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## Effect of Conditioning Variables on Impeller Power in Anionic Conditioning of Phosphate Slurries

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### ABSTRACT

The trends that promote anionic reagent adsorption and subsequent flotation were examined in a statistically designed study in an effort to determine their effect on reagent mixing during anionic conditioning on a bench scale. The impeller power was measured during conditioning, and a statistical model was developed to correlate impeller power to the various conditioning variables examined. It was observed that the impeller power was significantly affected by the conditioning variables. A range of solids loading and agitation speeds exists in which the classical power number–Reynolds number correlation was found to hold true. The impeller power was found to be influenced by the conditioning time, indicating a time-dependent variation in rheological properties of the phosphate feed slurry. It was suggested that the impeller power drawn during conditioning was an indication of the extent of mixing of the collector with the particles, which contributes to the reagentization of the phosphate particles and their subsequent flotation. Flotation recovery was found to decrease when conditioning was carried out in the regime where the impeller power deviated from the  $N_p$ – $N_{Re}$  correlation.

### INTRODUCTION

Separation of phosphates of value from quartz in phosphate ores is carried out by the Crago “double float” (1) process by using a mixture of fatty acid and fuel oil as the collector in the rougher flotation stage, followed by deoiling with  $H_2SO_4$  and quartz flotation using an amine collector. The flotation performance of phosphates in the rougher flotation

stage is determined to a large extent by anionic conditioning, the unit operation prior to flotation where the collector is adsorbed onto the phosphate particles. Process upsets and the resulting unpredictability, common to flotation, can be traced back to poor conditioning resulting from incomplete mixing of reagents.

Conditioning is essentially a mixing operation where the phosphate slurry is mixed (reagentized) with the collector, a mixture of fatty acid and fuel oil in a mixer. In the phosphate industry, concentrated slurries (65–75 wt%) of sand and phosphate mixtures are reagentized in conditioners, mostly of the stirred tank type, where the energy requirements vary with the feed. In a survey of agitated tanks used for conditioning phosphate flotation feed with anionic reagents, a statistically significant correlation between the BPL (bone phosphate of lime) recovery and relative slurry viscosity (monitored by power draw) was detected (2). It is believed that the anionic reagents affect the viscosity of the slurry upon emulsification, and hence the impeller power during conditioning. However, the authors are not aware of any published work on rheological studies of phosphate slurries or its effect on flotation performance.

In the past, anionic conditioning in phosphate flotation has often been judged on the basis of collector adsorption studies on a microflotation scale where mixing is on a molecular scale (from solutions of oleic acid). The lab scale results generally do not translate to the industrial scale without a loss in process efficiency. It has been reported that under plant conditions the kinetics of collector adsorption may differ from that in lab conditions mainly due to high solids loading (3). Additionally, the kinetics of adsorption in plant conditions is also governed by collector emulsification because of the use of a mixture of fatty acid and fuel oil in the plants as a collector as opposed to sodium oleate in fundamental studies. Thus mixing during conditioning operations in industry is on a mesoscopic scale and hence plays a deciding role in determining the floatability of phosphate ores.

Of the many rules of thumb that have been developed to represent mixing characteristics in the industrial application of agitators, the power per unit volume has been widely used because of its convenience (4). In the edible oil industry, the use of power per unit volume is a common practice in the design of agitators for hydrogenation of vegetable oil, which is a gas dispersion operation. In the steel industry, in quenching, pickling, and plating operations, the entire process is characterized by a low power per unit volume. Impeller power characteristics find wide use in the petroleum industry, where crude oil blending and suspension of sediment are required for efficient pipeline and refinery operations. Drilling mud mixing to prevent settling of solids and floating of gel is another example of an

operation where power input is used to characterize agitation of non-Newtonian slurries. Agitators are used in dispersion of high solids clay slurries in the clay industry, agitation of high viscosity pulp stock in the paper industry, and in wastewater treatment plants where activated carbon slurries are maintained in suspension. In all of the industrial applications mentioned above, power measurements are used mostly in the design of equipment and rarely for process control. However, power measurements can be used for obtaining rheological control as is done in the concrete industry for determining the workability of concrete mixtures (5). It was the objective of this study to identify a process parameter that could be used to monitor the mixing process and evaluate it as a function of conditioning variables.

