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TECHNICAL ARTICLE

Stability of Coatings on Sulfide Minerals in Acidic and Low-Temperature Environments

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Abstract The effectiveness of silicate- and phosphate-based surface coating agents (Na_2SiO_3, $CaSiO_3$, and KH_2PO_4) in inhibiting the oxidation of sulfide minerals in acidic and low temperature conditions was studied. Rock samples were coated using two oxidation methods: H_2O_2 was mixed with the coating agents in one, whereas in the second, H_2O_2 was applied to the rock surface prior to the coating agent. The second approach was ineffective, but with Method 1, the pH (5) was above the pH (3.6) of the uncoated control sample. Oxidation, calculated after 14 weeks, had been suppressed by up to 96 and 65%, as indicated by the release of Fe^{2+} and SO_4^{2-}, respectively. All three coating agents (at concentrations of 0.1 and 0.3 M) suppressed the release of Fe^{2+} (<3 mg L^{-1}). Na_2SiO_3 inhibited Fe^{2+} and SO_4^{2-} release by 94 and 65%, respectively. Surface protection (or oxidation inhibition) efficiencies of KH_2PO_4 (in terms of Fe^{2+} release) were 67, 94, and 96% with 0.05, 0.1, and 0.3 M, respectively. The Na_2SiO_3 and $CaSiO_3$ coatings, irrespective of their concentrations, reduced iron oxidation by 94 and 84%, respectively. Fourier transform infrared spectroscopy analysis of the Na_2SiO_3 treated samples showed the presence of iron silicate coatings. Based on this work, coating of rock samples with silicate or phosphate can reduce the oxidation rate of sulfide minerals in acidic and low temperature conditions.

Keywords Acid rock drainage · Metal sulfides · Oxidation · Surface coating agent · Silicate · Phosphate

Introduction

Control of acid rock drainage (ARD) generation, for example, by application of hydrofugants, complexation of Fe^{3+}, and microencapsulation/passivation has been studied by Van den Eynde et al. (2009). Hydrofugants prevent contact between sulfides and oxidants by forming a hydrophobic layer on the surface of sulfides, and inhibit the oxidation process. Sodium oleate and phospholipids are hydrofugants that act as protective agents (Jiang et al. 2000; Kargbo et al. 2004; Zhang et al. 2003). These agents can be effective but are very expensive; this limits their practical use (Van den Eynde et al. 2009). Triethylentetramine (TETA) and diethylene tetramine (DETA) have been used for complexation of Fe^{3+} (Cai et al. 2005; Chen et al. 2006); however, these materials are known to possess biological toxicity (Diao et al. 2013).

Microencapsulation/passivation can be achieved by formation of precipitates on the surface of sulfide minerals, which act as a protective barrier. Treatment of pyritic coal mine spoils with sodium silicate (Na_2SiO_3) or potassium phosphate (KH_2PO_4) has generated great expectations in terms of ARD control (Evangelou 1995, 2001; Kargbo and Chatterjee 2005). Addition of $CaSiO_3$ to acid sulfate soil also yielded promising results (Shamshuddin et al. 2004). Most of the previous studies have been conducted

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in a stable environment, with only a few exceptions (Ji et al. 2012; Kang et al. 2015; Table 1). Hence, monitoring and detailed studies of coating agents and their inhibition efficiencies under different conditions are necessary.

The objective of this study was to investigate the durabilities and effectiveness of selected coating agents at inhibiting sulfide oxidation under acidic and low temperature environmental conditions to mimic real weathering scenarios. Acidic (pH 3.5) and low temperature (− 10 °C) conditions were used to simulate the natural environmental settings; the low temperature and pH conditions may destroy coatings on sulfide minerals.

Materials and Methods

Chemicals and Sample Description

All required chemicals including KH_2PO_4, Na_2SiO_3, CH_3COONa, H_2O_2, HCl, and NaOH were purchased from Daejung Chemical Co. (Seoul, Republic of Korea). $CaSiO_3$ was purchased from Junsei Chemical Co. (Tokyo, Japan). All chemicals used in this study were of reagent grade.

