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The influence of electromagnetic fields from two commercially available water-treatment devices on calcium carbonate precipitation

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ABSTRACT: The marketing and implementation of commercially available pulsed-electromagnetic field (PEMF) devices to ostensibly control scaling, in processes such as reverse osmosis (RO) and cooling-tower installations, is based on the notion that such devices enhance the coagulation of inorganic particles such as calcium carbonate. In order to provide a scientific basis for such claims, the precipitation characteristics of calcium carbonate under the influence of the PEMFs from two commercially available devices has been investigated under controlled conditions. Thus the rate and profile of calcium carbonate precipitation in the presence and absence of PEMF exposure of parent calcium nitrate and sodium carbonate aqueous solutions was tracked in parallel by UV absorption at 350 nm and by turbidity measurements. The morphology of the corresponding crystalline precipitates was, at the same time, also assessed using SEM. From these studies, is apparent that exposure of the parent solutions to the PEMF from one of these devices in particular can influence both the profile of $CaCO_3$ precipitation and the morphology of the resulting microcrystals.

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1. INTRODUCTION

Calcium carbonate precipitation is a major problem in the operation of Reverse Osmosis (RO) membrane systems for desalination and water reuse applications, and also in cooling-tower installations and industrial refrigeration plants (Y.I. Cho, Fridman, Lee, & Kim, 2004; Young I. Cho, Lane, & Kim, 2005). The types of scale in RO membranes have been categorized as being alkaline (e.g. calcium carbonate), non-alkaline (e.g. calcium sulphate) and/or silica based (Antony et al., 2011; Greenlee, Lawler, Freeman, Marrot, & Moulin, 2009). Calcium carbonate, $CaCO_3$, is usually the main precipitate in seawater RO (Conway, 2002; Greenlee et al., 2009). When water temperature increases, the solubility of calcium carbonate decreases (Young I. Cho et al., 2005; Coetzee, Yacoby, Howell, & Mubenga, 1998) and when dissolved or suspended minerals precipitate they are attracted to the membrane surface due to their natural charges (Bisbee, 2003; Thompson et al., 2012) and form crystals (Kobe, Dražić, McGuiness, & Stražišar, 2001). Scaling results in permeate flux decline and crystals can damage the active membrane layer (Valavala, Sohn, Han, Her3, & Yoon, 2011). Harsh chemical cleaning cycles can also damage the membrane and shorten its lifetime (Conway, 2002).

The terms “non-chemical water treatment systems” or Non-Chemical Devices (NCDs) used for calcium carbonate precipitation management include magnetic (permanent/electro-magnetic)~~, Pulsed~~, Pulsed Electromagnetic Field (PEMF) technologies (Duda, Stout, & Vidic, 2011), solenoid coils, electrostatic devices, catalytic surfaces, ultrasound and devices, turbulence, and vortex flow (Y.I. Cho et al., 2004) and are considered as pretreatments rather than techniques for cleaning of existing scale. Anti-scale magnetic treatment of hard water has been employed for more than 100 years (Benson, Lubosco, & Martin, 2000; Harfst, 2010; Lipus, Acko, & Hamler, 2012). Magnetic water treatment consists of passing water through a magnetic field of certain characteristics (Gilart et al., 2013) and the magnetic field may be generated by fixed magnets or electromagnets.

The definitions of ‘AC induction methods’, electromagnetic water treatment,’ ‘Electronic Anti-fouling Technology (EAF)’ and ‘electronic water treatment’ are closely related and the general understanding is that the water being treated is passed through a PVC or stainless steel pipe where an energized solenoid cable is wrapped around the pipe; there is no connection between the coils and the treated solution (Bisbee, 2003; Huchler, 2002). Voltage on the coils is varied quickly (in the hertz (Hz) to megahertz (MHz) frequency range) in very complex ways (Dresty, 2012; Huchler, 2002) leaving detection or the measurement of the induced field difficult. Devices in this class utilize pulsed low (60Hz) and high (10 kHz to 100 kHz) frequency electromagnetic fields (Patton & Alley). Therefore devices in this category are distinguished from magnetic water treatment devices due to production of a pulsing, coil resonating (or “ringing”) harmonic field across the flow gradient as it passes through the system (Lane & Peck, 2003).