## MATERIALS AND METHODS

Phosphate feed ( $-35 + 150$  mesh) was obtained from IMC-Agrico, Four Corners Mine, Bartow, Florida. The size distribution and feed analysis are shown in Table 1. A mixture of fatty acid and fuel oil was used as the collector, and 10% caustic was used as the modifier. Reagents (collectors and modifiers) were obtained from the IMC-Agrico flotation plants.

### Conditioning

Conditioning was done in a 1.75-L stainless steel cell using a Lightnin Labmaster mixer. A four-blade cruciform impeller was used for mixing. The conditioning pH was maintained at 9.2, and the ratio of fatty acid to fuel oil were kept constant throughout the study. The torque required to keep the slurry in motion was measured directly by a built-in dynamometer in the mixer, and displayed on a digital readout as power. The power

TABLE 1  
Feed Size Distribution (5.62%  $P_2O_5$ , 82.43%  
insolubles)

Tyler mesh size	Weight fraction (%)
+ 35	10.23
- 35 + 48	25.58
- 48 + 65	35.08
- 65 + 100	21.55
- 100 + 150	5.30
- 150	1.62

measurements were recorded at the end of conditioning, when the reading had stabilized, in order to minimize fluctuations due to inertial effects.

### Flotation

Flotation was carried out in a Denver flotation machine (Model D-2), using a 5-L stainless steel float cell. The agitation speed was maintained at 1200 rpm, and an aeration rate of 8 L/min was used for all tests. Flotation was done for 1 minute at a pH of 8.2–8.4, using Gainesville tap water. The flotation pH was monitored using a digital pH meter (Fisher model). The float and sink fractions were dried in an oven at 110°C for 12 hours, and their respective weights were recorded. A 10-g sample was obtained by splitting the respective fractions and grinding in an electric alumina mortar.

### Chemical Analysis

1 g of the sample was dissolved in 25 mL of digestion acid (2 parts of HNO<sub>3</sub> and 1 part of HCl) by boiling for 25 minutes. The liquor was filtered using Whatman No. 40 ashless filter paper, and the filtrate diluted to 1 L. Analysis of the filtrate for P<sub>2</sub>O<sub>5</sub> content was done using an Inductively

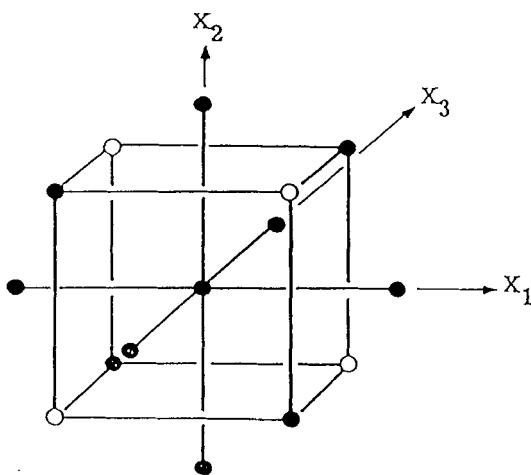


FIG. 1 Rotatable central composite design.

TABLE 2  
Conditioning Variables and Levels Used in Design

Variable	Levels (coded scale)				
	1 - $\alpha$	-	0	+	1 + $\alpha$
Solids loading (wt%)	71	72.2	74	75.8	77
Conditioning time (s)	128	210	330	450	532
Agitation speed (rpm)	366	400	450	500	534

Coupled Plasma Emission Spectrometer (Perkin Elmer II). The insolubles content was determined gravimetrically by burning off the filter paper.

### Statistical Design and Analysis

A three factor-five level rotatable central composite design (illustrated in Fig. 1) was used to conduct experiments in order to model the effects of the conditioning variables on impeller power. The conditioning variables examined in the study, along with their levels, are listed in Table 2. The response, impeller power, measured after conditioning was fitted into a three-variable second-order model of the form

$$P = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + a_{11}x_1^2 + a_{22}x_2^2 + a_{33}x_3^2 + a_{12}x_1x_2 + a_{13}x_1x_3 + a_{23}x_2x_3$$

where  $x_1$  = solids loading in wt%

$x_2$  = conditioning time in seconds

$x_3$  = agitation speed in rpm

The coefficients  $a_i$ ,  $a_{ii}$ , and  $a_{ij}$  were determined using the method of least squares.

