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Effect of Agglomerate Pore Structure on Efficiency of Solid-Liquid Separation by an Agglomeration Technique: Use of a Model System*

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Abstract

Solid-liquid separation is often difficult to achieve when the solids are finely divided. When the liquid involved is a hydrocarbon, the economics of any process involving such a separation will often depend on the ease and efficiency of liquid recovery. A process is described in which an enhanced solid-liquid separation is achieved by means of a size enlargement technique requiring agitation with a second immiscible liquid, which preferentially wets the solid surface. Potential applications for such a process include removal of ash particles in coal liquification and separation of solids from solvent extracted oil-sands. In this work a model system has been used in an attempt to better understand the process mechanics and the factors which affect the efficiency of liquid separation.

INTRODUCTION

It is well known (1, 2) that particle size is an important factor in solid-liquid separation processes with "dewatering" becoming more difficult as particle size decreases. In previous work (3-6), results have been presented relating to improved separation of a bitumen-rich solution from solvent extracted oil-sand by using a particle size enlargement technique requiring agitation with a second immiscible liquid (7). While this method markedly improves surface drainage of the solids, some miscella is occluded with the agglomerates during the size enlargement process. The results of the earlier work demonstrated that the amount of this occluded miscella was markedly

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dependent on the porosity of the agglomerates being produced under given conditions. The major factors affecting agglomerate porosity were identified as particle size distribution, agglomerate size, agglomeration time, condition of the solid surface, agglomerate compaction forces, and the mechanism of agglomerate growth (4).

Because of the lack of homogeneity of oil-sands feed materials, results were sometimes difficult to reproduce, even for feeds with nominally similar characteristics. It was therefore considered useful to reevaluate the earlier results using a model feed system more reproducible from batch to batch, thus allowing a more quantitative assessment of the factors affecting the extent of suspending medium occlusion by agglomerated solids. The information generated may be applied to solid-liquid separations for a variety of difficult-to-handle solid suspensions.

EXPERIMENTAL

Materials

The particulate solid used was pulverized agricultural limestone (Domtar). Two grades were blended to give a broader particle size range. Partial size distributions for the finer fractions are shown in Table 1. A mixture of equal parts of these materials was used in all experiments. The suspending liquid used was Stoddard solvent (Imperial Oil Varsol). The physical properties of this liquid are listed in Table 2. Unlike the earlier oil-sands work, there was no bitumen present in the model system.

Apparatus

Similar equipment to that used in this work has been described in detail elsewhere (3); a schematic representation of the unit, originally designed for oil-sands extraction, is shown in Fig. 1. In the absence of bitumen, the extraction unit, comprising a rotating drum containing a charge (10.6 kg) of steel rods, merely served as a mixer. The speed of rotation (about 25% of the critical speed) was such that little or no comminution of the solid occurred.

Water was added to the extraction drum in amounts sufficient to flocculate or microagglomerate the particulate feed material, thus reducing carry-over of fine dispersed solids with the overflowing suspending medium (Varsol). The overflow stream was recycled to the drum to maintain a constant liquid

TABLE 1
Size Distribution of -200 Mesh Fraction

Sieve size (μm)	Cumulative weight percent passing	
	Beachville "fine"	Beachville "coarse"
44	67.0	7.4
53	74.7	8.2
74	91.8	15.5

TABLE 2
Properties of Stoddard Solvent
(suspending medium)

Distillation ($^{\circ}\text{C}$):	
1BP	156
50%	162
FBP	177
Component analysis (vol%):	
Paraffins	44
Aromatics	5
Naphthenes	51
General:	
Specific gravity ($15/15^{\circ}\text{C}$)	0.777
Viscosity ($\text{Pa} \cdot \text{s}$) (25°C)	0.00089

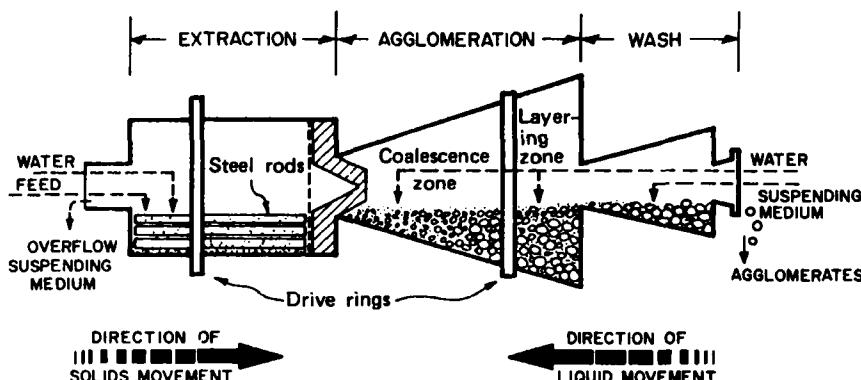


FIG. 1. Schematic representation of agglomeration apparatus.

level throughout the system. The solids were fed by a K-tron, microprocessor controlled, loss-in-weight screw feeder. Liquids were pumped by FMI reciprocating piston pumps. All streams could be controlled to $\pm 1\%$ of set values.

