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D. Murali^a; D. Bhattacharyya^a; R. I. Kermode^a

^a DEPARTMENT OF CHEMICAL, ENGINEERING UNIVERSITY OF KENTUCKY, LEXINGTON, KENTUCKY

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An Experimental Investigation of the Flotation of Residue Solids from Liquefied Coal

D. MURALI, D. BHATTACHARYYA, and R. I. KERMODE

DEPARTMENT OF CHEMICAL ENGINEERING
UNIVERSITY OF KENTUCKY
LEXINGTON, KENTUCKY 40506

Abstract

A semibatch flotation column has been used to investigate the feasibility of separating the mineral ash components from liquefied coal without the addition of a surfactant. The variables studied included the type of foaming gas and its flow rate, the ash concentration, the diffuser pore size, the length of foaming time, and the temperature profile along the column. For a suspension containing 4.6% ash, a separation up to 60% was achieved with a bulk liquid temperature of 110°C and a foam temperature of 65°C. A temperature gradient between the foam and liquid was essential if separation was to be achieved. The best separation was obtained with a 50- μm average pore size diffuser. Separation using smaller pore size diffusers was more erratic but statistically in the same range. Very poor separation resulted from a 150- μm diffuser.

INTRODUCTION

Heavy oil produced from the liquefaction of coal contains all of the inorganic ash-forming solids as well as any unconverted coal. Since coal liquefaction is generally carried out at a pressure of about $1.8 \times 10^8 \text{ N/m}^2$, separation of the lighter products can be achieved by several stages of pressure reduction and cooling. In each of these steps progressively higher molecular weight materials flash overhead while the remaining liquid becomes more concentrated with respect to ash and unconverted coal. When atmospheric pressure is reached, additional separation can be achieved by vacuum distillation.

TABLE 1
Analysis of the Solid Vacuum Bottoms^a

Moisture	0.41 % (weight)
Ash (mineral matter)	20.9
Volatile matter	55.8
Fixed carbon	22.9
Sulfur	0.89
Btu/lb value	13,000

^a Became liquid (highly viscous) at about 120°C.

The composition of a vacuum bottoms derived from an Illinois #6 coal by this type of process is shown in Table 1. This material, which is a solid at room temperature and becomes a viscous liquid upon heating, could be used:

- (1) Without modification as a source of hydrogen by partial oxidation
- (2) As a boiler fuel for ships if almost complete ash removal could be achieved
- (3) As a boiler fuel for large-scale electric power generation if the federal SO₂ emission standard could be met

The federal SO₂ standard, which is stated in terms of pounds of SO₂ per million Btu's, may be met either by lowering the sulfur content or by raising the Btu content of the fuel. Table 2 shows that if none of the sulfur is removed with the ash, then removal of 63.44 % of the ash will raise the Btu content sufficiently to meet the most stringent federal emission standard for a solid fuel of 0.6 lb of sulfur per million Btu.

The separation of fine ash particles (and unconverted coal) from the liquid product is a necessity in all coal liquefaction processes. The solid-liquid separation step is often a difficult and potentially expensive step (1).

TABLE 2
The Effect of Ash Content on Fuel Quality

Vacuum bottoms						
Ash content (%)	20.9	15.68	10.45	7.64	5.23	0
Ash removed (%)	0	25	50	63.44	75	100
Pound sulfur/10 ⁶ Btu	0.685	0.655	0.619	0.600	0.583	0.548
Btu/lb of fuel	13,000	13,580	14,370	14,830	15,250	16,250

The use of rotary drum precoat filtration, precoat leaf filtration, two-stage centrifugation, and solvent precipitation de-ashing has been reported. Filtration methods are often plagued by plugging problems, low filtration rates, use of expensive precoat materials, the difficulty of disposing or regenerating contaminated precoat material, and high capital investment. Batchelor and Shih (1) have reported the economics of the above processes and found the unit cost to vary in the range of \$2.66/bbl (rotary drum filters) to \$1.53/bbl (solvent precipitation de-ashing).

In the present study the removal by flotation of particulate matters (containing ash and unconverted coal) present in the liquid product prior to the vacuum distillation step of a liquefaction process has been experimentally investigated. The objectives are to determine the feasibility (without any addition of surfactant) of separating ash particles, and to establish the effects of key independent variables on the flotation process.

Literature Review

Flotation has in the past been used extensively for the separation of mineral matter during metallurgical processing and for the removal of suspended solids from various industrial wastes. Most of these separations have been carried out at room temperature from aqueous solution containing various surfactants and frothers as additives.