### RESULTS

Error analysis of the data indicated that the predicted model had a good fit to the experimental data. In order to visualize the response surface obtained, the predicted power was plotted as a function of solids loading and agitation speed at constant values of conditioning time. Three re-

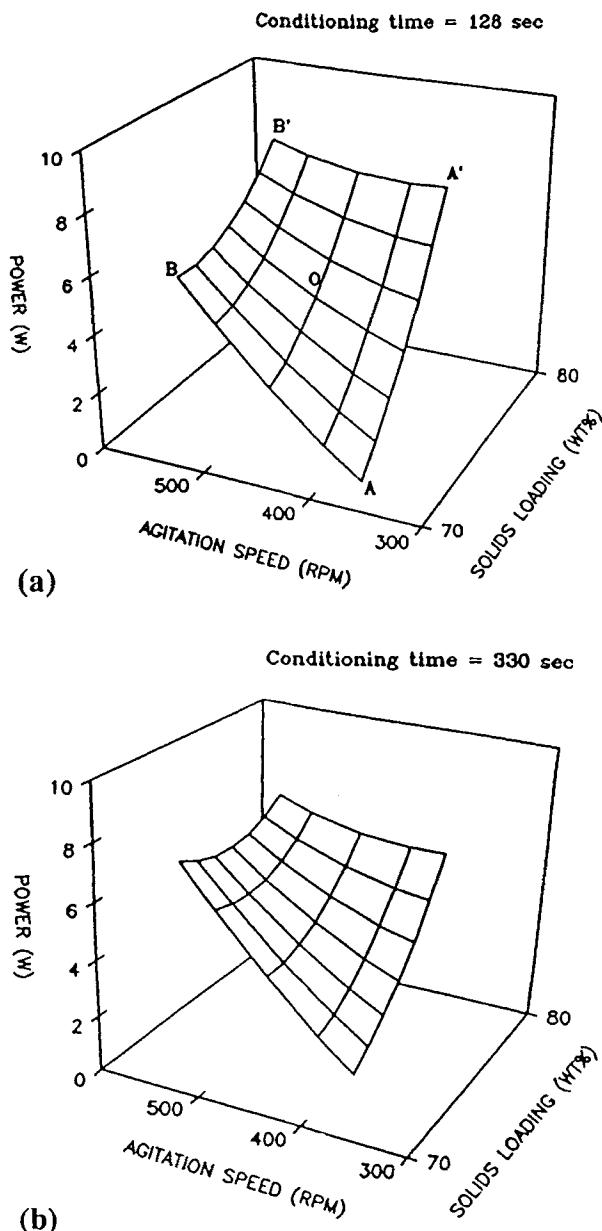


FIG. 2 Contour surfaces representing effect of conditioning variables on impeller power at different conditioning times.

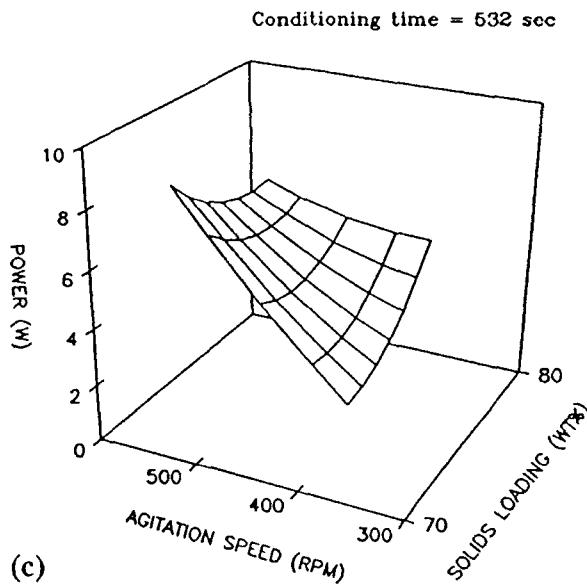
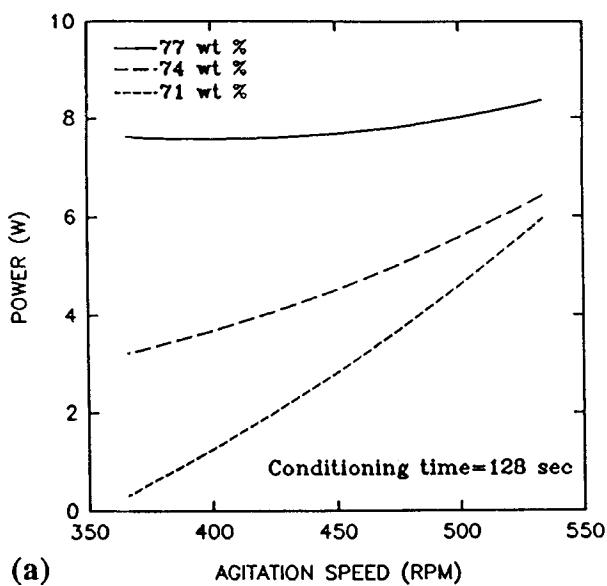