When a liquid flows through a pipe which is connected to a driver enclosure that produces a pulsating current, it is thought that the pulsating current creates time-varying magnetic fields inside the pipe and an induced electric field (Alley, Puckorius, & Kienle, 2008). Such an induced electric field is believed to provide “molecular agitation” where ions collide and precipitate in a unique way by modifying the natural surface charge of the particles (Alley et al., 2008), instead of depositing onto heated surfaces (Y. L. Cho, Fan, & Choi, 1997). Also, due to such treatment, it is reported that there might be a change in crystalline structure (Y. L. Cho et al., 1997). However, such theories remain controversial.

Manufactures of commercial Pulsed Electromagnetic Field (PEMF) devices that are available for water treatment also claim that their devices “activate” small-suspended particles in the water by removing their static electric charge (Tomczyk, 2011). Such activated particles seed the co-precipitation of dissolved minerals, which would otherwise remain in solution and precipitate onto equipment surfaces. Activated mineral-coated particles are thought to be easily removed by various physical means, such as filtration or centrifugal separation (ClearwaterSystemsCorporation, 2008; Griswold, 2011; Tomczyk, 2011). However, there are some reports that such treatment has no observable effect on boiler scaling (Y.I. Cho et al., 2004) other studies support such devices being used for scaling management (Young I. Cho et al., 2005).

The research described herein represents a “proof-of-principle” study to evaluate the efficacy of two different commercial PEMF devices for their ability to modify calcium carbonate precipitation.. Both devices were evaluated in terms of their comparative frequencies and wave form characteristics. Calcium carbonate, $CaCO_3$, precipitation was induced by mixing analytical grades sodium carbonate, Na_2CO_3, and calcium nitrate, $Ca(NO_3)_2.4H_2O$. Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ aqueous solutions were pre-exposed to PEMF (and then carefully mixed and the $CaCO_3$ was allowed to precipitate under controlled conditions). Precipitation profiles were tracked via changes in turbidity and uv absorbance at 350 nm and crystal size and morphology was assessed using light micrscopy and SEM.

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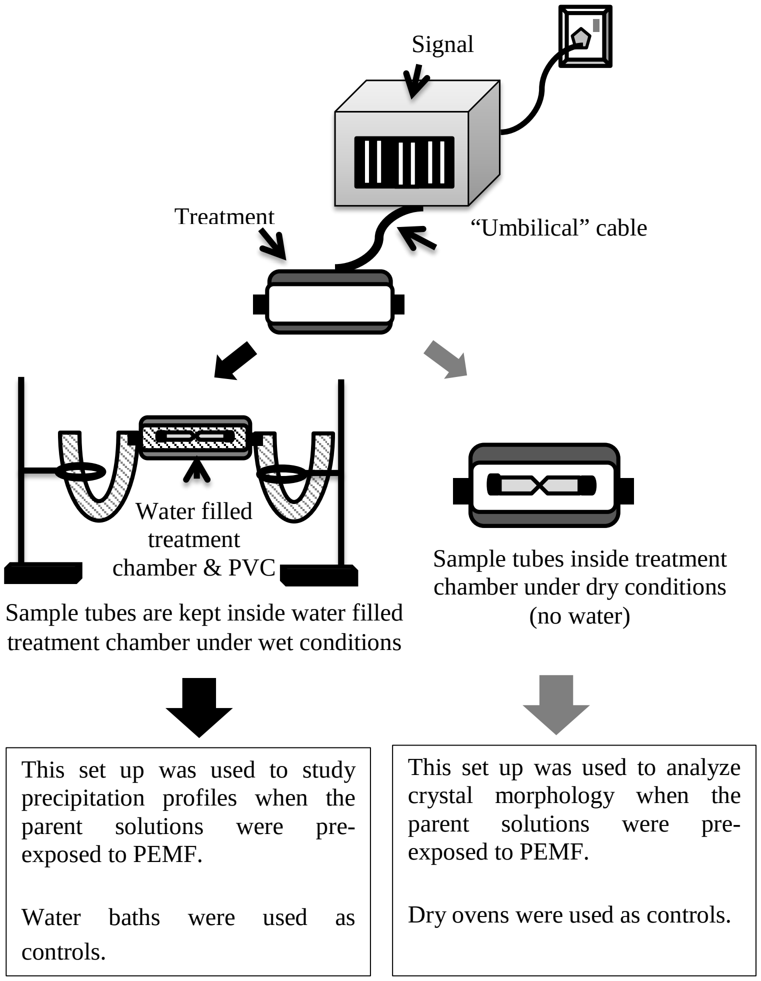
2. MATERIALS AND METHODS