The agglomeration unit was a conical drum directly coupled to and rotating with the extraction unit. Provision was made to add water at two points in the drum, as shown in the diagram. Two drum sizes were used to test the effect of charge bed height on agglomerate composition. The altitudes and top diameters of the truncated cones were the same in each case, but with bases of 36.2 and 48.6 cm, respectively, giving angles of 14 and 23° when positioned horizontally as shown. The volumes of the drums were 22.5 and 36.4 L; in operation about 25% of this volume was occupied by the solid and liquid phases. Because of the large effect that agglomerate size has on liquid drainage, it was necessary to produce agglomerates at some preselected diameter, regardless of the operating conditions. This was accomplished by making provision for the variable addition of artificial nuclei to the system. Each added nuclei provided a growth center for the agglomerating particulate solid and by adjusting the ratio of nuclei to feed, the agglomerate size could be effectively controlled. For convenience, the nuclei used were 3 mm glass beads and addition at a ratio to the feed of up to 1:50 produced agglomerates of 1.0 ± 0.1 cm for all conditions tested.

Analysis

Before analysis, all samples were allowed to drain to constant weight in sealed jars. The agglomerates were analyzed for water and solids by using a Soxhlet extractor in conjunction with a water separator (9); the volume of water was measured at a known temperature and then converted to mass using density values from the literature. The residual suspending liquid associated with the agglomerates could then be determined by difference.

In some cases the organic suspending liquid was determined directly by carbon analysis using a LECO CR12 Carbon Determinator. Agreement between the two methods was quite good; for example, a comparative test of the two methods gave values for the organic content of $1.9 \pm 0.2\%$ by the carbon analysis method and $1.8 \pm 0.3\%$ with the extraction method. In view of the congruency of the results obtained for the two methods, the values obtained by difference were considered acceptable.

Treatment of Results

Because of natural fluctuations in the agglomeration process itself, there is some variability in the composition of the produced agglomerates, even when the feed streams into the system are themselves constant. Small changes in the water content of agglomerates can have a large effect on suspending medium retention. Therefore it was virtually impossible to determine directly a true equilibrium agglomerate composition for any given fixed conditions. Consequently, a composition curve, comprising a plot of water content versus solid content (wt%) was constructed for each run. The water concentrations were normalized for each set of conditions by determining moisture content on a dry weight basis using the ratio of water to solid content (*W/S*).

For the range of moisture content studied, the data could be best represented by straight lines using least squares techniques. Average compositions could then be interpolated from the least squares plots at a given value of *W/S*, thus allowing direct comparisons for different operating conditions. In order to broaden the range used for the statistical analysis of composition data, deliberate changes in the rate of water addition were made during each run. Agglomerate analyses made during the period immediately following such changes were ignored. The significance of differences between composition plots was determined by a variance minimization technique (10).

As the agglomeration process occurs under a blanket of suspending liquid, it seems reasonable to assume that any agglomerate pore volume not filled with bridging liquid will be saturated with the suspending medium (4); that is, the agglomerates will be essentially air-free. Consequently, the amounts of the liquid components present in the agglomerates may be used as an estimate of the pore volume. For example, Fig. 2 shows the close correlation between apparent density calculated from the composition of oil-sands agglomerates and the apparent density of the same materials determined by mercury displacement (11). A slope of almost unity and an intercept of close to zero indicate that the apparent densities determined by the two different methods are essentially the same. Porosity, actually the fraction of the total agglomerate volume occupied by liquid, was then calculated from

$$\varepsilon = \left(1 - \frac{Sp_a}{100p_s} \right) \quad (1)$$

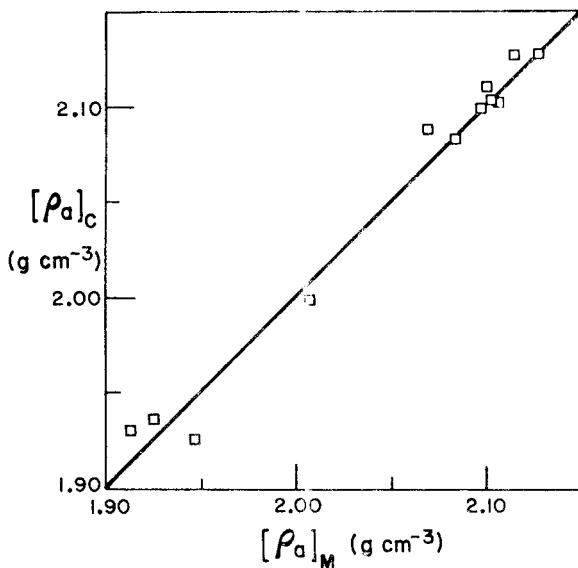


FIG. 2. Correlation between the apparent density determined by mercury displacement $[\rho_a]_M$ and that calculated from agglomerate composition $[\rho_a]_C$.

where ε = porosity, S = solids content (wt%) of agglomerates, P_a = apparent density of the agglomerates, and P_s = density of the solid component.

RESULTS AND DISCUSSION

The process of agglomerate formation is known (12, 13) to depend on the amount and distribution of the bridging liquid present in the system. Three distinct states of pore filling by bridging liquid have been postulated, namely the pendular, funicular, and capillary states, as represented pictorially in Fig. 3. Pendular bonding between particles, Fig. 3(a), occurs at low levels of bridging liquid and results in a loosely flocculated structure which collapses to a more compact form on addition of sufficient bridging liquid to give the continuous phase required by the funicular state, Fig. 3(b). At this point the particulate network still contains substantial amounts of entrapped suspending medium. Ultimately, the agglomerate pore volume becomes saturated with bridging liquid to give the capillary state, Fig. 3(c), which is usually considered to represent the most dense agglomerate form. As agglomerate

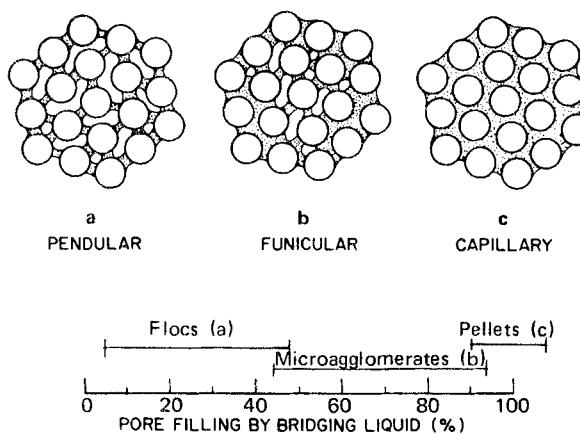


FIG. 3. Bridging liquid distribution in agglomerates.

strength depends on its density, there is also a very marked effect of water content on agglomerate strength.