Factors that control the surface chemistry of various minerals in aqueous media and the kinetics of bubble-particle attachment have been reviewed (2). The effect of particle size on the efficiency of flotation had been studied by various investigators. A study (3) with 10 to 230 μm hematite particles showed that the rate of flotation was directly proportional to the square of the particle diameter. A similar study (4) with different particle sizes of glass spheres also reported the effect of particle size on flotation. For relatively large particles (150 μm), flotation rate was directly proportional to the square of the particle diameter. For submicron particles the flotation rate was reported (5) to be inversely proportional to (particle diameter)^{2/3}. For submicron particles, bubble-particle collision becomes difficult and results in poor flotation.

A study of the effect of concentration in metalliferous flotation concluded that the best separation resulted when the solid concentration was 20 to 30% by weight (6). For the separation of fine coal particles from pyrites and other impurities by flotation, 12 to 25 wt-% solids gave the best separation (7). Klassen (8) showed that the extent to which gas-liquid and solid-liquid interfaces were replaced by gas-solid interface was character-

ized by the amount of ash associated with the coal. The reduction in the flotability of coal with increased ash content has been verified, and the rate of flotation was considerably higher with coal of low ash content. These effects were attributed to the density change and surface characteristics of the particles.

It has been shown qualitatively by many investigators (4, 9) that smaller bubbles tend to increase the flotation rate. Industrial wastewater treatment plants tend to use very small bubbles, often with a diameter of 100 μm or less (10, 11), because the effluent stream contains very fine particles. Mineral flotation cells use comparatively large bubbles (500 to 2000 μm) because the pulps are in concentrated form. Very large particles are floated by a cluster of several bubbles (9).

The effects of temperature on flotation are very pronounced because of the temperature dependence of viscosity, surface tension, etc. (12). Certain minerals, such as molybdenite from copper and iron sulfides, were found to be best separated at a temperature of 75°C (13). In the industrial operation of fluorspar flotation plants, boiling the pulp produced the best separation of fluorspar from silicious minerals. An increase in the temperature of the slurry was found to improve the flotation of fine coal particles slightly (14, 15). Temperature was also reported to reduce the stability of the foam due to the change in the surface and drainage characteristics of the liquid (16).

Although the literature survey did not uncover any references on organic-based flotation, variables such as particle and bubble size, slurry concentration, and temperature would be expected to be important regardless of the type of liquid.

EXPERIMENTAL

Apparatus

The 3.4-cm i.d. glass flotation column shown in Fig. 1 was used for all the experiments. The three foam sampling spouts, located at 12, 16, and 20 cm from the bottom of the column, provided a 2 to 10 cm variation of foam height above the liquid height of 10 cm. Feed and liquid sampling spouts were located near the bottom of the column. Since experiments were carried out at elevated temperature, the column was wrapped with heating tapes. The lower tape was wrapped around the liquid section and the upper tape around the foam section of the column. Two variables were independently adjusted to maintain the desired difference in tem-

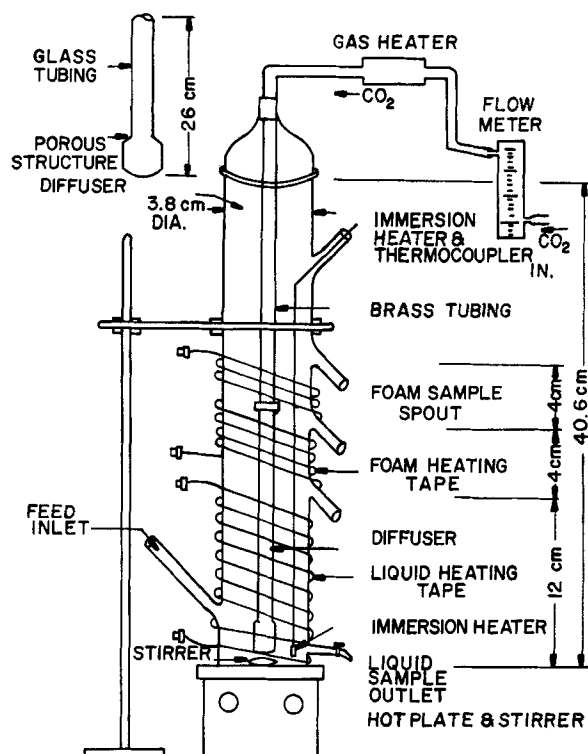


FIG. 1. Flotation column schematic.

perature between the liquid and foam. The predetermined Variac settings were not changed during a run. Finally, a heat exchanger in the carbon dioxide line could be used to heat the flotation gas between ambient and 110°C.

