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TECHNICAL ARTICLE

Synthesis of a Goethite Pigment by Selective Precipitation of Iron from Acidic Coal Mine Drainage

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Abstract Conventional treatment of acid rock drainage (ARD) from coal mines generates large volumes of sludge, which requires further treatment and disposal. The aim of this work was to study the recovery of iron by selective precipitation to synthetize goethite for use as pigment. Goethite particles were successfully produced, and presented a particle distribution in the range of nano- and micro-sizes, varying from 0.04 to 5.0 µm when produced as paste suspension, or from 0.04 to 25.0 µm when dried at 60 °C and converted to a solid powder. The pigment was used to color a white cement paste, giving it a yellow ochre color. ARD treatment plants can adopt this process to reduce waste disposal issues and produce a valuable mineral (e.g., yellow pigment for concrete).

Keywords Acid rock drainage · Wastewater sludge · Recycling · Iron oxide pigment

Introduction

Acid rock drainage (ARD) causes environmental damage in many coal mining regions. ARD treatment plants in Brazil and other parts of the world mostly use lime or sodium hydroxide for neutralization. The metal precipitates are removed in settling ponds, conventional settling tanks, lamellar tanks, and dissolved air flotation units (Silveira et al. 2009; Skousen et al. 1998). Although active treatment can provide effective remediation, it is expensive and produces a bulky sludge (Johnson and Hallberg 2005; Kontopoulos 1998; Skousen et al. 1998). This sludge typically contains 2–5% solids, high concentrations of iron and aluminum, and minor concentrations of manganese, zinc, and other metals, and represents a major environmental problem.

There have been promising indications for the selective precipitation of metals from ARD as a suitable process to generate industrially useful materials, including iron products (Simate and Ndlovu 2014). The sludge obtained from ARD has been previously considered for the production of coagulants (Finch et al. 1992; Menezes et al. 2009, 2010), adsorbents and catalysts (Flores et al. 2012) and magnetic particles like magnetite and other ferrites (Silva 2010; Silva et al. 2012; Wei et al. 2005).

Another group of minerals of commercial interest is iron oxide pigment (IOP). Natural iron oxides have historically been the major IOP source and still represent a significant

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portion of the IOP market. Synthetic IOPs are widely used though and compete with natural IOPs in many applications, in part because color can be more precisely duplicated and a substantially wider variety of colors can be produced (Allen 1971; Schwertmann and Cornell 1991). IOPs are used in construction materials, such as block, brick, mortar, paving stones, roofing tiles, and ready-mixed and decorative concrete (Tanner 2016). Concrete is the most important construction material. The estimated consumption of concrete worldwide is about 33 billion metric tons (t) per year, and pigments are often used for permanent decorative coloring (Mehta and Monteiro 2013). The pigments used in concrete are mainly iron oxide-based to provide permanent yellow and red colors (López et al. 2009, 2016).

Goethite is of special interest since it can be synthesized in nanometer (nm) and micrometer (µm) sizes that provide a yellow color or the precursor of a red pigment (hematite) by thermal decomposition. Goethite has been extensively used as a yellow pigment, with special interest in coloring construction materials (Cornell and Schwertmann 1996; Streltsova et al. 2013). However, goethite grains have been generally been produced with pure chemicals, i.e. $FeSO_4 \cdot 7H_2O$, $Fe(OH)_2$, $FeCO_3$, $Fe(NO_3)_3$, and $C_{33}H_{57}Fe_2N_3O_{24}$ (Cabral-Prieto et al. 1998; Frost et al. 2005; Kurokawa and Senna 2006; Ristić et al. 2013), and more recently, from waste materials like scrap iron (Nayak and Rao 2005), mill scale (Legodi and de Waal 2007), iron oxide sludge from abandoned coal mines (Hedin 2003), and wetlands constructed for treating coal mine drainage (Hedin 2008).

Important advances in the recovery of iron oxides from ARD were attained by Hedin (2003, 2008). The settled sludge from aerobic passive treatment systems was dewatered and screened to obtain a 70–95% pure goethite (FeOOH) and ferric hydroxide ($Fe(OH)_3$) with strong yellow pigmentary characteristics, high surface area, and small particle size. However, it was observed that organic impurities (vegetative debris, litter, and coal refuse) prevented its ready use as a finished yellow pigment. To overcome this issue, the material was calcined at ≈900 °C, which dehydrated the goethite to hematite (a red pigment), and volatized the organic contaminants. The final product could be used by itself or blended with other materials to yield a finished pigment, and the income generated by pigment production helps pay for the costs of long-term passive treatment.

