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TECHNICAL COMMUNICATION

Enhancement of Sludge Sedimentation Properties in a Concentrated Acid Mine Drainage Using Nano- and Micro-magnetite Particles

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Abstract

Conventional treatment of AMD involves neutralization with consequent precipitation of metals as hydroxides. In AMD with a high concentration of metals, the settling rate of the sludge/water interface is low. We investigated the use of nano- and micro-magnetite particles to assist the settling and thickening of floc particles. The magnetite was produced from ferrous sulphate crystals (melanterite, $Fe_2SO_4 \cdot 7H_2O$) obtained by leaching pyrite from a coal mine. AMD was obtained from the treatment plant at the same mine and the water was neutralized with $Ca(OH)_2$ at pH 8.7 ± 0.1. Laboratory studies were conducted in 1 L test tubes with and without the addition of magnetite particles and a flocculant. Sedimentation curves (interface settling) were generated to evaluate the rate of sedimentation. For the studied effluent, the best option was 4 g L^{-1} of magnetite particles and 5 mg L^{-1} of high molecular weight anionic polyacrylamide. The magnetite particles were recovered magnetically from the sludge with $\approx$ 90% efficiency. Thus, the combined use of magnetite and a flocculant increased the sludge settling rate and, consequently, reduced the area needed for settling basins.

Keywords AMD active treatment · Anionic polyacrylamide · Magnetic iron oxide · Sludge settling

Introduction

Acid mine drainage (AMD) is actively treated by the addition of an alkalinity source, which increases the pH and causes most of the dissolved metals to precipitate as hydroxides. Additionally, in the presence of Ca^{2+} and SO_4^{2-} ions, insoluble gypsum complexes ($CaSO_4 \cdot xH_2O$) form. Generally, the reagents used to adjust the pH to neutral to alkaline values—between pH 7 and 9—are $Ca(OH)_2$, CaO, NaOH, Na_2CO_3, and NH_3 (Akcil and Koldas 2006; Johnson and Hallberg 2005; Kefeni et al. 2017; Matlock et al. 2002; Pereira et al. 2020; Skousen et al. 2019). Afterwards, sedimentation of the formed sludge is promoted. This step is usually accelerated by the addition of high-molecular-weight polymers (Skousen 2014). However, often the sedimentation rate of metallic precipitates and the volumetric ratio between clarified water and sludge are unfavourable, which impedes the performance of the treatment process.

The addition of nano- or micro-particles can increase the sedimentation rate and sludge density (Chen et al. 2019). Magnetic particles are often used in similar circumstances, due to their high density and the ease of separating the material by magnetic attraction (Luo and Nguyen 2017; Stolarski et al. 2007). Examples of applications are sludge sedimentation in water and sewage treatment (Zieliński et al. 2018), ultra-fine tailings sedimentation (Li et al. 2016), acid mine treatment and settling of ultrafine tailings (Kefeni et al. 2018). Furthermore, magnetic particles can be obtained from the AMD itself and from the bioleaching of pyritic concentrates from mining tailings (Lopes 2017; Silva et al. 2012; Wei and Viadero 2007).

Thus, the hypothesis of this work is that nano- and micro-magnetite particles (N/M-MP), produced from AMD, can help increase the sedimentation velocity of the sludge generated in by conventional AMD treatment. For this purpose, the sedimentation velocities were evaluated in glass graduated cylinders with and without the use of magnetic

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particles. The N/M-MP were magnetically recovered from the sludge for reuse, and the treated AMD was characterized and compared with effluent discharge standards.