2.1 Test apparatus

Two PEMF devices, designated ‘D’ and ‘G’, were purchased from different commercial suppliers. Both the units share common features; namely a signal generator housing the power and control components and a treatment ~~chamber which~~chamber, which is connected to the signal generator via an “umbilical” cable, Figure 1. The treatment module allows water to pass through a tube whereby it can be exposed to the PEMF. The test apparatus was employed in either ‘wet’ or ‘dry’ conditions.

For the ‘wet’ conditions two lengths of approximately 2.54 cm diameter PVC tubing were attached to both ends of the treatment module and elevated at both ends to contain 600 mL of water, which was allowed to equilibrate to the ambient temperature of ~ 22 °C. The PEMF device was then switched ON. After 4 hrs it was determined that the setup was stabilized both thermally (and electronically). Interestingly, the thermal stabilization of the two devices was different, with the water temperature after 4 hrs being ~ 40 °C and ~ 27 °C for D and G, respectively. As controls, two static water baths were maintained at ~ 40 °C and ~ 27 °C and into which analogous tubes were placed. The relative characteristics of the two devices have been previously reported (Piyadasa et al., 2016). For static exposure of the Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ solutions two tubes were inserted into the treatment module, as depicted in Figure 2.

In order to study the effects of the PEMF without cooling mechanisms, the devices were used without the PVC arms or the cooling water, where the solution tubes were placed inside the treatment chamber in a ‘dry’ (without surrounded by cooling water) environment. No pre-stabilization period was used and the maximum treatment/exposure time was limited to 30 min to prevent the device from over-heating. As controls, two laboratory ovens were maintained at ~ 40 °C and ~ 27 °C into which analogous tubes were placed.



Sample tubes are kept inside water filled treatment chamber under wet conditions

Sample tubes inside treatment chamber under dry conditions (no water)

This set up was used to study precipitation profiles when the parent solutions were pre-exposed to PEMF.

Water baths were used as controls.

This set up was used to analyze crystal morphology when the parent solutions were pre-exposed to PEMF.

Dry ovens were used as controls.

Figure 1 Schematics of the two different static PEMF testing set ups developed for the study of the commercial devices in either wet or dry testing conditions.

Turbidity tests: 5 mM analytical grade Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ were prepared in chromatography grade pure water from 0.1 M stock solutions. 5.0 mL from each solution were simultaneously added into the 10 mL glass cuvette of a turbidity meter (Hanna

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Instruments, HI 88703 serial B0005085). The sample was vortexed for 10 secs just before t=0 and initial turbidity was measured within 50 secs after first mixing. The turbidity was then recorded for the next 30 min with 2 min sampling intervals.

Absorbance tests: 8 mM analytical grade Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ were prepared from 0.1 M stock solutions. 1.0 mL of each solution was added into 2 mL spectrophotometer cuvette and absorbance was recorded at 350 nm every 2 min for a duration of 30 min using a spectrophotometer (Biochrom, Model Libra S11, Cambridge, England).

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Effects of PEMF on turbidity and absorbance: When studying turbidity and absorbance from PEMF-pre-exposed parent solutions, 5 mL of either 5mM or 8mM Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ contained in two 10 mL screw cap graduated tubes (Techno Plas, St Mary's, South Australia 5042) were placed inside the treatment tube for each PEMF unit. Solution containment in plastic tubes during exposure has been reported in similar studies (Chibowski, Holysz, Szcześ, & Chibowski, 2003). Static PEMF treatment was carried out for 3 hr under wet conditions and 30 min under dry conditions. At the end of the exposure, the solutions were mixed and turbidity and absorbance profiles were recorded over 30 min. A similar set of solutions tubes were placed inside static water baths or an oven (water baths were used as controls for wet conditions and ovens were used as controls for dry conditions) that were maintained at similar temperatures to each device as control experiments.