Some possible growth mechanisms have been described elsewhere (8); a summary is given on Fig. 4. In the case of coalescence, Fig. 4(a), two or more agglomerates combine to produce a substantially larger entity. On further agitation or tumbling, the coalesced agglomerates again assume a spherical form. The layering mechanism occurs when free particles become attached to the surface of existing agglomerates, Fig. 4(b). This is the most controllable form of growth, resulting in optimum particle packing and agglomerate densification. The agitation required to drive the agglomeration process also creates turbulence which can result in agglomerate destruction. Because the smallest agglomerates are the weakest, this group is the most susceptible to breakdown. Broken pieces from this crushing process may be incorporated into larger agglomerates, as shown on Fig. 4(c). All three growth mechanisms can occur simultaneously, and the preponderance of any one mechanism will depend on the experimental conditions and the characteristics of the material being agglomerated.

BRIDGING LIQUID DISTRIBUTION

For conventional cylindrical balling drums, the total amount of bridging liquid is usually premixed with the solid feed to assure an ideal mix prior to agglomeration in the drum (15), thus giving maximum utilization of the drum

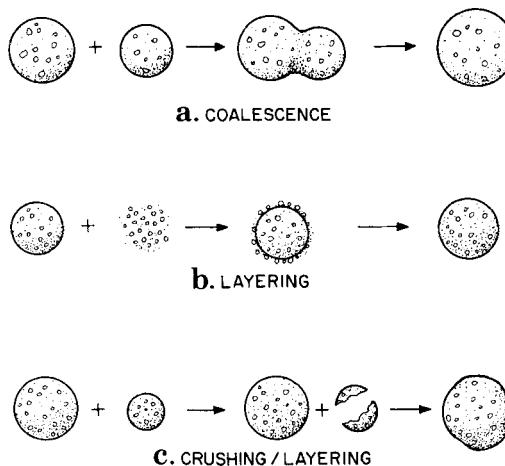


FIG. 4. Schematic illustration of agglomerate growth mechanisms.

space for balling. A cylindrical drum does not produce any size distribution of the charge and, as a result, it is necessary to screen the product and recycle the undersize to the drum. Material may pass through the drum several times before the desired pellet size is reached. In the case of balling disks, the action is such that the tumbling charge is segregated according to size, with the larger pellets being preferentially discharged in the product. Water is sprayed onto the charge during the actual balling process. The position of water and feed addition has a marked effect on pellet size. The conical-type agglomeration drum used in this work has attributes of both the cylindrical drum and the disk in that it combines both good rolling action and size segregation (15). Because of the latter feature, the addition of water to different zones of the conical drum should have significant effects on the agglomerated product in an analogous way to that found in balling disks.

Provision was made to add water to the agglomerating charge at three different points in the apparatus, corresponding to different growth regimens: 1) flocculation/nucleation zone, 2) rapid growth or coalescence zone, and 3) layering zone. The total amount of water added to the system, expressed as a ratio of water to solid, was usually maintained in the range 0.095 to 0.105, but the proportion added to the different zones was systematically varied.

Nucleation begins immediately after contact between the particulate solid and water. In this apparatus the initial water/solid contact could be made to occur either before or after entry to the mixer/extractor drum; that is, the

solid could be either water wet or solvent wet prior to commencement of agglomeration. Sufficient water was added at the mixer stage to ensure that most of the solids were microagglomerated, thus minimizing carryover of dispersed fines with the recycle stream of suspending medium. About 40–50% of the total water was required for this purpose. The remaining water was added to the agglomeration drum as follows: 1) split about equally between the coalescence and layering zones, 2) all added to the coalescence zone, or 3) all added to the layering zone.

A number of runs were carried out for each condition; the composition curves are shown on Fig. 5, 6, and 7, with the replicate runs being reproducible to within the experimental error. In order to simplify the calculations, the nondrainable suspending liquid, associated with the agglomerate surfaces and amounting to about 0.5 wt%, was assumed to be part of the internally trapped component. Comparison of the three sets of combined data indicated significant differences in the slopes of the regression lines, as illustrated in Fig. 8, where Curve 1 represents the split water case, Curve 2 the addition of water to the coalescence zone, and Curve 3 the addition of water to the layering zone only. Also shown, Curve 4, is the composition of solids over the same range of moisture content, but calculated on a suspending medium-free basis.