Methods

The AMD sample used in this work was provided by a company that mines coal in the municipality of Figueira, Paraná, Brazil (23°49′15″ S; 50°25′50″W). The magnetic particles were synthesized by dissolving 70 g of ferrous sulphate crystals (melanterite, $FeSO_4 \cdot 7H_2O$), produced from a pyrite concentrate from the same mine, in 1 L of deionized water. The solution was adjusted to pH 10.5 by dropwise addition of 4 mol/L NaOH, which was agitated mechanically at 370 rpm for four uninterrupted days to form the magnetite nano-microcrystals. The suspension was centrifuged, washed three times with deionized water and dried in an oven at 60 °C until it reached a constant weight. A summary of the properties of the synthesized particles is presented in Fig. 1. Details on the synthesis and properties of the N/M-MP, in terms of elemental chemical composition, particle size distribution, density, crystalline phase, and magnetic properties, can be found in Lopes (2017).

The addition of reagents for the treatment of AMD was conducted in a jar test apparatus (Quimis, Q305, Brazil) in 1 L containers. $Ca(OH)_2$ was added with rapid stirring at 60 rpm until pH 8.7 ± 0.1 was reached. The N/M-MP were added at this same level of agitation and the stirring speed was maintained for a minute longer. Subsequently, the flocculant (Floerger AN 934 SH, Chemtall Incorporated, USA), an anionic polyacrylamide of high molecular weight prepared in a concentration of 0.5 g L^{-1} in deionized water, here named PAA, was added, and the frequency of agitation was reduced to 20 rpm, which was maintained for 2 min to provide aggregation between the metallic oxides/hydroxides and the N/M-MP. Following floc formation, the effluent was carefully transferred to 1 L glass graduated cylinders, so that sedimentation could take place. The following situations were considered: (a) natural precipitation of metal hydroxides formed without the addition of N/M-MP nor PAA (control); (b) with the addition of N/M-MP; (c) with the addition of PAA; and (d) with the sequential addition of N/M-MP and PAA. The dosages applied were experimentally adjusted, and the best established conditions (4 g L^{-1} of N/M-MP and 5 mg L^{-1} if PAA) were applied. Chemical concentrations used in reagents dosages trials and their performance can be found in the supplementary material (Appendix 1). To understand the mechanisms involved and interactions between the sedimentation aid agents, an optical microscope (BEL Engineering®, B3) connected to a digital camera (Olympus—DP73—17 megapixel resolution) was used to capture digital images of the N/M-MP dispersed in water and sludge droplet samples in Neubauer plates (0.05 mm × 0.05 mm).

Sedimentation was monitored by the height of the clarified/sludge interface as a function of time, with the aid of a stopwatch. Each condition was performed in triplicate, with variations in the level of the clarified/sludge interface as a function of time of not more than 1.5% (which corresponds to 0.45 cm in the 30 cm sedimentation column); the values plotted on Fig. 2 correspond to the average values. The parameters of sludge volume and time at the critical point, as well as the subsidence velocity of the interface, were evaluated using the methods of Talmadge and Fitch (1955). The volume of sludge obtained was determined at the end of the experiment. A magnetized bar was inserted into the sludge for 5 s to attract the magnetic particles. The particles that adhered to the magnetic bar was carefully washed with water to remove the sludge and the N/M-MP were transferred to a clean glass beaker. The procedure was repeated consecutively 3 times. Finally, the sludge was filtered on qualitative filter paper and dried in an oven at 60 °C, to quantify the dry mass generated. N/M-MP mass recovery was calculated relative to the initial amount added to each experiment by two procedures: (a) by measuring the mass of the collected magnetic material, and (b) by the mass difference of the dried sludge in experiments with and without the addition of magnetic particles.

Furthermore, the liquid effluents generated in the AMD treatment processes were analysed to investigate possible chemical differences. Thus, the raw and clarified AMD of treatments using PAA and N/M-MP + PAA were analysed for pH, conductivity, turbidity, metal content (Al, Ca, Cu, Fe, K, Mg, Mn, Ni, Zn, Pb, and As), and sulphates. The metals were analysed by inductively coupled plasma optical emission spectrometry (ICP-OES) and the sulphate content was measured by the turbidimetric method. All analyses followed the procedures described in "Standard Methods for Examination of Water and Wastewater" (Eaton et al. 2005). The results were compared to CONAMA 430/2011(Brasil 2011), Brazilian legislation that determines standards for effluent discharge.

