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Recent Advances in Power Requirement and Powder (Product) Characteristics of Stirred Media Milling

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PLENARY

ABSTRACT

While applications of stirred media mills for fine particle production have continued to grow, there is a lack of understanding of power requirements, optimum operating conditions and powder (product) characteristics underlying stirred media milling processes. Recent results of tests in laboratory stirred media mills with media, limestone and yttria stabilized zirconia are presented and mill dynamics, performance and physico-chemical aspects are discussed. Four operational regions marked by sharp transitions are described: transition from static to dynamic friction; channelling; dispersion; and centrifugation. Equations, including power and modified Reynolds number, have been established for relating relevant operating and geometrical variables. Scale-up guidelines with respect to power consumption are also proposed. The best operating conditions for grinding limestone have been identified. Effect of additives including that on the ground product is discussed using the example of ultrafine grinding of zirconia, in which complexation by polymer was found to cause extraction of yttrium into solution with resultant changes in surface chemical composition of the product.

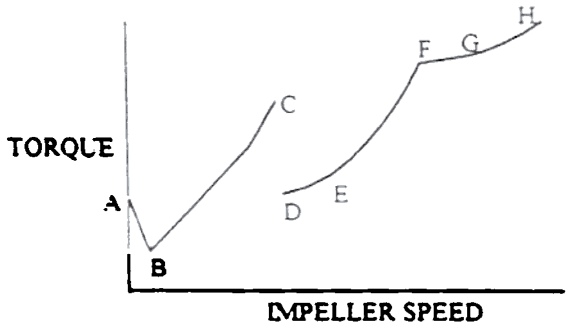
INTRODUCTION

It has been estimated that 25 billion kWh of U.S. electrical power production is consumed annually in ore beneficiation, with half of that spent in the grinding stage.[1] Tumbling, planetary, centrifugal, fluid energy, vibratory and stirred mills are primarily used in grinding operations. In comparison with other mills, stirred media mills (including Tower, sand, Coball and Perl mills) have especially attracted attention during recent years because of their reported high energy efficiency, ability for grinding into the micron and sub-micron range, and lower product contamination. Stirred media mills are used for fine particle production in many industries such as mineral, metallurgical, ceramic, electronic, pigments, paint and lacquer, chemical, bio-technology, rubber, agricultural, pharmaceutical, photographic, coal and energy.[2–4] However, there is very little design information available, and scale-up of stirred media milling processes rarely reaches the designed capacity.[5] Detailed operating and performance characteristics of stirred media mills are also proprietary. This situation is in contrast to that in tumbling mills, for which information including type, dimensions, loading, speed and power consumption is freely available and reliable estimates of mill power draw can be made by means of dynamic and semi-empirical models.[6] In this context, there is a need for fundamental understanding of power requirement, operating conditions, powder (product) characteristics, and the requirements for optimum performance in stirred media mills.

In this paper, recent results of tests in vertical stirred media mills with media, limestone and yttria stabilized zirconia in our laboratory are given. Mill dynamics, operating performance and physico-chemical aspects in stirred media mills are also discussed.

MILL DYNAMICS

Power characteristics of stirred media mill have been studied by an approach developed from work on stirred reactors operating under different conditions of impeller speed, design and dimensions, and media concentration, size and density.[7-8] Fig. 1 provides a schematic summary of distinct regions displayed by torque versus (4-pin) impeller speed curve for monosize glass spheres media with water as supernatant. This shows that the process passes through four regions: transition from static to dynamic friction; channelling; dispersion; and centrifugation.[7] Channelling is caused by cavities on the trailing edges of the impeller pins. The progress from the region of channelling to that of dispersion results in the drop in torque (Fig. 1 CD), which is due to media dispersion into supernatant causing a drop in concentration and consequently a sharp lowering of viscosity. If there is no supernatant, the concentration does not decrease and the discontinuity does not occur. Because the dispersion region is relatively uniform and of known concentration, it is especially amenable to analysis. To further understand channelling and dispersion flow behavior, a model has been developed to predict power draw of multiple pin impellers from results obtained for milling with one pin situated at depths corresponding to each pin in the multiple pin case.[8]