A small immersion heater in the liquid, which was actuated by an on-off controller connected to a thermocouple, kept the liquid temperature within $\pm 0.5^\circ\text{C}$. Two other thermocouples were used to record the foam temperature at two levels. A thermocouple inside the diffuser tube indicated the temperature of the gas.

The liquid was slowly stirred with a magnetic stirrer to prevent settling of particles. A calibrated rotameter was used to measure the gas flow rate through the 1-cm o.d. diffuser which had a porous length of 2.5 cm.

The Flotation Liquid

The number of potential variables in flotation using an organic medium is considerably greater than an inorganic medium, such as water. In particular, the temperature and pressure range that must be considered is very large. Solids in a liquefaction process are present in streams ranging from ambient conditions to 800°F and 2500 psi. Changes in temperature can change physical properties such as the viscosity of both the liquid and foam dramatically. Because of the multicomponent nature of the liquid phase, changes in temperature will alter the vapor-liquid equilibrium which in turn will modify the composition of both the liquid and foam. The analytical difficulties associated with describing these compositional changes are numerous and almost overwhelming when compared with aqueous systems. In addition, the somewhat unstable character of coal liquids leads to discernable changes in physical and chemical properties on a day-to-day basis.

It was therefore decided that for the initial phase of this research, some of the variables associated with coal liquids had to be eliminated or at least minimized. This was accomplished by using a petroleum-derived light cycle oil (LCO) plus 6% cresols. Some properties of these liquids are shown in Table 3. This mixture was chosen because physical properties such as boiling point, density, and viscosity were similar to the liquid product from an atmospheric still in a coal liquefaction process. The liquid contained no asphaltenes. Because of the stability of LCO, enough could be stored in 55 gal drums initially to complete all the runs. The use of this mixture insured that the starting organic flotation liquid was the same for all runs.

TABLE 3
Analysis of the Light Cycle Oil (LCO)^a

Saturates	30%
Olefins	12%
Aromatics	58%
Sulfur	0.2%
Boiling range	205–350°C
Viscosity	20.6 cP (at 25°C), 4.0 cP (at 100°C)
Surface tension	39.5 dyn/cm (at 25°C), 25.2 dyn/cm (at 100°C)
Specific gravity	1.061

^a Technical grade cresol: *m*-cresol, 54%; *p*-cresol, 29%; other phenols, 17%.

Mineral Matter—Hydrocarbon System

Table 1 shows the proximate analysis of a coal (Illinois #6) derived from a vacuum still bottoms product. This material contains most of the original coal mineral matter as shown by the 20.9% ash content. A sufficient quantity of this material was available to carry out all the experiments in this phase of the study. Before mixing the liquefaction solids with the organic liquid, they were ground to pass through a 150-mesh screen. Because of the low softening point of this material, care was taken to avoid high temperatures in the grinder. This was accomplished by hand feeding a very small quantity of solids at a time. Precautions were taken to minimize weathering of both the coarse and +150 mesh material by storing in dry sealed containers.

Operating Procedures

A flotation run was begun by preparing two identical batches of the solids-liquid mixture. Two predetermined weights of +150 mesh vacuum bottoms were mixed with two equal volumes of the LCO-cresol liquid. One batch was poured into the column shown in Fig. 1 while the other was kept in a beaker. Both batches were brought to temperature simultaneously and kept at this temperature for a predetermined length of time. Settling of the solids was prevented by a magnetic stirrer. The purpose of this dissolution period was to dissolve some of the vacuum bottoms in the LCO-liquid and liberate mineral particles for flotation. The initial dissolution period was set at 1 hr. Subsequent experiments showed that for the material being used, times as short as 15 min could be used without affecting the extent of separation. Most of the reported runs, however, used a 30-min dissolution period.

Samples of the liquid and foam were taken at predetermined times. A volume equal to the liquid plus foam collected was added from the feed beaker immediately after sampling. Thus all of the results are for a semi-batch constant volume process. The samples of liquid and foam were collected in weighted crucibles, dried slowly at 200°C to remove the solvent, and then ashed at 800°C.

After termination of a run the diffuser was removed from the liquid, cleaned thoroughly with hot LCO, and then bubbled continuously in LCO at room temperature until the next use. The transparent nature of the LCO was utilized for visual inspection of the bubbling characteristics of a used diffuser before each run. Visual changes in the bubble size or fraction of

the fritted area emitting bubbles were used to decide when to change diffusers. The character of the vacuum bottoms material virtually insured that some plugging of the diffuser would occur. Although all the diffusers were treated the same, some plugged after only one run while others lasted for as many as nine runs.