Scanning Electron Microscopy (SEM) Images of precipitated material: To examine crystal morphology, 1M parent solutions were ~~used,~~mixed, to obtain a generous amount of precipitate. 1M solutions (both PEMF exposed and control) were centrifuged at 4000 rpm for 10 minutes. The precipitate was washed in absolute ethanol and then with diethyl ether. The washed precipitate was transferred into small petri dishes and stored in a desiccator. The dried samples were mounted onto stainless steel studs of SEM and observed under High Vacuum 5kv X1000 magnification.

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3. RESULTS AND DISCUSSION

3.1 “Establishment” $CaCO_3$ precipitation profiles

In order to evaluate the appropriate experimental parameters for the precipitation process, preliminary laboratory studies were performed without PEMF treatment. Once reproducible conditions had been established, replicate experiments were conducted under the influence of PEMFs. Thus $CaCO_3$ was precipitated from appropriate concentrations of analytical grade aqueous Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ solutions, according to the reaction:

$$Na_2CO_3(aq) + Ca(NO_3)_2.4H_2O(aq) \rightarrow CaCO_3\downarrow + 2NaNO_3(aq) + 4H_2O$$

All the chemicals used were of analytical grade, including the water (Pang, 2013), and UV absorbance at 350 nm and turbidity were used to track the precipitation process. Parent aqueous solutions of 5 mM were used for the turbidity tests, as suggested by Colic and co-workers (Colic & Morse, 1998) and solutions of 8 mM were used for absorbance tests (Higashitani, Kage, Katamura, Imai, & Hatade, 1993). In both cases measurements were obtained over 30 min at 2 min intervals. These specific concentrations were required in order to obtain “well-behaved” profiles.

Figure 2 (a) and (b) depict the $CaCO_3$ precipitation profiles in the absence of exposure of the parent solutions to a PEMF, as tracked by turbidity measurements and UV absorption at 350 nm, respectively.

Turbidity: From Figure 2(a) it can be seen that when 5 mM Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ were mixed without any pre-exposure treatment, the turbidity increased after initial mixing at t=0 and then levelled off, finally decreasing - in agreement with Colic and co-workers (Colic & Morse, 1998). These workers suggest that, initially, the turbidity of the solution increases due to nucleation, then levels off due to sedimentation of the precipitate, finally decreasing as the larger aggregated particles sediment and the nucleation rate diminishes. They reported reaching a maximum turbidity value of 100-110 NTU in about 400 min - with no pre-treatment. Our own observations for the maximum peak ratio turbidity for non-treated samples was just above 50.6 NTU at 6 min and this was found to be reproducible. Therefore, based on our experiments, it would appear that the time of 400 min in Colic and Morse should have been 400 sec.

Absorbance: When 8 mM Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ were mixed without any pre-exposure treatment, absorbance gradually increased after initial mixing at t=0 and then levelled off, finally gradually decreasing in agreement with Higashitani et al (Higashitani et al., 1993), Figure 2(b). They suggest that the initial increase is due to formation of $CaCO_3$ particles and the later absorbance decrease is due to sedimentation of the formed particles. Curiously, they also report reaching a maximum absorbance value of 0.69 ~~in about~~in about 400 minutes with no treatment condition. Our own observation for peak absorption was ~~around~~ 0.81 at about ~~5 6~~8 min~~utes~~ and this was also found to be reproducible, Figure 2(b). This outcome is also consistent with our turbidity experiment. So here again we propose that the 400 mins in Higashitani et al should be seconds rather than minutes. We suggest that, since Colic and Morse extensively cites Higashitani et al, that the latter had incorrectly used min instead of sec and that this was inadvertently picked up by Colic and Morse.

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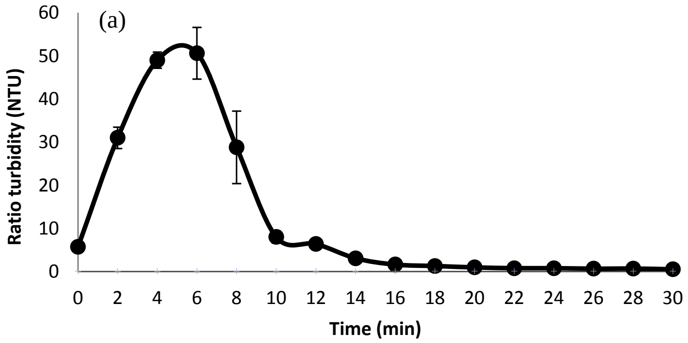

(b)

Figure 2 Establishment precipitation profiles from the mixing of ~~aueous~~aqueous 5 mM Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ over 30 min as a function (a) ratio turbidity (without PEMF involvement); ~~error bars are standard errors for five replicates~~ (b) UV absorbance at over 30 minutes as a function of absorbance at 350 nm (without PEMF involvement); error bars are standard errors in the mean for five ~~replicates~~replicate experiments.

