	1.74
10*	21.3	12.9	23.4	17.3	1.92	2.97	1.19	2.15
12*	21.7	10.5	17.7	/	2.09	2.88	2.59	/
	P. radiata		*E. regnans*		*P. radiata*		*E. regnans*	
2**	14.9	10.1	10.6	8.6	0.41	0.64	0.25	0.33
4**	20.2	11.9	14.2	10.5	0.64	1.52	0.51	1.00
6**	25.1	13.3	20.9	10.4	1.40	3.72	1.06	1.61
8**	30.3	14.8	20.3	8.4	1.93	4.51	1.37	1.49
10**	29.2	13.1	20.1	9.6	2.28	4.60	1.46	1.83
12**	27.6	10.4	20.6	9.3	2.25	3.60	1.68	1.79

* Autoclaved mortar ** Air-cured cement
†50 ± 5% RH, 22 ± 2°C

With respect to the aspect ratio of the fibres it has been noted by Coutts and Warden (1985) that air-cured WFRC samples reinforced with softwood fibres (l/d = 80 - 100) when compared to similar samples reinforced with hardwood fibres (l/d = 50 -60) (Coutts, 1987a) displayed higher flexural strength at the same fibre content by mass (Table 2.1). Conflicting with this observation is the fact that autoclaved samples of mortars reinforced with softwood fibres (Coutts, 1984a) and New Zealand flax (l/d = 200) (Coutts, 1983c) show very similar flexural strength, but, more importantly, lower fracture toughness (see 2.1.3). The reason that NZ flax composites do not produce better mechanical properties is related to the fact that the fibres are weaker and the most of the

fibres are broken (Page, 1985), and so σ_{fu} is a limiting factor and not l/d. When tested wet, the longer NZ flax fibres produce stronger samples than the short *P. radiata* fibres. The hydrogen bonds between fibres or between fibre and matrix are destroyed (by insertion of water molecules between the bridging hydroxyl groups) (see 2.2.3), so more flax fibres (long fibre) can be loaded up to failure. In keeping with this, we find the very short *E. regnans* fibre reinforced materials are weak when tested wet or at RH test conditions (50 ± 5% relative humidity, 22 ± 2°C) as short fibre is pulled out and cannot be loaded to failure.

2.1.6 Fracture toughness

As discussed in section 2.1.3, that the post-cracking ductility imparted to the composite by fibre addition can be considerable. Gordon (1976) states: "The worst sin in an engineering material is not lack of strength or lack of stiffness, desirable as these properties are, but lack of toughness, that is to say, lack of resistance to the propagation of cracks". The origin of fracture toughness in WFRC composites was claimed to come mainly from fibre pull-out (85% - 90%) (Andonian, 1979), although later studies by these same workers (Mai, 1983) considered fibre fracture must be of importance in agreement with other researchers in the field (Davies, 1981; Coutts, 1982b; Fördos, 1986).

A generalised theory has been proposed by Martson et al. (1974) where the specific fracture resistance (R) is given by the sum of; the toughness due to fibre pull-out ($R_{p.o.}$), redistribution of stresses (R_{re}) and fracture of surfaces (R_s). If the fibres are pulled out rather than fractured, then it seems appropriate to neglect stress redistribution R_{re} as a component contributing to the specific work of fracture (R). Thus

$$R = R_{p.o.} + R_s \qquad (2.10)$$

Where, $R_{p.o.} = 0.41 v_f l^2 \tau / 12d$ (2.11)

$$Rs = v_f R_f + v_m R_m + (0.41 v_f) l R_{if} / d \quad (2.12)$$

R_m, R_f and R_{if} are the fracture energies of the cement mortar matrix, fibre and fibre-matrix interface respectively. Normally, $R_f << R_m$ and $R_{if} \cong R_m$ (Martson, 1974) so that equation (2.12) is simplified to

$$R_s \cong 0.41 v_f l R_m / d + v_m R_m \quad (2.13)$$

The specific work of fracture R is thus given by

$$R = 0.41 v_f l^2 \tau / 12d + [v_m + 0.41 v_f l / d] R_m \quad (2.14)$$

Toughness measurements can be conducted in several ways (Hibbert, 1982); impact testers such as Charpy or Izod which involve stored energy in a pendulum, calculating the area under stress - strain curves, and the use of fracture mechanics involving stress intensity or similar parameters, or more practical tests such as dropped balls or weights, etc. Some of these techniques are more suited to particular composites, but all have limitations which render elusive the well-defined material properties useful to engineers and material scientists.

Mindess and Bentur (1982) studied the fracture of WFRC products and found that saturated samples were weaker and more compliant than air-dry specimens. It was found that the wet samples were completely notch-insensitive, while the air-dry specimens may be slightly notch-sensitive. Notch sensitivity is a requirement for the application of linear elastic fracture mechanics to cement composites, and so these workers concluded that LEFM could not be used for WFRC materials.

Mai and Hakeem (1984a, b) have studied the slow crack growth of WFRC composites for both dry and wet conditions, using a double-cantilever beam system. The results were analysed using K-solutions, and compliance measurements within the framework of LEFM. It was concluded that LEFM concepts can be used for WFRC products.

The use of LEFM requires a linear elastic homogeneous matrix. The introduction of fibres into matrix, to achieve ductility, must by their very nature result in a heterogeneous material with a non-linear stress-strain curve after matrix cracking.

The fracture toughness results given in Table 2.1 for a range of NFRC composites have all been obtained by the method of measuring the area bounded by the load / deflection curve. As the materials are non-linear and non-elastic, this approach is useful in that the total recorded energy will include the contributions of the work of fracture of the matrix, the debonding and frictional slipping of the reinforcement, and any strain energy released by fibre fracture.

For fibre cements and concretes the matrix work of fracture is virtually that of the unreinforced cement or concrete and is less than 50 J/m^2. Therefore it is assumed that any improvement in composite toughness will depend on whether the fibres bridging the crack are able to support the load previously carried by the matrix, and on whether the fibres break or pull out of the matrix.

Fibre pull-out is the most common mode of failure for steel and glass fibre cements and concretes. This is because, to incorporate sufficient fibre into the formulations without

fibre tangling, the fibre aspect ratio cannot be high enough to exceed the critical fracture fibre length.

It has been recorded that wood fibres can be loaded into the matrix material with fibre volumes in excess of v_{min} and can be fractured during failure, hence the "apparent critical fracture length" is exceeded. This behaviour does result in relatively high levels of fracture toughness (Table 2.1) but does not lend itself to a conventional form of theoretical analysis. The explanation of such properties is discussed in terms of fracture mechanics and bonding in section 2.2.

Recently, Hughes and Hannant (1985) have studied the reinforcement of Griffith flaws in WFRC in an attempt to explain their behaviour, and concluded that stresses developed are significantly higher than would be expected using the mixture rule theory.

It is hoped that as commercial production of NFRC becomes a global activity, more interest will be shown in the theoretical explanation of how this system works and hence in developing ways to optimise its use in design.

2.2 Bonding and microstructure of NFRC composites

As well as the physical properties of the fibre and the matrix, a major factor which controls the performance of a composite is the type and arrangement of bonds linking the two materials.

According to composite theory, the interface (that is the region of intimate contact between fibre and matrix) plays the dual role of transmitting the stress between the two phases and of increasing the fracture energy of the composite by deflecting cracks and delocalising stress at the crack tip.

The interfacial bond itself can be physical or chemical in nature, or a combination of both. Too strong a bond between fibre and matrix results in a brittle material which has strength whereas a weak bond results in a tough material lacking strength. The mechanical performance of a NFRC composite is therefore directly related to the nature and properties of the fibre-matrix interface.

2.2.1 Chemical bonding

The chemistry and morphology of the matrix material has been well documented (Lea, 1976) and will not be considered further, apart from stating that cement is strongly alkaline (pH > 12) and presents metal hydroxy groups at its surface, such as -Ca-OH, -Si-OH, -Al-OH and -Fe-OH (due to hydration and hydrolysis of silicates, aluminates and to a lesser extent ferrites of calcium that are present in the cement matrix). Cellulose fibres such as wood fibres contain covalent hydroxyl groups, -C-OH, either phenolic (from residual lignin) or alcoholic (from the cellulose component) and carboxylic groups, O=Ç-OH, due to oxidation of end groups. Hydrogen bonding and / or hydroxide bridges may play a major role in the bonding of NFRC composites. The chemical implications will not be considered quantitatively; it suffices to say that hydrogen bonds may form between fibres or between fibres and matrix.

Coutts and co-workers (Coutts, 1979a, b) considered the possibility of using chemical pretreatments of the wood fibres to enhance the bond between fibre and cement. The most acceptable theory for the development of coupling agents is the "chemical bonding theory", which suggests that the coupling agent acts as a link between fibre and matrix by the formation of a chain of covalent chemical bonds (Fig. 2.5).

Although small improvements in composite mechanical performance have been recorded from the use of pretreated fibres, the costs of such operations are currently considered prohibitive. The initial hypothesis that some form of coupling agent or additive was needed to achieve bonding stemmed from the general belief that the bond between wood fibre and cement would be weak. It has subsequently been suggested that this may not be the case (Coutts, 1984b) and that the presence of hydroxyl groups on the surface of both wood and cement may be sufficient for adequate chemical interaction to take place via hydrogen bonding.

2.2.2 Mechanical bonding

As well as the chemical bonding aspects of a fibre, the physical bonding potential must also be considered. Much of the theoretical data on fibre reinforcement is based on smooth cylindrical fibres of uniform sharp and dimensions (Section 2.1).

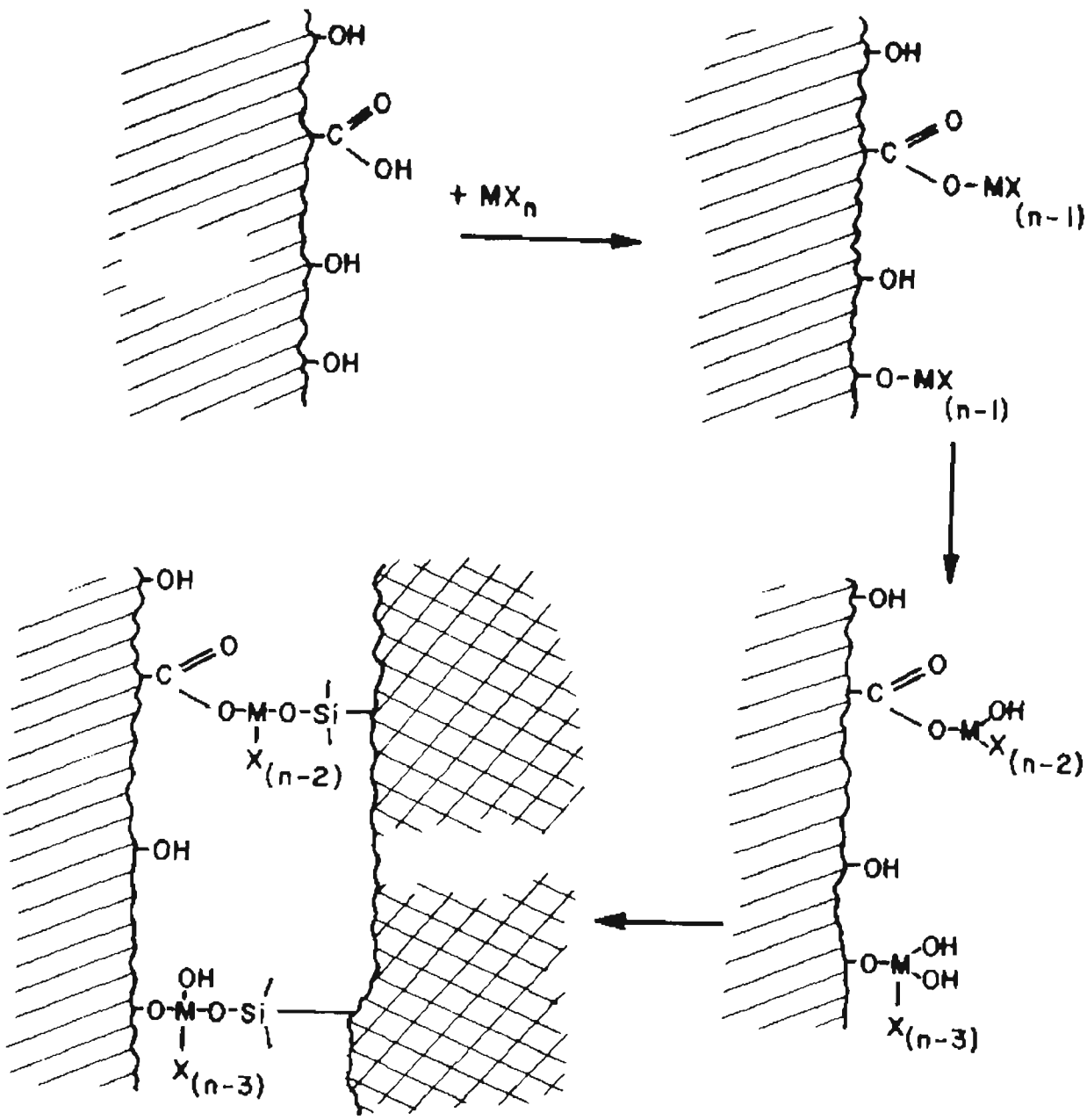

Fig. 2.5. Possible coupling mechanism between wood fibre and cement matrix (Coutts, 1979a).

Maximum fracture energy is often achieved if frictional energy is dissipated via fibre pull-out. Wood fibres are relatively long compared to their diameter and hence have aspect ratios of say 60 - 200 (depending on whether they are hardwood or softwood, early wood or late wood fibres), but, more importantly, the fibres are hollow and can be collapsed to ribbons and at the same time develop a helical twist along their length (like a cork-screw). When fibres such as these are used to reinforce a brittle matrix, an asymmetrical process will be taking place during pull-out (after interfacial debonding has occurred). In classical

pull-out of straight fibres (glass, steel, etc.) the forces are symmetrically distributed around the fibres; in the case of the contorted wood fibres, the leading edge of the helical fibre can experience considerable compressive stresses resulting in a ploughing action which can damage fibre or matrix resulting in increased fracture surfaces and hence increased fracture energy. Similar observations were recorded by Bentur *et al.* (1985a,b) when they studied the physical processes taking place during the pull-out of steel fibres, of various geometry, from Portland cement.

Coutts and co-workers (Coutts, 1982a, 1984a) reported that mechanical refining or beating of wood fibres resulted in improved flexural strength from autoclaved mortars reinforced with such fibres. This phenomenon can again be discussed in terms of mechanical bonding in that the external surface of the fibre is "unwound" and the fibrils so formed offer extra anchoring points by which the fibres can accept stresses from the matrix and so become more effective reinforcing elements. Although refining the fibre can assist in mechanical bonding, its main value is in improving the drainage rate and solids retention in the commercial Hatschek process (Anon, 1981; Coutts, 1982a).

Michell and Freischmidt (1990) studied curly fibre in the cement and silica matrix at the aim of improving fibre and matrix bonding. The use of curly fibres in reinforced cement and silica sheets gave sheets with improved wet interlaminate bond strengths, relative to sheets prepared from conventionally treated fibres but had little effect on the values of modulus of rupture and fracture toughness.

Mechanical bonding has been discussed in other systems, such as asbestos fibre cement, in which Akers and Garrett (1983a) showed that asbestos can be fiberized like wood fibres,

steel fibres can be kinked, as we have noted in the work of Bentur *et al.* (1985a), or polypropylene can be fibrillated into a netlike form, as reported by Hannant and co-workers (1978).

2.2.3 The effect of moisture on bonding

The effect of moisture on the strength properties of NFRC composites is of importance. Earlier in WFRC research, variation in test conditions produced variations in test results, and it was evident that standard conditions must be adopted. Coutts and co-workers reported that as the moisture content of a WFRC test specimen was increased, the flexural strength of a given sample decreased while the fracture toughness increased (Coutts, 1982a). These observations have been confirmed by Mai *et al.* (1983). This behaviour is common to both air-cured (Coutts, 1985) and autoclaved (Coutts, 1982a, 1984b) samples containing a range of cellulosic fibres, be they softwood (Andonian, 1979; Coutts, 1983a), hardwood (Coutts, 1987a), abaca (Coutts, 1987b) or NZ flax (Coutts, 1983c).

Coutts and Kightly (Coutts, 1982b, 1984b) suggested that these observations could be supported by the hypothesis that hydrogen bonds and / or hydroxide bridges play a major role in the bonding and hence in the mechanical performance of WFRC composites.

Wet or dry, a wood fibre has about the same tensile strength, but its stiffness is considerably lower when wet. Thus a dried WFRC composite has stiff, highly contorted fibres (see section 2.2.4) locked into a rigid cement matrix which could be bonded at the interface by a large number of hydrogen bonds or hydroxyl bridged sites. This system when stressed can transfer the stress from the matrix to the fibres via the many interfacial

bonds, and hence sufficient stress may be passed on to the fibre, after the matrix has cracked, to cause the reinforcing fibre to fracture under tensile load.

On the other hand, in a moist sample, the hydrogen bonds between fibres or between fibre and matrix are destroyed (by insertion of water molecules between the bridging hydroxyl groups); and, at the same time, the cellulosic fibres are swollen by water absorption and have become less stiff. Under stress this system allows the fibres to move relative to each other or to the matrix. However, due to the pressure of swelling and the highly contorted assemblage of fibres, considerable frictional forces are developed. If the forces are effective over sufficient length of a fibre, they can result in the fibres being loaded to failure; however, the number of fibres that pull out without failure is higher than when the sample is dry, and hence the observed values of fracture toughness for wet samples is higher than for dry samples.

2.2.4 Microstructure of NFRC composites

The examination of wood-cement composites by scanning electron microscope (SEM) is relatively recent. Ahn and Moslemi (1980) examined the manner in which wood particles and Portland cement bonded together in cement particle-boards and decided that mechanical interlocking plays a significant role. This interlocking is due to crystal growth during the hydration of cement. A similar crystal interlocking effect has been reported for wood fibre reinforced plaster products (Coutts, 1987c).

NFRC composites have been examined by different research groups with strong interests in the micromechanical behaviour of such composites. In a number of these SEM studies of NFRC materials, the microstructural features of the fracture surfaces of the broken

composites were described, with emphasis being placed on the surface of the fibre and how it had failed (Davies, 1981; Coutts, 1982b; Mai, 1983; Pavithran, 1987; Akers, 1989). More recently, interest has turned to the matrix, and in particular, the interfacial region adjacent to the fibre.

In a study of air-cured WFRC composites, containing Kraft or CTMP (chemithermeomechanical pulp) fibres, Davies *et al.* (1981) proposed that the fracture surfaces indicated that both fibre fracture and fibre pull-out were taking place during loading to failure under ambient conditions. Andonian *et al.*(1979), studying autoclaved WFRC samples, stated that fibre pull-out was the main source of fracture toughness (80-90%) even though the samples had been dried in an oven at 116°C for 24 h. Mindess and Bentur (1982) also considered that a pull-out mechanism was the main process taking place when commercially produced WFRC samples containing 20% by mass of wood fibres were tested. These workers used only photographic evidence, stating that at 18 X magnification (their highest), lack of focus restricted their observations due to the unevenness of the surface. As the diameter of a wood fibre is approximately 15 - 40 μm, the observation of end fractures would be difficult by optical means.

SEM studies by Coutts and Kightly (1982b, 1984b), using autoclaved WFRC samples, complement the earlier findings with air-cured samples, namely, that failure takes place by mechanisms of fibre fracture and fibre pull-out. More importantly, this study showed that the relative importance of these mechanisms is very dependent upon the moisture content of the test sample (Coutts, 1984a). Figure 2.6, 2.7 and 2.8 show the SEMs of fracture surfaces of WFRC samples obtained from the same formulation when tested dry, wet and at 50 ± 5% RH. Most of the fibres protruding from the fracture surface of a sample

preconditioned in an oven at 105°C for 24 h before testing have fractured ends (Fig. 2.6). By contrast, Figure 2.7 shows the fracture surface of a sample which has been preconditioned in water for 48 h before testing and indicates considerable fibre pull-out, although fibre fracture is still very obvious (Fig. 2.7). Figure 2.8 shows the fracture surface of the sample conditioned at near ambient conditions (50 ± 5% RH and 22 ± 2°C). Both fibre fracture and fibre pull-out appear to have taken place. Higher magnification (Fig. 2.8b) shows that considerable damage to some fibres of relatively short length has taken place while other fibres have been pulled out of the matrix. Mai *et al.*(1983), on re-examining their earlier work, are now of the opinion that fibre pull-out is not the major source of toughness for WFRC, but indeed that fibre fracture is of considerable importance.

The significance of moisture content, as evidenced in the above SEM work, has been adequately described in the section 2.2.3.

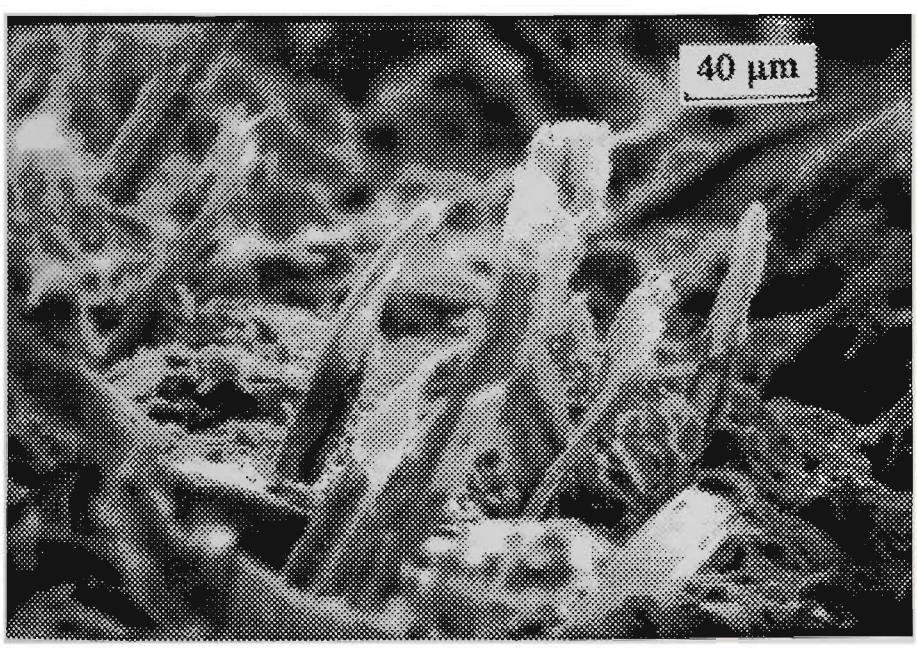

Fig. 2.6. SEM showing fracture surface of WFRC preconditioned at 100 - 105°C for 24h (Coutts, 1984b)

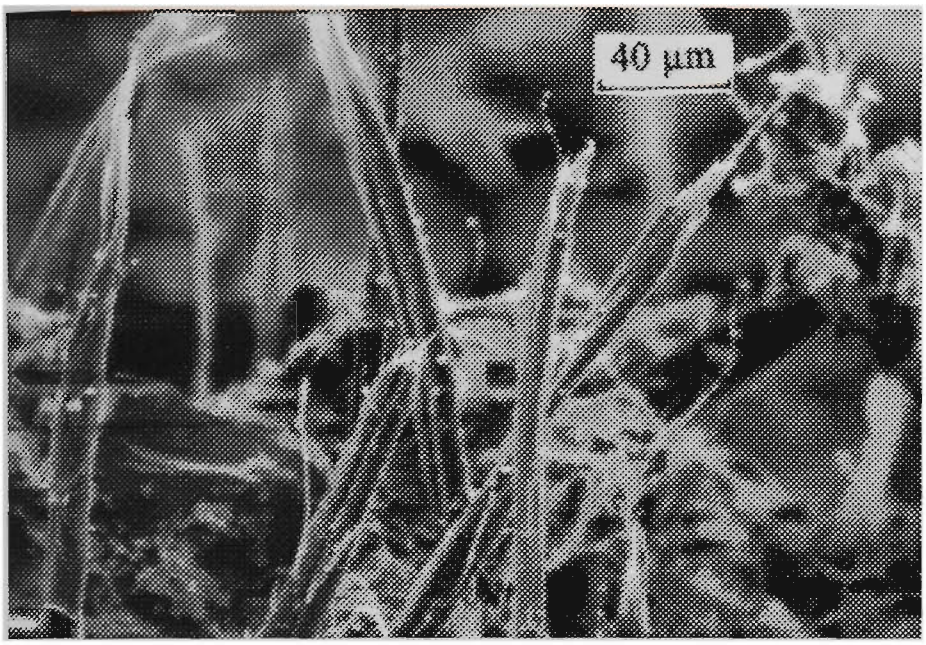

Fig. 2.7. SEM showing fracture surface of WFRC preconditioned by soaking in water for 48h (Coutts, 1984b)

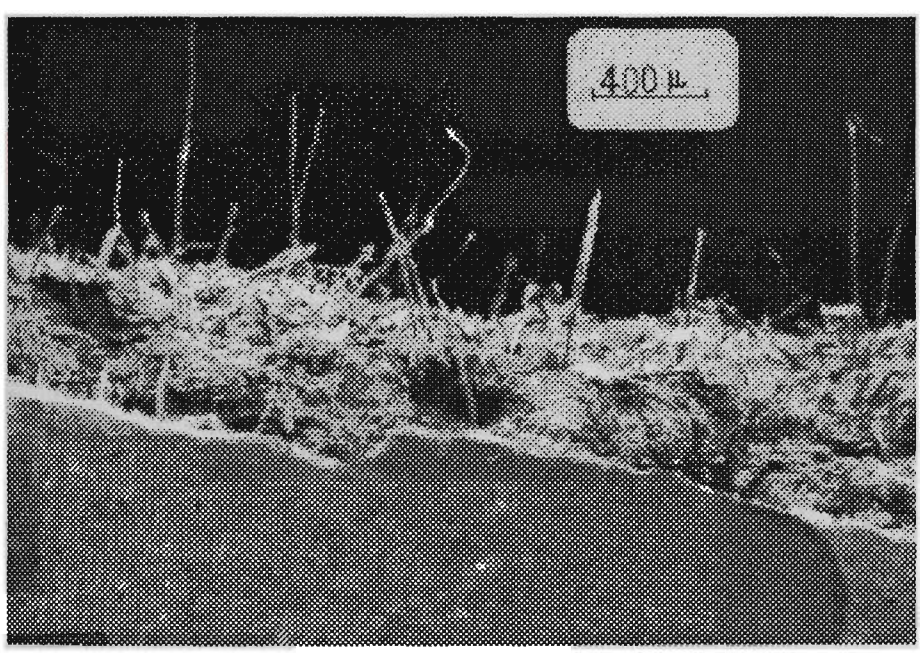

Fig. 2.8a. SEM showing fracture surface of WFRC preconditioned at 50 ± 5% relative humidity and 22 ± 2°C (Coutts, 1984b)

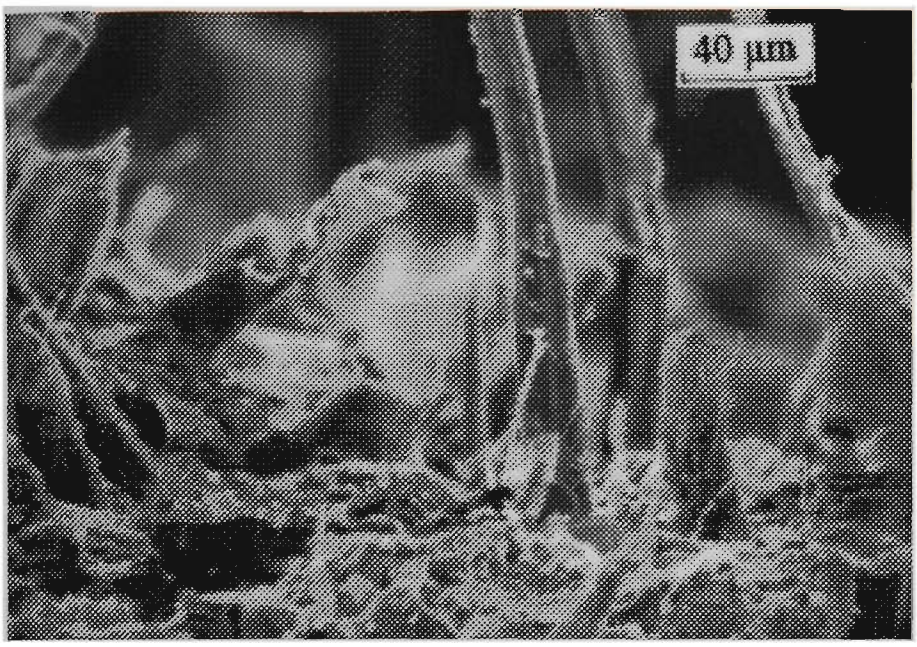

Fig. 2.8b. As (a) but higher magnification

Most of the foregoing studies have related to the condition of the fibre after failure and rather little has been said of the most important aspect of the composite - the interfacial region. In studies of air-cured cement matrices reinforced with aggregate particles (Barnes, 1978), steel (Pinchin, 1978; Bentur, 1985a,b) or glass fibres (Stucke, 1976; Singh, 1981) detailed reference has been made to an interfacial region with structure and porosity different to the bulk cement paste. This interfacial zone has, in several cases, been considered to be 50 - 100 µm thick.

In the cases of aggregate, steel or glass fibre there is a general similarity of behaviour. At the surface of the reinforcement a dense layer, consisting largely of hexagonal lamellar crystals of portlandite (calcium hydroxide or CH), forms and replicates the topography of the reinforcement over much of its surface area. At discontinuity in this dense layer, inclusions of hydration products such as calcium silicate hydrate (CSH) gel, ettringite and large crystals of CH occur.

With increasing distance from the reinforcement surface, the proportion of CH decreases, and a relatively porous layer (in comparison to the bulk of the matrix material) forms for some distance before gradually becoming more dense and merging into the bulk matrix.

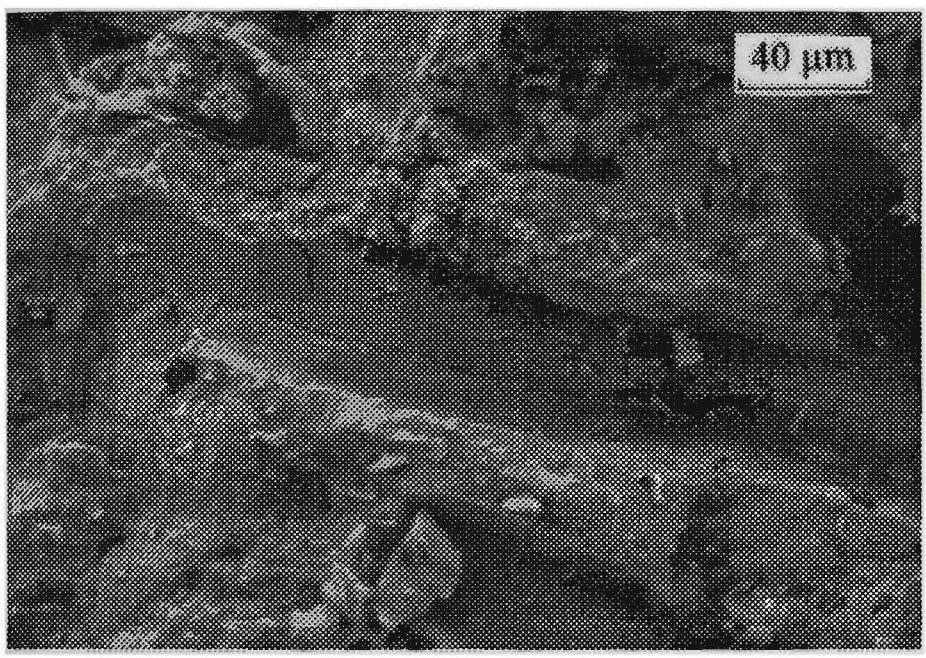

Fig. 2.9. SEM showing cement surface at interface contains dense matrix with some discontinuities (Coutts, 1987d).

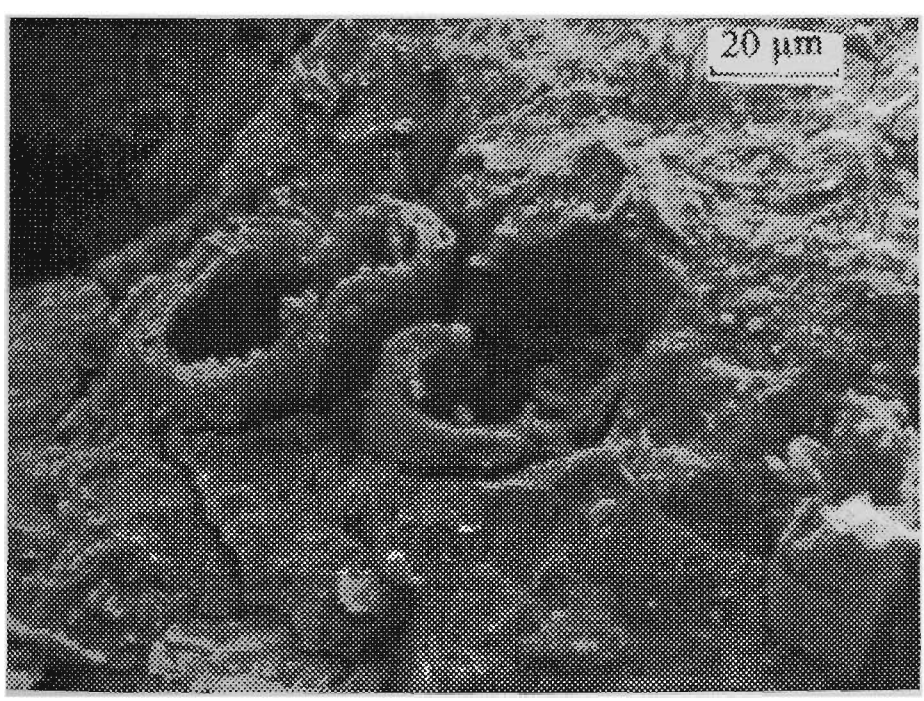

Fig 2.10. SEM shows fractured fibres with dense material from bulk of matrix up to fibre wall (Coutts, 1987d)

An SEM study of WFRC composites which focused on the region of the wood fibre-matrix interface (Coutts, 1987d,e), revealed that the general characteristics associated with the interfaces of other fibre reinforced cement composites were not observed. Coutts noted that the interface between wood fibre and matrix is usually dense and replicates the surface of the fibre, although discontinuities do occur, especially at higher fibre loadings when normal packing of the fibres becomes more difficult (Fig. 2.9). Again, if one looks at the intimate area of contact between fibre and matrix (Fig. 2.10), no obvious zone of weak matrix material exists at the interface. It was stated that during fabrication of WFRC composites the samples were placed under pressure to compact the structure and reduce the water - cement ratio. It was hypothesised that, unlike the glass or steel fibres, which are not compressible, the hollow wood fibres are compressed, and after the pressure is removed the water level immediately adjacent to the fibre is lowered even further as the sponge-like fibre draws excess water back into itself. This would reduce the occurrence of voids at the interface of fibre and matrix particles, because less free water would be present, and produce a homogenous dense matrix material from the bulk of the material right up to the point of contact with the fibre.

Another example of a dense interface is found in the work of Akers and Garrett (1983b) who studied the microstructure and failure mode of the fibre-matrix interfacial region in asbestos fibre reinforced air-cured cement. Also reported was a mutual interlocking of asbestos fibres with the cement hydrate, as had been observed with WFRC. But, more importantly, energy dispersive X-ray measurements showed no evidence of a calcium hydroxide enriched zone adjacent to the interface, as had been reported for other fibre cement materials. The study proposed that fiberizing asbestos fibres increased the fibre surface available for water absorption, which helped to distribute the water-filled voids in

the interfacial region. Thus the nature of the fibre-cement interface need not be the same as is described for materials such as glass, but appears to depend on the characteristics of the reinforcing fibre.

In the case of TMP pulp reinforced composites (Coutts, 1986 and Chapter 9), the high temperature of the autoclave (> 160°C) coupled with the alkali conditions of the matrix material (pH > 12) results in the extraction of polysaccharides and wood acids from the reinforcing fibre (see section 3.3), which, in the case of Kraft pulp have been removed during pulp preparation. These extractives contribute to "poisoning" of the cement (Singh, 1979; Thomas, 1983) and coating on the fibre surface thus causing poor interfacial bonds. Hence, TMP pulp-cement composites have low strength and poor fracture toughness (see section 1.1.4).

Chapter Three:

The Nature of Plant Fibres

3.1 Fibre Classification

Natural plant fibre can be classified into wood fibre and non-wood plant fibre, which consists of:

(a) Seed hairs;

(b) Bast fibres - fibres derived from the bark of dicotyledons, which include herbaceous plants, shrubs, and trees;

(c) Leaf fibres - fibres derived from the vascular bundles of very long leaves of some monocotyledons. Leaf fibres are also known as " hard " fibres because they are more lignified than bast fibres;

(d) Grass fibres - these are another group of monocotyledonous fibres where the entire stem together with the leaves are pulped and used in papermaking. Such pulps are composed not only of fibres, but of other cellular elements as well.

Woods are grouped into two main classes, namely *softwood* and *hardwood*. The names *hardwood* and *softwood* are based mainly on timbers produced in the northern hemisphere. More correctly, the softwoods or coniferous types (pines, firs and spruces) are called *gymnosperms*, and the hardwoods (gums, oaks and ashes) are all classed as *angiosperms*. The hardwood-softwood grouping has little meaning on a world scale, as some hardwoods (such as balsa wood) are extremely soft.

3.2 Structure of Natural Plant Fibres

Natural plant fibres can be derived from wood, bast, seed leaf and grass, needless to say there are far too many plants to describe in this thesis. There are four types of tissue in natural plants. They are parenchyma, fibre, tracheid and vessel (or pore). However some plants do not contain all of these four tissues, such that softwood does not contain vessels (or pores). Each type of tissue serves one or more special functions: the parenchyma cell conduct and store food and water; the fibre's main role is to provide mechanical support; tracheids and vessels (or pores) conduct water and dissolved mineral salts from the roots to the leaves as well as providing mechanical support.

In general terms it can be said that the fibre cells, which are themselves composite structures, have a cylindrical or ribbon-like shape made up of different layers with a hollow centre (lumen). The fibre can vary in length from less than 1 mm to greater than 250 mm. The diameter can be from less than 5 μm to greater than 80 μm (see section 3.5.1.1). When the fibres are separated from each other they can collapse flat, or the lumen may remain open, if the walls of the fibres are thick. Fibres may develop a spiral twist along their length, which can be of significance during fibre composite failure.

Plant fibres contain cellulose, a natural polymer, as the main material of reinforcement. In a simplified description we can say chains of molecular cellulose are held together by hydrogen bonds to form microfibrils, which in turn are held together by amorphous hemicellulose and form fibrils. The fibrils are assembled in various layers of differing thickness at different angles of orientation to build up the internal structure of the fibre, the main reinforcing element of interest to our research (see Table 3.5). Figure 3.1 and 3.2 show schematic structure of wood fibres and bamboo fibre, respectively.

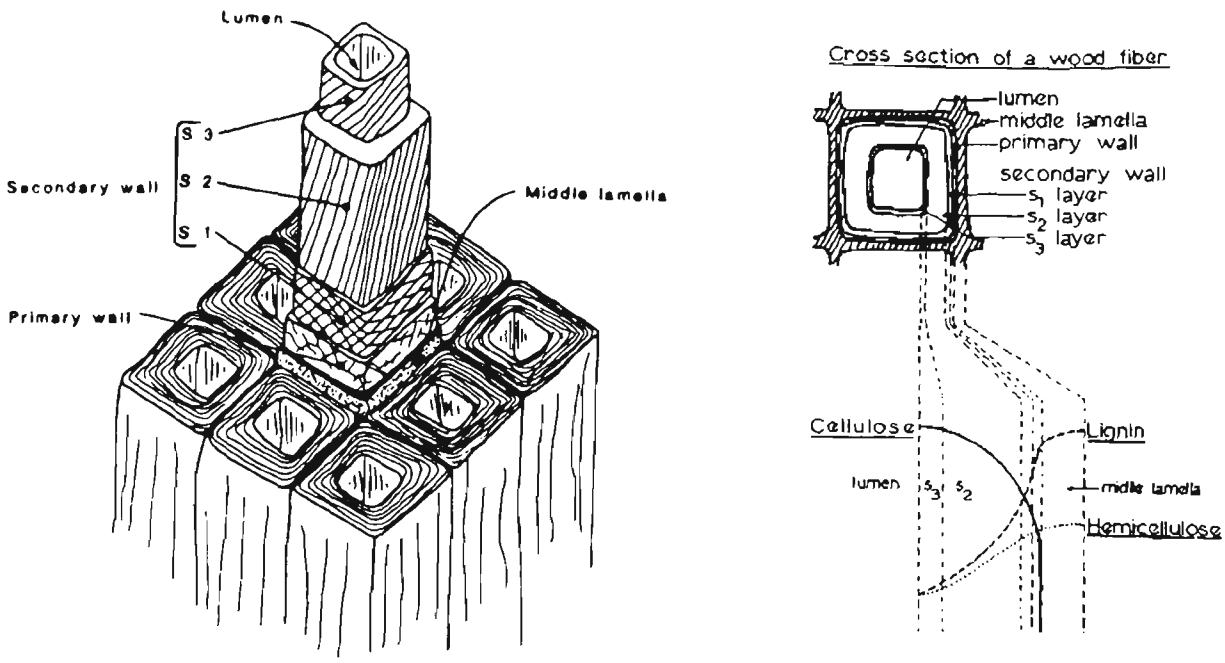

Fig. 3.1. The structure of wood fibre

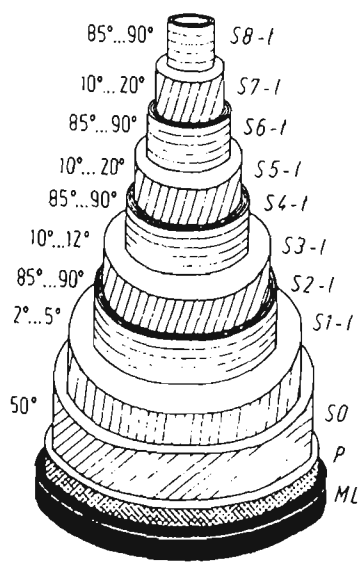

Fig. 3.2. The structure of bamboo fibre

Fibres are cemented together in the plant by lignin, a complex natural organic adhesive. In much of the work on plant fibres, we are really discussing aggregates of fibres, which are often incorrectly called " fibres". As we will see, when we discuss durability, much of the

lack of performance of certain " fibres" can be attributed to breakdown of these aggregates, due to the alkalinity of the matrix materials, and not to the fibre cell itself.

