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Effects of colloidal montmorillonite particles on froth flotation of graphite, galena and fluorite

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Abstract: The effect of colloidal montmorillonite (MMT) on froth flotation of graphite, galena and fluorite was investigated in this work. The results showed that the presence of sufficient amount of colloidal MMT particles in the mineral slurry would be detrimental of flotation by reducing the recovery of minerals. This observation was attributed to slime coating of MMT on the coarse valuable mineral particles and entrainment of MMT particles in the froth product together with water in the triangle froth zones. The former would reduce the recovery of the valuable minerals because of hydrophilic MMT coating. The latter would decrease the concentrate grade. The degree of slime coating depended on slurry pH, while the degree of entrainment was closely related to water recovery. It was also found that slime coating was a dominant factor in mineral flotation in acidic pH regions in the presence of colloidal MMT particles.

Keywords: montmorillonite, graphite, galena, fluorite, slime coating, entrainment.

Introduction

Montmorillonite (MMT) is a valued commercial commodity widely used as additives and fillers in plastics, rubber, adhesives, sealants, pharmaceuticals and paint (Wang et al., 2004; Chen et al., 2014; Yi et al., 2014). Also it is a commonly occurring gangue in mine tailings slurries, which occur in a variety of ore deposits, including porphyry copper ores, low grade nickel ores, platinum group metal deposits and diamond bearing ores (Aplan, 1997). In some rare cases, the content of MMT in such ores can reach as high as 80%. Processing of the soft sediment clay tailing wastes has attracted considerable attention in the mining industry in recent years owing to their environmental consequences. This is because some of the tailing slurries, containing as little as a few percent MMT, are difficult to dewater and require large areas for their disposal (Zhijun et al., 2013). Thickening and compression are significantly affected by the strength and nature of the interparticle forces and particle packing. Meanwhile

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the presence of MMT in flotation and comminution slurry causes a wide array of problems for the flotation operations, including higher reagent consumption, poorer selectivity and impeded flotation kinetics (Wang et al., 2015).

MMT belongs to the phylosilicate mineral family. Phylosilicate minerals are so named because they typically display a platy/leafy habit (Deer, W.A. et al., 1978). There are a large number of different types of phylosilicates in existence, with a variety of different properties, including: (*i*) a layer structure with one dimension in the nanometer range, the thickness of the1:1 (TO) layer is about 0.7 nm, and that of the 2:1 (TOT) layer is about 1 nm (Ndlovu et al., 2011); (*ii*) the anisotropy of the layers or particles; (*iii*) the existence of several types of surfaces: external basal (planar) and edge surfaces as well as internal (interlayer) surfaces. The faces and edges of phylosilicates carry different electrical charges, making these particles electrostatically anisotropic. The faces (or basal planes) tend to be negatively charged, while the edges carry a charge that alters from positive to negative as a function of pH (Teh et al., 2009). The surface can be modified by either adsorption, ion exchange or grafting.

The diverse characteristic of MMT can induce production of a large amount of colloidal particles and cause a wide range of different problems in mineral beneficiation circuits. They may either completely or partially attach to coarse hydrophobic mineral particles, preventing the particles from floating (Huynh et al., 2000). It is commonly referred by the generic term “slime coating”. The mechanism for such coating is widely believed to be the electrostatic attraction between oppositely charged mineral particles (Xu et al., 2003). In flotation, the presence of negatively-charged slime particles has no effect on positively-charged valuable minerals recovery, while the presence of positively-charged slimes almost completely depresses valuable minerals flotation (Arnold and Aplan, 1986). However, these results often neglect the effect of clay electrostatic anisotropy on the slime coating.

Another detrimental effect of colloidal particles on flotation is commonly referred by the generic term “entrainment”. Entrainment is the process by which particles enter the base of a flotation and are transferred up and out of the flotation cell suspended in the water between bubbles (Güler and Akdemir, 2012). Unlike true flotation, which is chemically selective, both gangue and valuable minerals alike can be recovered by entrainment (Cilek, 2009). One has stated that several origins of the gangue minerals could be recognized by the presence of the valuable minerals in the composite particles, unsuccessful preparation of particles surfaces and fine size fraction in the feed (Lynch et al., 1981). Entrainment is generally held to be damaging to product grade since the recovery of the more abundant gangue mineral reduces the quality of concentrate (Yianatos and Contreras, 2010). However, very little is known on the relative magnitude of the impact that both of two effect the flotation performance.