Results and Discussion

The magnetic particles used to assist the sedimentation process were identified in crystalline terms as magnetite (Fe_3O_4), with a particle size distribution ranging from 0.04 to 20.0 μm. Two populations were identified, the first with a peak in the nanometre range (550 nm) and the other in the micrometre range (8 μm). This particle size distribution was substantially wider and larger than that described by Wei and Viadero (2007), who obtained crystal sizes on the order of

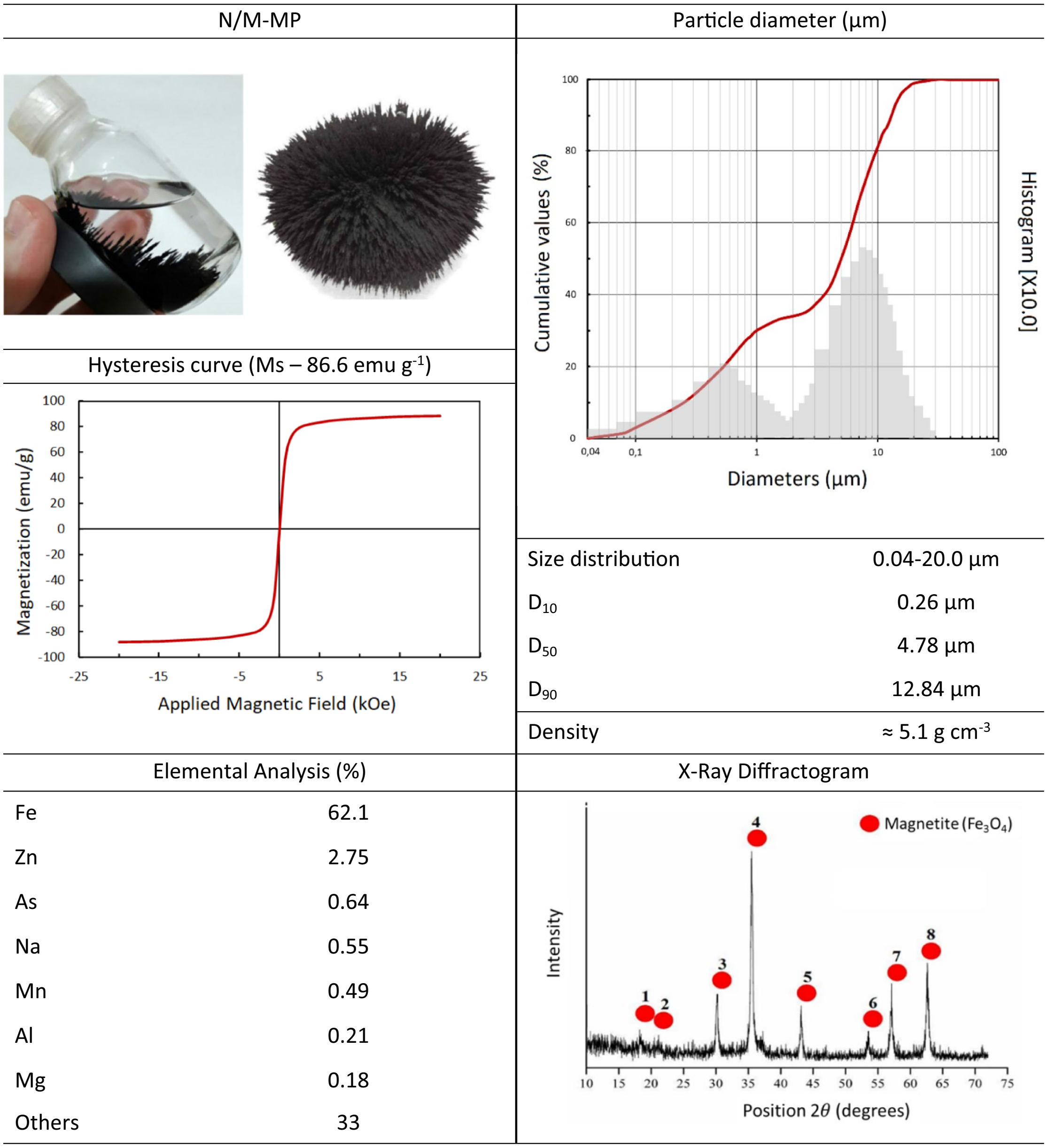

Fig. 1 Characteristics of nano/micro magnetite particles (N/M-MP) produced from melanterite ($FeSO_4{\cdot}7H_2O$). Source: Lopes (2017)