Pyrite was purchased from the Hansol Education Co. (Seoul, Republic of Korea). Rock samples were collected at the Geumsan (Chungbuk, Republic of Korea) construction site, where construction work was stopped by AMD formation, due to exposure of pyritic slate (Ogcheon group). Rock samples were crushed and sieved to between 5.6 and 9.5 mm. All pyrite and rock samples were washed with 1 N HCl and rinsed with distilled water 4–5 times to eliminate possible oxidation before the addition of coating agents.

Evaluation of Acid Production Potential

Net acid producing potential (NAPP) and net acid generation (NAG) pH are used to evaluate the AMD-generating potential of rock samples (Jha et al. 2012; Stewart et al. 2006). The ARD generation potential of the rock samples was evaluated in the absence of coating materials, prior to the experiments. Rock samples were ground with an agate mortar and sieved to obtain a −100 mesh fraction (150 μm). ARD generation potential was evaluated according to the protocol of Stewart et al. (2006). Total sulfur content was used to determine the maximum potential acidity (MPA). The rock sample was digested in 0.5 N HCl according to its fizz rating test, and the digest solution was titrated with 0.1 or 0.5 N NaOH to determine the amount of available acid-consuming minerals, such as carbonates and bicarbonates. NAPP was calculated by subtracting the acid neutralization capacity (ANC) from MPA (NAPP = MPA − ANC). The NAG test was performed by reaction with H_2O_2. The NAG pH of the reaction was analyzed after the reaction was completed (Lawrence et al. 1988).

Experimental Procedure

The surfaces of rock samples were coated with Na_2SiO_3, $CaSiO_3$, or KH_2PO_4, and the oxidation inhibition efficiencies were monitored in terms of Fe^{2+} and

Table 1 Efficiency of different coating agents at inhibiting sulfide mineral oxidation as reported in previous studies

Material	Coating agent	Efficiency	References
Pyrite	Na_2SiO_3	Fe^{2+}: 87% reduced (for 6 h)	Bessho et al. (2011)
	Na_2SiO_3	Total Fe: 73% reduced (for 5.7 h)	Kargbo and Chatterjee (2005)
	NPS	Total Fe: 96% reduced (for 70 h)	Diao et al. (2013)
	TEOS	Total Fe: 59% reduced (for 70 h)	
	Silicon and catechol	Total Fe: 63% reduced (for 28 days)	Jha et al. (2012)
	KH_2PO_4	Total Fe: 85% reduced (for 13.3 h)	Evangelou (2001)
Pyrrhotite	TETA	Total Fe: 79–83% reduced (for 90 days)	Cai et al. (2005)
Acid sulfate soil	KH_2PO_4	Total Fe: 67% reduced (for 84 days)	Shamshuddin et al. (2004)
	$CaSiO_3$	Total Fe: 68% reduced (for 84 days)	
Slate (1% volume of iron sulfides)	Na_2SiO_3 (pH 5–6)	SEM analysis: 40% less affected (for 21 days)	Van den Eynde et al. (2009)
	KH_2PO_4 (pH 5–6)	SEM analysis: 45% less affected (for 21 days)	
Mine (NAGpH: 3.6, NAPP: 3 kg H_2SO_4 ton^{-1})	KH_2PO_4 (pH 6)	$SO_4{}^{2-}$: 86% reduced (for 110 days)	Ji et al. (2012)
Sloping rock surface (NAGpH: 2.7, NAPP: 38 kg H_2SO_4 ton^{-1})	Na_2SiO_3 (pH 7)	Fe^{2+}: 99% reduced $SO_4{}^{2-}$: 98% reduced (for 449 days)	Kang et al. (2015)

NPS *n*-propyltrimethoxysilane, *TEOS* tetraethylorthosilicate

SO_4^{2-} concentrations in the leachates. $CH_3COONa{\cdot}H_2O_2$, which generates ferric ions from sulfide minerals, was needed to form a coating. In addition, the CH_3COONa buffer was required to maintain the solution pH during the coating procedure. Coated rock samples were used to fill columns (9 cm diameter, 15 cm height). The experimental conditions are summarized in Table 2. Briefly, 500 mL of each coating agent (KH_2PO_4, Na_2SiO_3, and $CaSiO_3$) was applied at initial concentrations of 0.05–0.3 M to 400 g of the rock samples for 24 h. Each coating agent was adjusted to pH 7 followed by mixing with 0.1 M H_2O_2 and 0.1 M. In the first set of experiments, the oxidizing agent (H_2O_2) was added to the coating material (mixed with the coating agent) followed by application of this mixture to the surfaces to be protected. In the second set of experiments, the rock surfaces were first oxidized with H_2O_2 (pre-oxidation), followed by application of the coating material.