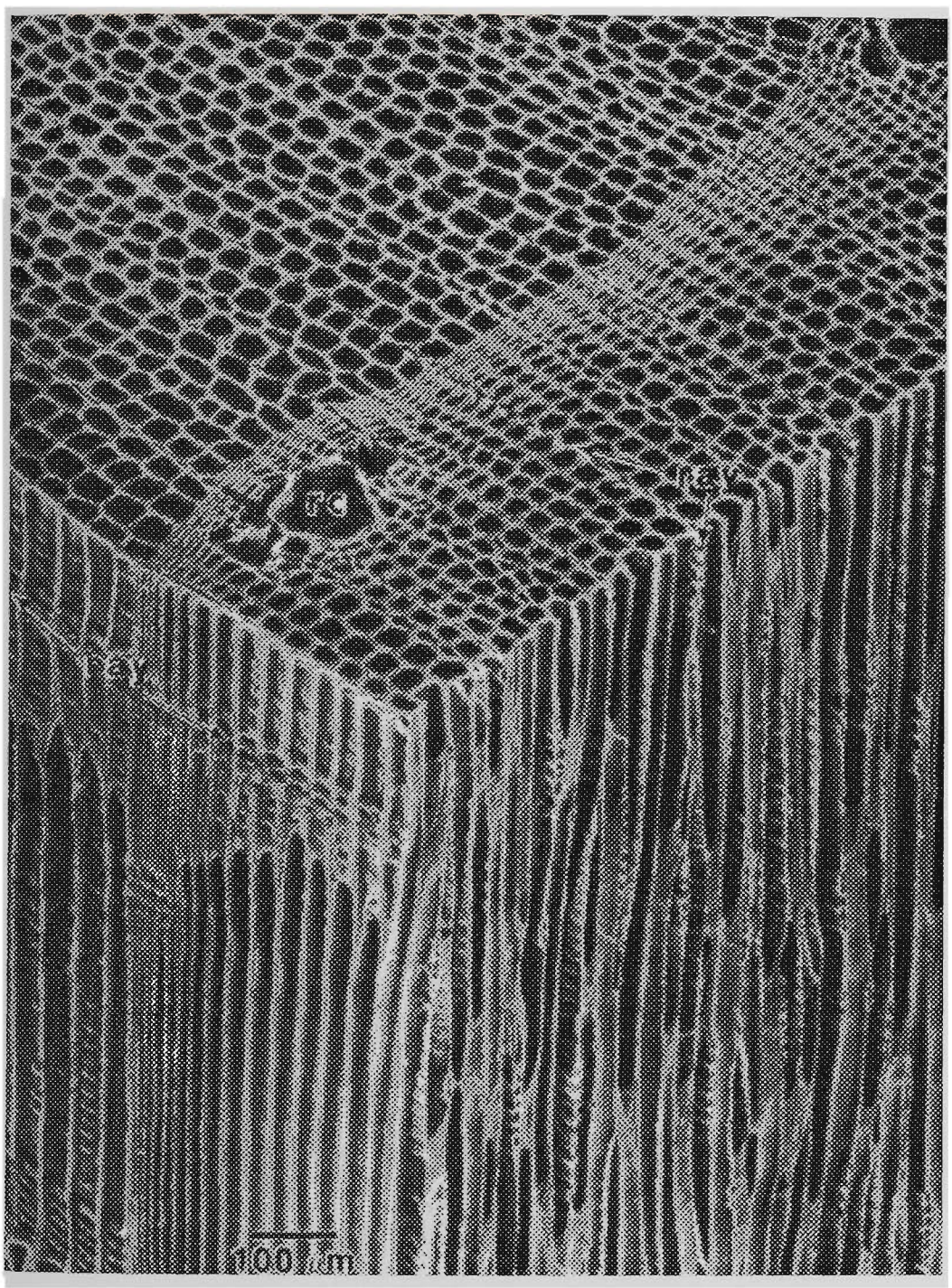

Fig. 3.3. Scanning electron micrograph of a cube of eastern white pine microtomed on three surfaces. Note the arrangement of longitudinal tracheids (tr) in radial files and the structure of the rays. In this species resin canals (rc) are rather prominent features (Côté, 1980).

The increase in size of a natural plant varies with the seasons and climatic conditions. Such as in trees, during late autumn and winter, there is little or no growth, but growth is at a maximum in spring. The spring or early wood usually has wood cells with larger diameter and thinner walls than those cells formed during periods of slow growth - summer wood or late wood (Fig. 3.3).

As will be seen later in this thesis, fibres with thick cell walls have different papermaking characteristics compared with thinner - walled fibres. Consequently, the ratio of later wood fibres to early wood fibres can have a big influence on the ability of a given wood fibre source to act as a reinforcing element.

3.3 Chemical Composition and Durability

The chemical components which constitute most of the mass of natural plants are cellulose, hemicellulose and lignin (Table 3.1). Cellulose is made up of thousands of glucose molecular units joined in long chains, and represents 40 - 45 % of plant. It contains 44.4 % carbon, 6.2 % hydrogen and 49.4 % oxygen, and is relatively unaffected by alkalis and dilute acids.

Lignin occurs in the form of very complex chain polymer of phenolic building blocks consisting of about 65 % carbon, 6 % hydrogen and 29 % oxygen, and is virtually impossible to dissolve without first breaking it down into simpler substances. Lignin comprises between 22 and 30 % of plant and varies in chemical composition and amount in different species, being somewhat more abundant in softwoods than in hardwoods.

Table 3.1 Chemical compositions of natural plant fibres (Atchison, 1983)

Type of fibre	Cross & Bevan cellulose	Alpha cellulose	Lignin	Pentosans	Ash	Silica*
Stalk-straw-rice	43-49	28-36	12-16	23-28	15-20	9-14
-wheat	49-54	29-35	16-21	26-32	4.5-9	3-7
-barley	47-48	31-34	14-15	24-29	5-7	3-6
-oat	44-53	31-37	16-19	27-38	6-8	4-6.5
-rye	50-54	33-35	16-19	27-30	2-5	0.5-4
-cane-sugar	49-62	32-44	19-24	27-32	1.5-5	0.7-3.5
-bamboo	57-66	26-43	21-31	15-26	1.7-4.8	0.69
-grasses-esparto	50-54	33-38	17-19	27-32	6-8	
-sabai	54.5		22.0	23.9	6.0	
-reeds-phragmites communis	57.0	44.75	22.8	20.0	2.9	2.0
Bast -seed flax tow	75.9-79.2	45.1-68.5	10.1-14.5	6.0-17.4	2.3-4.7	
-seed flax	47	34	23	25	5	
-kenaf	47-57	31-39	14.5-18.7	22-22.7	1.7-5.0	
-jute (1)	57-58		21-26	18-21	0.5-1.8	
Leaf -abaca (Manila)	78	60.8	8.8	17.3	1.1	
-sisal (agave)	55-73	43-56	7.6-9.2	21.3-24	0.6-1.1	
Seed hull -cotton linters		80-85				
Woods -coniferous	53-62	40-45	26-34	7-14	<1	
-deciduous	54-61	38-49	23-30	19-26	<1	

* The content of tramp (as distinguished from inherent) inorganics may be greatly increased by mechanical harvesting.

Hemicelluloses act as a matrix for the cellulose microfibrils, are of relatively low molecular weight and are soluble in alkalis. They occur as about 15 to 30 % of the weight of the natural plant.

All the chemical components of natural plants are formed from the sugars produced in the leaves by photosynthesis. The water obtained from the roots is combined with carbon dioxide from the atmosphere, by complex biochemical processes dependent on chlorophyll which is able to absorb from sunlight the energy needed, to form simple sugars. When these sugars are conveyed from the leaves to the plant cells, they are changed into the much more complex cellulose, hemicellulose and lignin.

In addition to cellulose, hemicellulose and lignin, natural plant also contains a wide range (5 - 30 %) of compounds known as extractives which can be extracted by solvents. These materials, contribute to colour, density, durability, flammability and moisture absorbency, and include polyphenols, oils, fats, gums, waxes, resins and starches.

These extractives plus hemicellulose and lignin can inhibit or retardate the hydration and strength development of Portland cement. In the case of TMP pulp reinforced composites, the high temperature of autoclave (> 160 °C) coupled with the alkali conditions of the matrix material (pH > 12) results in fibre chemical degradation and extraction of the fibres. These extractives contribute to "poisoning" of the cement and coating on the fibre surface. Hence, TMP pulp-cement composites have poor mechanical performance (see section 1.1.4 and 2.2.4).

Natural fibres are susceptible, under certain conditions, to biological degradation, the action of such decay resulting in, amongst other things, strength loss. However, in the fibre cement system, chemical degradation would be more significant than biological degradation due to high alkali environment (pH > 12). Sisal fibre (aggregate form) has acceptable initial strength. While, immersed in lime solution, the fibre suffers a 74% reduction in strength due to significant chemical degradation (Nilsson, 1975). Thus composites reinforced with aggregate fibres have poor durability due to fibre degradation (Lola, 1986).

3.4 Preparation of Natural Plant Fibres

3.4.1 Pulping

Natural fibre can be prepared by means of pulping. As stated, natural plant consists of parenchyma, tracheid, vessel (or pore) and fibre tissues. The fibre cell contains cellulose, hemicellulose and lignin. The purpose of pulping is to separate the fibres from the plant so that they are suitable for papermaking and composite reinforcement.

Wood pulp fibre is the plant fibre of most commercial importance at the present time. There are a number of ways of producing pulp which will be summarized briefly in the following sections.

3.4.1.1 Chemical pulping

Chemical pulps are made by heating the wood chips at high temperatures (about 170 °C) with a solution of chemicals that dissolve most of the lignin which bonds the fibres together in the wood. The chemicals dissolve carbohydrates as well as lignin removing about half of the wood substance, yield is typically 45 - 55 per cent. It is not possible to remove all the lignin during chemical pulping, therefore all unbleached chemical pulps contain some residual lignin and are brown in colour.

The pulp is washed with water to remove pulping chemicals and dissolved wood components. Most modern pulp mills have a chemical recovery system in which these washings are concentrated and then burnt in a specially designed furnace to recover the chemicals for re-use in the pulping process and to generate heat energy.

The Kraft process is used to produce a major portion of the world's chemical pulp. Sodium sulphide and sodium hydroxide are the active ingredients which attack the lignin. The process operates under alkaline conditions, and penetration of the wood is more rapid. It is applicable to all species and so has a wide industrial appeal, but it creates considerable effluent disposal problems and releases an unpleasant odour into the surrounding district.

The soda-anthraquinone (soda-AQ) process uses sodium hydroxide with a small quantity of anthraquinone as pulping chemicals. Pulps by this process are brown in colour and are no easier to bleach than Kraft pulps. In most respects soda-AQ pulps are similar to Kraft pulps. The process offers no advantage with regard to bleachability of the pulps, but the absence of sulfur offers the possibility of using new chemical recovery systems eg, the Direct Alkali Recovery System (DARS). The successful development of DARS may make it possible to produce soda-AQ pulp in economically viable mills of a smaller scale than is currently necessary if the Kraft process is used.

The alkaline conditions cause less harm to the cellulose than the acidic conditions of the sulphite process, since cellulose is more easily hydrolysed by acids than by alkalis.

The use of chemical pulps in WFRC products removes the problem of "extractives" which, interfere with the cure of autoclaved products and fibre - matrix bonding resulting in materials of low performance (Coutts, 1982b).

3.4.1.2 Mechanical pulping

Papermaking pulps can be manufactured by mechanically separating wood into its constituent fibres. Chips are first steamed to soften the lignin and to make the separation

of the fibres easier. The chips are fed into a refiner where they are ground between two plates. Alternatively billets may be fed onto a grinding stone.

After this treatment the freed fibres are screened and pumped to storage. The amount of pulp retained from mechanical processes is usually higher than 95 per cent as only the water soluble material in the wood is removed.

Mechanical pulping in its various forms has been claimed as the pulping method of the future (Kurdin, 1983). This method can solve the problems of limited wood supply, of restricted capital, of environmental restrictions and of efficient utilization of mixed forests. In its expansion from groundwood pulping, mechanical pulping has advanced into the domain of chemical pulping blurring what was once a clear line of distinction - there is now refiner mechanical pulp (RMP), thermomechanical pulp (TMP), chemimechanical pulp and chemithermomechanical pulp (CTMP).

Mechanical pulping is not without its weaknesses. Means must be found to reduce the energy consumption, increase the strength of the pulp and improve light stability. The last factor is important in paper manufacture, but not in the use of the pulp as a reinforcement in cement systems *etc.*

The principle of chemical and mechanical pulping is scheduled briefly in Figure 3.4.

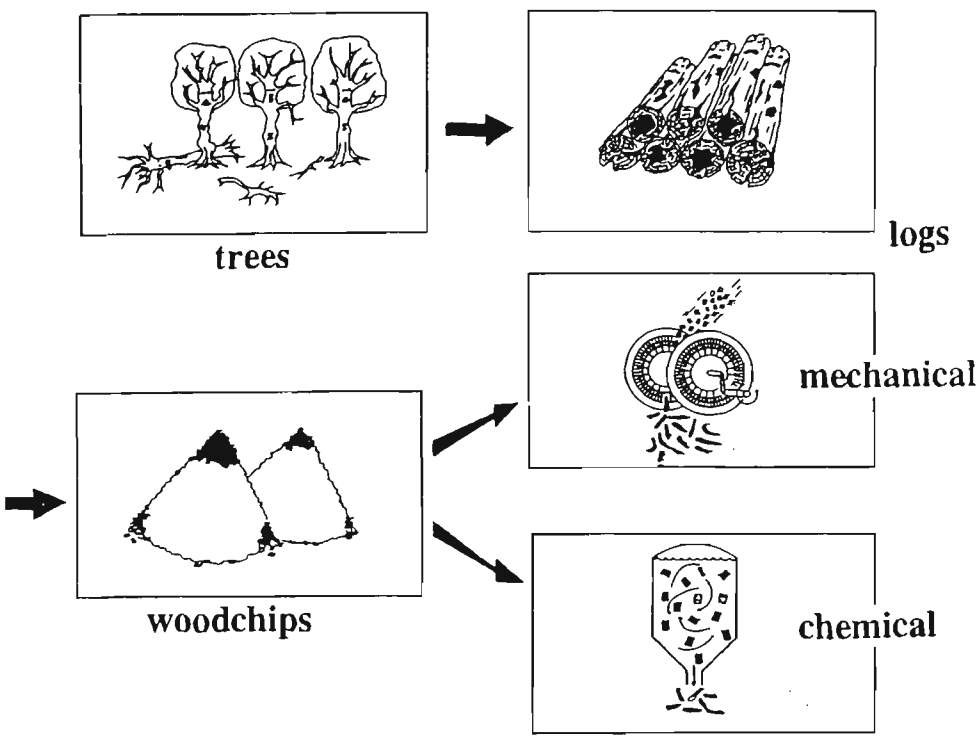

Fig. 3.4. The principle of chemical and mechanical pulping

3.4.1.3 Non-wood pulping

Some non-wood resources provide excellent papermaking fibres. Some bast fibres such as jute, flax and kenaf can be produced by a retting method, which allows bundles of fibres to be freed from cellular tissue surrounding them by the combined action of bacteria and moisture, then the fibrous material is crushed, washed and dried. Decortication, a process used for leaf plants such as sisal and abaca, involves crushing and scraping the leaf to remove cellular tissue, then washing and drying.

Besides these mechanical process, almost all natural plant fibres can be prepared by the Kraft pulping process. In countries where wood is scarce, it is quite common to find cereal straw, bagasse (from sugar cane), bamboo and similar materials being used as a source of papermaking fibre. These fibres have roughly the same quality as hardwood fibres, sometimes a bit less because the fibres have been damaged during harvesting /

processing, but usually adequate as a component of printing and writing papers (Li, 1992). However, there are a few difficulties that inhibit non-wood fibres from being used more often as a source of papermaking fibres (McKenzie, 1992).

The first difficulty is that one can harvest the crop at only one time of the year. This means that a whole year's supply of raw material must be collected and stored within a few weeks. Furthermore, it cannot merely be cut and stored, but must be protected from decay immediately after harvesting. Also, the prospect of a crop failure (for whatever reason) and the subsequent closure of a multimillion dollar manufacturing operation for lack of raw material is enough to deter the average investor.

Many agricultural residues have a high silica content (rice straw and bamboo are notorious in this respect) which dissolves in alkaline pulping liquors and redeposits throughout the pulping plant. The bulkiness of straws and similar materials compared to wood chips can also be a problem, as the productivity of a digester depends on the amount of raw material which can fit into it. This means that the cost of a mill designed to produce straw pulp will probably be much higher than the cost of a wood chip mill capable of producing the same amount of pulp of the same type.

Overall, the cost of producing pulp from non-wood material is high. It can only be justified economically if the pulp is of superior quality, such as is the case with pulp produced from textile or cordage grade fibres. It is significant that in many countries where wood is scarce but agricultural residues are plentiful, it is common to import wood pulp rather than to produce non-wood pulp from materials such as wheat straw. In other countries, straw pulp and the like is only used because of government restrictions on the

importation of wood pulp. It is estimated that China and India produce nearly half of the annual world total of 10 million tonnes of non-wood based paper.

3.4.2 Refining or beating

Refining or beating can be defined as the mechanical treatment of pulp carried out in the presence of water, usually by passing the suspension of pulp fibres through a relatively narrow gap between a revolving rotor and a stationary stator, both of which carry bars or knives aligned more or less across the line of flow of the stock. The term "beating" is usually applied to a batch treatment of pulp suspension, whereas "refining" is used when the stock is passed continuously through one or more refiners in series.

It should be pointed out that the refining of chemical pulp does not produce the same effects as it does on mechanical pulp. Chemical pulps are relatively pure cellulose, with the hydroxyl groups accessible. In mechanical pulps the hydroxyl groups are blocked by the presence of lignin. The refining of mechanical pulp is really a completion of the process of disintegration of fibre bundles down to individual fibres.

Changes observed in fibre structure as a result of the mechanical action on the fibrous material can depend on the type of refiner, the refining conditions used, the fibre type (hardwood or softwood) and the pulp (mechanical or chemical). However the main effects which are observed can be classified into four areas:

(a) Internal fibrillation or delamination

(b) External fibrillation of the fibre surface

(c) Fines formation

(d) Fibre shortening

Internal fibrillation effects (a) are difficult to observe under a microscope, but they can be considered by analogy with a piece of rope. Rope is a helical wrap of strands which themselves are helical wraps of fibres. If one twists a rope in the direction of the helical wrap the rope becomes stiffer; likewise, if the twist is in the opposite direction the rope unwinds (or delaminates) to open up the structure and becomes "floppy"; such is the case with internal fibrillation. The main effect of internal fibrillation is to increase fibre flexibility and swelling. The fibres may also undergo excessive curling and twisting.

External fibrillation (b) is easily observed by scanning electron microscopy (Fig. 3.5). The fibrils or fibrillar lamellae attached to the fibre surface can vary widely in size and sharp (but the process is again like unravelling a piece of rope at its surface).

The last stage (c) of external fibrillation is the peeling off of the fibrils from the fibre surface with the formation of fines. Depending on the forces acting on the fibre during refining, more or less of the fibrils will be removed from the surface of the fibre.

Fibre shortening (d) is the other primary effect attributed to refining. An indication that fibre shortening has occurred is the change observed in particle size distribution, which is a result of the cutting action of the blades or discs in the machinery on the single fibres. Refining plays an important role in producing a large surface area for fibre - to - fibre or fibre - to - matrix (in the case of composites) bonding and, more importantly, can assist in controlling the drainage rates of processing liquids during the fabrication of products. This is one of natural fibre's main advantages compared to synthetic fibres such as glass, steel, *etc.*, in asbestos replacement.

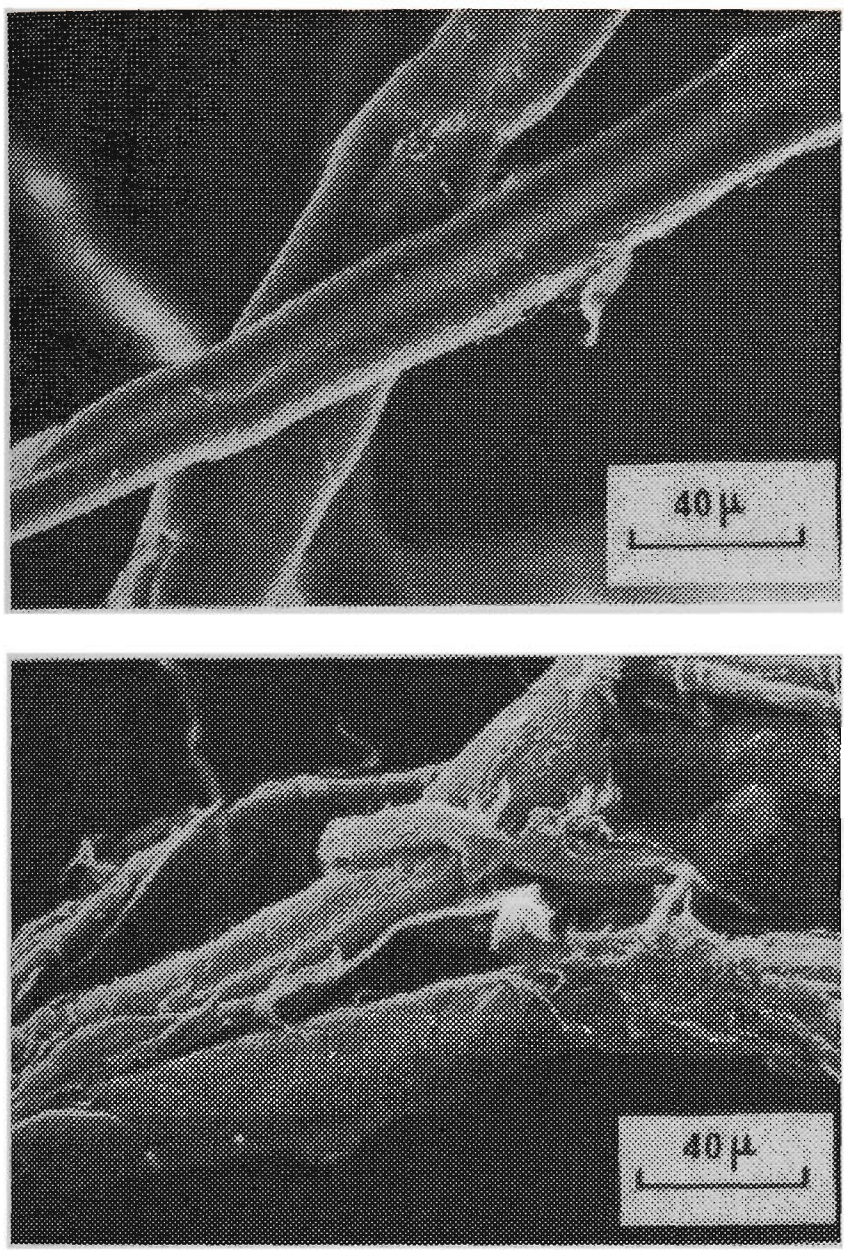

Fig. 3.5. (a, top) Unbeaten fibre of *P.radiata*, compared to (b) externally fibrillated fibre (Coutts, 1982b).

3.5 Properties of Natural Fibres

The significance of the properties of the natural fibres in the cement composite will be studied through out this thesis. The physical and mechanical properties of natural fibres, such as fibre size, morphology, surface charge, strength, *etc.*, play important roles in the manufacturing process of fibre cements.

3.5.1 Physical properties

3.5.1.1. Physical dimensions

The physical dimensions of fibres are exceedingly important in the application of fibres as reinforcement, be it in paper, medium density fibre board or in fibre reinforced cement.

Hardwood fibres are much shorter (av. 1.0 mm) and narrower (av. 20 μm) than softwood fibres (av. length 3.5 mm). With softwoods there is a difference in fibre diameters, between early and late wood (av. diameter 45 μm and 13 μm resp.). Hardwood fibres generally have a higher relative cell wall thickness than do early wood softwood fibres. This implies hardwood fibres are stiffer and have a greater resistance to collapse (see Table 3.2).

Table 3.2 Typical softwood and hardwood fibres physical dimensions (Coutts, 1988).

Fibre property	*P. radiata*	*E. regnans*
Fibre length (mm)	2 - 6, av: 3.5	0.5 - 2.5, av: 1.0
Fibre width (μm)		10 - 23, av: 20
Early wood	45	/
Late wood	13	/
Cell wall thickness (μm)		4
Early wood	3	/
Late wood	5	/
Type of cells (%)		
Fibres	> 90	65
Vessels	/	18
Parenchyma	< 10	17

It can be seen from Table 3.3 and 3.4 that natural fibres have great variation in length, diameter, lumen size and fibre wall thickness. Some of the fibre properties for reinforcing are better than those of wood fibres, for example bast fibres and leaf fibres have much greater length than that of softwood fibres.

Chemical pulping processes reduce natural plant to pulp, which is then composed of individual cells. For wood and most of non-wood pulp, the small percentage of non-fibre cells can be washing away and the pulp contains mainly pure fibre cells. However, the pulps of grasses (cereal straws, sugar cane bagasse, bamboo, *etc.*) contain fibres as well as a great variety of other cells, such as parenchyma, epidermal and vessel segments. These segments and pitted cell act as a filler rather than as a reinforcing material in the cement based composites.

Table 3.3 Some non-wood fibres physical dimensions (Strells, 1967)

Fibres		Length (mm)	Width (μm)	Shape	Lumen size	Wall thickness
Seed Hairs	cotton	10-40 (18)	12-38 (20)	ribbon-like	broad	thick
	coir	0.3-1.0 (0.7)	12-24 (20)	cylindrical	"	"
Bast	flax	9-70 (33)	5-38 (19)	"	fine	"
	hemp	5-55 (25)	10-51 (25)	"	broad	"
	jute	1.5-5 (2)	10-25 (20)	"	"	"
	ramie	60-250 (120)	11-80 (50)	"	"	"
	kenaf	2-6 (5)	14-33 (21)	"	"	"
Leaf	abaca	2-12 (6)	16-32 (24)	"	"	thin
	sisal	0.8-8 (3.3)	8-41 (20)	"	"	thick
	NZ flax	2-15 (7)	5-27 (15)	"	fine	"

Table 3.4 Some grasses pulps physical dimensions (Strells, 1967)

	Fibre		Parenchyma		Vessel	
	L. (mm)	W. (μm)	L. (mm)	W. (μm)	L. (mm)	W. (μm)
cereal straw	0.68-3.12 (1.48)*	6.8-23.8 (13.3)*	0.45	130	1.0	60
	0.8-2.9**	27-34**				
sugar cane	0.8-2.8 (1.7)	10.2-34.1 (20)	0.85	140	1.35	150
bamboo	1.45-4.35 (2.7)	6.8-27.3 (14)	0.25	65	/	100
	2.8-3.26†	20.5-40†				

* thick wall
** thin wall
†pitted fibres

3.5.1.2 Surface potential and surface area.

The Hatschek process is the major manufacturing method for fibre cement products and, as have been shown (section 1.1.2), is very much akin to a papermaking machine. The retention of cement particles by asbestos (or its replacement) and drainage properties of

the sheets during the dewatering stage are paramount importance, hence fibre surface potential and surface area are of considerable significance.

Zeta-potential - the electrokinetic charge on a colloidal particle - has been discussed in many areas of science. When the particle is a wood fibre it has been postulated that the zeta-potential plays a major role in the process of papermaking,. The zeta-potential is a controlling parameter in filler and fines retention, drainage and pulp flow behaviour (Arno, 1974).

Work in the field of paper science (Britt, 1974; Herrington, 1986) shows quite dramatically that zeta-potential is not a measure of surface charge, and cannot be used for comparison of the surface charge of even very similar materials, let alone two materials as distinct as asbestos and wood fibre. In this discussion it suffices to say that in the case of cellulose fibres, the present evidence indicates that dissociation of ionic groups is the main source of the charge. At very high pH values, dissociation of the hydroxyl groups can contribute to the particle charge, but at low pH values the charge must be due to dissociation of carboxyl groups or sulphonic acid groups, depending on the pulp yield and on whether a sulphite or sulphate pulp is used. A cellulose fibre dispersion is negatively charged at all pH values. When cationic hydroxylated metal species, such as occur in cement particles, are present, charge reversal may occur, as has been established in the case of wood fibres in the presence of papermaker's alum (Arno, 1974).

3.5.2 Mechanical properties

The use of natural fibres in paper, paperboard, and fibre reinforced composite materials has created a need for better characterization of fibre mechanical properties.

Unfortunately, researchers often report properties of the fibre when in fact they are studying aggregates of fibres with very different properties to those of the individual fibre. For example, bast and leaf fibres have been widely used in the textile and cordage industries. Tensile strength in the textile industry is defined in terms of tenacity which is the breaking load per unit mass per unit length; for cordage applications, it can be defined as the breaking length which is the length of fibre which can theoretically support its own weight when suspended at one end.

Most of the pioneering work in natural fibre (wood) testing was carried out by researchers from the paper industry, and much of the data on fibre properties interpreted from tests carried out on paper sheets. The information on single fibres is limited, although new techniques in recent times are providing more data as time passes (McKenzie, 1978).

3.5.2.1 Modulus of elasticity and tensile strength

Theoretical calculations suggest the modulus of elasticity of cellulose could be as high as 150 GPa. Experimental values for single fibres vary with change in fibril angle, drying restrains and defects but range between 10 - 100 GPa (Fig.3.6 and 3.7).

Like elastic modulus, the tensile strength of single fibres is dependent on the helical angle of the S_2 layer and the presence of defects. Page and co-workers (Page 1970, 1971; Kim, 1975) have reported a value of 2000MPa for defect-free black spruce fibres with a zero fibril angle; this figure should be considered as a maximum value. A more realistic value would be in the range 500 - 1000 MPa for selected fibre fractions carefully prepared to minimize fibre damage. Jayne (1959) found values for tensile strength between 350 - 1000 MPa.

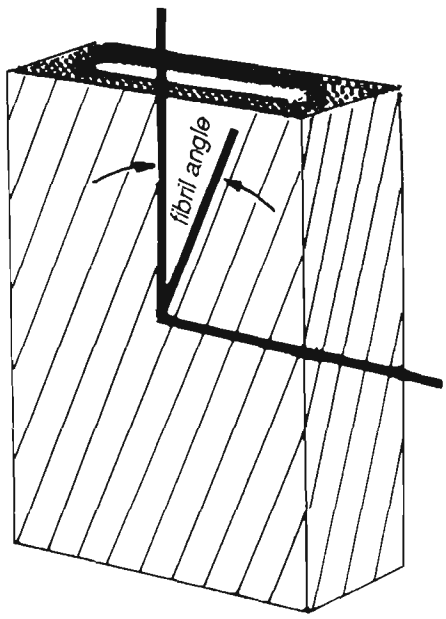

Fig. 3.6. A pulp fibre consists of cellulose fibrils in largely parallel array, embedded in a matrix of lignin and hemicellulose. The majority of the cellulose fibrils form a steep spiral around the axis of the fibre, and it is this structure which gives to the fibre its mechanical strength

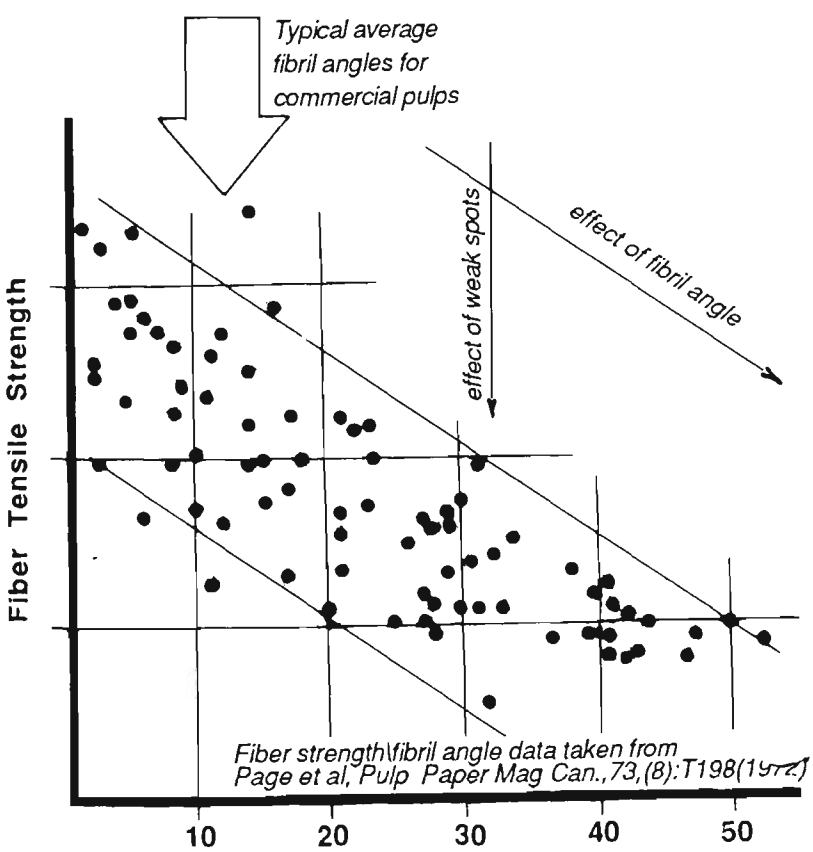

Fig. 3.7. The maximum strength of a pulp fibre is limited by the inherent properties of the cellulose fibril modified by the spiral angle of the fibril about the fibre axis. The actual strength exhibited by the fibre in relation to this limiting strength is then determined by the presence or absence of defects and dislocations which produce "weak spots" where premature failure is induced.

Considerable data from zero-span tensile testing of paper sheets have been equated to tensile values for single fibres. It would appear from all the available evidence that the likely average tensile strength of fibres commercially prepared from reasonable quality wood by normal methods would be not less than 700 MPa. Strengths of 800 - 900 MPa might be anticipated if the wood supply is carefully selected to contain a large proportion of late wood. This is not likely to be readily achieved in Australia or New Zealand where the main softwood species is young *P.radiata* (McKenize, 1978).

The more common fibres, such as abaca, sisal, flax, kenaf and bamboo, range in tensile strength from say 50 - 500 MPa, with densities of about 1.2 -1.5 g/cm^3. The elastic modulus of plant fibres can range between 5 - 100 GPa (Coutts, 1992b). Unfortunately, "fibres" are often characterized as an aggregate of fibres rather than as an individual fibre, thus information is limited in the scientific literature. Table 3.5 lists some fibre structure and strength values, it should be interpreted with care.

Table 3.5 Structure and strength parameters of non-wood fibres (Mukherjee, 1986)

Fibre		Cellulose%	Spiral angle	cell L.(mm)	L / W	UTS (MPa)	Elongation%
Seed	coir	43	45	0.75	35	140	15
	flax	71	10	20	1687	780	2.4
Bast	hemp	78	6.2	23	906	690	1.6
	jute	61	8.0	2.3	110	550	1.5
	remie	83	7.5	154	3500	870	1.2
	banana	65	12	3.3	150	540	3.0
Leaf	sisal	67	20	2.2	100	580	4.3

Pulp fibres strength properties can be reflected by comparing the pulp for tear, burst, tensile and other sheet properties at different levels of laboratory beating. The properties can be plotted and compared at different freeness levels; at different levels of beating time;

and against each other. When properties are plotted against each other, the freeness, or beating time, or sheet bulk, is indicated. These curves can then be compared with curves similarly obtained from other pulps, in order to assess the effects of type of fibrous raw material, and of cooking, bleaching and refining procedure, upon final strength.

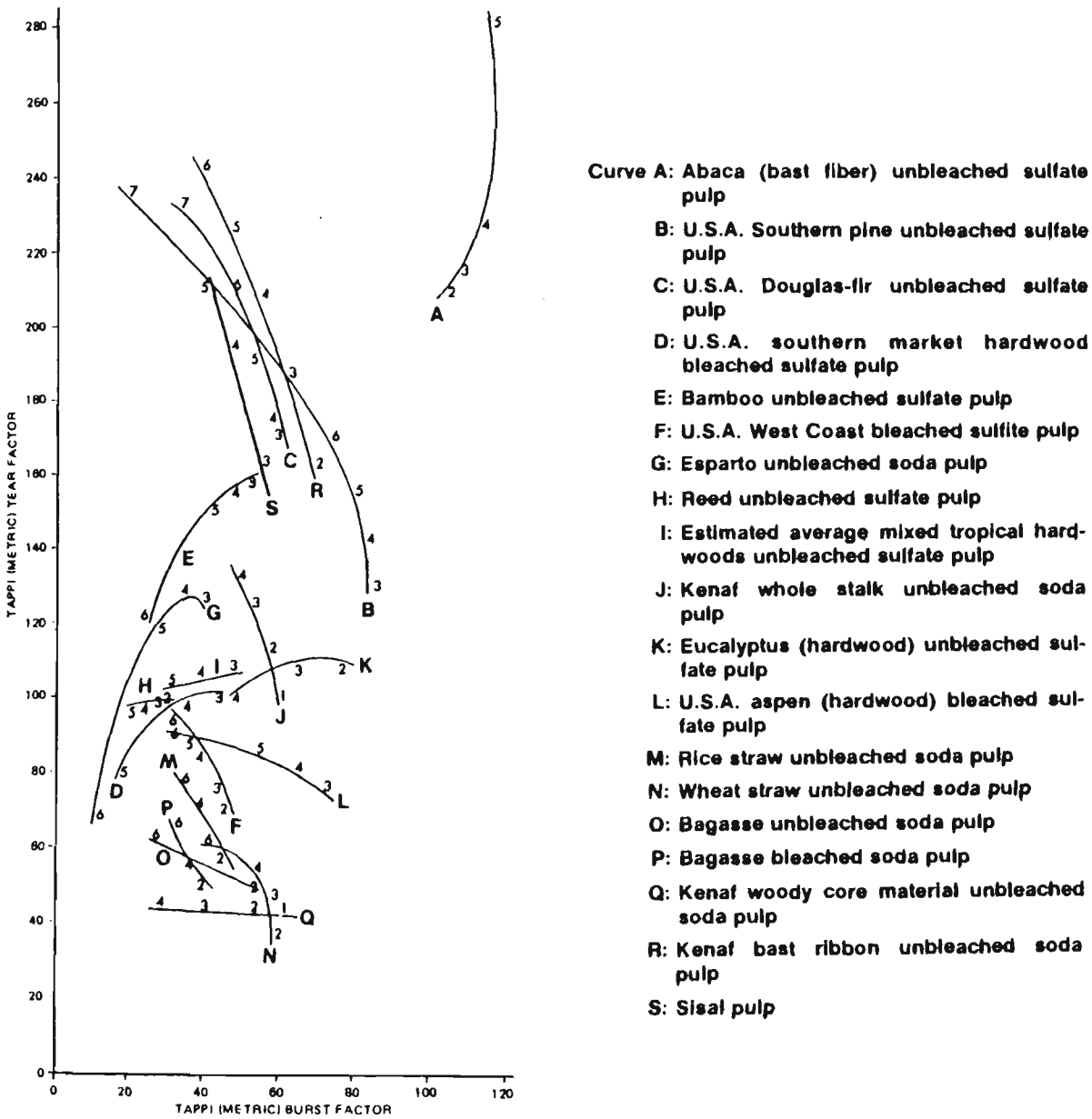

Fig. 3.8 Laboratory beating - strength tests on chemical pulps from various wood and non-wood plant fibres. Digits superimposed on the curves represent Canadian Standard Freeness tests ÷ 100 (Atchison 1983).

Figure 3.8 depicts one group of such curves, it can be seen that there are tremendous differences in strength between the pulps prepared from these different raw materials. For

example, the tear strength of abaca (curve A) is outstanding; and the tear strength of most of the softwood pulps is high compared to that of hardwood pulps and most of the non-wood plant pulps. Mixed tropical hardwood pulps compared favourable with plantation hardwoods and with temperate zone hardwoods. Also to be noted is the strength of kenaf bast fibre (curve R); its properties are comparable to those of softwood fibres. By contrast, the strength of kenaf core pulp (curve Q) is very low. Its initial freeness is also very low, meaning that it is a very slow-draining pulp.

3.5.2.2 Fibre flexibility

The flexibility of a fibre is of great importance during the preparation stage of a composite material and also during the process of composite failure. Certainly wet fibre flexibility has been known to be important for paper manufacture, but the property was seldom measured quantitatively.

Tam Doo (1982) and Kerekes (1985) have developed a single-fibre flexibility test method which eliminates the disadvantages of earlier methods. This has enabled the researchers to measure the effect of wood species and pulping conditions on fibre flexibility and confirm quantitatively what has long been known in a qualitative way. They showed that cedar fibres are flexible (1.33×10^{-2} Nm^{-2}), Douglas fir fibres are stiff (6.8×10^{-2} Nm^{-2}) and Southern pine pulp fibres are very stiff (11.7×10^{-2} Nm^{-2}). It was also shown that mechanical pulps are 20 - 30 times stiffer than chemical pulps from the same species. By chemically treating mechanical pulps, a marked decrease in stiffness can be noted.

3.5.2.3 Work of fracture

The energy needed to form a fracture surface is called work of fracture, and is related to fracture toughness by consideration of the area of the surface formed during fracture. Gordon and Jeronimidis (1980) showed that, weight for weight, the strength and stiffness of wood along the grain compares well with the best engineering metals, as does its work of fracture across the grain.

Earlier Page *et al* (1971) showed that for single wood fibres a pseudo-plastic deformation took place in tension. Wood fibres are hollow tubes composed of layers of fibrils wound in a steep spiral (see Fig 3.1), and so behave as a spirally wound fibre reinforced composite tube. Under axial tensile strain such structures can fail by buckling due to the induced shear stresses. Such a failure mechanism can lead to high values of fracture toughness. This process will be seen to be important in wood fibre reinforced cements, in generating fracture toughness in the composite, as the fibres fail in tension.

3.5.3 The effect of moisture on fibres

It has been shown that cellulose is the primary component of the cell wall and the crystalline microfibrils are the elements which give the fibre its tensile strength; however, there are disordered zones which are believed to play a significant role with respect to mechanical properties.

Hemicellulose and lignin act as matrix materials in wood fibres and are generally considered to be amorphous. They are hygroscopic thermoplastic substances, and so environmental conditions such as humidity and temperature have a strong influence on them and hence the mechanical properties of natural fibres. With increasing moisture

content, the torsional stiffness of fibres decreased by about 50% (between 25% and 90% RH at room temperature) (Nevell, 1984). This softening is related to the softening of the hemicelluloses in the cell wall. In the longitudinal direction this effect (up to 90% RH) results in a drop in modulus of only 11%, although the hemicellulose modulus is reduced by a factor of a thousand. This is clearly a reflection of the fact that the stiff cellulosic microfibrils are preferentially aligned along the fibre.

In many cases, a maximum in fibre tensile strength and tensile modulus is found at low moisture contents. When relatively dry, fibres show low tensile properties due to a poorer stress distribution within their structure. Such an effect is not restricted to natural fibres but also occurs in synthetic fibres.

When single fibres are immersed in water, the relative longitudinal stiffness drops considerably i.e. by about 70 -80% from the value at 50% RH. This has been explained as being due to softening of the disordered zones of the cellulose microfibrils and also to a reduction in cell wall cohesiveness leading to a slippage between fibrils. The relative decrease in modulus of pulp fibres, when immersed in water, is almost independent of yield.

Chapter Four:

Air-cured and Autoclaved Bamboo Fibre Reinforced Cement Composites (BFRC)

Recently, there has been a trend to use natural cellulose fibres to replace asbestos fibre in the fibre reinforced cement industry. In Australia wood fibre (*P.radiata*) has replaced asbestos fibre as a reinforcement in commercial cement product since 1981. This fibre has a reasonably high market price. Thus considerable research effect has gone into the study of fibre composites from fast growing cheap agricultural crops and crop residues, especially for those countries with limited forest resources.

