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ABSTRACT

Present studies on the electrochemical aspects of pyrite flotation in the presence of reducing agents were conducted to explain the effects observed during flotation of the separated organic and mineral-rich fractions of coal independently when sodium dithionite was used as an additive during and after grinding. In addition, the electrochemical phenomena occurring on the surface of pyrites of different origin was studied to aid in explaining the differences observed in the flotation of various coal-derived pyrite samples. Potentiometric and voltammetric measurements have been conducted on electrodes prepared from natural pyrite samples of mineral and coal origin. It was shown that the rest potential of pyrite electrodes decreases very significantly with increased concentration of sodium dithionite, increased pH, and stirring of solution. Mineral pyrite displayed lower potentials than pyrites of coal origin.

Voltammetric curves recorded on FeS_2 electrodes indicate the presence of elemental sulfur, iron-oxy, and iron-hydroxy compounds, even on the freshly prepared surfaces. Comparison of voltammetric curves with results of potentiometric measurements show that in the presence of dithionite several electrochemical processes are expected to take place on the pyrite surface: reduction of iron oxidation compounds, reduction of elemental sulfur, and cathodic decomposition of the mineral itself. It is also expected that these processes will result in changes of surface properties of pyrite during flotation and oil agglomeration.

Voltammetric curves recorded in solutions of different pH with and without sodium dithionite demonstrate that pyrite is a very good electrocatalyst in the anodic oxidation of dithionite. During the negative application of voltage, pyrite oxidation products are reduced and pyrite is decomposed. The increase of the pyrite potential after cathodic pretreatment results in the formation of elemental sulfur at potentials much lower than those required for anodic oxidation of pyrite.

The electrochemical results observed and discussed during this investigation are very suitable as preliminary tests of individual pyrite samples. They enable prediction of the behavior of pyrite in a flotation pulp containing a reducing agent as an additive for the modification of surface properties. According to these results, the reduction of elemental sulfur should decrease the floatability during flotation without a collector or with nonpolar collectors such as diesel fuel or kerosene. Simultaneously, when subsequent oxidation takes place on pyrite after its pretreatment with reducing agent, the floatability of pyrite is expected to increase as a result of the formation of elemental sulfur.

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OBJECTIVE

The primary objective of this research is to advance the application of surface science to improve the beneficiation of coal to minimize potential acid rain precursors. This study addresses techniques to alter and control the surface chemistry of coal and mineral particles, especially pyrite, during comminution in a stirred ball mill in order to enhance liberation and to improve subsequent separation using advanced physical beneficiation techniques applicable to ultrafine coal.

INTRODUCTION

In previous reports(1,2) the influence of introducing sodium dithionite ($\text{Na}_2\text{S}_2\text{O}_4$) during grinding and subsequent separation of the organic fraction from the mineral fraction of coal was described. Improvement in the flotation of coal from pyrite in the presence of sodium dithionite was shown using microbubble techniques. The observed influence of dithionite on the separation was explained as a result of electrochemical reduction processes taking place on the coal and mineral surfaces. The consequence of dithionite addition during grinding was an increase in floatability of the organic (coal) fraction and a decrease in floatability of the mineral (pyrite) fraction.

Voltammetry can be particularly useful for studying surface reactions. For our purposes, this technique involves applying a potential, scanned linearly with time across the regions of stability of electrode material and possible reaction products, and observing the resultant current. Reactions are indicated by linear or non-linear changes of the current, and the formation of monolayers or multilayers can be inferred from the shape of voltammetric curves. Such electrochemical investigations provide information on the kinetics and mechanisms of electrode reactions and have been also applied extensively to semiconducting sulfide minerals.

Electrochemical processes on pyrite in aqueous solutions have been investigated by Hamilton and Woods(3). Results indicated the presence of elemental sulfur and sulfate ions in the oxidation reaction products. The relative amount of hydrophobic elemental sulfur in these products was observed to decrease with an increase in pH and electrode potential. According to their investigation, the hydrophilic-hydrophobic properties of pyrite are expected to change with pH and redox potential of the flotation slurry. Electrochemical aspects of pyrite flotation and depression were also discussed by Janetski et al.(4). Other investigations by Ogunsola and

Osseo-Asare(5,6), Briceno and Chander(7), and Lai et al.(8) show essential differences in the electrochemical properties of coal-derived and mineral-derived pyrite.