Preliminary Experiments

The lack of prior investigations using organic liquids meant that some preliminary studies had to be carried out to establish a set of conditions under which measurable separation of mineral matter by flotation occurred. Initial experiments were necessary to establish the foaming characteristics of the mixture in terms of the percent cresol, temperature, flotation gas, and gas flow rate. The amount of cresol was initially set at 6.0%. This quantity produced physical properties similar to those of a stream in the liquefaction process which contains mineral matter. Viscosity measurements of the liquid (17) indicated that a temperature of at least 100°C was necessary to reduce the viscosity sufficiently. Experience with aqueous systems indicated that a single diffuser with a pore size range of 40 to 60 μm would provide adequate bubbles in a reasonable size range. Three gases (air, nitrogen, and carbon dioxide) were considered initially. Preliminary flotation runs indicated that carbon dioxide, in addition to being inert and readily available, yielded a stable foam and separated a measurable amount of solids. The effect of CO_2 flow rate on foam height and foam stability was also established (17).

Table 4 shows the conditions finally settled on as satisfying the criteria of providing reasonable separation. The same table shows the variables considered during the main experimental program as well as their range.

Although it is not evident from the runs shown in the next section, considerable initial difficulty was experienced in achieving separation. The original design of the apparatus provided for isothermal conditions. That is, the liquid and foam sections of the column were both wrapped with heating tapes. The foam heating tape was adjusted to keep the foam temperature the same as the liquid. The CO_2 gas was preheated and entered the mixture at the temperature of the liquid. Over a temperature range of 70 to 140°C, no measurable separation occurred. Considerable time was spent investigating other types of temperature conditions. Ultimately, heating was confined to the liquid only. The foam temperature without heating established an equilibrium some 30 to 40°C below that of the liquid. Separation did take place under these circumstances.

TABLE 4
Experimentally Investigated Variables

Variable	Range	Standard condition
(1) Type of gas	N ₂ , CO ₂ , air	CO ₂
(2) Gas flow rate	100 to 1000 cc/min	390
(3) Ash solids in liquid	2.2 to 7.5% by weight (10 to 30% by weight vacuum bottoms)	(20%)
(4) Mean diffuser pore size	6, 15, 50, >150 μ m	40–60 μ m
(5) Cresol added	0, 3, 4, 6.7, 13.4 cc	6.7 cc
(6) Time of run	160 min max	30
(7) Temperature of foam	Heated, unheated	Unheated
(8) Temperature of liquid	70–125°C	110
(9) Dissolution time	0 to 60 min	30

EXPERIMENTAL RESULTS

The extent of ash separation from the liquid during flotation is defined as:

$$\% \text{ ash separation} = \frac{\text{initial ash concentration in the liquid} - \text{ash concentration in liquid at time, } t}{\text{initial ash concentration in the liquid}} \times 100$$

Liquid loss as collapsed foam was only 5 to 10% of the initial liquid volume, and thus the percent ash separation based on concentration is an adequate criterion for determining flotation efficiency.

As with most flotation experiments, considerable qualitative information could be gained by careful observation of the foam during the progress of a run. For poor (less than 20%) separation the foam appeared watery and collapsed almost as soon as it entered the spout. As the percentage solids rose, the foam appeared thicker and did not collapse in the outlet spout.

For successful flotation of solids, a minimum stable foam height was maintained during the progress of a run by proper adjustment (within a predetermined range) of the gas flow rate. An ensuing period of unstable foam always resulted in decreased separation.

Another difficulty was the accumulation of particles near the outlet spout. The asphaltic nature of the vacuum bottoms in the thicker foams caused some particle sticking on the column surface. In some runs, solids would build up for a period of time near the top of the foam. During this

