



Effect of magnetic water treatment on calcium carbonate precipitation: Influence of the pipe material

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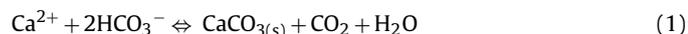
ABSTRACT

The main purpose of this work was to investigate the influence of the material of the pipe, through which scalant water (4 mM of CaCO₃) was magnetically treated, on the calcium carbonate precipitation process. Tested solutions were exposed to a magnetic field (MF) of 0.16 T for 15 min with different flow rates (0.54–0.94 L min⁻¹) and pH (6–7.5). Magnetic water treatment and pipe material (PTFE, Tygon, PVC, copper, and stainless steel) effects on CaCO₃ crystallization were assessed by means of a precipitation test based on the dissolved-CO₂ degasification method. It was shown that magnetic treatment (MT) affects calcium carbonate crystallization by increasing the total precipitate quantity and by favouring its formation in the bulk solution instead of its incrustation on the walls. This was observed for all used pipe materials but, it is strongly dependent on their physico-chemical properties. It was found that the homogeneous and total precipitation ratios were significantly influenced when MF was applied through non-conductive materials. It was also found that for the same pipe material, the surface roughness plays an important role on the effect of MT on CaCO₃ crystallization. In addition, it was shown that the pipe material strongly influences the nucleation process of CaCO₃ even in the absence of MF.

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1. Introduction

Calcium carbonate precipitation has been the focus of numerous investigations because of its importance in several industrial processes. For instance, it is involved in many industrial applications as pigment, brightener filler, adsorbent and in different biomedical uses [1–3]. However, calcium carbonate scale deposition induces important damage in the domestic, agricultural or industrial installations using natural waters (pipe blocking, membrane clogging, efficiency decay of heaters or heat exchangers, etc.). The main reaction involved in the precipitation of calcium carbonate is governed by the displacement of the calcoarbonic system equilibrium:



Various methods were used to prevent scaling, for example by water decarbonation using electrochemical processes, seeding or acid addition. Other way consists in blocking the nucleation and crystal growth by adding chemical inhibitors. However these chemicals are generally deleterious for human health and their use is forbidden in drinking water. Therefore, various physical methods were developed to avoid chemicals addition. In particular magnetic treatment (MT) methods of hard waters are currently used to pre-

vent mineral salts incrustation [4–6]. According to the review paper of Baker and Judd [7], in spite of a long experience, the efficiency of these treatments is still a controversial question and it is not possible to get a clear explanation of the phenomenon.

A number of mechanisms have been proposed to explain magnetic field (MF) effects on aqueous solutions and particle dispersions. Although it is almost certain that there are influential factors that are unrecognized at the present time, it can be summarized that the following types of effects most probably affect dispersion solubility and crystallization [8]: magnetically modified hydration of ions and solid surfaces [9–11] and Lorentz force effects on ions and dispersed particles (magnetohydrodynamic (MHD) phenomena) [12–16].

In addition of its effect on the physico-chemical characteristics of water and the crystallization process in solution, some researchers have shown that MF influences the hydrodynamics of fluid flow. In particular, according to Busch and Busch [17], depending on experimental conditions, the application of an orthogonal field to the flow of a conducting fluid may increase or decrease turbulence in the fluid, promoting aggregation or deaggregation of both ferromagnetic and diamagnetic colloids. This magnetohydrodynamic force on fluid flow can be promoted by the conductivity of the solution, the linear flow velocity of the fluid, and the flux density of the transverse field. Busch et al. [14] and Martemianov and Sviridov [15] have shown that the profile of the flow of a conducting solution in a pipe is perturbed when a MF is applied. At constant velocity, the flow

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rate is accelerated along the walls and therefore is reduced along the central axis of the pipe.

Other authors, in particular Gabrielli et al. [18], have claimed, by evaluating the scaling power of the treated water with an electrochemical scaling test, that the material of the pipe, in which the MF was applied, has an important influence on the calcium carbonate crystallization process: scaling rate and the precipitated calcium carbonate variety. It was deduced that, in addition to the water hardness and the flow velocity, the electrokinetic phenomena occurring in the vicinity of the tubing walls were also involved. However, it could be objected that the electrochemical precipitation test used by the authors induces exclusively a heterogeneous nucleation by a strong interfacial pH shift. This could be in contradiction with what happens in usual conditions where the homogeneous and heterogeneous precipitation are mainly due to the escape of carbonic gas from water, under the effect either of a change of pressure or an increase of temperature.

