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EFFECT OF FERRIC NITRATE AND PAX ON WET MILLING OF QUARTZ

The effect of selected inorganic and surface active grinding additives on wet ball milling of quartz has been studied as a function of pH and additive concentration. The results revealed that the effects of ferric nitrate and potassium amyl xanthate on wet ball milling of quartz were dependent on pH of the slurry and the concentrations of these additives. It was found that potassium amyl xanthate had marked beneficial effects on the production of –68 micron material at pH values between 5 and 8 at dosages between $5\cdot10^{-5}$ M and $5\cdot10^{-6}$ M.

In ferric nitrate solutions, at pH 6.5 a decrease in the production of fines was observed. However, there was a marginal improvement on grinding efficiency at pH 5.

INTRODUCTION

In mineral processing, the decrease in ore grade and accompanying increase in the degree of fineness of values in the ore increase the need for fine and ultrafine grinding. Consequently, the energy consumption is higher and grinding cost represents a relatively higher portion of the total beneficiation cost. Therefore, it seems to be an important task to improve the energy utilization inside the grinding mill. Although there is direct experimental verification of the advantageous effect of grinding additives, no sound scientific explanation has yet been suggested to predict the behaviour of ginding aids.

Boozer et al. (1963) examined the effect of surface active agents on rock deformation under compression loads. They claimed that when rock pores were saturated by oleic acid the ultimate strength of sandstone was decreased. This was attributed to the adsorption of surfactant molecules on rock grains even though no adsorption tests was carried out. Wang (1966) studied the effect of dodecylammonium chloride, $AlCl_3$ and Na_2CO_3 on the compressive strength of sandstone. They attributed these results to reduction in surface energy upon the adsorption of the additives on the surface of sandstone. Vutukuri (1972) reported that the tensile strength of quartzite

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immersed in 0.06% $AlCl_3$ solution to be 11.1% lower than that in water. The influence of dodecylammonium chloride on grinding efficiency of quartz in a porcelain ball mill was investigated by Ryncarz and Laskowski (1977). They argued that grindability was minimum at pzc. However, the researchers did not take into account possible surface contamination by aluminum species from porcelain mill. Mishra et al. (1982) studied the grinding characteristics of quartz in the presence of inorganic and organic additives. They reported that Fe^{3+} and Cu^{2+} ions as well as organic electrolytes such as dodecylamine hydrochloride and potassium amyl xanthate increased the production of fines.

Physical properties of the pulp such as pulp fluidity, state of dispersion and flocculation of the pulp, solid and medium density determine transport conditions of particles to grinding zone and have an effect on hydrodynamic behaviour of particles as well as on the grinding media and consequently on grinding efficiency (Somasundaran and Lin, 1972). Hockings et al (1965) studied the effect of pulp viscosity on the production rate of quartzite fines in a rod mill. They observed that an increase in the pulp viscosity from 1 to 1000 centipoise caused as much as a 50% decrease in the rate of grinding when the grinding was performed for 1 minute. The effect diminished with an increase in grinding time.

In this study, the effect of grinding aids such as ferric nitrate and potassium amyl xanthate on wet ball milling of quartz is discussed in terms of pH and chemical composition of solution.

EXPERIMENTAL

The grinding tests were carried out in a 0.4 m long and 0.538 m in diameter mild-steel ball mill equipped with twelve lifter bars. The grinding medium was alumina ceramic balls averaging 25 mm in diameter, with a total mass of 60.5 kg. The following operational variables were kept constant throughout the experimentation: mill filling: 0.4; mill speed: 80% of critical speed; grinding time: 60 min; percent solids: 70%; ionic strength: $3 \cdot 10^{-2}$ M (KNO_3).

The quartz sample used in the study was a mine dump sand sieved at 600 micron. Figure 1 illustrates the particle size distribution of the mill feed. During experiments, measurements of the pH and samples of the slurry in the circuit were taken at prescribed intervals over each 60 minute mill run. The pH was adjusted using 0.1M NaOH. The particle size distribution of the ground material was determined by the Malvern Master Sizer. In order to check the reproducibility of the particle size measurements, experimental runs were repeated several times under identical conditions. It was found that the variation in percent net production of minus 68 micron material between run to run was less than 2 percent.

The additive concentrations were $5 \cdot 10^{-3}$ M; $5 \cdot 10^{-5}$ M; and $5 \cdot 10^{-6}$ for both ferric nitrate and potassium amyl xanthate in the pH range 5 to 8.

