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Influence of operating variables on continuous flocculation of mineral fines

K. V. S. SASTRY, T. WILSON and S. K. MOOTHEDATH
University of California, Berkeley, CA 94720, USA

ABSTRACT

The present study was undertaken to delineate the effect of common process variables such as agitation speed, slurry flow rate, slurry percent solids and slurry pH on the continuous flocculation. Results are discussed in relation to the known results from batch flocculation experiments. It is found that the best operating performance of a continuous flocculation system could be obtained under short residence times and lowest possible agitation speed.

Key words : *Flocculation, Continuous flocculation, Operating variables.*

INTRODUCTION

Removal of fine solids from process plant circulation water and densification of solids are items of growing importance in the mineral and coal industries. Current and future environmental protection measures require that waste slurry discharges from chemical and mineral processing plants to streams and waterways be avoided, and that ponds for the containment of waste slurries be either minimized or eliminated. As a consequence, plant water circuits must be effectively closed, and removal of fine solids from circulating plant must be done to a degree that is operationally acceptable in terms of time and cost. In this context, flocculation is generally accepted as a pretreatment step to solid - liquid separation for the purpose of destabilizing the suspension, thus facilitating the separation process. It is defined as the agglomeration of fine particles into a larger, loosely aggregated mass using high molecular weight long chain polymer.

Surface chemical aspects of flocculation have been investigated in detail, and the appropriate conditions that favor the formation of stable flocs have been documented[1-7]. The process engineering principles of flocculation have not been investigated adequately, and most of such studies are essentially limited to batch flocculation with constant operating conditions[8-18], except that in some cases the polymer is added continuously[15-18]. To the best of our knowledge there is no detailed published study on the continuous flocculation of thick slurries, generally applicable for mineral processing systems. This is obviously due to the complexity of continuous flocculation experiments. However, some studies on continuous coagulation have been reported in the context of water treatment where the solid content in the suspension is very small[19,20] and as such the findings are not too relevant to thick slurries.

Considering the industrial importance of continuous flocculation, we have conducted a detailed process engineering study. Here we present the results of our study on the effect of different operating variables on the continuous flocculation in a mechanically agitated single stage flocculation system and draw conclusions of industrial significance.

BACKGROUND

Flocculation takes place due to the bridging of particles by long chain polymers. The effect of different operating and design variables on the flocculation characteristics of different batch flocculation systems have been studied[8-16] and some of the important results are enumerated in the following.

- The polymer dispersion, adsorption and flocculation takes place within a few seconds after the addition of the polymer. Subsequently, the flocs start degrading till they reach an equilibrium size distribution.
- The floc formation and degradation are found to be strongly dependent on the operating variables such as polymer dosage, slurry pH, solids concentration in the slurry, agitation intensity, size distribution of the feed particles etc.
- There exists some particular ranges of polymer dosage, slurry pH, and solids concentration, beyond which the flocculation was not quite satisfactory.
- While certain amount of agitation is required to promote particle interaction, too much agitation results in the breakage of the flocs due to the excess shear forces.
- The size distribution of the feed particles has significant effect in that finer feeds yield coarser floc size distributions and undergo slower the degradation. In an investigation on the influence of step changes in operating variables on the batch flocculation of limestone Moothedath and Sastry[22] found that multiple stage addition of solids and polymer gives a much coarser floc size distribution when compared to a normal single stage batch flocculation.

MATERIALS AND METHODS

Materials

Commercial limestone (specific gravity - 2.6) was used as the model system in all the experiments. This material, 97 percent calcium carbonate pulverized to nominally 96 percent passing 44 was purchased from the C.Pfizer Company. The complete size distribution and other properties of the limestone powder are given in Table 1. The flocculating agent used for all experiments was a 35 percent anionic polyacrylamide manufactured by the Calgon Corporation, designated as "Calgon 590", prepared as a 0.2 percent aqueous solution.

