AIR AGGLOMERATION OF HYDROPHOBIC PARTICLES*

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ABSTRACT

The agglomeration of hydrophobic particles in an aqueous suspension was accomplished by introducing small amounts of air into the suspension while it was agitated vigorously. The extent of aggregation was proportional both to the air to solids ratio and to the hydrophobicity of the solids. For a given air/solids ratio, the extent of aggregation of different materials increased in the following order: graphite, gilsonite, coal coated with heptane, and Teflon. The structure of agglomerates produced from coarse Teflon particles differed noticeably from the structure of bubble-particle aggregates produced from smaller, less hydrophobic particles.

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INTRODUCTION

A number of processes have been employed to separate hydrophobic particles from hydrophilic particles by the selective attachment of either gas bubbles or oil droplets or both to hydrophobic particles in an aqueous suspension. These processes have ranged from froth flotation of naturally hydrophobic solids and solids treated with oily collectors, to froth flotation of particles agglomerated or emulsified with oil, to separation of particles agglomerated with oil by screening. The fundamental physicochemical principles underlying various processes involving the application of oil including extender flotation, agglomeration flotation, emulsion flotation, and oil agglomeration have been reviewed extensively by Laskowski (1).

In the future the list of processes for separating hydrophobic and hydrophilic particles may include the air agglomeration of hydrophobic solids and recovery of the agglomerates by some means other than flotation, although flotation may still be the most appropriate method of recovery. This process will arise from the discovery that highly hydrophobic solids such as Teflon and gilsonite form stable agglomerates in aqueous suspensions when fine air bubbles are introduced into the vigorously agitated suspensions. The discovery arose from an investigation of the effect of air on the oil agglomeration process for cleaning coal which showed that small amounts of air promote the agglomeration process (2-4). During this investigation it was observed that the wetting and dispersion of highly hydrophobic, fine, dry particles in water was facilitated by vacuum degassing of the mixture of particles and water. Without degassing, the particles tended to float on the surface of the liquid, and the dispersion of the particles in the liquid was extremely difficult. Therefore, it was reasoned that once dispersed in water, such particles would have a tendency to collect at an air-water interface if a gas were dispersed in the liquid. This could lead to the formation of air bridges between particles similar to oil bridges between particles in oil agglomeration. Alternatively, the particles could collect around gas bubbles as in froth flotation.

To investigate these possibilities, several hydrophobic solids were selected for testing including Teflon, gilsonite, sulfur, Ceylon graphite, and Pittsburgh No. 8 coal. Aqueous suspensions of these materials in finely divided form were prepared by degassing and agitation, and then the agitated suspensions were treated by introducing air in a series of small increments. Evidence of particle agglomeration was obtained by observing the change in turbidity of a suspension. In addition, samples of the suspension were collected at the end of the experiment and examined with an optical microscope.

EXPERIMENTAL

A number of materials with varying degrees of hydrophobicity were utilized for this study including Teflon powder, gilsonite, Ceylon graphite, flowers of sulfur, and bituminous coal. The Teflon or polytetrafluoroethylene powder was obtained from Dupont which identified the material as No. 8 compression molding resin. The average particle size was 0.575 mm according to the manufacturer, and the particles appeared to range from 0.1 to 1.0 mm in size. When examined with a scanning electron microscope, the individual particles appeared to be compact, potato-shaped agglomerates or pellets composed of much finer flake-like particles (see Figure 1). Hence, the particles appeared to have been formed by some type of balling or agglomeration process.

The gilsonite and graphite were obtained from Ward's Natural Science Establishment. The gilsonite, which originated in the Uinta Basin of Utah, was ground with a high speed impact mill so that 99% passed a 0.180 mm screen. The graphite was also ground with a high speed impact mill, and the fraction which passed a 0.038 mm screen was utilized. The sublimed flowers of sulfur were obtained from Fisher Scientific Company, and a screened portion was used which was 85% below 0.038 mm in size.

The high volatile A bituminous coal came from the Pittsburgh No. 8 seam in Belmont County, Ohio. The dried coal had an ash content of 27% and total sulfur content of 5%. The coal was prepared by first crushing with a double roll crusher to produce particles smaller than 1.4 mm in size. This material was

Figure 1 — Electron photomicrograph of untreated Teflon particles.

mixed with water and ground in a stirred, stainless steel ball mill so that 99% of the product was finer than 0.038 mm in size. When the product was examined with a scanning electron microscope, the average particle size appeared to be about 10 μm . The product was partially dewatered with a Büchner filter funnel and then stored as a paste containing 56% solids in a refrigerator set at 5°C to minimize oxidation.