For this reason, in a recent work [19] a survey on the effect of MT on CaCO_3 crystallization has been undertaken with the same magnetic device as in [18], but to evaluate the scaling potentiality of treated waters a precipitation method (scaling test), based on the extraction of the dissolved carbonic gas (reaction (1)), was developed. The main advantage of this method consists in simulating the natural scaling phenomenon and makes possible the evaluation of the effect of the MT on calcium carbonate crystallization kinetics, solubility and adhesion on walls in contact with water. It was shown that the magnetic treatment decreases the solubility of CaCO_3 and promotes its precipitation in bulk solution instead of on the reactor walls. This effect depends on the solution pH, the flow rate and the treatment duration. It was advanced that MT affects the dehydration process of the ionic pair associations which are involved in the nucleation process of CaCO_3 precipitation. During this last investigation, calcocarbonic pure water was circulated at a constant flow rate in a permanent MF. All treatments were made before the scaling test using a Tygon tube in the working section of the magnetic device. However, as shown by Gabrielli et al. [18], the pipe material has significant effects on CaCO_3 crystallization.

In the present study, the same experimental method as in [19] was used in order to evaluate the effect of the pipe material, through which scalant water is exposed to the MF, on the potentiality of the calcium carbonate incrustation. Two material types were tested: conductive and insulating materials. The scaling test was performed by slowly degassing the dissolved- CO_2 gas in an independent cell made of polyamide.

2. Experimental

The used experimental procedure is detailed in [19], we recall briefly in this paper its principle. It consists in (i) preparing the working solution, (ii) magnetically treating this solution and (iii) proceeding on the calcium carbonate precipitation test.

The tested solutions were calcocarbonic pure waters prepared by dissolving 0.4 g L^{-1} of reagent grade CaCO_3 in deionised water by bubbling pure CO_2 . The resulting pH was about 5.7. The hardness of this water ($[\text{Ca}^{2+}] = 4 \text{ mM}$) can be considered as moderate by comparison with the very high values (8–12 mM) used by many authors [9,20–23].

The treatment step consists in circulating 0.5 L of solution in closed loop through a 7 mm in diameter pipe for 15 min in the presence or absence of MF. Five pipes made of different materials were tested: Tygon, PVC, Teflon (PTFE: polytetrafluoroethylene), copper and stainless steel (SS) (67% Fe, 22% Cr and 9% Ni). The working solution was circulated in each pipe at three fluid flow rates in laminar

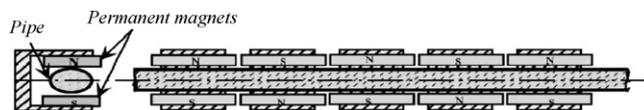


Fig. 1. Sketch of the set-up of the permanent magnets for the magnetic water treatment.

regime (0.54, 0.74 and 0.94 L min^{-1}). Three pH values (6, 7 and 7.5) of the working solution were tested. To apply the MF, a section of the pipe made of the tested material ($L = 20 \text{ cm}$) was inserted between the polar pieces of the used magnetic device (0.16T): a series of 5 pairs of permanent magnets with north and south faces facing each other are associated alternately. For each pair, the magnetic circuit was closed with a U shaped iron yoke [18] (Fig. 1).

Magnetically treated and untreated solutions were thereafter submitted to a precipitation test. It consists in slowly increasing the pH of the solution by bubbling at a constant flow a neutral gas (nitrogen) during 90 min in a reactor made of polyamide. By this way, the carbon dioxide was exhausted from the water and CaCO_3 was precipitated according to reaction (1). The pH of the solution and the calcium concentration, determined by EDTA complexometry titration, were continuously recorded. The induction time t_{ind} was determined from a discernable change in the slope of the pH and $[\text{Ca}^{2+}]$ vs. time experimental curves using the method of Dedieu et al. [24] (Fig. 2).