AB static to dynamic friction region
BC channelling region, media rotation commences near C
CD media begins to suspend into the supernatant
DE media dispersing into available supernatant
EF media fully dispersed and uniform in concentration
FG vortexing
GH centrifuging

Figure 1: Schematic summary of regions displayed by torque versus impeller speed curve in media with supernatant liquid.

In order to establish the relationship between power consumption and the process variables, dimensionless groups including power and Reynolds number are used. Several researchers[9-10] use the viscosity and density of the liquid phase rather than those of slurry and media combination to calculate power and Reynolds numbers. An alternative method which is adopted in the current work considers the mixture of pulp and grinding media as a non-Newtonian power law liquid. Combining the power law equation with the viscosity definition and the assumption that the average liquid shear rate is proportional to impeller speed,[11] the effective viscosity can be obtained:

$$\mu = K\alpha^{n-1} N^{n-1} \tag{1}$$

A detailed procedure for determining the effective viscosity has been developed based on the two assumptions of proportionality: shear rate to impeller speed; and shear stress to torque.[7] Relationships between effective viscosity (m), flow index (n), consistency (K), impeller speed (N), and concentration have been evaluated for a number of design geometries, media particle size and solid concentration.[7-8] The viscosity value can be incorporated into the Reynolds number:

$$N_{Re} = \frac{N^{2-n} D^2 \rho}{K\alpha^{n-1}} \tag{2}$$

Therefore, the power number equation for laminar conditions can be expressed as:

$$N_P = C\left[\frac{N^{2-n} D^2 \rho}{K\alpha^{n-1}}\right] \tag{3}$$

where C is a design factor dependent on impeller/tank geometry which can be determined by employing the power and Reynolds number correlation evaluated using Newtonian calibration liquids. For the full dispersion condition [Fig. 1 EF], the average density can be calculated from the volumetric concentration, c, using the equation

$$\rho = c\rho_S + (1-c)\rho_l \tag{4}$$

Power and Reynolds number can be calculated using the viscosity and the density determined with equations (1) and (4). The relationship between power number and modified Reynolds number for 4-pin, 1-pin and disc impeller at different conditions has been correlated.[7-8] Figure 2 presents a summary of plots of power number vs. Reynolds number for tests with 4-pin impellers for different impeller speed and dimensions, media size, concentration and density. The data is correlated by a straight line of slope −1 for the wide range of variables studied. Based on this result, certain scale-up guidelines with respect to power consumption are proposed: using a small mill to determine the values of C, K, n and a parameters and assuming that they remain constant in scale-up, power consumption for large units can be calculated using the equation:

$$P = CK\alpha^{n-1} N^{n-1} D^3 \tag{5}$$

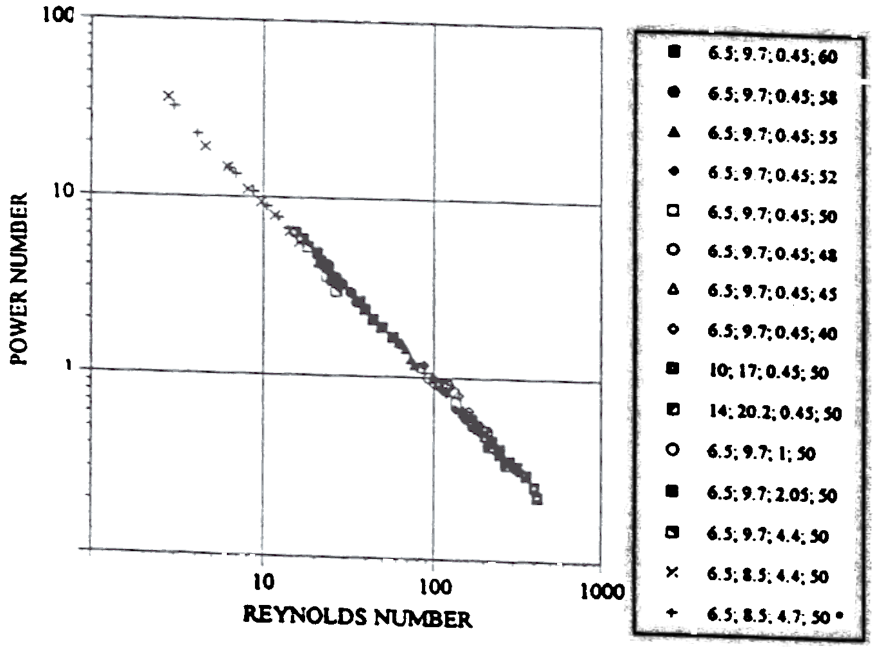


Figure 2: Power number versus Reynolds number for 4-pin impeller for different conditions. The order of the tabular data is D(cm): T(cm): d(cm): c(%) by volume. All refer to glass media except the last entry for steel balls.

MILL PERFORMANCE

Power requirements and product characteristics for stirred media milling of limestone with and without supernatant have been studied with respect to grinding time, impeller speed, total solid (media and particles combined), ratio of media to particle volume, media size and density, impeller and tank dimensions, impeller design, pulp viscosity and so on.[12]

Without supernatant, lower stirring speeds have resulted in better energy efficiency (ratio of specific surface area to specific energy) for grinding limestone . This finding is in accordance with the results for grinding coal[13] and for grinding dolomite.[14] However, relatively higher speeds give higher specific surface area in a given grinding time.[12] Considering the combined effects of grinding time and stirring speed, both lower speed and shorter grinding time give better energy efficiency. Energy efficiency is found to be proportional to the number of revolutions (product of speed and time) raised to −0.57 power.

The results shown in Fig. 3 obtained for grinding limestone with initial supernatant liquid are different from those for the cases without supernatant. The average torque versus speed curve displays several distinct regions, in agreement with the results for a loading of glass beads only with supernatant liquid [Fig. 1]. Product surface area is found to increase with stirring speed, and this result is similar to that in the case without supernatant. However, because of the drop in torque due to the transition from channelling to dispersion, the best energy efficiency occurs at the stirring speed corresponding to the lowest point in torque versus speed, in the "onset of dispersion" region.