The hydrophobicity of the different materials was evaluated by measuring the three-phase, air/water/solid contact angle. The captive bubble technique was employed which required a smooth, flat surface. Gilsonite, sulfur, graphite, and coal were prepared by wet polishing lumps of the materials with No. 600 sand paper and then wet polishing with a fine cloth. These materials were rinsed subsequently with deionized water. A second sample of graphite was prepared by dry polishing. A piece of Teflon sheet was cleaned with Nochromix and then rinsed with deionized water but was not polished. The prepared samples were placed individually in deionized water and a small air bubble was introduced under the smooth surface with a syringe. Both the water advancing and water receding contact angles were measured with an NRL model 100 goniometer made by Rame-Hart. The contact angle was measured on each side of the bubble, and the measurement was repeated at six different locations on each sample. In the case of a second coal sample, the polished specimen was suspended for 15 min. in a suspension of fine coal particles, and then the slime-coated specimen was placed in deionized water and the contact angle measured in the usual way.

For conducting agglomeration tests the solid particles were suspended in deionized water having a resistivity of 17.9 megohm-cm. For some of these tests the coal was first treated with n-heptane obtained from Burdick and Jackson Laboratories which reported that the heptane had been distilled in glass and that it had a boiling point of 98-99°C.

The agglomeration tests were conducted with a specially designed, closed mixing system which consisted of a cylindrical tank fitted with baffles and an agitator together with equipment for monitoring the turbidity of the suspension. The tank had an inside diameter of 15.24 cm and height of 15.24 cm, and it was fitted with four vertical baffles. Each baffle projected inward a distance of 1.27 cm. Except for being slightly concave to facilitate drainage, the top and bottom of the tank were essentially flat. The walls of the tank and baffles were made of Plexiglas, whereas the top and bottom were made of stainless steel. The contents of the tank were stirred with a 5.05 cm diameter, Rushton-type, stainless steel, turbine impeller mounted on

a vertical shaft which was driven by a controllable speed motor. The impeller had six vertical flat blades mounted on a horizontal disk, and it was located 1.27 cm above the bottom of the tank. For the agglomeration tests the agitator was operated at 2400 rpm. The measured net volume of the mixing system fitted with baffles and agitator was 2870 cm³.

The turbidity of a suspension undergoing agglomeration was determined continuously by pumping a stream of material from the mixing tank through a tubular glass measuring cell where the light transmittance of the suspension was determined with a photometric dispersion analyzer (PDA 2000) manufactured by Rank Brothers Ltd. The suspension was then returned to the mixing tank. The suspension was removed through an opening in the side of the tank which was located 1.27 cm above the bottom and was returned through an opening in the top of the tank. A peristaltic pump was used for pumping the suspension.

To prepare a suspension for agglomeration, a measured quantity of solids was placed in the mixing tank and 2800 cm³ of water was added. The tank was sealed and connected to a vacuum pump. The suspension was degassed by applying a vacuum corresponding to -95 kPa for 20 min. Since the more hydrophobic materials including Teflon, sulfur and gilsonite tended to float on the surface of the liquid, it was necessary to apply the vacuum in short bursts followed by vigorous shaking of the tank in order to disperse the particles in the liquid. After degassing the suspension, the tank was topped off with previously degassed water, and any remaining gas bubbles were removed from the system.

To conduct an agglomeration test, a suspension containing 1-2% solids was first conditioned for 2 min. by operating the agitator at 2400 rpm. As agitation was continued, air was introduced in a series of measured increments to cause particle agglomeration. Small amounts of air (e.g., 1-3 cm³) were introduced with a syringe, while larger volumes were introduced by withdrawing corresponding amounts of water and allowing the water to be replaced by air. The progress of agglomeration was monitored with the photometric dispersion analyzer. After each increment of air was added, the output signal from the instrument increased due to an increase in the intensity of the transmitted light as a result of a decrease in turbidity. Between each addition of air the system was allowed to stabilize as indicated by a constant output signal. The process of introducing air was continued until it was apparent that considerable agglomeration had taken place. After some tests a sample of the suspension was collected and examined with an optical microscope. For the examination the sample was placed in a 3 mm deep by 25 mm diameter cavity of a specially made slide, and the agglomerates were viewed and photographed with an Olympus model SZH stereo microscope with camera attachment.