At the end of each precipitation test, the formed precipitate in the bulk of the solution was recovered by filtration on a $0.45 \mu\text{m}$ membrane. This CaCO_3 quantity (m_h) is considered to be precipitated by following a homogeneous nucleation process. By measuring the calcium ions remaining in the solution, the total precipitated calcium carbonate (m_t) was determined. The total precipitation ratio and the homogeneous precipitation ratio were determined as follows:

$$\text{total precipitation ratio } \tau_{TP} = \frac{m_t}{m_i} \times 100$$

$$\text{homogeneous precipitation ratio } \tau_{HP} = \frac{m_h}{m_i} \times 100$$

where m_i is the total calcium carbonate initially dissolved.

All experiments were replicated three times from which average values were calculated ($\sim 3\%$). After each experiment the tubing was thoroughly cleaned, first, by recirculating a low-concentration acid solution for about 0.5 h, after which deionised water was recirculated for 10 min.

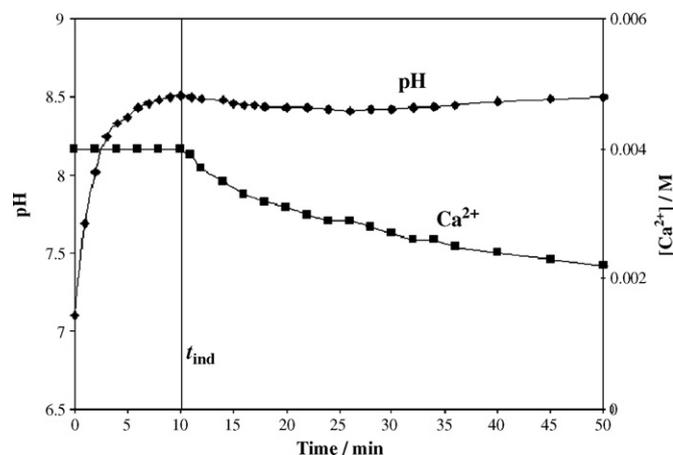


Fig. 2. Variations of the solution pH and the calcium ion concentration during a precipitation test ($[\text{Ca}^{2+}] = 4 \times 10^{-3} \text{ M}$, initial pH = 7) by CO_2 degassing.

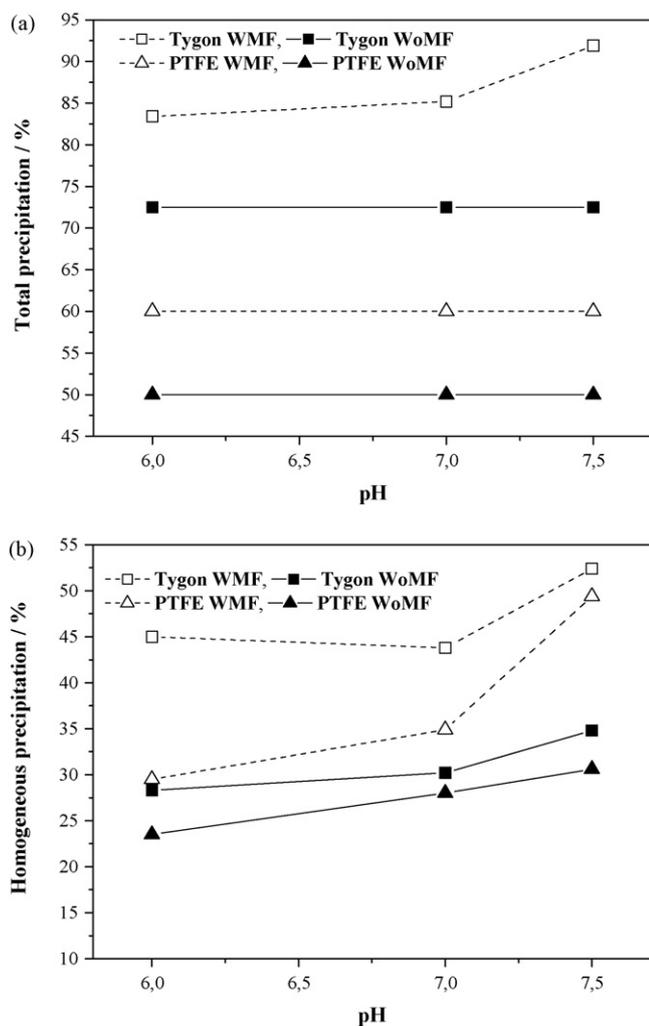


Fig. 3. Effect of pH on (a) total and (b) homogeneous precipitated CaCO_3 ratios with and without magnetic treatment ($[\text{Ca}^{2+}] = 4 \times 10^{-3} \text{ M}$; $T = 30^\circ \text{ C}$; pipes: Tygon and PTFE). WMF: with magnetic field, WoMF: without magnetic field.