The most interesting feature of these results is the drastic change in slope of the composition curves which occurs as water addition is progressively switched from one zone of the agglomeration drum to the other, with increased slope resulting as the proportion of water to the layering zone was raised. The effect of change in slope of the composition curves on the extent of suspending medium entrapment is shown in Fig. 8 (inset). The curves show the normalized suspending medium content (sm/S) as a function of moisture content. The values were calculated by difference using inter-

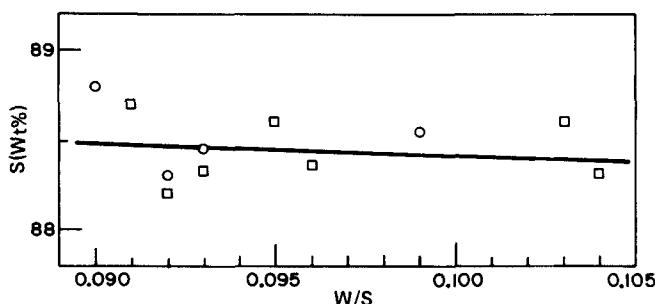


FIG. 5. Composition curves for agglomerates formed by coalescence mechanism for two runs.

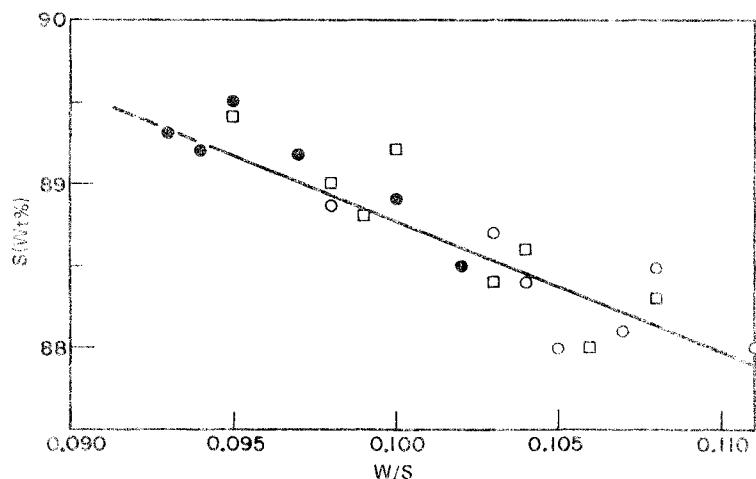


FIG. 6. Composition curves for agglomerates formed by layering mechanism for three runs.

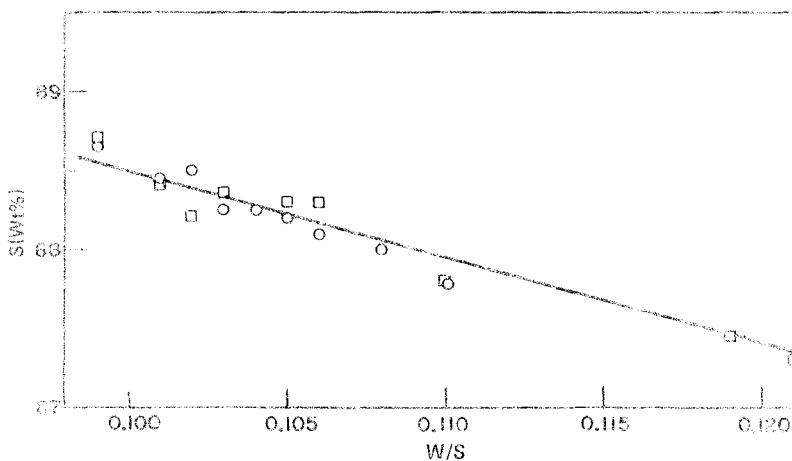


FIG. 7. Composition curves for agglomerates formed by intermediate mechanism for two runs.

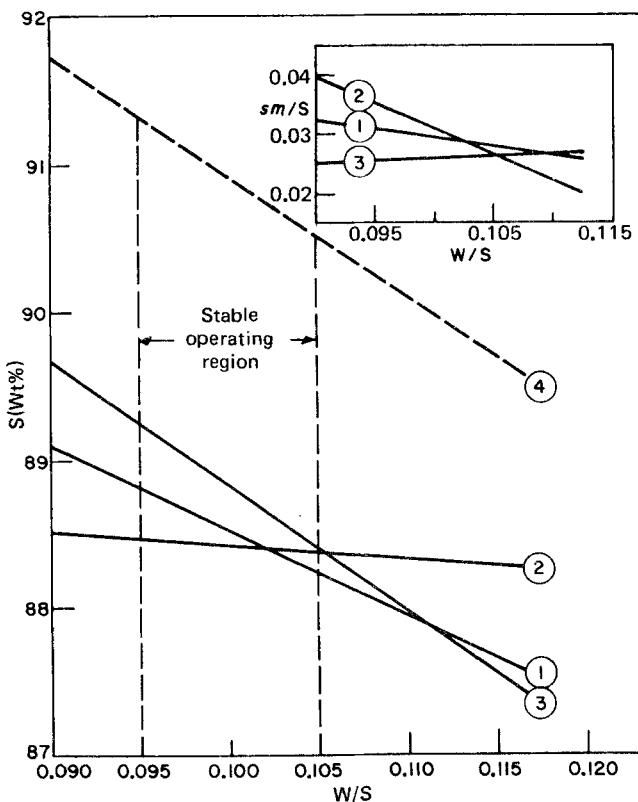


FIG. 8. Comparison of agglomerate composition curves for different growth mechanisms. Inset: Corresponding suspending medium retention levels as a function of bridging liquid content.

polated values of solids and moisture contents from the corresponding composition curve. As expected, the plots are the inverse of the solid composition curves, with the least solvent being retained by agglomerates with the highest solid content at the same normalized moisture content. Addition of the bridging liquid to the layering zone appears to be most effective for elimination of solvent at low moisture contents, whereas at high moisture levels, addition to the coalescence zone gives the best results. However, in the latter case it is necessary to work outside the most stable operating region to obtain a noticeable improvement. This effect on agglomerate composition may be explained, qualitatively at least, by a change in the mechanism of agglomerate growth as the point of water addition was changed.