Table 1 : Size distribution and other properties of limestone feed

Size, µm	62	44	31	22	11	5.5	2.8
Cum. Wt.% Passing	100	96	90	76	43	22	7

Experimental Set Up

A flow diagram of the experimental system used in this study is shown in Fig. 1. The system consisted of (i) a 220 liter slurry preparation cum storage tank equipped with a suitable agitator, (ii) a variable speed slurry transfer pump with a pumping capacity of 1.8 to 8.9 liters/min, (iii) a 6 liter cylindrical flocculation vessel, (176 mm diameter and 250 mm height) provided with four baffles and a 4-blade pitched blade impeller, (iv) polymer storage vessel, (v) polymer metering pump, (vi) a slurry sampler and (vii) a sedimentation column for floc size analysis. Details of flocculation vessel, sampler and floc size analysis column are described by Nayar[11].

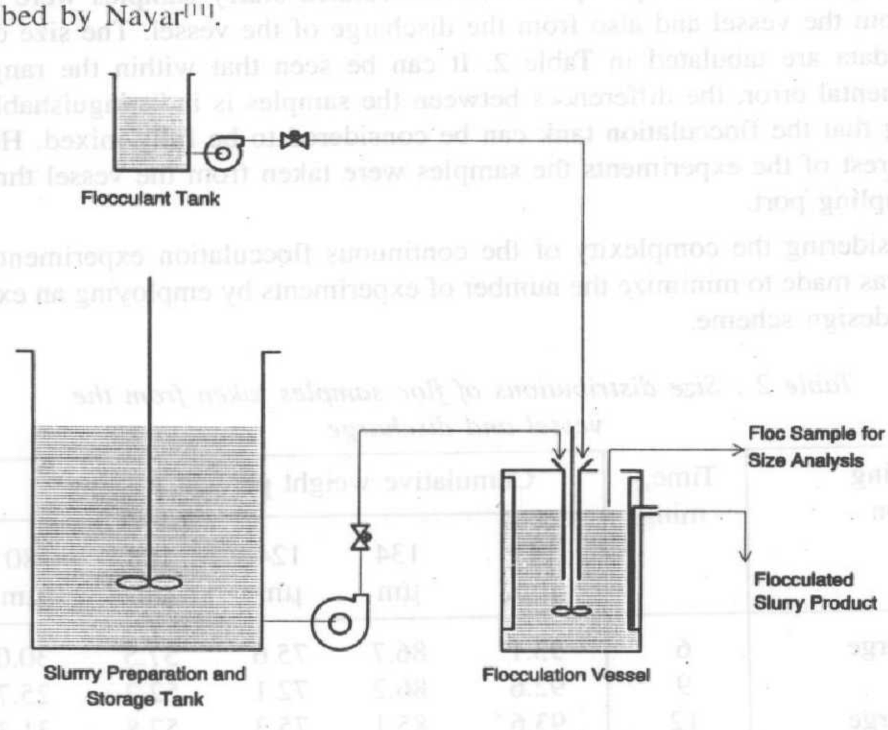

Fig. 1: Flow diagram of the continuous flocculation experimental system

Experimental Procedure

The flocculant solution was prepared one day in advance because of the slow dissolution rate of the polymer. It was found during the trial tests that a polymer batch stored for more than two days has reduced efficacy as a flocculant. A flocculant addition rate of 0.8 milligrams flocculant solids per gram of slurry solids (hereafter written as mg/g) was used for all the experiments to insure adequate dosage.

Limestone slurry was prepared according to the needs of the experiment by weighing an appropriate amount of fine limestone and combining it with a calculated volume of water in the agitated slurry tank. In all cases an extra 50 liters of slurry was prepared to occupy the dead volume in the bottom of the slurry tank. The natural pH of the slurry (8.3) was used except in the study of the effect of this variable, where the pH is adjusted with a saturated solution of lime.

The start up procedure consisted of establishing the flocculant solution and limestone slurry flows at the desired rates in an open circuit mode. After the flow rates are properly adjusted, the experiment was initiated by beginning the addition of flocculant to the slurry tank. Samples were taken at predetermined times and the size distributions are determined using a sedimentation column.

During the preliminary experiments flocculated slurry samples were taken both from the vessel and also from the discharge of the vessel. The size distribution data are tabulated in Table 2. It can be seen that within the range of experimental error, the differences between the samples is indistinguishable indicating that the flocculation tank can be considered to be fully-mixed. Hence, for the rest of the experiments the samples were taken from the vessel through the sampling port.