3. Results

During the magnetic water treatment, calcium concentration, conductivity and pH were followed for each tested material. It was shown that conductivity and calcium concentration remained constant. The variation of pH in all cases did not exceed 0.15 units for a 15 min processing time. It means that the treatment did not induce any significant CO_2 departure due to water agitation, and that no CaCO_3 particles could be formed during the treatment time.

In order to evaluate the effect of MT on CaCO_3 crystallization, two parameters were followed: total (τ_{TP}) and homogeneous (τ_{HP}) calcium carbonation precipitated ratios. According to our previous study [19] where treatment was made on Tygon pipe, the most important effect of MT on τ_{TP} and τ_{HP} was registered for treatment of working solutions at pH = 7.5. This was confirmed in the present work with all tested pipe material. For example, in Fig. 3 are reported the effect of pH on the total and the homogeneous precipitation ratios for water treated using Tygon and PTFE pipes for a flow rate of 0.94 L min^{-1} . For this, in the following, we chose to present only results obtained for pH = 7.5.

3.1. Effect of the pipe material on the MT

Fig. 4 shows experimental measurements of τ_{TP} in the presence or absence of MF. The flow rate was varied from 0.54 to 0.94 L min^{-1} .

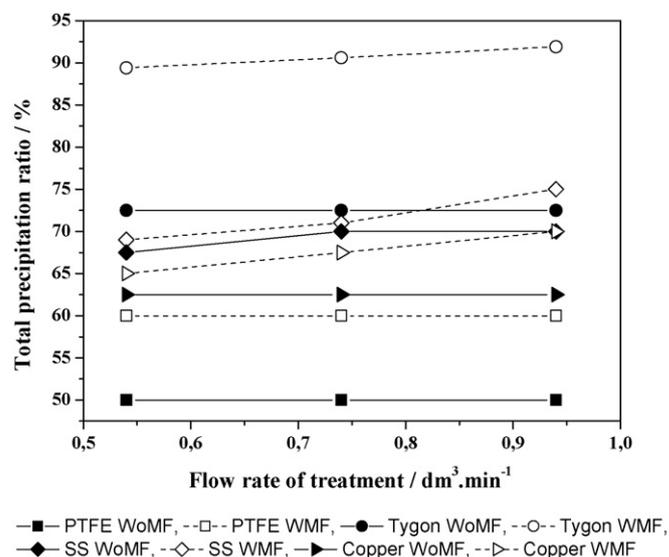


Fig. 4. Effect of all tested pipes material on total precipitation rate of calcium carbonate with and without magnetic treatment ($[\text{Ca}^{2+}] = 4 \times 10^{-3} \text{ M}$; $T = 30^\circ \text{ C}$).

This figure demonstrates clearly that the application of the MF increases significantly the total precipitation ratio for all used pipes material. This increase in τ_{TP} was more pronounced in the case of PTFE and Tygon where it increased by about 10 and 17%, respectively. This MF effect on τ_{TP} is less important for copper, SS and PVC (not presented in Fig. 3) pipes. However, the influence of the pipe material in the absence of MF is somewhat surprising. For example, for a flow rate of 0.54 L min^{-1} , τ_{TP} decreased from 73% for Tygon to 50% for PTFE pipe. To our knowledge this effect has never been mentioned until now. Moreover, in the absence of MF the total amount of precipitated CaCO_3 did not depend significantly on the flow rate, whatever the pipe material. Nevertheless, τ_{TP} increases with the flow rate (φ) when water was magnetically treated in conductive materials (SS and copper). This increase was about 5% when φ passed from 0.54 to 0.94 L min^{-1} .