The amount of bridging liquid required to form microagglomerates is much less than the void volume of the microagglomerated solids (13). As mentioned earlier, the void volume not occupied by bridging liquid will tend to be saturated by the suspending phase. Therefore, the microagglomerated solids transferred from the mixer to the agglomeration drum contain comparatively large amounts of entrapped suspending medium and tend to be in a "surface dry" condition. Thus, when water was added only to the layering zone, there was little tendency for microagglomerates to grow until they arrived at the point of water addition. Because the bridging liquid was added as a continuous stream rather than a spray, the resulting local high concentrations of water led to selective rapid growth of that part of the agglomerate charge in the immediate vicinity of the point of addition. This size increase, resulting from coalescence of microagglomerates, provided nuclei for subsequent agglomerate growth. The greater proportion of the remaining charge would then tend to be collected by the larger nuclei particles according to the proposed crushing/layering mechanisms. Because the bulk of the layering process occurred in a high compaction zone (greatest bed depth in the conical drum), the layered particles were packed to give minimum void space and thus maximum exclusion of suspending medium under the prevailing conditions. However, the agglomerate nucleus, comprising a number of relatively uncompacted, coalesced microagglomerates would contain significantly higher amounts of suspending medium compared to the compacted outer layers. This structure is illustrated in Fig. 9(a). This type of pore volume distribution is supported by the fact that smaller agglomerates contain a greater proportion of internally entrapped suspending medium than do larger agglomerates (3).

When all the bridging liquid is added to the neck region (coalescence zone) of the conical drum, most of the microagglomerated solids quickly attain a "surface wet" condition allowing them to combine, by coalescence, to form

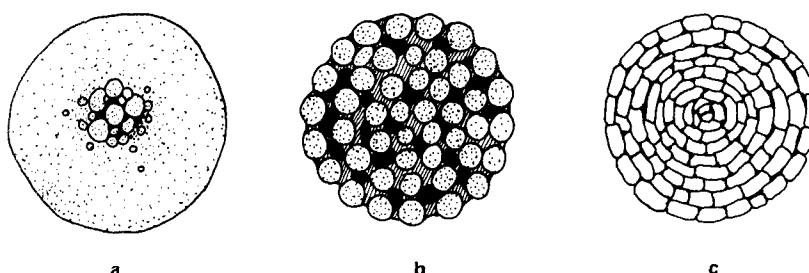


FIG. 9. Postulated structures for agglomerates produced under different conditions.

larger agglomerates very rapidly. Such coalesced agglomerates will therefore contain two types of porosity: 1) fine, interparticle pores due to the porosity of the microagglomerates themselves; and 2) coarser pores due to the porosity that occurs between microagglomerates when they coalesce, as shown in Fig. 9(b). This structure will be very dependent on the moisture content because of its effect on agglomerate strength. At lower moisture contents, agglomerates tend to be comparatively rigid as opposed to the plastic behavior observed at higher water levels. Therefore, at lower water content, one would expect the individual microagglomerates to resist deformation during compaction, thereby retaining their individual integrity and giving a higher value for the coarse porosity. Addition of more water will result in deformation of the microagglomerates, allowing them to conform to each other more closely (16, 17) in the compacted, coalesced state, as shown in Fig. 9(c). Thus the coarse type porosity will tend to be progressively eliminated as the moisture content is increased. Because suspending medium retention is related to this total internal porosity, the amount of entrapped nonwetting liquid will also be reduced as the porosity decreases. When the bridging liquid is split between the two addition points, a combination of the two mechanisms occurs and intermediate behavior is observed.

The objective of the separation step is to achieve a condition in which the agglomerate "pore volume" is entirely occupied by the bridging liquid. The degree of pore filling, actually the volume of water expressed as a percentage of the total liquid volume, over a range of moisture content is shown in Fig. 10 for the three modes of bridging liquid addition. The solid lines connect points of equal water content, and the intercepts with the broken lines occur at the corresponding pore volume and degree of pore occupation for the three sets of results. The degree of pore filling by water was found to be less than expected for pellet formation in the capillary condition (1).

For Case 3, water to layering zone only, the pore volume is initially lowest but increases as water is added to the system. This demonstrates that an increase in the water content has caused an expansion of the matrix structure rather than a displacement of the entrapped suspending medium. Thus there is only a small overall increase in pore filling. When agglomerate growth by coalescence is encouraged, Case 2, an increased water content actually results in a decrease in pore volume, possibly corresponding to the disappearance of macropores due to the collapse of the original microagglomerate structure. If the bridging liquid is divided between the two zones, Case 1, then intermediate behavior is again observed. This evidence suggests that displacement of the nonwetting phase from the agglomerate porosity, by increased bridging liquid, is most effective if accompanied by particle rearrangement to give decreased pore volume.