Column experiments were conducted to assess whether the surface coatings were resistant to an acidic (pH 3.5 with H_2SO_4) and low temperature environment. A rainy environment was simulated by pumping 380 mL of deionized (DI) water for 70 min at a rate of 5.4 mL min^{-1} once every 2 weeks. These experiments were conducted at room temperature (25 ± 2 °C) for 14 weeks. Control (without coating agents) experiments were also performed.

Coated rock samples were also subjected to a low temperature (− 10 °C), acidic environment (again, pH 3.5) to mimic site conditions. To simulate oxidation by precipitation on a waste rock dump, DI water was fed into the column using a metering pump once every 2 weeks. The amount of DI water pumped (380 mL) was based on the annual precipitation (1438 mm) in Taebaek (Gangwon, Republic of Korea). The solution obtained after passage through the column was collected once every 2 weeks. The pH, and Fe^{2+} and SO_4^{2-} concentrations of each sample were then monitored for 14 weeks.

Analytical Methods

The pH of the collected leachates was measured using a multiparameter meter (A215, Thermo Scientific, USA). Concentrations of Fe^{2+} and SO_4^{2-} were determined by 1,10-phenanthroline and the SulfaVer 4 method (USEPA method 375.4 and HACH method 8051), respectively, using a spectrophotometer (DR-3900, HACH, USA). The elemental compositions of the rock samples was determined by X-ray fluorescence (XRF, ZSX Primus II, Rigaku, Japan). The silicate coating on the surfaces was characterized by Fourier transform infrared (FT-IR, Vertex 80/80v, Bruker, USA) spectroscopy. The analysis was conducted in duplicate and average values are reported.

Table 2 Experimental conditions under acidic and low temperature environments

Coating agent		CH_3COONa (M)	H_2O_2 (M)	Application of H_2O_2
Species	Concentration (M)			
Control				
Na_2SiO_3	0.05	0.1	0.1	Pre-oxidation
	0.05	0.1	0.1	Mix
	0.1	0.1	0.1	Pre-oxidation
	0.1	0.1	0.1	Mix
	0.3	0.1	0.1	Pre-oxidation
	0.3	0.1	0.1	Mix
$CaSiO_3$	0.05	0.1	0.1	Pre-oxidation
	0.05	0.1	0.1	Mix
	0.1	0.1	0.1	Pre-oxidation
	0.1	0.1	0.1	Mix
	0.3	0.1	0.1	Pre-oxidation
	0.3	0.1	0.1	Mix
KH_2PO_4	0.05	0.1	0.1	Pre-oxidation
	0.05	0.1	0.1	Mix
	0.1	0.1	0.1	Pre-oxidation
	0.1	0.1	0.1	Mix
	0.3	0.1	0.1	Pre-oxidation
	0.3	0.1	0.1	Mix

Results and Discussion

Evaluation of ARD Generation Potential

The rock sample in this study was composed of Si (30.7 wt.%), Fe (26.2 wt.%), Al (18.8 wt.%), K (15.7 wt.%), Ti (2.24 wt.%), and S (2.09 wt.%). A positive NAPP value indicates that a rock sample has the potential to be acid-forming. A low NAG pH is also a sign of ARD generation potential: <3.5, high possibility; 3.5–5.5, low possibility; >5.5, no possibility (Lawrence and Wang 1997). The NAPP values of the samples in the present study ranged from 2.70 to 64.41 kg H_2SO_4 ton^{-1}. The low NAG pH values (2.19–2.46) of the rock samples indicated a high ARD generation potential; therefore, the rock samples were categorized as potentially acid forming (PAF).