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3.2 PEMF-~~-~~influenced $CaCO_3$ precipitation under static 'wet conditions'

Figure 3~~ ~~(a) & ~~and ~~(b); and (c) ~~and~~& (d), ~~(a) & (b) ~~demonstrates the turbidity curves and the absorbance curves, respectively, after both parent solutions were pre-exposed to PEMF under static 'wet conditions', for both the ~~devices ~~D and G devices in turn. From Figure 3~~ ~~(~~a~~b), when both 5 mM parent solutions were exposed to ~~static ~~D ~~static ~~PEMF treatment for 3 hrs, and mixed at room temperature (22 °C)~~, the~~, the maximum turbidity is significantly higher than its DWB control. However, w~~W~~hen the parent solutions were exposed to G ~~static ~~PEMF treatment for 3 hrs, the maximum turbidity is ~~slighlty higher~~not significantly different ~~than~~ the GWB control, Figure 3(a).~~.~~ ~~Notably, both DWB and GWB maximum turbidities are similar to those recorded at room temperature Figure 2 (a).~~ These results are reflected in the corresponding absorption curves. Thus in Figure 3(d) the absorbance over time when exposed to the D PEMF is significantly different to the control, showing a sharp drop-off in absorbance after the maximum. However, the absorbance over time when exposed to the G PEMF is not significantly different from the control, Figure 3(c). These results are consistent with the PEMF from the D Device having a significant influence on the $CaCO_3$ precipitation process and suggest that this PEMF enhances particulate aggregation.

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Scanning electron microscopy (SEM) was employed to gain more information on the particulate nature, and possible crystal morphology, of the above precipitates when the parent solutions were exposed to PEMF from the two devices.

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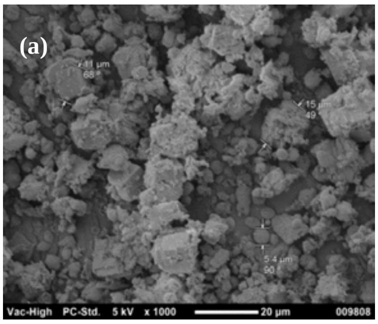

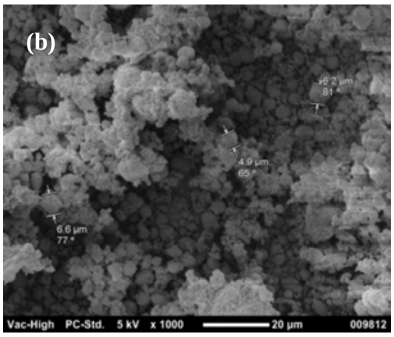

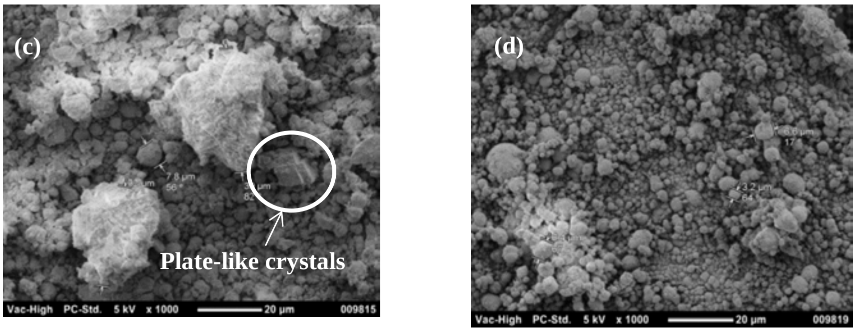

Figure 4: ~~dried~~ Dried precipitates from 1M parent solutions pre-exposed to **(a)** D PEMF **(b)** D control~~oven~~ **(c)** G PEMF **(d)** G control~~oven~~ for 30 min and mixed ~~under~~ at room temperature (22 °C).