Bamboo is a rapid grown agricultural crop, which has good fibre qualities and is widely used in the paper industry throughout the Asian region. Although bamboo has been used in various forms in the construction industry, there is limited information in the scientific literature concerning the use of bamboo pulp fibre. Sinha *et al* (1975) and Pakotiprapha *et al* (1983) reported that air-cured bamboo fibre reinforced cement composites had flexural strength values close to 20 MPa, at a fibre loading of 10% by mass. However, there was no report of the values of fracture toughness, which is as important a mechanical property as strength or stiffness when considering building materials.

The Hatschek process followed by curing in a high pressure steam autoclave has been commercially applied to the production of wood fibre reinforced cement (WFRC) products. Steam curing at temperatures close to 180°C enables the replacement of between 40% to 60% of ordinary Portland cement by less expensive silica, the latter reacts

with the cement to form a calcium silicate matrix of acceptable strength. The reaction is completed within 6 to 8 hours instead of 3 to 4 weeks required with air-cured products.

This chapter discusses the preparation and mechanical and physical properties of air-cured and autoclaved BFRC composite to establish their suitably as an alternative to wood pulp fibre for asbestos replacement in fibre cement building products.

4.1 Experimental work

4.1.1 Materials

The bamboo fibre was unbleached Kraft pulp of Kappa No.26, and was prepared from commercial packaging paper (Chang Jing Paper Mill, China). The bamboo species was *Sinocalamus affinis (Rendle) McClue*. The matrix was prepared from ordinary Portland cement or from equal proportions of ordinary Portland cement and finely ground silica (Steetly brand, 200 mesh, washed quartz).

4.1.2 Fibre modification

The fibres used in the composites were obtained by soaking the commercial packing paper overnight followed by disintegration for 10 min at a speed of 2850 RPM. After disintegration, the fibres were subjected to 3 different modifications:

1. vacuum-dewatering and crumbing [unbeaten bamboo pulp (400 Canadian Standard Freeness {CSF})];
2. beating in a Valley Beater to 100 CSF, then vacuum-dewatering and crumbling [beaten bamboo pulp (100 CSF)];

3. disintegrating the original pulp with hot water (90-95°C) for 2 min in a 3 litre NORAM disintegrater followed by screening on 0.83 mm hole size Somerville screen (yield 46%) then dewatering and crumbling [screened bamboo pulp (550 CSF)].

4.1.3 Fabrication and characterisation

Air-cured and autoclaved BFRC samples were produced by a slurry / vacuum dewatering and press technique which had proved most successful with wood-pulp fibre reinforced cement composites (see Appendix A.3.2).

Mechanical and physical properties of BFRC such as flexural strength (MOR), fracture toughness, void volume, water absorption and density were determined by the methods described in Appendix A.4.

The fibre length weighted average was measured on a Kajanni FS-200 fibre length analyser (see Appendix A.2.3). The fibre length mass distribution was converted from reported fibre length population distribution.

Canadian Standard Freeness test method is a measurement of pulp drainage. The freeness test used in this study was to indicate the degree of beating (see Appendix A.2.2).

4.2 Air-cured bamboo fibre reinforced cement

4.2.1 Mechanical properties

Air-cured BFRC composites were prepared from two different bamboo pulps, one beaten (100 CSF) and the other unbeaten (400 CSF). The physical and mechanical properties of the BFRC composites are reported in Table 4.1. These properties are also compared with those of both softwood and hardwood pulp reinforced cements in Figures 4.1- 4.4.

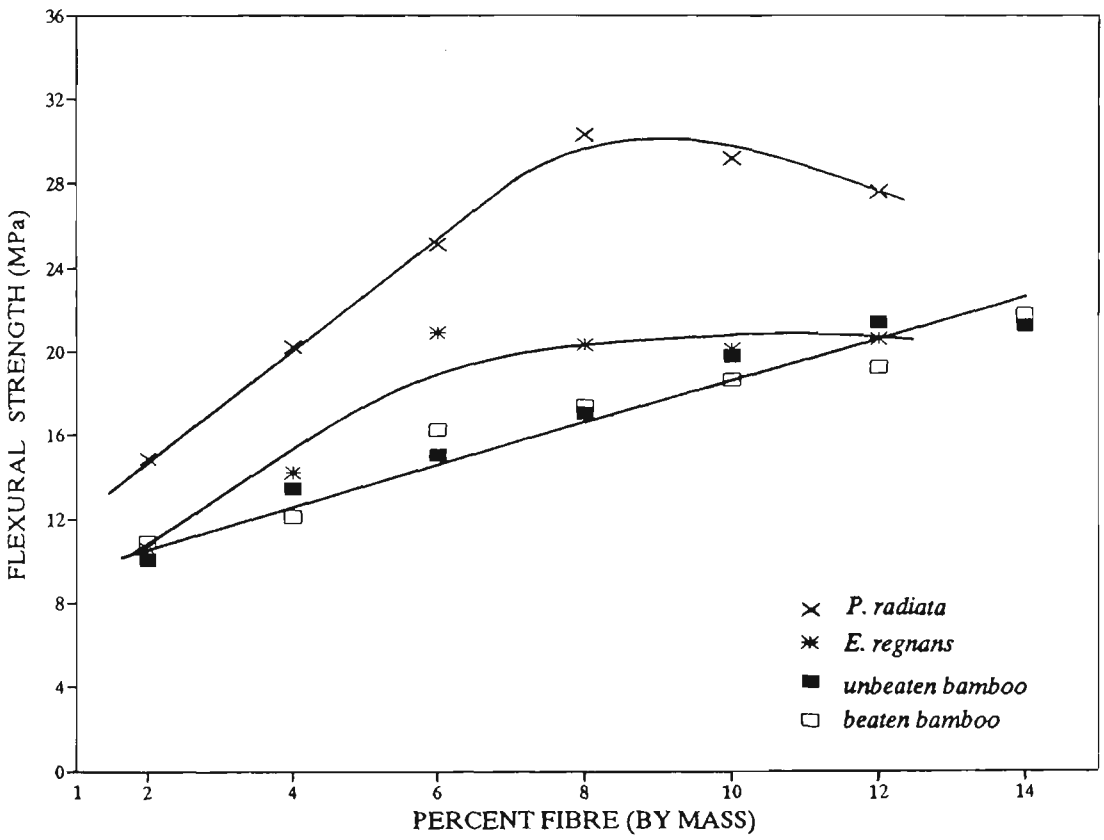

Fig. 4.1. Effect of fibre content on flexural strength for air-cured WFRC and BFRC

Figure 4.1, shows the variation in flexural strength, of BFRC composites, as the fibre content is increased. This same graph contains published data for air-cured WFRC reinforced with softwood (*P. radiata*) (Coutts, 1985) and hardwood fibres (*E. regnans*) (Coutts, 1987a). Flexural strength values for BFRC increased from about 10 MPa up to 22 MPa as the fibre content increased from 2 % - 14 % by mass. The flexural strength value

for unreinforced cement board was suggested about 9 MPa from the graphic. Unlike softwood and hardwood fibres which indicated maximum values of flexural strength for the composites at about 8 % by mass, the bamboo reinforced material was still increasing in flexural strength at loading of 14% by mass for both the unbeaten and beaten pulps.

Table 4.1. Properties of air-cured bamboo-fibre-reinforced cement

Fibre (w%)	MOR (MPa)	Frac. Tough(kJ/m2)	Void Vol (%)	Water Abs.(%)	Density (g/cm³)
Unbeaten Pulp					
2	10.1±2.0	0.10±0.04	26.3±0.9	14.6±0.7	1.81±0.03
4	13.4±1.9	0.15±0.02	26.3±0.6	15.2±0.5	1.73±0.02
6	15.0±2.3	0.23±0.03	26.5±0.6	15.9±0.5	1.67±0.02
8	17.0±1.7	0.34±0.05	26.6±1.1	16.7±0.3	1.59±0.06
10	19.7±1.0	0.49±0.05	26.0±1.1	16.8±0.7	1.55±0.02
12	21.4±3.8	0.80±0.23	25.6±0.5	17.6±0.4	1.46±0.03
14	21.2±2.4	0.97±0.23	25.3±0.9	17.9±0.8	1.42±0.02
Beaten Pulp					
2	10.9±1.4	0.07±0.01	26.8±0.7	14.6±0.6	1.83±0.03
4	12.1±1.3	0.15±0.02	28.3±0.6	16.7±0.4	1.69±0.03
6	16.2±1.0	0.23±0.02	27.6±0.4	16.7±0.4	1.65±0.03
8	17.4±0.8	0.32±0.03	26.6±0.9	16.5±0.6	1.61±0.03
10	18.6±1.1	0.45±0.07	26.5±0.9	16.9±0.6	1.57±0.05
12	19.2±1.4	0.54±0.05	27.1±0.7	18.2±0.3	1.49±0.03
14	21.8±1.7	0.70±0.06	28.0±0.6	19.4±0.4	1.44±0.04

*All composite were fabricated using ordinary portland cement, air-cured for 28 days, tested at 50±5 per cent RH and 23±2°C.
* 3 standard deviation, sample size n=9.

The fracture toughness values of BFRC increased with increasing fibre content (Table 4.1 and Fig. 4.2). At a fibre loading of 14% by mass the fracture toughness values were ~ 1.0 kJm^{-2} and ~ 0.7 kJm^{-2}, for samples containing unbeaten and beaten fibres respectively. These values are low compared to softwood and hardwood fibre reinforced WFRC's at fibre loading of 12% by mass which are 2.25 $kJm^{-2}$and 1.68 kJm^{-2} respectively (Fig. 4.2). The initial indication is that bamboo fibres are unsuitable on their own as a reinforcement for cement products as they lack the essential property of fracture toughness. The use of hybrid fibre cement formulations containing both bamboo and long softwood fibres have been investigated in an attempt to improve fracture toughness properties and will be reported in Chapter 5.

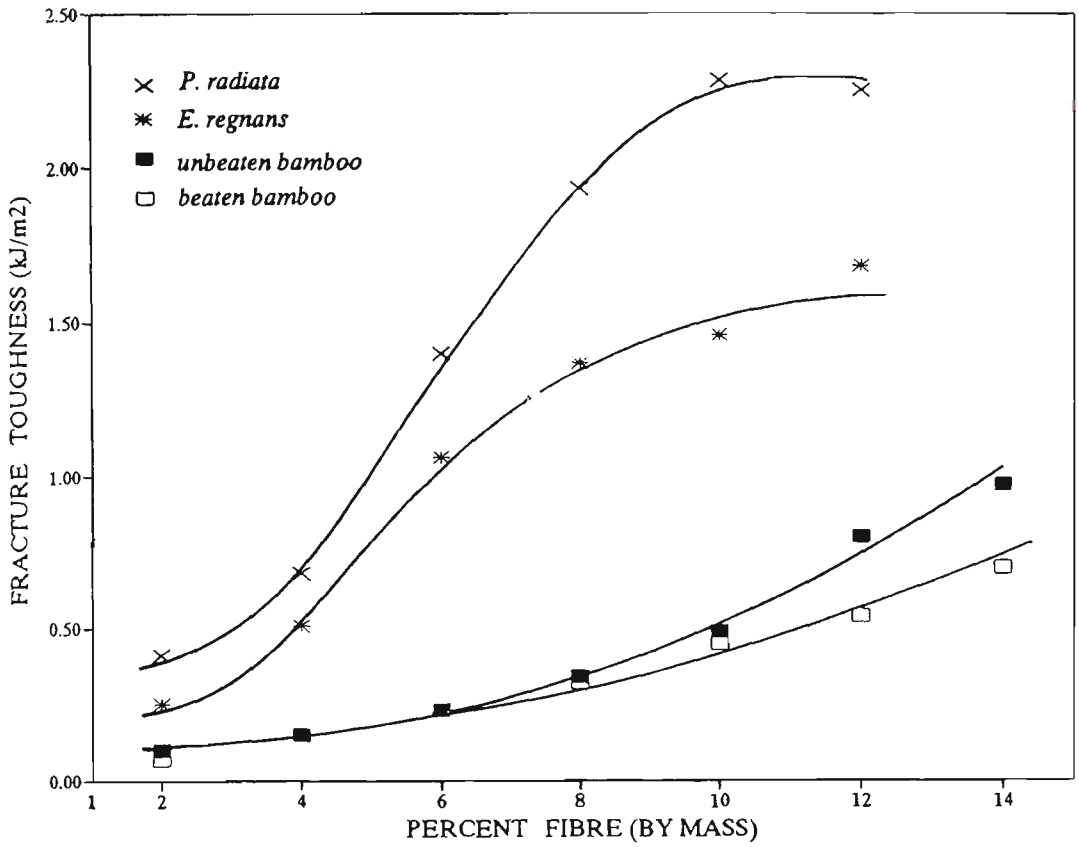

Fig. 4.2. Effect of fibre content on fracture toughness for air-cured WFRC and BFRC

An important observation with regard to the mechanical performance of the BFRC compared to softwood and hardwood WFRC's is that the long softwood pulp fibres produced superior properties of flexural strength (> 30 MPa) and fracture toughness (> 2.28 kJm^{-2}) than either BFRC or hardwood WFRC, at similar loading of 8 % by mass of fibre. This behaviour has been explained by the fact that the long softwood fibres (as measured on a Kajanni FS-200) had a fibre length weighted average of ~2.8 mm and were more able to contribute to the reinforcement and to the process of fibre pull-out during fracture. The shorter bamboo fibres (average fibre length weighted 1.1 mm) or hardwood fibres (average fibre length weighted 1.0 mm) are not as effective as reinforcement. However, this is not the complete explanation as BFRC and hardwood WFRC have similar flexural strength values (see Fig. 4.1), yet BFRC materials have much lower fracture toughness values than hardwood WFRC (see Fig. 4.2).

In natural fibre reinforced cement composites fibre length plays a major role in the mechanical performance of the material. At the same time other fibre properties such as fibre coarseness, wall thickness (lumen size), fibre wall structure and fibre strength *etc.* also effect the composite properties. For example in this study, bamboo fibre is much weaker than softwood or hardwood fibre. Zero-span tensile index as measured on handsheets made from pulps of bamboo, *P.radiata* and *E.regnans* were 71 Nm/g, 138 Nm/g and 138 Nm/g respectively. Bamboo fibre has a different microstructure from wood fibre, there is a very small lumen (compared to softwood fibres) and the fibre's primary wall is easily pealed off during refining(Wai, 1985; Wang, 1993). Furthermore bamboo pulp contains a considerable number of segments and pitted cells (see section 3.5.1.1.), which act as a filler rather than as a reinforcing material in the cement based composites. The complexity of these parameters is currently being studied by investigating the relationships between pulp fibre properties and the properties of the cement composites derived from such pulps and some of the preliminary results will be discussed later in this thesis.

4.2.2 Physical properties

The physical properties of void volume, water absorption and density are reported in Table 4.1. There appears to be little difference in the physical properties of the composites containing either beaten and unbeaten bamboo fibre. As the fibre content was increased from 2 % - 12 % fibre by mass the water absorption only increases from 14 % to about 18 % by mass (see Fig. 4.3). It can be seen that as the fibre content increased the rate of increase in water absorption was much lower for bamboo fibre containing materials than for softwood (14 % - 25 %) and hardwood (18 % - 30 %) fibre reinforced products.

Likewise, the density of the bamboo reinforced materials decreased at a lower rate than either softwood or hardwood WFRC (Fig. 4.4), while the void volume remained fairly constant over the range of fibre contents studied.

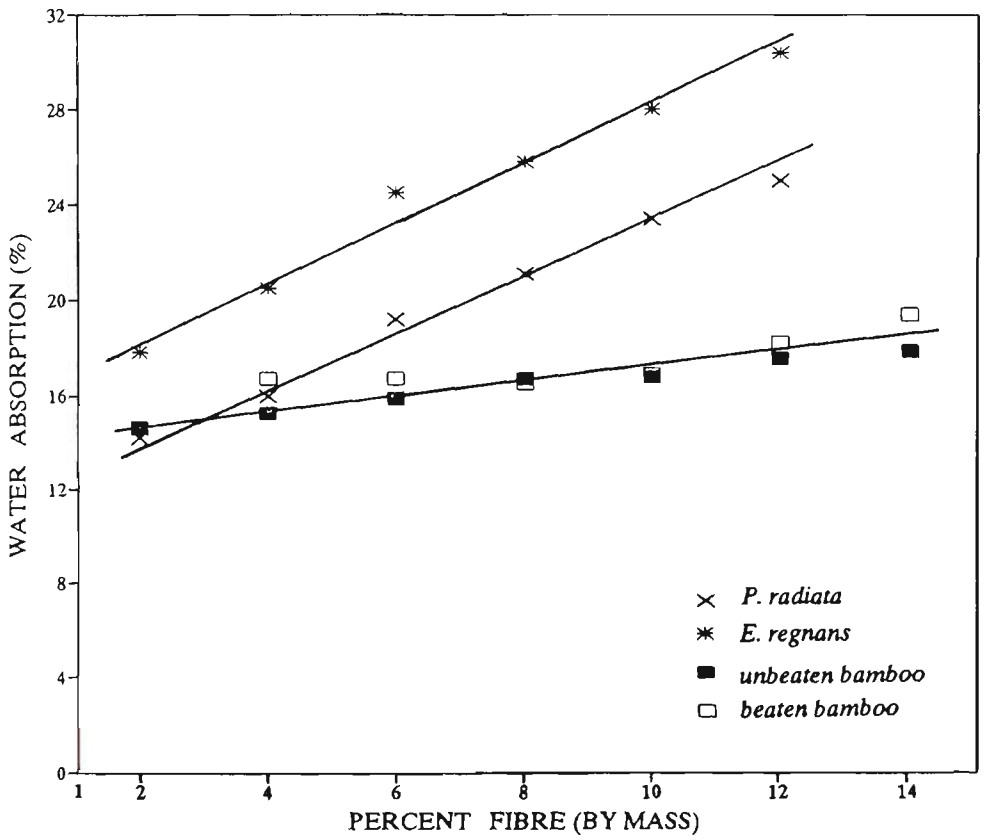

Fig. 4.3. Effect of fibre content on water absorption for air-cured WFRC and BFRC

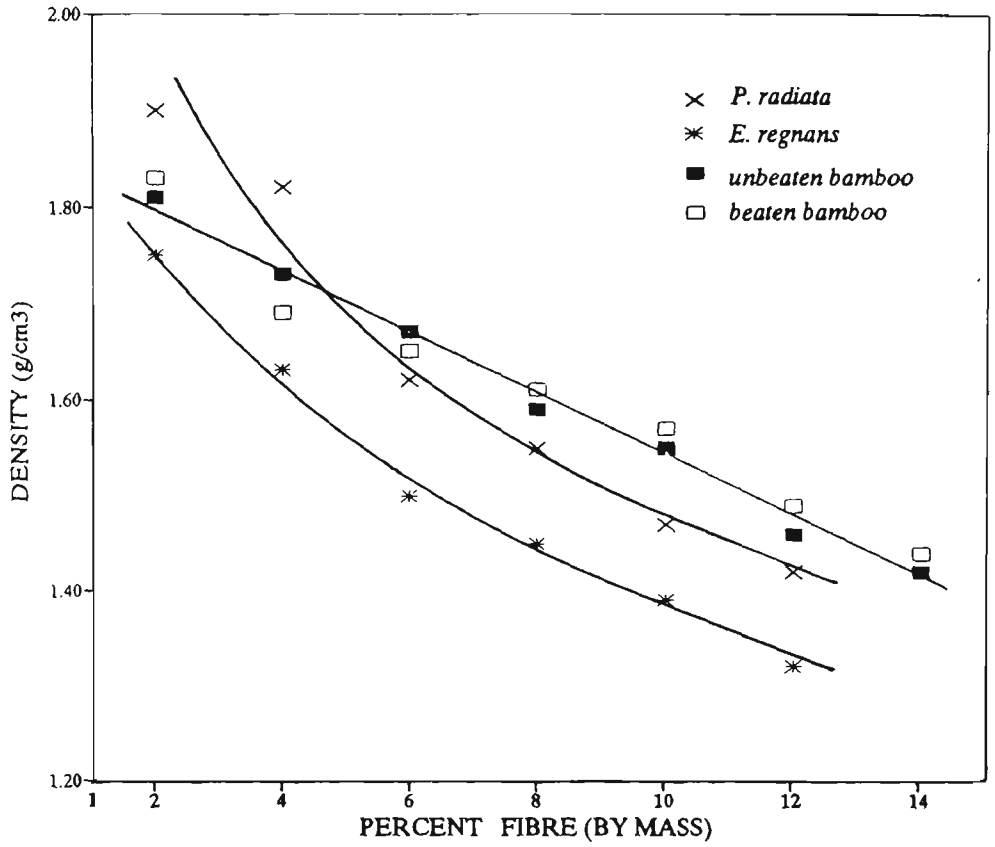

Fig. 4.4. Effect of fibre content on density for air-cured WFRC and BFRC

4.3 Autoclaved bamboo fibre reinforced cement

4.3.1 Mechanical properties

4.3.1.1 Flexural strength

1. Unbeaten fibre reinforced composites

Table 4.2 and Figure 4.5 show the variation in flexural strength of BFRC composites, as the fibre content is increased from 2% to 22% in steps of 2%. Figure 4.5 contains reference data for autoclaved WFRC composites reinforced with Kraft *P.radiata* fibres, as this fibre is commercially used in Australia and is the preferred fibre. Flexural strength values for autoclaved BFRC composites increased from about 12 MPa up to 18 MPa as the fibre content was increased from 2% to 14% at which point the strength of the composite products start to decrease due to poor fibre distribution throughout the matrix material.

This observation was in general agreement with the change in flexural strength observed with autoclaved WFRC composites.

2. Beaten fibre reinforced composites

Beaten (100 CSF) and unbeaten (400 CSF) BFRC material gave similar flexural strength values of approximately 18 MPa at 14% fibre (Table 4.2). With the WFRC composites it was found that beaten *P radiata* (550 CSF) gave a maximum flexural strength of 24.3 MPa at 10% fibre loading. This was an improvement over products reinforced with unbeaten fibres.

Table 4.2 Properties of autoclaved bamboo-fibre-reinforced cement

Fibre (w%)	MOR (MPa)	Frac. Tough(kJ/m2)	Void Vol (%)	Water Abs.(%)	Density (g/cm^3)
Unbeaten Pulp					
2	13.3±0.5	0.08±0.01	35.7±0.5	22.1±0.5	1.62±0.02
4	13.2±1.8	0.13±0.01	38.0±0.9	24.9±0.9	1.52±0.03
6	13.8±0.9	0.20±0.02	39.1±1.0	26.7±1.0	1.46±0.02
8	15.5±1.0	0.29±0.05	43.1±2.2	30.7±1.1	1.41±0.04
10	16.0±0.8	0.39±0.04	45.1±1.7	33.9±1.5	1.33±0.02
12	16.5±1.2	0.49±0.05	45.7±0.9	35.9±1.3	1.27±0.02
14	18.3±1.6	0.62±0.01	45.4±4.9	36.6±6.5	1.26±0.11
16	17.2±1.5	0.77±0.07	48.6±1.4	41.1±2.3	1.18±0.03
18	17.7±1.0	0.96±0.14	49.2±1.2	42.8±2.3	1.15±0.03
20	16.1±0.6	1.08±0.11	50.8±0.8	45.1±2.1	1.13±0.05
22	14.9±1.3	1.20±0.31	50.9±0.6	49.0±2.3	1.00±0.05
Beaten Pulp					
2	12.1±0.5	0.10±0.01	34.2±0.6	21.0±0.6	1.63±0.02
4	12.2±1.0	0.13±0.02	37.7±1.1	24.8±1.0	1.52±0.02
6	14.6±1.3	0.22±0.03	39.8±1.5	27.6±1.4	1.44±0.02
8	14.9±1.2	0.29±0.04	41.1±1.1	29.1±1.1	1.41±0.02
10	16.1±0.9	0.40±0.04	44.2±1.3	33.1±1.6	1.34±0.03
12	17.1±1.4	0.50±0.02	44.3±1.4	34.1±1.6	1.30±0.03
14	18.2±1.3	0.50±0.01	42.8±0.8	32.5±1.2	1.32±0.03
16	18.2±1.4	0.56±0.06	44.7±1.5	35.9±2.0	1.25±0.03
18	16.4±0.9	0.82±0.08	46.9±1.1	40.6±1.8	1.16±0.04
20	17.1±1.5	0.97±0.08	47.0±0.8	40.9±1.1	1.15±0.01
22	16.7±1.2	1.03±0.11	47.4±1.0	42.2±1.4	1.12±0.02

*BFRC composites were fabricated using ordinary Portland cement and silica at the ratio of 1:1, autoclaved at 1.25MPa steam pressure for 7.5h, tested at 50±5 per cent RH and 22±2^{o}C. BFRC composites maximum flexural strength at 14% by mass. 3 standard deviation, sample size n=9.

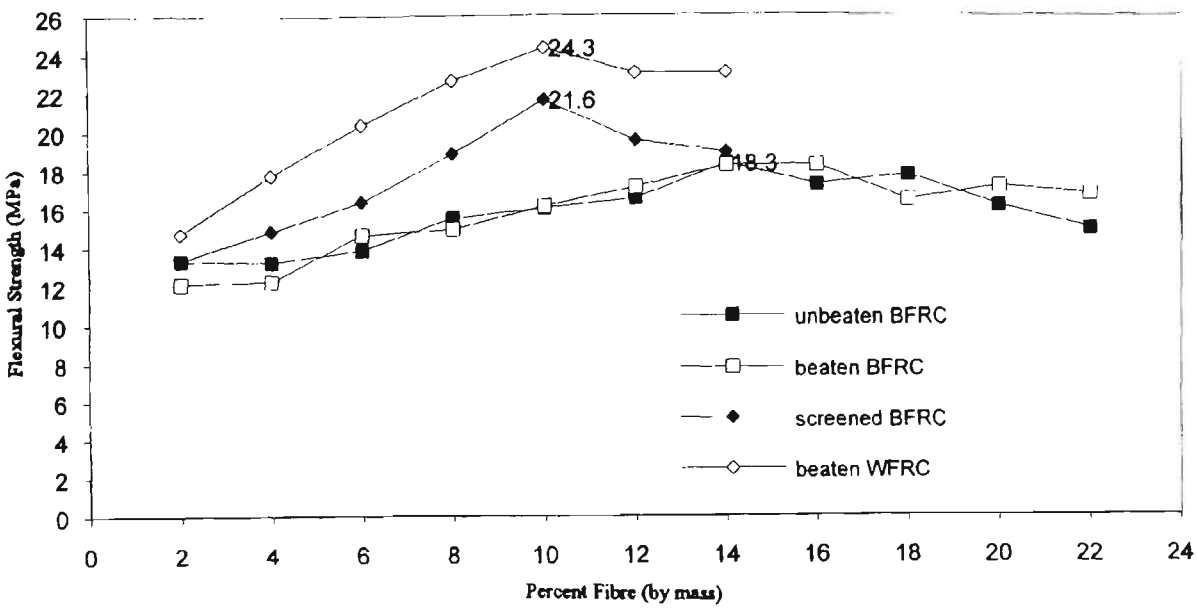

Fig. 4.5. Flexural strength as a function of percent fibre loading for autoclaved BFRC and WFRC composites

Table 4.3 Fibre weighted average length (mm)

beaten *P.radiata*	unbeaten bamboo	beaten bamboo	screened bamboo
2.4	1.0	0.8	1.3

Bamboo has a fibre length less than half of that of *P.radiata* (Table 4.3). The bamboo fibre used in this study was separated from commercial packaging paper and there was a high fines content in both the beaten and unbeaten pulps. The beaten and unbeaten bamboo pulp fines (lengths < 0.4 mm) accounted for 32.7% and 28.8% of the mass respectively (Table 4.4). This suggests little damage occurred during beating.

Table 4.4 Fibre length mass distribution percentage

Length (mm) longer than	beaten *P.radiata*	unbeaten bamboo	beaten bamboo	screened bamboo
0.05	100	99.7	99.7	100
0.4	**95.6**	**71.2**	**67.3**	**94.8**
0.8	90.0	51.6	42.1	74.3
1.2	81.8	32.2	22.6	51.1
1.6	72.1	19.2	11.4	30.3
2.0	61.3	10.9	5.3	15.4
2.4	48.9	5.9	2.5	7.6
2.8	36.8	3.1	1.3	3.6
3.2	28.0	1.7	0.6	1.6

Fibre length mass distribution percentage was analysed on Kajanni FS-200 fibre length analyzer. More than 15,000 fibres were measured and analysed. Length less than 0.4mm fibre mass distribution percentage for *P.radiata*, unbeaten bamboo, beaten bamboo and screened long bamboo pulp was 4.4%, 28.8%, 32.7% and 5.2% respectively.

3. Screened fibre reinforced composites

Bamboo pulp was washed through a Somerville screen (0.83 mm hole size, yield 46%) to remove fines and to obtain the longer fibres for use as reinforcement. Compared with unscreened unbeaten fibre the screened fibre showed improved composite flexural strength at the same fibre loading. The maximum flexural strength of screened BFRC was 21.6 MPa at 10% fibre loading (Table 4.5).

Fibre length can make a significant contribution to the composite flexural strength. Softwood *P.radiata* fibre, and screened and unscreened unbeaten bamboo fibres, had fibre lengths (length weighted average) was 2.4 mm, 1.3 mm and 1.0 mm respectively (Table 4.3). The flexural strengths of materials reinforced with wood fibre, and screened and unscreened unbeaten bamboo fibres were 24.3 MPa, 21.6 MPa and 16.0 MPa respectively at 10% fibre by mass (Fig. 4.5).

Table 4.5 Properties of autoclaved screened long bamboo fibre reinforced cement

Fibre (w%)	MOR (MPa)	Frac. Tough(kJ/m2)	Void Vol (%)	Water Abs.(%)	Density (g/cm^3)
2	13.3±1.4	0.10±0.01	39.1±0.8	24.7±0.8	1.59±0.02
4	14.8±1.5	0.23±0.02	41.2±1.0	27.7±1.1	1.49±0.02
6	16.3±2.2	0.37±0.06	42.6±1.0	30.0±1.3	1.42±0.03
8	18.8±1.2	0.54±0.12	42.6±0.9	30.1±0.9	1.42±0.07
10	21.6±2.2	0.71±0.13	42.7±1.4	32.0±1.4	1.33±0.02
12	19.5±1.5	0.79±0.06	46.6±0.6	37.8±1.4	1.23±0.03
14	18.9±1.9	1.09±0.14	47.0±0.9	39.8±1.7	1.18±0.03

* Unbeaten bamboo pulp was screened on 0.83mm hole size Sommerville screen (yield 46%) fibre length weighted average (Kajanni FS200 fibre analyser) was 1.3 mm and the freeness was 550CSF. BFRC maximum flexural strength was at 10% by mass fibre loading, which was similar fibre loading to WFRC. 3 standard deviation, sample size n=6.

The flexural strength of unscreened BFRC materials (beaten and unbeaten) increased up to 14% fibre before the maximum value was reached. This could be due to the fact that the fine material (length < 0.4 mm) offers little reinforcement to the composite and so a greater mass of pulp was needed to have sufficient numbers of long fibres. When fines (lengths < 0.4 mm) were removed, maximum strength was achieved with fibre loading between 8 - 10%, which is in keeping with the early study of WFRC composite. The

percentage of screened bamboo pulp fibre lengths longer than 0.4 mm, was 94.8% of its mass. This is similar to the data for *P.radiata* pulp (Table 4.4).

A fibre length fractionation study of *P.radiata* wood pulp supports this belief that fragments with length less than 0.3 mm, act more as a filler-diluent than as a reinforcing fibre when used to make WFRC products (see Chapter 6). A similar behaviour regarding the maximum flexural strength value with respect to fibre mass content (about 12%) was noted for waste paper fibre reinforced cement products (Coutts, 1989). In that instance, the high fibre content was required to provide sufficient mass of the longer reinforcing fibres. This was due to the high fines content generated (lengths < 0.6 mm constituted 21.2% by mass) during processing and recycling.

There is little difference between beaten and unbeaten BFRC composites with respect to flexural strength, which contrasts with the observation reported in the earlier research on beaten and unbeaten *P.radiata* WFRC composites (Coutts, 1984a). Similar behaviour to that of autoclaved BFRC was found in the case of autoclaved NZ flax composite (Coutts, 1983c) and air-cured bamboo reinforced cement products (Coutts, 1994b). This might be associated with the fact that the round and small diameter NZ flax and bamboo fibres do not have the same ability to collapse as do softwood fibres which form flat ribbons. Alternatively, it may be due to the beating, generating fines which do not contribute to the composites strength.

4.3.1.2 Fracture Toughness

The mechanism that takes place when a fibre composite is loaded to failure include fibre fracture and fibre pull-out. The latter can have considerable influence on the value of fracture toughness. If the fibre is short then the energy required to pull the fibre through the matrix, after the fibre to matrix bond is broken, is low and can contribute little to the dissipation of energy contained in the advancing crack. Therefore the crack continues through the sample and the material appears brittle.

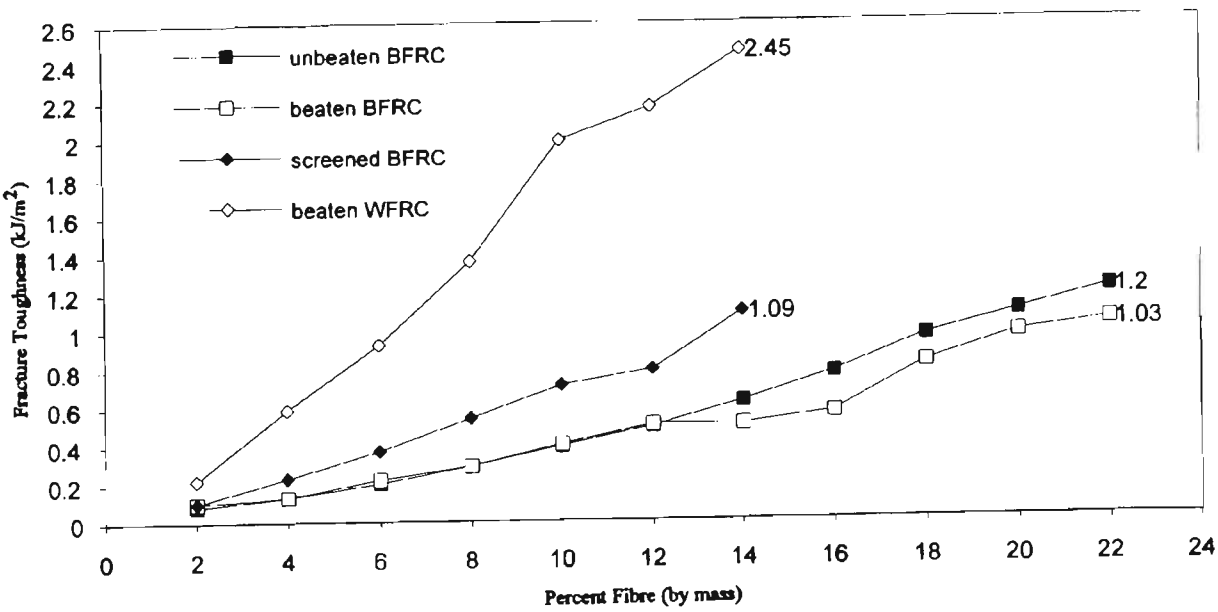

Fig. 4.6. Fracture toughness as a function of percent fibre loading for autoclaved BFRC and WFRC composites

Figure 4.6 shows the increment in fracture toughness values of beaten and unbeaten BFRC composites (from 0.10 kJ/m² to 1.03 kJ/m² and from 0.08 kJ/m² to 1.20 kJ/m² respectively) for fibre loading from 2% to 22%. The same graph has reference data for WFRC composite fracture toughness, which are seen to have higher values (Coutts, 1984a). Screened BFRC composites which contain more long fibres are tougher [from 0.10 kJ/m² to 1.09 kJ/m² for fibre loadings from 2% to 14%, (Fig. 4.6)] than unscreened composites. Unbeaten BFRC composites are slightly better than beaten ones at the high fibre loadings.

WFRC composites indicated better properties of fracture toughness than BFRC composites in the three point bending load/deflection curve as shown in Figure 4.7. After reaching the maximum load, the curve for WFRC composites went through a gradual "tailing off" which indicated that a greater amount of fracture energy was needed to pull the long fibres though the matrix. The curves for the BFRC composites did not show such behaviour and were more brittle. This is indicated by a sharp "drop off" of load carrying capacity in the load/deflection curve. Fracture toughness behaviour is related to fibre length and fibre morphology, and comparison is only valid for specimens of the same thickness.

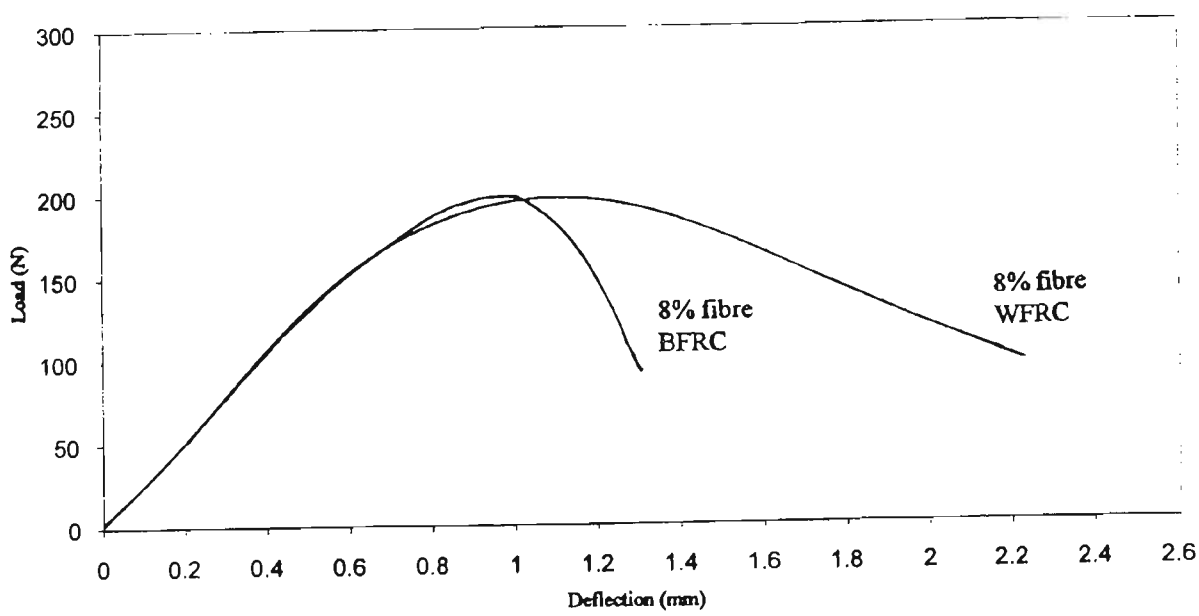

Fig. 4.7. Typical Load / Deflection graph for autoclaved WFRC and BFRC composites.

Beaten BFRC composites did not vary greatly in fracture toughness values from those of the unbeaten BFRC composites. At high fibre loadings the beaten BFRC composites showed slightly lower values due probably to the increased amount of fines present in the formulation (Table 4.4). As stated above, the presence of fines provides less opportunity

for fibre pull-out being a major component in the mechanism of failure, and hence lower fracture toughness values are observed (see Chapter 6).

4.3.2 Physical Properties

The changing proportions of the constituent fibres and matrix affect void volume, density and water absorption (Table 4.2 and 4.5). There is little difference in the density of the composites when the bamboo fibre was beaten compared with the materials containing unbeaten bamboo fibre (Fig 4.8). However, the same graph shows a slight decrease in density, at a given fibre content, when the pulp has been screened to remove the fines. This effect is possibly due to the increase in fines, present in the unscreened pulps, which allows closer packing of the fibres and matrix, and hence less void volume in the composite.

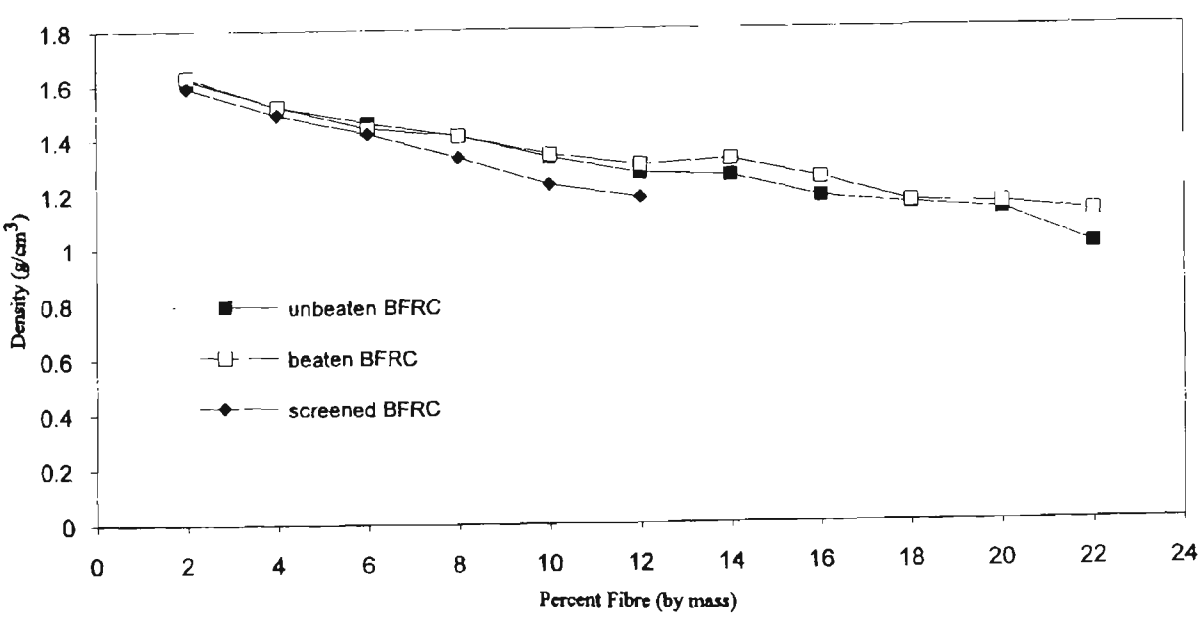

Fig. 4.8. Density as a function of percent fibre loading for autoclaved BFRC composites.

The relationship between water absorption and density is depicted in Figure 4.9. The amount of water absorbed by the cellulose fibre reinforced cement composites depends on

their void volume and the amount of cellulose material present; both these parameters have an effect upon density. Thus one would expect the density to decrease and the water absorption to increase as the fibre content is increased, due to the nature of the hydrophilic, low density bamboo fibres. At the same time the packing of fibres and matrix becomes less efficient, as the fibre content is increased and so void volume increases accompanied by decreased density and increased water absorption.