Based on the hypothesis that electrochemical properties of a particular pyrite sample control its floatability, potentiometric and voltammetric measurements were made using pyrite electrodes prepared from different specimens of mineral pyrite and coal-derived pyrite. Present studies on the electrochemical aspects of pyrite flotation in the presence of reducing agents were conducted to explain the effects observed during flotation of the separated organic and mineral-rich fractions of coal independently when sodium dithionite was used as an additive during and after grinding. In addition, the electrochemical phenomena occurring on the surface of pyrites of different origin were studied to aid in explaining the differences observed in the flotation of various coal-derived pyrite samples. The influence of a reducing agent on the processes taking place on the pyrite surface was simulated electrochemically by imposing a potential from an external source using a potentiostat. The observed current resulting from the applied potential was a measure of the rate of surface processes.

The results of our electrochemical measurements are expected to be useful in explaining the role of processes taking place on the pyrite surface in solutions containing different reducing agents (e.g., $\text{Na}_2\text{S}_2\text{O}_4$ and Na_2S). Based on the electrochemical results, we also expect to explain some differences observed in the behavior of individual mineral and coal-derived pyrites during either flotation or oil agglomeration.

PROGRESS

MATERIALS AND METHODS

Electrode Preparation

Electrodes were prepared from natural samples of coal-derived pyrite (from an Iowa mine, from the Illinois No.6 seam, and from an unknown origin) and mineral pyrite from Huanzala, (Peru). Electrical contact between the samples and an external circuit was achieved by attaching an insulated copper wire to the sample using a conducting epoxy resin (TRA-DUCT 2902, TRA-CON, Inc., Medford, Mass). The sample was then encapsulated in a nonconducting epoxy resin. After curing, the lower face of the pyrite was exposed by grinding away the epoxy. The electrode surface was freshly prepared before each experiment by wet polishing on 600-grit silicon-carbide paper. The working surface was then rinsed with distilled water and the electrode was introduced to the electrochemical cell containing a deoxygenated solution of constant pH.

Measurements

Electrochemical measurements were carried out at ambient temperature using a conventional three-electrode cell. Potentials were measured relative to a saturated calomel electrode which has a potential of +0.244V on a hydrogen electrode scale. All potentials in this report are referenced to this electrode. A platinum foil electrode, separated from the working electrode compartment by a glass frit, was used as an auxiliary electrode. Stationary pyrite electrodes were used in these investigations. All experiments were conducted in solutions prepared from reagent-grade chemicals and deionized water. Prior to the electrochemical measurements, the solutions were deoxygenated by bubbling argon through them. Solutions of different pH (4.6, 9.3, and 11.8) were used in electrochemical pyrite examinations without and with sodium dithionite of different concentrations (5×10^{-4} M, 5×10^{-3} M, 2×10^{-2} M, and 1×10^{-1} M).

Voltammetric curves, initiated from the open circuit potential (rest potential) and polarized in cathodic and anodic directions, were recorded using freshly polished electrodes at a linear potential sweep rate of 25 mV/s. Polarization curves were recorded using a Pine Instrument Corp. model RDE-3 potentiostat equipped with a program generator and an XY recorder. Potentials were measured with a model 8050A Fluke digital multimeter. The analysis of electrochemical measurements on pyrites was based on oxidation or reduction of the surface during a linear potential sweep and analysis of the products from subsequent reverse potential scans. Interpretation of electrochemical behavior of pyrite was also made on the basis of literature data.

RESULTS AND DISCUSSION

Rest Potentials

A pyrite electrode, immersed in an aqueous solution with no external potential imposed, generates an open circuit potential (rest potential). The magnitude of the rest potential, which significantly depends on the composition of the solution, is a function of various electrochemical processes taking place on the electrode surface. In our experiments, rest potentials were measured on freshly polished coal-derived pyrite and mineral pyrite electrodes in buffered solutions of pH 4.6, 9.3, and 11.8, and in the same solutions containing sodium dithionite, a reducing agent. Figures 1a-d show the potential vs. time relationships recorded on four pyrite electrodes in buffered (pH 9.3) sodium dithionite solutions of different concentration. Figures 2a and 2b show the potentials of pyrite electrodes versus dithionite concentration recorded after 20 s in unstirred solutions and after 120 s in stirred solutions, respectively. It is easily seen in Figure 2 that the rest potentials of pyrite decrease as the concentration of sodium dithionite increases. Moreover, the rest potentials observed for mineral pyrite were

Figure 1. Potential vs. time relationships for mineral pyrite (a) and coal-derived pyrites (b,c,d) electrodes at pH 9.3 in the presence of $\text{Na}_2\text{S}_2\text{O}_4$.

lower than those observed for coal-derived pyrite. Only the rest potentials of pyrite of unknown origin recorded in stirred $\text{Na}_2\text{S}_2\text{O}_4$ solution at concentration above 10^{-2} M displayed the lower potentials.