An examination of Figure 4(a) reveals evidence for cuboidal crystal morphology in the precipitated material that is not observed in the control (b). This is consistent with the D PEMF pre-exposure having an effect on the crystal morphology and is also consistent with our observations that this PEMF has a significant effect on the precipitation profile, *vide supra*. ~~Dried precipitate of D PEMF (both parent solutions were pre-exposed for 30 min to device D under dry conditions, and then mixed at room temperature) results in significantly cuboidal crystals shape which~~shape, which ~~was not observed with other treatments. With D oven (Fig 4b) (where only the temperature factor was there without presence of PEMF), no such coexistence was observed; nor a lot of well-defined crystals, only aggregates of micro crystals. Some large aggregates were observed with G PEMF (Fig 4c) including a few plate-like crystals, but majority was microcrystals clumped together.~~ For G PEMF pre-exposure, there is some evidence of plate-like crystal formation, Figure 4(c) but, for the most part, no well-defined crystal morphology is apparent and the particulate matter appears similar to that in the control (d).

~~3.3 Morphology of CaCO$_3$ precipitated from pre exposed Na$_2$CO$_3$ and unexposed Ca(NO$_3$)$_2$.4H$_2$O~~

~~In the previous section both parent solutions were exposed to the PEMF of devices D & G and respective controls. Here only Na$_2$CO$_3$ was pre-seposed to PEMF under ‘dry conditions’ for 30 minutes and mixed with unexposed Ca(NO$_3$)$_2$.4H$_2$O under room temperature conditions. Figure 5 shows SEM images of the precipitates formed under treatments and respective controls.~~

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crystallization and deposition of $CaCO_3$. For example,

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~~However, magnetic treatment has been sometimes proven to be ineffective for retarding scale formation (Lipus et al., 2012). Available magnetic water treatment studies can be inconsistent as a result of a number of factors, including use of non-standardized methods, variations in water composition, differences in the course of the treatment (A. Szkatul/a, M. Bal/anda, & Kopec', 2002) and use of different pipe materials, which has been shown to affect the efficiency of magnetic water treatment (Gabrielli, Jaouhari, Maurin, & Keddam, 2001). Changes in crystal structure may reduce scaling by facilitating deposition rather than adherence but this will be a function of the system design as hydrodynamic shear effects will alter from system to system, as will the presence of biofilms and other causes of scale adhesion. Such an explanation might also explain why EMF is able to prevent scaling in some systems but not in all systems.~~

4. CONCLUSION AND RECOMMENDATIONS

In this study, two commercially available devices that, ostensibly, control scaling in water systems were tested under controlled laboratory conditions, for their ability to influence $CaCO_3$ precipitation. These experiments were conducted to establish a scientific basis for the above claims. Thus the effects of PEMF pre-exposure of ‘parent’ Na_2CO_3 and $Ca(NO_3)_2.4H_2O$ aqueous solutions were investigated with respect to the $CaCO_3$ precipitation profiles and precipitate characteristics upon subsequent mixing.~~carbonate precipitation in the presence and absence of commercial PEMF exposure was studied.~~ The precipitation profiles were tracked by replicate turbidity and UV absorbance experiments and the precipitate characteristics were examined using SEM. ~~There were two commercial PEMF devices were employed under wet or dry conditions as and water baths and ovens were employed as controls as explained in text. The morphology of the precipitated crystals were studied with SEM.~~

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One of the commercial devices, but not the other, was found to have a significant influence on the $CaCO_3$ precipitation profile and also was also found to have an effect on the precipitate characteristics and crystalline morphology. We have previously reported that these two devices have different waveforms (Piyadasa et al., 2016) and it is possible that this could be the reason why one device appears to have an effect but not the other. In this regard, we

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have recently demonstrated that these two devices have quite different effects on microbial

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~~Device D has influenced CaCO$_3$ precipitation when either parent solutions or both was pre exposed to its PEMF, as the crystal morphology was changed, perhaps due to and influence on solution characteristics under device D PEMF. Device D has also influenced precipitation process itself and also showed antimicrobial activity as in our previous publication (Piyadasa et al., 2016).~~

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