RESULTS AND DISCUSSION

The results in Figure 2 shows that ferric nitrate can influence wet ball milling of quartz both beneficially and detrimentally at pH 5, depending on additive concentration. It can be seen that the ferric nitrate addition to the ball mill is beneficial at $5 \cdot 10^{-5}$ M.

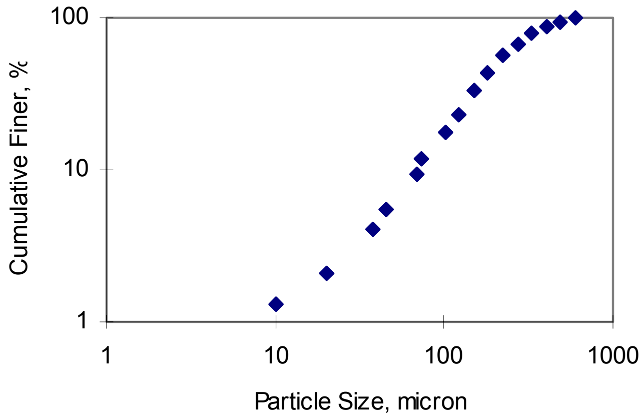

Fig. 1. Particle size distribution of the mill feed

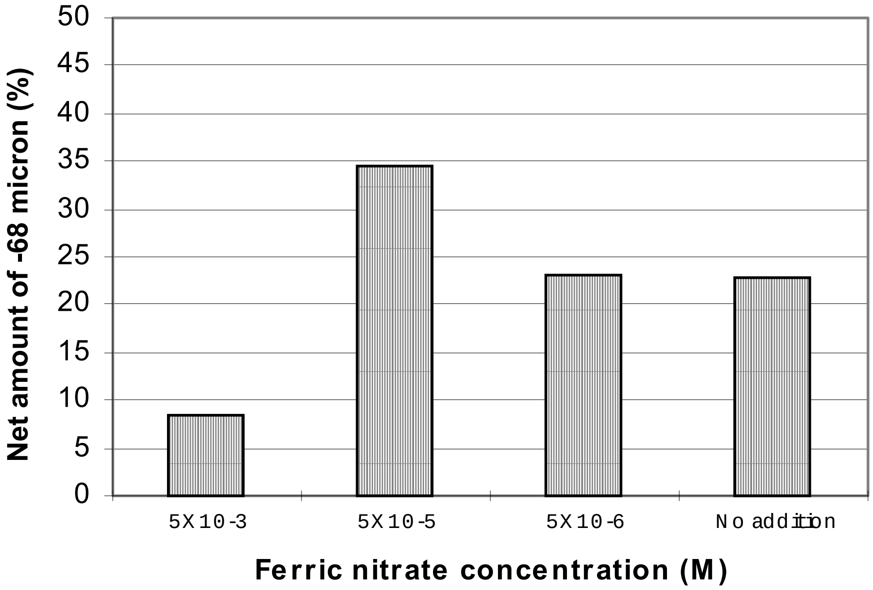

Fig. 2. The effect of ferric nitrate on the production of –68 micron material at pH 5

In Figure 3, the use of potassium amyl xanthate at $5\cdot10^{-6}$ M at pH 5 yielded the most favourable result in terms of the total amount of –68 micron material produced. For both additives concentration of $5\cdot10^{-3}$ M detrimental effects in the production of –68 micron material are observed.