In this work, the effect of colloidal MMT on flotation of graphite, galena and fluorite was investigated. These three minerals used in this study are representatives natural hydrophobic, sulfide and oxide minerals. Montmorillonite is also the representative of clay mineral. The objective was to obtain more understandings of the impact that both of slime coating and entrainment have on flotation and quantify their relative magnitudes, in order to provide a fundamental theoretical guidance for overcoming the problem caused by colloidal clay particles.

Experimental

Materials

The original MMT used in the present study was obtained from Sanding Technology Co., Ltd, Zhejiang province, China. A common method for obtaining purified colloidal MMT is fractionation by sedimentation after removal of carbonates, oxides and organic materials as well as smashed by the ultrasonic grinder. Figure 1 gives the X-ray diffraction (XRD) image of the sample, showing that the colloidal MMT particles were very high grade and contained negligible impurities. Diffraction patterns were verified according to JCPDS PDF pattern no. 13-0135 for montmorillonite. A Malvern Zetasizer Zeta-Nano was used to determine the zeta potential of graphite, galena, fluorite and MMT particles in aqueous solutions.

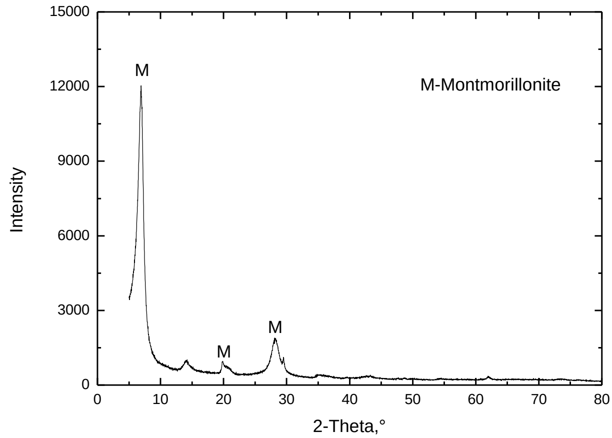

Fig. 1. XRD trace of the colloidal MMT

The samples of graphite, galena and fluorite were obtained from Sanchaya (Yichang, China), Fankou (Guangdong, China) and Yunfeng (Jiangxi, China), respectively. The particle size distribution of the samples of graphite, galena and fluorite is illustrated in Fig. 2. All of them have more than 95% purity analyzed by XRD. Kerosene, ethyl xanthate and oleate (as collectors), MIBC (as a frother); sodium hydroxide (NaOH) and hydrochloric acid (HCl) (as pH regulators) were from the Sinopharm Chemical Reagent Co., Ltd (China). All of them were of analytical purity.

The water used in this work was produced using a Millipore Milli-Q Direct 8/16 water purification system with 18.2 MΩ.

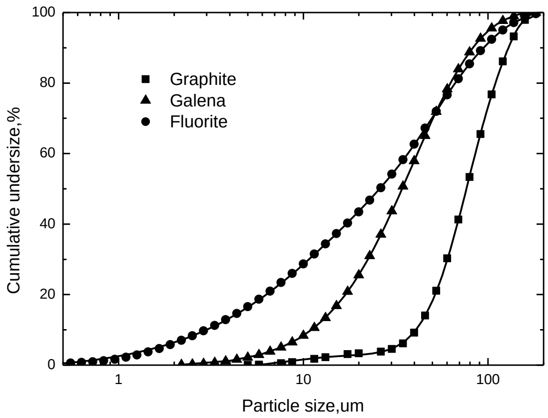

Fig. 2. Particle size distribution of graphite, galena and fluorite

Measurements

The particle size distribution of the flotation tailings was estimated by light scattering, using a Malvern Mastersizer 2000. A sample was placed in an ultrasonic bath for 1 min before measurement. The particle size distribution of the colloidal MMT sample is illustrated in Fig. 3, showing that the particle size at 50% cumulative undersize (D_{50}) and the particle size at 85% cumulative undersize (D_{85}) were of 0.25 μm and 0.5 μm, respectively.

