DOSING AND MIXING PROPORTION OF COAGULANT AND FLOCCULANT IN THE ASSORTMENT OF ARUFU LEAD ORE COLLOIDS FOR WATER CLARIFICATION

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Abstract
Generally, effluents are highly undesirable and unsafe to use. Treatment of tailing effluents requires the removal of the suspended solids for purification and possible re-usage in the mill or in other processes, was the main focus in this work. The research examines the possibility of agglomerating Arufu galena colloids and determination of the appropriate mixing and dosing of the coagulant and flocculant which ranges from 0-10ml of 2.5ml interval for both. The work revealed that 5% lime solution with 1% starch solution dosage and mixing ratio of 1:1 produced a well-agglomerated suspension of the Arufu galena colloids as the particles settled after 5 minutes without leaving suspended solids in the supernatant liquid. Furthermore, the initial pH of the suspension was controlled from highly acidic level (pH of 4) to alkalinity state (pH of 12).

Keywords: Colloids, coagulant, flocculant, dosing, mixing and clarification

INTRODUCTION

Clarification of water containing colloids particles arising from mineral processing operation is a serious task for settling rate and overdosing can be a serious problem, which may create an established suspension that will be extremely difficult to separate (Damisa and Akindele, 2015). In all mining operations, solids and liquids must be separated which can be facilitated by flocculants in the thickening of froth flotation concentration and clarification steps (Prakash *et al*., 2014). Mostly in mineral processing, the suspended fines are impurities arising from crushing and grinding. They are separated as the solid phase rather than the mineral of interest, which remains in solution, coal being an exception. The mining industries requiring flocculants include coal, iron ore, bauxite and uranium (Sresty *et al*., 1978; Tripathy and De, 2006). Both cationic and anionic flocculants are used including some natural polymers. Cationic polymers are of quaternary ammonium type, for example poly (DADMAC) or polyamine especially in the recovery of coal (Wang and Muhammed, 1999; Crittenden *et al.,* 2005). The principal anionic synthetic flocculants are poly (acrylamide-acrylate) copolymers although nonionic polyacrylamide is also utilized. Among natural polymers used are starch, guar gum, animal glue, lignin (sulfonate) etc (Tripathy and De, 2006).

Most mineral separation processes involve the use of substantial quantities of water and the final concentrate has to be separated from a pulp in which the water-solids ratio may be high (Wills, 2006). Water is used to process about 80-90% of the tonnage of minerals and coal (Dahlstrom, 2003). A small percentage of minerals and coal is, of course, processed dry, which implied that coal is crushed and screened in dry form. Industrial minerals such as diatomite and bentonite can also be processed dry. High water-solid ratio inevitably leads to suspension whose sedimentation can be a serious problem in mineral processing operations (Damisa and Akindele, 2015). Accordingly, dewatering is a major cost in mineral processing, probably exceeded only by the cost of comminution (grinding to be specific), flotation and endothermic reactions (Dahlstrom, 2003). Suspended solids may be in the form of colloids.

Colloidal Particles

Colloidal particles are particles that fall into a general category of very fine material that has electrostatic surface charges. In general, most colloidal materials have negative charge. Particles with like charges tend to repel each other, preventing the forming of coagulated particles. These characteristics cause the colloidal particles to remain in solution.

Destabilizing colloidal material to allow coagulation and settlement to occur is achieved by adding reagents that develop positive charges. Positively charged ions in the solution act to destabilize the colloidal matter and allow settlement of coagulated material to occur (Auckland, 2004). Coagulation and flocculation occur in successive steps intended to overcome the forces stabilizing the suspended particles, allowing particle collision and growth of flocs, which then can be settled and removed (by sedimentation) or filtered out of the water (Tzoupanos and Zouboulis, 2008). Often the terms coagulation and flocculation are used synonymously in spite of a subtle difference between the two (Tripathy and De, 2006). If destabilization is induced through charge neutralization by addition of inorganic chemicals, the process is called coagulation. On the other hand, the process of forming larger agglomerates of particles in suspension or of small agglomerates already formed as a result of coagulation through high molecular weight polymeric materials is called flocculation (Tripathy and De, 2006).