10–15 nm. These authors performed a similar synthesis process, but their crystallization time was only 30 min, while in this work, the time taken was four days. The saturation magnetization obtained in the particles synthesized in this work was 86.6 emu g^{-1}, approximately 93.7% of the saturation magnetization value of magnetite (92 emu g^{-1}) in the form of massive (bulk) material (Cornell and Schwertmann 2003), considerably superior to that of the 29.2 emu g^{-1} zinc ferrite ($ZnFe_2O_4$) synthesized by Lopes (2017) or the 5.6 emu g^{-1} α- Fe_2O_3 particles produced by Kefeni et al. (2018).

Appendix 1 provides information obtained during the screening stage of the work. The addition of N/M-MP without the flocculating polymer provided only a small benefit to the sedimentation properties after a pH adjustment to 8.7 with $Ca(OH)_2$. The best dosage for promoting an increase in sedimentation was 4 g of N/M-MP per L^{-1} of AMD. In the absence of N/M-MP, a dosage of 2.5 mg L^{-1} of PAA was sufficient for good flocculation. However, in the presence of magnetic particles, a slightly higher dosage of 5 mg L^{-1} produced an even better effect. Considering that the mass of precipitates was 10 g L^{-1}, 4 g of N/M-MP per L^{-1} corresponded to a ratio of 0.4 g of N/M-MP per g of sludge. Thus, Fig. 2 shows the sedimentation test (solid/liquid interface × time) for the four proposed treatments: $Ca(OH)_2$ at pH 8.7 without settling agents (control); with the addition of N/M-MP; with the addition of PAA; and with the sequential addition of N/M-MP and PAA. In all treatments, an interface was readily established between the clarified zone and the sludge. There was a large difference in the sedimentation speed (Table 1), relative to the control, when using the PAA, which increased even more with incorporation of the N/M-MP in the flocs. We also observed that the flocculant is essential for thickening the aggregates resulting from the precipitation of metal hydroxides and gypsum and for promoting the bond between them and the magnetic particles.

Regarding the benefit of adding the magnetic particles, the N/M-MP addition nearly doubled the sedimentation rate, from 3.8 cm min^{-1} with the use of PAA to 6.2 cm min^{-1} with the use of N/M-MP and PAA. The addition of N/M-MP also improved the clarified/sludge volume ratio at the critical point (Table 1), even though the volume ratio stabilized at 0.27 for times longer than 25 min. Among the main benefits of the increase in sedimentation speed are the reduced retention time of the effluent and the area occupied by the settling tanks (Metcalf and Eddy 2003). Regarding the reuse of N/M-MP, of the 4.00 g used in the experiment, 3.6–3.8 g were collected magnetically (90–95% recovery). This percentage was also maintained when the same sample of N/M-MP was used sequentially, which ended with a recovery of 79% after 3 consecutive treatment batches with very similar settling performances (supplementary material—Appendix 1).

