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Simplified removal of Fe(II) and Mn(II) from groundwater via oxidative coprecipitation with portlandite and subsequent neutralization with CO₂ bubbling

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Abstract

Groundwater enriched with Fe(II) and Mn(II) at concentrations exceeding 0.3 mg/L and 0.1 mg/L, respectively, may pose serious health risks upon consumption and therefore requires treatment. This study developed a simple, rapid, and effective method to remove Fe(II) and Mn(II) from groundwater (with removal efficiencies greater than 99%) by accelerating their precipitation and oxidation (particularly for iron) under alkaline conditions (at pH of approximately 12.2), constrained with calcium hydroxide (Ca(OH)₂). Alkaline water is then neutralized by CO₂ bubbling for 3–5 min. This method requires short treatment time (shorter than 1 h), a low Ca(OH)₂ dosage (0.5–3.0 g/L), and ambient temperature, and can be easily scaled up to treat large volumes of water because only existing conventional technology is needed (e.g., tanks with agitators at atmospheric pressure, pumps, and injectors). In addition, the advantage of this method is that other metallic pollutants can also be removed during the treatment, and the resulting red solid residue, mainly composed of calcite and iron oxyhydroxide (the FeOOH type) nanoparticles, can be reused as pigments, sorbents, or mineral filler powders for industrial applications. This integrated method can be successfully used to remove toxic dissolved ions from water while generating solid residues with industrial uses.

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Keywords: Groundwater; Removal; Toxic ions; Water treatment; Coprecipitation; Mineral nanoparticles

1. Introduction

Iron and manganese are two metals present as oxides and oxyhydroxide minerals in most tropical ferruginous soils. The dissolution of these minerals by recharging waters accounts for the high concentrations of iron and manganese in groundwater (Lanciné et al., 2005). At concentrations above 0.3 mg/L, iron stains laundry and plumbing fixtures. Below 0.3 mg/L, a metallic taste is generally not detectable, although turbidity and deposits may still occur. Manganese at concentrations

exceeding 0.1 mg/L in water can impart an undesirable taste to beverages and stain both sanitary appliances and fabrics. Similar to iron, the presence of manganese in drinking water can lead to the accumulation of deposits in distribution systems (WHO, 2017). Iron also promotes the development of iron bacteria, which derive energy from the oxidation of ferrous iron to ferric iron, thereby depositing a slimy layer on pipes (Benefield et al., 1982; Ahmad et al., 2005).

Numerous methods have been reported in the literature for the reduction or removal of Fe(II) and Mn(II) ions from drinking water, including oxidation by atmospheric oxygen (Stumm and Lee, 1961), oxidation by strong oxidants (Knocke et al., 1991; González-Davila et al., 2005), biological treatment (Godard, 1987; Mouchet, 1992), electrocoagulation (Ghosh et al., 2008; Vasudevan et al., 2009;

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Doggaz et al., 2018), ion exchange (Vaaramaa and Lehto, 2003; Keller, 2004), membrane technologies (Fakhfekh, 2017), and adsorption on various materials (Wang et al., 2013; Mettler et al., 2009; McBride, 1979). Each of these techniques has its own advantages and disadvantages. However, most approaches are not applicable in certain developing countries, particularly in rural areas, due to their high cost, implementation complexity, and significant energy consumption.

This study is a continuation of three previous works conducted by Montes-Hernandez et al. (2023, 2009) and Hamdouni et al. (2016). These studies focused on the removal of dissolved pollutants, including ferrous ions, from synthetic waters via carbonation of portlandite under CO₂ pressure (greater than atmospheric pressure). Within this framework, this study proposed a simpler method to simultaneously remove iron(II) and manganese(II) from enriched synthetic waters using portlandite and a simple CO₂ bubbling process. To this end, batch experiments were carried out at the liter scale and consisted of two successive steps: portlandite–solution interaction under stirring for 1 h in contact with atmospheric air, followed by neutralization of the solution or slurries by CO₂ bubbling for 3 min (i.e., rapid carbonation). Residual Fe and Mn concentrations were measured by inductively coupled plasma atomic emission spectrometry (ICP-AES). In addition, the solids and/or precipitates were characterized by X-ray diffraction (XRD), scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM-EDX), and Fourier-transform infrared spectroscopy (FTIR) to better understand the trapping and/or sequestration mechanisms of Fe and Mn.