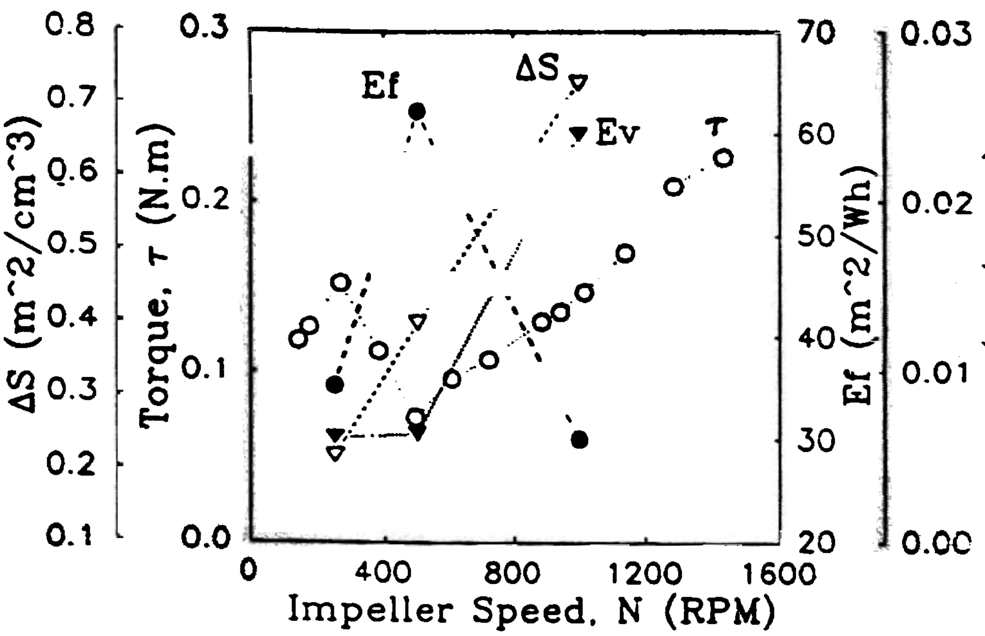


Figure 3: Effect of impeller speed with the supernatant (conditions: D = 6.5cm; T = 11.8cm; t = 15min; c = 60%; R = 3; d = 3; d = 2.05mm; V = 150cm^3).

The results with and without supernatant indicate that solid concentration is a very important factor in the grinding process. Effect of total solids (media and particles) concentration on grinding limestone is shown in Fig. 4. The torque or power increases with increasing solid concentration, and the best energy efficiency is obtained at the solid concentration corresponding to the value at which there is minimal supernatant liquid in the system. At the very high concentration (80%), only those solids (media and particles) around the center of the impeller are stirred causing grinding of the contained particles, while beyond the impeller pins the solids remain almost stationary and particles in that region remain unground. Also wet grinding is more energy efficient than dry grinding (c=100%).

In the study of the effect of solids concentration, solids include both media and particles, and therefore, the effect of their relative proportions must be evaluated. Specific energy increases with increasing ratio of media to particle volume, and the ratio for the best energy efficiency and product fineness is 2.8 for grinding limestone using glass beads as media. This corresponds to the voids in the media packing being just occupied by the limestone particles. This critical ratio of filling is given by

$$R = \frac{1-\varepsilon_m}{\varepsilon_m(1-\varepsilon_p)} \qquad (6)$$

and confirmed in this case as $e_m = 0.4$ and $e_p = 0.47$ giving R = 2.8.

Just as the ratio of media to particle volume has an effect on grinding, the ratio of media to feed particle size may also have an effect. In fact, the optimum ratio of media to feed particle size is found to be 12:1 for the case of grinding limestone using glass beads. Furthermore, glass media is found to be more energy efficient for grinding limestone than steel. The best ratio may depend on media density and minerals being ground. The optimum range of media to feed size suggested in the literature[15] is 7:1 to the maximum 20:1. For the case of grinding coal using steel balls, the optimum ratio is reported to be 20:1.[16]