The combination of coagulation and flocculation processes is known as agglomeration, which means bringing together very small particles to form bigger ones. In practice, coagulation and flocculation processes are combined for greater effectiveness in the agglomeration of fine particles (Pillai, 2014). This research therefore seeks to determine the dosing and mixing ratio of combination of lime as a coagulant and starch as a flocculant for galena colloids. Figure 1show how surface charges of particles are neutralized and microflocs are formed, although flocculant are often added to bring the microflocs particle together.

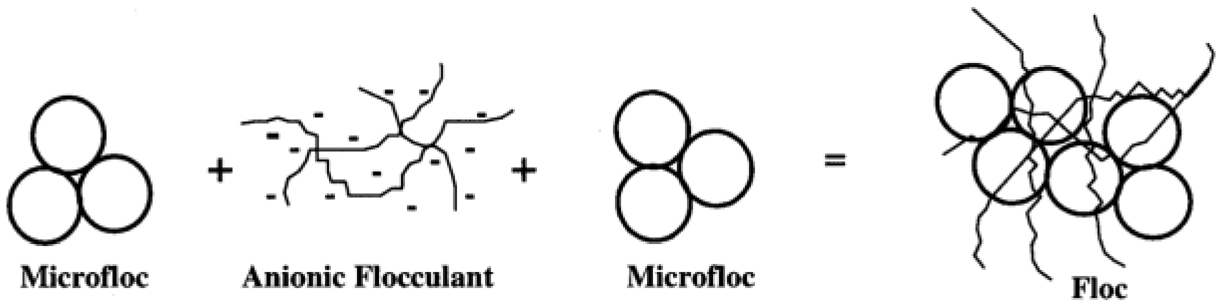

Figure 1: Flocculation of coagulated fine clay particles

Solid-Liquid Separation

According to Silverblatt and Easton (2002), solid-liquid separation is an important, and many times critical, step in a mineral processing plant. There are many processes in the minerals industry that require some type of solid-liquid separation. Some examples of typical separations are as follows:

a. Operation of a seven to eight stage counter-current decantation (CCD) washing /thickening circuit to effect high recovery of the caustic and aluminium values in an alumina plant. This must be accomplished in a closed-circuit system with highly scaling slurries at starting temperatures of 100°C

b Operation of a vacuum filter to produce an iron concentrate cake with a moisture content required for pelletizing in order to minimize bentonite addition and maximize pellet quality. A good quality filtrate must be maintained to prevent abrasion of the internal parts of the filter.

c. Operation of a closed water system in a coal washing plant so that reclaimed water containing less than one percent suspended solids is produced for reuse to maintain beneficiation efficiency. Refuse solids must also be thickened so that they can be effectively dewatered to meet environmental protection agency (EPA) requirements.

d. Recovery of phosphoric acid values from slurry containing gypsum and other gangue solids at an elevated temperature. This must be done while minimizing wash water usage, maximizing P_20_5 recovery and minimizing scale problems.

Classification of Solid in Suspension

Solids in suspension may be classified into three classes (Abouzeid, 1991).

i. *Macroscopic Particles*: The particles are large enough to settle according to Stoke's Law of settling or modifications of it. This type of particles settles fairly rapidly. They settle rapidly because the specific charge density on the surface of the particles is negligible compared with other types of forces acting on the particles such as gravitational force.

ii. *Microscopic Particles:* These are small particles with low specific surface charge density but having such a slow settling rate that enormous settling tanks (thickeners) would be required.

iii. *Colloidal Particles:* The material in this case is extremely fine and carries a high surface charge density, positive, negative or both. The mutual repulsion between particles of similar charges prevents particles from actually adhering to each other. This type of suspension causes serious problems in settling, due to its high stable conditions. Figure 2 provides range of particle sizes in microns that expected to dissolve or in suspension as indicated on the scale.