SEM studies of graphite sample coated with colloidal MMT were done using a JSM-5610LV scanning electron microscope (Japan). The coated graphite sample was received from the tailings in graphite flotation.

A Malvern Zetasizer Zeta-Nano was used to determine the zeta potential of the MMT in aqueous solutions. This instrument works with the technique of laser Doppler electrophoresis. 0.01 g MMT sample was mixed with 200 cm^3 water to prepare a suspension of 0.05 g/dm^3 solid concentration, while a given electrolyte solution was added. The mixing was performed on a stirrer at 10000 r/min for 5 min. Then, the suspension was poured into the measuring cell of zeta meter. The temperature was kept at 25±1 ℃ throughout the measurement. The average value from 10 individual measurements was reported in this paper.

Viscosity measurements of the MMT suspension were performed using an AR-2000 advanced rheometer (TA Instruments, America). The measurements were carried out at a shear rate of 700 s^{-1} and temperature of 25 ℃. Each viscosity measurement was repeated 30 times and the results are reported as the arithmetic average.

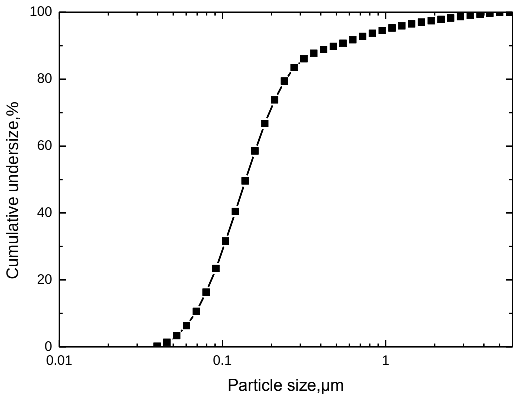

Fig. 3. Particle size distribution of colloidal MMT samples

Flotation

The composition of the flotation slurry was a mixture of pure minerals and colloidal MMT at a variety of proportion, ranging from 0% to 40% pure minerals (60% to 0% MMT). The ground samples were transferred into a batch cell and made up to a volume of circa 80 cm^3 for each test to give a flotation slurry density of 5 wt% solids. An impeller speed of 2500 r/min was applied to all flotation tests. Conditioning time for collector and frother was 1 min. At this stage, the slurry was aerated under the natural aeration conditions, i.e., the valve was fully open in all the cases.

Flotation tests were conducted on a RK/FGC laboratory type flotation machine. The froth was scraped every 2 s for the first minute of flotation and every 5 s thereafter. A Sartorius P8-10 laboratory pH meter was used to measure the pH. Eppendorf Research plus Pipette were used to add acid/base to set and maintain pH. Afterwards chemical analyses were performed for C, Pb and CaF_2 to calculate the grade and recovery values.

Results and discussion

Effect of slime coating on flotation

The recovery of graphite at a variety of MMT proportions in mineral slurry is presented in Fig. 4. All tests were performed at pH 10. The result shows that when the suspension consisted entirely of graphite (representing a baseline flotation condition), graphite exhibited a high level of floatability, achieving over 92.7% recovery. This result is entirely expected as graphite is known to be a naturally floatable mineral. As the proportion of MMT in the slurry increased, the recovery of graphite decreased dramatically. When the slurry phase consisted of 60% MMT, only 40% graphite recovery was achieved. The slurry chemical conditions remained constant among tests. Therefore, the most likely explanation is that the detrimental flotation performance results from the effect of slime coating caused by the increased proportion of colloidal MMT.

The same results were also obtained for galena and fluorite flotation, as shown in Fig. 4, in which the recovery as the function of the MMT proportion is illustrated. The effect on graphite and fluorite flotation well coincided in different MMT proportions in the slurry, indicating that the MMT affected the recovery of galena and fluorite in the similar way. As the proportion of MMT in the slurry increased, the recovery of galena and fluorite decreased dramatically. In other words the MMT has a negative impact on different minerals from the sulfide and oxide ore.