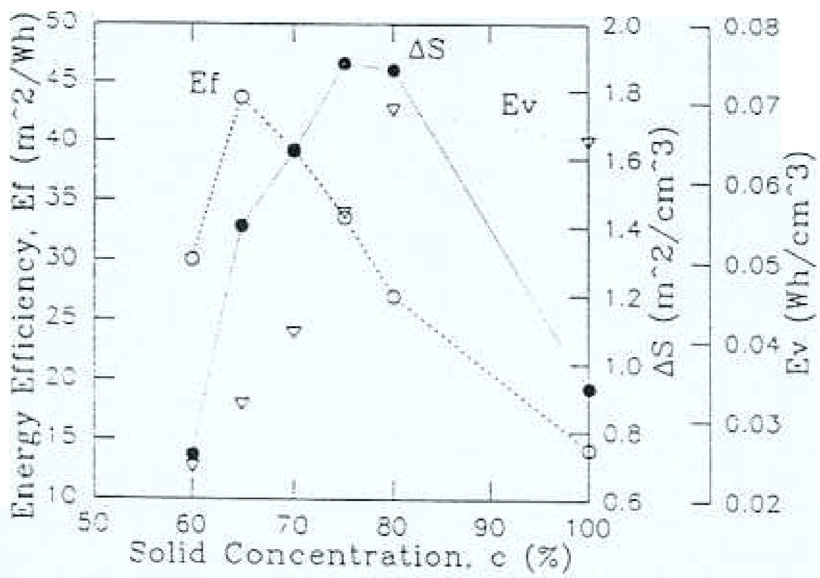


Figure 4: Effect of total solid concentration (conditions: D = 6.5cm; T = 11.8cm; t = 15min; N = 1000 rpm; R = 3 d = 2.05mm; V = 150cm^3).

Dimensional relationships between impeller, which displaces the media to cause particle fracture, and tank containing media and particles also affect grinding. The torque depends principally on the impeller diameter but hardly on the tank diameter (see Fig. 5). The lower the ratio of tank to impeller diameter, the finer the product. However, the maximum energy efficiency is obtained at the highest ratio of tank to impeller diameter.

It is clear from above discussion that the grinding process is affected by many operating variables, which in turn influence pulp viscosity. A procedure to determine the effective viscosity during the grinding process rather than at its termination was described earlier in this paper. Based on this procedure, the effective viscosity values were calculated and the relationship between effective viscosity and solid concentration for the cases of media only, and media and particles combined are compared and shown in Fig. 6 together with the best fit equations.

The correlation between grinding and energy input is described by the equations relating energy efficiency and increase of specific surface area respectively as a function of volume-based energy for most cases studied (see Figs. 7 and 8). The results using the half 4-pin impeller are included in the correlations, suggesting that grinding may not be very dependent on the particular design of this type of impeller. However, some conditions, namely, lower solid concentration, smaller media size, higher density and dry grinding, are excluded from the present correlations, probably due to different and less efficient grinding.

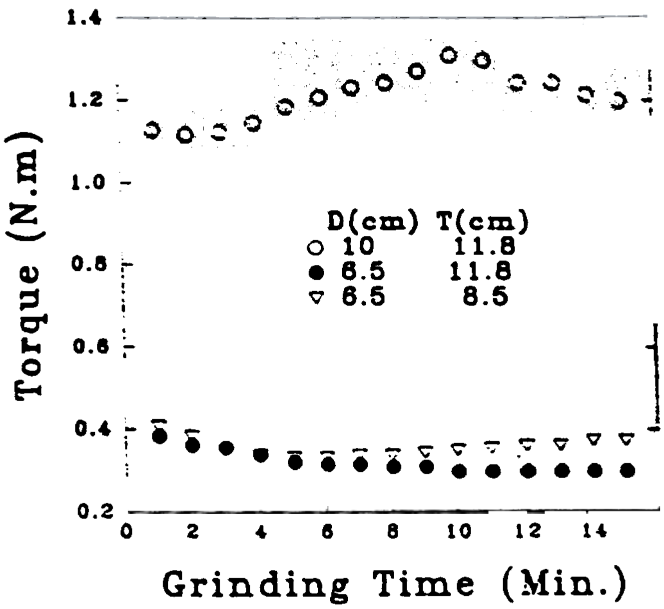


Figure 5: Effect of impeller and tank diameter on torque.