Active ARD treatment systems by neutralization/metal precipitation also need proper sludge management. We assessed the feasibility of iron recovery by selective precipitation and the synthesis of goethite particles with consideration for the peculiarities of conventional ARD treatment systems. The iron oxide was characterized in terms of fine particle properties (size distribution, grain shape, specific surface area, and crystalline and elemental composition) and applied to color a cement paste, which is the base material for producing colored concrete. This research could potentially reduce ARD sludge generation.

Reactions

The conventional synthesis of goethite by means of chemical precipitation includes the following steps (Cornell and Schwertmann 1996; Schwertmann and Murad 1983; Schwertmann et al. 2004):

Step 1 Selective iron precipitation: hydrolysis of soluble iron by addition of alkali to precipitate the metal as ferric hydroxide at pH 3.6 allows the separation of iron from other metals present in ARD. The process should be followed by successive washes with an aqueous solution at the same pH to remove any undesirable contaminants present in the interstitial water (Reaction 1):

$$Fe^{3+}{}_{(aq)} + 3OH^{-}{}_{(aq)} \rightarrow Fe(OH)_{3(s)}. \tag{1}$$

Step 2 Iron dissolution: the iron hydroxide should be dissolved in water by the addition of nitric acid to form a complex called iron-hexa-aquo ion (Reaction 2).

$$Fe(OH)_{3(s)} + 3HNO_{3(aq)} \rightarrow \left(\left[Fe(OH)_6\right]^{3+}\right)_{(aq)}. \tag{2}$$

Step 3 Goethite crystallization: the synthesis of goethite from Fe^{3+} can be obtained under alkaline conditions by the precipitation of the ferric nitrate solution (iron-hexa-aquo-ion), producing a precipitate called ferrihydrite ($Fe_5HO_8 \cdot 4H_2O$) by the addition of potassium hydroxide at pH 12.0. (Reaction 3):

$$\left(\left[Fe(OH)_6\right]^{3+}\right)_{(aq)} + KOH_{(exc)} \rightarrow \left(Fe_5HO_8 \cdot 4H_2O\right)_{n(s)}. \tag{3}$$

Under these conditions, the initially formed precipitate was dissolved, and released an ionic species ($Fe(OH)_4^-$), which forms crystalline goethite by nucleation, and starting the process of goethite growth (FeOOH) (Reactions 4 and 5).

$$\left(Fe_5HO_8 \cdot 4H_2O\right)_{n(s)} \rightarrow \left[Fe(OH)_4{}^{-}\right]_{(aq)}, \tag{4}$$

$$\left[Fe(OH)_4{}^{-}\right]_{(aq)} \rightarrow FeOOH_{(s)}. \tag{5}$$

Materials and Methods

ARD Sample

ARD was collected from a drainage channel near a coal tailings deposit in the state of Santa Catarina (Brazil) and sealed in high-density polyethylene bottles (Fig. 1a). In the laboratory, the solids and debris in the water samples were removed by settling and the remaining suspended solids were removed by filtration through a 0.45 µm membrane and stored at 4 °C. The pH was measured and the ARD was analyzed for dissolved metals (total Fe, Fe^{2+}, Fe^{3+}, Al, Mn, Zn, Ca, Mg), and sulfate, following the procedures described in standard methods for the examination of water and wastewater (Eaton et al. 2005).

Iron Recovery and Solution Preparation

Iron recovery was achieved by selective precipitation of 1 L of ARD at pH 3.6. The method used for iron precipitation was the same as Wei et al. (2005) and Menezes et al. (2009). The pH of the ARD was increased to, and maintained at 3.6 ± 0.1, with the addition of a 4 M NaOH solution to precipitate the iron as ferric hydroxide/oxyhydroxide. This was further separated from the ARD by centrifugation at 3000 rpm. The supernatant was analyzed for pH and concentrations of dissolved metals and sulfate. Subsequently, the pH was adjusted to 7.0 to reach the wastewater discharge standards. The precipitate obtained at pH 3.6 was washed with distilled water at pH 3.6 ± 0.1, resuspended, and centrifuged; this cycle was repeated three times. The final precipitate was dissolved in nitric acid to achieve a clear acidic iron solution to form an iron-hexa-aqua-ion complex (Reaction 3) (Fig. 1b, c).