Considering the complexity of the continuous flocculation experiments, an effort was made to minimize the number of experiments by employing an experimental design scheme.

Table 2 : Size distributions of floc samples taken from the vessel and discharge

Sampling position	Time, min	Cumulative weight percent passing				
		141 μm	134 μm	124 μm	108 μm	80 μm
Discharge	6	93.1	86.7	75.6	57.5	30.0
Vessel	9	92.6	86.2	72.1	52.2	25.7
Discharge	12	93.6	85.1	75.3	57.8	31.3
Vessel	15	91.7	85.1	72.1	54.1	29.9

RESULTS

Fig. 2 gives a plot of the complete floc size distribution from a typical test at different times after the start of the experiment. It is seen that the floc size distribution at 15 sec (which is the first floc sample that could be taken after starting the addition of the flocculant) is the coarsest of all. Actually, as can be seen from Fig. 3 in which the cumulative weight percent larger than the stated size is plotted as a function of time, that by about 3 min the flocs degrade and come to steady state conditions.

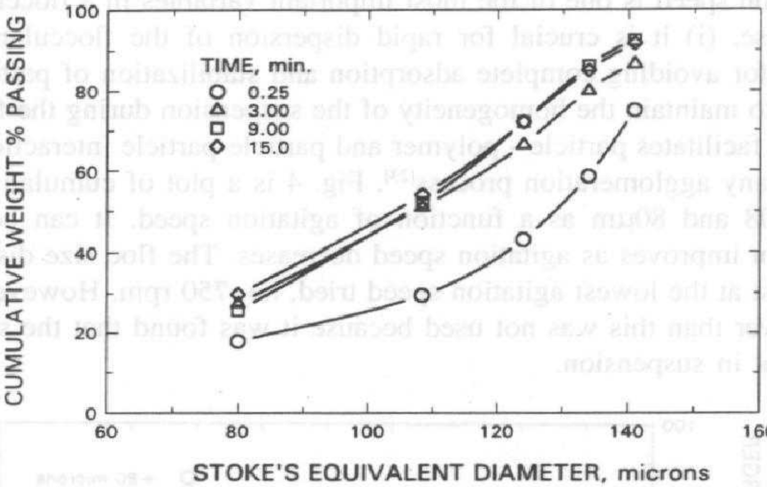

Fig. 2: A typical plot of the cumulative size distribution of the flocs at different times (Solids conc. - 5%, Slurry flow rate - 3 l/sec, Slurry pH - 8.3, Agitation speed - 1000 rpm

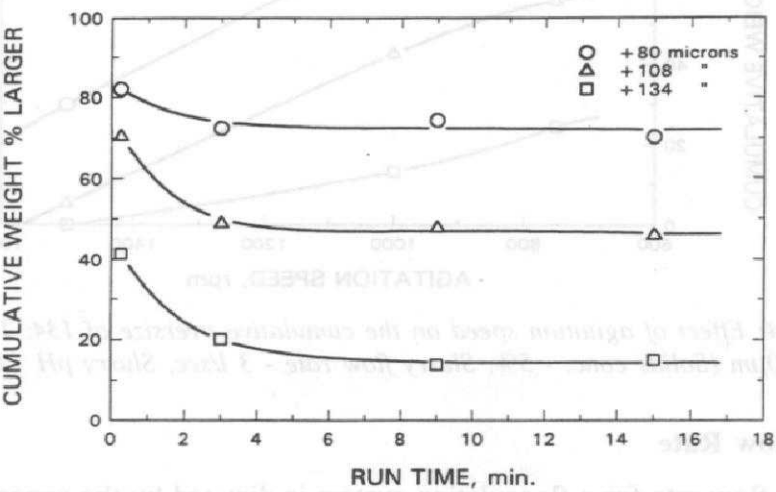

Fig. 3: Effect of run time on the weight percent +134μm, +108μm, and +80μm flocs (Solids conc. - 5%, Slurry flow rate - 3 l/sec, Slurry pH - 8.3, Agitation speed -1000 rpm)

As it is much easier to evaluate the effect of different variables on the basis of weight percent above certain size when compared to the whole size distribution, we have chosen to use the weight percent +134μm, +108μm and +80μm flocs six minutes after the beginning of the experiment for the rest of the study. A discussion on the practical significance of these results presented in the next section.