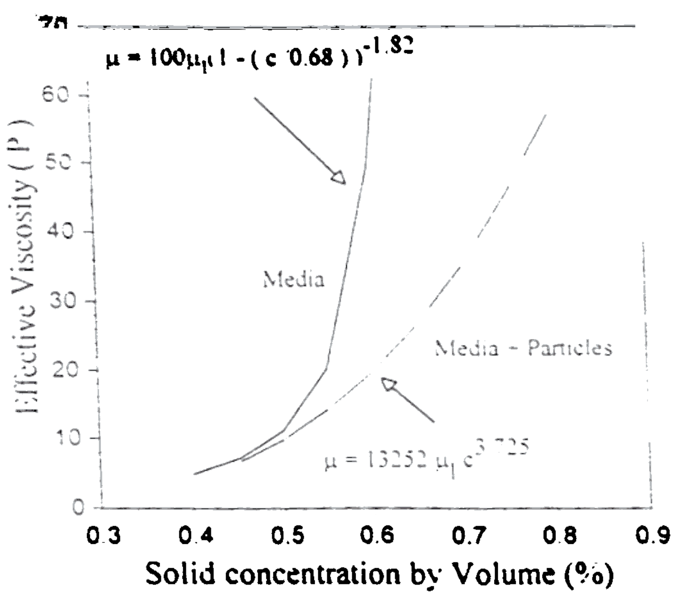


Figure 6: The relationship between effective viscosity and solid concentration.

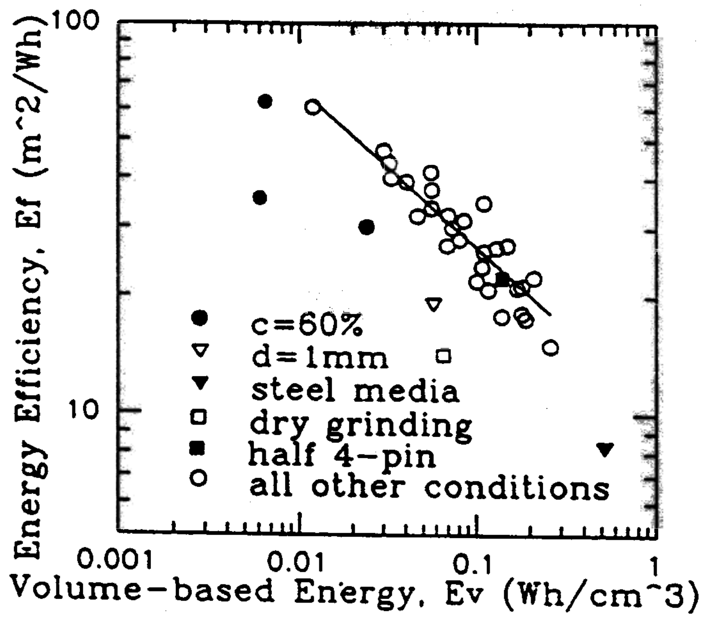


Figure 7: Relationship between energy efficiency and volume-based energy.

Specific energy (volume or weight based energy) has been used as the basic criterion for the scale-up of stirred media mills.[2,5,17] This specific energy is obtained by integrating average power over experiment time, from which the power number is calculated. Modified Reynolds numbers are calculated using

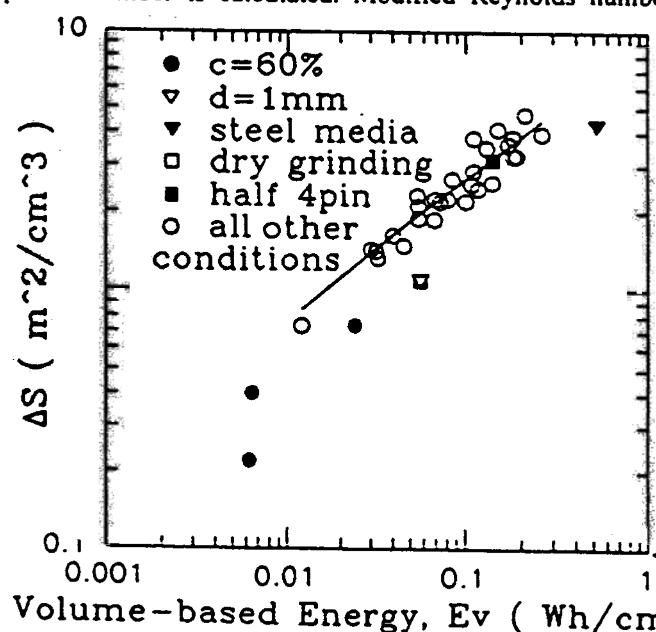


Figure 8: Relationship between increase of specific surface area and volume-based energy

equation (2) and the procedure developed earlier.[12] Finally, dimensionless group correlations of power and modified Reynolds number are established for relating the parameters influencing the power consumption for media, particles and liquid system in stirred mills.