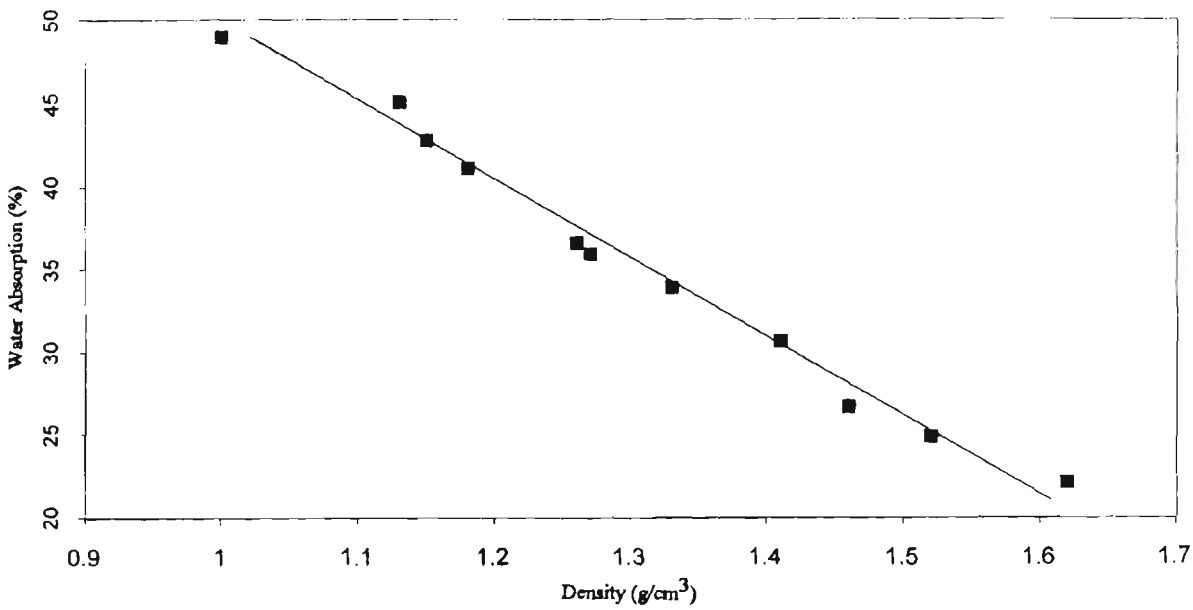

Fig. 4.9. The relationship between density and water absorption for autoclaved BFRC composite

4.4 Conclusions

Bamboo fibre is a satisfactory fibre for incorporation into the cement matrix. Air-cured BFRC composites at 14% by mass had flexural strength values of about 22 MPa. Alternatively, autoclaved products had strength values of about 18 MPa at same fibre loading. However, the fracture toughness was low due to short fibre length and high fines content of the bamboo pulp.

By screening out "fines" contained in the original bamboo pulp, autoclaved composites flexural strength could be improved to greater than 20 MPa while fracture toughness exceeded 1.0 kJ/m^2 at a loading of 14% fibre .

In contrast to softwood fibre reinforced cement composites, beaten bamboo fibre composites did not vary greatly in flexural strength and fracture toughness values from those of the unbeaten fibre composites.

Chapter Five:

Bamboo and Wood Hybrid Fibre Reinforced Cement Composite Materials (BWFRC)

As we have discussed in Chapter 4 composites reinforced with bamboo fibre have lower mechanical properties than composites reinforced with wood fibre due to relative short fibre length of bamboo fibre. This study includes in blending furnishes of bamboo pulp and long softwood fibre pulp to increase the average fibre length thus hopefully improving composites mechanical properties.

Pulp blending idea is used widely in the pulp and paper industry with the aim of modifying the final product specifications. For example, long fibre pulp blended with short fibre pulp can improve short fibre paper properties of tear, tensile and burst strength; short fibre pulp blended with long fibre pulp can improve long fibre paper properties of formation and surface smoothness. Blended furnish of some pulps at certain proportion would even generate synergistic effect for some paper properties (Kuang, 1992).

Over the years, the combined use of different types of fibres to optimise the performance of a material has also been studied and commercialised by a number of material scientists and technologists. A considerable amount of work has been done in hybridising polypropylene fibre, PVC fibres, glass fibre and cellulose fibre with the aim of improving fibre-cement products fracture toughness and durability (Walton, 1975; Mai, 1980; Simatupang, 1987; Gale, 1990). However, the poor temperature resistance of

polypropylene fibre and poor alkali resistance of glass fibre inhibit their use in autoclaved cement product (see section 1.1.3).

James Hardie Industries in Australia has a patent, which reported the combination of cellulose fibres and a small amount of chopped cellulose fibre (preferred proportion at about 0.1% of the total mass, and preferred length about 10 mm) (Aus Patent, 1982), but there is no further information available regarding their products reinforced with hybrid fibre. Fordos at Dansk Eternit in Denmark reported a study of hybrid composites properties (1986). Kraft softwood fibre combined with eucalypt fibres at 1:1 ratio showed interesting high flexural strength in air-cured cement, although detailed information was not provided in the paper.

The purpose of this chapter is to investigate the combination of long softwood fibre with short bamboo fibre to improve the composites properties, particularly in terms of fracture toughness development.

5.1 Experimental work

5.1.1 Fibre Preparation

Bamboo and softwood pine fibres were used in this study. Bamboo fibre was prepared from Kraft-unbleached commercial packaging paper (Jian Xi Paper Mill, China). This is a different source of bamboo pulp to earlier work although the species is the same [*Sinocalamus affinis (Rendle) McClue*]. Pine fibre was obtained from Australia APM Maryvale mill Kraft-unbleached dry lab pulp. The packaging paper and the dry lab pulp were soaked separately in water over-night then disintegrated into individual fibres. After disintegration the fibres were subjected to de-watering, crumbling and the moisture

content was measured. The wood pulp was blended with bamboo pulp in a small mixer into various proportions as shown in Table 5.1.

5.1.2 Fabrication and characterisation

Fibre cement composites reinforced with above prepared hybrid fibre were produced and characterised as the method described in Appendix A.3 and A.4. Air-cured samples were reinforced with 6%, 8% and 10% of blended pulp. Whereas, the autoclaved samples were reinforced with 8%, 10%, 12% and 14% of blended pulp.

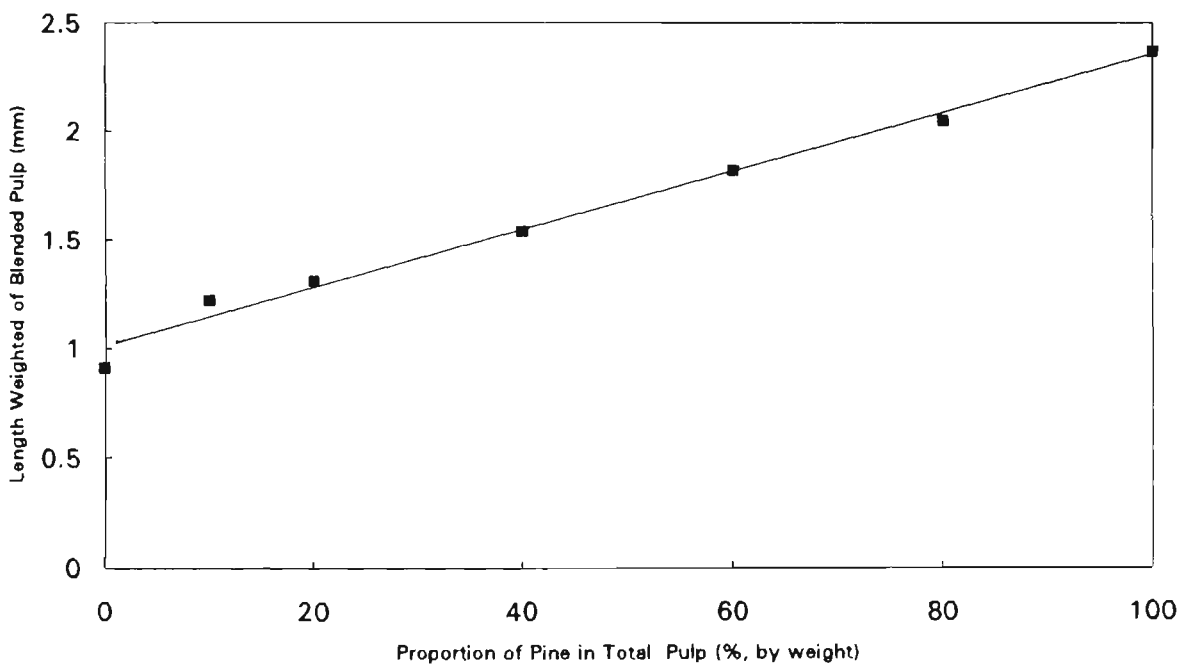

Fig. 5.1. Relationship between pine fibre proportion and furnish pulp length weighted average.

5.2 Results and discussion

5.2.1 Length and freeness of blended pulp

The value of length weighted average and freeness of blended pulps is listed in Table 5.1 and depicted in Figure 5.1 and 5.2. These furnishes were prepared from different proportions of bamboo and pine pulp. Both fibre length and freeness of blended pulp increased almost linearly as the proportion of pine fibre increased. For example, bamboo pulp alone had length about 0.91 mm and freeness around 330 CSF; while, 60% bamboo and 40% pine blended furnish had increased length to 1.54 mm and draining ability to 470 CSF. As discussed in Chapter 5, BFRC had fairly poor strength and toughness values due to the short length of bamboo fibres. Blended pulp has increased average fibre length thus one would expect composites reinforced with such hybrid fibre to have better mechanical properties.

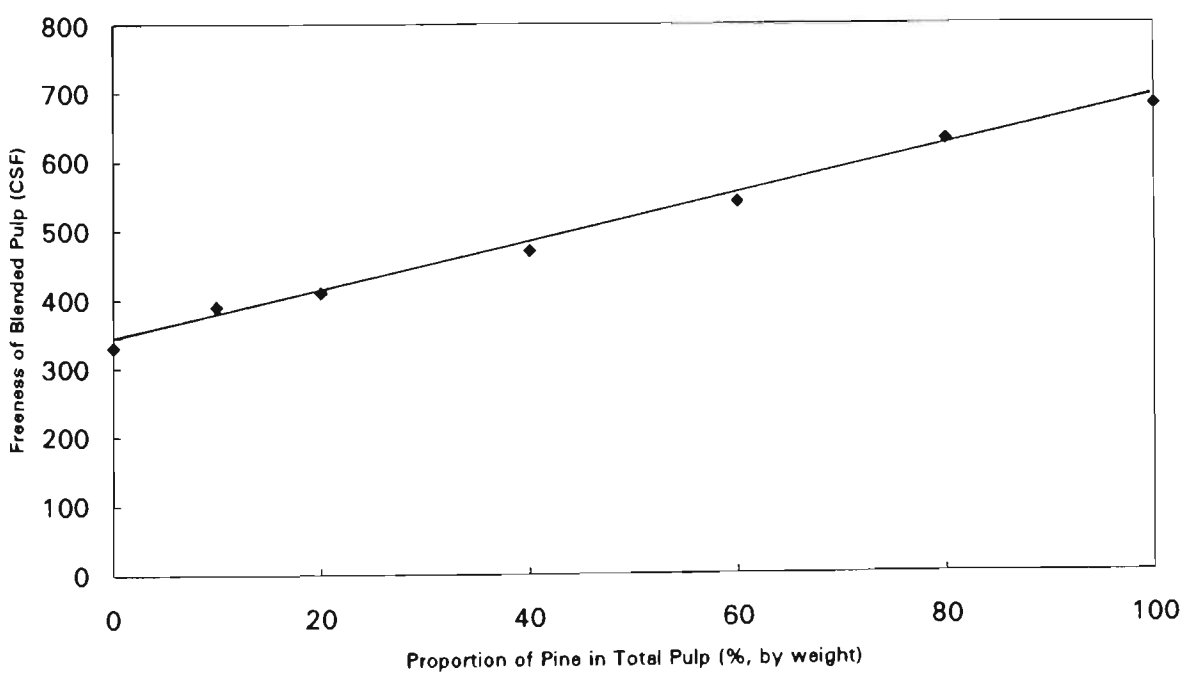

Fig. 5.2. Relationship between pine fibre proportion and furnish pulp freeness value.

5.2.2 Air-cured BWFRC composites

Results of air-cured BWFRC composites mechanical and physical properties are listed in Table 5.2 and shown in Figure 5.3, 5.4 and 5.5, 5.6.

Table 5.1 Length and freeness of blended pulp

Proportion of Blended Pulp	Length Weighted Av. (mm)	Freeness (CSF)
100B*+0P	0.91	330
90B+10P	1.22	390
80B+20P	1.31	410
60B+40P	1.54	470
40B+60P	1.82	540
20B+80P	2.05	630
0B+100P	2.37	680

* B and P represent bamboo and pine pulps respectively. The number before B and P is the weight proportions of bamboo and pine pulp in the blended furnish.

Table 5.2 Properties of air-cured BWFRC

Fibre (w%)	MOR (MPa)	Frac. Tough(kJ/m2)	Void Vol (%)	Water Abs.(%)	Density (g/cm^3)
100B+ 0P					
6%	17.8±2.3	0.82±0.06	33.1±0.9	20.6±0.7	1.64±0.02
8%	18.1±1.1	0.97±0.05	33.6±0.5	21.9±0.4	1.56±0.01
10%	18.5±2.0	1.24±0.22	35.3±1.4	23.2±1.2	1.50±0.02
90B + 10P					
6%	18.8±1.7	1.11±0.21	32.6±0.9	20.1±1.0	1.63±0.04
8%	20.4±1.8	1.26±0.17	33.7±0.7	21.6±0.7	1.57±0.03
10%	20.1±1.5	1.34±0.09	35.3±0.6	23.6±0.6	1.50±0.01
80B + 20P					
6%	17.9±1.5	0.95±0.22	31.9±0.7	19.4±0.6	1.64±0.02
8%	21.0±2.2	1.35±0.19	33.8±0.5	21.5±0.6	1.57±0.02
10%	23.3±2.0	1.67±0.11	33.9±0.9	22.3±0.9	1.52±0.02
60B + 40P					
6%	20.3±1.5	1.21±0.20	32.5±0.5	20.0±0.4	1.64±0.02
8%	21.3±1.9	1.36±0.23	34.6±0.5	22.2±0.6	1.56±0.02
10%	22.7±1.2	1.78±0.25	35.6±1.0	23.7±1.1	1.50±0.03
40B + 60P					
6%	22.9±1.4	1.47±0.27	32.0±0.7	19.2±0.8	1.66±0.03
8%	24.4±1.9	1.69±0.38	33.6±0.8	21.4±0.3	1.57±0.02
10%	25.8±1.8	2.38±0.58	33.8±0.3	22.7±0.6	1.52±0.03
20B + 80P					
6%	23.8±1.8	1.46±0.27	32.0±0.4	19.2±0.4	1.66±0.02
8%	26.6±2.7	2.15±0.33	33.3±1.2	21.0±1.2	1.58±0.04
10%	25.0±2.4	2.49±0.46	35.0±0.8	23.3±0.9	1.50±0.03
0B + 100P					
6%	24.3±1.4	1.97±0.21	32.5±0.6	19.1±0.6	1.66±0.02
8%	25.5±1.5	2.38±0.38	32.0±0.4	19.9±0.4	1.61±0.02
10%	26.5±0.9	2.96±0.37	33.1±0.3	22.2±0.4	1.53±0.02

*All composite were fabricated using ordinary portland cement, air-cured for 28 days, tested at 50±5 per cent RH and 23±2°C.
* 3 standard deviation, sample size n=9.

Figure 5.3 shows that flexural strength of BWFRC increases with increasing of hybrid fibre contents which is in keeping with early results in natural fibre reinforced cement. More importantly that strength develops almost linearly as the long pine fibre content is increased. This behaviour was associated with increasing average fibre length which results from adding long pine fibre.

Composites reinforced with a blend of 20 % bamboo and 80 % of wood fibre (by weight) had similar strength values than those reinforced with wood fibre alone. The reason for this is not clear. This could be attributed to experiment error. Coutts and Warden (1985) demonstrated that wood fibre reinforced cement composites could achieve a flexural strength of about 30 MPa at 8% fibre content. Their results are in good agreement with strength developing trend shown in this work (dot lines shown in Figure 5.3).

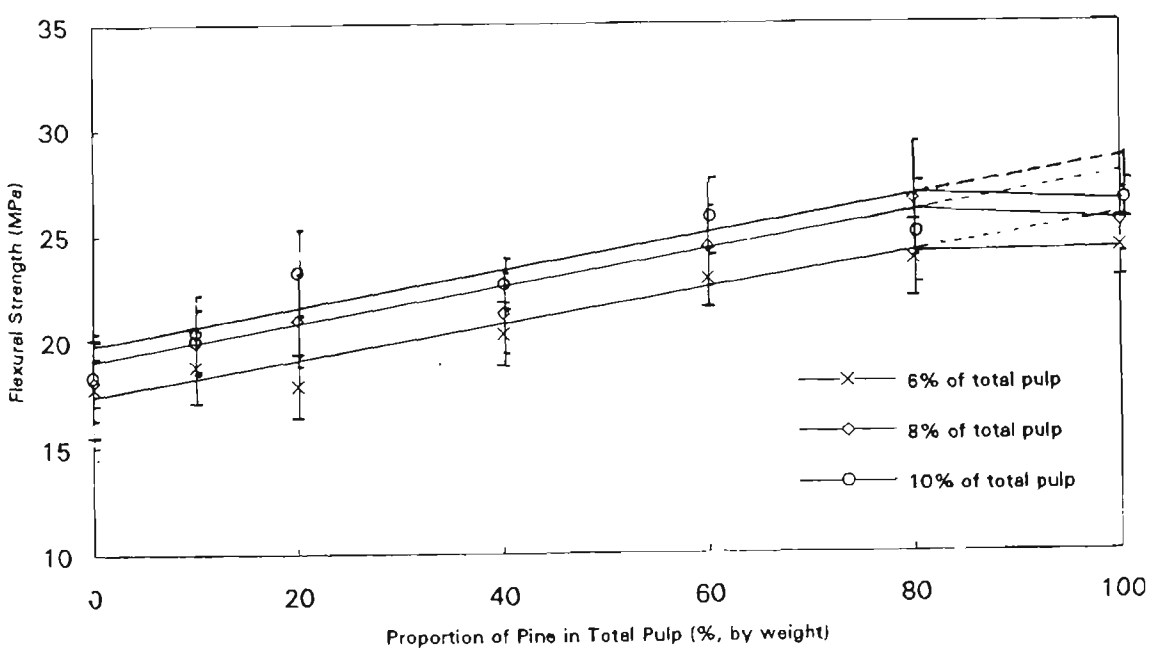

Fig. 5.3. Influence of long fibre (pine) proportion on the air-cured composites flexural strength.

Short fibre blended with some long fibre will greatly improve the pulps reinforcing ability. The influence of such reinforcing ability on the composite fracture toughness property is

more pronounced than on the strength property. If the fibre is longer, more energy will need to be consumed to pull the fibre out from the matrix, after fibre-matrix bond is failure; thus, improves composite fracture toughness. Figure 5.4 shows that fracture toughness value of composite increases very rapidly with increasing of long softwood fibre proportion in the reinforcing fibres.

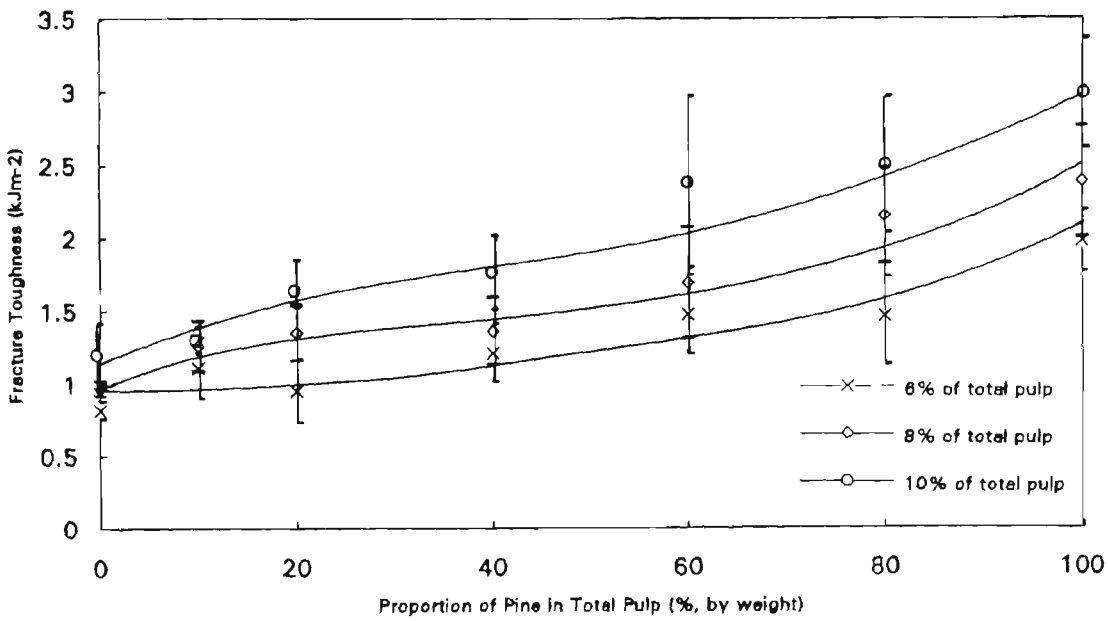

Fig. 5.4. Influence of long fibre (pine) proportion on the air-cured composites fracture toughness.

Influence of long fibre proportion on the strength and toughness properties of composite can be understood in terms of fibre length. The relationship between fibre length and composite flexural strength or fracture toughness is shown in Figure 5.5 and 5.6, respectively. The observation will be seen more clearly in Chapter 7. In that instance, studies on the composites reinforced with various length modified pulps show the significance of fibre length to the composites strength and toughness development.

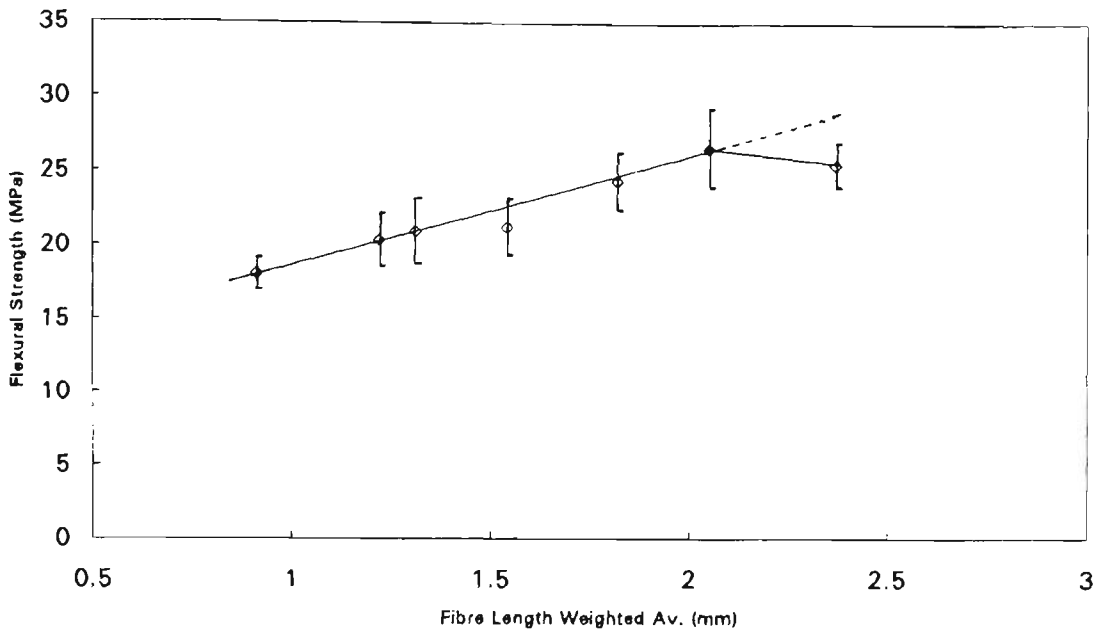

Fig. 5.5. Influence of fibre length on the air-cured composites flexural strength at total 8% fibre content.

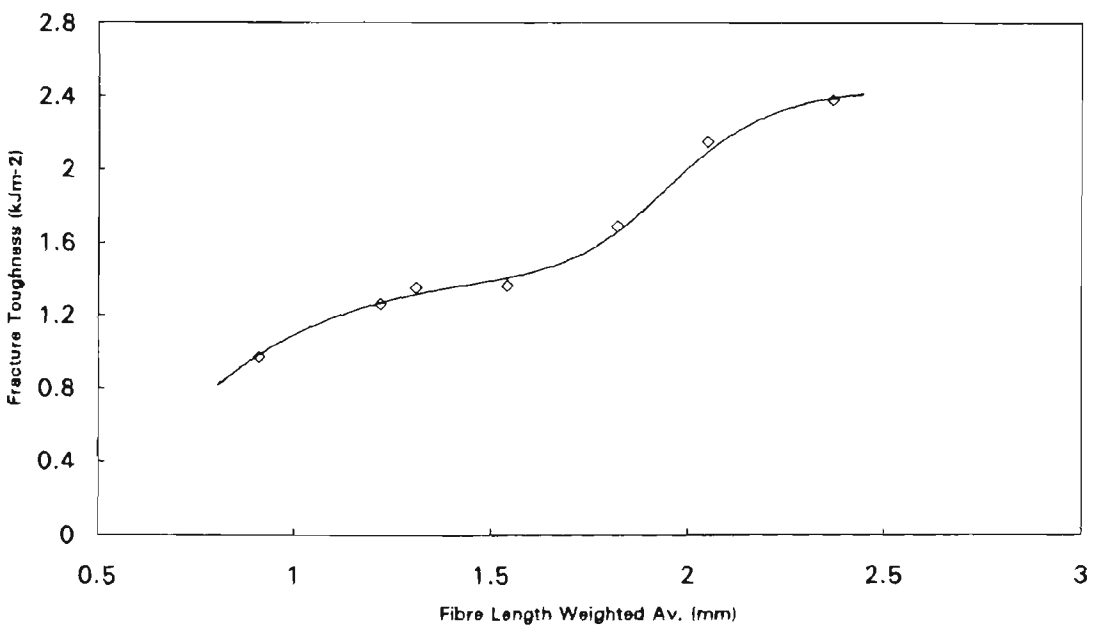

Fig. 5.6. Influence of fibre length on the air-cured composites fracture toughness at total 8% fibre content.

The changing of wood fibre proportion of the constituent hybrid fibre is not expected to affect composite physical property such as void volume, density and water absorption.

Table 5.2 shows fair constant value of composites physical properties if the total reinforcing fibre content remains the same.

This work is very important for utilising natural fibres as reinforcing material in fibre-cement products. Most natural plant fibre (including some wood fibre) do not have good reinforcing potential due to their short fibre length. As discussed above, short fibre blended with certain proportions of long fibre such as Kraft pine would increase the average fibre length thus significantly improve its reinforcing ability. One of the weaknesses of waste paper is its unsatisfactory length distribution when utilised as a reinforcing material (Coutts, 1989). This weakness could be overcome by adding a certain amount of long fibre pulp. On the other hand, one would modify the products properties by adding cheap short fibre to reduce the cost of fibre production.

In WFRC manufacture practice, natural fibre (eg. softwood) requires some degree of refining (beating) to enhance its reinforcing ability, more importantly to control the drainage rates of processing liquids during the fabrication of products (Coutts, 1982a). Unrefined (unbeaten) wood fibre has very high freeness value about 700- 800 CSF. Refining can reduce this freeness down to 500-550 CSF, which is preferred for the manufacture process. Such an effect could also be achieved by blending long fibre with short fibre without refining (see Table 5.1 and Figure 5.2). It might be possible to reduce the reinforcing cost by means of fibre blending. However, this idea needs further work to confirm in both laboratory and pilot-plant scale before it is adopted by the industry.

5.2.3 Autoclaved BWFRC composites

As in the case of the air-cured BWFRC, the strength property of autoclaved BWFRC composites also increased with an increase of long wood fibre proportion as seen in Table 5.3 and Figure 5.7, 5.8. However, strength improvement was not as high as that in the case of air-cured BWFRC. Strength of air-cured composites improved from 18.1 MPa reinforced with bamboo fibre alone to 26.6 MPa reinforced with blended 80 proportion of wood fibre at 8% of total fibre content, about 47 % increase. But in autoclaved

composites only a 37% increase of flexural strength can be observed. Over the range of fibre loadings studied there was little change in the strength properties of a given hybrid formulation. However, as the pine fibre content increased there was a gradual increase in composite strength.

Table 5.3 Properties of autoclaved BWFRC

Fibre (w%)	MOR (MPa)	Frac. Tough(kJ/m2)	Void Vol (%)	Water Abs.(%)	Density (g/cm^3)
100B+ 0P					
8%	13.8±1.1	0.27±0.02	43.8±0.8	32.5±1.5	1.35±0.04
10%	14.5±1.5	0.37±0.04	45.6±1.1	35.3±1.6	1.29±0.03
12%	14.1±1.2	0.48±0.04	46.9±1.1	38.2±1.8	1.23±0.03
14%	13.7±2.1	0.54±0.11	48.6±1.1	41.4±2.3	1.17±0.04
90B + 10P					
8%	14.6±1.1	0.42±0.07	43.9±1.7	33.5±2.3	1.33±0.04
10%	15.3±2.6	0.61±0.10	46.4±1.9	36.1±2.7	1.29±0.05
12%	16.4±2.0	0.80±0.07	46.9±1.6	38.9±2.7	1.21±0.04
14%	15.4±2.3	0.95±0.13	47.8±1.0	40.5±2.0	1.18±0.03
80B + 20P					
8%	15.5±0.8	0.47±0.04	44.9±1.5	33.4±2.1	1.34±0.04
10%	16.7±1.9	0.63±0.08	45.6±1.7	35.3±2.2	1.29±0.04
12%	16.9±1.1	0.84±0.15	47.1±1.0	38.3±1.7	1.23±0.03
14%	16.5±1.3	1.08±0.15	48.8±1.1	41.7±2.6	1.17±0.05
60B + 40P					
8%	17.2±1.2	0.65±0.07	42.8±2.2	31.7±2.5	1.36±0.04
10%	17.6±1.5	0.97±0.15	44.4±2.0	34.2±2.5	1.31±0.04
12%	17.7±2.3	1.17±0.20	46.0±2.2	36.9±2.8	1.25±0.04
14%	17.8±1.8	1.50±0.18	48.4±1.7	40.3±2.2	1.20±0.03
40B + 60P					
8%	16.5±1.5	0.77±0.10	44.3±1.2	32.5±1.6	1.34±0.03
10%	18.0±1.4	1.09±0.11	45.3±1.3	35.7±1.8	1.27±0.03
12%	19.3±1.5	1.28±0.18	46.3±1.4	37.0±2.0	1.25±0.03
14%	18.5±2.5	1.69±0.10	47.6±1.2	39.9±2.0	1.19±0.03
20B + 80P					
8%	18.6±3.6	0.95±0.30	42.8±2.1	31.1±2.4	1.37±0.04
10%	20.4±2.9	1.19±0.13	45.8±1.2	35.0±1.6	1.29±0.03
12%	19.8±2.5	1.55±0.21	45.9±2.1	36.2±2.9	1.25±0.04
14%	20.1±4.6	1.87±0.50	47.8±1.9	39.6±2.7	1.20±0.04
0B + 100P					
8%	19.9±2.8	1.58±0.37	42.4±1.4	31.6±1.7	1.34±0.03
10%	20.6±3.1	1.90±0.32	43.1±1.8	33.6±2.0	1.27±0.03
12%	20.9±3.3	2.75±0.40	44.0±1.4	35.2±1.8	1.25±0.03

*BWFRC composites were fabricated using ordinary Portland cement and silica at the ratio of 1:1, autoclaved at 1.25MPa steam pressure for 7.5h, tested at 50±5 per cent RH and 22±2^{o}C. 3 standard deviation, sample size n=9.

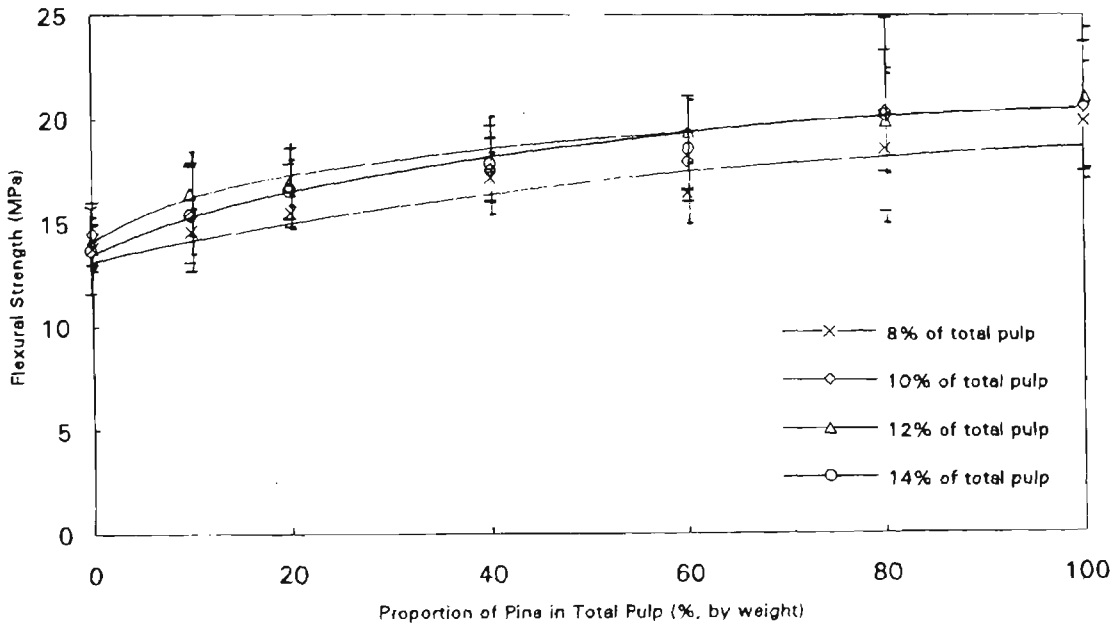

Fig. 5.7. Influence of long fibre (pine) proportion on the autoclaved composites flexural strength.

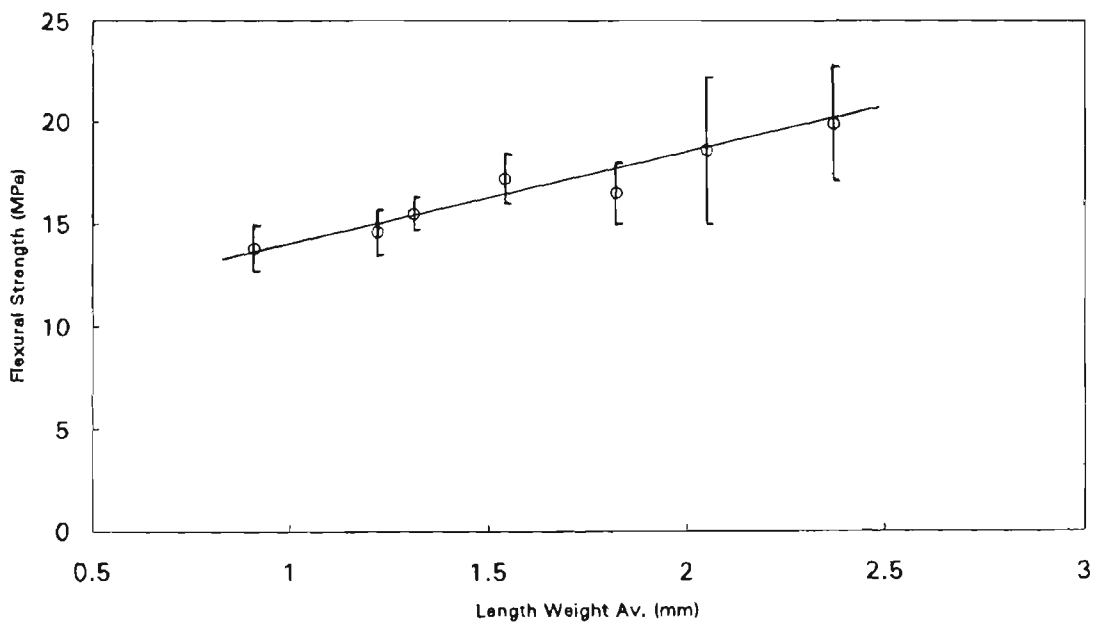

Fig. 5.8. Influence of fibre length on the autoclaved composites flexural strength at total 8% fibre content.

Fracture toughness value of autoclaved hybrid composites increased very rapidly with increasing long fibre (wood) proportion as seen in Figure 5.9 and 5.10. This behaviour has already been observed in the case of air-cured composites. Fracture toughness values of

the composites increased over 5-fold as the pine fibre was increased in the hybrid fibre formulation. In contrast to strength values, the fracture toughness values were seen to increase, for any given hybrid pulp, as the fibre content increased. This increase was of the order of twofold as the content increased from 8% - 14%.

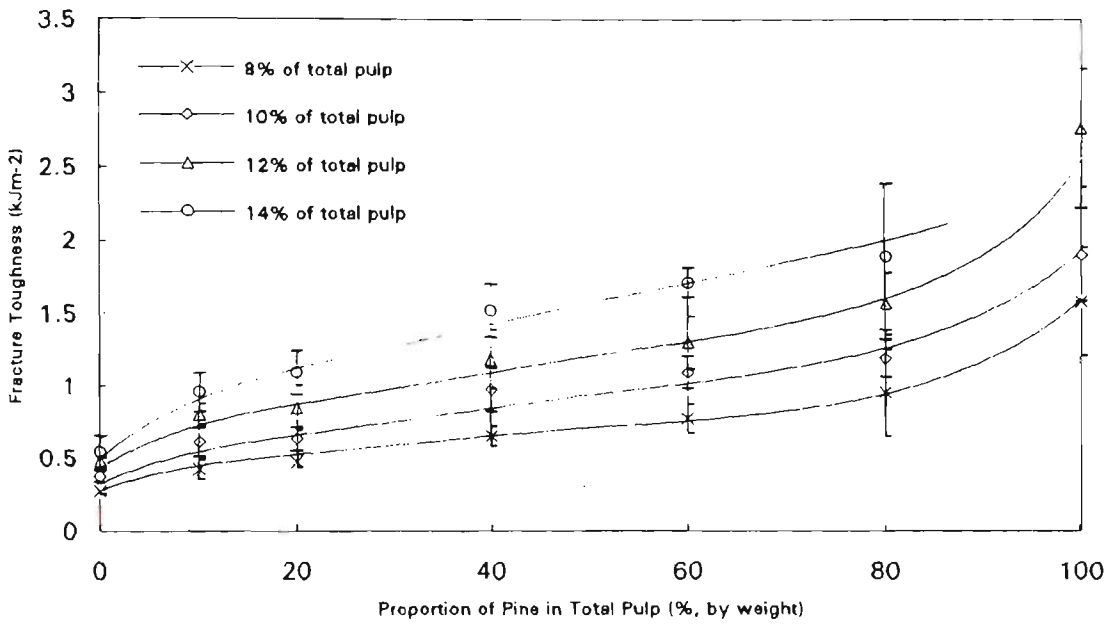

Fig. 5.9. Influence of long fibre (pine) proportion on the autoclaved composites fracture toughness.

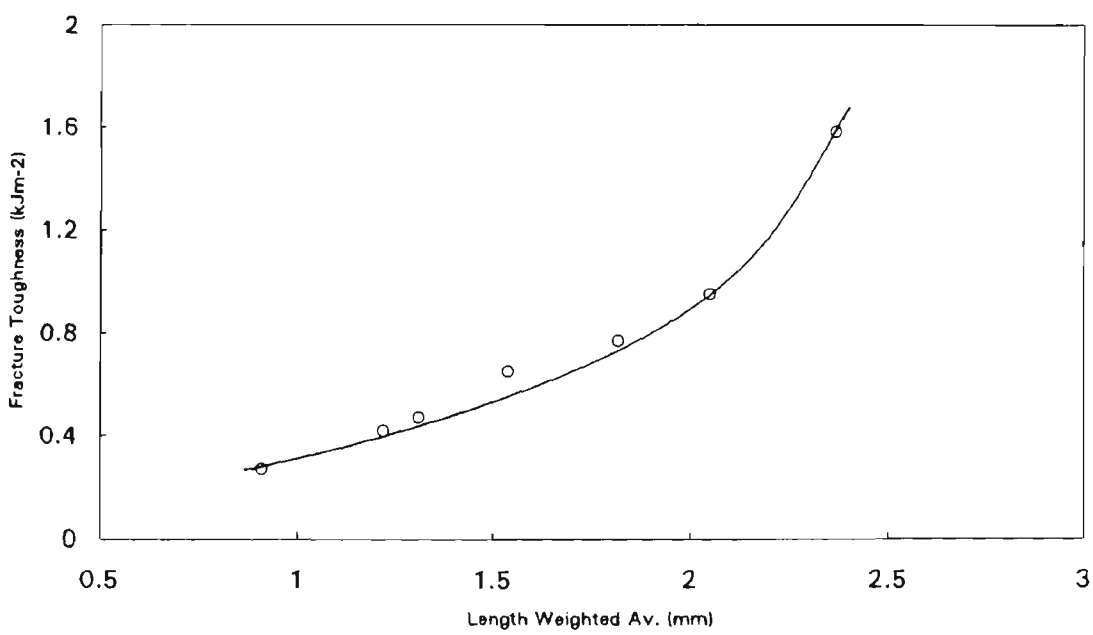

Fig. 5.10. Influence of fibre length on the autoclaved composites fracture toughness at total 8% fibre content.

Physical property of autoclaved products remained fairly constant at the same total reinforcing fibre contents. But the density values was about 18% lower than that of the air-cured counterpart. Water absorption and void volume always have reversed relationship with density. Hence, higher density samples would have low water absorption and void volume.

5.3 Theoretical predication and the experimental results

We have discussed early in section 2.1 that the composite strength and toughness can be calculated from the equations (2.4) and (2.14) respectively.

$$\sigma_b = [\alpha/\beta]\ \sigma_{mb} v_m + 0.82(\alpha\tau) v_f (l/d) \qquad (2.4)$$

$$R = 0.41 v_f l^2 \tau / 12d + [v_m + 0.41 v_f l / d] R_m \qquad (2.14)$$

Where α, β, τ, σ_{mb} and R_m are constants which can be determined experimentally. The fibre diameter d is suggested to remain constant for certain types of fibre. Thus at a constant fibre content v_f and v_m, the composite strength increases linearly, while the toughness increases as a second order polynomial with increasing fibre length. Refer to equations (5.1) and (5.2).

$$\sigma_b = K_{11}(l) + K_{12} \qquad (5.1)$$

$$R = K_{21}(l^2) + K_{22}(l) + K_{23} \qquad (5.2)$$

Where K_{11}, K_{12}, K_{21}, K_{22} and K_{23} are constants.

The experimental results support the above theoretical prediction. A simple regression analysis was performed on the data in Table 5.2 and 5.3. The results are presented in Table 5.4 which displays a relationship of fibre length, composite strength and toughness. The relationship is valid as the statistical multiple coefficient (R^2) is approximately 0.95.