Agitation Speed

Agitation speed is one of the most important variables in a flocculation system because, (i) it is crucial for rapid dispersion of the flocculant which is necessary for avoiding complete adsorption and stabilization of particles, (ii) it is needed to maintain the homogeneity of the suspension during the flocculation and (iii) It facilitates particle - polymer and particle-particle interaction which is critical in any agglomeration process[23]. Fig. 4 is a plot of cumulative oversize of 134, 108 and 80μm as a function of agitation speed. It can be seen that flocculation improves as agitation speed decreases. The floc size distribution is the coarsest at the lowest agitation speed tried, i.e. 750 rpm. However, agitation speeds lower than this was not used because it was found that the slurry could not be kept in suspension.

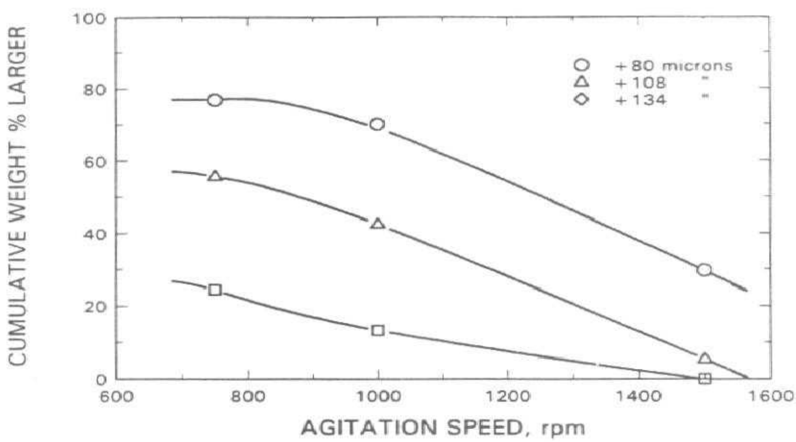

Fig. 4: Effect of agitation speed on the cumulative oversize of 134, 108 and 80μm (Solids conc. - 5%, Slurry flow rate - 3 l/sec, Slurry pH - 8.3)

Slurry Flow Rate

Slurry flow rate for a flocculation system is dictated by the capacity requirements, and for any given system it determines the average residence time of the slurry in the system. The main purpose of studying the effect of slurry flow rate in this case was to understand the effect of average residence time on the extent of flocculation. Fig. 5 shows the effect of varying the slurry flow rate in the range of 2 - 6 l/min on the cumulative oversize percentages after attaining equilibrium. This corresponds to reactor mean residence time in the range 2.35 - 0.78 minutes. It is clear that a higher flow rate results in larger quantities of coarse flocs. In other words flocculation performance improves as residence time decreases. Also the trend of the curves indicate a sharper reduction in the floc sizes in the vicinity of 2 l/min and which corresponds to an average residence time of 2.35 minutes and more.

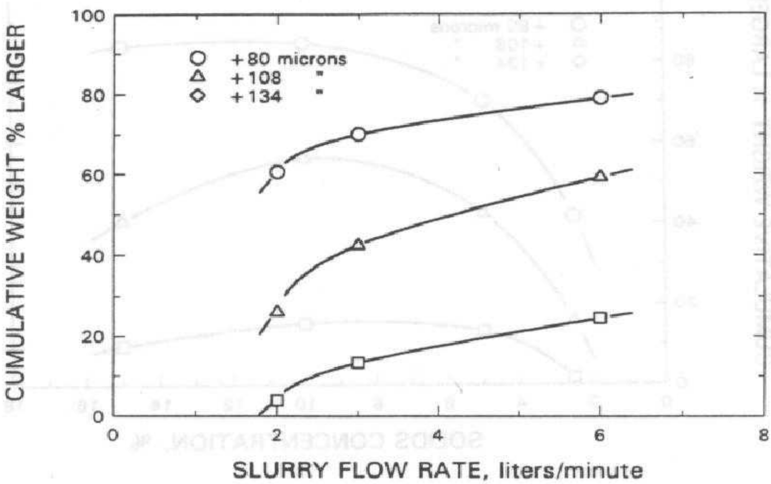

Fig. 5: Effect of slurry flow rate on the cumulative oversize of 134, 108 and 80μm (Solids conc. - 5%, Slurry pH - 8.3, Agitation speed - 1000 rpm)