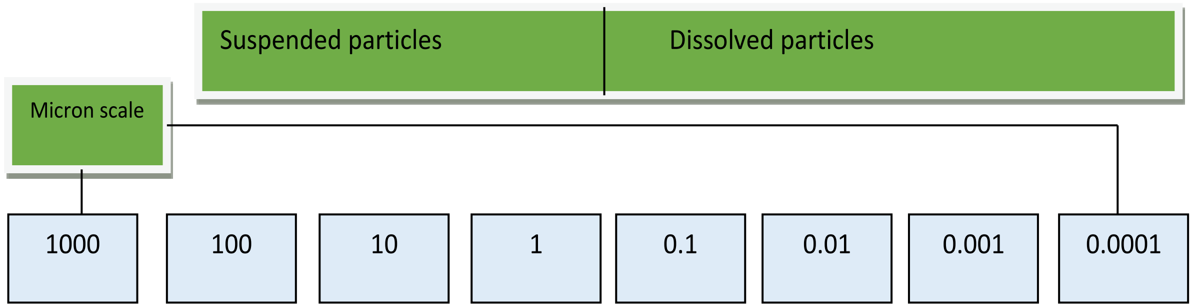

Figure 2: Particles size spectrum (Shemanski, 2014)

Effects of dosing and mixing conditions

The degree of flocculation achieved can be markedly affected by dosing and mixing conditions. It has been found that for high solids concentrations and relatively low polymer doses, flocculation occurs rapidly, but the flocs are not stable and can be broken at moderate stirring rates (Gregory and Li, 1994). By reducing the rate of stirring shortly after polymer dosing, floc size (and settling rate) can be held at plateau levels, without subsequent decline, which, however, is difficult to achieve in practice because of the precise control required (Gregory and Li, 1994). It has been suggested that optimum flocculation occurs when half the area of solid is covered with polyelectrolytes (Holt *et al.,* 2002). At higher concentration, the degree of flocculation decreases and the particles may be completely covered by the absorbed polymer layer (Hogg, 1992). Thus, overdosing can be a serious mistake in that it may create a well-established suspension that is extremely difficult to separate (Tripathy and De, 2006). But, in principle, a substantial degree of flocculation can be obtained with much lower polymer dosage than is usually required. Hydrodynamic factors arising from mechanical agitation play a significant role in flocculant adsorption (Gregory, 1988). Vigorous agitation of flocculating suspension causes floc breakage and the exposure of fresh surfaces to polymer adsorption thereby increasing adsorption capacity (Ayazi and Titchener-Hooker, 1993). At the same time, however, increased agitation leads to the production of smaller flocs, indicating that enhanced adsorption does not compensate for increased floc breakage. In fact, the general rule seems that, provided adsorption does occur, the actual amount adsorbed varies inversely with the extent of flocculation (Moudgil *et al.,* 1994).

MATERIALS AND METHODS

The materials used include:

- Ø Twenty kilogramme of Lead ore obtained from Arufu, Nasarawa State.
- Ø Pulverizer
- Ø Laboratory jaw crusher
- Ø Jones riffle
- Ø 5 No of 250 ml beakers
- Ø 5 No of 500 ml beakers
- Ø 25 No of 250 ml graduated cylinders
- Ø 5 No of 1 ml pipettes
- Ø 5 No of 5 ml pipettes
- Ø Lime powder
- Ø Starch powder and
- Ø pH indicator.

The following techniques and procedure were employed;

1.*Sample Collection*

In order to have a true fraction representation of lead ore from the bulk, samples were collected from different points at different depth ranges using the random sampling techniques within the study area.

2.*Sample Preparation*

The samples collected in lumps size were broken manually with sledge hammer to provide the required size acceptable to laboratory jaw crusher. The sample after initial pulverization was subjected to coning and quartering and riffled to obtain a representative sample (Akindele *et al*., 2017).