Table 5.4 Regression analysis results

Fibre (w%)	MOR (MPa) Relation	R^2	Fract. Toughness (kJ/m^2) Relation	R^2
Air-cured				
6%	5.29 L + 12.35	0.917	$0.24 L^2 - 0.06 L + 0.71$	0.944
8%	5.74 L + 13.26	0.900	$0.25 L^2 + 0.15 L + 0.70$	0.970
10%	6.16 L + 13.48	0.830	$0.33 L^2 + 0.24 L + 0.70$	0.951
Autoclaved				
8%	4.12 L + 9.98	0.931	$0.50 L^2 - 0.81 L + 0.64$	0.973
10%	4.47 L + 10.43	0.947	$0.35 L^2 - 0.19 L + 0.30$	0.958
12%	4.49 L + 10.68	0.969	$0.89 L^2 - 1.56 L + 1.29$	0.947
14%	5.44 L + 8.98	0.977	$-0.45 L^2 + 2.53 L - 1.42$	0.990

Table 5.1 shows that the blended pulp fibre length increased almost linearly as the proportion of pine fibre increased. Therefore the composite strength increased almost linearly with increasing proportions of long fibre content. On the other hand, the composite fracture toughness improved as a second order polynomial function.

5.4 Conclusions

Bamboo pulp when blended with softwood pine pulp improves the furnish pulp's average fibre length and drainage ability. The improvement increased with increasing the proportion of wood pulp (long fibre).

The advantage of introducing the hybrid fibre was to improve and modify the composite properties. In this study increasing the proportion of wood fibre caused improvement of both air-cured and autoclaved composites mechanical properties, but had little effect on their physical properties. The improvement was minimal but more obvious in the case of the strength property of air-cured products than on the autoclaved counterpart. The

improvement in the property of fracture toughness was more pronounced than that of strength for the same formulations containing hybrid fibres.

Chapter Six:

Influence of Fibre Length on Composite Properties

The development of asbestos free fibre cement industry has made it most desirable to obtain definite information on the relationship between the nature of the natural plant fibres and their composite properties. It has been generally recognized that fibre length and fibre strength are two of the most important factors but, because various features of fibre morphology and chemical composition can influence composite properties, it has been difficult to obtain a clear picture of the effect of any one property.

Excellent work has been carried out in the field of paper science regarding fibre length and papermaking properties (Watson, 1961; Page, 1969; Seth, 1990). Apart from improving sheet formation, which indirectly affects many sheet properties, reducing fibre length has little direct influence on the structural and optical properties of the sheet. The major effect of decreased fibre length is on the strength properties; most are severely reduced. Thus, longer fibres will benefit all tensile properties, tearing resistance and folding endurance, particularly of weakly bonded sheets or sheets wet strength. However, fibre length can be less important if the sheets are well bonded, because the failure in the sheet can become controlled by the strength of the fibres.

Various methods have been used for achieving fractionation of fibre length and these methods are designed to isolate the effect of fibre length from that of other morphological and chemical factors. The usual method has been to separate a pulp into fractions of different fibre length by screening. A more direct approach was used by Brown (1932) in

which handsheets, formed with pulps from which the fines had been removed by screening, were cut into narrow strips by a sharp knife. The shortened fibres so obtained were reformed into handsheets; these had lower strength properties than the original handsheets.

Another method by which the effects of fibre length variation may be investigated is to prepare pulp from individual growth rings. This has been done by Watson for *P.radiata* (growth rings 2 to 12) (1952) and for *P.taeda* (growth rings 2 to 11, each separated into late and early wood) (1954). This procedure gave fibres of *P.radiata* ranging from 1.6 mm (growth rings 2) to 3.1 mm (growth ring 11), and for *P.taeda* from 2.2 mm (growth ring 2, late and early wood) to 3.2 mm (growth ring 11, late wood). An increase in fibre length produced an improvement in tearing strength but it was difficult to draw any definite conclusion regarding other strength properties. Variation in cell wall thickness as is found between early wood and late wood of one growth ring, also had a marked influence on strength properties.

Watson and Dadswell (1961) developed another technique by cutting delignified *Araucaria klinkii* chips into different lengths prior to pulping. The smaller size chip has more cutting ends thus its pulp has more short fibres. Again their work found longer fibre had much better tear strength than that of short fibre.

6.1 Experimental work

6.1.1 Fibre length fractionation work

The fibre was prepared from unbleached high-tear *P.radiata* dry lab pulp (APM, Maryvale mill, Australia). The lab pulp was soaked in water over-night, disintegrated and Valley beaten to 550 CSF freeness then subjected to fibre length fractionation.

Various fibre length fractions were prepared using the Bauer-McNett screening method, guillotined handsheets method and Wiley grinding method.

Weighed beaten pulp was first disintegrated on the British disintegrator for 500 counter revs and diluted to 0.2% concentration. The diluted pulp (about 10g o.d. weight) was poured into the Bauer-McNett fibre length classifier, equipped with four series screen compartments, for 20 minutes (10#, 30#, 50# and 80# US standard screen) (see Appendix A.2.3). Ideally, the apparatus is designed to produce four length fractions, however in this study only the fibres remaining on screen 10# and 80# were collected and used in the composites due to the insufficient separation of fractions by the screen technique. This procedure was repeated few times until the desired amount of pulp was obtained.

Handsheets made from beaten pulp were also used in the preparation of cut fibres. Dried handsheets were cut into 1.0 mm strips with a sharp guillotine. Strips were soaked in water over night, disintegrated for 1,000 rev. counts in the British disintegrator and used for composite fabrication.

Air-dried beaten pulp was ground in a Wiley mill to fibre length weighted av. 0.3 mm as the shortest fraction. Thus five fibre length fractions were ready for composite fabrication (2 from Bauer-McNett, 1 from guillotine, 1 from Wiley mill and 1 original).

6.1.2 Fabrication and characterisation

The fibre cement composite samples were produced by a slurry / vacuum dewatering and press technique, followed by air-curing up to 28 days (see section A.3.2). Each sample was based on a 130g dry weight of ingredients, ordinary Portland cement was used as matrix.

Mechanical and physical properties of the composites such as flexural strength (MOR), fracture toughness, void volume, water absorption and density were determined by the methods described in section A.4. The fibre length fractions were measured on a Kajanni FS-200 fibre length analyser reported as fibre length weighted average.

6.2 Results and discussion

6.2.1 Fractionation of fibre length

A single pulp source was separated into 5 length fractions using three methods and these length fractions are listed in Table 6.1. As mentioned before, the Bauer-McNett apparatus is designed to produce four length fractions. In our experience, however, it was unwise to utilise all four fractions due to the fact that diluted pulp fibre has high flexibility and some long flexible fibres are able to elude the small opening mesh (screen) barrier so that there is a considerable amount of overlap in the four fractions length population distributions as seen in Figure 6.1. Furthermore, fractionation not only separates the pulp into fractions of different length but also effects a separation between fine and coarse fibres.

Table 6.1 Fibre length fractions

Length Fraction (mm)	Experiment Technique
3.13	Bauer-McNett screening
2.66	original pulp length
1.59	cutting dry handsheets
0.74	Bauer-McNett screening
0.30	Wiley mill grinding

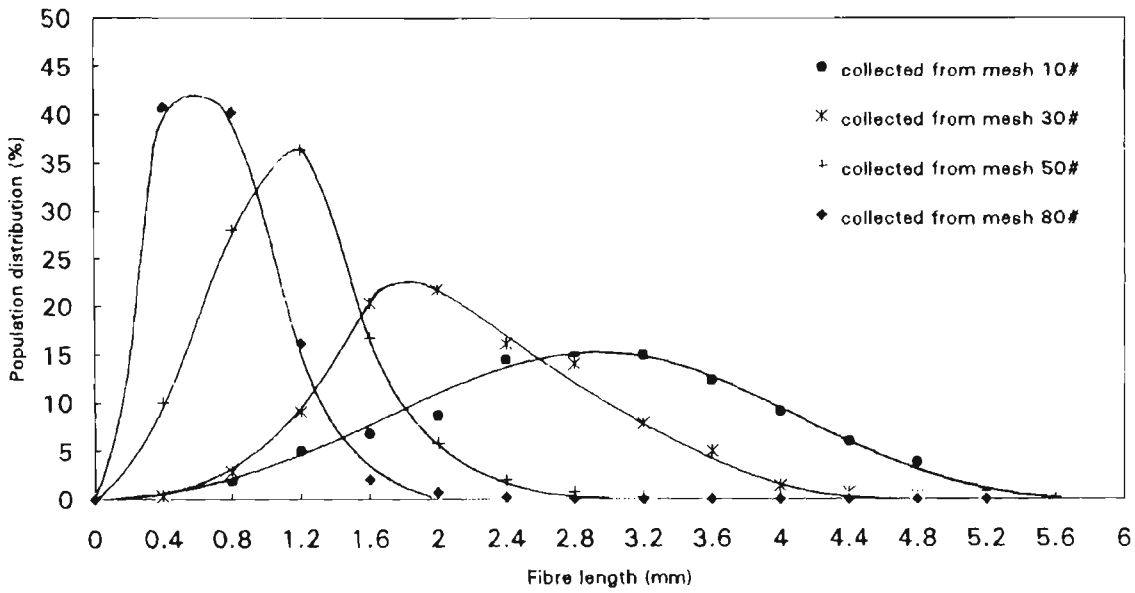

Fig. 6.1. Fibre length population of Bauer-McNett technique four length fraction.

Using a guillotine to cut handsheets can reduced fibre length to 1.59 mm from its original 2.66 mm. Attempts have been made to further reduce fibre length by employing a second cut to the cut fibre formed handsheets, however, it was not successful as the second cut only had very small amount of reduction in length compared to that from the first cut (e.g. first cut: 1.59mm and second cut 1.50mm).

Mean fibre length may not provide the best measure of effective fibre length in both paper sheets and composite products. The best measurement would be the fibre length distribution. It is quite possible two different population distribution pulps would have

same mean arithmetic fibre length (or even length weighted average). The observation used in paper science suggests that of two pulps with the same mean fibre length, the one with the greater proportion of long fibres will give the higher tearing resistance (Watson, 1961 and Colley, 1973). The influence of length population distribution to composite properties requires further investigation.

6.2.2 Influence of fibre length on composite mechanical properties

Table 6.2 Relationship between fibre length and air-cured composite performance.

Fibre (w%)	MOR (MPa)	Frac. Tough(kJ/m2)	Void Vol (%)	Water Abs.(%)	Density (g/cm^3)
L: 3.13 mm**					
2	18.0±5.1	0.33±0.03	33.3±0.7	18.4±0.6	1.81±0.02
4	18.6±3.3	1.07±0.34	33.6±1.0	19.7±0.6	1.70±0.03
6	25.9 ±2.3	1.68±0.28	33.0±0.9	20.3±1.0	1.63±0.03
8	27.4±2.7	2.39±0.40	34.6±0.4	22.3±0.4	1.55±0.01
10	30.4±3.3	2.89±0.61	35.0±0.8	23.3±0.8	1.50±0.02
L: 2.66 mm**					
2	18.8±1.1	0.38±0.10	29.2±0.4	15.64±0.3	1.86±0.02
4	20.4±1.9	0.87±0.12	30.5±0.3	17.46±0.3	1.75±0.02
6	23.4±2.3	1.35±0.24	32.6±0.9	19.84±0.8	1.65±0.03
8	24.3±1.9	1.87±0.19	33.4±1.2	21.57±0.8	1.55±0.05
10	28.1±3.1	2.80±0.45	34.6±1.0	22.47±1.1	1.54±0.03
L: 1.59 mm**					
2	16.2±1.1	0.22±0.02	29.5±1.0	15.9±0.7	1.86±0.02
4	18.1±2.1	0.59±0.07	29.4±0.8	16.8±0.6	1.78±0.02
6	20.9±1.8	0.89±0.11	30.6±0.6	18.1±0.3	1.70±0.05
8	22.3±0.7	1.37±0.17	32.4±0.6	20.2±0.6	1.60±0.02
10	22.6±2.3	1.68±0.28	34.3±0.8	21.9±0.6	1.57±0.06
L: 0.74 mm***					
2	13.3±0.7	0.24±0.03	32.9±0.5	18.3±0.4	1.80±0.01
4	17.6±3.7	0.58±0.19	34.2±0.4	20.5±0.4	1.67±0.01
6	22.4±0.9	0.91±0.10	35.4±0.7	22.5±0.8	1.58±0.02
8	23.6±2.5	1.23±0.27	36.7±0.6	23.1±0.6	1.55±0.02
L: 0.30 mm**					
2	13.6±1.0	0.07±0.01	28.9±0.6	15.2±0.4	1.90±0.01
4	14.3±0.5	0.09±0.01	29.9±1.0	15.5±0.7	1.82±0.02
6	14.1±0.7	0.13±0.02	31.8±0.8	18.4±0.6	1.72±0.02
8	14.3±1.3	0.18±0.01	33.3±0.5	20.5±0.6	1.62±0.02

* Pulps of fibre length weighted average 3.13 mm and 0.74 mm were generated from Bauer-McNett classifier screens; pulp of fibre length weighted average 1.59 mm was prepared from guillotined dry handsheets and pulp of fibre length weighted average 0.30 mm was made from Wiley mill. The initial pulp for this study was Freeness 550 CSF, length weighted average 2.66 mm and kappa number around 30 *P.radiata* Kraft pulp.

** 3 standard deviation, sample size n=9. *** 3 standard deviation, sample size n=6.

The results of effective of fibre length to air-cured composite mechanical and physical properties are shown in Table 6.2 and Figure 6.2 to 6.6. Fibre lengths between 0.3 mm to

3.13 mm were used at weight content between 2 per cent to 10 per cent and the comparison of properties was made at 28 days.

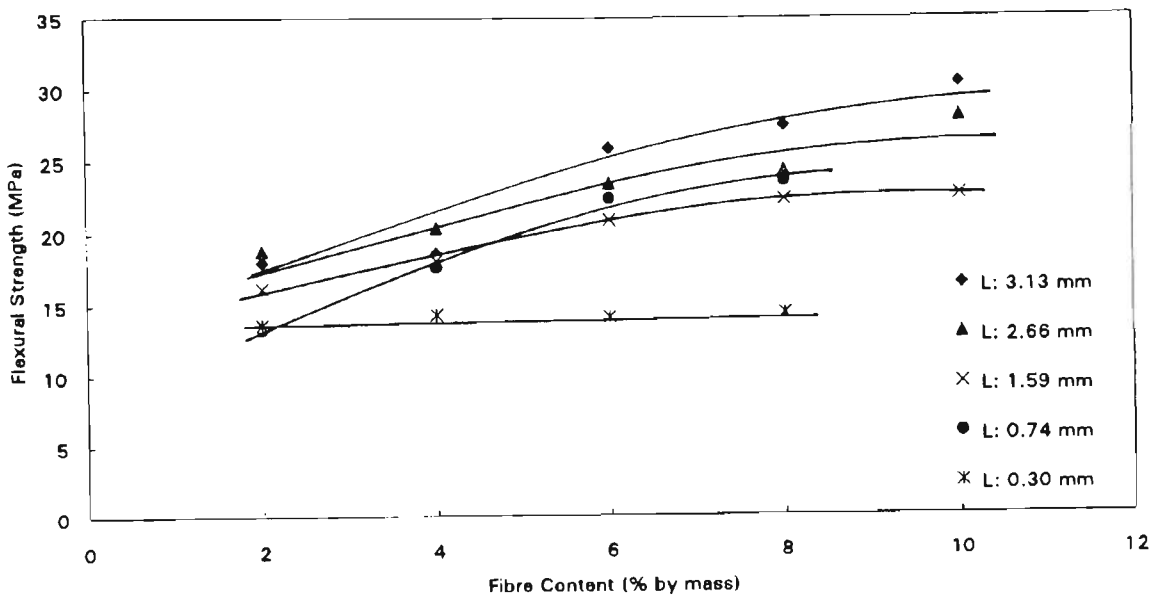

Fig. 6.2. Effect of fibre content on composite flexural strength for different fibre lengths.

Fig. 6.2 shows the effect of fibre content on flexural strength for different fibre lengths. Except the very short length fraction (L: 0.3mm), the higher strength of the composites with longer fibre fractions can be attributed to the increased length efficiency factor (see section 2.1.1). The longer fibre fraction had better strength property. The length contribution however was not that significant to the strength improvement. For example at 8% fibre content, with length fibres nearly doubled (1.59 mm and 3.13 mm) the composites strength values increased from 22.3 MPa to 27.4 Mpa, respectively.

The porosity of the composite increases as the fibre content increases and fibre length increases (see Table 6.2). This effect may be the cause of the reduction in flexural strength of some of the composites at fibre content greater than 8 to 10 per cent by weight.

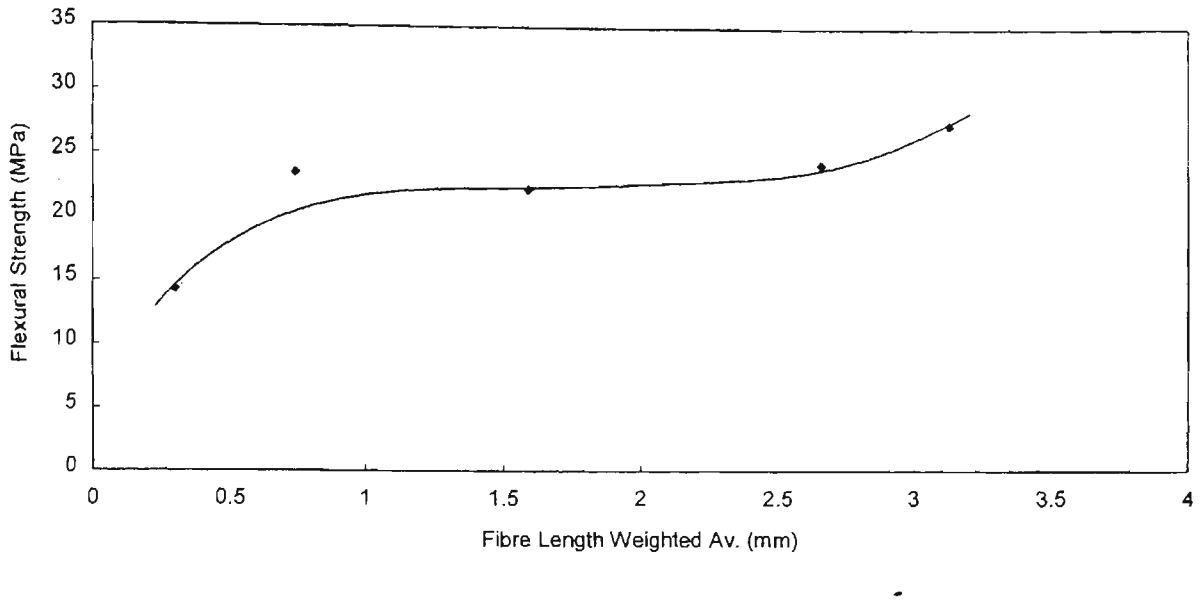

Fig. 6.3. Influence of fibre length on composite flexural strength at 8% fibre content

Again Figure 6.3 shows composite strength increased with fibre length increased at 8 % fibre by mass, although there were some small strength variations to composites made from fibre length 1.59 mm and 0.74 mm. This might be explained by the fact that mean fibre length may not be the best measure of effective fibre length. The 1.59 mm length fraction might have a greater percentage of fines than the 0.74 mm fraction (due to different fractionation techniques) and such fines do not contribute to composite strength development (see results of the 0.3 mm fraction). Density results in Table 6.2 and Figure 6.7 seen to support the above argument as composites made from the 1.59 mm fraction are denser than those from the 0.74 mm fraction.

Fibre length contributes greatly to composite fracture toughness development. Over the whole range of fibre contents, the longer fibres have higher values of composite toughness (Fig. 6.4 and 6.5). The length contribution to the fracture toughness is much greater than that to the flexural strength.

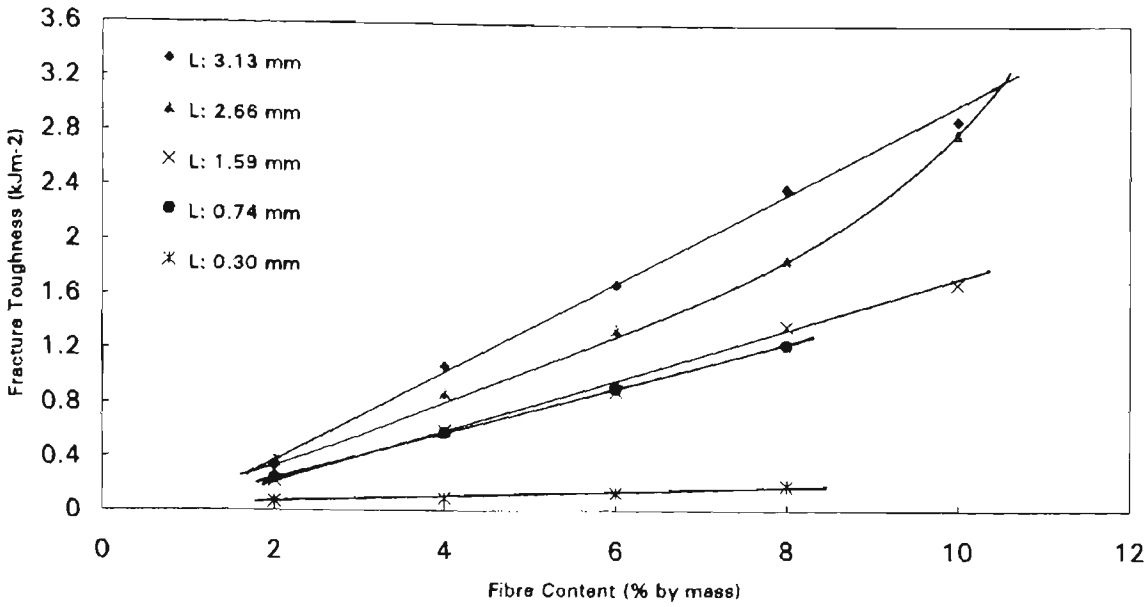

Fig. 6.4. Effect of fibre content on composite fracture toughness for different fibre lengths.

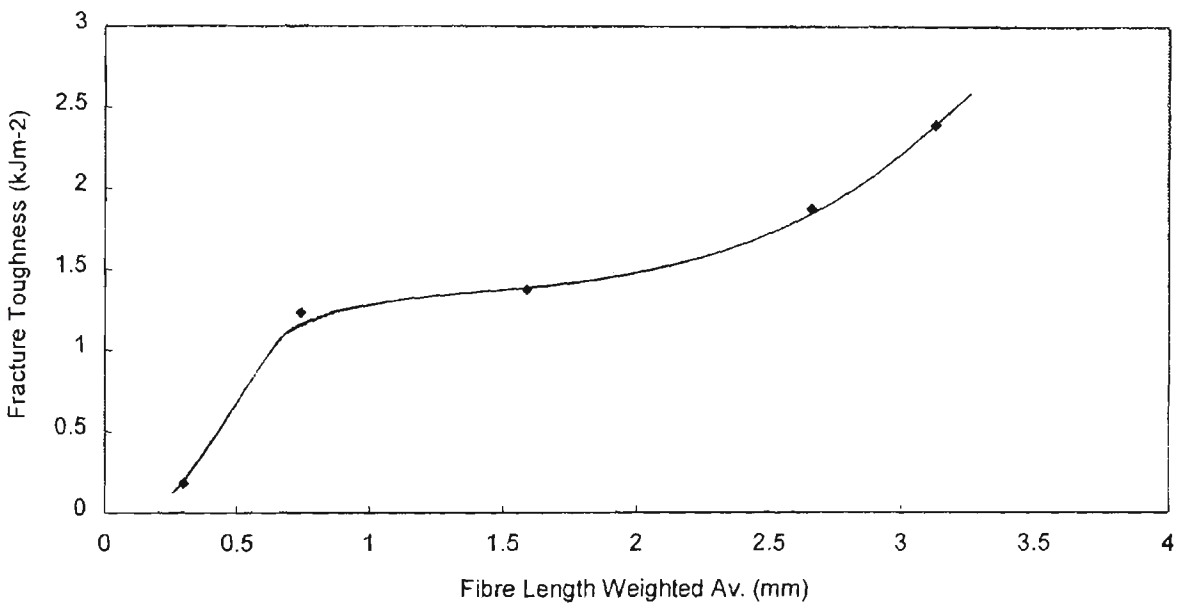

Fig. 6.5. Influence of fibre length to fracture toughness at 8% fibre content.

The mechanism that takes place when a fibre reinforced composite is loaded to failure include fibre fracture and fibre pull-out. If the fibre is strong or fibre-matrix bond is weak, the pull-out mechanism can have considerable influence on the value of fracture toughness. The longer the fibre the greater the energy required to pull the fibre through

the matrix, after the fibre to matrix bond is broken and the greater the contribution to the dissipation of energy contained in the advancing crack.

The same proposition was developed by the Institute of Paper Chemistry (Staff of IPC, 1944) on the studies of the influence of fibre strength to paper tearing resistance. The tearing resistance is the sum of the work done in breaking some fibres and the work done in pulling the remainder out of the fibre mat. The maximum tear is obtained at a degree of interfibre bonding such that the greatest number of fibres required a force just short of their breaking load to pull them free, while the level of the maximum depended on fibre length as the parameter controlling the distance over which that force is applied.

Studies of synthetic fibres such as steel, polyproylene, glass and more recently kevlar and carbon reinforced cement based composites led to the same conclusion that composites reinforced with long fibre have better strength and toughness properties. The area under a 3-point bend load-deflection curve is often described as a measure of the toughness or energy absorbing capability of the material. Various values for toughness can be calculated for the same curve depending upon whether the complete load-deflection curve is used including the descending portion or whether a cut-off point is chosen. From the point of view of serviceability of a structural unit a more meaningful value for toughness can be obtained from the area up to the maximum load or up to a specified deflection depending on the degree of cracking allowed in service. Hannant (1978) in his book *Fibre Cement and Fibre Concretes* reported that Johnston (1975) surveyed the data for the complete load-deflection curve and also for the area up to the maximum stress (Fig. 6.6). It can be seen that the ability of a composite unit to absorb energy is substantial and

increases with fibre content and fibre length (or with length and width aspect ratio), even if the cut off point is taken at the maximum stress.

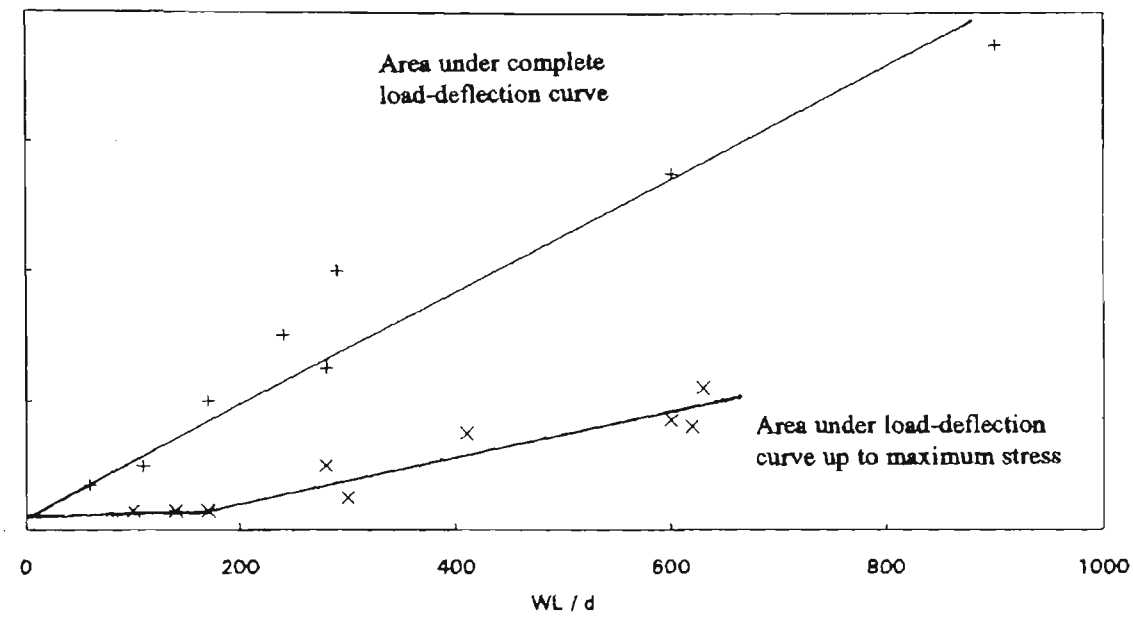

Fig. 6.6. Influence of fibre WL / d on composite fracture toughness. Where W = fibre content by weight x 100, L = fibre length, d = fibre diameter (Hannant, 1978).

The interesting finding in this work is that very short fibre (fragments) say, with length less than 0.3 mm, act more as a filler-diluent then as a reinforcing fibre when used to make composite materials. So a greater mass of pulp is needed to have sufficient number of long fibres in the high proportion short fibre pulp reinforced composites such as bamboo fibre reinforced cement and waste paper fibre reinforced products (Coutts, 1994a and 1989).

Due to the variation of wood fibre diameter (early wood and late wood) and the lack of fibre diameter (coarseness) values no attempt is made to determine fibre aspect ratio influence. Further, no definite critical fibre length value has been concluded in this study because there is a wide length gap between 0.3 mm and 0.74 mm. Further work is needed

to resolve these unanswered questions, however, the general picture of the effect of fibre length is obvious.

6.2.3 Theoretical and experimental conflict in results

We have discussed early in section 2.1 that the composite strength can be calculated from the equation (2.4) when the fibre content is less than 8% by mass.

$$\sigma_b = [\alpha/\beta]\ \sigma_{m,b} v_m + 0.82(\alpha\tau) v_f (l/d) \qquad (2.4)$$

Where the α, β, τ, $\sigma_{m,b}$ are constants which can be determined from experiments (Andonian, 1979). Furthermore, the fibre diameter d is suggested to be constant for a certain type of fibre. Thus at a constant fibre content v_f and v_m, the composite strength increases lineally with increasing fibre length [equation (6.1)].

$$\sigma_b = K_1(l) + K_2 \qquad (6.1)$$

Where K_1 and K_2 are constants.

However, the experimental results from this study do not support the theoretical equation (6.1). A simple regression analysis of the data presented in Table 6.2 leads to a relation (6.2).

$$\sigma_b = 3.3(l) + 16.9 \text{ (at 8\% fibre content)} \qquad (6.2)$$

The relationship shown in equation (6.2) is statistically unacceptable since the statistical multiple coefficient (R^2) is only about 0.66 which is very low compared to the theoretical estimation of approximately 0.99.

The non-conformability of experimental results with the underlying theory could be attributed to the theoretical assumptions made. The fibres are pulled out of the cement matrix instead of broken at failure. And, the fibres have uniform diameter, the fibre-matrix interfacial bond strength is constant. The natural fibre, however, violates these assumptions.

The fracture mechanics concept as discussed in Chapter 2 can not be used to correlate the experimental results obtained in this study. If LEFM has to be valid, the stress intensity factor, K_c in general and the fractural toughness, K_{Ic} in particular have to be defined in terms of crack geometry and crack location which can not be ascertained for the kind of composites used in this study, ie., the K_{Ic} is difficult to establish based on standard K_{Ic} test procedure.

6.2.4 Influence of fibre length on composite physical properties

The changing of fibre length and proportions of the constituent fibres and matrix affect void volume, density and water absorption (Table 6.2). Short fibre (0.3 mm) results in close packing of ingredients, thus the composites are more dense than those made with long fibre as showed on Figure 6.7. In general, long fibres cause formation and running ability problems in paper making and have "balling up" and drainage problems in cement products. However, the fact these problems did not appear might be due to greater control of the sample preparation at the laboratory level. There were some density variation for composites made from fibre length 0.74 mm and 1.59 mm fraction. This variation had caused composite strength inconsistency, the reason for this could be explained by the fibre length population distribution.

Water absorption and void volume change with density changes in keeping with the early results. If the composite is more dense, the amount of porosity in the composite and hence water absorption and void volume values are lower.

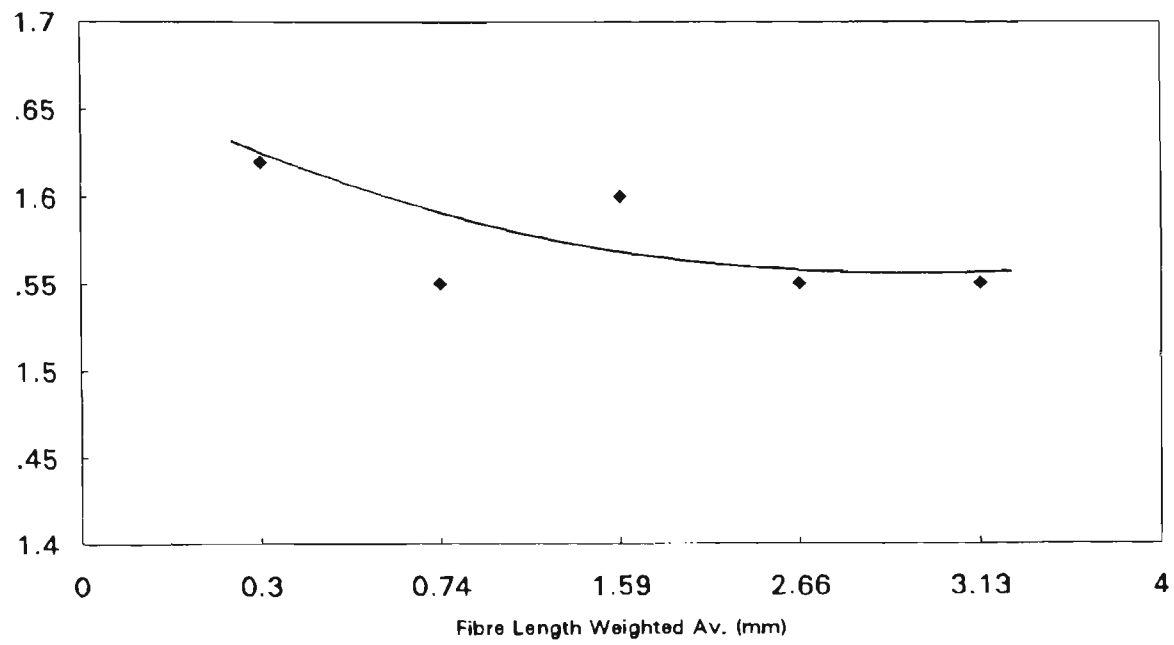

Fig. 6.7. Influence of fibre length on composite density.

6.3 Conclusions

A single *P.radiata* Kraft pulp has been divided into 5 different length fractions varying from 0.3 mm to 3.13 mm. Fibre length makes a major contribution to the mechanical and physical properties of air-cured cement composites. Both composite flexural strength and fracture toughness values increase with increasing fibre length over the range of fibre contents examined. The fracture toughness can be significantly improved with long fibres. The very short fibres (fragments) say, with length less than 0.3 mm, act more as a filler-diluent then as a reinforcing material when used to make composite materials.

Composites have lower water absorption, lower void volume and higher density value with decreasing fibre length. The conclusion concerning the effect of fibre length for natural fibres is in general agreement with the findings of the effect of fibre length for synthetic fibre in cement products although the composite strength doesn't increase lineally with fibre length increases.

Chapter Seven:

Influence of Fibre Strength on Composites Properties

In section 2.1.1 we discussed the "rule of mixture" with respect to composite strength which suggests that stronger fibre and higher fibre loadings lead to greater composite strength. The influence of fibre content has been studied by a number of people and the results suggest an optimum fibre content of about 8 - 10% by weight for softwood fibre (Coutts, 1979a and AU Patent 1981). As well as fibre content, there is a need to study natural fibre strength as a parameter affecting composite performance.

Lhoneux and Avella (1992) plotted a wide range of wood pulp fibre tensile strengths against the fibre-cement composites flexural strengths. A nearly linear relationship was observed and they suggested the stronger fibres provided better reinforcement than the weaker fibre. The results seemed in agreement with the theory of mixture rule; however, their work ignored the influence of other fibre factors which might also have impact on composite strength. For example, they ignored the influence of fibre length between the softwood and hardwood; ignored the influence of fibre chemical composition between the chemical pulp and mechanical pulp fibres; ignored the influence of fibre fibril angle and cross-dimension between different species.

The objective of this chapter is to investigate the influence of fibre strength on fibre-cement composite properties. Therefore, the experiment is designed to vary the fibre strength while maintaining constant as many fibre parameters as possible.

7.1 Experimental work

7.1.1. Fibre strength fractionation

As discussed before the key point of the experiment is to fractionate fibre intrinsic strength without changing other parameters (eg. fibre length and freeness). This could be achieved by means of cellulose degradation. Wood cellulose fibre can be reduced in strength by alkaline, oxidative, hydrolytic and thermal methods or combinations of these conditions which result in degradation reactions of cellulose. There are two methods used in this study to prepare degraded fibre, namely extensive alkaline cooking and liquid phase acid hydrolysis.

7.1.1.1 Extensive alkaline cooking method

The fibre used in this method was holocellulose fibre, which was prepared from Kraft pulped *P. radiata* (~2.6% lignin content) by chlorite deligninification method (see Appendix A.1.4). After delignification, the fibres were subjected to further alkaline (Kraft) cooking to produce fibres of various strengths. The details of cooking conditions and fibre strength, length and freeness values are reported in Table 7.1.

7.1.1.2 Liquid phase acid hydrolysis method.

Liquid phase acid hydrolysis method was applied to *P.radiata* Kraft pulp (~2.6% lignin). Each 100g of pulp was slushed in 7 litre water. 1 litre hydrochloric acid (HCl), 10M was added with stirring. The fibre was attacked by the acid for various periods of time, such as 50h, 70h, 140h and 528h. After each desired period, the pulp was filtered and washed free of acid.

7.1.2 Fibre quality evaluation

Fibre strength was evaluated as Zero-span wet (unbonded) tensile index (ZSTI) and the degree of polymerisation (DP). ZSTI was conducted on Pulmac Zero-span Tensile Tester. It is a standard measurement used in pulp and paper science to evaluate pulp fibre tensile strength (see Appendix A.2.5.2). Holocellulose fibre polymers are subjected to degradation during further alkaline cooking process. This degradation results in fibre strength loss and can be evaluated by means of the degree of polymerisation (DP). DP was determined using 0.5M cupriethylenediamine as a solvent and a capillary viscometer in this study (see Appendix A.2.5.1).

The fibre weighted average length and freeness value was measured on Kajaani FS-200 Fibre Length Analyser and Canadian Standard Freeness Tester, respectively(see Appendix A.2.3 and A.2.2). Fibre length and freeness are two important parameters associated with reinforced composites (see Chapter 6; Coutts, 1982). It was desirable to maintain the constant length and freeness while the strength was altered by chemical treatment.

7.1.3 Composite fabrication and evaluation

Composite specimen were produced by a slurry / vacuum dewatering and pressing technique with 8% (by mass) strength modified fibres as the method described in Appendix A.3. Both air-cured and autoclaved samples were made. In order to minimise the matrix and curing effect, composites reinforced with different strength fibres were cured at the same time to eliminate variations in the curing regime.

Mechanical and physical properties of composites such as flexural strength (MOR), fracture toughness, void volume, water absorption and density were determined by the methods laid down in Appendix A.4.

Specimens after fracture were straight pulled into parts and stored in the oven at 60°C over night to release moisture. The fracture surfaces were examined with a Philip XL 30 FEG scanning electron microscope.