Solids Concentration

In an industrial system the solids concentration varies widely depending on the specific application of the process, ranging from less than 0.1 percent in water treatment to 10 percent or more in the mineral processing[24]. The effect of varying the solids concentration in the feed slurry over a range of 2.5-15 percent solids was studied and the results are presented in Fig. 6. The curves indicate the existence of larger quantities of coarser flocs at about 10 percent solids. A lower or higher solids concentration resulted in comparatively finer size distributions of the flocs.

Slurry pH

Slurry pH alters surface properties of particles and hence it is very important in flocculation as polymer adsorption on particle surface would be affected by the same. The experiments for studying the effect of pH was carried out at 5 percent solids, slurry flow rate of 3 l/min and 1000 rpm. Three different levels of pH values, 8.1, 10.6 and 11.5; was studied and Fig. 7 shows a plot of weight percent +134μm, +108μm and +80μm flocs at equilibrium against pH. It can be seen that the flocculation performance increases up to a pH of about 10 - 10.5 and declines afterwards. Similar results were observed in the case of batch flocculation of limestone also[11].

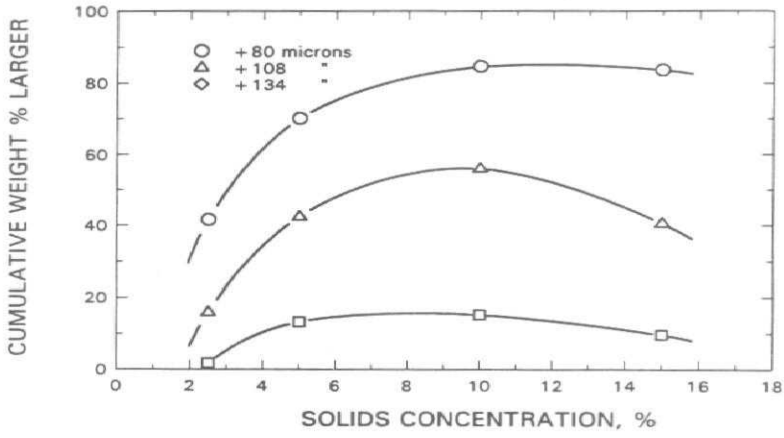

Fig. 6: Effect of solids concentration on the cumulative oversize of 134, 108 and 80μm (Slurry flow rate - 3 l/sec, Slurry pH - 8.3, Agitation speed - 1000 rpm)

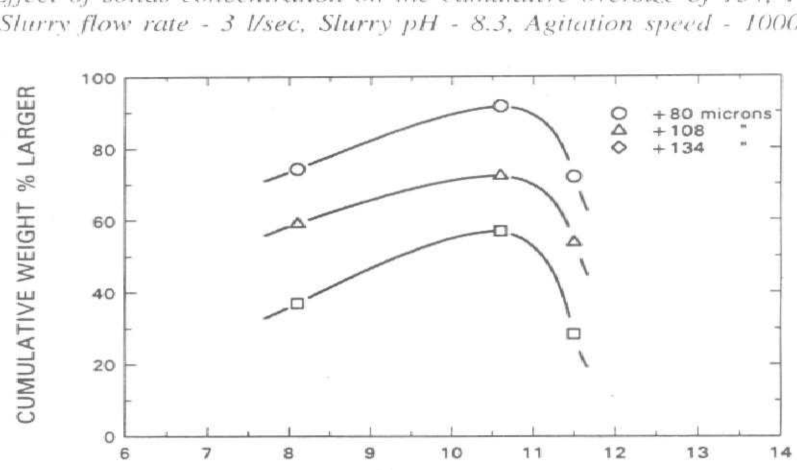

Fig. 7: Effect of slurry pH on the cumulative oversize of 134, 108 and 80μm (Solids conc. - 5%, Slurry flow rate - 3 l/sec, Agitation speed - 1000 rpm)