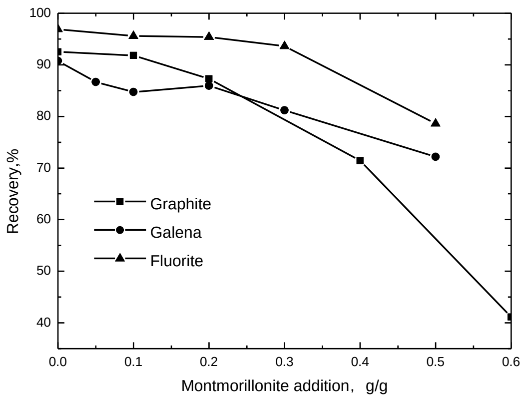

Fig. 4. Effect of various MMT additions in the slurry on the recovery of three minerals

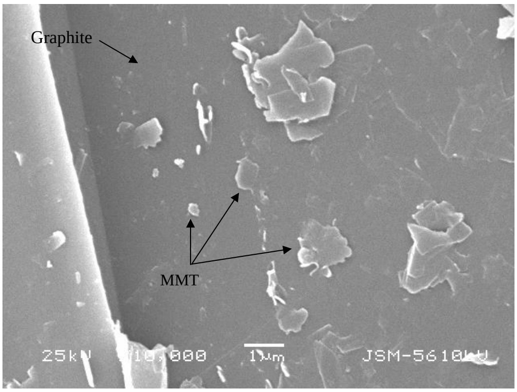

Fig. 5. SEM of graphite coated with colloidal MMT

The SEM study was undertaken to investigate the possible interaction of colloidal MMT with the graphite surfaces (Fig. 5). Figure 5 shows that the relatively coarse graphite mineral particles in the tailings become either completely or partially coated by a layer of hydrophilic montmorillonite, thereby rendering the valuable mineral hydrophilic and inhibiting collector adsorption.

Once the floatability of graphite as a function of the MMT proportion in the slurry was established, the tests were performed to evaluate the effect of pH value in the presence of stable quantities of MMT (20% proportion). The results are presented in Fig. 6. The recovery decreased monotonously with decreasing the pH value. However, the natural floatability of graphite was not affected by pH and remained relatively high in the pH range 2–10. Therefore, it is likely that the noticeable decrease in flotation performance results from the effect pH on the slime coating.

The similar results were obtained for galena and fluorite flotation, as shown in Figs. 7 and 8. The recovery of galena and fluorite decreased with decreasing the pH value. This observation is not beyond expectation as the slime coating is largely governed by the electrostatic interactions. With increasing slurry pH, the pH-dependent surface potential is anticipated to be increasingly negative, resulting in a stronger electrostatic repulsive force between the minerals and MMT clay particles, and hence a reduced degree of slime coating.