7.2 Results and discussion

7.2.1 Fibre strength variation work

The properties of the fibres after chemical treatment are shown in Table 7.1 and Figures 7.1, 7.2 and 7.3. It can be seen from these results that the extensive cooking successfully reduced the holocellulose fibre strength to various values. With more intensive treatment the cellulose DP was reduced with reduction in viscosity from 16.27 mPs.s to 6.5 mPs.s; while the strength dropped from ZSTI 100 Nm/g to 44.0 Nm/g. In addition, there was a strong relationship between fibre zero-span tensile strength and viscosity [degree of polymerisation (DP)] as shown in Fig. 7.1, which was in general agreement with the findings of other workers (Gurnagul, 1992). The results also showed that such a Kraft cooking did not significantly change the other parameters of the fibres, such as length and freeness (Figure 7.2 and 7.3). It was noticed that the length of the weak fibre (ZSTI 44 Nm/g, 2.18 mm) was only slightly shorter than that of the strong fibre (ZSTI 100 Nm/g, 2.48 mm). However this small length variation would not be expected to have a great influence on the composite properties (see Chapter 6)

Table 7.1 Properties of *P.radiata* fibre after alkaline cooking

Further cooking condition	ZSTI (Nm/g)	Viscosity (mPa.s)	W. Length (mm)	Freeness (CSF)
holocellulose untreated	100	16.27	2.48	680
E.A13% 30 mins to 140°C	89.1	12.96	2.32	680
E.A26% 30 mins to 140°C	81.2	13.66	2.11	700
E.A26% 60min to,10min on 180°C	44.0	6.50	2.18	700

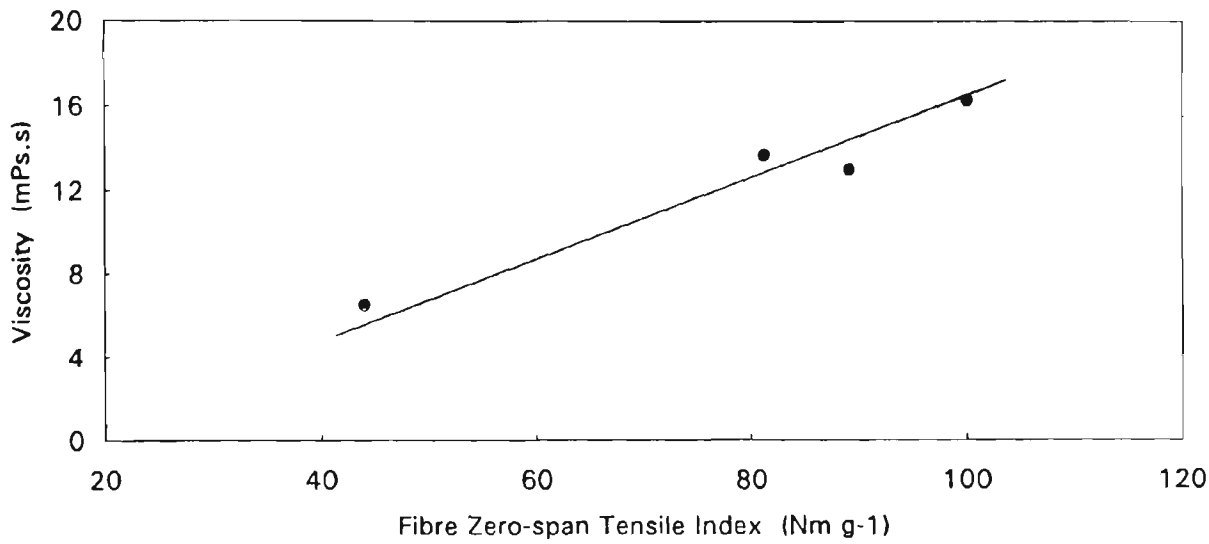

Fig. 7.1. Relationship between fibre zero-span tensile strength and pulp viscosity

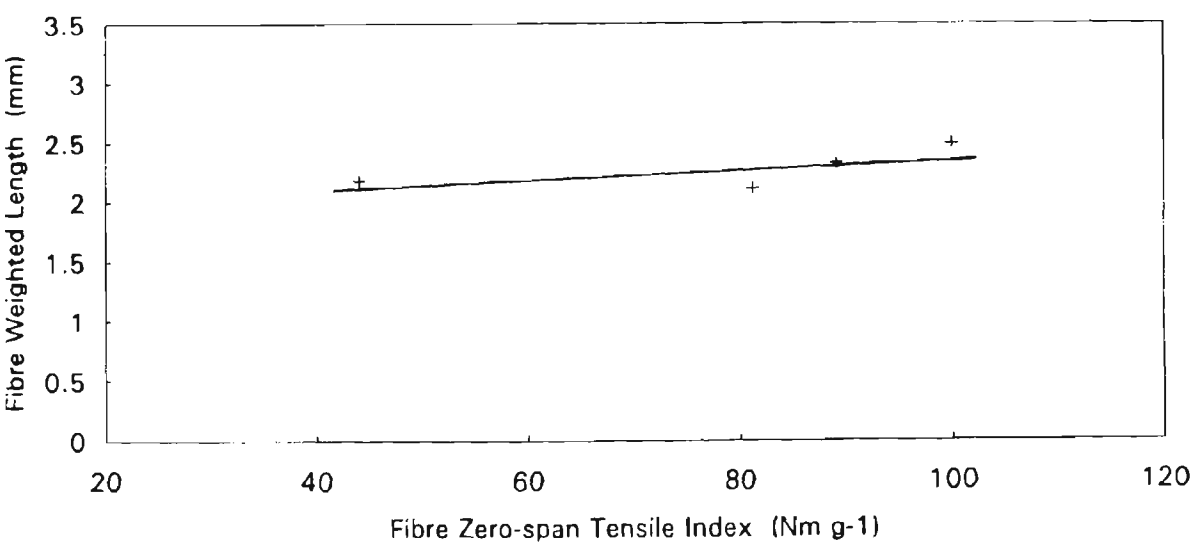

Fig. 7.2. Relationship between fibre strength and fibre length

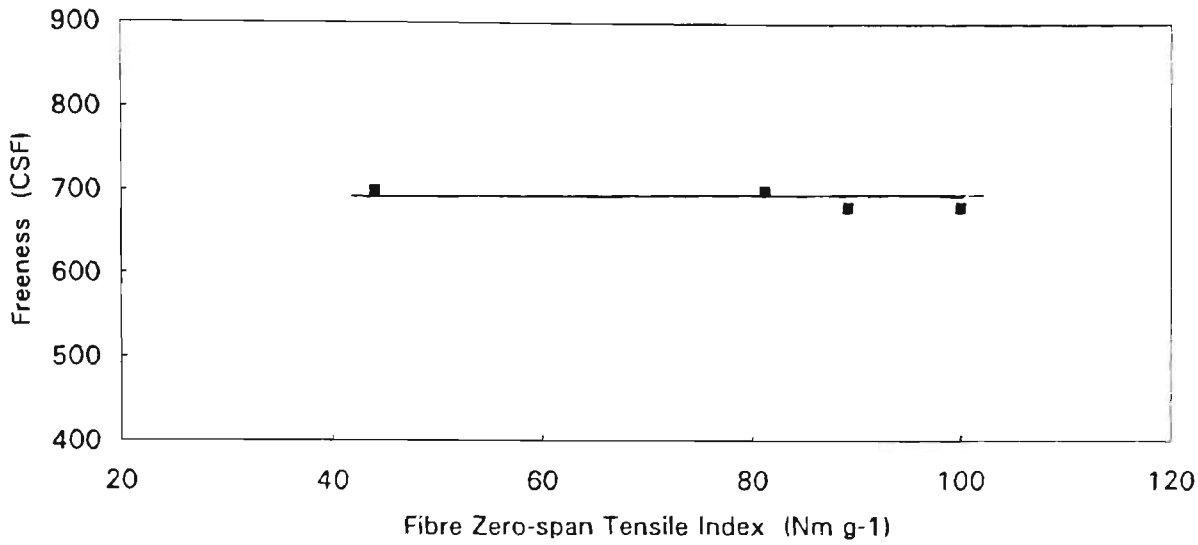

Fig. 7.3. Relationship between fibre strength and pulp freeness

Holocellulose fibre was treated with extensive alkaline cooking to produce pulps of various strength values. There were two reasons to use holocellulose fibre. Firstly these lignin-free fibres allowed further treatment to various fibre strengths without the complication of products with different lignin levels, which would affect the fibre strength determinations and composite fabrication. Secondly holocellulose fibre was found easier to subject to further alkaline degradation than any other mechanical or chemical pulped fibre. This may be related to the larger quantity of effective alkali (on a sample basis) in the holocellulose cooking, or to the influence of lignin on the pulping process or to the consumption of alkali (McKenzie, 1985).

Due to limited pulp source, the properties of pulp treated with hydrochloric acid were not determined. However, the evidence of fibre strength loss caused by acid attack was observed by a number of researchers (McKenzie, 1985 and Gurnagul, 1992). Gurnagul (1992) suggested that there were two types of degradation processes which control the extent of the strength loss. Homogeneous and random degradation causes little strength

loss, but localised degradation weakens fibre significantly. The above two treatments would possibly cause localised degradation resulting in a significant loss of fibre strength.

7.2.2 Influence of fibre strength on composites properties

7.2.2.1 Alkaline treated holocellulose fibre

Table 7.2 and Figure 7.4, 7.5 shows the variation of composite flexural strength when reinforced with fibres possessing a wide range of strength values. It is surprising that the composites flexural strength did not decrease significantly as fibre strength reduced from ZSTI 100 Nm/g to ZSTI 44 Nm/g in both air-curd and autoclaved WFRC specimen. The strength of the specimens reinforced with very weak fibre (ZSTI 44 Nm/g) started to drop off, but at the same time that point the fibre length was somewhat shorter which could have an effect on the strength. This observation was inconsistent with the rule of mixture of composites strength development.

Table 7.2 Composites properties reinforced with alkaline treated holocellulose fibres

ZSTI (Nm/g)	MOR (MPa)	Tough (kJ/m^2)	Void Vol (%)	Water Abs.(%)	Density (g/cm^3)
Air-cured WFRC					
100	25.1±1.3	2.40±0.39	32.9±1.0	20.7±1.1	1.59±0.03
89.1	24.0±2.2	1.90±0.28	33.3±0.2	21.1±0.1	1.58±0.01
81.2	25.6±1.3	1.42±0.26	32.3±0.9	19.9±1.0	1.62±0.03
44.0	21.4±1.5	0.90±0.14	31.7±0.5	19.2±0.6	1.65±0.02
Autoclaved WFRC					
100	19.9±2.9	1.62±0.51	42.5±0.8	30.9±1.2	1.37±0.03
89.1	21.4±1.7	1.46±0.44	41.9±0.5	30.4±0.7	1.38±0.02
81.2	21.1±1.3	0.92±0.19	41.8±1.2	30.3±1.5	1.38±0.03
44.0	18.6±1.2	0.51±0.12	42.1±0.8	30.5±1.0	1.38±0.02

* 3 standard deviation, sample size n=9.

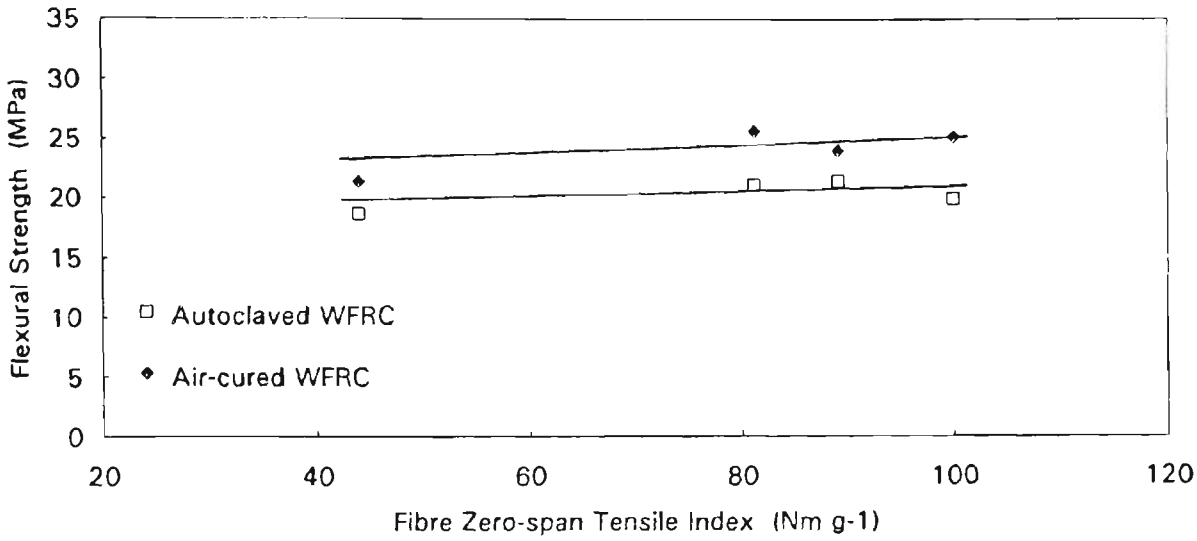

Fig. 7.4. Relationship between fibre strength (0-span) and composite strength

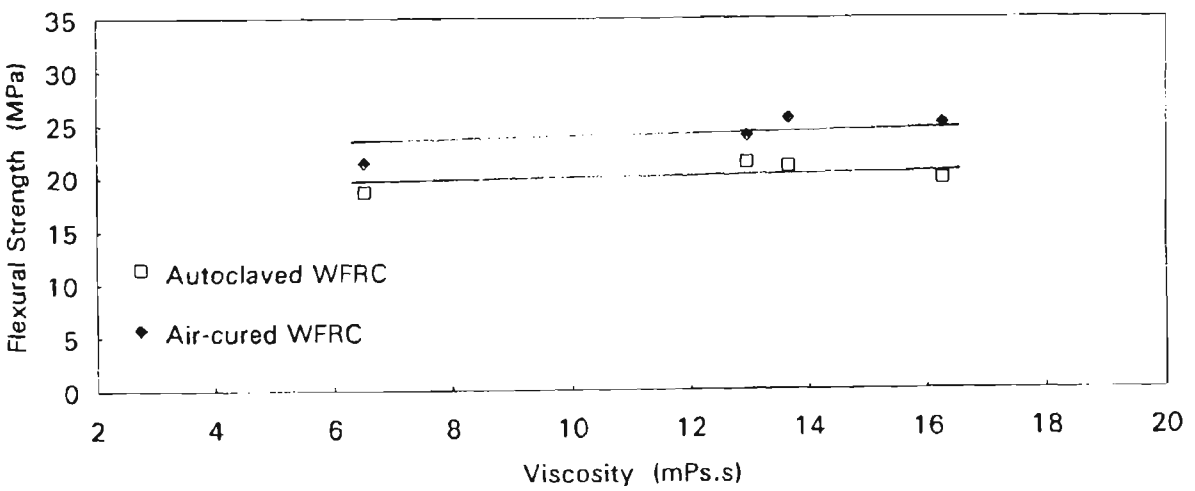

Fig. 7.5. Relationship between fibre strength (viscosity) and composite strength

The composite fracture toughness values decreased greatly when the reinforcing pulp contained weak fibres (Figure 7.6 and 7.7). At a fibre loading of 8% by mass and fibre strength decreasing from ZSTI 100 Nm/g to ZSTI 44 Nm/g, the composite fracture toughness values were reduced from 2.40 kJ/m^2 to 0.90 kJ/ for air-cured samples (about 62% reduction) and from 1.62 kJ/m^2 to 0.51 kJ/m^2 (about 69% reduction) for autoclaved samples, respectively. This result suggests that the weak fibres are not suitable as a

reinforcement for cement products, due to the composites lack of essential fracture toughness, even when having acceptable strength values.

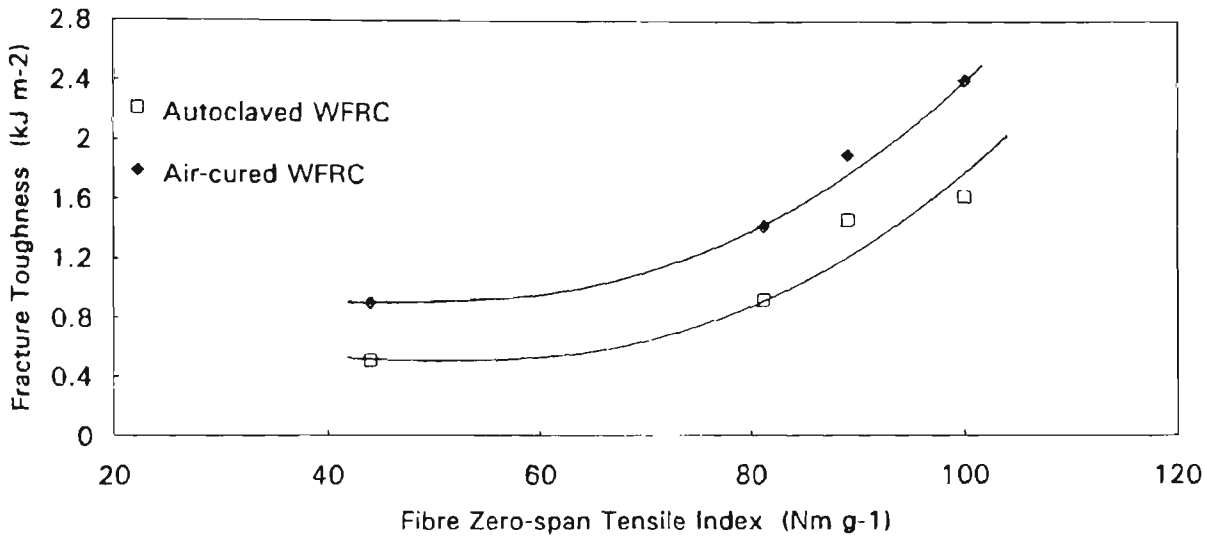

Fig. 7.6. Relationship between fibre strength (0-span) and composite fracture toughness

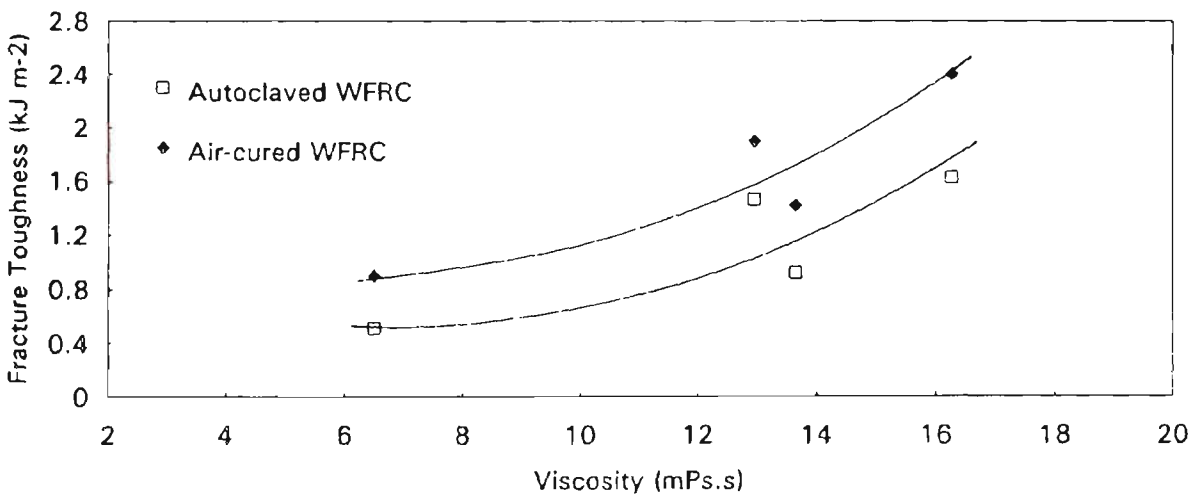

Fig. 7.7. Relationship between fibre strength (viscosity) and composite fracture toughness

The mechanisms that take place when a cement based fibre composite is loaded to failure include fibre fracture and fibre pull-out. The fracture toughness value is determined by a combination of these two mechanisms. If the fibre is weak, then the energy required to fracture the fibre is less than that to break the fibre-matrix bonds plus pulling the fibre out

of the matrix. Thus the weak fibre is likely to break before being pulled out and the composite is brittle.

The above explanation of fracture toughness was confirmed by examination of the composite fracture surface using scanning electron microscopy. When composites were reinforced with strong fibres, fibres were both fractured and pulled out during the fracture (Figure 7.8) and the specimen had an irregular fracture surface and high fracture toughness values resulted. Alternatively, composites reinforced with weak fibres showed a fracture surface that was flat, most fibres broke at the crack front and the crack could continue right through the matrix. The composite showed brittle behaviour and had low values of fracture toughness (Figure 7.9). In this study other factors such as fibre-matrix bond, chemical composition and fibre morphology were all constant, so the difference of composite properties were mainly caused by the difference of reinforcing fibre strength.

Fig. 7.8. Fracture surface of composite reinforced with strong fibre shows fibre pull-out.

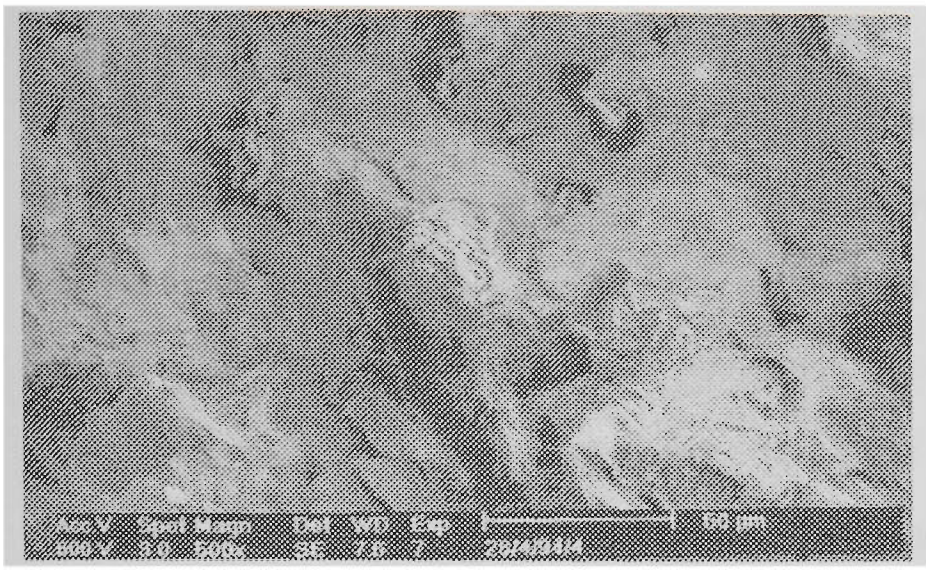

Fig. 7.9. Fracture surface of composite reinforced with weak fibre shows fibre fracture.

By contrast it is difficult to explain the behaviour of composite strength. It could be suggested, however, that for the high modulus cement based composites even a low percentage of low modulus fibres has an initial crack-stopping effect, but for further strength development a strong fibre and good fibre-matrix bond is required. In the current study the fibre strength (even ZSTI 100 Nm/g fibre) or fibre-matrix bonding might not be sufficient enough to produce further strength increasing. Thus the composites showed an initial strength which was similar.

The constant density, water absorption and void volume of composites were expected from the same matrix, fibre content and morphology. The slightly higher value of density and lower value of void volume and water absorption for the composite reinforced with weak fibre maybe due to the conformability which was caused by heavy degradation.

7.2.2.2 Liquid phase hydrochloric acid (HCl) treated fibre

The study of composites reinforced with hydrochloric acid treated fibre provided the same conclusion that weak fibres have little influence on the composite strength but can significantly reduce the composite toughness (see Table 7.3 and Figure 7.10, 7.11). In this instance, cellulose degradation was not as easy as that for the holocellulose. The results showed that both composite strength and toughness values were not greatly affected although the fibre had been attacked by acid for more than 140 hours. But after 528 hours treatment, composite strength and toughness were reduced from 22.3 MPa to 19.1 MPa and from 0.79 kJ/m^2 to 0.39 kJ/m^2, loss about 14% and 51%, respectively.

Extensive discussion in the next chapter will show that the same source of fibre pulped to different Kappanumber (lignin level) can cause composites to have various toughness values (see Chapter 8). This study will help us to better understand the mechanics of the composite fracture, because different Kappa number pulp has been strongly associated with its fibre strength (Page, 1985).

Table 7.3 Composites properties reinforced with acid hydrolysis fibres

Fibres	MOR (MPa)	Tough (kJ/m2)	Void Vol (%)	Water Abs.(%)	Density (g/cm^3)
Un-treated	22.3±1.8	0.79±0.08	30.6±1.2	42.5±1.0	1.39±0.02
50 hours treated	21.7±2.3	0.77±0.09	31.6±0.6	42.7±0.5	1.36±0.06
70 hours treated	20.8±1.6	0.87±0.10	28.9±0.7	40.2±0.6	1.39±0.01
140 hours treated	20.9±1.6	0.77±0.13	28.7±0.9	40.2±1.2	1.40±0.02
528 hours treated	19.1±1.4	0.39±0.03	26.1±1.3	37.8±1.2	1.45±0.03

* 3 standard deviation, sample size n=9.

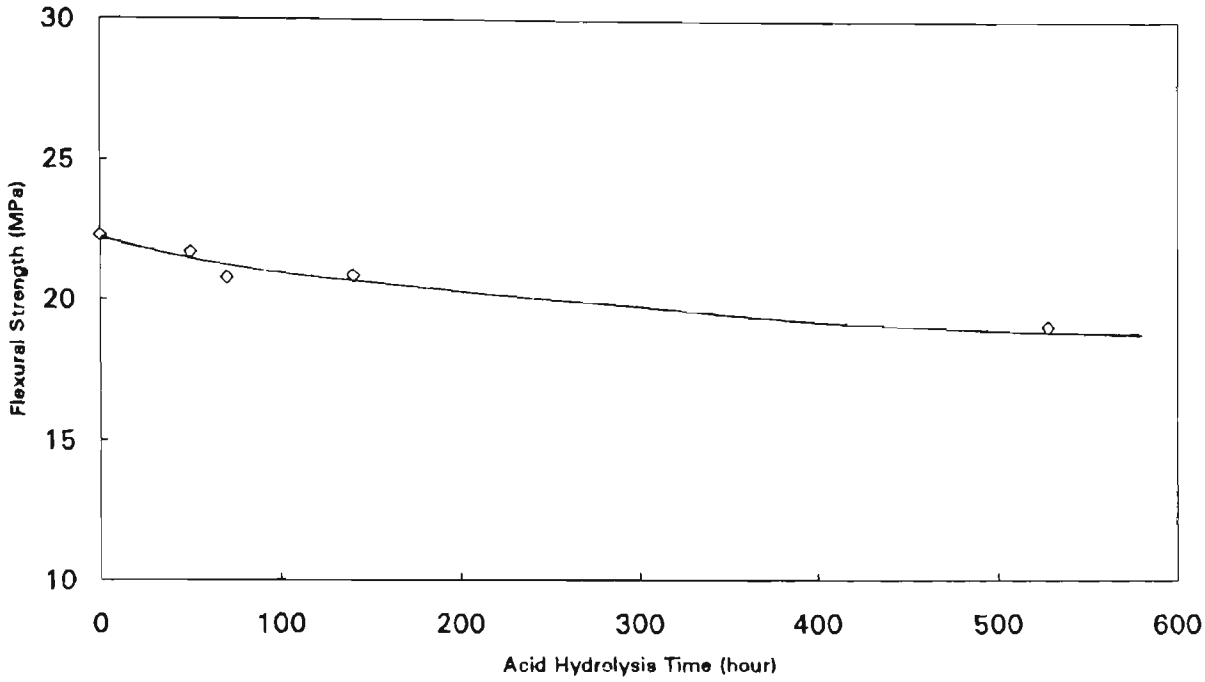

Fig. 7.10. Relationship between fibre acid treated time and composite strength

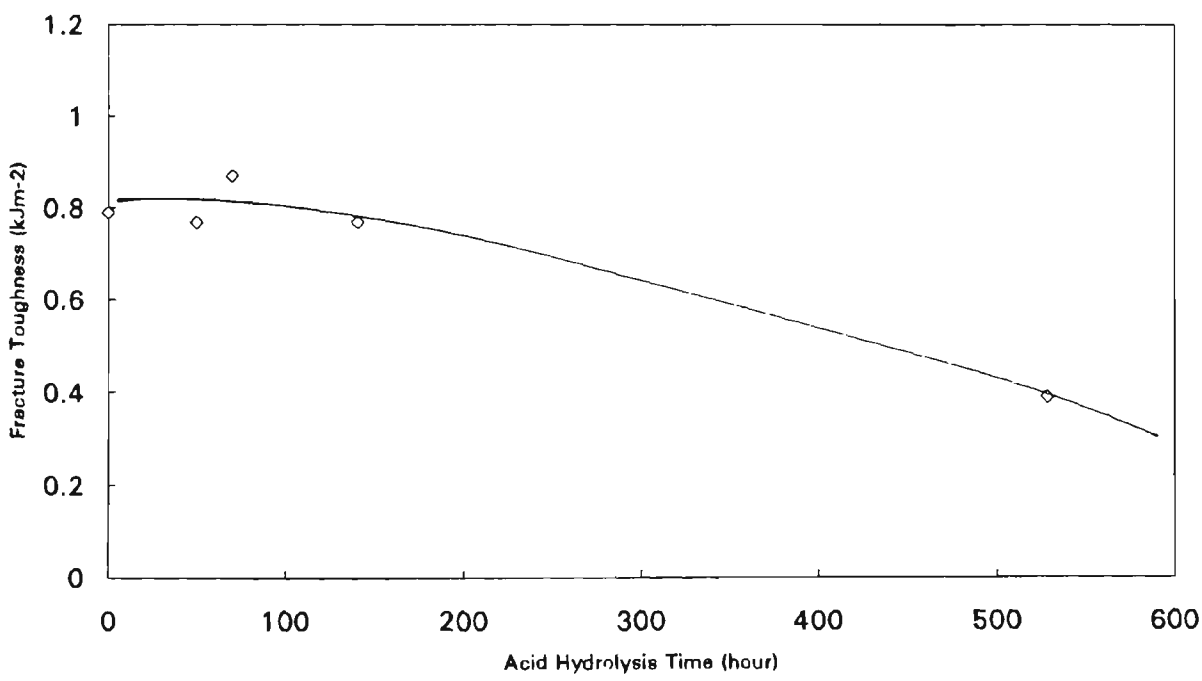

Fig. 7.11. Relationship between fibre acid treated time and composite fracture toughness

Coutts studied composites reinforced with NZ flax fibre(Coutts, 1983c). The composites showed similar flexural strength values, but, lower fracture toughness values compared to

the composite reinforced with softwood fibre (Coutts, 1984a). This was possible due to the fact the strength of NZ flax Kraft pulp fibre was weaker than that of softwood fibre.

In addition, Stevens (1992) reported that autoclaving cycle had a significant effect on composite toughness. In his work the first autoclaving cycle produced specimens which were quite tough. However, after ten autoclaving cycles the samples were extremely brittle. The results could be explained by the fact that cellulose degraded and the fibre strength reduced gradually after each autoclaving cycle. These weakened fibres were not able to provide the composite with sufficient fracture toughness.

7.3 Conclusions

Holocellulose and Kraft wood pulp fibres were chemically treated to various fibre tensile strength without changing other parameters. The results suggested that the fibre strength was particularly important to composite toughness but not as important to the strength. In both air-cured and autoclaved cases, when fibre strength was reduced to about 40%, the composites had a toughness loss more than 60%, but the strength was virtually unchanged. Examination of SEM micrographs, of the composite fracture surfaces, showed that the samples containing the weaker fibre had produced the expected higher population of broken fibres than samples containing the stronger fibres.

Chapter Eight:

Influence of Fibre Lignin Content on Composites Properties

During the course of this study, we found that pulps with different Kappa numbers (made from the same wood chip source) could result in a significant variation of composite properties, particularly values of fracture toughness. This finding suggested further investigation of the influence of lignin content (Kappa number) on the composite properties was warranted.

Along with cellulose and hemicelluloses, lignin is another major chemical component of plants. It is present to the extent of about 24 to 32% in softwood, lower amount in non-wood plants and very little in cotton and bast fibres (see section 3.3). The influence of lignin content on pulp and paper properties has been studied by several researchers (Gieriz, 1961; Page, 1985), despite the extremely complex nature of lignin chemistry. In general, in a non-degrading pulping process, fibre strength improves with decreasing lignin content due to the higher cellulose content. Paper formed with these strong fibres has better mechanical properties. However, further reduction in lignin content such as pulping to a yield corresponding to an α-cellulose content higher than 80% or bleaching tends to reduce fibre strength, apparently due to the elimination of this stress-equalising matrix lignin or due to cellulose degradation. Furthermore, because of the hydrophobic character of lignin, it binds the fibrils firmly together (fibres become stiffer) even after wetting. Lignin also decreases the interfiber bonding in paper.

Information regarding the influence of lignin content on composites is very limited. Mai and co-workers (1983) studied low lignin content bleached pulp and Kraft pulp autoclaved composites. The composites were reinforced with commercial Wisakraft bleached pulp and Kinleith Kraft pulp and fabricated by the Hatschek process. They found that bleached pulp composites had higher flexural strength but lower fracture toughness values than those of unbleached Kraft pulp.

Coutts (1986) studied high lignin content TMP and CTMP pulps prepared from *P. radiata* chips as reinforcement for autoclaved and air-cured composites (fabricated by slurry / vacuum de-watering technique). Both pulps were unsatisfactory, for use as a fibre reinforcement alternative to Kraft pulp in the autoclaved cement mortars, but some gave acceptable air-cured products. It was suggested that during autoclaving the extracted chemicals (eg. lignin) formed could cause an inhibiting effect on the curing of the cement and the deposit of such products could effect fibre-matrix bonding.

The fibre-cement industry has already committed itself to the use of Kraft softwood pulp as the asbestos alternative source in Australia. The level of pulping yield (lignin content) is not identified, but Kraft pulp at kappa number around 25 (lignin content about 3.7%) is well suited for autoclaved WFRC products.

Base on the above data, it was considered appropriate to study the relationship between fibre lignin content and fibre cement composite properties.

8.1 Experimental work

8.1.1 Fibre preparation

This study was designed to investigate the effect that different lignin containing pulps had on reinforced fibre cement composite properties. The pulps studied contained various levels of lignin and included a thermomechanical pulp (TMP), a chemithermomechanical pulp (CTMP), three Kraft pulps and an oxygen delignification pulp. The pulps were prepared from selected *P.radiata* chips (APM, Maryvale mill, Australia) in the CSIRO, Division of Forest Products pulping laboratory. The detailed pulping techniques have been discussed in Appendix A.1. While, a brief description of these techniques is listed in Table 8.1.

Table 8.1 Pulping techniques

Pulp	Pulping Technique
TMP	presteam chips 1-2 min at 120-125°C, followed by Asplund defibrillator 2-3 min, then refined in Bauer refiner
CTMP	presoak chips in 10% casustic soda over 18 hours, then processed as in the case of TMP pulp
Kraft (Kappa No.44.56)	E.A. 11.51% (NaOH), 4:1 liquor to wood ratio, 2 h to 170°C and 2 h at 170°C in air-bath (3L pulping vessels)
Kraft (Kappa No.27.49)	E.A. 13.56% (NaOH), 4:1 liquor to wood ratio, 2 h to 170°C and 2 h at 170°C in air-bath (3L pulping vessels)
Kraft (Kappa No.17.33)	E.A. 14.00% (NaOH), 4:1 liquor to wood ratio, 2 h to 170°C and 2 h at 170°C in air-bath (3L pulping vessels)
Bleached	Kraft pulp (E.A.16.63%) mixed with 1% $MgCO_3$, 2% NaOH at 10% consistency cooked with O_2 (780kpa), 75 min to 115°C and 30 min at 115°C

All pulps were lightly beaten in a Valley beater at condition of each 360g (o.d.) with 23 litre water without load for 20 min then with 1.5 kg bed-plate load for another 20 min. The beating effect was evaluated by means of Canadian Standard Freeness tester (see Appendix A.2.2).

8.1.2 Fibre lignin content and other properties evaluation

The pulps prepared from above techniques cover a wide range of lignin contents and there is no single lignin evaluation method available. For high yield pulp such as TMP and CTMP pulps, Klason lignin method is more suitable, while for Kraft and oxygen bleached pulps, Kappa number test is normally applied. The percentage of lignin present is then estimated as 0.147 x Kappa number value. Because softwood contains only a small percentage of soluble lignin, there was no attempt to correct for soluble lignin in Klason lignin measurement. The detailed lignin content evaluation methods should be refereed to in Appendix A.2.1.

Other fibre properties such as strength, length and freeness values are measured as wet zero-span tensile index, Kajanni length weighted average and Canada Freeness respectively. The testing methods are described in Appendix A.2.

8.1.3 Composite fabrication and characterisation

Air-cured and autoclaved composites were fabricated and characterised by the methods described in Appendix A.3 and A.4.

8.2 Results and discussion

8.2.1 Fibre lignin content and other properties

Table 8.2 Fibre lignin content, freeness and fibre strength, fibre length

Pulp	Lignin (%) / method	Length (mm)	Freeness (CSF)	Strength as ZSTI
TMP	25.52 / Klason	2.17	700	low
CTMP	26.04 / Klason	2.43	730	low
Kraft 44.56*	6.47 / Kappa	2.53	730	109
Kraft 27.49	4.04 / Kappa	2.60	700	113
Kraft 17.33	2.55 / Kappa	2.36	670	101
Bleached	0.69 / Kappa	2.29	670	98

* The number after Kraft is its Kappa number

The variation of pulp lignin content, freeness, fibre strength and fibre length are summarised in Table 8.2. As mentioned before due to the softwood only containing a small percentage of soluble lignin, there was no attempt to correct for such soluble lignin in Klason lignin measurement. There was an inconsistency between TMP and CTMP lignin content as the values of 25.52 % for TMP and 26.04 % for CTMP could be expected to be reversed. Klason lignin measures insoluble lignin on pulp base, CTMP pulp could show a higher lignin content than that in the same amount of TMP pulp due to pretreatment of caustic soda extracting other soluble materials from the fibres. The principle of mechanical pulping is to mechanically separate wood into its constituent fibres. Mild chemical treatment during CTMP pulping would not cause much lignin loss, thus TMP and CTMP could be expected to have similar lignin content as that of the wood. However, the difference in other properties of these pulps would have some influence on the final composite performance.

Although it would be desirable to maintain a constant length, so as to limit the variables when studying lignin effect, some variation was recorded. It would not be expected to have a major influence (see Chapter 6).

In addition, the difference of fibre strength could also have some effect on composite performance particularly the fracture toughness as studied in Chapter 8. The pulp with Kappa number around 27 had the highest strength index of the six pulps. The number of un-degraded cellulose fibres in the pulp makes the major contribution to the strength development of the pulp. Besides cellulose content, mechanical pulps (TMP and CTMP) and high Kappa number pulp (Kraft 44) contain a high percentage of lignin and

hemicellulose. There are less cellulose fibres present in the high yield pulp compared to the low yield pulp (e.g. Kraft 27), thus a lower strength index is expected. The decrease in strength of the pulps with the Kappa number below 27 such as Kraft 17.33 and bleached pulp could be due to degradation of cellulose from strong alkali cooking or bleaching.

The high freeness value suggested that 20 min, with low bed-plate load, Valley beating would probably only improve fibre conformability rather than fibrillating fibre surface. Although the TMP and CTMP pulps contained high fines proportion, they still showed relative high freeness values because of the stiff nature of the fibres.

8.2.2 Influence of lignin content on air-cured composites

The influence of fibre lignin content on air-cured composite flexural strength, fracture toughness and density are delineated in Figure 8.1, 8.2 and 8.3, respectively. The detailed results is also listed in Table 8.3.

Figure 8.1 shows the flexural strength variation as fibre content was increased from 2% to 12% by mass for six different lignin content fibre-cement composites. The strength of the composites start to decrease for fibre content over 10 % due to poor fibre distribution throughout the matrix material. This observation was in general agreement with the change in flexural strength observed with early studies in WFRC and other NFRC (Coutts,1985, 1987b, 1994b).

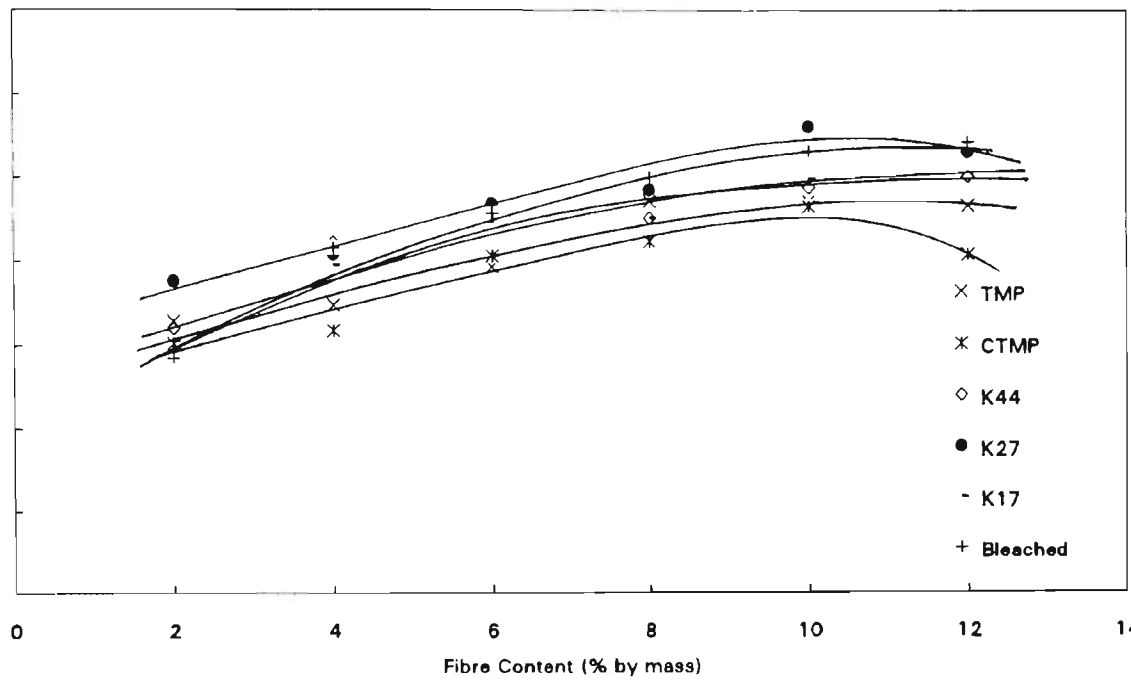

Fig. 8.1. Influence of fibre lignin content on air-cured WFRC flexural strength at different fibre content

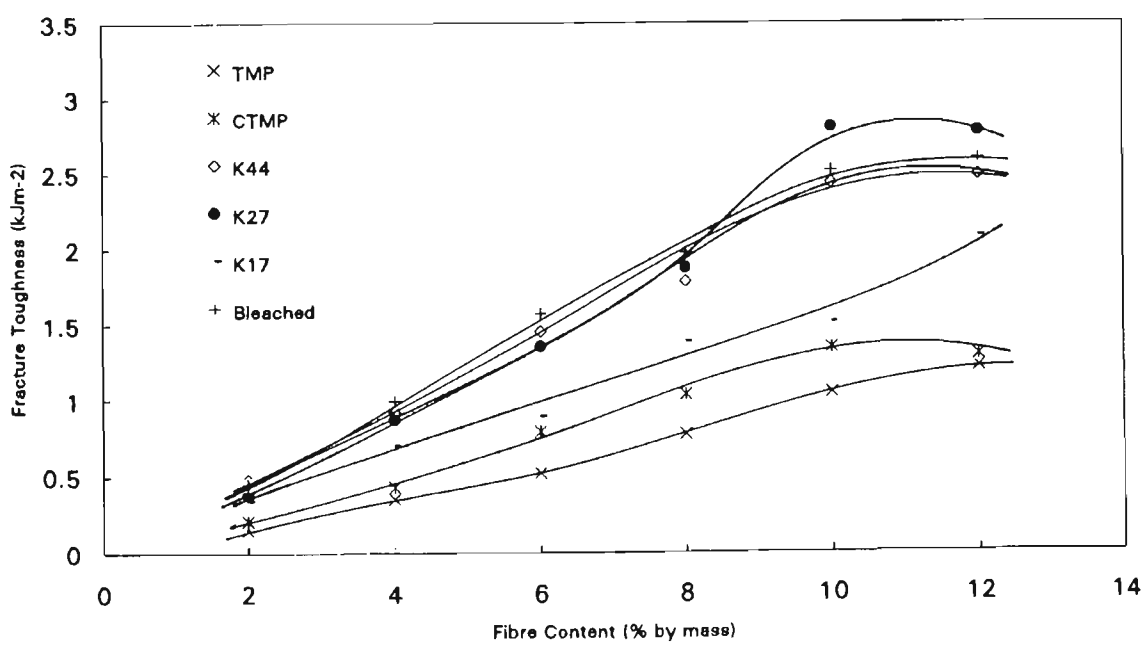

Fig.8.2. Influence of fibre lignin content on air-cured WFRC fracture toughness at different fibre content

Table 8.3 Influence of fibre lignin content on air-cured WFRC.