On the other hand, the precipitated CaCO_3 was formed both by homogeneous and heterogeneous nucleation processes and hence was deposited either in bulk solution and/or on the walls of the reactor made of polyamide, respectively. Fig. 5 shows experimental measurements of the amount of calcium carbonate precipitated in the bulk of the solution τ_{HP} .

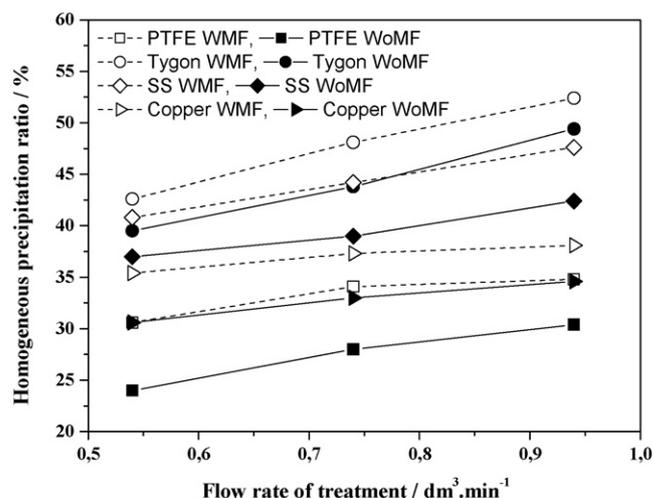


Fig. 5. Effect of all tested pipes material on CaCO_3 homogeneous precipitation rate with and without magnetic treatment ($[\text{Ca}^{2+}] = 4 \times 10^{-3} \text{ M}$; $T = 30^\circ \text{ C}$).

This figure proves that the application of MF increases the homogeneous precipitation ratio and, as for τ_{TP} , its effect on τ_{HP} is pipe material-dependent. Contrarily to τ_{TP} , the ratio of CaCO_3 formed in the bulk solution is flow rate-dependent. It increases with the flow rate. This means that the magnetic treatment reduces the heterogeneous nucleation and can, therefore, protect the wall against scaling. It can be also concluded that when water passes through the PTFE pipe in the magnetic device, the increase of τ_{HP} is the greatest ($\sim 7\%$).

3.2. Influence of the material conductivity on the MT

According to the results presented in Figs. 4 and 5, the MF effect on total and homogeneously precipitated CaCO_3 is less significant if water is treated by using conductive materials (copper or SS). Moreover, it was shown that the total precipitated calcium carbonate in magnetically treated solutions is flow rate-dependent. To confirm this relationship between material conductivity, precipitated CaCO_3 ratios and hydrodynamic conditions and to avoid any besides effects such as oxydo-reduction reactions which can occur on the walls [17], two pipes made of Teflon were tested: non-conductive white PTFE and an electroconductive anti-static black PTFE.

Fig. 6a shows that the treatment flow rate has an important influence on the total precipitation ratio when a conductive material was used (black PTFE): e.g., the determined τ_{TP} in magnetically treated