3.*Sieve Analysis*

Sieve sizes were arranged in a stack with the coarsest sieve on the top and the finest at the bottom. A tight-fitting pan was placed below the bottom sieve to receive the final undersize and a lid was placed on top of the coarsest sieve to prevent escape of the sample (Obasi *et al*., 2014). The sieved sample (63 um) of 50 g was introduced into 500 ml of distilled water to form a suspension of finely ground galena.

4.*Preparation of Solutions*

(i) *Lime solution*: A 5 % lime solution was prepared by adding 25g of freshly prepared chemically pure lime and this was stirred until almost all the solid was dissolved. The solution was filtered and made up to 500 ml.

(ii) *Starch solution:* measured 5 g of the dry starch mixed into a thin slurry with a small quantity of distilled water. Boiling distilled water was added to the slurry, completing the volume to about 500ml and stirred, giving 1 % starch solution. This was allowed to cool.

5.*Control test*

The suspension of finely ground galena 1/6 filled two bottles with exact measurement. Initially, test was carried out on the galena slurry by adding coagulants (lime) and flocculant (starch) as sample under investigation (1st bottle) and another galena slurry of the same volume as control but without the coagulant and flocculant (2nd bottle) and both were observed for three days.

6.*Procedure*

The suspension was thoroughly shaken and 25ml of the suspension was transferred into a 250ml graduated cylinder and diluted to 100ml using distilled water and then the appropriate amount of lime solution was added. The suspension was then agitated and the required amount of starch solution was added. This was diluted to 150ml and mixed thoroughly by placing stopper over the top of the cylinder and inverting it five times. Care was taken to avoid vigorous shaking or stirring. The suspension (in each case) was allowed to settle, thereafter, the depth of the settled suspension at the bottom of the cylinder after 5 minutes of settling and also the state of this top liquid (clear, turbid, or highly turbid) were recorded.

RESULTS AND DISCUSSION

Control Test

It was observed that even after three (3) days, the control was still turbid while the experiment (galena slurry and coagulants/flocculant) had clear liquid on top of settled particles after about twenty minutes (Figure 3a & 3b).

Figure 3a: Control test after 3 days

Figure 3b: Set of settling test cylinders

Presentation of Results

Table 1: Result of investigated parameters: pH readings, degree of turbidity and height of settled particles

Vol. of settled particles / State of liquid		Volume of starch (ml)				
		0	**2.5**	**5**	**7.5**	**10**
Vol. Of lime Solution(ml)	**0**	1.5/highly turbid (pH =4.0)	2.5/highly turbid (pH =5.0)	3.8/highly turbid (pH =5.0)	4.3/highly turbid (pH =6.0)	5.2/Highly turbid (pH =6.0)
	2.5	4.5/turbid (pH =6.0)	5.6/turbid (pH =6.0)	6.3/turbid (pH =7.0)	6.9/turbid (pH =6.0)	7.5/turbid (pH =7.0)
	5	15.0/slightly turbid (pH =12.0)	16.4/slightly turbid (pH =12.0)	17.6/slightly turbid (pH =14.0)	18.3/slightly turbid (pH =11.0)	19.2/slightly turbid (pH =14.0)
	7.5	36.5/clear (pH =14.0)	46.5/clear (pH =14.0)	25.5/clear (pH =12.0)	60.6/clear (PH =12.0)	61.0/clear (pH =9.0)
	10	66.5/very clear (pH =11.0)	62.5/very clear (pH =14.0)	30.4/very clear (pH =11.0)	70.5/very clear (pH =13.0)	59.8/very clear (pH =12.0)

When the lime and starch were introduced into the cylinders, an interface was formed in each case and after 2 minutes, liquid of varying turbidities formed on top, while particles settled with different degrees in heights (ml). As time proceeds, the interface descends and heights of settled particles increase as well. The data was collected as shown in Table 1. The Table shows that as volumes of both coagulant and flocculant increases, the heights of settled particles increased with varying degrees of turbidity. For different mixing ratios, the rates of settling differ for each dose in the settling slurry in each cylinder as shown in the table after 15 minutes.