It would appear, therefore, that the best mode of operation would be to

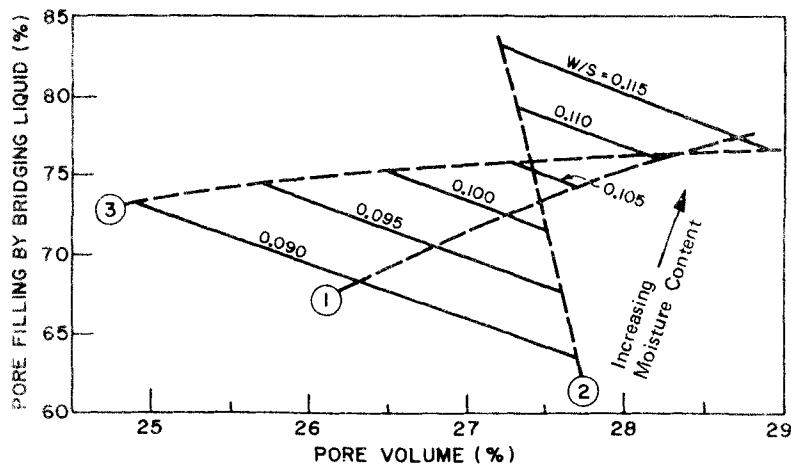


FIG. 10. Relationship between porosity and degree of pore filling by bridging liquid for agglomerates prepared by different growth mechanisms.

encourage coalescence growth at high water levels. Unfortunately, the moisture content required ($W/S > 0.105$) to yield an improvement is outside the region of stable operation for this material with the paste-like consistency of the agglomerates making them difficult to handle. An alternative would be to split the feed, with a minor portion being agglomerated by coalescence at high water levels to form substantial nuclei or seeds. The remaining bulk of the material could then be layered onto these cores at a lower water content to give optimum particle packing. This approach would have the advantage of reducing the quantity of solvent entrapped in the agglomerate core while maintaining the bulk of the agglomeration in a region of moisture content in which greater operational stability could be achieved. Alternatively, the use of solid seed particles onto which the particulate solid is built up under conditions of high compaction could be considered.

AGGLOMERATE COMPACTION

Residence Time and Drum Size

As discussed in the previous section, the retention of suspending medium by the agglomerated solids is related to both the total pore volume and the degree of pore filling by the bridging liquid. Other factors which affect the

pore volume are the bed depth of the tumbling charge and the residence time in the drum (13, 14, 18). Comparison of results from different size drums must therefore be made at the same residence time and vice versa.

A series of residence time tests were carried out in the two different sized agglomeration drums. Agglomerate composition curves at approximately the same residence time for the two drums are shown in Fig. 11. A summary of all results at different residence times is shown in Fig. 12, where Curves 1

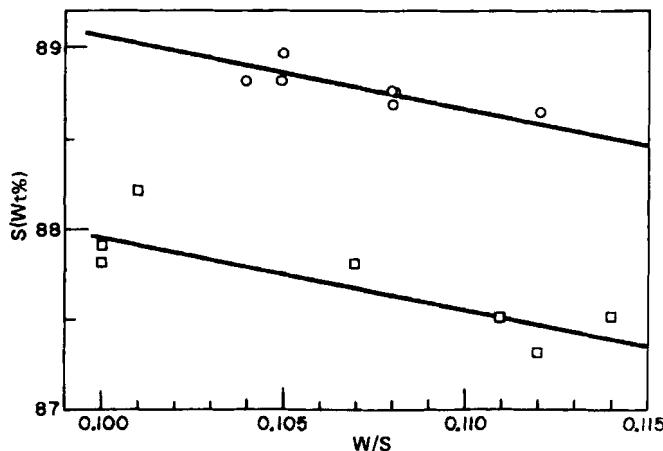


FIG. 11. Composition curves for agglomerates prepared in drums of different diameters: residence time 10 min.

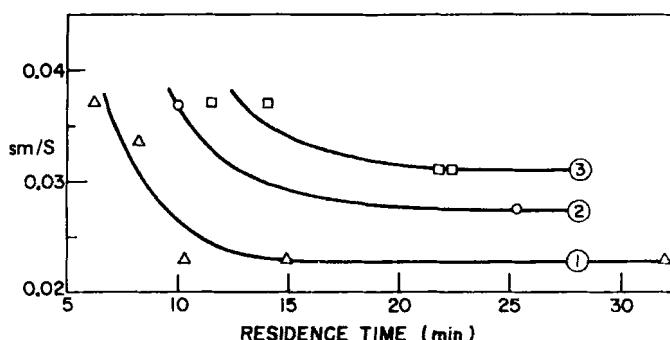


FIG. 12. Effect of residence time and bed depth on suspending medium retention by agglomerated solids. (1) High bed depth (water wet solids). (2) Low bed depth (water wet solids). (3) Low bed depth (oil wet solids).

and 2 were obtained for the larger and smaller diameter drums, respectively. As can be readily appreciated, there is a significant decrease in the retention of the suspending medium (sm/S) by the agglomerated solids processed in the larger drum. Obviously, the agglomeration process begins to break down as the residence time is reduced to less than 10 min. Ultimately, the curves should intersect at some critical residence time at which only micro-agglomerates are produced with the same characteristic suspending medium content. The value of the critical residence time will depend, to a great extent, on the rate at which the bridging liquid is adsorbed by the particulate solid. Where water is premixed with the solid feed, residence times of 2 min or less can be achieved for some systems (19).