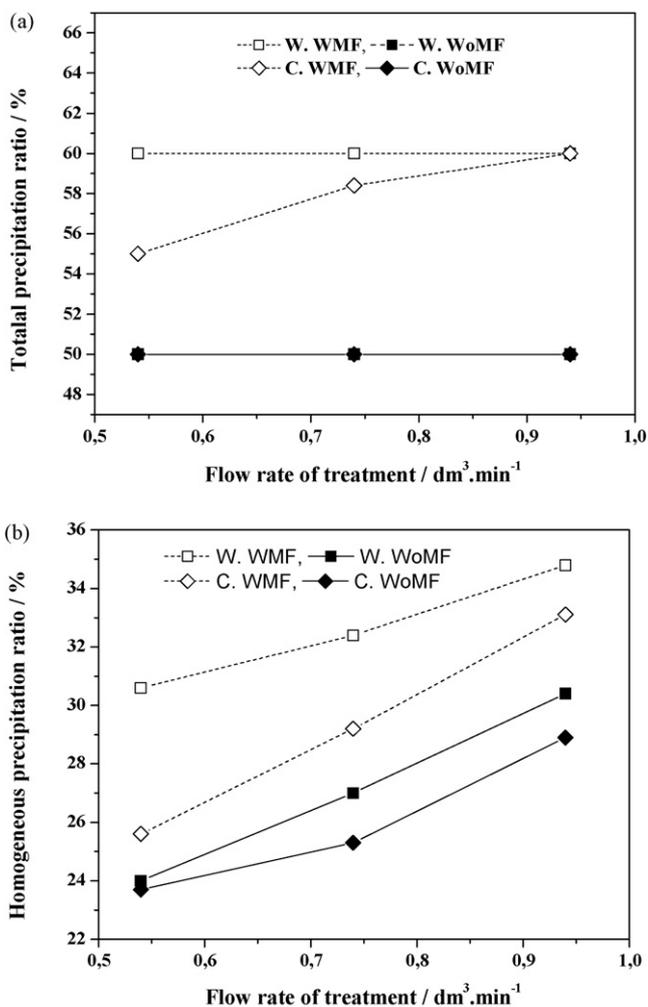


Fig. 6. Influence of material conductivity on (a) total and (b) homogeneous CaCO_3 precipitation ratios ($[\text{Ca}^{2+}] = 4 \times 10^{-3} \text{ M}$; $T = 30^\circ \text{C}$; pipe: PTFE), W: non-conductive PTFE, C: conductive PTFE.

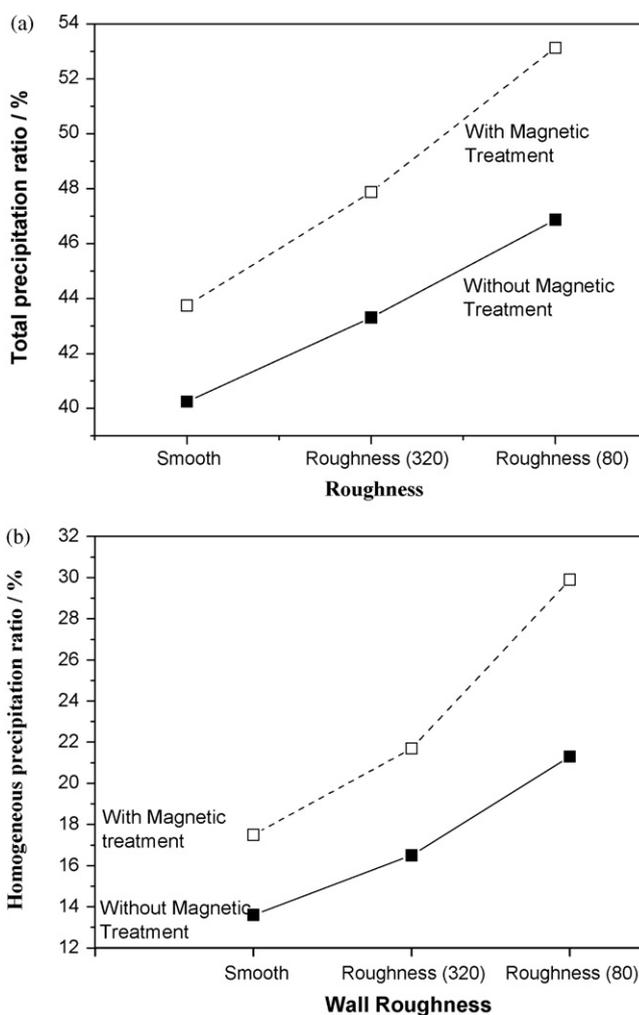


Fig. 7. Influence of the wall roughness on (a) total and (b) homogeneous CaCO_3 precipitation ratios ($[\text{Ca}^{2+}] = 4 \times 10^{-3} \text{ M}$; $T = 22^\circ \text{C}$; pipe: PVC).

solutions at the lowest flow rate are about 55 and 60% when conductive and non-conductive materials were, respectively, used. This physical parameter influences in the same way the effect of MT on CaCO_3 precipitated in the bulk solution as shown in Fig. 6b: e.g., for a flow rate of 0.54 L min^{-1} , τ_{HP} increased by 28 and 8% with respect to non treated water through non-conductive and conductive PTFE pipes, respectively. In agreement with results obtained with metallic pipes, this material property (conductivity) inhibits partially the effect of MF on τ_{TP} and τ_{HP} especially toward the lower flow rates.