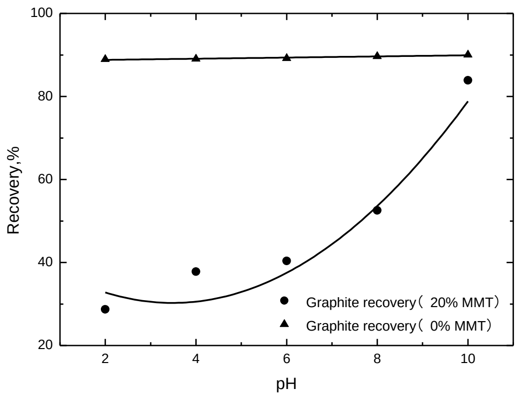

Fig. 6. Effect of pH on flotation of mixed mineral and pure graphite

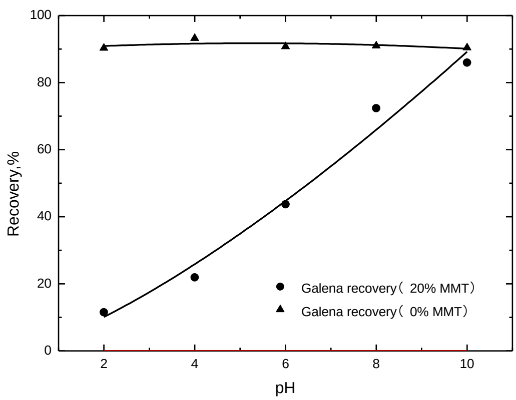

Fig. 7. Effect of pH on flotation of mixed mineral and pure galena

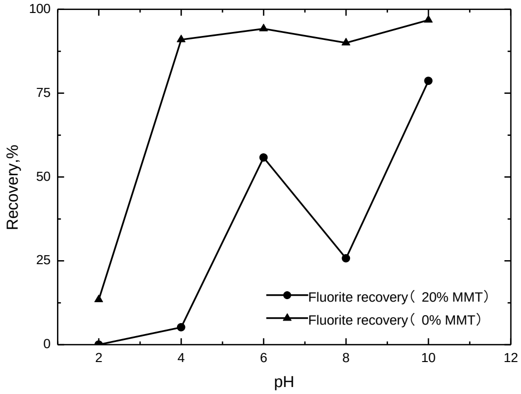

Fig. 8. Effect of pH on flotation of mixed mineral and pure fluorite

The graphite flotation recovery as a function of flotation time performed at natural slurry pH 6 is shown in Fig. 9. It is evident that graphite exhibited a fast flotation kinetics. Under the current flotation condition, the cumulative recovery attained 90% at the flotation time of 2.5 min. It should be noted that in the current study, we were not concerned about the optimal flotation conditions, as the objective was to further explore the role of MMT in graphite flotation. The addition of MMT up to 20% by weight of total solids showed a significant reduction of graphite flotation kinetics. It appears that the MMT clay has a stronger affinity to graphite, resulting in a severe slime coating of MMT clay particles on graphite surface. The presence of hydrophilic MMT clay particles on graphite surface hinders air bubble–graphite attachment and, consequently, causes a significant reduction in graphite flotation recovery.

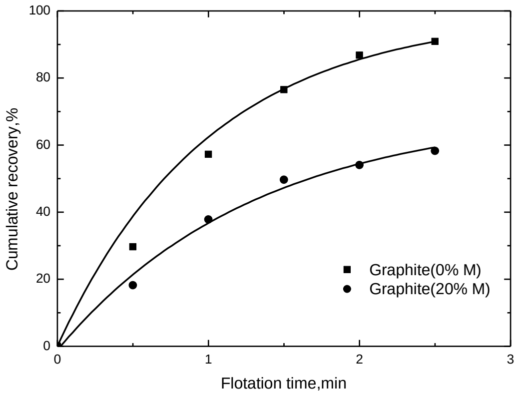

Fig. 9. Effect of MMT addition on graphite recovery in dependence on flotation time

The same result were also obtained for galena and fluorite flotation, as shown in Figs. 10 and 11. The flotation kinetics of galena and fluorite decreased seriously with addition of MMT up to 20%. In other words, the decrease of flotation kinetics was mainly caused by the slime coating when the MMT clay was added to the flotation slurry, which corresponded well with the results for coal flotation (Arnold and Aplan, 1986).

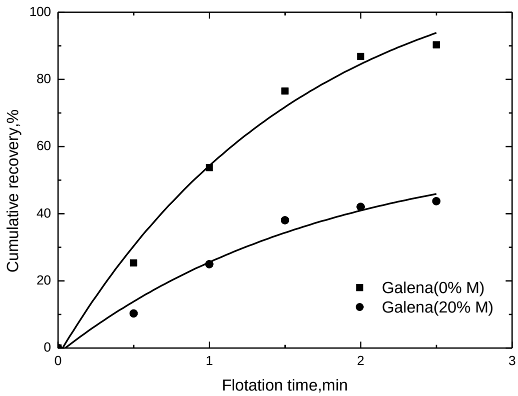

Fig. 10. Effect of MMT addition on galena recovery in dependence on flotation time