Significant decreases in electrode potentials were observed when the solutions were stirred during measurement. This suggests that the rate of electrode processes on the pyrite electrodes is limited by mass transport to or from the electrode surface. The influence of stirring was less pronounced at higher dithionite concentrations.

Similar rest potential measurements were conducted with the same

Figure 2. Potential vs. $\text{Na}_2\text{S}_2\text{O}_4$ concentration plots for coal-derived pyrite and mineral pyrite electrodes after 20 seconds in an unstirred solution (a) and after 120 seconds in a stirred (b) solution at pH 9.3.

Figure 3. Potential vs. time relationships for mineral pyrite (a) and Iowa coal-derived pyrite (b) electrodes at pH 11.8 and in the presence of $\text{Na}_2\text{S}_2\text{O}_4$.

electrodes in solutions of pH 11.8 containing sodium dithionite of different concentrations (Figure 3). Results presented in Figures 3 and 4 indicate

Figure 4. Potential vs. $\text{Na}_2\text{S}_2\text{O}_4$ concentration plots for coal-derived pyrite and mineral pyrite electrodes at pH 11.8 after 20 seconds in an unstirred solution and after 120 seconds in a stirred solution.

that rest potentials of pyrite in sodium dithionite solutions are strongly affected by pH and are lower at higher pH. The rest potential for mineral pyrite was lower than the potential observed for coal-derived pyrite. Measurements in the solutions of pH 4.6 (Figures 5 and 6) show that both mineral pyrite and coal-derived pyrite samples exhibit higher potentials in relation to those observed at pH 9.3 and 11.8. It can also be seen from Figure 6 that the potential variations of pyrite electrodes at pH 4.6 observed for sodium dithionite concentrations exceeding 5×10^{-3} M are insignificant. Relative potential changes observed for dithionite concentrations below 5×10^{-3} M were more pronounced. This suggests that for lower concentrations of reducing media, its transport to the pyrite surface is a rate determining step. At higher $\text{Na}_2\text{S}_2\text{O}_4$ concentrations the surface reaction on pyrite controls the rate of the process.

The relationship of potential vs. $\log[\text{Na}_2\text{S}_2\text{O}_4]$ recorded for both coal-derived pyrite and mineral pyrite electrodes were not straight lines, indicating that the values most likely represent mixed potentials which are characteristic of corrosion processes. The potential vs. time curves measured for coal-derived pyrites at pH 9.3 and 11.8 are very similar (Figures 1,3). However, the potential-time relationships observed for

Figure 5. Potential vs. time relationships for mineral pyrite (a) and coal-derived pyrite (b,c,d) electrodes at pH 4.6 in the presence of $\text{Na}_2\text{S}_2\text{O}_4$.

coal-derived pyrite and mineral pyrite at pH 4.6 (Figure 5) are very different from those for pH 9.3 and 11.8.

Voltammetric Measurements

Figures 7-12 show the voltammetric curves measured for electrodes prepared from mineral pyrite and coal-derived pyrite. The potential sweeps commenced from rest potential and each sweep was carried out on a freshly polished surface.

The cathodic curve recorded in a unstirred solution (pH 4.6) for mineral pyrite (Figure 7) exhibited a small peak at -0.06 V and a more distinct peak at -0.50 V. On the cathodic curve for coal-derived pyrite (Figure 8) the corresponding peaks were observed at very similar potentials but the

Figure 6. Potential vs. $\text{Na}_2\text{S}_2\text{O}_4$ concentration plots for coal-derived pyrite and mineral pyrite electrodes at pH 4.6 after 20 seconds in an unstirred solution and after 120 seconds in a stirred solution.

Figure 7. Voltammetric curves for a mineral pyrite electrode in an unstirred (a) and a stirred (b) solution at pH 4.6. ($dE/dt=25$ mV/s, 1-start in cathodic direction, 2-start in anodic direction).