This technique could be applied in Niger (a low-income country), particularly for water supply in villages and pastoral areas, to meet the essential needs of populations and livestock. Furthermore, it would help reduce the costs associated with replacing pipelines encrusted with ferric oxide deposits and improve access to safe drinking water at lower cost for rural communities.

2. Materials and methods

2.1. Preparation of solutions

This study was carried out using two types of solutions. The first category consisted of solutions prepared from ultrapure water with an electrical resistivity of 18.2 MΩ·cm. The second category was prepared from natural water (Grenoble water) enriched to better replicate the actual conditions of an aquifer. Ultrapure water was used instead of natural water to limit the risk of premature oxidation of Fe(II) and Mn(II).

Experiments were conducted with these two categories of enriched solutions: the first containing only Fe(II) at a concentration of 35 mg/L, prepared from FeSO₄·7H₂O (ferrous sulfate heptahydrate; Sigma–Aldrich); and the second

containing both Fe(II) and Mn(II) at respective concentrations of 35 mg/L and 2.5 mg/L.

2.2. Test procedure

1 L of the prepared solution was introduced into a 2-L beaker and mixed with a known amount of Fe(II) and/or Mn(II) salt. Then, 0.5 g of commercial portlandite (Ca(OH)₂; with 96% purity, containing 3% CaCO₃ and 1% other impurities; Sigma–Aldrich) was added. The suspension was stirred at 500 r/min for 1 h.

To monitor the evolution of Fe (or Mn) and calcium concentrations, samples were collected at regular intervals using syringes and filtered through a membrane with a pore size of 0.22 μm. The filtrates were acidified by adding one drop of the 3-mol/L nitric acid solution to prevent any subsequent ion precipitation prior to ICP-AES analysis. pH and redox potential were also monitored during the experiment.

After the portlandite–solution interaction, under stirring and in contact with atmospheric air, the alkaline suspension was subjected to CO₂ bubbling (with 99.995% purity; supplied by Linde Gas SA) for 3–5 min. A sample was also collected at this stage for ICP-AES analysis. At the end of the bubbling process, the solution–mineral suspension was left to settle for approximately 12–24 h.

On the following day, after decantation in the beaker, the solid phase was recovered and dried in an oven at 60°C for 24 h. The obtained solids were then stored in vials for subsequent characterization. All experiments were carried out at room temperature (approximately 20°C) and in the presence of atmospheric air.

The removal efficiency (*E*) of iron in the solution at the portlandite interface was determined using Eq. (1):

$$E = \frac{C_0 - C_e}{C_0} \times 100\% \quad (1)$$

where *C*₀ is the initial iron concentration, and *C*_e is the equilibrium iron concentration.

2.3. Physicochemical analysis of filtered solutions

The filtered solutions were analyzed by ICP-AES using a Varian 720 ES spectrometer. Calibration was performed with standard solutions prepared by diluting certified commercial single-element stock solutions in 2% nitric acid. The standard solutions were stored in dedicated tubes to ensure their stability. pH and redox potential were measured using a multi-function device (Mettler Toledo).

2.4. Characterization of solids

2.4.1. XRD analyses

Powder XRD analyses were performed using a Bruker D8 diffractometer equipped with a vertical goniometer and a rotating sample holder. Diffractograms were recorded using Cu Kα₁ (with a wavelength of 1.540 6 Å) and Kα₂ (with a wavelength of

1.544 4 Å) radiation over a 2θ (where θ is the diffraction angle) range of 5° – 90° , with a step size of 0.026° and a counting time of 5 s per step. Crystalline phases were identified using the DiffracPlus Eva software and its associated database.

2.4.2. FESEM observations

The samples were dispersed in absolute ethanol by ultrasonic treatment for 6–12 min. 1–2 drops of the resulting suspension were then deposited on an aluminum stub and coated with a thin layer of platinum for field emission scanning electron microscopy (FESEM) observations. The morphology of the crystal faces was examined using a Zeiss Ultra 55 scanning electron microscope, which provided a maximum spatial resolution of approximately 1 nm at 15 kV. The semi-quantitative elemental compositions of the precipitates were determined by energy-dispersive X-ray spectroscopy (EDX).

2.4.3. FTIR measurements

Selected powdered samples were analyzed by transmission infrared spectroscopy using a Bruker Hyperion 3000 infrared microscope. The infrared beam was focused through a 15-time objective, with an analysis area of approximately $50\ \mu\text{m} \times 50\ \mu\text{m}$. Spectra were recorded in transmission mode between 4000 cm^{-1} and 500 cm^{-1} , at a spectral resolution of 4 cm^{-1} and an acquisition time of 100 s. For certain measurements, fine aggregates rich in calcite were manually compressed prior to analysis.