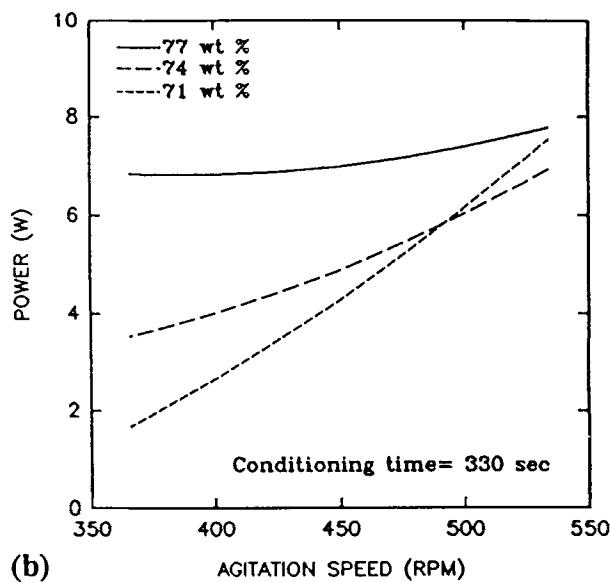
FIG. 2 Continued.

spose surfaces representing power at conditioning times of approximately 2, 5.5, and 9 minutes are shown in Figs. 2(a), 2(b), and 2(c), respectively. The figures show only the experimental regime of the response surface; the mathematical center of the response surface is located away from the center of the design and is not discussed here. The response, power, is plotted on the vertical axis versus the agitation speed (366 to 534 rpm) and solids loading (71 to 77 wt%) on the horizontal plane. As can be seen from the figures, the response surface is concave upward.

The effect of agitation speed on the impeller power is shown in Fig. 3. The impeller power increased with an increase in agitation speed. However, the increase in power was less prominent at higher solids loading. Figure 4 shows the effect of solids loading on impeller power. An increase in solids loading was generally seen to increase the impeller power. It is interesting to note that at a conditioning time of 9 minutes, an increase in



(a)



(b)

FIG. 3 Effect of agitation speed on impeller power.

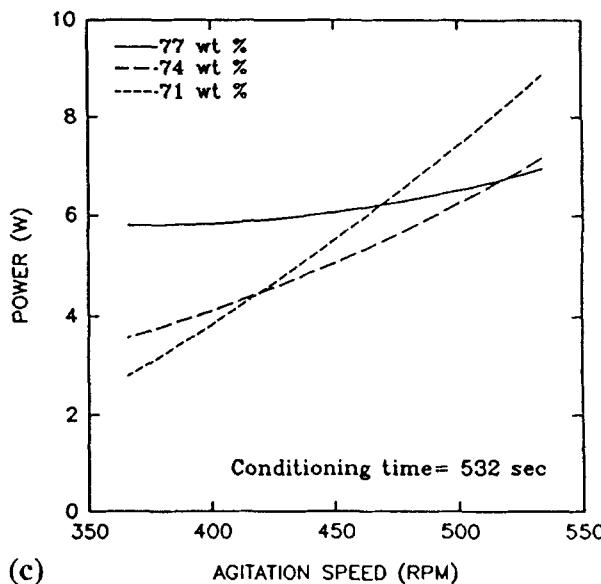
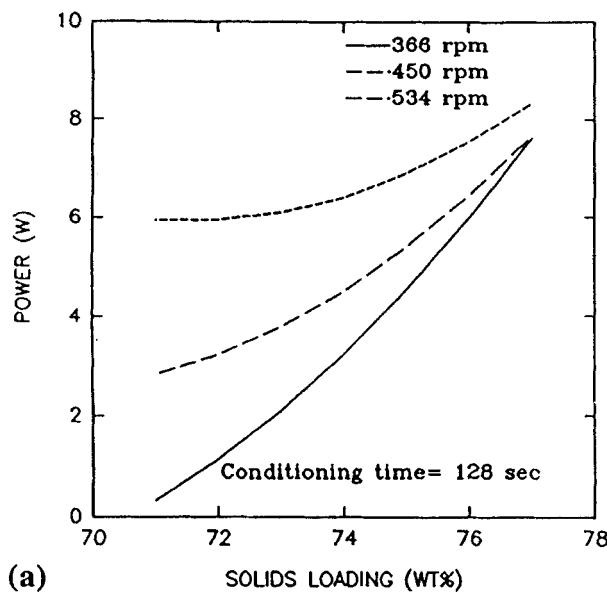