At zero volume of lime solution and increasing volume of starch solution, sedimentation occurred with varying degree of turbidity and this could be explained by the fact that with no lime (coagulant), repulsion forces of the electrical double layer charge around the particles prevented very fine colloidal particles from settling. In other words, the mutual attraction forces such as London van der Waal's, surface tension, magnetic attraction etc., among the colloidal particles could not be enhanced due to the absence of coagulant. At zero volume of starch and different volumes of lime, the extremely fine particles were able to adhere directly to each other by reducing the repulsion forces. Overall, the fastest rate of settling was with 10ml of flocculant (starch) in combination with 10 ml of coagulant (lime) giving a ratio of 1:1. Figure 4 indicates different levels of pH reading as lime and starch being introduced to the suspension, but most importantly it was observed that before addition of the reagents the suspension was highly acidic (pH of 4) while the ratio 1:1shows pH of 12 (alkalinity state) which has an implication on corrosion control processes.

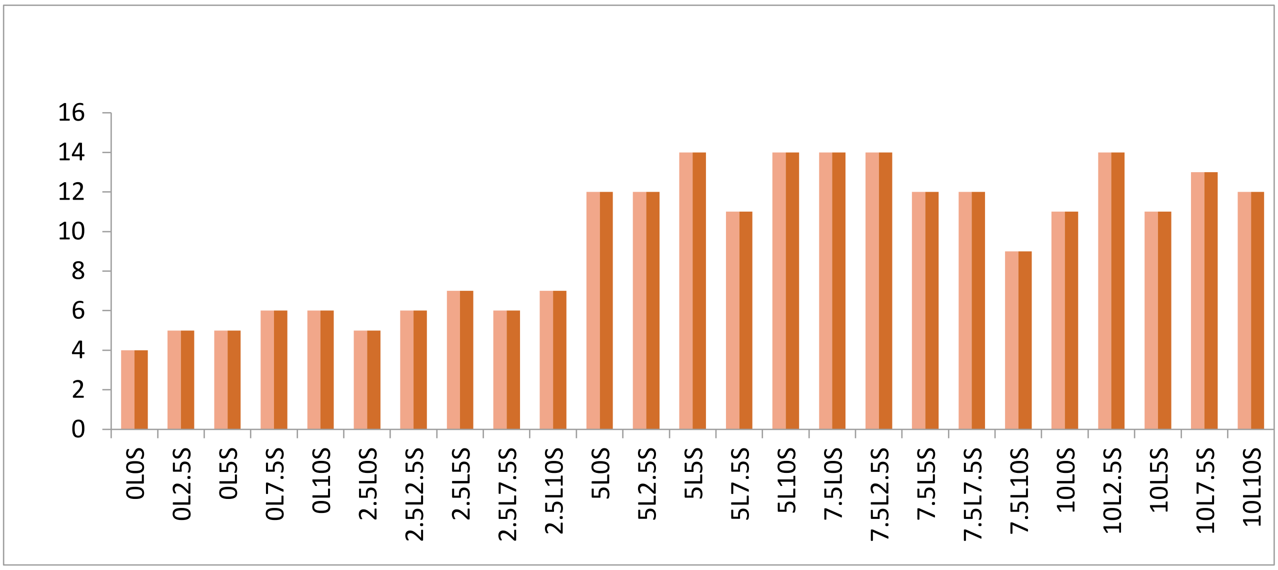

Figure 4: The pH reading against respective mixing ratio

CONCLUSION

Based on the results obtained from the experimental work carried out, the following conclusion can be made;

i. The performance of both lime (coagulant) and starch (flocculant) with dosages ranged from 0 to 10ml were determined

ii. It was found that starch addition to lime enhanced coagulation process even at various starch doses.

iii. The work shows that a substantial degree of agglomeration of the galena and by extension its settling rate can be obtained with much lower polymer (starch) dosage than the electrolyte (lime)

iv. The fastest rate of settling was with 10ml of flocculant (starch) in combination with 10ml of coagulant (lime) giving a ratio of 1:1.

v. The pH reading shows the lowering of acidic level of the wastewater before discharging or recycling which could lead to efficient corrosion control measure.

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