Carbonaceous mudstone was classified by Stewart et al. (2006) as PAF, with a total sulfur content of 2.02 (wt.%), a NAG pH of 2.5, and a positive NAPP value (52 kg H_2SO_4 ton^{-1}). The sulfur content, NAPP value, and NAG pH of the rock samples in the present study were comparable to those reported for that mudstone.

Effectiveness of Coating Agents at Inhibiting ARD Generation

The solution pH for the uncoated rock sample was <4, while the concentrations of Fe^{2+} and SO_4^{2-} in the initial leachates were 6.4 and 20 mg L^{-1}, respectively on the 14th day (Fig. 1). At the end of the experimental period of 14 weeks, the levels of Fe^{2+} and SO_4^{2} were significantly higher than in the initial leachates (22.4 and 55 mg L^{-1}, respectively). The extent of sulfide mineral oxidation when coating agents mixed with H_2O_2 were used decreased (10^{-5}, 2, and 40 mg L^{-1} in terms of H^+, Fe^{2+}, and SO_4^{2-} production, respectively) at the end of the 14 weeks (Fig. 2). The results indicated that sulfide mineral oxidation was suppressed by the formation of a coating by reaction of iron and silicate or iron and phosphate (Bessho et al. 2011; Kollias et al. 2014, supplemental information, Fig. S1). The H_2O_2 can interact with the iron sulfide surface, releasing Fe^{3+}. A precipitate of Fe^{3+}-hydroxide on sulfide minerals can then react with the $Si(OH)_4$ or PO_4^{3-} to form a Fe^{3+} hydroxide-silica barrier or a Fe^{3+} phosphate barrier.

The efficiencies of different concentrations (0.05, 0.1, and 0.3 M) of coating agents on oxidation inhibition were also investigated. The pH after application of the first method was higher (pH 5) than that of the uncoated rock sample (pH 3.6) due to less pyrite oxidation; the increased pH might also have lowered the amount of dissolved iron. Higher concentrations of coating agents increased surface stability. KH_2PO_4 had efficiencies of 72, 95, and 96% at concentrations of 0.05, 0.1, and 0.3 M, respectively, as measured in terms of lower Fe^{2+} concentrations in the leachate. Inhibition efficiencies (measured the same way) of Na_2SiO_3 and $CaSiO_3$ coatings were above 94 and 84%, regardless of their concentrations (Figs. 2, 3). Iron silicate inhibited oxidation better than iron phosphate under acidic conditions. An iron silicate coating consists of a combination of an outer silica layer and an interior iron-oxyhydroxide layer, and the outer silica layer prevents its destruction (Evangelou 2001). Na_2SiO_3 showed slightly better inhibition efficiency of Fe^{2+} release (96% at a concentration of 0.05 M) than $CaSiO_3$ (85%). Therefore, the application of coating agents mixed with H_2O_2 significantly inhibited sulfide oxidation. In contrast, no sulfide protection efficiency was observed with the pre-oxidation method, as the Fe^{2+} and SO_4^{2-} levels in the leachates of samples were higher than that of the control.

Temperature changes, especially low temperatures at field conditions such as strip mines, can destroy coatings (Evangelou 2001). The solution pH was below 4 when the uncoated rocks were exposed to a sub-freezing temperature