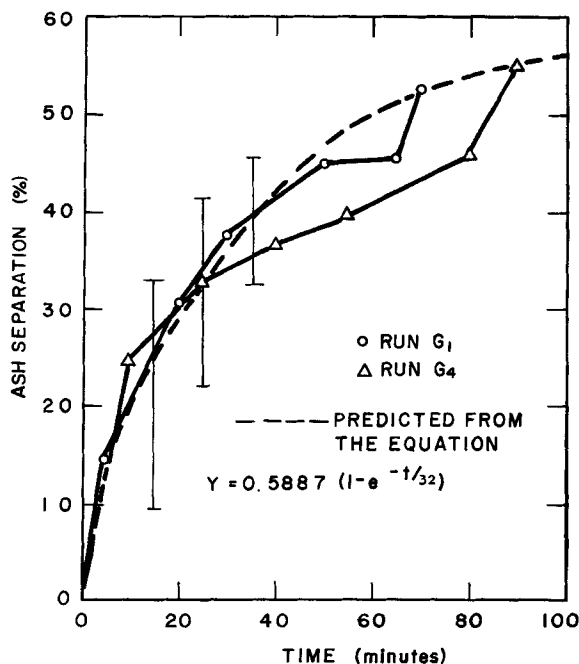


FIG. 2. Transient semibatch separation curves.

time, samples would show lower separation. For subsequent samples some of these accumulated solids might be washed out with the foam. This would result in an abnormally high degree of separation. On several occasions individual samples showed separations above 60%. These were attributed to high foam stability and some washing out of the accumulation of solids near the sampling spout.

Figure 2 shows typical flotation results for Runs G₁ and G₄. These two runs were carried out under the standard operating conditions shown in Table 4. Run G₁ used a new diffuser labeled G while Run G₄ was the 4th run on that same diffuser. No difference in diffuser behavior is shown even after four runs. A total of 75 runs of this type was carried out. Most of the successful runs were terminated before 60 min; however, one run was 160 min in length. The maximum separation during this time period exceeded 60%.

Because of the difficulty in obtaining identical separations in similar runs, it was necessary to establish confidence limits on the extent of

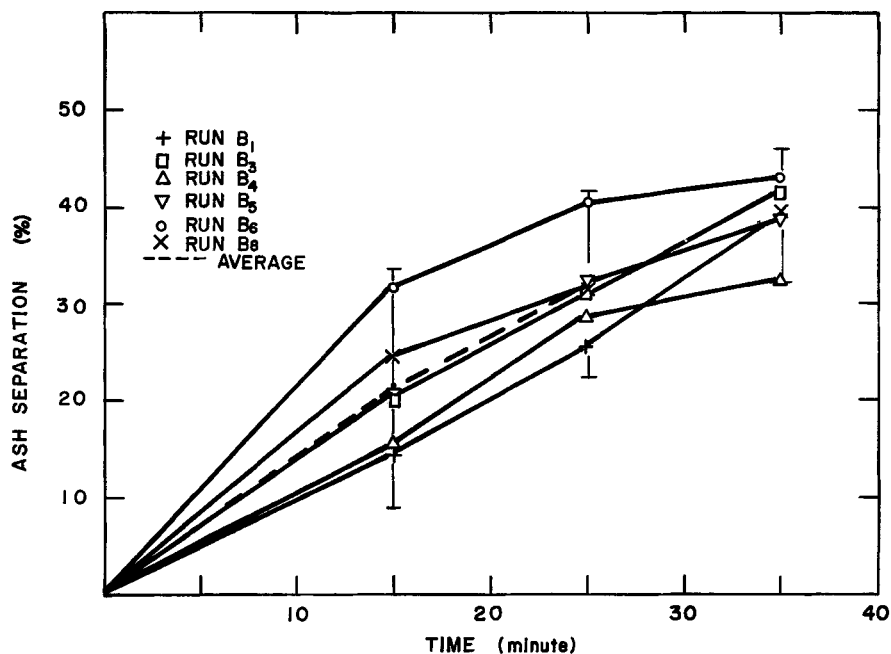


FIG. 3. Transient separation curves for the standard 40–60 μm diffuser.

separation as a function of time. One of the 40 to 60 μm diffusers used in this study functioned satisfactorily for nine runs, six of which were under the standard conditions. The six transient curves up to 35 min for Diffuser B are shown in Fig. 3. No systematic change in the diffuser is evident in these data. The average values and two standard deviations are shown in this figure. These average separations are 21.2, 31.9, and 39.2% with standard deviations of 5.8, 4.9, and 3.4% for 15, 25, and 35 min, respectively. This same band is also shown in Fig. 2 and clearly indicates that Diffuser G performed the same as Diffuser B. Unfortunately, insufficient runs with the same diffuser were carried to the point where the inherent standard deviation in the steady-state measurement could be estimated. However, the 35-min data indicate that it should be less than 3.4%. On subsequent figures, for comparison purposes, the average curve and the two standard deviation limits are shown.