Fibre (w%)	MOR (MPa)	Frac. Tough(kJ/m2)	Void Vol (%)	Water Abs.(%)	Density (g/cm³)
TMP, 25.52%					
2	16.4±1.4	0.15±0.02	28.7±0.8	15.4±0.5	1.86±0.02
4	17.4±2.0	0.35±0.07	30.5±1.4	17.8±1.1	1.72±0.03
6	19.7±2.5	0.52±0.06	30.9±1.2	18.9±1.1	1.64±0.03
8	23.6±2.5	0.77±0.13	32.0±1.6	20.5±1.4	1.56±0.04
10	23.6±2.6	1.04±0.17	33.1±1.1	22.2±0.8	1.49±0.02
12	23.3±3.3	1.21±0.24	35.1±1.8	24.7±1.6	1.42±0.02
CTMP, 26.04%					
2	15.1±1.7	0.21±0.05	29.5±0.6	16.0±0.4	1.84±0.02
4	15.9±2.2	0.43±0.07	30.6±1.1	17.7±0.9	1.73±0.03
6	20.3±2.7	0.79±0.16	33.1±0.8	20.6±0.8	1.61±0.03
8	21.2±3.4	1.03±0.29	35.1±0.5	23.6±0.6	1.49±0.02
10	23.3±4.6	1.34±0.34	37.5±1.5	26.7±1.4	1.40±0.02
12	20.4±3.5	1.29±0.26	38.4±1.1	28.9±6.6	1.34±0.07
K44, 6.47%					
2	16.0±1.6	0.48±0.06	28.4±0.4	15.4±0.4	1.84±0.03
4	21.2±1.4	0.92±0.12	29.1±0.7	16.6±0.7	1.76±0.02
6	22.7±2.3	1.45±0.20	31.5±0.7	19.4±1.0	1.63±0.05
8	22.6±2.8	1.78±0.28	32.5±1.1	20.6±1.0	1.58±0.03
10	24.4±2.2	2.43±0.67	35.0±0.8	23.2±1.0	1.51±0.03
12	25.1±1.2	2.48±0.09	35.5±0.8	24.0±0.8	1.48±0.02
K27, 4.04%					
2	18.8±1.1	0.38±0.10	29.2±0.4	15.6±0.3	1.86±0.02
4	20.4±1.9	0.87±0.12	30.5±0.3	17.5±0.3	1.75±0.02
6	23.4±2.3	1.35±0.24	32.6±0.9	19.8±0.8	1.65±0.03
8	24.3±1.9	1.87±0.19	33.4±1.2	21.6±0.8	1.55±0.05
10	28.1±3.5	2.80±0.45	34.6±1.0	22.5±1.1	1.54±0.03
12	26.5±5.4	2.77±0.67	34.8±1.0	22.9±0.7	1.52±0.02
K17, 2.55%					
2	15.3±0.9	0.34±0.03	29.1±1.0	15.9±0.7	1.84±0.03
4	19.8±0.9	0.70±0.10	30.2±0.7	17.5±0.7	1.73±0.03
6	22.6±2.9	0.89±0.26	32.0±0.7	19.5±0.7	1.64±0.03
8	22.6±2.5	1.38±0.09	33.2±1.1	20.8±1.1	1.59±0.04
10	24.9±2.3	1.51±0.15	34.1±1.0	22.0±0.7	1.55±0.03
12	27.2±1.4	2.08±0.10	34.7±0.4	22.9±0.4	1.51±0.01
Bleached, 0.69%					
2	14.2±1.1	0.45±0.09	29.7±3.3	16.1±2.7	1.86±0.09
4	20.8±1.5	0.99±0.20	30.4±0.8	17.1±0.7	1.78±0.02
6	22.9±2.5	1.57±0.29	30.0±0.8	19.1±0.7	1.68±0.08
8	25.0±4.1	1.97±0.32	32.9±0.8	20.1±0.8	1.63±0.03
10	26.6±2.4	2.51±0.32	34.4±0.9	22.1±1.0	1.56±0.03
12	27.1±4.2	2.49±0.46	34.7±0.3	22.3±0.4	1.55±0.01

*All composite were fabricated using ordinary portland cement, air-cured for 28 days, tested at 50±5 per cent RH and 23±2°C.
* 3 standard deviation, sample size n=9.

Of the six different pulp reinforced composites studied, the sample pulp which contained pulp with a Kappa number about 27 had highest strength value. This could be due to the fact that Kraft 27 pulp contained the strongest fibre as measured by zero-span tensile index (see Table 8.2). If the standard deviations are considered, one would not expect a significant strength variation among the pulps at the same fibre content. The small amount

of variation would be caused more by different fibre length rather than by different lignin content. The influence of lignin content will be seen more clearly when discussing composite fracture toughness property.

The fracture toughness values of the composites increased with increasing fibre content in all cases. However, the rate of increase and the toughness values obtained varied greatly between pulps as shown in Figure 8.2. Again Kraft pulp at Kappa number 27 had the highest toughness value and a high rate of increase. This could be due to the pulp having the strongest and longest fibres in addition to good flexibility for mixing with the cement matrix. TMP and CTMP pulp had similar high values for lignin content, low strength index and high fines content, one would not expect composites reinforced with these pulps to have good fracture toughness. The slightly longer CTMP pulp composite had better fracture toughness than that of TMP pulp. The unsatisfactory fracture toughness value of Kraft 17 WFRC might be attributed to the smaller and weaker reinforcing fibres compared to those in Kraft 27. Air-cured Kraft 27 WFRC had better toughness at high fibre contents (say > 8%), although not much difference was observed between Kraft 44 and Kraft 27 WFRC at low fibre contents.

Bleached pulp composites even though they contain relatively short and weak fibres had very competitive fracture toughness with that of Kraft 44 and 27 composites. This could be due to bleached pulp fibre being more flexible and forming strong bonds to the cement matrix. The high density values of WFRC reinforced with bleached pulp supports this statement. Fracture energy is the combination of the fibre-matrix bond breaking, fibre fracture and fibre pull out. If the fibre is strong enough to withstand a pulling force, then a well bonded composite should have good fracture toughness property. It was discussed in

Chapter 7, that fibre strength had great impact on composite toughness and that the toughness decreases rapidly with reduction in fibre strength. In that case, the fibre was not strong enough to withstand pulling force and the fibre failure was the controlling factor during specimen fracture. The properties of air-cured WFRC reinforced with bleached pulp suggested that the oxygen bleaching to lignin content down to about 0.7% (in addition to wet zero-span tensile index about 100 Nm/g) was still appropriate for air-cured products.

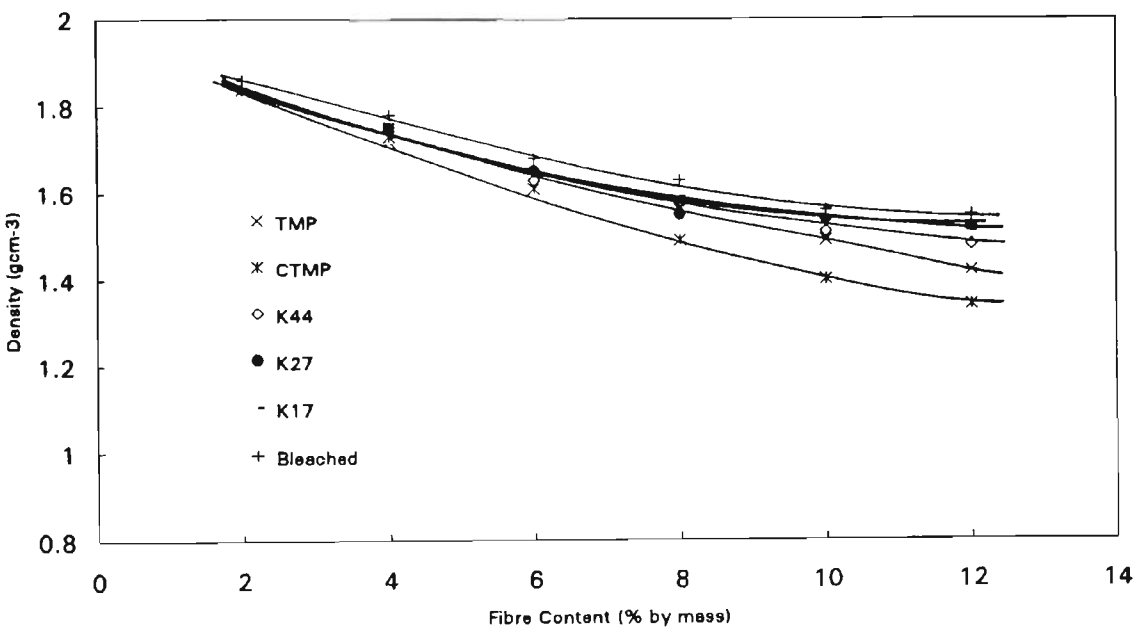

Fig. 8.3. Influence of fibre lignin content on air-cured WFRC density at different fibre content

The variation of composite density, water absorption and void volume was expected from the flexibility natural of the pulps. The stiffness or flexibility of the fibre increases with the removal of the lignin, with flexibility gradually improving from TMP, CTMP, high yield Kraft pulp, low yield Kraft pulp through to bleached pulp. Fibres with a high degree of flexibility are easy to form in the composite and provide good interfacial bonds with the cement matrix. Such composites have high density and low water absorption and void volume values as seen in Table 8.3 and Figure 8.3.

8.2.3 Influence of lignin content on autoclaved composites

Table 8.4 and Figure 8.4, 8.5, 8.6 shows the variation of autoclaved WFRC mechanical and physical properties influenced by fibre lignin content.

It can be seen from Table 8.4 and Figure 8.4 that the flexural strength of autoclaved composites reinforced with low yield Kraft pulp and bleached pulp increased with fibre content increasing which was in agreement with that of the air-cured products. However, the strength of WFRC reinforced with high yield TMP pulp, at all fibre contents showed little improvement. CTMP pulp composite at above 8% fibre by mass content showed some small strength development.

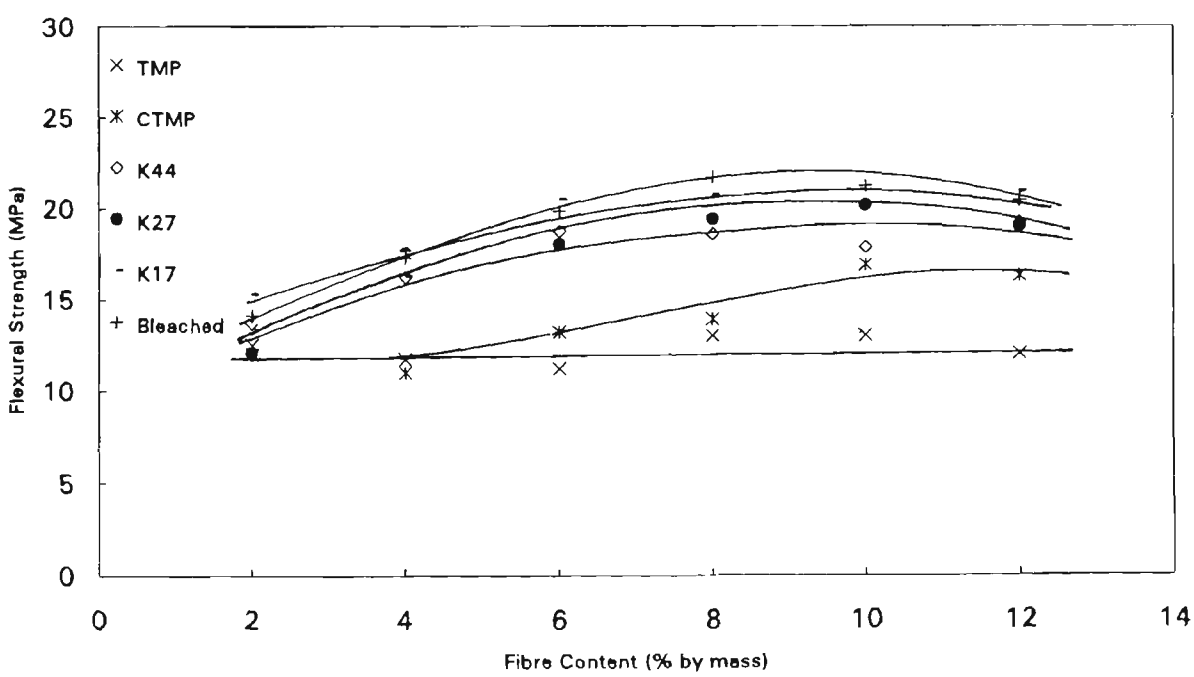

Fig. 8.4. Influence of fibre lignin content on autoclaved WFRC flexural strength at different fibre content

Table 8.4 Influence of fibre lignin content on autoclaved WFRC

Fibre (w%)	MOR (MPa)	Frac. Tough(kJ/m2)	Void Vol (%)	Water Abs.(%)	Density (g/cm³)
TMP, 25.52%					
2	12.5±1.8	0.07±0.01	36.9±0.9	23.3±0.8	1.59±0.02
4	11.7±1.9	0.17±0.01	39.1±1.0	26.6±1.2	1.47±0.03
6	11.2±2.1	0.33±0.05	41.6±1.4	30.4±1.9	1.37±0.05
8	13.0±1.6	0.46±0.05	42.1±1.1	31.9±1.4	1.32±0.03
10	13.0±2.5	0.73±0.16	46.3±1.2	39.1±1.9	1.19±0.03
12	11.8±2.3	0.77±0.16	48.1±1.3	42.9±2.2	1.12±0.03
CTMP, 26.03%					
2	12.0±1.4	0.13±0.04	39.1±1.0	25.0±1.2	1.56±0.03
4	11.2±0.8	0.46±0.07	41.0±0.6	28.7±0.8	1.43±0.02
6	13.2±0.7	0.66±0.09	42.9±0.6	32.2±0.9	1.33±0.02
8	13.9±2.3	0.79±0.18	44.9±0.6	35.5±1.0	1.27±0.02
10	16.9±2.8	0.89±0.20	46.5±0.7	39.4±0.4	1.18±0.06
12	16.2±2.3	1.06±0.18	47.8±0.4	41.6±1.1	1.14±0.02
K44, 6.47%					
2	13.7±0.8	0.13±0.02	38.1±0.8	23.8±0.7	1.60±0.02
4	16.2±0.9	0.45±0.10	40.0±0.4	26.6±0.6	1.50±0.02
6	18.7±1.7	1.09±0.29	40.6±1.2	28.1±1.4	1.44±0.03
8	18.6±1.6	2.18±0.20	41.7±1.2	30.4±1.4	1.38±0.03
10	17.8±1.2	2.62±0.21	44.1±1.1	33.1±1.3	1.34±0.02
12	19.2±2.0	2.65±0.67	44.9±1.4	34.8±1.9	1.29±0.03
K27, 4.04%					
2	12.1±0.7	0.07±0.02	39.3±1.3	25.2±1.3	1.56±0.03
4	17.5±2.0	0.38±0.05	41.5±0.7	28.0±0.8	1.48±0.02
6	18.0±1.4	0.64±0.09	42.7±0.8	30.5±1.0	1.40±0.02
8	19.4±1.9	2.36±0.41	43.2±0.5	32.0±0.9	1.35±0.02
10	20.1±1.4	2.89±0.55	44.4±1.1	34.3±1.4	1.30±0.02
12	19.0±0.9	2.82±0.12	44.3±0.6	34.9±0.9	1.27±0.02
K17, 2.55%					
2	15.3±1.1	0.15±0.02	39.2±1.2	24.8±1.0	1.58±0.02
4	16.3±2.8	0.32±0.06	41.2±1.2	27.7±1.3	1.49±0.03
6	20.5±1.0	0.68±0.03	41.6±0.8	29.0±1.0	1.44±0.02
8	20.7±1.5	0.88±0.02	41.8±1.7	30.2±1.8	1.39±0.03
10	20.2±1.1	1.38±0.02	42.9±1.3	32.4±1.6	1.32±0.03
12	20.9±1.2	1.84±0.01	43.4±2.1	34.5±1.9	1.26±0.04
Bleached, 0.69%					
2	14.1±0.5	0.12±0.07	39.1±0.6	24.7±0.7	1.58±0.02
4	17.3±1.9	0.44±0.06	41.5±1.1	27.7±1.3	1.50±0.03
6	19.8±2.2	0.55±0.09	41.0±1.0	28.2±1.1	1.46±0.02
8	21.7±2.8	0.81±0.27	41.3±1.9	29.4±2.0	1.41±0.03
10	21.2±2.4	1.71±0.47	44.7±1.2	34.1±1.7	1.31±0.03
12	20.4±1.1	2.53±0.27	44.4±0.6	34.0±0.9	1.30±0.01

*The composites were fabricated using ordinary Portland cement and silica at the ratio of 1:1, autoclaved at 1.25MPa steam pressure for 7.5h, tested at 50±5 per cent RH and 22±2°C. 3 standard deviation, sample size n=9.

It was discussed in section 3.3 and 3.4 that natural plant fibre (wood) consists of considerable amount of lignin and this lignin can be dissolved by some chemical solutions and the high temperature can accelerate the reaction rate. This is the principle of chemical pulping. Composites subjected to autoclaving could easily dissolve lignin due to the high

curing temperature (above 175°C) and severe alkaline cement environment. This dissolved lignin would either poison the matrix curing or deposit on the surface of the fibre causing poor interface bonding, or the combination of these two. The mechanism of dissolving lignin is still not fully understood.

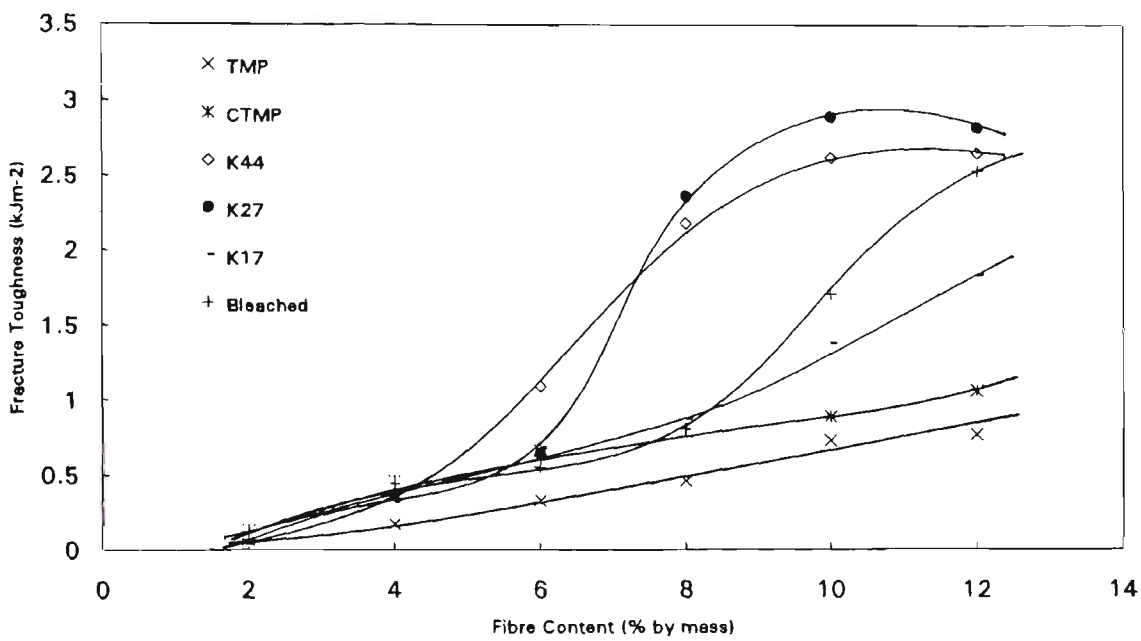

Fig. 8.5 Influence of fibre lignin content on autoclaved WFRC fracture toughness at different fibre content

Autoclaved composites reinforced with fibres containing low lignin appear to be more stable than those with higher lignin content. Thus autoclved WFRC reinforced with bleached pulp and Kraft 17 showed high flexural strength as seen in Table 8.4 and Mai's work (1983). Autoclaved TMP fibre reinforced composites had the lowest flexural strength values due to the effect of lignin dissolving / depositing. The same observation was reported by Coutts (1986), he suggested that high yield TMP and CTMP pulps were unsatisfactory for use as a alternative fibre reinforcement to *P.radiata* Kraft pulp in autoclaved cement mortars, but acceptable for use in air-cured products.

The effect of lignin content on the property of fracture toughness could be understood in terms of fibre strength. Fibre strength plays a dominant role to the composite fracture toughness as discussed in Chapter 7. Figure 8.2 and 8.5 shows composites reinforced with strong fibre (Kraft 27) had the highest toughness value.

This remark is strengthened by the results of the fibre strength study in Chapter 7. The fibres in the present study had a variation in strength properties (Table 8.2). In addition to dissolving lignin, autoclaving is akin to a severe alkaline "pulping" and can further reduces strength values due to degradation of fibre cellulose. Although the degree of such strength loss is uncertain, the fracture toughness loss of autoclaved composites reinforced with bleached pulp was significant compared with air-cured products. A few workers (Lhoneux, 1991; Stevens, 1992) have studied fibre strength loss during autoclaving, mainly using lime solution to simulate the matrix environment. This study attempts to investigate the "real pulping" conditions present in the autoclave by means of a reconstituted handsheet technique. Reinforcing fibre was first formed into handsheets, then inset into cement blocks subjected to autoclave; after autoclaving, the matrix was broken apart, the handsheets were carefully removed out and disintegrated into pulp; then characterised for change of pulp strength and other properties. In our preliminary study it was observed that the fibre strength and lignin content were reduced during autoclaving, however extensive work is required and results will not be reported in this thesis.

The variation in autoclaved composites physical properties were similar to those of air-cured cement products. The fibre stiffness or flexibility, which is influenced by the fibre lignin content, became the key factor in regarding materials density, water absorption and void volume as listed in Table 8.4 and shown in Figure 8.6.

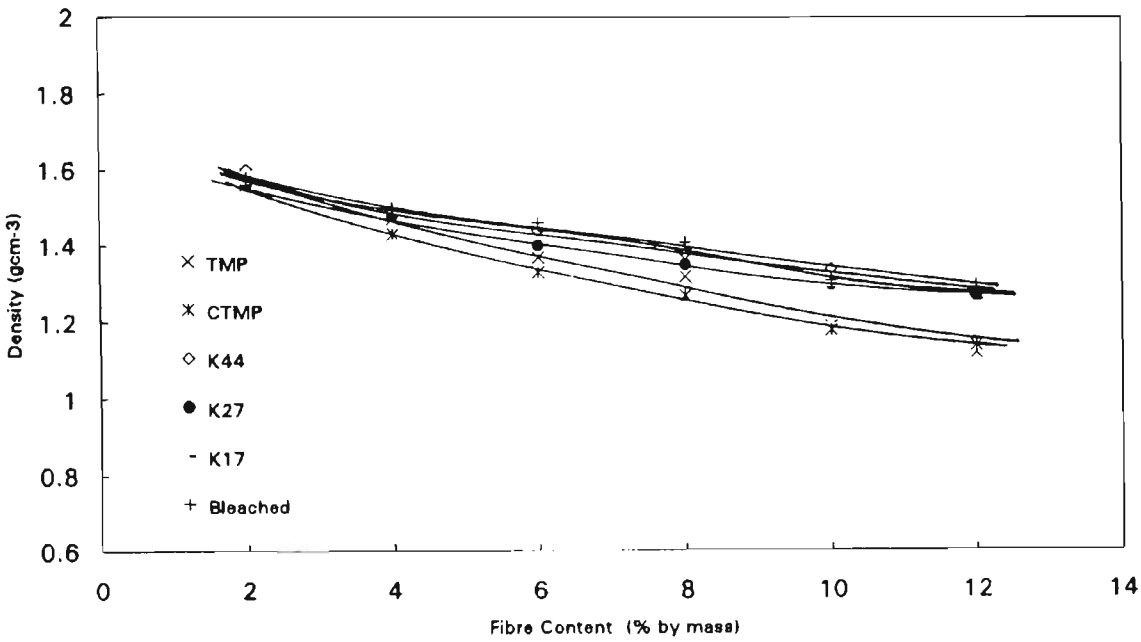

Fig. 8.6. Influence of fibre lignin content on autoclaved WFRC density at different fibre content

8.3 Conclusions

The results showed that both TMP and CTMP pulps did not have satisfactory mechanical properties when used to reinforce autoclaved cement composites. Air-cured products have better mechanical properties and could be possible used in a number of applications such as renders, moulded articles or sheet products.

The lignin component of the pulp fibre had considerable influence on the composites mechanical and physical performances. Such influences could be understood in terms of fibre strength, fibre stiffness, fibre-matrix interface bonding, fibre number and lignin dissolving mechanics during the autoclaving process.

Experimental results also showed that composites reinforced with Kraft pulp with Kappa number around 27 had the best mechanical and physical properties in both air-cured and autoclaved products.

Chapter Nine:

Conclusions and Further Work

9.1 Conclusions

The following conclusions can be drawn from this work:

1. Chemically pulped bamboo fibre is a satisfactory fibre for incorporation into a cement matrix. Mechanical and physical properties of both air-cured and autoclaved bamboo fibre cement composites were studied. Bamboo fibre cement products have reasonable flexural strength and competitive physical properties to their wood fibre cement counterparts. However, the fracture toughness values are low due to short fibre length and high fines content of the bamboo pulp. Improved properties of composites reinforced with screened long bamboo fibre confirm this belief.

There is little difference between the properties of composites reinforced with beaten and unbeaten bamboo pulp. This could be due to the average short fibre length or the structure of bamboo fibre when compared with wood fibres.

2. Properties of bamboo fibre cement composites (and the other natural fibre products) can be improved by blending long fibres (such as softwood fibre) with the bamboo reinforcement to increase the average fibre length. Such hybrid fibres improve the composite strength and more importantly the fracture toughness value. The extent of such improvement is related to the increasing proportion of long fibres.

3. The influence of fibre length on composite performance has been extensively studied. Fibre length plays a significant role in the behaviour of fibre cement composites. If the fibre is long, a greater amount of fracture energy is needed to pull the fibre through the matrix and the composite can be tougher and stronger. This work was carried out by fractionating a single pulp into a range of lengths. The results showed that when short fragments of fibre (weighted length 0.30 mm) were used as reinforcement and compared with longer fibres (weighted length 1.59 mm), a drastic reduction in composite fracture toughness and strength was observed. For example, at 8% by mass the short fibre samples showed a seven fold decrease in fracture toughness and possessed only half the flexural strength of samples containing the same amount of the longer fibre.

4. Research with holocellulose fibre pulp, chemically treated to vary the tensile strength of the individual fibres, indicated that fibre strength was particularly important to composite toughness but not to composite strength. In both cases of air-cured and autoclaved fibre composites, when fibre zero-span tensile index (wet) and pulp viscosity values were reduced to about 40% of full strength, the composites fracture toughness value also were observed to fall to about 40% of original value. Surprisingly, the flexural strength values were virtually unchanged. The reason behind this behaviour is not clear. Examination of SEM micrographs, of the composite fracture surfaces, showed that the samples containing the weaker fibre had produced the expected higher population of broken fibres than samples containing the stronger fibres.

5. Air-cured and autoclaved composites reinforced with a wide range of lignin content pulps were evaluated. The lignin component of the pulp fibre had a great influence on the composites mechanical and physical performances. Such influences could be understood

in terms of fibre strength / fibre stiffness (cellulose content), fibre-matrix interface bonding, fibre number and lignin dissolving mechanics during the autoclaving process.

Both TMP and CTMP pulps did not have satisfactory mechanical properties when used to reinforce autoclaved cement composites. Bleached pulp reinforced composites had high strength but low toughness values (compare to Kappa number 27 pulp). The composites reinforced with Kappa number of about 27 kraft pulp had the best mechanical and physical properties in both air-cured and autoclaved products.

9.2 Recommendations for further work

9.2.1. Pulp supply

The reinforcing potential of kraft fibres depends mainly on the properties of the raw material used. Although fibre properties can be affected by the cooking conditions - alkali charge affecting wet fibre flexibility and sulphidity affecting intrinsic fibre strength - these factors do not compensate for the variations caused by the raw material. It is therefore essential for both the final product quality and production costs that the interactions between raw material and end-products properties are well understood and the wood fibre property variations can be measured.

The basic natural (eg. wood) fibre properties are wall thickness, fibre width, fibre length, "weak points" in the fibre wall, S_2 fibril angle, the index of crystallisation and chemical composition. These properties vary between different wood species; within one annual ring (springwood and summerwood); between different parts of the trunk and are due to the different growing conditions.

Modern forestry has accelerated forest growth, and the trend is to harvest younger trees. The raw material is also used more efficiently (sawmill chips, whole tree chips and wood residue), and the amount of imported chips from plantations seems to be increasing. This means that quality variations in raw material are also increasing significantly. If this is not taken into account in pulping and stock preparation, the reinforcing potential of the raw material is not utilised in full, and at the same time final product quality variations increase.

Tasman Pulp and Paper Co. Ltd., New Zealand has made progress in matching the highly variable wood supply to various end-products, i.e. papermaking and fibre-cement, by means of segregation of their fibre supply into density ranges. However, there is still a long way to go to understand the true relationship between fibre quality and end-products properties and to modify the fibre quality to match the products requirement (Williams, 1994).

9.2.2. Fibre length population distribution and cross-dimensions (coarseness)

Among the wood fibre basic properties, fibre length and fibre cross-dimensions, i.e. wall thickness and fibre width, might be the most important factors regarding the preparation of paper and fibre composite products. The influence of fibre length has been studied in this thesis, however, the influence of fibre length population distribution would be more meaningful to fibre-cement composites. This is because of the nature of non-uniformity of fibre length, the nature of stock preparation and pulp blending.

The fibre cross-dimensions, i.e. wall thickness and fibre width, has often been characterised using the coarseness value. Because of the close relationship between the

cross-dimensional properties of softwood fibres and fibre length, the variation in paper (and fibre-cement) properties caused by the cell wall thickness and fibre width have many times been erroneously explained in terms of fibre length. The different cross-dimensional properties of fibres, for example thick-walled, narrow, stiff and strong fibres and thin-wall, wide, flexible with a large lumen and excellent bonding ability fibres, has great impact on their end-products properties. The influence of fibre cross-dimensional characteristics on paper properties has been studied by a number of researchers (Clark, 1962; Seth, 1987; Kibblewhite, 1989), some people even suggested that in practice the cross-dimensions (coarseness value) was the most identical characteristic to predict the papermaking potential of different softwood fibres (Paavilainen, 1993).

Very limited work has been done regarding the effect of fibre cross-dimensions to the fibre-cement products quality. Vinson and Daniel (1990) demonstrated that high density summerwood fibres (coarser) were better suited for fibre cement reinforcement than low density fibres. Coutts (1987) in studying natural fibre reinforced cement composites suggested the importance of fibre dimensions in respect to its length and diameter aspect ratio. However, more extensive work is required before a clear picture can emerge.

9.2.3 Conformability - flexibility and collapsibility

Fibre conformability, namely fibre flexibility and collapsibility, characterises the ability of wet fibres to deform (plastic and elastic deformation). Both fibre flexibility and fibre collapsibility are dependent on cross-dimensional fibre properties and on the elasticity of the cell wall. At the same time the fine structure, chemical composition and water content of the cell wall as well as the drying history and the chemical and mechanical treatments to which the fibres are subjected all play a role in how the fibre will respond.

In the case of chemical pulp fibres, it is widely agreed that collapsibility and flexibility are important both for paper and composites properties . Furthermore the conformability of the fibre also affects stock preparation and machine run-ability.

Although the importance is widely recognised, knowledge of the factors affecting conformability is limited and not systematic. Also, experimental verification of the relationship between conformability and end products properties is difficult to find in the scientific literature (Paavilainen, 1993).

9.2.4 The impact of pulp medium consistency treatment on fibre-cement products

The processing of pulps at medium consistency (MC) has become common in the pulp and paper industry. The MC technology, which employs fibre mass concentrations typically in the range 8-14%, allows considerable reduction in the volume of pulp suspension handled during processing operations. This has led to reduced equipment size, and saving in capital and operating costs.

The conditions required to fluidise the suspensions lead to beating, as well as the introduction of curl and microcompression in the fibres (Seth, 1991). The MC treatment will result in a higher sheet stretch and lower elastic modulus of the sheet. Sheet porosity and tear will also be enhanced by the increased curl and microcompression (Page, 1985; Seth, 1993). Although the influence of curl on the fibre cement composites has been studied by Michell (1990), the implications of MC processing nevertheless needs further investigation with both bench-scale and commercial equipment.

9.2.5 Theoretical modelling

There is limited work available on model systems in the literature, dealing with fibre reinforced cement composites. A consideration should be given to the development of models to predict composite properties based on an understanding of fibre and matrix and their inter-relationship. Modelling allows the engineer to take into consideration scaling and size effects. It also has the advantage of being able to indicate the direction for future work in achieving optimum strength and toughness performance for a given system.

Appendix A:
Fabrication and Characterisation Methods

A.1 Pulp Fibre Preparation

A.1.1 Chemical pulping (Kraft pulping)

The Kraft process is the predominant pulping process in industry today; it is tolerant to variations in wood chip dimensions and wood quality and, because of its high strength, Kraft pulp can be used in a wide range of end products.

The Kraft process involves heating the wood at 165-175°C with a solution of sodium hydroxide (NaOH) and sodium sulphide (Na_2S) for 0.5-3 hours. Time to cooking temperature is in the range 1-2 hours. Pulp yield 45-55%. On an industrial scale, the spent cooking liquor (called black liquor) is concentrated and then burnt in a furnace, allowing the chemicals to be recovered and at the same time providing energy for the pulp mill.

In the laboratory assessment of pulpwoods, pulping conditions (temperature, time) are chosen to resemble those of industry. Wood sample size are usually constant and specified in terms of oven dry mass. As the volume of wood contained in each vessel fluctuates with the density of the wood sample, sample sizes must be chosen so as to remain within the capacity of the pulping vessels. In the air-bath used in this work six 3-litre pulping vessels are used (Fig. A.1). Typical conditions for a cook would be two hours at 170°C, 25% sulphidity and 4:1 liquor to wood ratio.

The alkali charge is varied to achieve the required degree of delignification, usually described in terms of Kappa number (a measure of the residual lignin content, see section A.2.2.1). Unbleached packaging grade pulps are usually delignified to Kappa numbers around 40, bleachable hardwood pulps are lower, about Kappa 20. Pulps for reinforcement in the cement composites their Kappa number are in the range of 30 to 20.

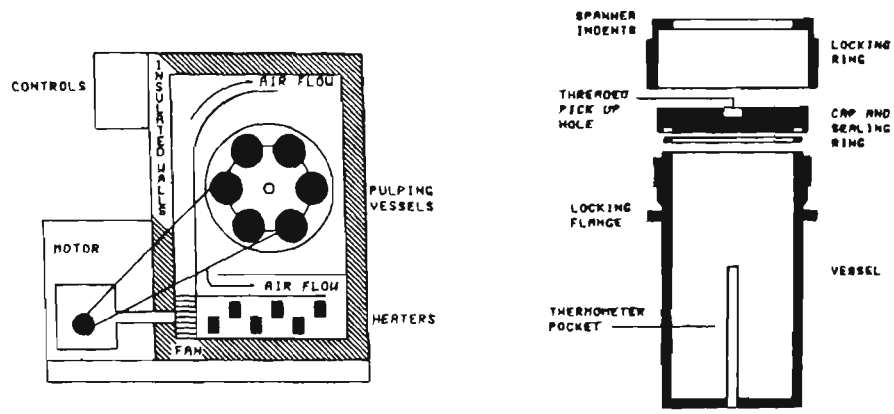

Fig. A.1. Schematic illustration of Air-bath and 3-litre pulping vessels.

The active chemicals in the Kraft pulping liquor are sodium hydroxide (NaOH) and sodium sulphide (Na_2S). The alkali charge is usually expressed in terms of the equivalent quantity of sodium oxide (Na_2O), although that particular chemical entity is never actually encountered. Three parameters are used to define the chemical make up of a Kraft pulping liquor: active alkali, effective alkali and sulphidity.

1. The active alkali (abbreviated AA) is NaOH + Na_2S, expressed as Na_2O, and usually as a percentage relative to the weight of oven-dry (o.d.) wood chips to be cooked.

2. The effective alkali (EA) is defined as NaOH + 1/2Na_2S, expressed as Na_2O.

3. The sulphidity is the percentage ratio of Na_2S to active alkali, expressed as Na_2O.

For most new wood samples the alkali charge required to attain the target Kappa number will not be known. It is usually necessary to find the proper alkali charge by trial. As the change in Kappa number with alkali charge is frequently non-linear at low Kappa numbers, experience with similar wood samples is often the only guide. The normal alkali requirement for softwood pulping is about 12 to 14% effective alkali on o.d. wood [8 to 10% for hardwoods (Smook, 1982)].

The alkali charge is usually made up from stock solutions of sodium hydroxide and sodium sulphide. These stock solutions have to be standardised before use because the chemical concentrations change as the chemicals react with carbon dioxide in the atmosphere.

Another essential piece of information is the moisture content of the wood sample. Not only does the moisture content affect the weight of wood required in each pulping vessel, but also the liquor to wood ratio. Details of the calculations required to determine the amount of wood, chemicals and water required for a Kraft cook are given in Appendix B.

The 3-litre air bath can be preheated to a temperature well in excess of the final cooking temperature before the pulping vessels are loaded. The air bath can also be heated after loading the pulping vessels. It usually takes 1.5 - 2 hours to reach the cooking temperature in this case. Although the air bath can be thermostatically controlled when finally at the cooking temperature, the rise-to-temperature portion of the cycle, especially from about 160°C on, is controlled manually, and some degree of judgement is required to effect a smooth approach to the cooking temperature without significant overshoot. It is suggested that the maximum temperature up to 180°C does not significantly affect the cooking result. From 180 to 190°C, there appears to be a small reduction in yield; above 190°C, the yield and strength loss may be substantial due to attack on cellulose (Smook, 1982).

Temperature reading are taken at intervals throughout the cook to allow the calculation of the H-factor. H-factor, first developed by Vroom in 1957, is a means of representing the times and temperatures of any cooking cycle as a single numerical value. Its values lies in allowing the comparison of pulping procedures which incorporate differing temperature / time profiles.

At the completion of the cooking cycle, the pulping vessels are promptly removed from the air bath and cooled in a bath of cold water to halt further delignification.

After cooling, the spent cooking liquor (black liquor) is drained off. The softened chips are given a brief wash in cold water to reduce foaming, then the chips are disintegrated. The method used for disintegration often depends on the industrial process being modelled. At CSIRO, the softened wood chips from the 3-litre air bath are disintegrated in a mixer at 2850 revolutions per minute for 10 minutes.

Following disintegration, the pulp is washed with cold water in vacuum / de-water funnels to remove residual black liquor. This is a critical step because residual black liquor will affect the Kappa number and pH of the pulp. The washed pulp is then screened to remove uncooked fibre bundles. At CSIRO, a Packer screen with 0.2 mm wide slots is used for this purpose. The screened pulp is then dewatered, usually with a press, to about 25% consistency, follow with crumbing. The crumbing, which can be done in a large Hobart mixer, assists in distributing the moisture in the pulp evenly. This allows an accurate estimate of the moisture content of the pulp from small samples. The moisture content of the pulp can be measured at this stage. The crumbed pulp is bagged in a sealed plastic bag and stored in a refrigerator to maintain the moisture content.

A.1.2 Mechanical pulping (TMP, CTMP)

A.1.2.1 Asplund defibrator

Mechanical pulping in its various forms has been claimed as the pulping method of the future. The principle of mechanical pulping is mechanically separating wood into its constituent fibres. Chips may first be steamed or pretreated with 6-10% sodium hydroxide (Na^{++}) or calcium hydroxide (Ca^{++}) to soften lignin and to make the separation of the fibre easier.

In this experimental study, thermomechanical pulp (TMP) and chemithermomechanical pulp (CTMP) are prepared in Asplund Type D Laboratory Defibrator, which is equipped with facilities for pre-steaming and subsequently disintegrating wood chips under gauge pressures up to 12 atmospheres (190°C) as described by Higgins (1977). The main variables in TMP (CTMP) are the raw material (species, basic density, chip moisture

content and chip geometry), steam pressure (and corresponding temperature), pre-heating time and defibration time.

The chip charge can be as high as 400g (o.d.) wood, but in practice (e.g. hardwoods) it was sometimes necessary to run with a charge as low as 100g to avoid overload on the 7.5 kw motor. *P.radiata* low temperature range pulps (125-135°C LT-TMP) was reported to yield well fibrillated fibres with good paper-making properties; high temperature pulps (150-170°C HT-TMP) to yield smooth, lignin encased, unfibrillated fibres (Higgins, 1978).

To operate the Asplund defibrator, wood chips are placed in the four compartments of the inner vessel in approximately equal amounts, tighten the lid, check the temperature. After presteaming the chips for 1-2 minutes at low temperature range (120-125°C), start defibrator for 2-3 minutes (1440 rpm) to break chips down into fibre bundles. These fibre bundles are fed through Bauer refiner to form individual fibres (see section A.1.2.2).

For chemithermomechanical pulping (CTMP), chips are soaked in 10% caustic soda (NaOH) over 18 hours at ambient temperature and then processed as in the case of LT-TMP pulps.

A.1.2.2 Bauer refiner

Refining reduces a fibre "bundle" to individual fibres. The key part of a refiner is the refining plates. The refining plates transmit the mechanical energy into the wood fibres, so the primary function of plates is to keep the pulp between the plates.

Refining of Asplund defibrated pulp is done in the Bauer refiner fitted with 203 mm (8 inch) diameter plates (rotor and stator). Although there are two types of refining plates (open periphery and closed periphery plates) (Fig. A.2), Asplund defibrated TMP (CTMP) fibre bundles can be refined to individual fibres by use of closed periphery plates.

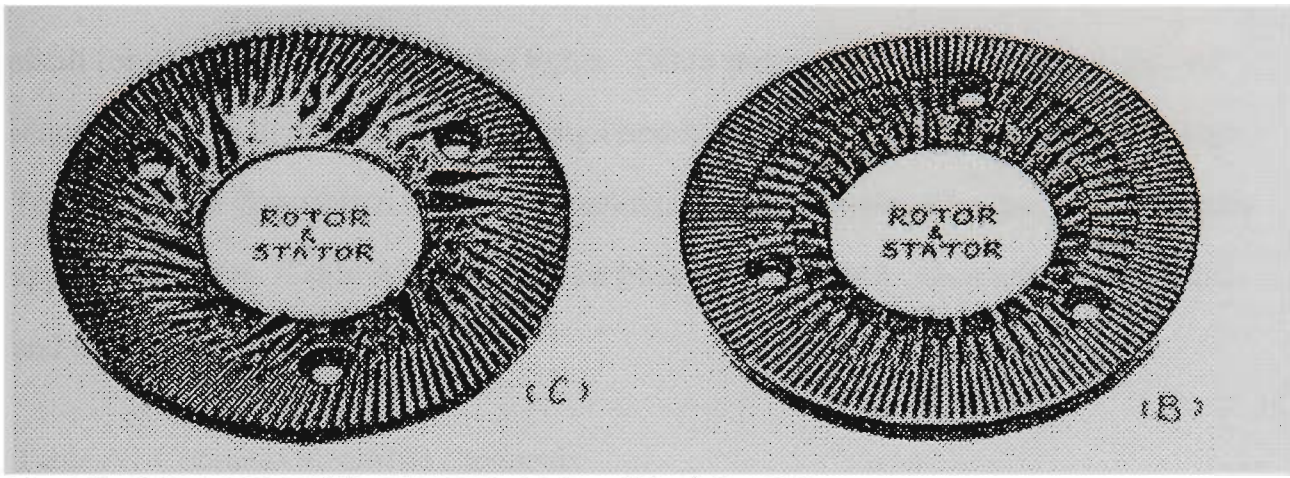

Fig. A.2. Open periphery (C) and closed periphery (B) refining plates.

Refining is achieved by successive passes of the pulps at various plate clearance until the required freeness level is reached. Refining Asplund defibrated *P.radiata* LT-TMP pulps in Bauer refiner fitted with closed periphery plates (rotor: 8117-4122p, stator: 8117-4121p) can be done using the following passes. One pass is made at a plate clearance of 2 mm (0.08 inches), one at 1.25 mm (0.05 inches), one at 0.625 mm (0.025 inches) and two at 0.125 mm (0.005 inches).

Pulp moisture consistency is a major factor to refining efficiency. Water should be kept to a minimum, if too much water is used (consistency < 8%), no work is done on the fibres and therefore no refining takes place. Care should be always taken during reducing the plates clearance not to clash the plates.

A.1.3 Bleaching pulps (Oxygen delignification)

The main objective of bleaching in papermaking is to convert a dark coloured pulp into one which is much lighter in colour. Bleached pulp improves fibre cement composite flexural strength due to fibres are more flexible and better fibre-matrix bonds (Mai, 1983).