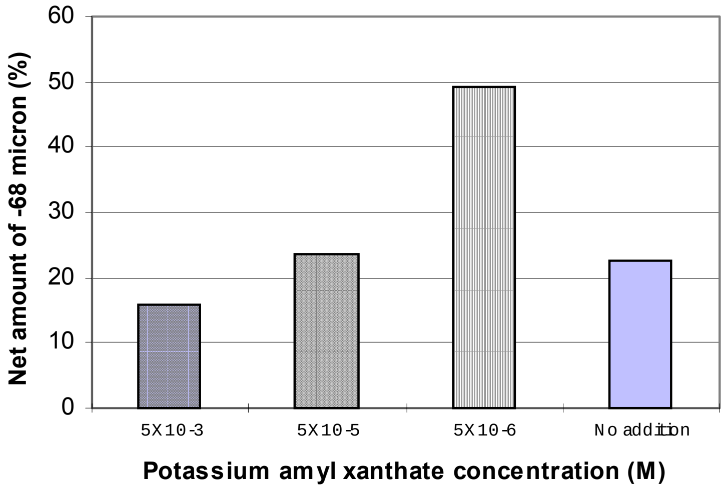

Fig. 3. The effect of PAX on the production of –68 micron material at pH 5

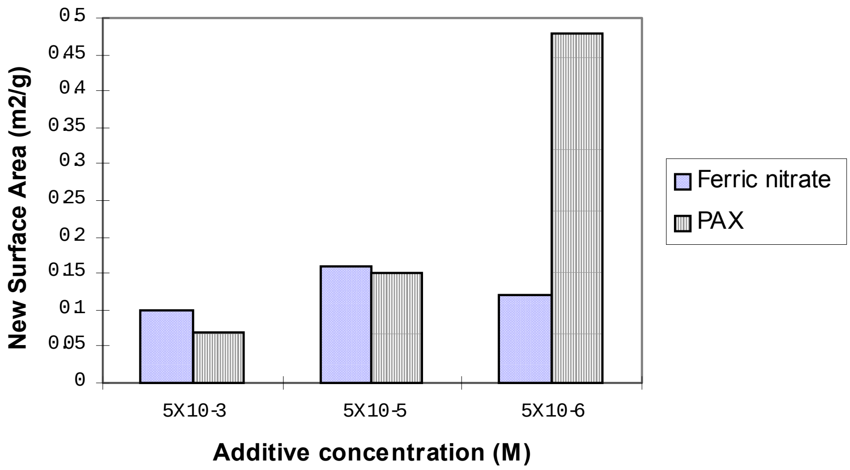

Fig. 4. The effect of additive type and concentration on the production of new surface at pH 5

The Malvern analyses of specific surface area were used in determining the new surface area produced which is an important parameter in determining grindability.

Figure 4 depicts new surface area produced per gram of new –68 micron material plotted against additive type and concentration.

It can be seen that the PAX containing slurry yielded the highest production of new surface area at a concentration of $5\cdot10^{-6}$ M. In general the effect of PAX on the production of new surface area increased as it is concentration was diminished, but was improved over the use of no additive at it is lowest concentration alone. The use of ferric nitrate at the intermediate concentration yielded a marginal improvement whereas detrimental effects were observed at the remaining two concentrations.

The net amount of new –68 micron material produced at different pH values in the presence of the two additives was analysed and compared. The resulting graphs are plotted in Figure 5.

It was observed that an increase in the pH of ferric nitrate containing slurry from 5 to 6.5 effected a lower production of fines, more specifically, a decrease from 7.5% to 4.5%. The implications are that the use of ferric nitrate at $5\cdot10^{-3}$ M at higher pH impacts adversely on the production of new –68 micron material.

The results for PAX, however, reveal the opposite effect. The PAX containing slurry, milled at pH 8 improved the production of fines as compared to the results obtained at the lower pH of 5. This suggests that increasing the pH of the pulp to a fixed value for the entire grinding period improved the production of new –68 micron material from 15% to about 19% at a concentration of $5\cdot10^{-3}$ M.