Synthesis and Characterization of Goethite

This solution was alkalized with potassium hydroxide and the pH was adjusted to 12.0 to form ferrihydrite (Reaction 4). Immediately, the system was diluted with water and heated to 70 °C for 60 h for goethite crystallization (Reaction 5). The synthesized goethite particles were prepared in two different forms: (a) as a paste—the goethite particles were centrifuged and prepared as a water suspension containing about 50% solids; and (b) as a powder—the goethite particles were dried at 60 °C (Fig. 1d, e). The solids were further analyzed for particle size (laser diffraction, in aqueous solution with 1% sodium polyacrylate dispersing agent), specific surface area (BET), crystalline compounds (X-ray diffraction, XRD) and elemental chemical composition (atomic absorption spectroscopy, AA).

Cement Paste Production and Color Measurements

The colored pastes were produced with commercial white Portland cement in a water/cement ratio of 1:2.5. Goethite powder was added at a powder/cement mass ratio of 1:10. The pastes were hand mixed for 2 min and placed in plastic containers with subsequent curing in a chamber of 95% humidity for 28 days. Figure 1f shows the consolidated cement paste produced.

The color of the cement was measured by reflectance spectra using a Minolta CM-2600D spectrophotometer with an integration sphere associated with an ultraviolet filter. As defined by CIELAB (CIE 1986), illuminant D_{65}, which simulates daylight and the standard observer at 10°, were chosen. At the beginning of the experiment, the calibration was done with two reference points, the zero and a white standard. The color parameters corresponding to the uniform color space (CIE 1995) were obtained directly from the apparatus. Within the uniform space, two color coordinates, a* and b*, as well as a psychometric index of lightness, L*, were defined. a* takes positive values for reddish colors and negative values for greenish ones, whereas b* takes positive values for yellowish colors and negative values for bluish ones. L* presents an approximate measurement of luminosity; according to this property, each color can be considered as equivalent to a member of the grey scale, ranging between black and white, taking values within the range of 0–100. Three measurements were taken at randomized places on the surface of the consolidated cement paste and the mean and standard deviation of L*, a*, and b* parameters were calculated.

Results and Discussion

Table 1 presents the dissolved metal concentrations in the ARD before and after metal recovery. The raw ARD had a low pH and contained elevated concentrations of dissolved metals (Fe, Al, Mn, Zn, Ca, and Mg). When the pH was increased from 2.8 to 3.6, the dissolved Fe concentration dropped from 3200 mg L^{-1} to about 65.7 mg L^{-1}, indicating that approximately 98% of the iron was recovered as iron hydroxide/oxyhydroxide. Moreover, at pH 3.6, the concentration of other metals remained nearly unchanged or dropped slightly, which ensured that the iron precipitated selectively. When the pH was adjusted to pH 7.0, all of the residual Fe and other metals were precipitated. The water quality met the Brazilian Standards for Wastewater Discharge (CONAMA 2011).

The iron hydroxide/oxyhydroxide precipitate from ARD at pH 3.6 was solubilized with nitric acid to obtain a ferric nitrate solution. The predominant metal was Fe (93.5%), present exclusively as Fe^{3+}; other metals were present at very low concentrations. Consequently, Fe^{3+} with relatively high purity was successfully recovered and used to synthesize the pigments. Table 2 presents the main physical (particle size distribution, specific surface area, particle shape), and chemical properties of the goethite.