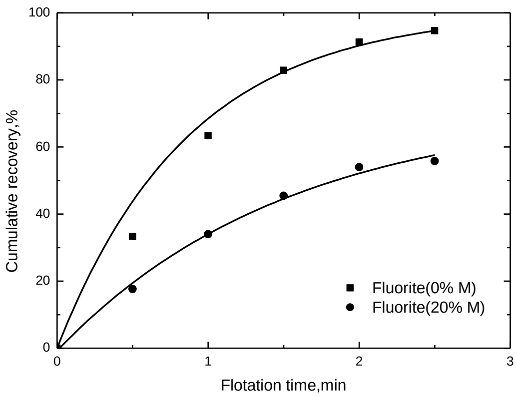

Fig. 11. Effect of MMT addition on fluorite recovery in dependence on flotation time

By analogy with chemical kinetics, the equation representing flotation kinetics may be expressed as:

$$\frac{dC}{dt} = -kC^n \tag{1}$$

where C is the concentration of solids, t is the flotation time, n is the order of the process, and k is the flotation rate constant.

The integration for obtaining the constant of flotation rate is described in a previous work (De Castro and de Hoces, 1996). The analysis results are summarized in Table 1. The results show that the flotation rate constant (k), decreases steadily as the proportion of MMT in the gangue phase increases. The proportion of valuable minerals in the flotation pulp, as well as the pulp chemical conditions, remains constant in the tests. Therefore, the most likely explanation is that the detrimental flotation performance results from the increased proportion of MMT that influences the pulp characteristics.

Table 1. Flotation kinetic analysis, as a function of MMT addition.

	n = 1		n = 2		n = non-integral	
	k_1 (min^{-1})	r^2	C_0k_2 (min^{-1})	r^2	k_q (min^{-1})	r^2
Graphite (0% M)	1.0055	0.9922	4.0152	0.8832	1.7936	0.9603
Graphite (20% M)	0.3605	0.9519	0.5843	0.9821	1.5237	0.9966
Fluorite (0% M)	1.2286	0.9914	6.9406	0.8368	1.9355	0.9735
Fluorite (20% M)	0.3423	0.9672	0.5588	0.9697	1.9047	0.8916
Galena (0% M)	2.0334	0.9828	3.8428	0.8920	2.0334	0.9511
Galena (20% M)	1.0018	0.9662	0.3422	0.9501	1.9514	0.9663

The results obtained for zeta potential values of four minerals measured against pH are plotted in Fig. 12. The graphite and fluorite had the isoelectric point, while MMT and galena were found to have negative zeta potential in the all pH range (2 to 10). Nonetheless, this trend was similar to typical observations in previous studies (Delgado et al., 1985; Kelebek and Smith, 1989; Oberndorfer and Dobiáš, 1989; Wakamatsu and Numata, 1991; Ndlovu et al., 2014). MMT was stable, ranging in zeta potential value from –20 to –35 mV. Galena was always negatively charged, but it appeared less negative at about pH 5. This observation is not beyond expectation as the slime coating is largely governed by the electrostatic interactions but taking into account the clay electrostatic anisotropy. A noticeable increase in the negative zeta potential at pH > 5 is thought to be related to the point of zero charge (pzc) of the clay edge at about neutral pH. In other word, the faces (or basal planes) of MMT tend to be negatively charged, while the edges carry a charge that alters from positive to negative as a function of pH. The result are of a great interest in analyzing the effect of pH value on graphite, galena and fluorite flotation. In this phenomenon, relatively coarse valuable mineral particles become either completely or partially coated by a layer of hydrophilic MMT (at pH < 5), thereby rendering the valuable mineral hydrophilic and inhibiting collector adsorption, which is in a good agreement with the flotation results. As illustrated in Fig. 13, it is concluded that the degree of slime coating can also simply be described as the function of pH. With decreasing slurry pH, electrostatic repulsive force between the minerals and MMT clay particles decreased, even reversed to attractive force. It means that the degree of slime coatings increased with the slurry pH decrease. Specifically, it is a dominant factor in mineral flotation in the acidic range.