3.3. Influence of the wall roughness

Since the flow rate has an important effect on CaCO_3 precipitation (Figs. 5 and 6b) and that the surface state of the tubing can influence the hydrodynamic parameters [25], we have tested the effect of a water treatment in PVC pipes with different roughness on the CaCO_3 crystallization process.

Three levels of roughness were tested: smooth pipe, polished pipes using abrasive papers of 320 and 80. The temperature, pH and flow rate of treatment were fixed at 22°C , 7 and 0.74 L min^{-1} , respectively. Fig. 7 shows the results of the precipitation tests.

In absence of MT, the quantities of the total and the homogeneous precipitates increase with roughness. The τ_{TP} increase is about 7% for the roughest surface with respect to the smoothest one. The application of MF encourages both total and homogeneous CaCO_3 precipitations. The raise of τ_{TP} and τ_{HP} are, respectively,

about 4 and 7% for the smooth pipe and reach 4 and 9% for the roughest one.

4. Discussion and conclusion

In this paper, it was proved that a magnetic water treatment affects calcium carbonate crystallization by increasing the total precipitate amount and by favouring its formation in the bulk solution instead of its incrustation on the walls. This was shown by means of a precipitation test based on the dissolved- CO_2 degasification method. Moreover, it was proved that the pipe materials and their physical properties (surface state and conductivity), through which water was circulated in the treatment step, has a noteworthy effect on CaCO_3 precipitation. Without MF, the lowest effect on total precipitated CaCO_3 amount was registered for the PTFE pipe and the highest effect was for the Tygon pipe. Metallic pipes present an intermediate behaviour. The MF application amplifies precipitation some either the tested material. These observations were also valid for the results obtained for homogeneous precipitates. The main difference was that the homogeneous precipitation ratio was flow rate-dependent in presence, or not, of MF whereas the total precipitation amount was flow rate-dependent only for conductive pipes and in presence of MF.

Without magnetic treatment it seems that the physical and chemical properties of the pipe material influence the homogeneous and total precipitation ratios. In Fig. 4, it was shown that the total precipitation ratio is pipe material-dependent. The highest τ_{TP} was registered for the Tygon pipe and the lowest for the PTFE one. This can be attributed to the alteration of the water properties after circulate through tubing as claimed by several researchers: Junk et al. [26] found that PE (polyethylene), PP (polypropylene), black latex, and six formulations of PVC (among them Tygon) tubing leached constituents even though they had been prerinsed. Many of the contaminants that leached from the PVC tubing, due to an erosion mechanism occurring at the polymer/water interface, were identified as plasticizers and other additives where the amount was related to the linear velocity of the water flowing through the tubing. They concluded that Tygon tubing contained a nearly inexhaustible supply of contaminants because of its high concentration of plasticizers with respect to the other tested tubing. This was in agreement with results obtained by other researchers [27–30]. By comparing the leachates from a variety of pipes, they found that leachate concentrations were the highest for Tygon and PTFE did not leach any detectable contaminants. Recently, Amiri and Dadkhah [31] have clearly showed that the surface tension of water passed through a Tygon pipe decreased from 72.54 to $\sim 52 \text{ mN m}^{-1}$. This was attributed to both soluble and insoluble impurities diffused in sample waters during its passing through the plastic pipe.

From these observations it can be concluded that the effect of the pipe material on the total precipitation ratios might be related to the presence of contaminants in water after passing through the tubing. In fact, these contaminants can play the role of seeds which promote the precipitation of CaCO_3 in the bulk of the solution. This can explain the lower precipitation ratio observed for PTFE which did not liberate any substance (Fig. 4) and the higher homogeneous precipitation ratio for Tygon in Fig. 5.

On the other hand, it was shown in Fig. 7 that the total and homogeneous precipitation ratios increase simultaneously by about 7% for the roughest surface with respect to the smoothest one. This shows that wall roughness influences only the quantity of calcium carbonate formed in the bulk of the solution. Indeed, internal roughness generated turbulence areas in the vicinity of the walls and than can create local eddy currents [32,33]. This can accentuate the release of impurities by erosion and therefore the precipitation of calcium carbonate in the bulk of the solution instead of the reactor walls.