The average line for Diffuser B shown in Fig. 3 was fit using an equation of the form:

$$y = A(1 - e^{-t/\tau})$$

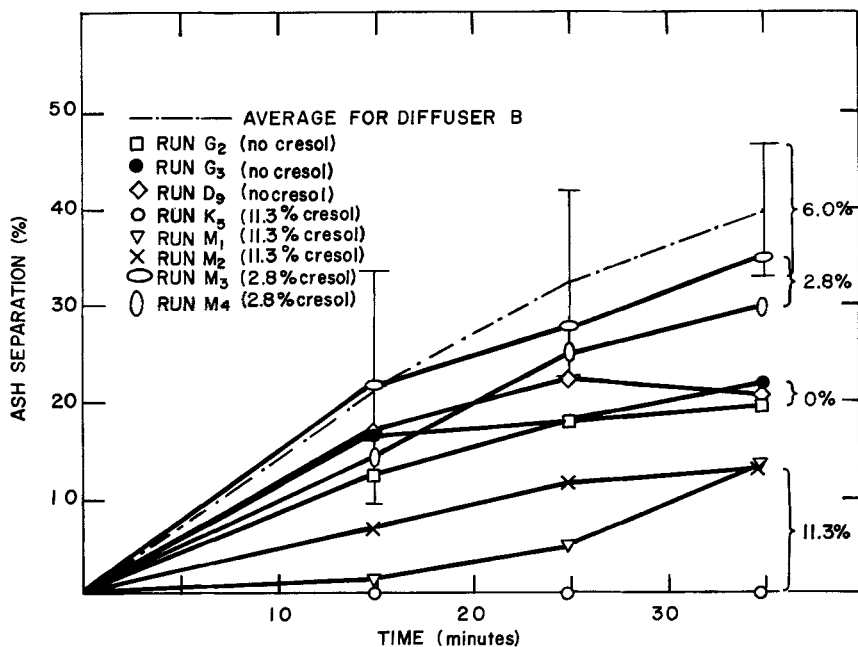


FIG. 4. The effect of cresol on the extent of ash separation.

where y is the conversion at any time t , and A and τ are the asymptotic conversion and time constant, respectively. Since there are data points at $t = 15, 25$, and 35 min, any two can be used to solve for A and τ . Using the $t = 15$ and 25 min points gave $A = 0.749$ and $\tau = 45$ min while using $t = 25$ and 35 min points gave $A = 0.5887$ and $\tau = 32$. The second set of values are plotted on Fig. 2 up to times $t = 100$ min for comparison with the Diffuser G results.

Figure 4 shows the effect of changing the amount of cresol added to the LCO on the extent of separation. All of the data points for the 11.3% cresol in the mixture lie outside confidence limits on the 6.0% cresol runs. A somewhat higher separation was achieved using no cresol, still less than the standard runs. Two curves are shown using a 2.8% solution. While it appears that these results are lower than the standard runs, conclusive statistical evidence is lacking. The importance of cresol is strengthened by remembering that standard Runs G₁ and G₄ were made before and after the 0% cresol Runs G₂ and G₃. Figure 2 shows that both of these runs were comparable with the results achieved from the standard Diffuser B. Similar standard runs were not made for Diffusers K and M.