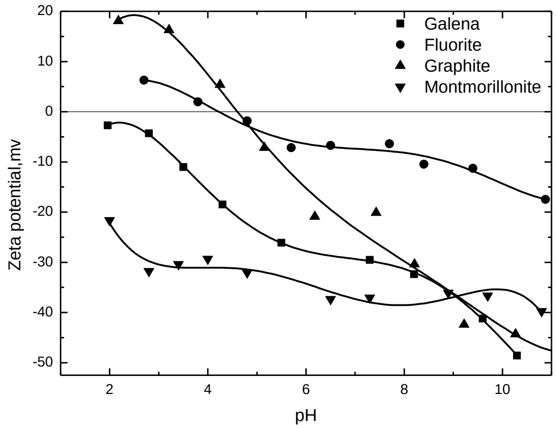

Fig. 12. Zeta potentials distributions of MMT and three minerals containing 0.1 mM KCl as a function of pH

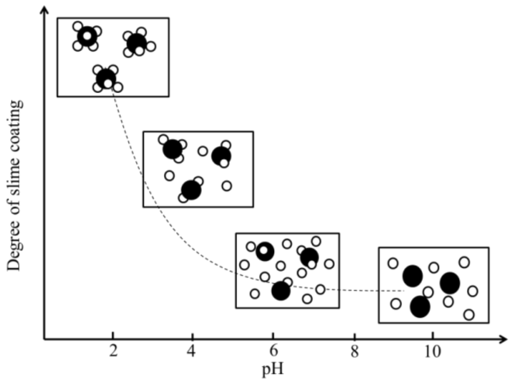

Fig. 13. Diagram of slime coating degree as a function of pH

Entrainment MMT particles

Entrainment is the primary mechanism for the recovery of fully liberated and dispersed gangue particles. Finely sized liberated particles are carried out in thin water layer surrounding air bubble, and recovered in concentrate launder regardless of the degree of hydrophobicity of particles. As it is known entrainment is a non-selective sub-process. The amount of the colloidal mineral particles (valuable or gangue) to be recovered by entrainment is proportional to their specific gravity, shape and amount in the feed. The grade of graphite at a variety of MMT proportion in the slurry phase is presented in Fig. 14. All tests were performed at pH 10. As the proportion of MMT in the slurry increased, the recovery of three minerals decreased dramatically. In other word, the colloidal MMT particles were also recovered by flotation resulting in the decrease of three minerals recovery.

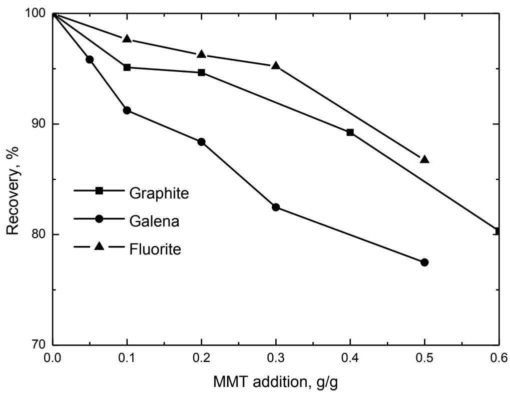

Fig. 14. Effect of various MMT additions in the slurry on the recovery of three minerals

Many authors have found that the recovery of gangue (R_g) increases with the recovery of water (R_w) and that for a flotation slurry with a wide distribution of particle sizes the R_g vs. R_w curves are parabolic. For narrow wide particle sizes ranges straight line are often obtained. Figure 15 shows that there is a relationship between the recovery of gangue and recovery of water. The degree of entrainment is almost stable, mainly depending on the water recovery (Zheng et al., 2006).