With magnetic treatment, a clear increase of the total and the homogeneous precipitation ratios toward higher values was registered for all tested pipes (Figs. 4 and 5). For results related to PTFE pipe which does not release any impurity in the solution, the influence of MF on the two ratios was clear and could be intervene on the ions and ionic associations present in the solution. Indeed, the experimental investigations on the calcocarbonic system $\text{CaCO}_3\text{--H}_2\text{O--CO}_2$ equilibrium showed the occurrence of a large supersaturated domain with respect to calcite (most stable variety of calcium carbonate) called the metastable domain, where the precipitation does not occur. It has been advanced [34–36] that the breakdown of the metastable state of a supersaturated solution is initiated by the nucleation of CaCO_3 hydrated forms (amorphous, monohydrate or hexahydrate) when the corresponding solubility product (or equilibrium pH_{eq}) is exceeded. In this investigation, the hard water was treated at pH values of 6, 7 and 7.5 which were lower than the pH_{eq} (7.83) of the monohydrated calcium carbonate ($\text{CaCO}_3 \cdot \text{H}_2\text{O}$), most insoluble hydrated form. Therefore, it can be assumed that, during the treatment step, the solutions do not contain any CaCO_3 nucleus or solid particles. This can be supported by experimental values of pH_{ind} ($\text{pH}_{\text{ind}} > 8.5$) recorded at t_{ind} corresponding to the formation of the first calcium carbonate nucleus. For example, Fig. 2 shows that precipitation occurs at $\text{pH}_{\text{ind}} = 8.55$ for a solution treated at $\text{pH} = 7$ in a Tygon pipe.

Nevertheless, since 1941, it is known that hard waters contain various ionic associations such as CaCO_3 ion pair [37,38]. Therefore, MT can intervene on these ionic pairs, ionic complexes (CaHCO_3^+) and hydrated ions Ca^{2+} , HCO_3^- and CO_3^{2-} . According to Lungader Madsen [10,39], MF induces faster proton transfer from hydrogen carbonate to water, due to proton spin inversion in the field of diamagnetic salts. The increased formation of CO_3^{2-} ions would explain the beneficial effect of MF on the amount of precipitate (Figs. 3, 4, 6a and 7a). Another explanation proposed by Higashitani et al. [20] is related to the specific influence of MF on the hydration of CO_3^{2-} ions which could directly modify the polymorph phase equilibrium during precipitation. The same phenomenon could also affect the dehydration process of the ionic pair associations and the hydrated calcium carbonate forms which act as precursors for the formation of the anhydrous forms which constitute the calcium carbonate precipitate [19].

On the other hand, MF application has a magnetohydrodynamic (MHD) effect. Lorenz forces exerted on charged species induce local convection movements in the liquid which could contribute to accelerate associations between ions or colloidal particles [14,15]. Moreover, MHD phenomena induce eddy currents which flattened the fluid velocity profile in the tube. This effect would result in a larger velocity gradient, along the walls. This can accentuate the release of impurities by erosion. In addition, the streaming potential along the walls should increase with the magnetic field. This phenomenon, by changing the surface charge, could throw out of balance the calcocarbonic equilibrium in the vicinity of the tubing walls [23]. However, hydrodynamic forces as well as the electrokinetic phenomena along the walls, which could favour the process of ionic pair micellization, are pipe material-dependent. This can explain the net difference on the effect of MT on homogeneous and total precipitation ratios with pipes material (Figs. 4 and 5).

To conclude, the magnetic water treatment decreases scaling by encouraging the homogeneous nucleation process of calcium carbonate. The choice of the pipe material seems a determinant parameter for the success of this MT. Non-conductive materials can be considered as the most efficient in this application. As PTFE gives the lower total precipitation (Fig. 4) and the higher homogeneous precipitation ratio (Fig. 5), it seems the most appropriate comparing to all others tested materials.

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Appendix A. Nomenclature

MHD	magnetohydrodynamic
MF	magnetic field
MT	magnetic treatment
t_{ind}	the induction time
m_i	mass of CaCO ₃ initially dissolved
m_t	mass of the total precipitate
m_h	mass of homogeneous CaCO ₃ precipitate
φ	flow rate of treatment
τ_{TP}	the total precipitation ratio
τ_{HP}	the homogeneous precipitation ratio

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