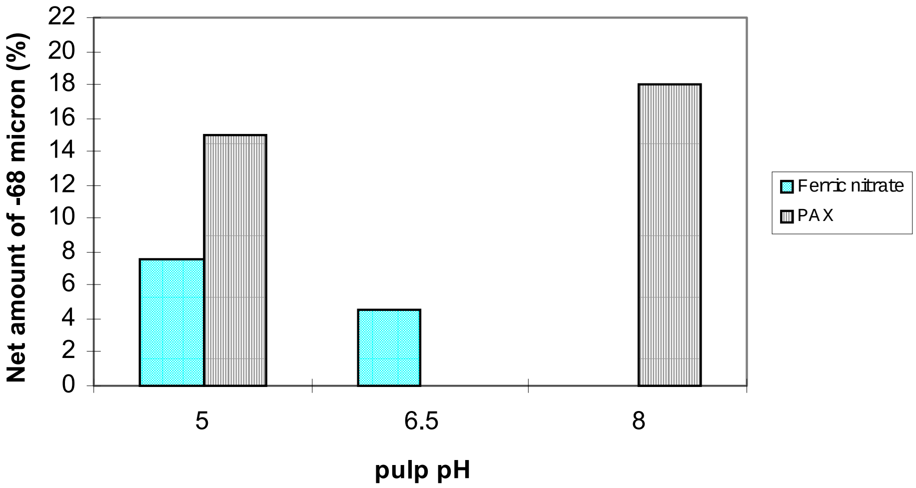

Fig.5. Effect of pH and additive type on fines production at $5\cdot10^{-3}$ M

In the presence of ferric ions, it has been reported by MacKenzie (1966) and El-Shall (1980) that the isoelectric point of quartz shifted from 2.1 to ~pH 6.5 showing that ferric ions can reverse the charge on quartz surface. This charge reversal consequently affects the flocculation properties and pulp fluidity. El-Shall and

Somasundaran (1984) studied flocculation behaviour of quartz suspensions in ferric nitrate solutions and found that pH values between 6 and 8 degree of flocculation of quartz particles reached maximum. They further confirmed by stability ratio calculations that at around pH 6.5 rapid coagulation occured. These studies are in very good aggreement with the results of the present study where the production of fines decreased when the pH of the pulp was altered from 5 to 6.5 which coincides with the maximum flocculation and i.e.p point. Mishra et al (1982) reported that in ferric nitrate solutions, the maximum production of fines took place when the concentration was $5.7 \cdot 10^{-4}$ M. However, in the present study in which the maximum grindability in ferric nitrate pulps was found to be $5 \cdot 10^{-5}$ M.

In their study, Mishra et al (1982) also found a peak point at about pH 8 for the production of –53 micron material at 10^{-4} M PAX. They argued that a change in the dosage did not have a significant effect at pH 9.

In the present investigation, the effect of potassium amyl xanthate on the fines production was more pronounced at pH 8. The production of –68 micron fraction fell at pH 5 and was found to be dependent on the concentration of PAX. As the dosage increased from 10^{-6} to 10^{-3} the fines production decreased considerably. This might be due to the change in the pulp fluidity and viscosity at higher concentrations which effect the fines prduction adversely. The other important point is the possible surface contamination by iron and aluminum species from the mill lining and grinding media which are known to reverse the zeta potential of quartz and shift the isoelectric point to around pH 6. In this study, the amount of iron and aluminum species transferred from the mill were not determined. However, if this argument is true at pH 5 positively charged quartz surface may interact with xanthate ions causing flocculation and deteriorating the grinding efficiency. At pH 8 quartz surface is negative, therefore the only explanation for the positive influence of xanthate ions might be the change in the fluidity of the pulp which needs to be verified.

CONCLUSION

Analysis of the grinding results in ferric nitrate and potassium amyl xanthate solutions from this study together with results of the studies conducted under similar conditions has led to the following conclusions:

i) the decrease in grinding rate on ferric nitrate additions ($5 \cdot 10^{-3}$ M) at pH 6.5 might be due to the change in pulp fluidity possibly caused by flocculation which coincides with the isoelectric point in the presence of Fe^{3+} species;

ii) a marginal improvement at pH 5 indicates the importance of the degree of coagulation on grinding in presence of ferric nitrate;

iii) beneficial effects were obtained on the addition of potassium amyl xanthate particularly at pH 8.

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Akdogan G., Johns D., Wp³yw azotanu ¿elaza (III) i ksantogenianu amylowo-potasowego na mielenie kwarcu na mokro. *Fizykochemiczne Problemy Mineralurgii*, 32, 165–171(w jêz. angielskim)

Badano wpływ wybranych związków nieorganicznych i powierzchniowo czynnych na mielenie kwarcu na mokro w młynie kulowym w zależności od pH i stężenia dodawanych odczynników. Badania wykazały, że wpływ azotanu żelaza (III) i ksantogenianu amylowo-potasowego zależy od pH układu. Stwierdzono, że pomiędzy pH 5 i 8 ksantogenian ma znaczny, pozytywny wpływ na powstawanie zawiesiny o uziarnieniu poniżej 68 μm przy stężeniu ksantogenianu pomiędzy $5 \cdot 10^{-5}$ i $5 \cdot 10^{-6}$ M. Zmniejszenie ilości drobnych ziarn zaobserwowano w roztworach azotanu żelaza przy pH = 6.5, ale zaobserwowano nieznaczny wzrost efektywności mielenia przy pH = 5.