Figure 8. Voltammetric curves for coal-derived pyrite electrode in stirred (a) and unstirred (b) solution at pH 4.6 $dE/dt=25$ mV/s, 1-start in cathodic direction, 2-start in anodic direction, 3-curves recorded at potential below +0.2 V.

recorded current was higher than for the mineral pyrite. According to Hamilton and Woods(3) and Lekki and Chmielewski(9), the only cathodic process expected at a potential of -0.06 V and pH 4.6 is the reduction of iron-oxo and iron-hydroxy products formed on the pyrite surface during electrode preparation in contact with air. Elemental sulfur, formed simultaneously on FeS_2 during oxidation, was observed to reduce at lower cathodic potentials (-0.5 V, Figures 7,8) according to the following reactions:

For higher pH values the formation of hydrosulfide ions is expected during the sulfur reduction:

More cathodic polarization results in a decomposition of the pyrite itself and a liberation of H_2S or HS^- (3,10) in the reactions:

In the reverse anodic scan, in an unstirred solution, a formation of elemental sulfur is the most likely process, according to numerous literature data (3,10-13):

Consequently, the corresponding anodic peak appears on the voltammetric curves at -0.2 V (Figure 7). When the anodic sweep was reversed at potential +0.2 V (Figure 8b) only the cathodic peak of reduction of

Figure 9. Voltammetric curves for coal-derived pyrite electrode in unstirred (a) and stirred (b) solutions at pH 9.3. ($dE/dt=25$ mV/s, 1-start in cathodic direction, 2-start in anodic direction, 3-curves recorded at potentials below 0.0 V).

elemental sulfur was observed at -0.5 V. This indicates the formation of elemental sulfur on mineral pyrite and coal-derived pyrite when the potential of pyrite changes from reductive to slightly oxidative. Elemental sulfur can not be formed on a pyrite surface when it is oxidized at potentials below +0.1 V without cathodic pretreatment.

Similar measurements conducted in a stirred solution at pH 4.6 did not exhibit any anodic peak at -0.2 V (or this peak was essentially reduced) as a result of the dispersion of cathodic electrode products from the pyrite surface. The anodic peak observed on the reverse curve at potentials of +0.2 to +0.3 V in both stirred and unstirred solutions (Figure 8) can be attributed to the transformation of solid species on the pyrite surface, most likely Fe(OH)_2 to Fe(OH)_3 :

When the polarization of coal and mineral pyrites at pH 4.6 was first conducted in an anodic direction to +0.6 V, the reverse cathodic peaks were

Figure 10. Voltammetric curves for mineral pyrite electrode in stirred (a) and unstirred (b) solution at pH 11.8. ($dE/dt=25$ mV/s, 1-start in cathodic direction, 2-start in anodic direction).

shifted to about -0.15 V, and were observed for both mineral and coal-derived pyrite electrodes as a result of FeS_2 oxidation (see Figure 8). The cathodic current after previous anodic oxidation was observed to be higher in relation to those recorded on fresh prepared pyrite electrode. This increase was evident for both cathodic peaks.

Figure 11. Voltammetric curves for mineral pyrite (a) and coal-derived pyrite (b) electrodes with anodic dithionite oxidation curves on the same pyrite electrodes in unstirred solution at pH 4.6.

Cathodic curves recorded at pH 9.3 in unstirred (Figure 9a) and stirred (Figure 9b) solutions did not exhibit any peak at -0.15 V. The cathodic peak appeared, however, at a potential of -0.6 V, and was correlated with the reduction of elemental sulfur and iron oxidation products since both these reactions are expected to take place at similar potentials in basic solutions. The presence of S^0 on the pyrite surface was the result of oxidation of HS^- ions at -0.3 V and oxidation of pyrite itself. In stirred solutions the anodic peak at -0.3 V was observed to decrease as a result of the dispersion of soluble cathodic reaction products (HS^-) from the electrode surface. Unlike the more acidic solutions (pH 4.6), at pH 9.3 two electrochemical processes are responsible for the anodic peak at a potential of -0.3 V, the formation of elemental sulfur, and the oxidation of iron(II) to iron(III) in solid state. The latter was confirmed by the presence of a peak at -0.3 V recorded in stirred solution. This process was observed to be less pronounced on the coal-derived pyrite.

At pH 11.8, a peak at -0.4 V appears on the reverse anodic scan for the mineral pyrite in both stirred and unstirred solutions (Figure 10). This is

Figure 12. Voltammetric curves for mineral pyrite (a) and coal-derived pyrite (b) electrodes with anodic dithionite oxidation curves on the same pyrite electrodes in unstirred solution at pH 11.8.

evidence of a solid state reaction, most likely the oxidation of $\text{Fe}(\text{OH})_2$ to $\text{Fe}(\text{OH})_3$. The magnitude of this peak decreased slightly when the solution was stirred since the dissolved species, most likely HS^- ions, were also considered to be dispersed in the course of this reaction. The formation of elemental sulfur is also expected along with $\text{Fe}(\text{OH})_3$ in this potential range. The shape of the anodic peak recorded on the coal-derived pyrite electrode is different from the peak recorded on mineral pyrite and suggests a higher occurrence of elemental sulfur formation according to reaction (6).