3. Results and discussion

3.1. Iron removal efficiency as a function of portlandite dosage

Fig. 1 illustrates the Fe(II) removal efficiency as a function of portlandite dosage in both the synthetic solution and enriched natural water. In the single-ion system (Fe(II) alone in the solution), whether in the synthetic solution or enriched natural water, the Fe(II) removal efficiency was close to 100%, regardless of the portlandite dosage (0.5 – 3.0 g/L). The high alkalinity generated by hydroxide ions released from portlandite accelerated the oxidation of Fe(II) to Fe(III) at the portlandite–solution interfaces (Hamdouni et al., 2016).

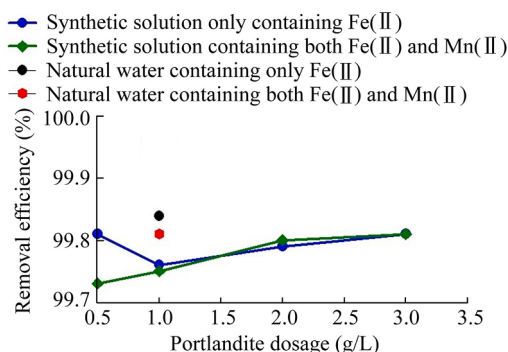


Fig. 1. Fe(II) removal efficiency as a function of portlandite dosage.

Portlandite particles thus appeared to efficiently catalyze this process, with complete Fe(II) oxidation observed in less than 1 h (Fig. 2). Furthermore, neutralization of the solution by bubbling pure CO_2 promoted the formation of calcite, accompanied by the coprecipitation of Fe(III) as FeOOH on the surface of the newly formed calcite (Fig. 3). Colloidal iron oxyhydroxide particles remain fixed on the calcite surfaces, facilitating their separation from water.

In the competitive system (Fe(II) in the presence of Mn(II)), the Fe(II) removal efficiency also remained close to 100% (Fig. 1). Therefore, the presence of Mn(II) did not affect the catalytic properties of portlandite with respect to Fe(II) oxidation to Fe(III). After CO_2 neutralization, the oxidized Fe persisted as FeOOH, still closely associated with the calcite surfaces, as in the single-ion systems.

Finally, the Fe(II) removal efficiency observed with the enriched natural Grenoble water was slightly higher than that obtained with the synthetic solution. The slight difference observed between the natural water and the synthetic solution can be attributed to the more complex composition of the natural water (dissolved oxygen, ionic strength, and a diversity of dissolved species), which may promote iron oxidation and precipitation processes from the earliest stages, even before the addition of portlandite.

3.2. Manganese removal efficiency as a function of portlandite dosage

The Mn(II) removal efficiency in the presence of Fe(II) at the solution–portlandite interfaces was evaluated across the two experimental steps. Fig. 4 shows that at the end of the solution–portlandite interaction phase under stirring, the Mn(II) removal efficiency exceeded 99% in the enriched synthetic solution and approximately 97% in the doped natural Grenoble water. After the neutralization phase, the removal efficiencies were 99% for the enriched synthetic solution and 96% for the doped natural water.

Notably, neutralization by CO_2 bubbling induced a slight re-dissolution of Mn. However, this re-dissolution is negligible relative to the limit set by the World Health Organization (WHO) for drinking water (0.1 mg/L). Indeed, even after neutralization, the residual Mn concentration remained well below this limit.

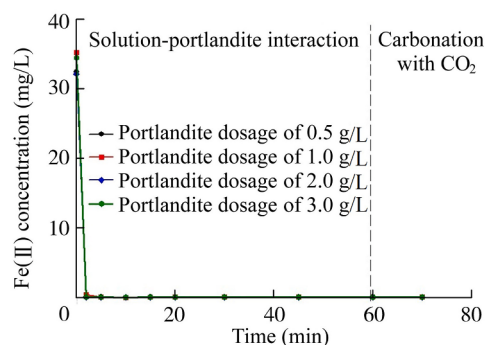


Fig. 2. Evolution of Fe(II) concentration with different portlandite dosages.