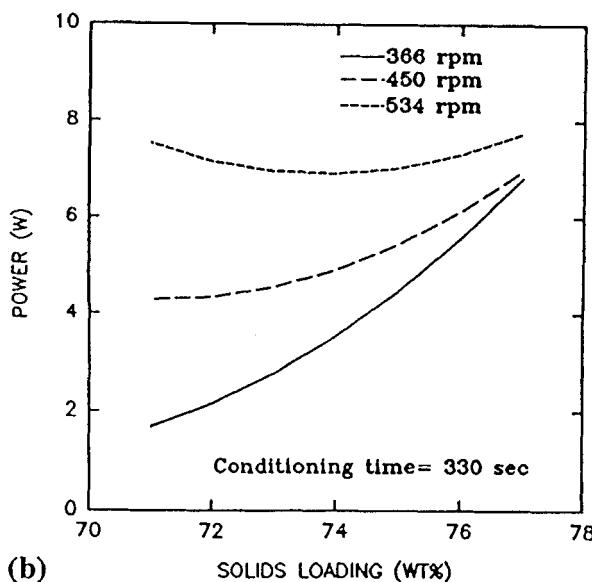
FIG. 3 Continued.

solids loading resulted in a decrease in the impeller power at high agitation speeds as shown in Fig. 4(c).

To obtain quantitative information about the effect of conditioning time on impeller power, power was also plotted as a function of conditioning time at selected values of agitation speed and solids loading. The significant effect of conditioning time on impeller power is evident from the difference in the nature of the response surfaces shown in Fig. 2. The trends shown by the time-dependent variations in impeller power at high solids loading is different from that observed at low solids loading (Fig. 5). The impeller power increases with an increase in conditioning at 71 wt% solids (see Fig. 5a). At 74 wt% solids, impeller power does not show any significant change with conditioning time as shown in Fig. 5(b). However, at 77 wt% the impeller power is observed to decrease with an increase in conditioning time as illustrated in Fig. 5(b).



(a)



(b)

FIG. 4 Effect of solids loading on impeller power.

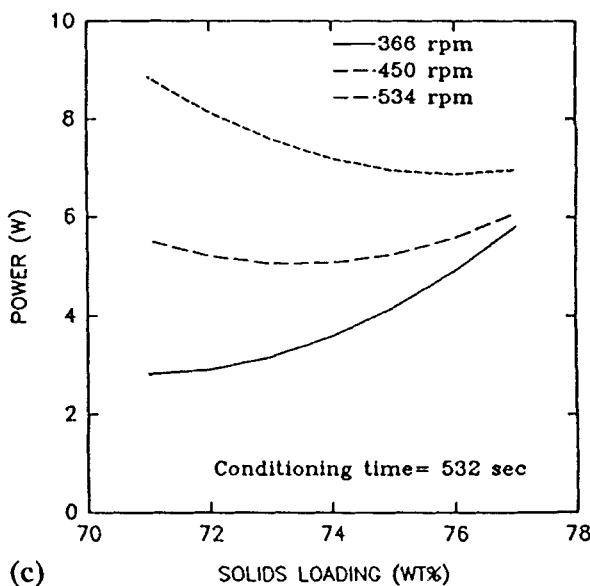


FIG. 4 Continued.