The above procedure was modified slightly for Pittsburgh No. 8 coal since the hydrophobicity of this material was insufficient for the material to be agglomerated by air alone. After preparing a suspension of the coal particles in the usual way, a small, measured amount of heptane was introduced, and the mixture was conditioned for 5 min. by operating the agitator at 2400 rpm. During conditioning the particles became coated with heptane, and an increase in the output signal from the photometric dispersion analyzer indicated that some particle agglomeration took place. Following conditioning, the agglomeration process was continued in the usual way by introducing small increments of air.

The turbidity (τ) of each particle suspension was determined by applying the following relation based on the Beer-Lambert law to the observed output signal from the photometric dispersion analyzer:

$$\tau = (1/L) \ln(I_0/I) \quad (1)$$

I represents the measured intensity of a narrow beam of light after passing a distance L through the suspension, while I_0 represents the intensity of a similar beam of light which has traveled the same distance through clear water. Since the turbidity is proportional to the number concentration and scattering cross section of the various particles which make up a suspension, it is a good indicator of particle agglomeration which produces a large decrease in overall particle concentration. For studying the effects of various parameters on agglomeration performance it has proved useful to determine the relative turbidity change

caused by a change in a given parameter (5). The relative turbidity change ($\Delta\tau_r$) in percent is defined by the following expression:

$$\Delta\tau_r = [(\tau_o - \tau)/\tau_o] 100 \quad (2)$$

where τ_o represents the initial turbidity of the unagglomerated suspension and τ represents the turbidity after agglomeration has taken place. It is apparent that as agglomeration takes place the relative turbidity change will increase while the absolute turbidity decreases.

RESULTS AND DISCUSSION

The measured three-phase contact angles for the air/water/solid system are listed in Table I for the various materials. The water advancing (θ_A), water receding (θ_R) and arithmetic average (θ_{AV}) values of the contact angle are shown. In each case the value represents the contact angle measured through the water phase.

The measured contact angles were in general agreement with values reported by other workers. The advancing contact angles of 109° for Teflon, 85° for sulfur, and 81° for dry polished graphite compared well with the corresponding equilibrium values of 112° for Teflon, 78° for sulfur, and 82° for graphite reported by Janczuk *et al.* (6) and also the reported equilibrium values of 108° for Teflon and 82° for Graphon (graphitized carbon black) determined by Chessick *et al.* (7). On the other hand, the measured values for sulfur and graphite were high compared to the values reported by Sun (8). However, the measured advancing contact angle of 89° for gilsonite compared well with the equilibrium value of 90° reported by Sun (8), and the measured advancing contact angle of 58° for Pittsburgh No. 8 coal was in line with equilibrium values reported for other high volatile A bituminous coals (9).

Although the cause of the large difference between the contact angles measured on wet and dry polished graphite has not been established with certainty, the results suggest that the surface of the dry polished material may have retained some air in pores, cracks, and crevices which made it more hydrophobic than the wet polished material.

While a clean, freshly polished surface of Pittsburgh No. 8 coal exhibited an average contact angle of 41° , a polished coal surface which had been suspended in a slurry of the finely ground coal and then resuspended in deionized water exhibited a contact angle of zero. The drop in the contact angle was due most likely to a strongly adhering slime coating of clay or other ash-forming material. This phenomenon has been reported previously (10).

To compare the response of different hydrophobic materials to agglomeration with air, a series of tests was conducted in which the relative change in turbidity caused by increasing amounts of air was determined for each of the materials. The results of these tests are presented in Figure 2. It can be seen that for each material except untreated Pittsburgh coal the relative turbidity change of the corresponding suspension increased as more air was introduced. Therefore, it is apparent that with the exception of untreated coal, air promoted the agglomeration of the various materials. Moreover the increase in relative turbidity change produced by a given air/solids ratio was roughly proportional to the hydrophobicity of the material as indicated by the three-phase contact angle. This relationship is also indicated by the data in Table I where the relative turbidity change corresponding to an air/solids ratio of 0.20 is listed together with the contact angle for the various materials. Teflon, being the most hydrophobic material, had the greatest response to the introduction of air. Gilsonite and sulfur being less hydrophobic than Teflon were less responsive, but the response of gilsonite was similar to that of sulfur which matched the contact angles for these materials. The response of graphite to air agglomeration appeared to be more in line with the contact angle measured with dry polished graphite than with that measured with wet polished graphite. Although untreated Pittsburgh coal did not respond to air agglomeration, this result may have been due to the presence of a slime coating on the surface of the coal particles since the material had a large ash content which probably was due in part to the presence of clay.