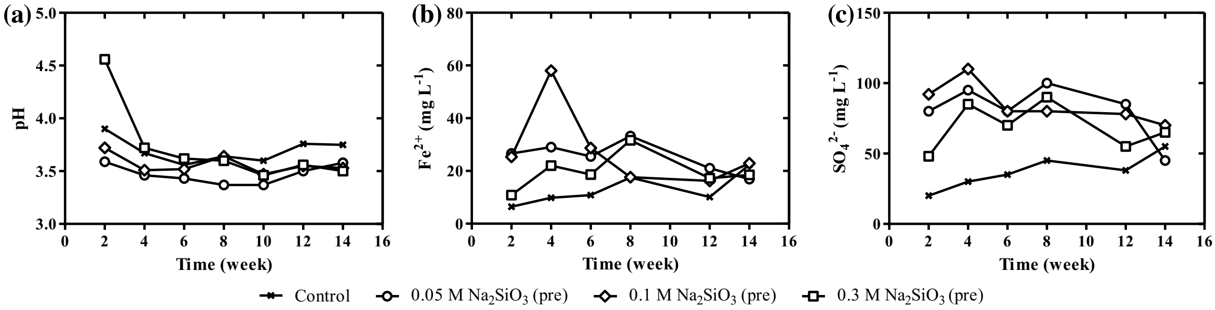

Fig. 1 Solution pH (**a**), Fe^{2+} concentration (**b**), and SO_4^{2-} concentration (**c**) of rock samples surface-coated with Na_2SiO_3 (pre-oxidation) as a function of reaction time under acidic conditions (pH 3.5)

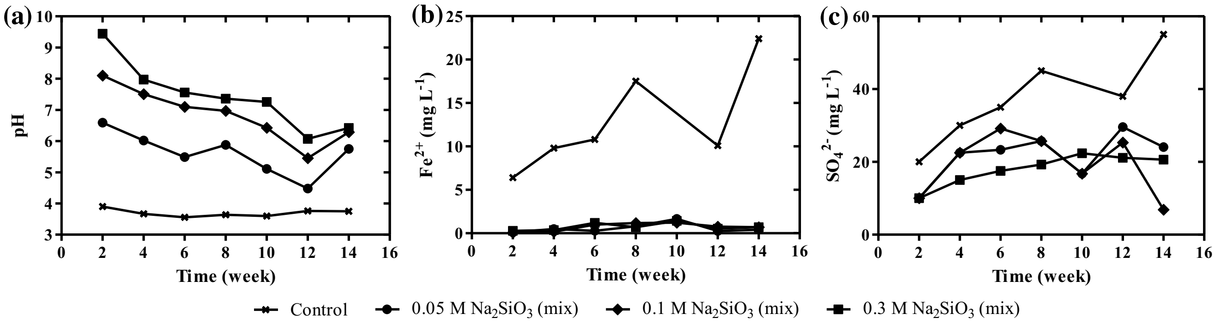

Fig. 2 Solution pH (**a**), Fe^{2+} concentration (**b**), and SO_4^{2-} concentration (**c**) of rock samples surface-coated with Na_2SiO_3 (mix) as a function of reaction time under acidic conditions (pH 3.5)

of − 10 °C (Fig. 4a). The solution pH after application of 0.1 and 0.3 M coating agents stayed above 6, though concentrations of 0.05 M of the coating agents were less effective (lower pH). KH_2PO_4 showed 67% inhibition of oxidation (eluted concentration of Fe^{2+}) at a concentration of 0.05 M, but above 94% inhibition at concentrations of 0.1 and 0.3 M, whereas the inhibition efficiencies of Na_2SiO_3 and $CaSiO_3$ were above 94 and 99% after application, regardless of their coating concentration (Figs. 4, 5). The rate of decrease of solution pH with 0.05 M Na_2SiO_3 and $CaSiO_3$ was greater than that with 0.1 and 0.3 M Na_2SiO_3 and $CaSiO_3$. The eluted concentration of Fe^{2+} was less than that of Fe^{2+} release after KH_2PO_4 treatment. Application of Na_2SiO_3 inhibited SO_4^{2-} generation by 58–65%, while $CaSiO_3$ and KH_2PO_4 inhibited SO_4^{2-} generation by 16–30 and 34–49%, respectively. Iron silicate-based coatings form due to the reaction between ferric ion and soluble silica on the surface of iron sulfide minerals (Bessho et al. 2011). The formation of iron phosphate-based coatings occurs by reaction of ferric ion and phosphate (Kollias et al. 2014). These coatings may inhibit diffusion of oxygen to the surface (Van den Eynde et al. 2009).

Previous studies were conducted under oxidizing conditions with hydrogen peroxide or oxygen, and the compounds evaluated had inhibition efficiencies of 67–99% based on the eluted iron concentration (Table 1). The three coating agents used in this study had inhibition efficiencies of 94–96% under acidic conditions and 94–99% at a low temperature. Furthermore, the cost of coating agent (Na_2SiO_3) was also estimated (supplementary information, Table S1).