Calculated values for the agglomerate pore volume and degree of pore filling by water were determined for the two cases (large and small drums). These results are summarized in Fig. 13. It is apparent that the increased compaction forces in the larger drum have resulted in lower pore volumes (Curve 1) as compared to agglomerates prepared in the smaller drum (Curve 2). More importantly, the degree of pore filling by the bridging liquid is significantly increased in the former case for the same W/S ratio. This

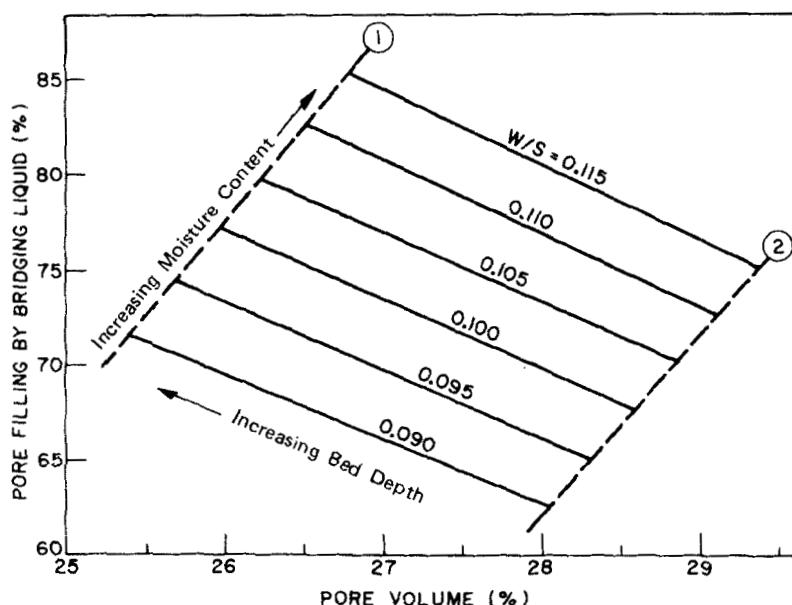


FIG. 13. Relationship between agglomerate porosity and degree of pore filling by bridging liquid as a function of bed depth and moisture content.

clearly illustrates that increased compaction has resulted in selective exclusion of the suspending liquid (4).

In geological terms, the *in situ* compaction of clay/silt sediments has been found by Athy (20, 21) to have a relationship of the form

$$\varepsilon = \varepsilon_0 e^{-ch} \quad (2)$$

where ε is the porosity at depth h , ε_0 is the porosity at the surface, and c is a constant term. This decrease in porosity is attributed to both mechanical and recrystallization effects, presumably with the former mechanism predominating at shallower depths. The mechanical compaction of agglomerates has been found to give a similar relationship (4) between agglomerate porosity and the depth of the bed of cascading solids. In fact, mechanical compaction of porous, particulate solid matrices has been described by a number of relationships between pressure and porosity; however, the equation of Athy is considered to be generally valid (22).

Assuming that such a relationship holds for the system investigated here and that the variable h may be equated to bed depth, then the constants of Eq. (2) may be estimated from the experimental results to give

$$\varepsilon = 35.1 e^{-0.018h} \quad (3)$$

Experimental determination of the porosity of the uncompacted solids (ε_0) gave values of $35.3 \pm 0.8\%$ and $34.6 \pm 0.6\%$ for dry and water saturated beds, respectively. These values may be compared to the calculated value of $\varepsilon_0 = 35.1\%$ obtained by Eq. (2). The close agreement between these calculated and measured values is an indication of the validity of the equation for this system in the range studied. Unfortunately, insufficient data were available for a rigorous statistical treatment of the results.

The composition of agglomerates having their pore volume completely filled with water alone may be determined from Curve 4 in Fig. 8 for any moisture level. The volume of water present in a given volume of agglomerates will then give the porosity for this ideal case. In this way a porosity of 21.7% was calculated at a *W/S* value of 0.100 for 100% pore filling. Substituting this value for porosity into Eq. (3) allows an estimate of the bed height necessary to achieve this degree of compaction to be made. A value of 26.7 cm was determined, corresponding to a drum diameter of about 75 cm. Similar results (4) have been obtained for a different particulate system using curve fitting techniques. It is obvious, therefore, that a large-scale test will be required to demonstrate the validity of the correlation.

Speed of Rotation and Drum Loading

The speed of rotation of the agglomeration drum can affect the size enlargement process in several ways. In terms of solid-liquid separation efficiency, the two most important factors are the mechanical energy input to the system and the drum loading. Both of these factors will have an effect on the degree of agglomerate compaction and hence on the retention of suspending medium. For example, in Fig. 14 are shown composition curves for agglomerates prepared under similar conditions, except that the drum speed was different in each case. At the lowest rotational speed the agglomerate composition indicates a much lower degree of compaction compared to the other cases which essentially gave the same result.

It is well known (23) that in a cascading charge of balls the kinetic angle of repose of the mass increases with the speed of rotation. This change can also affect the drum loading, as shown by reference to Fig. 15, where it is apparent that increased rotational speed has resulted initially in a larger hold-up of material in the drum (increased residence time). In order to facilitate removal of material from the drum, a skimming device or deflector was positioned in the exit port. This constraint prevented the charge level from increasing beyond this maximum point and explains why the residence time did not increase continuously with drum speed.

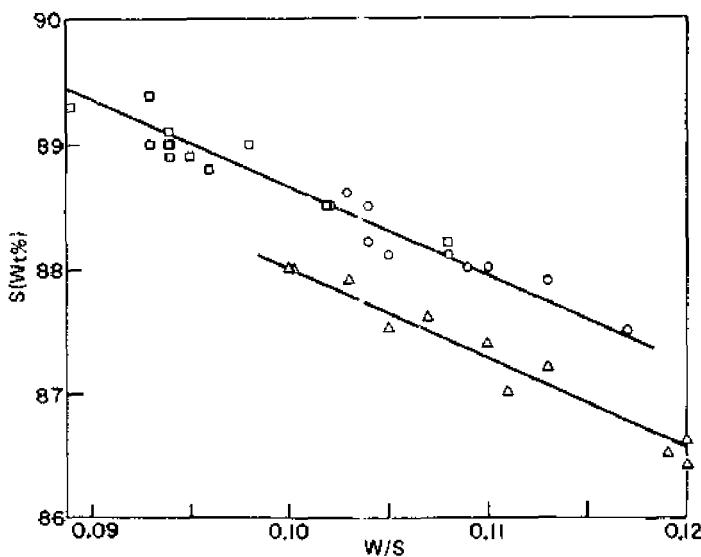


FIG. 14. Composition curves for agglomerates prepared at different speeds of rotation: 15 rpm (O), 12 rpm (□), 9 rpm (△).