Table I. The Three-Phase Contact Angle and Relative Turbidity Change of Different Materials

Material	Contact Angle			$\Delta\tau_r^*$ (%)	Remarks
	θ_A	θ_R	θ_{AV}		
Teflon	$109^\circ \pm 3^\circ$	$86^\circ \pm 4^\circ$	98°	46.5	Cleaned/Not Polished
Gilsonite	$89^\circ \pm 5^\circ$	$43^\circ \pm 5^\circ$	66°	13.0	Wet Polished
Sulfur	$85^\circ \pm 5^\circ$	$45^\circ \pm 5^\circ$	65°	13.0	Wet Polished
Graphite	$81^\circ \pm 7^\circ$	$42^\circ \pm 6^\circ$	61°	7.0	Dry Polished
Graphite	$13^\circ \pm 5^\circ$	$13^\circ \pm 3^\circ$	13°	7.0	Wet Polished
Pitts. Coal	$58^\circ \pm 8^\circ$	$25^\circ \pm 5^\circ$	41°	0	Wet Polished
Pitts. Coal	0	0	0	0	Slime Coated

*Relative turbidity change corresponding to an air/solids = 0.20.

When the hydrophobicity of Pittsburgh No. 8 coal was enhanced by the addition of heptane, the response of the material to air agglomeration improved greatly and was greater than most of the naturally hydrophobic materials. Furthermore, the response was proportional to the amount of heptane added. The results suggest that the mechanism of attachment of a gas bubble to heptane-covered coal is similar to the mechanism of attachment of a gas bubble to a naturally hydrophobic solid. This suggestion is consistent with an earlier observation (11).

In order to study the air agglomeration mechanism, samples of agglomerated particles in water were examined and sometimes photographed with an optical microscope having a camera attached. The character of the agglomerates varied greatly depending on the hydrophobicity and particle size of the material. Examples of the agglomerates formed with Teflon particles by using small and somewhat larger amounts of air are shown in Figures 3 and 4, respectively. The agglomerates produced with a small amount of air tended to be smaller and consisted of fewer particles than those produced with a larger amount of air. The presence of air bubbles within the agglomerates or air bridges between particles cannot be seen. Nevertheless the presence of air within the agglomerates became obvious when a sample of agglomerates in water was warmed gently by the light applied to view the sample with a microscope. As the sample was warmed, air was expelled from several agglomerates causing gas bubbles to form on the surface of the agglomerates.

When smaller particles of a less hydrophobic material such as gilsonite were agglomerated with air, the structure of the resulting agglomerates differed from that noted above. In this case the small particles tended to cover the surface of gas bubbles which appeared to range from 0.050 to 0.50 mm in size. The particles tended to stabilize the gas bubbles, but with time some of the smaller bubbles coalesced to form larger, particle-covered bubbles. The small particles also tended to clump together forming numerous small but rather loose clusters or flocs which often appeared to be associated with small gas bubbles. It was not possible to discern whether air was involved in bonding the small clusters or flocs. However, when a sample of the agglomerates in water was heated gently on a microscope slide, the gas bubbles expanded and the particles associated with any given bubble formed a cluster attached on one side of the bubble.

Figure 2 — Results of air agglomeration tests of different materials.

Figure 3 — Appearance of Teflon agglomerates produced with a small amount of air.

Figure 4 — A Teflon agglomerate produced with a larger amount of air.

With an even less hydrophobic material such as graphite, it appeared that the particles were incapable of stabilizing gas bubbles long enough to collect a sample and view the bubbles with a microscope. Therefore, the nature of particle aggregation which took place with weakly hydrophobic particles was not determined.

From the general appearance of the Teflon particle agglomerates and knowing that air bubbles as small as 0.050 mm were present in an agitated suspension, it seems likely that the Teflon agglomerates were held together by an interfacial force analogous to the force that is thought to hold oil agglomerated coal particles together. To characterize this force, a model for air agglomeration is proposed which is similar to the model proposed by Good and Islam (12) for oil agglomeration.