Chemical pulps contain residual lignin. This lignin has been extensively modified by the severe conditions used to make the pulps and can be quite dark in colour. It is extremely difficult to change this lignin into a colourless form with bleaching chemicals. So the way to bleach a chemical pulp is to completely remove the residual lignin. This is done in multistage processes using chlorine compounds or oxygen for lignin degradation, and

alkali for extraction of the degraded lignin. Often mixtures of bleaching chemicals or sequential addition of different bleaching chemicals is used in the same bleaching stage. To simplify the description of these sequences, each chemical can be designated with an appropriate letter. Table A1 lists common bleaching chemicals, their usual designation and their bleaching action.

Table A1 Common bleaching chemicals

Chemical	Designation	Bleaching action
Chlorine, Cl_2	C	Lignin degradation
Hypochorite, (NaClO or CaClO)	H	Lignin degradation
Chlorine dioxide, ClO_2	D	Lignin degradation
Alkali, NaOH	E	Extraction of degraded lignin
Oxygen, O_2	O	Lignin degradation or improved delignification in E-stages
Hydrogen peroxide H_2O_2	P	Improved delignification in E-stages

The choice and the conditions of use of the bleaching chemicals are limited because carbohydrate degradation must be avoided. Otherwise, pulp yield would be reduced and strength properties impaired. There are few common laboratory bleaching sequences such as CEHD and $OD_1(EO)D_2$.

The effect of fibre lignin content level on WFRC properties is studied. Low lignin content in the fibres was achieved under oxygen delignification conditions. The pulp responds well to oxygen bleaching and about half of the lignin can be removed easily. The general rule for oxygen bleaching, particularly when applied to softwood Kraft pulps, is that about 40 per cent of the lignin can be removed before the strength properties of the pulps are affected. The viscosity of the oxygen bleached pulps was measured as this property can be indicative of fibre damage if the value is below a threshold level. A viscosity of about 22 m.Pa.s is regarded as the lower limit for an oxygen-bleached northern hemisphere softwood Kraft pulp (Teder, 1991).

Oxygen delignification of the Kraft pulps was carried out with the 3L pulping vessels which were fitted with lids incorporating valves to introduce oxygen into the vessels. Pulp samples (100g o.d.) were mixed with magnesium carbonate (1% pulp basis), sodium

hydroxide (in the range 0.7-2.5 per cent, pulp basis) and water to give a pulp consistency of 10 per cent. The mixtures were placed in the pulping vessels which were pressurized with oxygen (780 kPa) and heated at 115°C for 30 min (time to temperature was 75 min).

A.1.4 Holocellulose pulp

Holocellulose is defined as lignin free, pure cellulose fibres. Holocellulose is used to study influence of fibre strength on WFRC properties. This lignin free fibre allows further Kraft cooking to various fibre strength without the complication of different lignin content products, which might effect the fibre strength determinations.

Sodium chlorite solution at room temperature was used for the delignification. The solution consisted of 60 g sodium chlorite, 20 g anhydrous sodium acetate and 40 ml of glacial acetic acid, made up to a litre with purified water. The moist, unbleached Kraft pulp at about 20 per cent consistency is mixed with sufficient of the chlorite solution to give about 5 per cent consistency and allowed to react at room temperature with occasional mixing and shaking for 24 hours. The pulp was then washed and the chlorite treatment is repeated with fresh solution for another 12 hours. After chlorite treatment the pulp is washed thoroughly with purified water (Stone, 1960).

A.1.5 Beating

The basic purpose of beating is to mechanically condition the fibres for papermaking and manufacturing WFRC. Addition to this, beating plays an important role in the Hatschek process to retain cement and silica particles (Coutts, 1982a). A more general term for mechanical working of pulp is "refining". The term "beating" actually denotes a specific type of refining, but is now commonly used to describe refining in the laboratory. Most laboratory beating methods have a more selective action than mill refiners and produce results that normally cannot be duplicated in the mill. So it is necessary to optimize the refining level with mill equipment and conditions.

A number of laboratory beating devices are in use around the world. The two devices most commonly used are the Valley beater and the PFI mill. The Valley beater (Fig. A.3) is essentially a miniature version of the Hollander beater. Although this device has a long tradition of use, it has some definite limitations and is gradually being displaced by the PFI mill. The principal disadvantage is the difficulty in obtaining reproducibility with respect to other Valley beaters and with respect to the same beater over long periods of time. The problem relates to variable wear patterns on the metal cutting edges. However, a Valley beater is used in our experiments to prepare pulps for composite fabrication because sufficient amount of pulp can be treated in a single run. To operate the Valley beater, a soaked 360g o.d. pulp with 23 litre water is beaten without load for 20 minutes then beaten for further period of time with the bed-plate load of 5.5 kg until the desired freeness level is obtained. After beating the pulp is subjected to dewatering, pressing and crumbing.

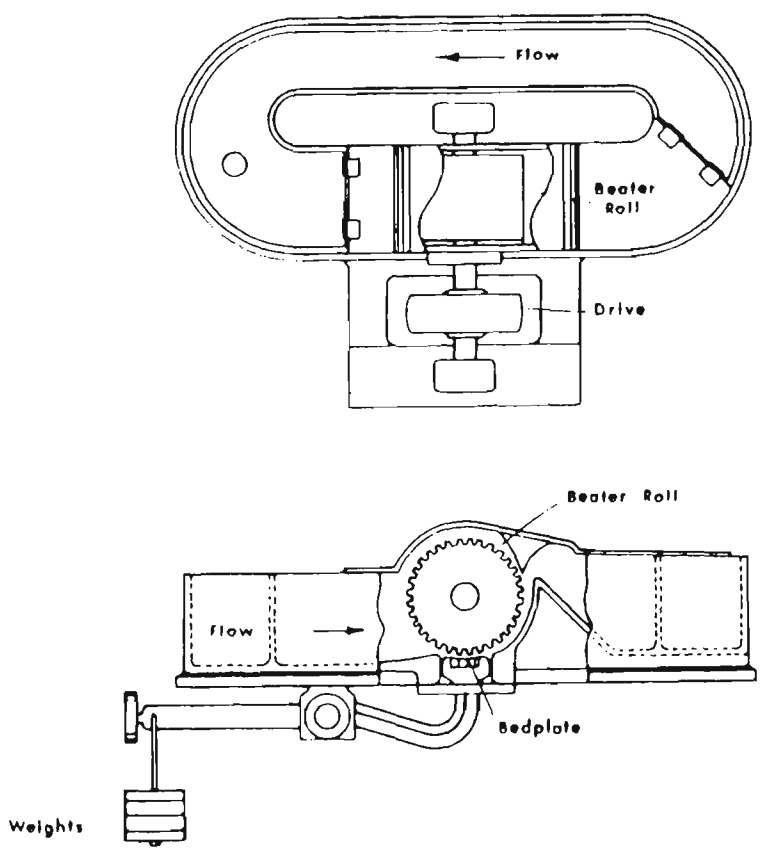

Fig. A.3. The Valley Niagara beater

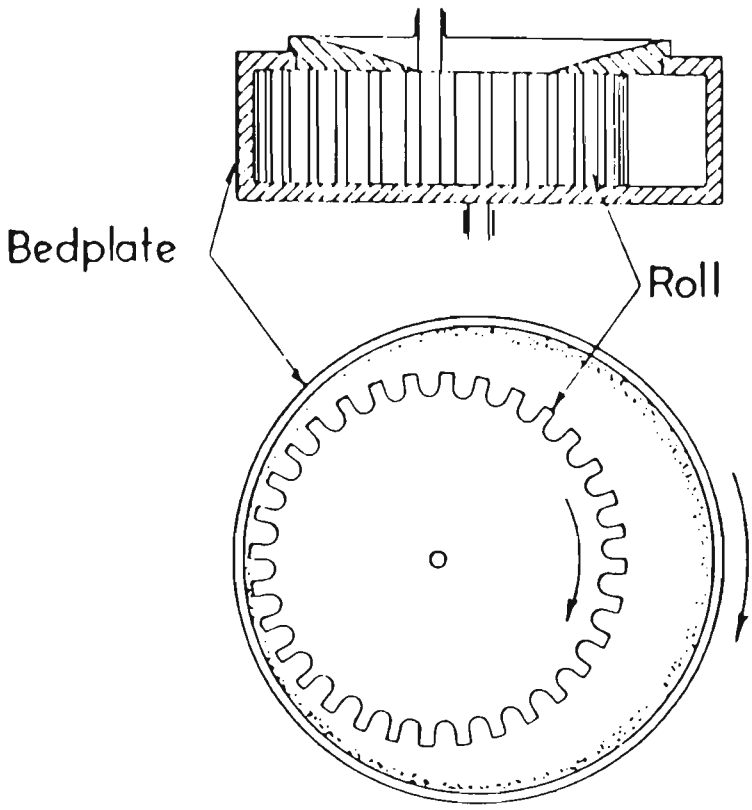

Fig. A.4. The PFI laboratory beater tackle

The PFI mill utilizes a grooved roll eccentric to a smooth trough, as illustrated in Fig. A.4. Both the roll and "bed-plate" rotate at high speed but at different peripheral velocities; this action induces friction, rubbing and crushing of the fibres to produce the beating effect. Since there is no metal-to-metal contact and no edges to wear, the device does not require calibration. It also has the advantage of requiring a relatively small amount of pulp to carry out a complete evaluation.

A.1.6 Preparation of fibres from dry lap-pulp

In mill practise, fibres are usually prepared in the pulp mill and transported in a dry lap-pulp form. In the fibre-cement plant, the dry lap-pulp is soaked in the water to release hydrogen bonds then disintegrated into pulp. Fibres experienced dry-lap distingentation process may lose some initial strength. However, it is more economic and convenient for the fibre-cement plant which has no pulping facility.

In our experimental work, some fibres were prepared from commercial dry lap-pulps or packaging paper. These lap-pulps or paper were torn into relatively small pieces and

soaked in cold water over night. Then they were disintegrated in a mixer at 2850 revolutions per minute for 10 min. Following disintegration, the pulp was dewatered, crumbled and stored in the refrigerator until composite fabrication. The pulp moisture content can be calculated at this stage.

A.2 Pulp Fibre Characterisation

A large number of testing methods are in common use to characterize pulps with respect to quality, process ability and suitability for various end uses. Many of these test procedures are empirical in nature and provide useful behaviour information. Other more "fundamental" tests provide the means to predict behaviour, or to explain and rationalize the empirical test results. A summary of common test methods is given in Table A2. Some of these methods were carried out during work for this thesis, the others are recommended for further study (see Chapter 10).

Table A2 Pulp test methods (Smook, 1989)

Fundamental Properties	Empirical Tests
* weighted average fibre length	* Kappa number
* intrinsic fibre strength	* CED viscosity
* fibre coarseness	* colour and brightness
* specific surface area	* cleanliness
* wet compactability	* drainability
* pulp chemical compositions	* beater evaluation

A.2.1 Lignin content

A.2.1.1 Kappa number

The non-cellulosic components (especially lignin) react readily with acidic permanganate solution ($KMnO_4$). This reaction provides the bases for the Kappa number test. At a controlled temperature, an excess of acidic permanganate is added to the pulp to be tested, and allowed to react for a set time interval, after which the unreacted permanganate is reduced by an excess of iodine. The liberated iodine is then determined by reaction with thiosulfate. The Kappa number of the pulp is equal to the number of ml of acidified 0.02 M potassium permanganate solution which would be consumed by one gram of moisture

free pulp in 10 min at 25°C. The results are corrected to 50% consumption of the permanganate added, which ensures a satisfactory relationship to the lignin content of the pulp.

The test procedures to be followed is described in detail in the TAPPI T236cm-85. This method may be used for all types and grades of chemical and semi-chemical unbleached and semibleached wood pulps in yields under 60 per cent. However, it should be noted that reproducibility is less for high yield pulps than for low yield pulps. For pulps such as TMP and CTMP, Klason lignin method will be more suitable.

A.2.1.2 Klason lignin

Klason lignin is defined as those components of wood or pulps which are insoluble after treatment with 72 per cent m / m sulphuric acid followed by boiling in 3 per cent sulphuric acid. TMP and CTMP pulps are more suited to the Klason lignin method (APPITA P11s-78). In this standard, the lignin content should not be less than 1 per cent to provide a sufficient amount of lignin, about 20 mg, for accurate weighting. It is not applicable to bleached pulps containing small amounts of lignin.

Most woods contain some lignin which is rended soluble by the above treatment and which is not determined by this standard. In softwoods and sulphate pulps this soluble lignin content is small, about 0.2 to 0.5 per cent, but in hardwoods it can amount to 5 per cent. Thus hardwood which has had any alkali treatment, may give a lower results than would be obtained from the untreated wood.

A.2.2 Drainability (Freeness)

The resistance of fibres to the flow of water is an important property with respect to pulp processing, papermaking and fibre-cement composite materials fabrication. The classical method of determining this property in North America and Australia is by means of the Canadian Standard Freeness (CSF) test (Fig A5). The CSF is defined as the number of ml

of water collected from the side orifice of the standard tester when pulp drains through a perforated plate at 0.30% consistency and 20°C.

Measurement of pulp drainage are know as freeness, slowness, wetness or drain time, according to the instrument or method used. If a pulp drains rapidly, it is said to be "free". If it drains slowly, it is said to be "slow". Freeness and slowness scales have an inverse relationship. The Schopper-Riegler Slowness test is the principal drainage measurement used in Europe and Asia.

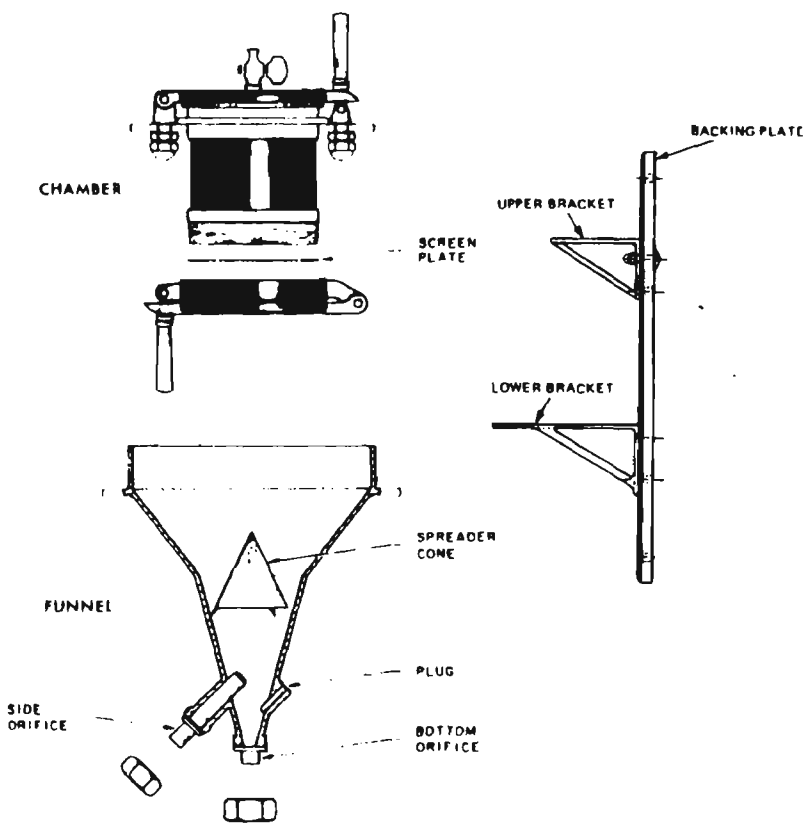

Fig. A.5. Canada Freeness tester

Freeness measurements are widely used as an indication of quality for mechanical pulps and as a measure of the degree of refining (beating) for chemical pulps. Studies have shown that the fines fraction (-200 mesh) is primarily responsible for changes in drainage. The removal of the fines fraction from beaten pulps can restore the original drainability, while the pulp retains its beaten strength properties. These findings are sometimes used as an argument against the use of drainage measurements as an index of pulp quality (Smook, 1982)

Although freeness measurements provide a basis for comparing similar pulps, the test does not simulate what happens on the paper machine wire. For example, groundwood pulp gives a lower freeness than highly beaten chemical pulp but shows faster drainage on the paper machine. Furthermore, the same freeness value does not indicate the same degree of fibrillation. Bamboo pulp might have same freeness value as a softwood pulp, but the degree of fibrillation for the bamboo pulp would be much less than that for wood pulp due to the fact that the bamboo pulp has or average short fibre length, massive pitch fines and sensitive response to the beating force.

A.2.3 Fibre length

Fibre length is measured or indicated either by microscopic examination of a representative number of fibres or by screen classification of a sample into different length fractions. In the microscopic method, a known weight of fibres is projected onto a grid pattern; all the fibres are measured and the average fibre length is calculated mathematically.

In the classification method, a dilute dispersion of fibres is made to flow at high velocity parallel to screen slots, while a much slower velocity passes through the slots. In this way, the fibres are presented lengthwise to a series of screen with successively smaller mesh openings, and only the fibres short enough not to bridge the opening pass in to the next chamber. The Bauer-McNett Classifier is one of the traditional instruments (Fig. A.6).

In the last few years, a new optical device, the Kajanni FS-200 Fibre Length Analyzer has become available for measuring fibre length. It is now widely used and is becoming accepted as a standard laboratory fibre length measurement (TAPPI T271pm-91). The FS-200 measures fibre length by an optical technique using polarized light and is based on the birefringence of the wood fibres (Fig. A.7). The machine employs a measurement range of 0 - 7.2 mm, divided into 144 classes, each of which represents a 0.05 mm interval in length. When the pulp sample (average number of fibres 15,000 to 30,000) passes through

the analyzer, the number of fibres in each classification is counted. This data is fed to a microprocessor unit which routinely records, calculates and displays the fibre length average in three modes: arithmetic, length-weighted and weight-weighted.

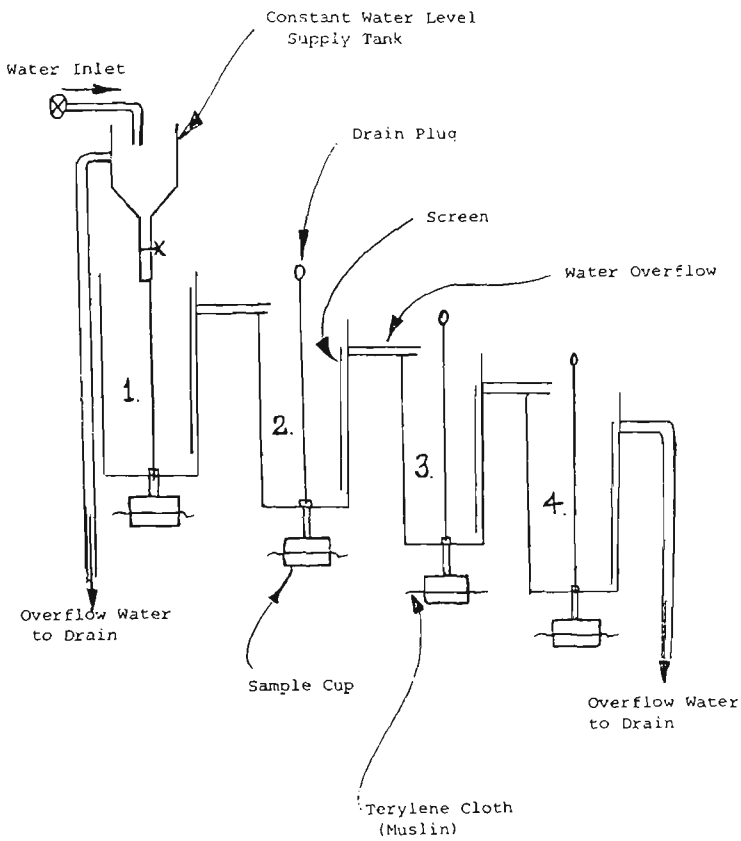

Fig. A.6. The Bauer-McNett Classifier

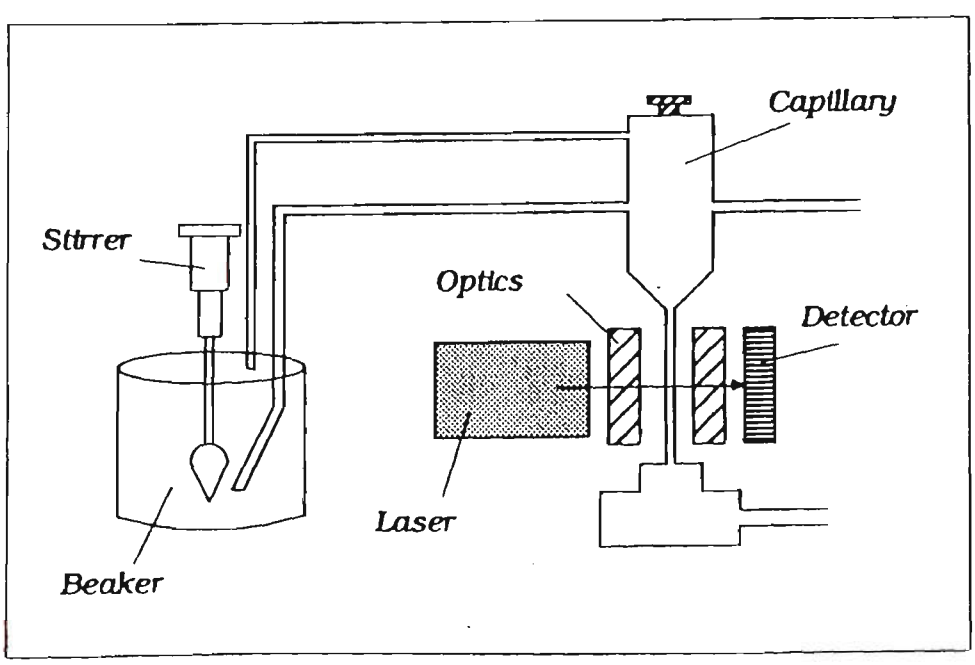

Fig. A.7. Kajanni FS-200 measurement principle

There is no singularly accepted definition of what is the average fibre length of pulp in a sample. The generally agreed approach is to the definition best suited to the nature of the sample under consideration.

If the sample is made up of fibres which are of fairly uniform length, the numerical or arithmetic, average fibre length L_n is most applicable (1). However, this definite is not applicable when, as is most frequently the case, the sample contains a high proportion of short fibres or fines. The use of a measurement which is weighted according to the weights of the fibres is then preferred. The length-weighted average fibre length L_{lw} is defined for the case where the fibre coarseness, the weight per unit length, f_i is assumed to be constant (e.g. $f_i = C$) (2); the weight-weighted average fibre length, L_{ww} is defined for the case where the fibre coarseness is assumed to be proportional to the fibre length (e.g. $f_i = C \cdot l_i$) (3).

$$L_n = \sum l/N = \sum n_i l_i / \sum n_i \qquad (1)$$

$$L_{lw} = \sum w_i l_i / \sum w_i = \sum (n_i l_i C) l_i / \sum (n_i l_i C) = \sum n_i l_i^2 / \sum n_i l_i \qquad (2)$$

$$L_{ww} = \sum w_i l_i / \sum w_i = \sum (n_i l_i f_i) l_i / \sum (n_i l_i f_i) = \sum (C n_i l_i^2) l_i / \sum (C n_i l_i^2) = \sum n_i l_i^3 / \sum n_i l_i^2 \qquad (3)$$

Where l_i represents the average length of fibres in the i^{th} fraction, n_i represents the number of fibres in the i^{th} fraction and w_i represents the weight of fibres in the i^{th} fraction.

According to Clark (1985), the coarseness of natural fibres increases with fibre length and he therefore favour the use of the weight-weighted form. However, the use of the length-weighted average fibre length is preferred by Kajanni researchers who consider it to give a better prediction of the paper making potential of the fibres. Accordingly, in the current study the length-weighted average fibre length is employed.

A.2.4 Handsheet preparation

Many papermaking tests require more than one sheet of paper, so it is convenient to prepare, under identical conditions, several paper sheets from each pulp sample. These paper sheets, called handsheets, must be as uniform as possible. Two sheetmaking

systems are refered to in the ISO standards, conventional sheet former and the Rapid-Köthem former. The method to make handsheets on the conventional sheet former is introduced below.

The first step in preparing pulp for handsheets is to decide what beating points will be used. Since beating changes the properties of pulp and paper, it is usual to make a set of handsheets from pulp beaten to at least four different levels, one unbeaten and three at progressively higher beating revolutions. This allows curves to be drawn which can be used to interpolate paper properties.

Having decided on the amount of beating, the next step is to disperse the specified quantity of pulp thoroughly in water. This is usually achieved with a disintegrator of specified design [ISO 5263-1979(E)].

The amount of pulp needed is determined by the grammage (mass per unit area) of the handsheets and the number of sheets to be made. Typical handsheet grammages are 60.0±3.0 grams per square metre (calculated on an oven dry basis) for ISO standards 5269/1-1979 (E) using a conventional sheet former.

After disintegration, the pulp is subjected to beating and measurement of the freeness. The pulp is diluted with water to a stock concentration suitable for the preparation of handsheets at the selected grammage. This is conveniently done using a stock divider, which provides continuous agitation of the pulp suspension to maintain stock uniformity. An appropriate volume of stock is taken from the stock divider to make the first handsheet. The oven dried weight of this first handsheet can be used to adjust the amount of water in the stock divider so that later sheets have the desired grammage. The details of the method used for sheet preparation should refer to ISO 5269/1-1979 (E).

After the handsheets are prepared, they are dried under conditions designed to prevent shrinkage. In the conventional sheetmaking equipment, this is achieved by attaching the

wet sheet to a rigid drying plate using a press, then allowing the sheet to air-dry in contact with the plate.

A.2.5 Fibre strength

The strength of a fibre is fundamentally attributable to the fibril angle (the angle at which the cellulose molecules spiral about the fibre axis) and the absence of "weak spots" in the fibre due to excessive breaks in the cellulose molecular chain. Such cleavage is predominantly a consequence of the pulping and bleaching processes employed to isolate and purify the pulp fibre from the wood structure where it originates (see section 3.5.2). Since breaks in the molecular chain will reduce the average chain length, it follows that the viscosity test will monitor the extent of molecular chain breakage.

A standard viscosity test is conventionally used to measure pulp strength. Technically, it measures no such thing, when carefully done, it provides a measure of the average chain length of the cellulose molecules obtained by dissolving the pulp sample in a suitable solvent, such as cupriethylenediamine solvent.

A zero-span tensile test on a sheet of paper measures the average strength of the fibres which are carrying the tensile load when failure occurs. It thus depends on both the number of fibres and their average strength and is a basic measure of fibre quality. Comparison of fibre quality, particular in a given mill environment, are generally made at close to the same fibre conditions. When this is the case, the zero-span tensile test is a comparative measure of fibre strength.

It is generally understood that the loss of fibre strength at high α-cellulose contents results from the degradation of cellulose. Homogeneous degradation is random, causes little strength loss and can be indicated by either zero-span tensile strength or pulp viscosity values. However, localized degradation in strength loss is unlikely to be recoverable and measurements of pulp viscosity may be misleading (Gurnagul, 1992). Thus care should always be taken when interpreting the strength results.

A.2.5.1 Viscosity of pulp (capillary viscometer method)

TAPPI T230 om-89 viscosity of pulp method describes a procedure for determining the viscosity of 0.5% cellulose solution, using 0.5M cupriethylenediamine as a solvent and a capillary viscometer.

To measure pulp viscosity the general procedure includes:

(1) weigh 0.2500 g air-dried, moisture free pulp into dissolving bottle;

(2) add 25.00 ml distilled water and shake to disperse the pulp;

(3) add 25.00 ml cupriethylenediamine solution and purge with nitrogen for 1 min;

(4) cap the bottle and shake for few hours until the fibre is completely dissolved;

(5) conduct viscosity measurement, the viscometer size is selected to give efflux times of over 100s, but less than 800s, and two specimens' efflux time should within ±2s range.

(6) viscosity is calculate as $V = Ctd$

where V: viscosity of cupriethylenediamine solution at 25.0°C, m·Pa·s(cp)

C: viscometer constant found by calibration

t: average efflux time, sec

d: density of the pulp solution, g/cm^3 (=1.052)

A.2.5.2 Zero-span tensile test

The zero-span breaking length appears to be an index of the breaking length (or tensile strength) of a pulp beaten to its maximum value under ideal conditions. Consequently, it is an excellent measure of the "maximum strength" of a pulp, and is almost completely independent of the laboratory beating procedure used. However, zero-span test as a direct measure of fibre strength suffers from the fact that changes in fibre length and / or interfibre bonding influence the test value. A simple way to eliminate the influence of interfibre bonding is to wet the sheet of paper prior to testing (wet zero-span test). Fibre length influence can be eliminated by adjusting the initial span to zero without any finite value.

In our laboratory, wet zero-span tensile strength is conducted on a Pulmac Zero-span Tester (Fig. A.8). To do this, cut the handsheet into a 20 mm by 100 mm strip, wet the strip and clamp between two jaws (at zero span position). Then apply the tensile force to fracture and calculate zero-span tensile strength (ZSTS, kN / m) and its index (ZSTI, Nm / g) as follows:

$ZSTS = (P - P_0) \times C \times 0.654$

ZSTI = ZSTS x 1000 / Grammage (g/m^2)

Where: P is instrument reading, P_0 is the conversion factor constant for zero opening and C is the instrument factor, about 0.370.

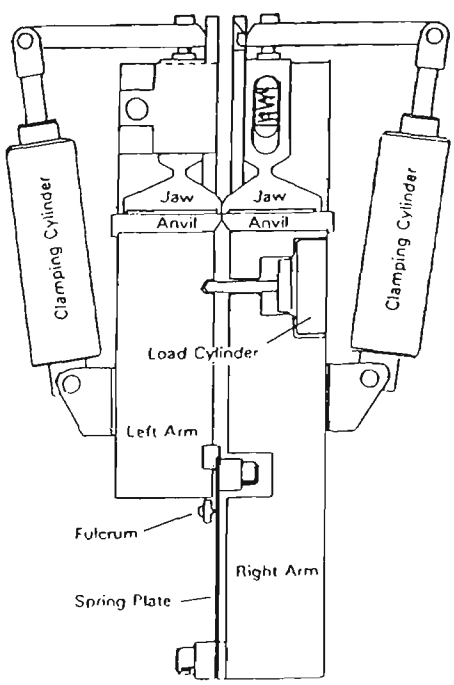

Fig. A.8. Schematic illustration of Pulmac Zero-span tester.

A.3 Fabrication composite material

It has been reported in Chapter one that cellulose fibre-cement sheets are commercially manufactured on a Hatschek machine. In our small scale laboratory work the composite materials are prepared by "slurry / vacuum dewatering" technique which emulates the commercial Hatschek process. This technique includes slurrying the fibre-cement formulation, vacuum dewatering, press and autoclaving (or air-curing).

A.3.1 Materials

The matrix was made from fresh commercial grade ordinary Portland cement (OPC) (Australian Cement, Geelong, Type A) and finely ground silica (Steetly brand 100WQ). The matrix grade and particle size might be important to material final properties, however, such study is not included in this work. Considerable work in this area would be done by the manufacturing companies themselves.

During the manufacturing process or laboratory work, attention should be paid to storage of the cement. At all stages up to the time of use, cement must be kept dry so as to prevent or minimize deterioration from the effects of moisture, atmospheric humidity and carbonation. In our experimental work, cement was purchased from local building materials store, then batched and sealed tightly into several plastic jars. It is suggested that for cement which is old than four months should be classified as "aged" and re-tested before use (Taylor, 1969).

Table A3 Natural fibre reinforced cement composites ingredients

Fibre % by mass	water (ml)	28-day Air-curing	175°C, 7.5h Autoclaving
2	300	OPC	OPC:Silica 1:1
4	350	"	"
6	400	"	"
8	500	"	"
10	500	"	"
12	500	"	"

It is usually necessary to add other raw materials or special additives to the fibre cement furnish / products, such as flocculating agents (processing aid), PFA (cheap filler / pozzolanic), ball clay (interlaminate bond agent), microsilica (void filler / pozzolanic / interlaminate bond agent), AH_3 (reduction moisture movement). Hence the choice of one or more of these special additives enables the designed properties of the end products to be obtained. However, no additives were used in this thesis work.

The reinforcing fibres were prepared by the method described in section A.1. Wood fibre (*P.radiata*) was supplied from Australia APM, Maryvale mill as specially selected high tear wood chips and high tear Kraft pulped unbleached dry lap paper forms. Bamboo fibre [*Sinocalamus affinis (Rendle) McClue*] was obtained from Kraft pulped unbleached commercial packaging paper from Chang Jiang Paper Mill and Jian Xi Paper Mill, China.

A.3.2 Slurry / Vacuum dewatering and press technique

Each sample is based on a 130g dry weight of ingredients (Table A3). As the moisture content of the pulp is known, thus the appropriate weight of pulp fibre (oven dried base) can be determined and then suspended in 300 to 500 ml of water (see Table A3) with stirring for 2 minutes. In some cases, weighed moist crumbled pulp was suspended in much more water and disintegrated for a total of 12,500 revolutions to ensure fibre dispersion, then dewatered to required amount. The preweighed matrix ingredients were mixed thoroughly and then slowly added to the stirred suspension of fibres. Rapid stirring took place for 5 minutes and the mixture was poured into an evacuable casting box (125 mm by 125 mm) with filter paper and fine wire screen (Fig. A.9) so that it could be distributed over the screen. An initial vacuum was drawn until the sheet appeared dry on the surface and then the sample was flattened carefully with a tamper. A vacuum of 60 KPa (gauge) was applied for a total of 2 min. The sheet was then removed from the filter screen. The sheet and screen were stored between two steel plates and the procedure repeated until a stack of four sheets had been prepared. The stack of sheets was then pressed for 10 minutes at a pressure of 3.2 MPa. During the pressing extracted water was blown away by compressed air and the load was applied and / or released slowly to prevent any damage to the sheets.

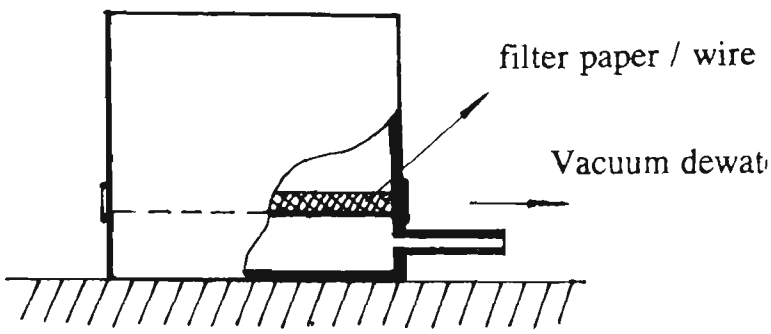

Fig. A.9. Vacuum dewatering casting box

Coutts and Warden (1990) studied the influence of casting pressure and time on the WFRC mechanical and physical properties for the slurry / vacuum dewatering technique. They found that increasing the casting pressure and time resulted in improving composites' mechanical properties and increasing the density of the composites. They suggested that composites containing 8-10% fibre loadings can usually achieve satisfactory properties when subjected to pressure for a short application time, whereas high fibre loadings might need longer press time.

After pressing, the screens were carefully removed from the sheets which were then stacked flat in a sealed plastic bag for 24 hours prior for curing.

A.3.3 Air-curing and autoclaving

There are few cement curing models such as water curing, air-curing and autoclaving. Air-curing and autoclaving are used for fibre reinforced cement products. Cement composites are cured under ambient conditions and will take 28 days to achieve their "full" strength (samples remained in the plastic bag for 7 days then cured under the ambient conditions). The air-cured method saves on capital investment in an autoclaving facility and has superior properties than those of autoclaved products. Air-curing model is suitable for on-site fabrication and for manufacturing polymer fibre reinforced cement products due to mild curing conditions.

In both Australia and Europe, the manufacture of natural fibre reinforced cement composites have been based on sheet products formed by techniques akin to the Hatschek process and cured in high-pressure steam autoclaves. Steam at temperatures close to 180°C enables the replacement of between 40 to 60 % of ordinary Portland cement by the less expensive silica, which can react with the cement to form a calcium silicate matrix of acceptable strength (Lea, 1976). The reaction is completed within hours [125 psi (180°C), 8 hours] instead of air-curing which takes weeks to achieve full strength. So the storage facilities can be less as turnover is faster.

The conditions for autoclaving were initially studied by Coutts and Warden (1984b). Composites autoclaved under 180°C, 125 psi steam, 8 hours at cement / silica ratio 1:1 demonstrated the best mechanical properties (see Fig. A.10 and A11). At that condition, fibre was not expected to be subjected to significant degradation while the composite achieved adequate strength. However, there is a need to optimize the autoclaving condition with different fibre sources, matrix ratios and fabrication process.

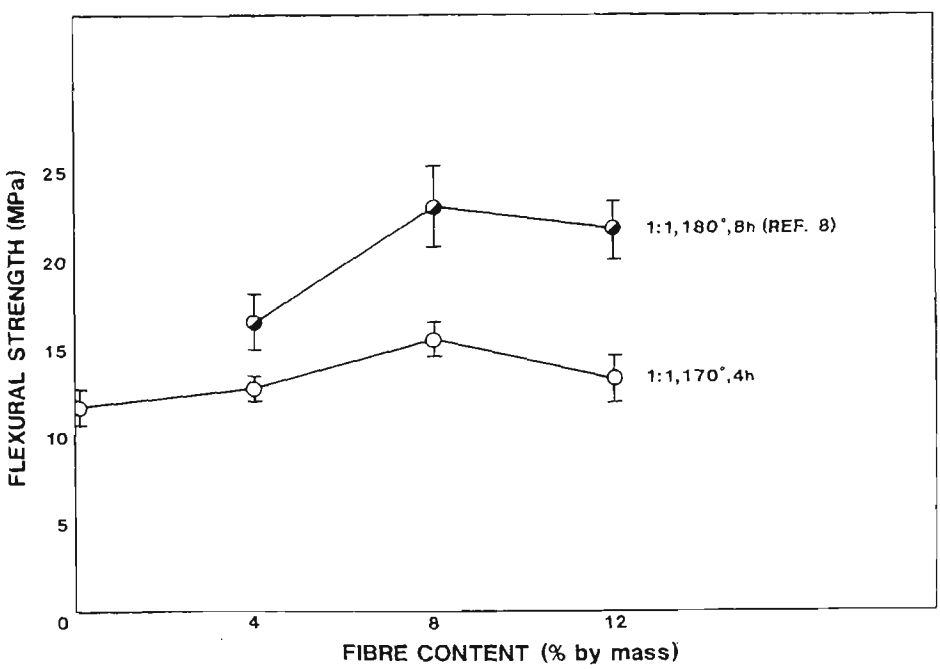

Fig. A.10. Optimize autoclaving temperature and time

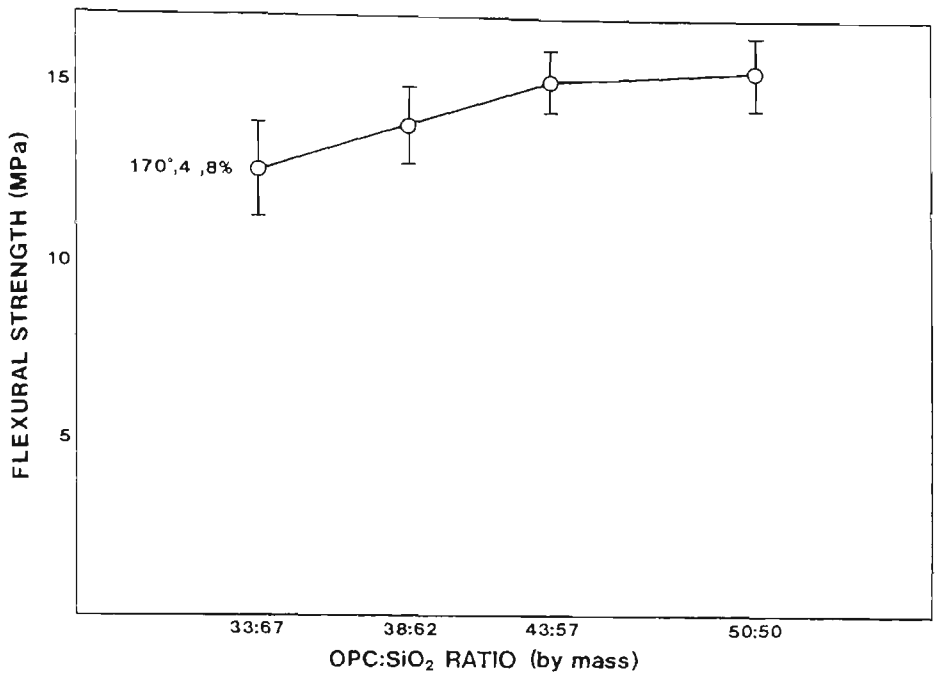

Fig. A.11. Optimize composite OPC:Silica matrix ratio for autoclaving

A.4 Characterisation of composite materials

Specimens were cut with a diamond saw to specified dimensions and stored under a controlled atmosphere of 50±5% relative humidity and 22±2°C for 7 days before testing.

Specimens measuring 125 mm by 40 mm (of varying thickness) were tested for flexural strength and fracture toughness values. The flexural strength was measured as the modulus of rupture (MOR) in three-point bending as: $3PL / 2bd^2$, where P is the maximum load recorded during the test, L is the specimen span, b is the specimen width and d is the specimen depth. A span of 100 mm and a deflection rate of 0.5 mm/min was used on an Instron testing machine (Model 1114). The results of the tests were obtained using automatic data collecting and processing equipment. The fracture energy was calculated from the area under the load / deflection curve when the failed specimen reached 50% maximum load. The fracture toughness is given by the fracture energy divided by the cross-section area of the specimen. The comparison of fracture energy or fracture toughness is strictly only valid for specimens of the similar thickness.

Water absorption, density and apparent void volume physical properties were obtained using the methods laid down in ASTM C948-81, which measures specimen dry weight, wet weight and amount of water the specimen displaced.

In all cases, at least six specimens were tested for MOR, fracture toughness, density, void volume and water absorption. 3 standard deviations have been included for all properties measured.

Appendix B:
Determination of Kraft Pulping Parameters

Consider a wood chip sample of X% moisture content. The requirement is to pulp A gm equivalent oven dried chips at Y% active alkali and Z% sulphidity at a liquor to wood ratio of R:1.

1. Wt of air dried chips required = A/(100-X)*100 = B gms;
2. Grams of active alkali required = A*Y/100 = C gms;
3. At Z% sulfidity, grams sulfide required = C*Z/100 = D gms;
4. Vol. of Na_2S soln., = D*1000/ [Na_2S] = E mls;

   where Na_2S is concentration of Na_2S solution in g/l (as Na_2O);
5. For NaOH in Na_2S soln; gms NaOH in Na_2S soln = E*$[NaOH]_2$/1000 = F gms;

   where $[NaOH]_2$ is concentration of NaOH in Na_2S solution in g/l (as Na_2O);
6. Grams of NaOH reqd. = C-D-F = G gms;
7. If concentration of NaOH solution is $[NaOH]_1$,

   then vol. NaOH = G*1000/$[NaOH]_1$ = H mls;
8. Total liquid required = A*R = I mls;
9. Mls of H_2O in wood chips = B-A = J mls;
10. Vol. of to H_2O be added = I-(J+H+E) = K mls.

Hence charge per bomb is:

1. B gms of air dry chips equivalent to A gms oven dried;
2. H mls of $[NaOH]_1$, of NaOH solution;
3. E mls of Na_2S solution;
4. K mls of water.

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