Figure 3 schematically illustrates the situations studied. The addition of $Ca(OH)_2$ and adjustment of the pH to 8.7 promotes the formation of a sludge mass that settles. The simple addition of N/M-MP promoted a limited gain in sedimentation. In this situation, we observed that an expressive fraction of the added magnetic material settles through the sludge to the bottom of the container. Still, this was the only situation in which the clarified sludge had a turbidity greater than 5 NTU. The addition of PAA, in doses above 2.5 mg L^{-1}, provided a substantial benefit. This relatively high dosage was necessitated by the amount of sludge generated by this concentrated AMD. The addition of magnetic particles followed by PAA allowed even faster sedimentation. In this case, the polymer bonds the precipitate and the magnetic particles, promoting greater sludge density. These observations can be seen in the photographic records carried out with the aid of a microscope and in the image in the supplementary material (Appendix 2). Figure 4a shows an image of the precipitate attained by pH adjustment. In Fig. 4b, it can be seen that without PAA, N/M-MP are dispersed and not necessarily included in the metal hydroxide/gypsum aggregates. Figure 4c shows floc formation and Fig. 4d the evident incorporation of magnetic particles inside the flocs. Regarding the interactions, it can be observed that magnetic particles, when suspended in pure water, magnetically attract each other, forming a relatively structured arrangement (Fig. 5a), such as the one shown in Fig. 5b. The attractive forces between the particles promotes compaction/densification of the floc. In turn, aggregation by the N/M-MP particles increases the particle diameter, increasing the effect of the gravitational force.

The parameters of the raw and treated effluents are compared to the standards established in CONAMA 430 (Brasil 2011) in Table 2. The treatment was efficient, since one of the main treatment goals is the removal of harmful metals. The metals Fe, Al, Zn, and Mn, which have a higher concentration in the raw AMD, were drastically reduced in all situations, fitting the effluent into the release pattern provided for in the legislation. The Ca concentration increased due to the addition of the reagent $Ca(OH)_2$, and its concentration, together with that of sulphate, was established based on the solubility product of $CaSO_4{\cdot}2H_2O$. The addition of N/M-MP did not affect the composition of the treated effluent. In this respect, the study conducted by Kefeni et al. (2018) points out that nanoparticles of α-Fe_2O_3, with paramagnetic properties with a saturation magnetization of 5.6 emu g^{-1}, efficiently removed several elements contained in the AMD. This effect was not evident in the present study using the

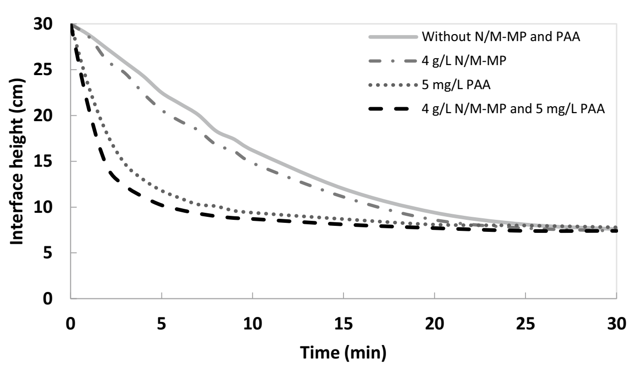

Fig. 2 Sludge settling as a function of time in glass graduated cylinders in the treatment of AMD at pH 8.7 ± 0.1 with $Ca(OH)_2$, considering the control (without the addition of PAA and N/M-MP), the addition of 4 g L^{-1} of N/M-MP, the addition of 5 mg L^{-1} of PAA, and the sequential addition of 4 g L^{-1} of N/M-MP and 5 mg L^{-1} of PAA

Table 1 Parameters of the sedimentation process

Parameter	Treated with $Ca(OH)_2$ at pH 8.7 ± 0.1	
	PAA	N/M-MP + PAA
t_{cp} (min)	4.4	2.8
v_{ct} (cm min^{-1})	3.8	6.2
$(Vf/Vi)_{cp}$	0.42	0.38
$(Vf/Vi)_{\alpha}$	0.27	0.27

t_{cp} settling time to the critical point, v_{cp} average settling rate of the interface until the critical point, $(Vf/VI)_{cp}$ volumetric settle slurry ratio at the critical point, $(Vf/VI)_{\alpha}$ volumetric settle slurry ratio at the endpoint