FT-IR Studies of Silicate-Based Coatings

The FT-IR studies performed to characterize the silicate-coated surfaces of the pyrite and rock samples suggest the presence of adsorbed monodentate silicate [Fe–O–Si–$(OH)_3$; Fig. 6b, d]. The peaks at 468, 905, and 928 cm^{-1} correspond to the bending vibrations of Fe–O–Si–(OH), in agreement with Vempati and Loeppert (1989). The Si–O–Si bending vibrations of the Na_2SiO_3-treated samples were present at 1030 and 1071 cm^{-1}. The absorption bands at 1013, 1030 and 1071 cm^{-1} correspond to asymmetric stretching of Si–O–Si bonds formed due to condensation of free residual silanol groups (Diao et al. 2013).

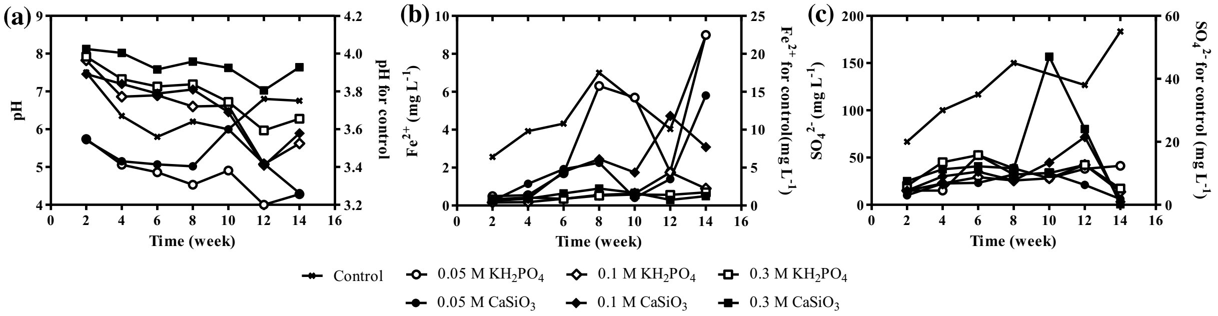

Fig. 3 Solution pH (**a**), Fe^{2+} concentration (**b**), and SO_4^{2-} concentration (**c**) of rock samples surface-coated with KH_2PO_4 or $CaSiO_3$ (without pre-oxidation) as a function of reaction time under acidic conditions (pH 3.5)

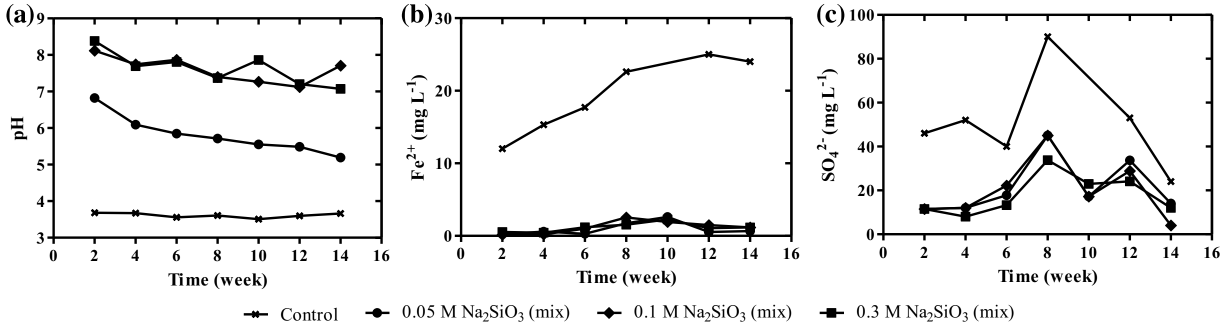

Fig. 4 Solution pH (**a**), Fe^{2+} concentration (**b**), and SO_4^{2-} concentration (**c**) of rock sample surface-coated with Na_2SiO_3 as a function of reaction time at low temperature (– 10 °C)