MILL PHYSICO-CHEMICAL ASPECTS

The effects of chemical additives on grinding have been explained mainly by two kinds of mechanisms.[18] One mechanism is based upon alterations of surface and mechanical properties of individual particles, such as reduction of surface energy[19] and modification of surface hardness,[20] while the other considers the arrangement of particles and their flow in suspensions, for example, improvement of pulp fluidity.[21] These mechanisms have been examined for many tumbling milling cases.[22–24] However, very few examples concern stirred media mills.

It is important to ensure that chemicals used as grinding aids do not lead to detrimental side-effects. In this regard, ultrafine grinding of yttria stabilized zirconia in polyacrylic acid solutions studied in this laboratory using a Netzch batch attrition mill showed some important effects.[18] Relatively high grinding rates-as measured by production of new surface-were demonstrated using zircon media balls of 1.2 mm diameter. The absence of contaminating materials such as steel grinding media provides an additional advantage: high purity product is important in material preparation for high performance ceramic applications. Interestingly, it was found that the polyacrylic acid added as the grinding aid caused changes in the chemical composition of the product due to preferential extraction of yttrium by the polymer. This finding is shown in Fig. 9 where yttrium concentration in the supernatant is plotted as a function of equilibrium polymer concentration. It is noted that no dissolved yttrium was detected in the supernatant in the absence of polyacrylic acid. For both polymers, the yttrium concentration in the supernatant gradually

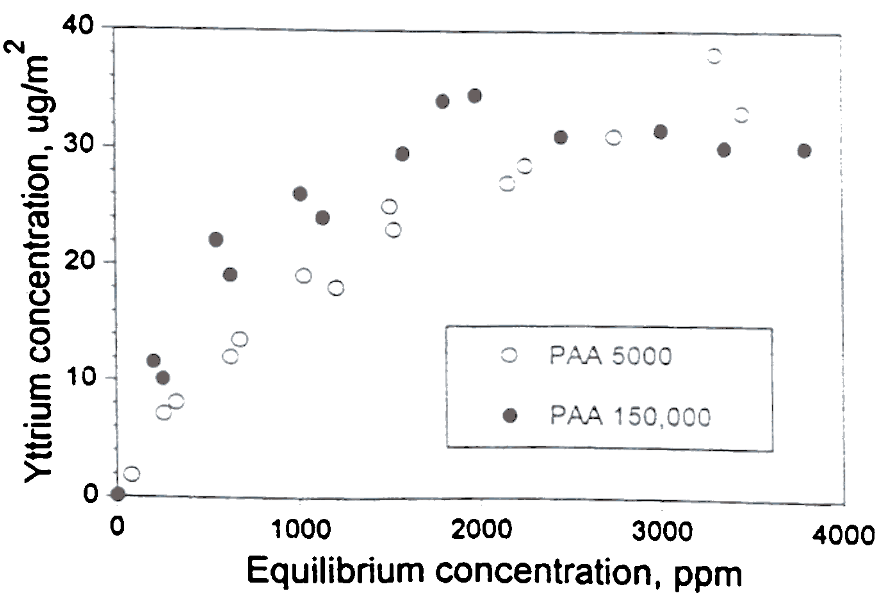


Figure 9: Amount of yttrium released by the surface of zirconia vs the equilibrium concentration of polyacrylic acid.[18]

increases with the residual concentration and then reaches a plateau. Reasons for this are not yet known even though complexation between polyacrylic acid and yttrium can be expected to play a role. The higher rate of yttrium extraction was observed for lower molecular weight polyacrylic acid in the kinetic study (see Fig. 10). Such interactions between polyelectrolyte and multi-valent ions are similar to those reported in the literatures.[25-26] A slight increase of adsorption at very low supernatant pH was observed, and this is in agreement with the expected release of hydroxyl ions upon the formation of the ion-polymer complex.