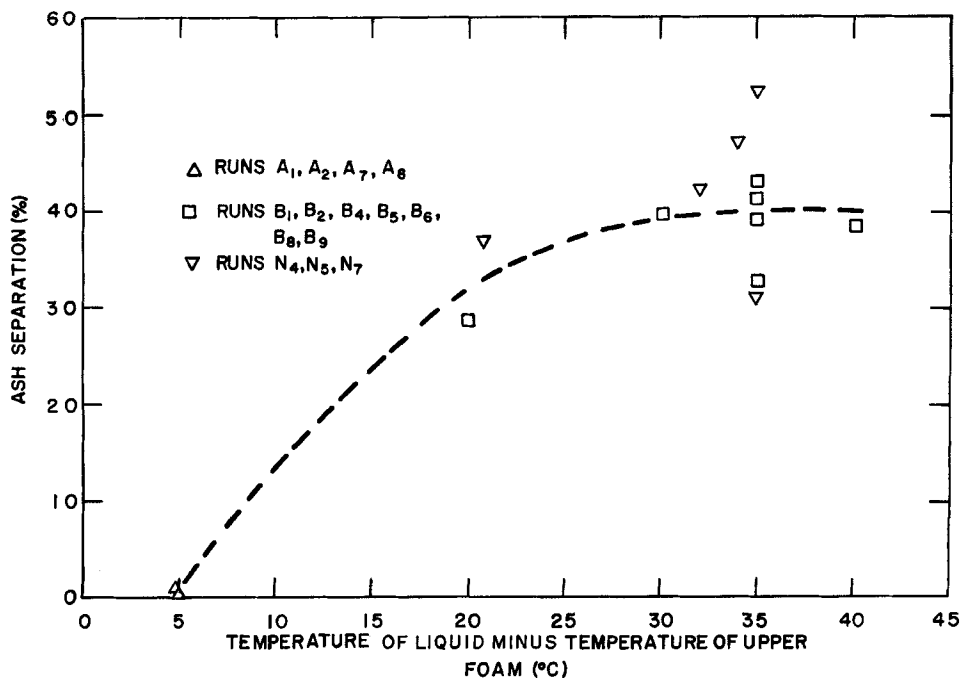


FIG. 5. The effect of the difference in foam and liquid temperatures on the extent of ash separation.

As previously mentioned, numerous attempts were made without success to separate ash under isothermal conditions. That is, the liquid, foam, and carbon dioxide were all maintained at the same temperature. Figure 5 shows the influence of the difference in temperature between the top of the foam and the liquid on the separation at $t = 35$ min. All these runs were carried out with an initial dissolution time of 60 min and a liquid temperature of 110°C . The dotted line shows the trend of the data; statistically, no difference exists for any $\Delta T > 20^{\circ}\text{C}$. Under standard conditions a $\Delta T = 35^{\circ}\text{C}$ was considered normal. Runs with a $\Delta T < 30^{\circ}\text{C}$ or $\Delta T > 40^{\circ}\text{C}$ were rejected. The three $t = 35$ min data points shown for Diffuser N appear higher than for the reference Diffuser B. The 15, 25, and 35 min separations for these three runs are N_4 (9.7, 31.1, 42.7); N_5 (28.5, 43.0, 47.5); and N_7 (27.5, 45.2, 52.7). The data points for Runs N_5 and N_7 are all near or above the corresponding $\bar{X}_t + 2\sigma_t$ for Diffuser B. N_4 shows a somewhat lower separation; however, this is strongly influenced by the very poor initial rate of flotation. In spite of this the separations at $t = 25$

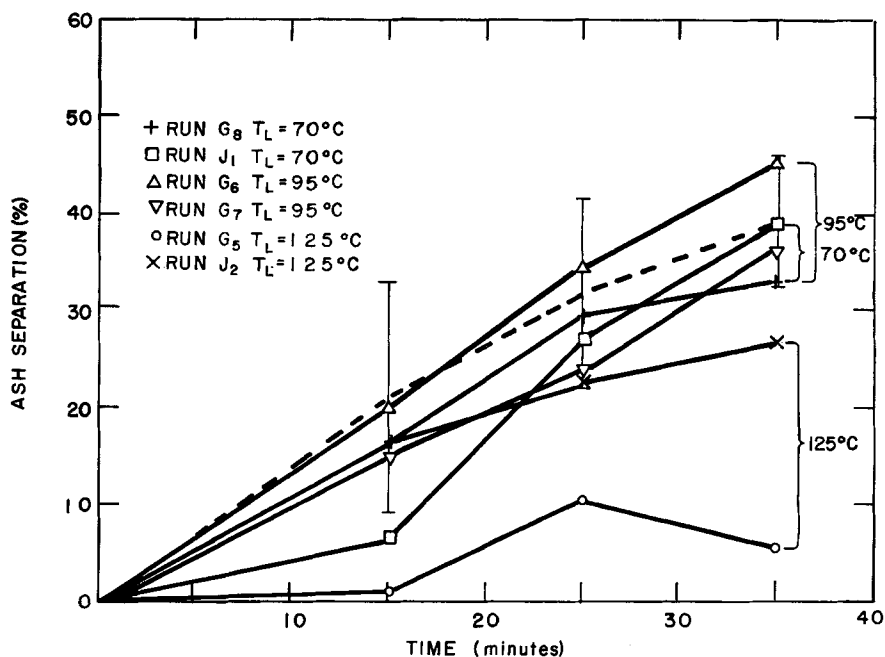


FIG. 6. The effect of liquid temperature on the extent of ash separation.

and $t = 35$ min are average or better. It is not unreasonable to conclude that this diffuser gave somewhat better separation than B. The flat dotted line has been drawn through the remaining points at $\Delta T = 35$ min.