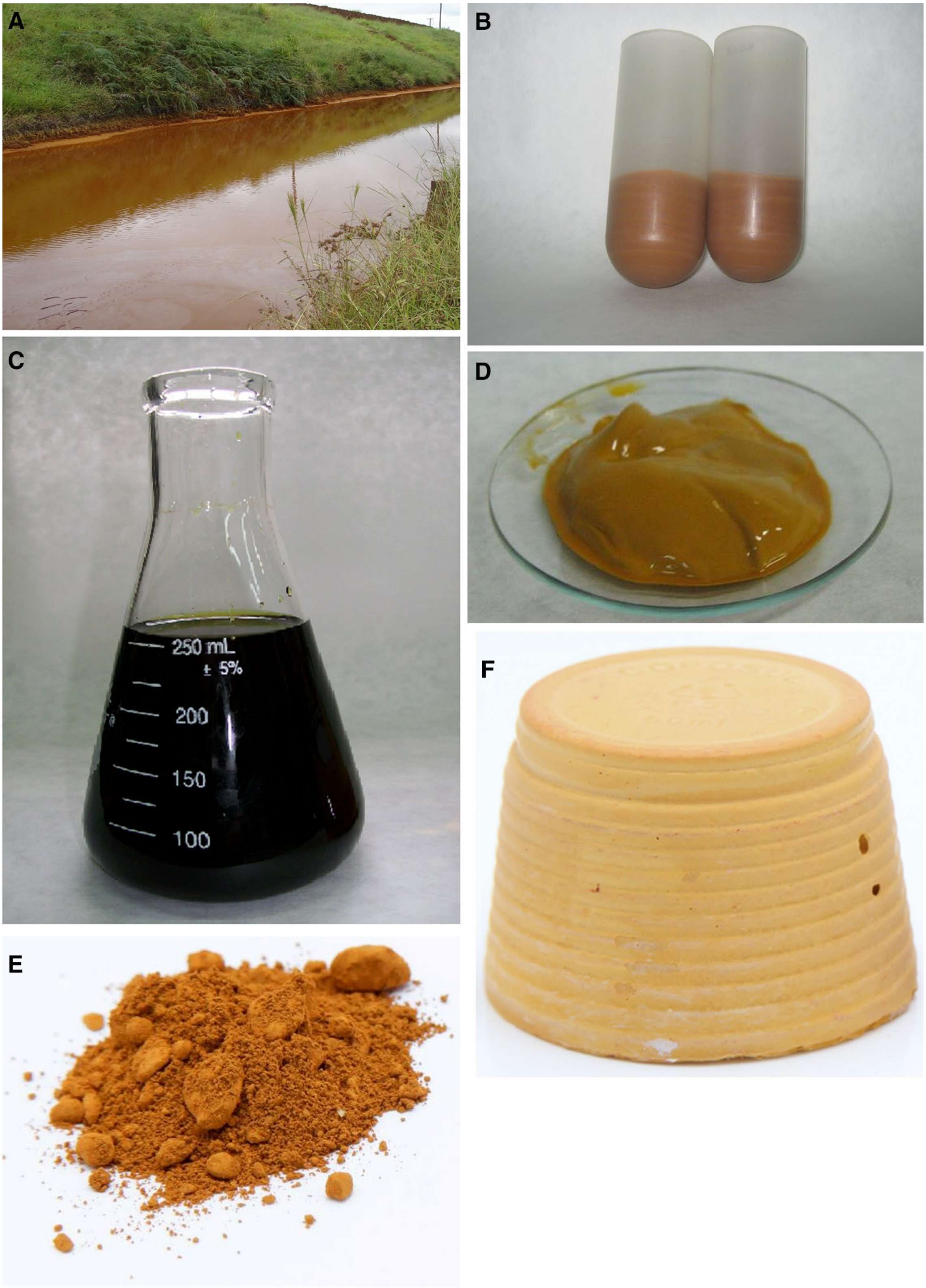

Fig. 1 Steps involved in goethite synthesis by selective precipitation of iron from an ARD: **a** acid rock drainage, **b** iron sludge obtained from selective precipitation at pH 3.6, **c** iron sludge solubilized, **d** goethite—paste, **e** goethite—powder, and **f** colored cement paste −10% goethite

Table 1 Chemical analysis of the raw ARD sample and after metal precipitation at pH 3.6 and pH 7.0

Parameters	Raw ARD	After pH adjustment at 3.6	After pH adjustment at 7.0	Brazilian Standards for wastewater discharge
pH	2.8	3.6	7.0	5–9
$Fe_{(total)}$ ($mg\ L^{-1}$)	3200.0	65.7	0.5	15.0
Fe^{3+} ($mg\ L^{-1}$)	3200.0	65.7	0.5	–
Fe^{2+} ($mg\ L^{-1}$)	ND	ND	ND	–
Al ($mg\ L^{-1}$)	1269.5	1006.7	1.3	–
Mn ($mg\ L^{-1}$)	114.6	91.5	0.6	1.0
Zn ($mg\ L^{-1}$)	80.0	62.0	0.03	5.0
Ca ($mg\ L^{-1}$)	11.8	10.2	10.5	–
Mg ($mg\ L^{-1}$)	8.4	5.3	5.5	–
SO_4^{2-} ($mg\ L^{-1}$)	12600	10,470.0	9236.7	–

ND not detected

Table 2 Properties of goethite particles synthesized by selective precipitation of acid mine drainage

Property	Goethite—from ARD	
	(Paste)	(Powder)
Size distribution (μm)	<0.04–5.0	<0.04–25.0
D90 (μm)	1.74	11.23
D50 (μm)	0.41	2.75
D10 (μm)	0.08	0.73
Surface area ($m^2\ g^{-1}$)	50.8	
Particle shape	Acicular	
Color	Yellow	
Mineralogical composition	Goethite—FeOOH (unique crystalline phase)	
Elemental composition (%) (metals)		
Fe	60.34	
Al	0.79	
Zn	0.17	
Mn	0.10	
Ca	0.13	
Cu	0.004	
Ni	0.013	
Pb	0.006	
Cr	0.002	