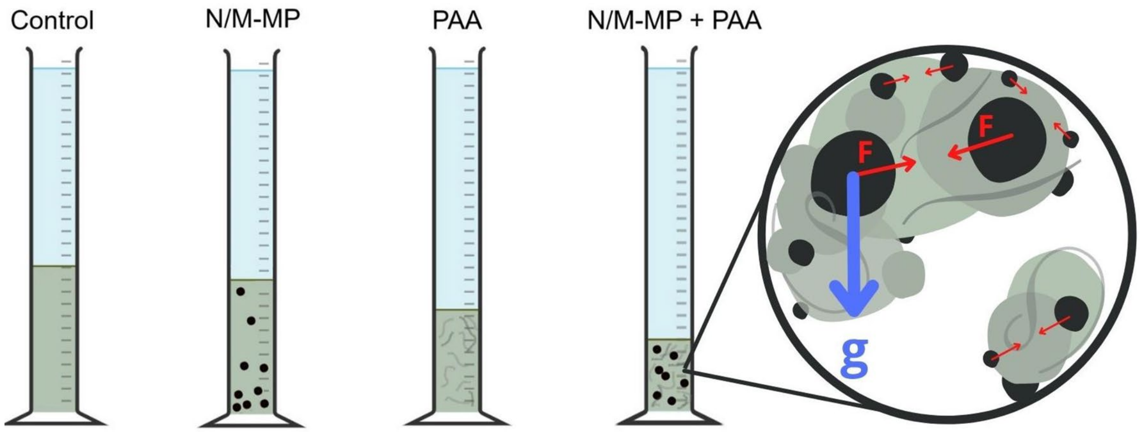

Fig. 3 Sludge settling in glass graduated cylinders after the treatment of AMD at pH 8.7 ± 0.1 with $Ca(OH)_2$: **a** considering the control (without the addition of PAA and N/M-MP), **b** the addition of N/M-MP, **c** the addition of PAA, and **d** the sequential introduction of N/M-MP and PAA