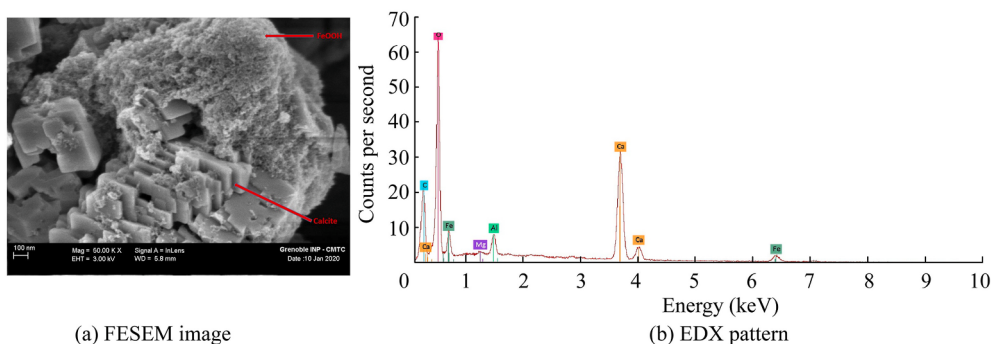


Fig. 3. FESEM and EDX analysis of solids obtained during solution–portlandite interaction after rapid CO₂ bubbling in Fe(II) and/or Mn(II) enriched solutions.

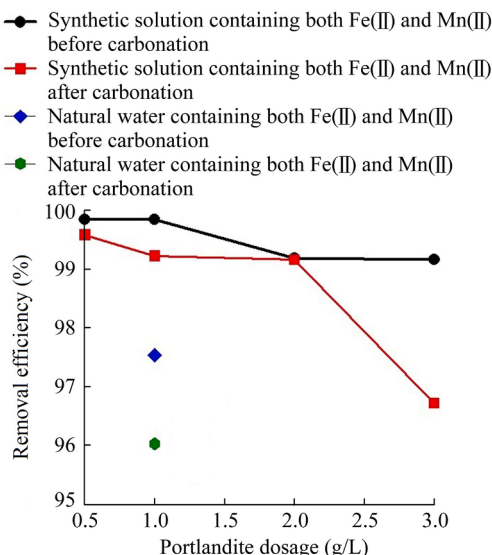


Fig. 4. Mn(II) removal efficiency as a function of portlandite dosage before and after carbonation.

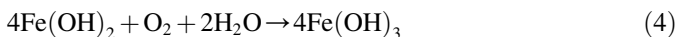
Overall, these results suggest that manganese predominantly precipitated as Mn(OH)₂ under the experimental conditions. However, CO₂ neutralization is associated with partial re-dissolution of manganese, an effect that became more pronounced at higher portlandite dosages (3.0 g/L), where increased release of manganese into the solution was observed.

3.3. Mechanisms of Fe(II) and Mn(II) removal at solution–portlandite interfaces

The elimination of Fe(II) by the portlandite particles occurs in various steps. The first step is the dissolution of portlandite:



The second step is the formation of the intermediate or unstable Fe(OH)₂, followed by the iron oxidation process and the formation of ferric oxyhydroxide:



The neutralization of the solution by CO₂ bubbling allows the precipitation of calcite and a pH change from 12.2 to approximately 6–7 depending on the bubbling time. Then the CO₂ gas is dissolved in the alkaline solution, forming carbonate ions:



The last step is the precipitation of calcite:



The mechanism of Mn(II) elimination is summarized in two main steps. The first step, common to both pollutants, is the dissolution of portlandite represented by Eq. (2). This is followed by the precipitation of manganese(II) hydroxide:



3.4. Iron behavior at solution–portlandite interfaces as a function of time

Fig. 2 shows the evolution of the Fe(II) concentration over time during the two-step removal process at the solution–portlandite interfaces. Initially, during the solution–portlandite interaction under stirring, the initial Fe(II) concentration (35 mg/L) declined to nearly zero within less than 5 min and remained constant until the end of this step (60 min). This result indicated that Fe(II) was rapidly oxidized to Fe(III) under the catalytic effect of portlandite, as high alkalinity promoted the oxidation–precipitation mechanism. After the first 5 min, stirring was no longer necessary, as its role was primarily to ensure good system homogenization. Rapid carbonation (shorter than 3 min) by bubbling pure CO₂ then neutralized the alkaline solution (at pH of approximately 12.45) to pH values between 6.57 and 7.13, which fell within the potability range recommended by WHO.