Table 3 Colorimetric parameters of the cement paste colored with goethite particles produced from ARD

	Colorimetric parameters		
	L*	a*	b*
Mean ± standard deviation	72.55 ± 0.78	5.10 ± 0.15	41.76 ± 0.04

Goethite prepared as a paste had a nm/μm size distribution; most of the particles (D_{10}–D_{90}) ranged from 0.08 to 1.74 μm, with a D_{50} of 0.41 μm. After drying at 60 °C, the grains agglomerated into clusters and most particles (D_{10}–D_{90}) ranged from 0.73 to 11.23 μm, with a D_{50} of 2.75 μm. Agglomeration while drying could create a problem in several particle systems, i.e., pigment for paint production (Lambourne and Strivens 1999). However, it does not seem to be a major problem for concrete. In fact, Silva (2010) characterized a commercial goethite pigment where most particles presented a D_{10}–D_{90} range of 2.70 to 215.88 μm with a D_{50} of 84.59 μm. These data agree with Dunnous (1997), who emphasized the importance of recognizing that particles of dry IOP do not exist individually. Electrostatic forces and packing compaction cause individual particles to clump together and form agglomerates ranging in size from 20 to 150 μm, with most falling between 60 and 120 μm. It should be noted that the reduced particle size obtained in this work led to fine goethite grains with a BET-specific surface area of 50.8 $m^2\ g^{-1}$, inside the range of 10–130 $m^2\ g^{-1}$ for most pigments (Lambourne and Strivens 1999). The produced goethite was yellow, with acicular grains and FeO·OH as the unique crystalline phase. By elemental analysis, the Fe content in the ARD goethite was measured as 60.34%; the major contaminants were Al, Zn, Ca, and Mn. This agrees with the stoichiometry, since pure goethite is 62.8% Fe.

The goethite powder was used to prepare a cement paste with a yellow ochre color; the colorimetric parameters had a mean value of 72.55 for L*, 5.10* for a*, and 41.76 for b* (Table 3). Standard deviation values of the color parameters were low, which indicates good color homogeneity for all surfaces of the consolidated cement paste. This homogeneity is largely due to proper mixing of the components and the pigment grain size, which was much less than the cement particles, which are normally less than 75 μm (Mehta and Monteiro 2013). As a result, the goethite pigment produced in this work, dispersed in the cement paste in a pigment/white cement mass ratio of 1:10, effectively provided a yellow color. It is important to mention that the practical limit for color saturation, according to literature, is reached using 6 to 12 parts of pigment to 100 parts of cement by weight (Dunnous 1997).

In terms of the environment benefits, considering this specific mine site, the precipitation of Fe^{+3} at pH 3.6 ± 0.1 leads to the formation of a centrifuged iron-rich sludge with 60 mL L^{-1}, with about 10% total solids. About 68% of the overall mass of sludge was recovered in this step, significantly reducing the amount of waste material to be disposed of in landfills.

This sludge can be processed, providing about 5.9 g of pigment per liter of ARD. Considering the average flow rate of 50 m^3 h^{-1}, approximately 2600 t of goethite could be produced annually at this mine. Accounting for the use of 300 kg of white cement to produce 1 m^3 of concrete and a pigment/white cement mass ratio of 1:10, we estimate that it is possible to produce yellow pigment for more than 80,000 m^3 of colored concrete per year at this single ARD site. If used for 10 cm high paving blocks, this would cover a building surface of 80 ha.

The IOPs market is expected to continue to grow during the next several years on a global scale, mostly because of increased construction in China, India, Indonesia, Latin America, and the United States (Tanner 2016). Hence, waste-based pigment production can now be considered an emerging field of research with potential for various industrial and construction applications.

Conclusion

Iron was recovered from ARD using a selective precipitation process at pH 3.6 based on the dissolution of iron hydroxide. The iron was used to synthetize goethite particles. The produced particles ranged in size from nm to µm, varying from 0.04 to 5.0 µm when produced as paste suspension and 0.04 to 25.0 µm when dried at 60 °C to convert to a solid powder, respectively. The pigment can be potentially used in a 10% pigment/cement paste mix, providing a yellow color. Active ARD treatment plants can adopt the process, thereby reducing sludge waste disposal issues and producing a valuable mineral (e.g., yellow pigment for concrete). The next step is to model this process so that the costs of producing pigment in this manner can be estimated.

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