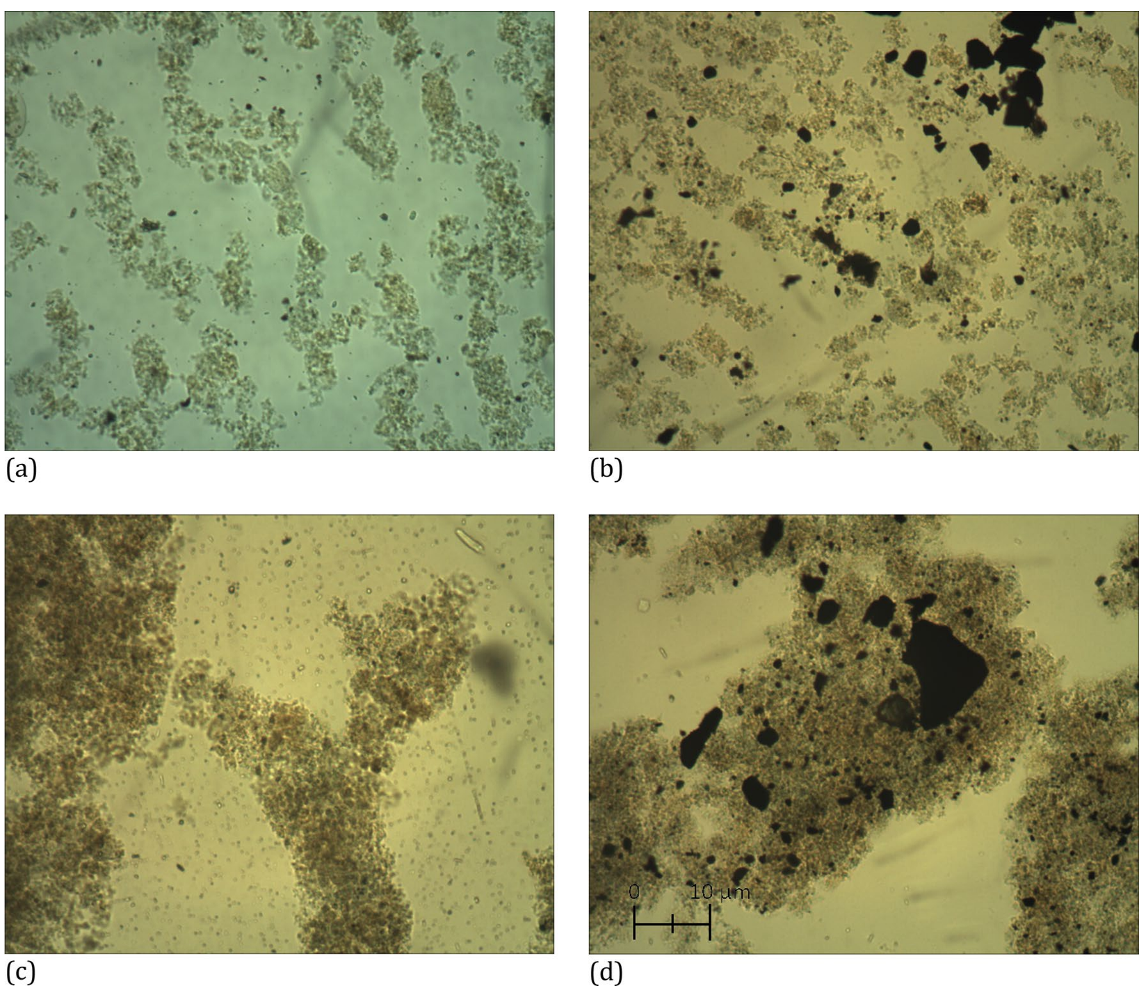

Fig. 4 Optical microscope photographs depicting the appearance of the precipitate attained by AMD treatment at pH 8.7 ± 0.1 with $Ca(OH)_2$, considering the following conditions: **a** without N/M-MP and PAA (control), **b** addition of 4 g L^{-1} of N/M-MP, **c** addition of 5 mg L^{-1} of PAA, and **d** the sequential addition of 4 g L^{-1} of N/M-MP and 5 mg L^{-1} of PAA

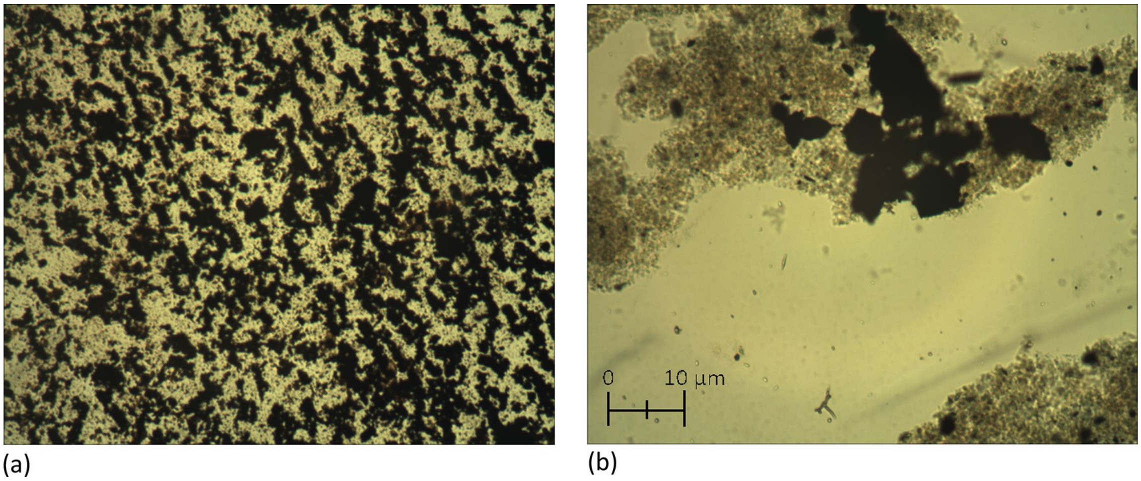

Fig. 5 Optical microscope photographs (100×) revealing: **a** the N/M-MP suspended in water without reagents, showing the magnetic interaction among the particles, and **b** N/M-MP particles aggregated inside a floc by the dual effect of polymer bridging and the magnetic attraction force. After treatment carried out with $Ca(OH)_2$ at pH 8.7 ± 0.1, 4 g L^{-1} of N/M-MP and 5 mg L^{-1} of PAA

N/M-MP of Fe_3O_4, since the effluent attained using the PAA alone had very similar chemical characteristics to the effluent using both N/M-MP + PAA.