3.5. Calcium behavior at solution–portlandite interfaces

The addition of portlandite to remove Fe(II) and/or Mn(II) ions resulted in a significant increase in the Ca²⁺ concentration in the water, thereby increasing its calcium hardness. Therefore, monitoring this concentration is essential. Upon introducing

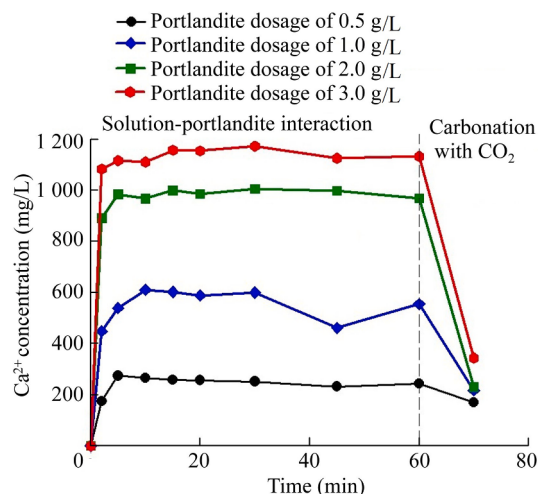


Fig. 5. Evolution of Ca^{2+} concentration during Fe(II) removal across different portlandite dosages.

portlandite into the working solution under continuous stirring (at 500 r/min), the Ca^{2+} concentration (Fig. 5) increased rapidly and then gradually reached a maximum and stabilized until the end of the first experimental step (interaction between the solution and portlandite). This concentration was directly proportional to the portlandite dosage and approached a limit of approximately 950 mg/L, corresponding to the solubility of portlandite at room temperature.

During the second step, neutralization via bubbling with pure CO_2 caused a sharp decrease in the Ca^{2+} concentration. This decrease is explained by the precipitation of calcite induced by CO_2 addition, following the reaction mechanisms described previously. Notably, in the competitive system, the presence of Mn(II) did not affect the variation in the Ca^{2+} concentration.

3.6. Evolution of pH and redox potential at solution–portlandite interfaces

As shown in Fig. 6(a), the addition of portlandite caused an immediate increase in pH to a stable value of approximately 12.45, regardless of the portlandite dosage (0.5–3.0 g/L). This high alkalinity, resulting from the release of OH^- ions during the

dissolution of portlandite, can simultaneously promote the oxidation–precipitation of Fe(II) to Fe(III), as well as the precipitation of Mn(II). However, at such a high pH value, the treated water is not suitable for consumption. Neutralization by CO_2 bubbling (for approximately 3 min) rapidly lowered the pH value to 6.6–7.1, a range within the WHO-recommended drinking water limit, through the establishment of the carbonate equilibrium.

As shown in Fig. 6(b), the evolution of pH directly influenced the redox conditions of the medium. Specifically, the increase in pH modifies the equilibrium of chemical species and promotes oxidation–reduction reactions involving dissolved oxygen. Monitoring the redox potential in the reaction medium could provide an indication of dissolved oxygen levels during the two phases of Fe(II) and/or Mn(II) removal following the addition of portlandite.

During the first phase, corresponding to the solution–portlandite interaction, the redox potential rapidly decreased to negative values immediately after the addition of portlandite, indicating accelerated consumption of dissolved oxygen in this highly alkaline medium. Dissolved oxygen participated in the oxidation of Fe(II) to Fe(III) and was entirely consumed by ferrous ions, as illustrated by the Fe(II) removal mechanism. It can therefore be inferred that Mn(II) is poorly oxidizable by dissolved oxygen under these conditions (Charles, 2008).

During the second phase, corresponding to neutralization by CO_2 bubbling, the decrease in pH was accompanied by a significant increase in the redox potential toward positive values, close to those of drinking water. This evolution reflects the complete oxidation of Fe(II) and, following neutralization, the establishment of more stable redox conditions promote intense gas exchange with atmospheric air and the re-equilibration of dissolved oxygen.

3.7. Characterization of precipitated solids

XRD analyses, combined with the determination of mineralogical composition from available databases, revealed the presence of calcite particles (Fig. 7).