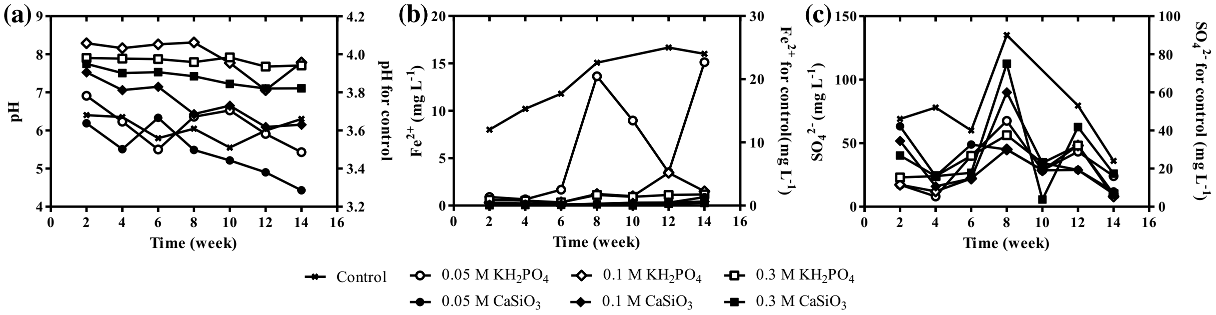

Fig. 5 Solution pH (**a**), Fe^{2+} concentration (**b**), and SO_4^{2-} concentration (**c**) of rock samples surface-coated with KH_2PO_4 and $CaSiO_3$ (without pre-oxidation) as a function of reaction time at low temperature (−10 °C)

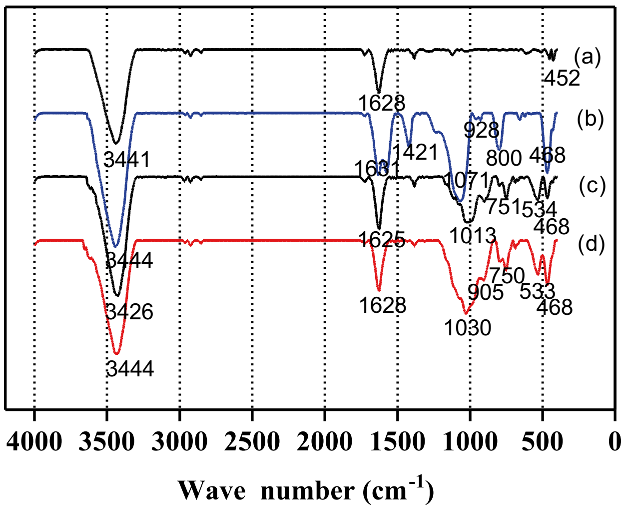

Fig. 6 FT-IR spectra of uncoated pyrite (*a*), Na_2SiO_3-coated pyrite (*b*), uncoated Geumsan rock sample (*c*), and Na_2SiO_3-coated Geumsan rock sample (*d*)

Conclusions

The selected silicate- and phosphate-based coating agents (Na_2SiO_3, $CaSiO_3$, and KH_2PO_4) react with ferric ion (Fe^{3+}) to form a protective coating on mineral surfaces. FT-IR spectra of Na_2SiO_3-treated pyrite showed the presence of iron–silicate coatings. The coating agents were effective at inhibiting chemical oxidation under acidic and low temperature conditions. Application of a coating agent mixed with an oxidizing agent (H_2O_2) is recommended, as application of coating agents after pre-oxidation of the surface was not effective at protecting against the oxidation of sulfide minerals; in fact, it accelerated oxidation rather than inhibiting it. Acidic and low temperature conditions did not affect the effectiveness of the coatings. Higher concentrations (0.1 and 0.3 M) of coating agents were better at inhibiting surface oxidation than lower concentrations (0.05 M). Iron silicate-based coatings are more suitable than iron phosphate-based coatings for field application at ARD sites. Further pilot-scale studies to assess the ability of various coating agents to inhibit the oxidation of pyrite minerals at ARD generation sites are recommended.

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