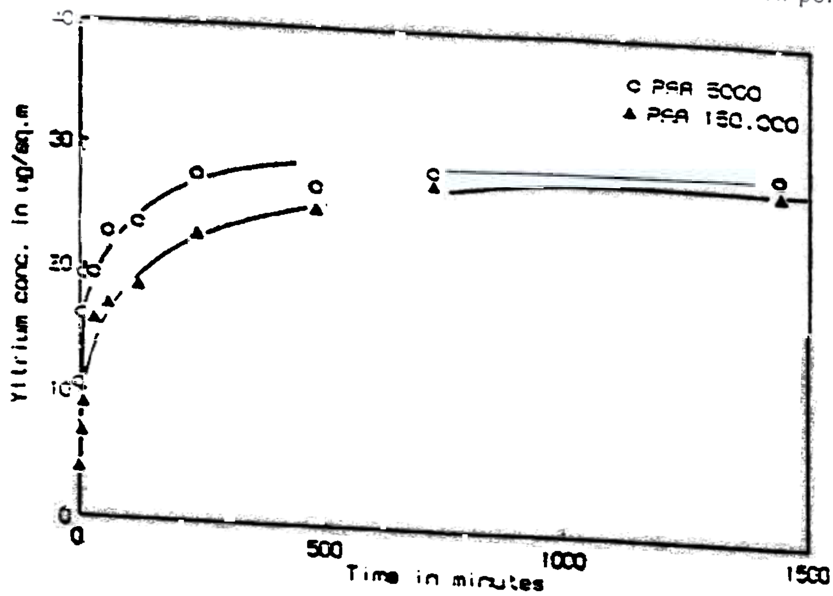


Figure 10: Kinetics of yttrium extraction from the oxide surface.[18]

CONCLUSIONS

Power characteristics of stirred media mills have been studied as a function of relevant operating and geometrical variables. The torque versus speed curve has been found to display four regions marked by sharp transitions: transition from static to dynamic friction, channelling, dispersing, and centrifuging. Equations, including power and modified Reynolds numbers, have been established for correlating the variables studied. Based on this work, scale-up guidelines have been proposed.

Power requirements and product characteristics of grinding limestone in laboratory-scale stirred media mills have been studied with respect to several operating variables. The best conditions for grinding limestone have been identified. Correlations between grinding and energy input for most conditions have been established using the relationship between energy efficiency and the increase of specific surface area respectively as a function of volume-based energy.

Polyacrylic acid as grinding aid in ultrafine grinding of yttria stabilized zirconia causes extraction of yttrium into solution with significant changes in the chemical composition of the product. Complexation between polyacrylic acid and yttrium is proposed to be the reason for this effect.

which stresses the importance of the effects, particularly those that are detrimental on downstream processes in the over-all industrial operation.

ACKNOWLEDGEMENT

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SYMBOLS USED

c Volumetric concentration of solids
C Impeller geometry factor constant
d Media particle diameter
D Impeller diameter
E Energy input
Ef Energy efficiency (= ΔS/Ev)
Ev Volume-based energy (= E/V)
K Consistency coefficient
n Power law index
N Impeller rotational speed
N_p Power number
N_{Re} Reynolds number
P Power
R Ratio of media to particle volume
S Specific surface area
ΔS Increase of specific surface area
t Grinding time
T Tank diameter
V Volume of ground material
α Impeller shear rate constant
ε_m Media packing porosity
ε_p Particle packing porosity
μ Viscosity
ρ Average media density
ρ_l Density of liquid
ρ_s Density of solid particle
τ Torque

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