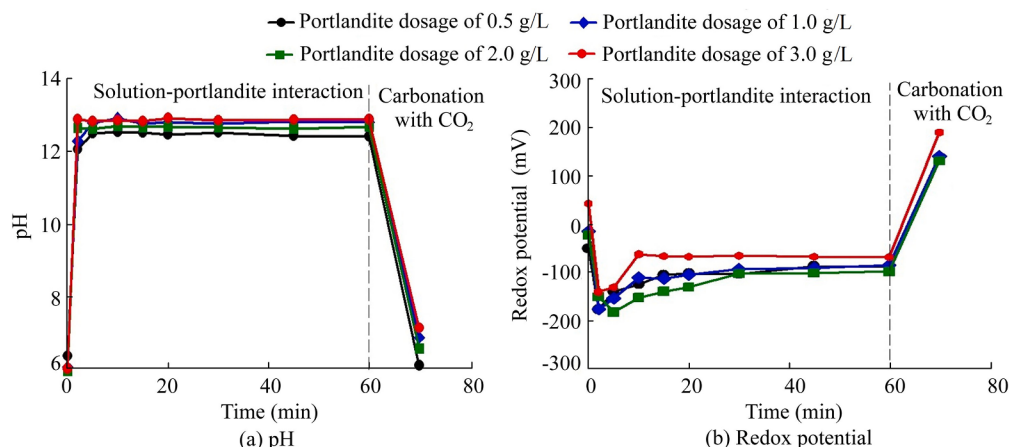


Fig. 6. Evolution of pH and redox potential at solution–portlandite interfaces.

As shown in Fig. 3, SEM-EDX analysis revealed very fine iron precipitate nanoparticles adhering to the calcite crystal faces. This formation manifested macroscopically as a homogeneous pink-red coloration of the solids. However, these nanoparticles were not detected by XRD, likely due to their extremely small size and/or low degree of crystallinity.

The transmission infrared spectra (Fig. 8) exhibited a broad band between $3\,000\text{ cm}^{-1}$ and $4\,000\text{ cm}^{-1}$, associated with the $\cdot\text{OH}$ group bound to the structure, as well as an extended band between $1\,050\text{ cm}^{-1}$ and $1\,200\text{ cm}^{-1}$, which may correspond to an iron oxyhydroxide of the lepidocrocite type (Cakić et al., 2002).

4. Conclusions

This study demonstrated that the use of portlandite is a highly efficient method for removing iron(II) and manganese(II). At low dosages of portlandite ($0.5\text{--}3.0\text{ g/L}$), removal efficiencies were high (close to 100%) within a relatively short treatment time (shorter than 1 h). This approach offers a twofold advantage. On the one hand, the solids obtained after treatment, which are rich in calcite and iron oxyhydroxide, can be used in other industrial applications or be reused for the capture of metallic and metalloid pollutants in solutions. On the other hand, the use of portlandite before neutralization can play a softening role by

reducing the hardness following the precipitation of $\text{Mg}(\text{OH})_2$ and after neutralization by eliminating the calcium hardness in the form of CaCO_3 . Future work will focus on evaluating the process at a larger scale and under real water treatment conditions.

Declaration of generative AI in scientific writing

No generative AI tools or AI-assisted technologies were used during the preparation of this work. The authors take full responsibility for the content of the published article.

Declaration of competing interest

The authors declare no conflicts of interest.

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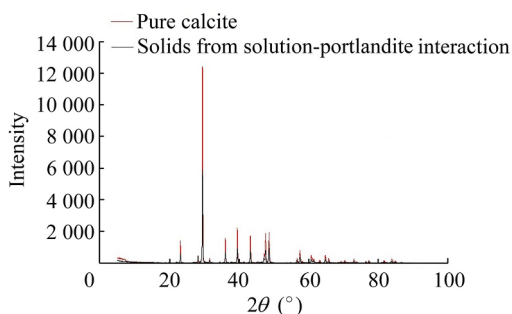


Fig. 7. XRD patterns of pure calcite and solids from solution–portlandite interaction after rapid bubbling with CO_2 during Fe(II) and Mn(II) removal on portlandite in batch reactor (with θ denoting diffraction angle).

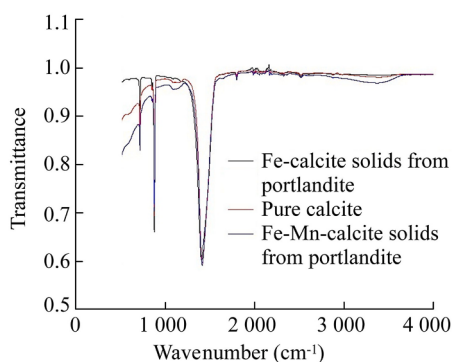


Fig. 8. Transmission infrared spectra of solids obtained during solution–portlandite interaction after rapid CO_2 bubbling.

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