The average recovery and grade of the concentrate (float fraction) from the rougher flotation at a conditioning time of 3.5 minutes are reported in Table 3. The range of solids loading from 71 to 74 wt% and that of agitation speed from 366 to 450 rpm will be referred to as "low" in the discussion that follows. "High" values of solids loading and agitation speeds are

TABLE 3  
Phosphate Recovery and Grade at a Conditioning Time of 3.5 Minutes

Variables	Range	Recovery (wt%)	Grade (wt% P <sub>2</sub> O <sub>5</sub> )
Solids loading	71-74 wt%	89.2	29.4
	74-77 wt%	68.3	32.5
Agitation speed	366-450 rpm	85.1	30.1
	450-534 rpm	58.1	31.3

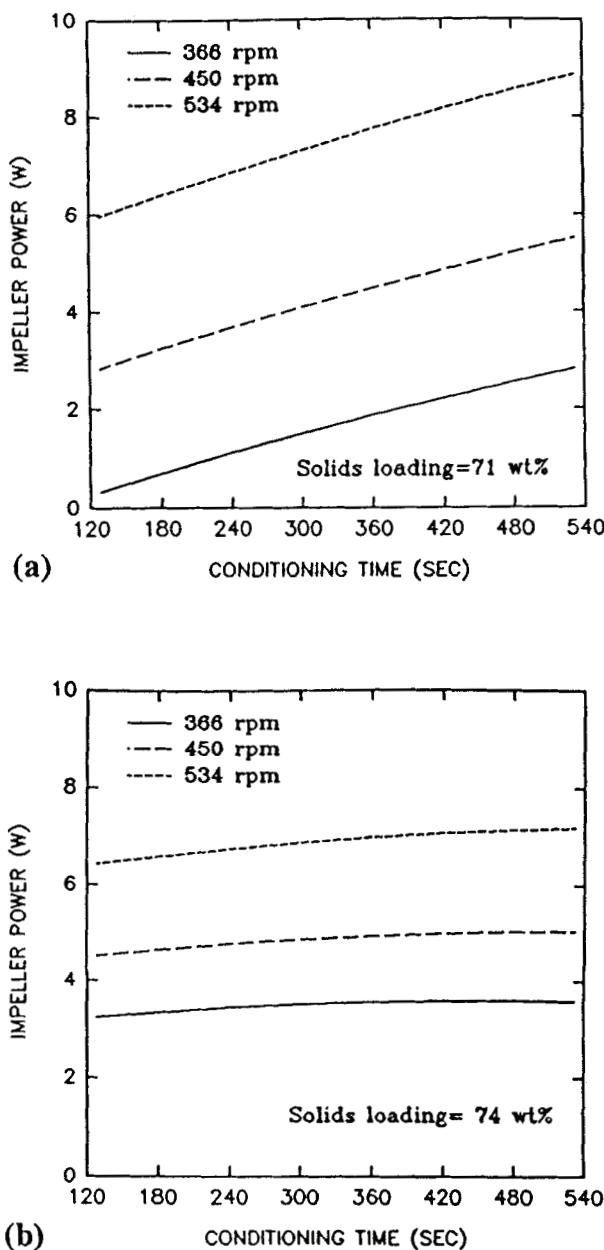


FIG. 5 Effect of conditioning time on impeller power.

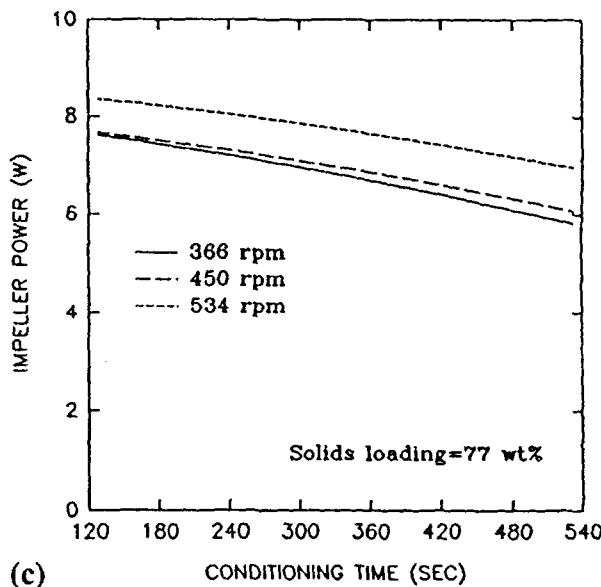


FIG. 5 Continued.

those in the 74 to 77 wt% and 450 to 534 rpm ranges, respectively. The average recovery is found to be lower at 74-77 wt% and at 450-534 rpm for a conditioning time of 3 minutes. The recovery and grade of the concentrate at high solids loading and agitation speed (for 9 minute conditioning time) is given in Table 4. The recovery is seen to increase significantly when mixing times are longer.