For the purpose of analysis it is assumed that two particles in close proximity can be approximated by two smooth, parallel, flat plates. Gravitational effects are neglected because the gravitational force is much smaller than surface forces. If the plates are immersed in water and an air bubble is introduced between them, the air will displace water from a portion of the surface of each plate as shown in Figure 5, and a three-phase contact angle θ will be established where the solid, liquid, and gas phases meet. The line of contact between the phases will form a circle (having a perimeter p) on the face of each plate. A resolution of the forces due to interfacial tension is indicated in a direction perpendicular to the two solid surfaces, and the net force, f , between the plates is given by the expression

$$f = 2\gamma p \sin\theta \quad (3)$$

where γ is the air-water interfacial tension. The net force must be overcome to break the bond between the two plates. For contact angles between 0 and 90° , the net force will increase as θ increases. If an external force is applied which increases the separation distance d , θ will increase which will cause f to increase counteracting the effect of the applied force. Therefore, the system is inherently stable. On the other hand, for a contact angle greater than 90° , the net force will decrease as θ increases so the application of an external force causing d to increase will not be met by a greater resisting force, and the system will be unstable. For a hydrophobic solid exhibiting a contact angle greater than 90° , a stable system will result when a water drop forms a bridge between two flat plates surrounded by air. Contributing to the stability of any of these systems is contact angle hysteresis which permits θ to vary over a wide range without changing the area of contact between an air bubble or a water drop and a solid surface.

Figure 5 — An idealized bridge formed by an air bubble confined between two flat particles immersed in water.

When typical values of interfacial tension and contact angle are substituted into equation 3, it becomes apparent that the net force per unit of perimeter length (f/p) which must be overcome to separate two flat particles will be roughly similar for particles bonded by an air bridge in water as for particles bonded by a heptane bridge in water. At 20°C the surface tension of water is 72.9 mN/m and the interfacial tension between water and heptane is 50.2 mN/m (13). Previously it was observed that the receding contact angle for the heptane/water/graphite system is 113° based on Ceylon graphite which had been wet polished (14). Substituting this contact angle and the interfacial tension between heptane and water into equation 3 yields the following value: $f/p = 92$ mN/m. Substituting the advancing contact angles shown in Table I for wet and dry polished graphite together with the surface tension for water provides the following values: $f/p = 33$ mN/m for wet polished graphite and $f/p = 144$ mN/m for dry polished graphite. Since these values bracket the preceding value of f/p for oil-bonded particles, it seems likely that an aggregation of particles held together by air bridges in water could be as strong as an aggregation held together by heptane bridges providing other conditions such as particle size and relative amounts of bonding materials are similar or equivalent.

The difference between the structure of Teflon particle agglomerates and the structure of the bubble-particle aggregates observed with other materials seems due in part to the difference in particle size in relation to bubble size. Whereas the majority of the Teflon particles were much larger than the smallest gas bubbles, other particulate materials were much smaller than the smallest observed gas bubbles. Therefore, it was not possible for small particles of other materials to be bonded by air bridges in the way suggested above for the bonding of Teflon particles. The finer size hydrophobic particles tended to coat gas bubbles and form stable bubble-particle aggregates which were probably very similar to those observed in microbubble flotation (15). Future experiments will address the effect of particle size to bubble size to see whether it is possible to produce the type of agglomerates made with Teflon by using other hydrophobic materials which are similar in size to the Teflon particles employed in this work.

CONCLUSIONS

By treating degassed suspensions of various types of hydrophobic particles in water with increasing amounts of air while employing vigorous agitation, it was shown that particle aggregation or agglomeration was promoted by the addition of air. The extent of aggregation was proportional to the amount of air introduced per gram of solids, and it was also proportional to the hydrophobicity of the solids as indicated by the air/water/solid contact angle. In the case of coarse Teflon particles, relatively large agglomerates were produced which may have been held together by air bridges between particles. A model for this type of bonding is presented which is similar to a model proposed previously for explaining the bonding of oil agglomerated coal particles. In the case of other hydrophobic materials which were small in size compared to the size of gas bubbles, the particles tended to coat the surface of the gas bubbles and to form loose clusters or flocs which were associated with small gas bubbles.

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