From the context described in this work, it is understood that the sedimentation process of the sludge generated by the active treatment of AMD can be accelerated by incorporating magnetic particles. The addition of N/M-MP, due to its high specific density (5.1 g cm^{-3}), increases the sludge sedimentation speed and ease the solid–liquid separation process. Furthermore, investigations could be conducted applying a magnetic field on the generated sludge, with or without the addition of magnetic particles (Zaidi et al. 2014). These arrangements have not yet been tested for the system established in this work. Considering the application of a magnetic field, theoretical and practical considerations proving its effectiveness were established by Stolarski et al. (2007). However, in studies conducted by Zieliński et al. (2018) with activated sludge, where an iron and aluminium coagulant were applied, only aluminium-treated sludge responded with an increased sedimentation rate. In turn, Kamizela et al. (2020) obtained good results and outlined considerations regarding the process in a study on the application of a magnetic field on the sedimentation and filtration properties of a biological sludge treated with a ferric coagulant and a polyelectrolyte. Regarding the joint use of particles and a magnetic field, the potential for success is

Table 2 Parameters analysed for raw AMD and treated considering different sedimentation methodologies

Parameter	AMD Raw	Treated with $Ca(OH)_2$ at pH 8.7 ± 0.1 with		CONAMA 430/2011
		5 mg L^{-1} PAA	4 g L^{-1} N/M-MP + 5 mg L^{-1} PAA	
pH	2.3	8.7	8.7	5–9
Conductivity (µS cm^{-1})	6110	3500	3590	–
Al (mg L^{-1})	157.53	0.34	0.28	–
Ca (mg L^{-1})	357.9	699.9	635.0	–
Cu (mg L^{-1})	0.09	0.60	0.23	1
Fe (mg L^{-1})	478.43	0.13	0.08	15
K (mg L^{-1})	< 0.07	3.51	3.34	–
Mg (mg L^{-1})	167.35	116.66	120.95	–
Mn (mg L^{-1})	43.62	0.36	0.91	1
Ni (mg L^{-1})	0.59	0.06	0.02	2
Zn (mg L^{-1})	53.9	0.03	0.02	5
Pb (mg L^{-1})	0.02	< 0.02	< 0.02	0.5
As (mg L^{-1})	< 0.02	< 0.02	< 0.02	0.5
Sulphates (mg L^{-1})	7410	2835	2959	–

great, and studies must be encouraged to develop the process, whether for the treatment of AMD or for effluents generated in other productive sectors.

Finally, it should be mentioned that the use of N/M-MP could be incorporated into the conventional high density sludge (HDS) method, in which part of the underflow sludge is recycled back to the treatment process and the excess underflow sludge is disposed as sludge wastage (Aubé and Lee 2015; Kuyucak 1998, 1999). This would promote an even denser sludge, with the advantage of easy separation of the magnetic particles by a magnetic field.

Conclusion

The use of nano- and micro-magnetite particles in the AMD treatment process showed promising effects on the settling rate of sludge. The addition of a flocculant polymer is essential for the formation of bridges between the precipitated hydroxides (and other components of the sludge) and the magnetic particles. The addition of magnetic oxides increased the sedimentation speed and the thickening of sludge, allowing for higher process rates and, therefore, a smaller area of settling basins for the same treatment flow. The magnetic property facilitates the recovery of the N/M-MP, allowing it to be returned to the process. AMD can be a source for magnetic particle synthesis, and its own active treatment can benefit from it.

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