TABLE 4  
Phosphate Recovery and Grade at a Conditioning Time of 9 Minutes

Variables	Recovery (wt%)	Grade (wt% P <sub>2</sub> O <sub>5</sub> )
High solids loading	71.8	32.1
High agitation speed	84.9	29.7

## DISCUSSION

The impeller power is described in terms of  $N_P$ , the power number,  $N_{Re}$ , the Reynolds number, and  $N_{Fr}$ , the Froude number, by the well-known fluid mechanics equation (4):

$$N_P = K(N_{Re})^a(N_{Fr})^b \quad (1)$$

where

$$N_P = P/\rho N^3 D^5$$

$$N_{Re} = D^2 N \rho / \eta$$

$$N_{Fr} = DN^2/g$$

The constant  $K$  depends on the geometry of the system, and "a" and "b" depend on the flow characteristics of the slurry. On rearranging Eq. (1), the following relationship for power is obtained:

$$P = K \frac{(D^{2a+b+5})(N^{a+2b+3})(\rho^{a+1})}{(\eta^a)(g^b)} \quad (2)$$

From the above equation it is clear that in the batch test of anionic conditioning where the geometry of the system was unchanged, the impeller power is a function of the impeller speed, the specific gravity, and the viscosity of the slurry. The value of the constant "a" is experimentally found to be -1, while "b" is a real number for both Newtonian and non-Newtonian slurries (6-9). Upon substitution of -1 for "a" in Eq. (2), the relationship for impeller power reduces to

$$P = K_1(N^{2b+2})\eta \quad (3)$$

where  $K_1$  is also a constant. The effect of conditioning variables on the impeller power in batch conditioning tests will now be discussed on the basis of Eq. (3).

### Effect of Design Variables

According to Eq. (3), an increase in agitation speed should increase impeller power. This is in accordance with experimental observations (see Fig. 3). Physically, higher impeller power measurements at higher agitation speeds are due to increased hydrodynamic resistance to flow of the slurry. Generally during agitation of fluids, the impeller power varies as the cube of the impeller speed. In the conditioning study, such a dependence is observed at lower solids loading only. It is interesting to note that at a solids loading of 77 wt%, the increase in impeller power with

agitation speed is less prominent (indicated by solid lines in Fig. 3) and deviates from the cubic relationship. This is attributed to the use of extremely high intensity agitation. At such high intensities it is observed that the mixer approaches its maximum power limit and slippage between shearing planes begins. The peripheral layer of the slurry was found to be almost immobile, resulting in lower power readings than the actual values. Thus, at extremely high solids loading, the mixing characteristics are seen deviate from that predicted by Eq. (1).

The effect of solids loading on impeller power is not directly evident from Eq. (3), but it should be noted that for a non-Newtonian slurry, the apparent viscosity,  $\eta$ , is a function of solids loading (10). The apparent viscosity of a non-Newtonian slurry at a defined shear rate depends on the viscosity of the liquid  $\eta_0$ , the particle size distribution (represented by the mean particle size  $x_d$  and standard deviation  $\sigma$ ), and the solids loading  $\phi$ . In general, the viscosity of the phosphate slurry can be described by the following relationship:

$$\eta = \eta_0([X_d, \sigma], \phi) \quad (4)$$

The apparent viscosity of a non-Newtonian slurry depends on such other factors as the temperature, surface properties, electrical charges (state of dispersion or aggregation), and the nature of the flow fields. The apparent viscosity of non-Newtonian slurries are known to increase with solids loading (11). This is evident in the increase in impeller power detected with an increase in solids loading (Fig. 4a). However, it was observed that with an increase in agitation speed, the expected increase in impeller power diminishes. The anomalous affect of lowering of impeller power at high solids loading is particularly evident at 534 rpm as illustrated in Figs. 4(b) and 4(c). This anomalous behavior was again attributed to the occurrence of slippage at high shear. The impeller power, and hence the mixing characteristics, were found to deviate from the  $N_p-N_{Re}$  correlation at extremely high solids loading and agitation speed.