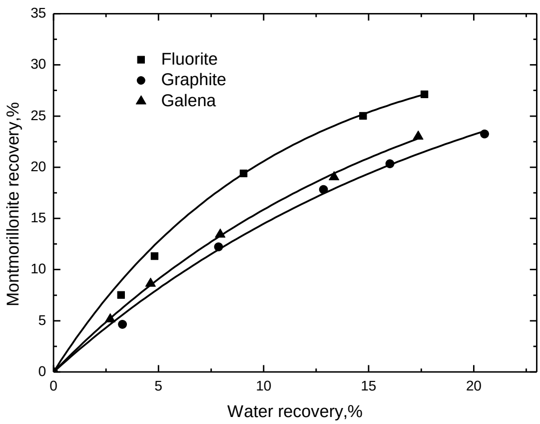

Fig. 15. Effect of water recovery on MMT recovery in three minerals flotation

Effect of pulp viscosity on flotation

According to some researchers in suspensions of colloidal particles (less than 1μm) the interface separating the phases is very large and it makes that the stability and rheology can be strongly affected by non-hydrodynamic electrical double-layers forces which interact with the prevailing chemistry and state of shear (Yang et al., 2001; He et al., 2004). This type of suspensions produces yield stress and steady shear viscosity values higher than the graphite, galena and fluorite particles. Changes in viscosity affect the hydrodynamics within flotation cells, and therefore affect various sub-processes necessary for efficient flotation, such as gas dispersion, particle suspension, bubble–particle collision, attachment and detachment. For example, the increasing presence of swelling clays in the flotation pulp has a strong effect on the flotation pulp rheology, with the suspension becoming increasingly viscous and deviating from Newtonian behaviour. Montmorillonite was found to have significant effect on the copper recovery. The final recovery decreased from 90 to about 80% in the presence of 15% montmorillonite (Farrokhpay et al., 2016).

The viscosity of aqueous MMT suspension was investigated in this work. Figure 16 shows the viscosity of MMT suspensions as a function of mass fraction. As the proportion of MMT in the suspension increases, the viscosity starts to increase. This is expected, as MMT particles have an irregular (platy) shape, with larger effective particle volume fraction, which leads to more viscous slurries. The presence of high pulp viscosity has a negative impact on the slurry rheology, with the suspension becoming increasingly viscous and deviating from Newtonian behaviour. High pulp viscosity causes a negative impact on flotation, which increased pulp viscosity resulted in a decreased probability of particle/bubble attachment and velocity of rising bubble. This phenomenon corresponded well with the results for flotation experiments in Figs. 4 and 14. As the proportion of MMT in the slurry increased, the recovery of graphite, galena and fluorite decreased dramatically. The resulting retardation in floatability of graphite, galena and fluorite can be attributed to reduction in the probability of particle/bubble collision through turbulence damping.

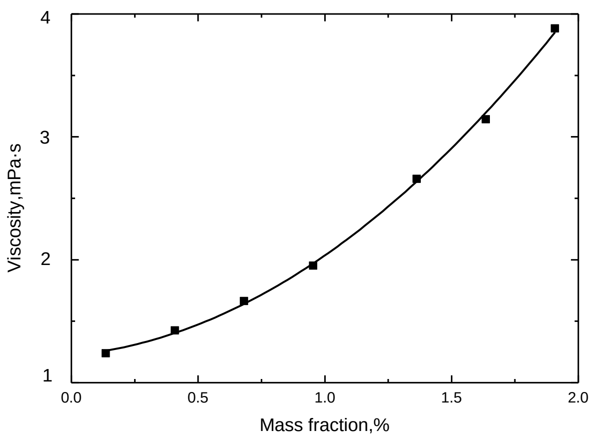

Fig. 16. Effect of mass fraction on viscosity of MMT suspensions

Conclusions

It was found that MMT featured a strong negative effect on floatability of graphite, galena and fluorite. The overall flotation recovery decreased significantly with increasing MMT addition. Slime coating and water carryover of colloidal clays were responsible for deteriorating recovery of minerals. The degree of slime coating could simply be described as the function of the pH, but the degree of entrainment was relative stable. Also, slime coating was a dominant factor in colloidal particle flotation in acidic pH regions (below pH 6). The presence of MMT in the suspension greatly increased the viscosity of the mixed mineral pulp, which might also be attributed to the retardation of floatability of three minerals because of reducing the probability of particle/bubble collision through turbulence damping.

Acknowledgments

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