DISCUSSION

The results from the present experiments indicate that the kinetics of floc formation is very fast for all combinations of solids concentration, slurry flow rate, slurry pH, agitation speed and polymer concentration. For the first sample at 15 sec the flocs have not been affected much by the shear forces in the system and hence the size distribution is the coarsest. This result is in agreement with the batch flocculation results of Nayar[11], Tomi and Bagster[9] and Williams et al[15]. Continuous addition of the solids and polymer will result in reflocculation of the degraded flocs and fines as well as formation and growth of new flocs[22] by mechanisms of nucleation, layering and coalescence. At the same time the flocs will degrade by the breakage or erosion due to the excessive shear. Due to the simultaneous growth and degradation, the system will finally come to a state of dynamic equilibrium when the rate of growth equals the rate of degradation. In almost all the cases studied during the present investigation the steady state was reached within about 3 min after the beginning of the experiment.

When the solids concentration was low (2.5 percent) and the agitation speed was just sufficient to keep homogeneity of the slurry the equilibrium floc size distribution was found to be coarser than the 15 sec distribution. Under these conditions the rate of floc formation seems to be limited by the frequency of particle collisions, which obviously is not adequate.

The effect of different operating variables has considerable process engineering significance. The average residence time in continuous flocculation has a direct connection with the time of flocculation in batch systems. It is well known that in batch flocculation the floc formation is instantaneous and subsequent agitation resulted in continuous degradation until an equilibrium size distribution is reached[9,11,15,16]. Also, in a study on the influence of step changes in operating conditions on the batch flocculation[22], it was found that multiple stage addition of solids and polymer resulted in a much coarser floc size distribution when compared to single stage batch experiment. Based on these results it can be concluded that a completely stirred tank reactor with a short residence time or a plug flow type reactor with provisions for multiple stage addition of solids and polymer are most suitable for a flocculation system. The present study proves that in a continuous system flocculation performance improves with decrease in the residence time. This has fascinating implications on the design and operation of flocculation systems in that shorter retention time means smaller flocculation vessels or higher throughput, either of which is economically advantageous. The agitation speed should be selected so as to maintain the slurry is suspension and keep it homogeneous throughout the vessel. This would depend on the geometry of the vessel and the impeller, as well as the slurry rheology. In the present study a speed of 750 rpm was sufficient to provide a homogeneous suspension. It is to be kept in mind that higher agitation speeds are not desirable as it would result in higher degradation of the flocs. The fact that floc samples from the vessel and the vessel discharge had the same size distribution is a clear evidence that the flocculation vessel is a completely stirred tank reactor (CSTR), wherein perfect mixing takes place. This information can be very useful in the modeling and scale up of the flocculation systems.

The existence of an optimum solids concentration in the range of about 5 - 10 percent was noticed in the case of batch flocculation of limestone[11], and this seems to be valid in the case of continuous flocculation also. It is to be noted that the results merely provide an indication of the influence of solids concentration on flocculation, but it may be of no practical significance. The slurry may have to be accepted as it comes. But this information is certainly be useful for the process engineering analysis and the design of the flocculation systems.

Similarly, it may not be usually possible to change the pH to a large extent due to economic reasons and sometimes environmental reasons. Still, the information on the effect of pH is valuable to account for the minor pH variations during the operation. A pH of around 10.5 (the PZC of limestone), which was determined to be the optimum in a detailed study in batch flocculation[11] happens to give the coarsest size distribution in the case of continuous flocculation also.

CONCLUSION

The kinetics of flocculation in a continuously operated completely stirred tank reactor (CSTR) have been investigated. A slurry consisting of finely ground limestone and a polyacrylamide flocculating agent were employed, and the effects of variable slurry percent solids, reactor retention time, and agitation rate were examined. The settling column technique for determining the floc size distribution of slurry samples provided a new analytical tool for assessing the performance of the flocculation system.

The generation of large flocs early in each test (15 sec.) under all experimental conditions indicated that the kinetics of flocculation fine limestone with polyacrylamide were rapid. After a few minutes under steady state conditions, the floc size distributions reached equilibrium and remained nearly constant.

A general conclusion is that short retention time and a higher concentration of particles (about 5-10 percent) in a zone of medium shear, which is just sufficient to keep the slurry homogeneous, results in best flocculation performance for this system.

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