Electrochemical Interpretation of the Role of Sodium Dithionite in Surface Processes on Pyrite

According to the measurements of rest potentials on pyrite electrodes in the solutions of $\text{Na}_2\text{S}_2\text{O}_4$ (Figures 1-6) and voltammetric measurements carried out on FeS_2 in the solutions of the same pH (Figures 7-10), the potentials exhibited by pyrite correspond to the range of reduction of pyrite oxidation products and to the cathodic decomposition of pyrite itself. Therefore, it can be expected that when sodium dithionite is present in the pyrite flotation pulp, the particles of FeS_2 being in contact with $\text{Na}_2\text{S}_2\text{O}_4$ solution

will display the potential corresponding to the reduction of pyrite oxidation products and of pyrite. The potential of pyrite particles will decrease to the value where the surface of FeS_2 can be "cleaned" in the course of electrochemical processes involving the reduction of pyrite and pyrite oxidation products during a cathodic process and of dithionite oxidation during an anodic process. The surface properties of the pyrite particles depend on the final composition of the surface layer after pyrite-dithionite interaction. After oxidation, the surface of pyrite grains is expected to be covered by different products (S^0 , $\text{Fe}(\text{OH})_2$, $\text{Fe}(\text{OH})_3$, Fe_2O_3 , Fe_3O_4); the composition of the surface layer is a function of several parameters. At the very low potentials exhibited by pyrite in dithionite solutions, the oxidation products will be removed from the surface of mineral particles and the surface properties of pyrite are expected to be changed.

In Figure 11 we compared the voltammetric measurements for coal-derived pyrite and mineral pyrite electrodes in a solution of pH 4.6 with anodic polarization curves recorded on the same electrodes in the presence of sodium dithionite of different concentration. According to these results the anodic oxidation of dithionite is observed in the potential range corresponding to cathodic reduction of iron oxidation products and elemental sulfur from the pyrite surface. An observed decrease of pyrite electrode potential with increasing $\text{Na}_2\text{S}_2\text{O}_4$ concentration and stirring of the solution (Figures 1-6) indicates that the anodic reaction is the rate determining step, particularly at low concentrations of dithionite. For dithionite concentrations exceeding 10^{-2} M the observed potential changes seem to be insignificant. In this range of concentration the superficial reduction of pyrite and pyrite oxidation products is expected to be the rate determining step.

CONCLUSIONS

The rest potential of individual pyrite electrodes in solutions of sodium dithionite decreases with an increasing concentration of the additive, increasing pH of the solution, and in stirred solutions. Mineral pyrite exhibited lower electrode potentials in $\text{Na}_2\text{S}_2\text{O}_4$ solutions than coal-derived pyrites. Voltammetric measurements show that at the pyrite surface freshly prepared by wet polishing in open to air atmosphere, presented are the mineral oxidation products (elemental sulfur and iron-oxy and iron-hydroxy compounds). The potential of cathodic reduction of these products decreases with increasing pH.

Voltammetric and potentiometric experiments demonstrated that in the presence of sodium dithionite the potential exhibited by FeS_2 electrodes corresponds to the reduction of pyrite oxidation products and cathodic

decomposition of pyrite itself. It was also demonstrated that both coal and mineral pyrites are very good electrocatalysts in the anodic oxidation of dithionite.

When the potential of the pyrite electrode was increased after cathodic pretreatment at pH 4.6, the formation of elemental sulfur on the FeS_2 surface was the most likely reaction at a potential -0.2 V. At higher pH the simultaneous formation of ferric hydroxide was also observed in the potential range from -0.2 to -0.3 V after cathodic polarization.

Potentiometric and voltammetric measurements on FeS_2 electrodes are very useful in predicting the behavior of various pyrite samples during flotation at different pH levels in the presence of sodium dithionite or other reducing agents introduced to modify the surface properties. Comparison of the potentiometric and voltammetric measurements conducted on pyrites in solutions containing sodium dithionite suggests that in the presence of this additive the chemical composition of pyrite surfaces will be altered due to the reduction of FeS_2 oxidation products. Consequently, we also expect a substantial decrease in pyrite floatability when $\text{Na}_2\text{S}_2\text{O}_4$ is present.

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