### Effect of Conditioning Time

The impeller power in Eq. (3) is independent of time for Newtonian and non-Newtonian fluids which do not show a time-dependent variation in viscosity. It was initially suspected that the increase in impeller power with time could be due to a change in the particle size distribution resulting from generation of fines at high agitation intensity. It was found that at 77 wt% solids loading and 534 rpm agitation speed (maximum agitation intensity), as much as 3.16 g of fines (–325 mesh) were generated from 1 kg of phosphate feed in 9 minutes of conditioning. However, the addition

of up to 6 g of fines (−325 mesh) resulted in no detectable change in power measurements. This indicated that any change in impeller power with conditioning time at high agitation speeds was not due to generation of fines.

The variation in impeller power with conditioning time is attributed to plausible time-dependent variations in the rheological properties of the slurry. The effect of conditioning time on the impeller power drawn by the slurry is thought to be due to reagentization of the phosphate particles by the collector. It is believed that as conditioning progresses, emulsification of the collector occurs, resulting in an increase in the number of droplets of the organic phase (fatty acid and fuel oil) and hence an increase in the collector–particle interaction, leading to adsorption of the collector on phosphate particles. In the absence of the collector, the slurry showed no time-dependent variations in impeller power. In the presence of reagents, such an increase is observed up to a solids loading of 74 wt%.

The time-dependent increase in impeller power is seen to be higher at 71 wt% solids (see Fig. 5a). At 74 wt% solids, impeller power shows a slight increase with conditioning time as shown in Fig. 5(b), suggesting that mixing and adsorption times are shorter (less than 2 minutes) at higher solids loading. This lends credence to the current industry practice of conditioning at higher solids loading. However, at still higher solids loading (77 wt%), mixing is expected to decrease due to slippage at the walls, resulting in lower shearing. The decrease in impeller power with conditioning time (Fig. 5c) at 77 wt% solids is suspected to be due to occurrence of slippage. The possibility of reduction of viscosity of the slurry due to the lubricating effect of the collector on the particles at high solids loading cannot be ruled out. However, further investigations are warranted to establish the reasons for decreasing impeller power at high solids loading.

In the regime of solids loading from 71 to 74 wt% and that of agitation speed from 366 to 450 rpm, the  $N_p$ – $N_{Re}$  correlation was found to hold true. The higher phosphate recovery (shown in Table 3) when conditioning was carried out in this regime was attributed to better mixing. This regime of better mixing and higher phosphate recovery is represented in Fig. 2(a) by the region bounded by the letters AA'OB. The power numbers were found to deviate from the  $N_p$ – $N_{Re}$  correlation at higher solids (77 wt%) and agitation speeds (534 rpm), indicating incomplete mixing. This is reflected in the lower phosphate recovery achieved under these conditions. The regions closer to point B' in Fig. 2(a) represent regimes of solids loading and agitation speeds where incomplete mixing of reagents and subsequently lower phosphate recovery were detected. Longer conditioning times were required to achieve better mixing and subsequently higher

recovery when conditioning was carried out at higher solids loading and agitation speeds.

## CONCLUSIONS

A statistical design of experiments was conducted to model the effect of conditioning variables on impeller power. The data predicted by the model were in close agreement with the experimental data. The effect of conditioning variables on the impeller power was explained on the basis of the well-known empirical equations for mixing of non-Newtonian fluids. An interesting feature was that the impeller power of the reagentized slurry was found to increase with time. This was attributed to the increase in the viscosity of the slurry due to emulsification of the collector and subsequent adsorption onto the phosphate particles. The results indicate that the conditioning variables have an interactive effect on the mixing of slurries during conditioning. There exists a regime of agitation intensities and solids loading in which mixing is characterized by the well-known  $N_P-N_{Re}$  correlation. The flotation recovery was found to be higher when conditioning was carried out in this regime. The present work suggests the possibility of estimating reagent adsorption upon mixing by monitoring the viscosity of the slurry during conditioning.

## NOMENCLATURE

$P$	impeller power
$N$	impeller speed
$D$	impeller blade diameter
$\rho$	slurry specific gravity
$\eta$	slurry viscosity
$\eta_0$	fluid viscosity
$g$	acceleration due to gravity
$x_d$	mean of particle size distribution
$\sigma$	standard deviation of particle size distribution
$\phi$	solids volume fraction

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ments. All opinions expressed are solely those of the authors and do not necessarily reflect the views of FIPR or Jacobs Engineering Group Inc.

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