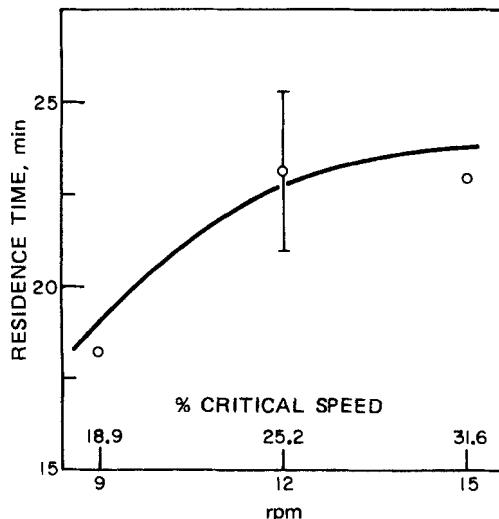


FIG. 15. Effect of speed of rotation on retention time in the agglomeration drum.

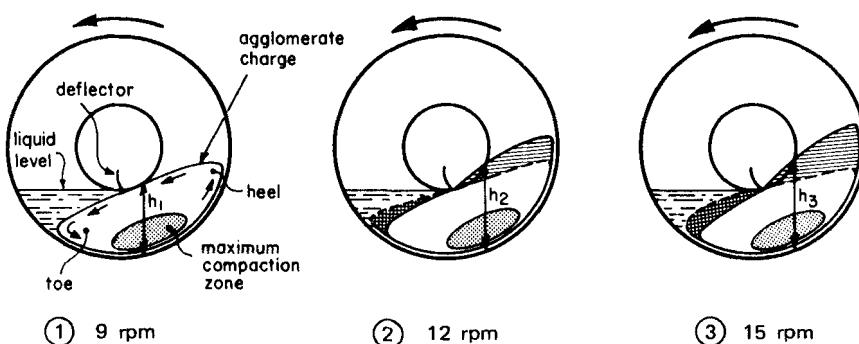


FIG. 16. Schematic representation of cross sections through the cascading agglomerate charge at different speeds of rotation.

Figure 16 is a qualitative representation of cross sections through the agglomeration drum illustrating observed charge distributions at different drum speeds. The zone of maximum compaction occurs in the region where agglomerates, being carried along by the drum wall, are contacted by agglomerates cascading downward from their highest point of travel and corresponds to the deepest cross section of the charge bed. As the speed of the drum is increased, the cross section through the charge changes as shown by the shaded areas in the toe and heel regions, with the center of the mass of the charge being displaced in the direction of rotation. The observed increase in charge hold-up as the drum speed was raised from 9 to 12 rpm therefore resulted in an increased bed-depth h_2 compared to h_1 . At 15 rpm the charge was carried even further up the side of the drum wall, but the positioning of the deflector plate prevented any significant change in the maximum bed-depth so that $h_2 \approx h_3$. The gain of charge mass in the heel region must therefore be compensated for by a decrease in charge mass at the toe as there was no significant change in the charge hold-up between the 12 and 15 rpm cases. Thus the increased agglomerate compaction observed was probably due to a bed-depth effect resulting from an initial increase in drum loading. The mechanical energy input into the system appeared to have little effect on agglomerate composition, as indicated by the congruency of results obtained at 12 and 15 rpm, where the bed-depth was similar.

Condition of Solid Surface

The analysis of results so far has presupposed that the solid surface is preferentially wetted by the bridging liquid, water in this case. This assumption may not always be true, as for example with oil-sands, where the solid surface may be contaminated by adsorbed bitumen fractions. Even in the model system considered here, problems of incomplete wetting may be encountered. On Fig. 12, for instance, Curve 3 was obtained under the same conditions as Curve 2, except that in the former case the solids were prewetted with the suspending medium and in the latter case with the bridging liquid.

The increased retention of the hydrophobic component in the case of Curve 3 must therefore be the result of incomplete displacement of this phase from the solid surface by water. Surface-active agents may be used to improve wetting of the solid particles, but any reduction in the interfacial tension will affect the agglomerate strength and therefore the agglomeration process itself.

CONCLUSIONS

It has been demonstrated that the "dewatering" of a particulate solid can be considerably enhanced by an agglomeration technique in which the suspending phase is selectively displaced from the agglomerate pores by a second, immiscible liquid which preferentially wets the solid surfaces. The efficiency of separation is markedly dependent on the porosity of the agglomerated solid and is increased by those factors, such as mechanical compaction, which tend to reduce the total agglomerate void volume during formation. The results obtained suggest that as the porosity is reduced, the remaining pore volume is preferentially occupied by the wetting phase, resulting in selective exclusion of the nonwetting phase from the porous solid matrix.

The pore structure of agglomerates has also been shown to be affected by the mechanism of agglomerate growth. This observation leads to the conclusion that separation efficiency is also dependent on the mode of addition of the bridging or surface wetting liquid.

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