The viscosity of the liquid and the drainage characteristics of the foam are strongly influenced by temperature. The series of runs shown in Fig. 6 was carried out to establish the extent of separation for various liquid temperatures. In all cases the foam was unheated. All other conditions, such as percent cresol, dissolution time, and diffuser size, were the same as the standard conditions shown in Table 4. In general, as the liquid temperature T_L was reduced, the temperature difference between the liquid and unheated foam also decreased. As an example, at $T_L = 70^\circ\text{C}$ the differences were $\Delta T = 35^\circ\text{C}$ for both Runs G₁ and G₈. Of the six runs only one shows separations at or above the average for the standard diffuser; however, statistically there is no difference between liquid temperatures of 70 and 110°C .

All of the standard runs were made by mixing 20 g of vacuum bottoms with the standard batch of LCO-cresol mixture (107 ml). This corresponded approximately to 4.6% ash, as determined by high temperature

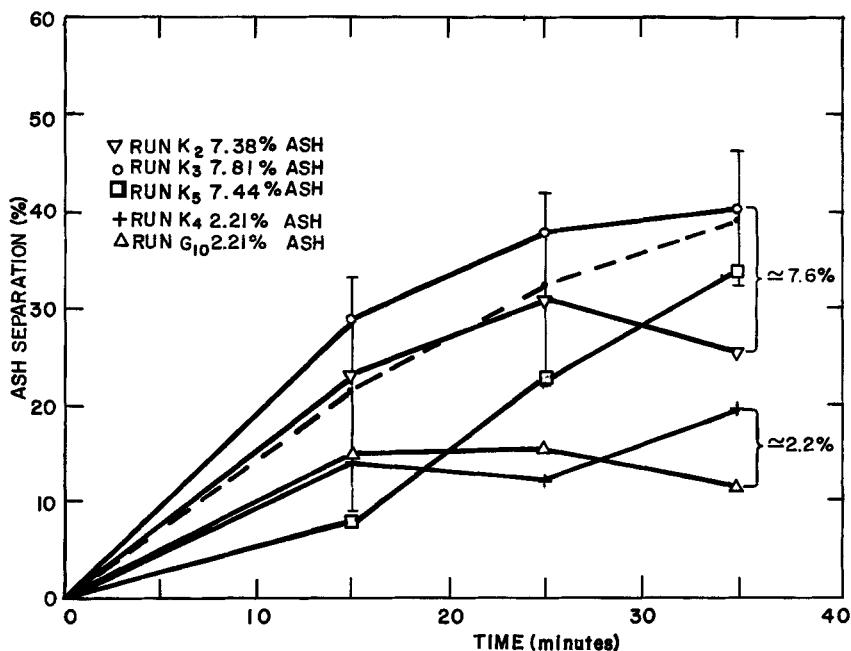


FIG. 7. The effect of the initial ash concentration on the extent of separation.

ashing, in the feed liquid. Several runs were made using 10 and 30 g of solid. These are shown in Fig. 7 and correspond to approximately 2.2 and 7.6% ash. The lower (2.2%) ash shows smaller separations which are statistically different. Lower ash concentrations also resulted in unstable foam and thus poor separation.

Finally, a series of runs were made using 4 to 8 μm , 10 to 20 μm , and greater than 150 μm diffusers. The two runs made using the 150 μm diffuser resulted in stable foam but poor separation. Both the $t = 25$ and $t = 35$ points were well below two standard deviations from the mean. For these runs the $t = 35$ min separations were 16.0 and 14.3%. The other two diffuser sizes produced results in the range of the standard diffuser; however, considerably more scatter was present. Visual observations indicated that the foam level was more difficult to keep within an acceptable range.

CONCLUSION

A laboratory scale semibatch flotation study has demonstrated the feasibility of separating coal mineral matter from an organic liquid with

physical properties similar to a liquefied coal product. The variables studied included the percent cresol added to the light cycle oil, the temperature of the liquid during flotation, the temperature difference between the foam and liquid, the diffuser pore size, and the percentage ash.

Preliminary studies were carried out to establish the relationship between the liquid and foam temperature on the extent of separation. Initial experiments with no temperature difference between the liquid and foam yielded no separation for temperatures between 70 and 125°C. A series of experiments with the liquid temperature at 110°C and the foam temperature below this established that a minimum ΔT of 20°C was necessary for separation. When all external heating of the foam was removed, the ΔT was approximately 35°C, and good separations were achieved under these circumstances.

The amount of cresol added to the light cycle varied between 0 and 11.3%. Statistically smaller ash separations were shown with 0 and 11.3% cresol when compared with the 6.0% results. Adding 2.8% cresol gave below average separation, but the results were within two standard deviations of the 6.0% line.

Use of the largest diffuser size (pores $> 150 \mu\text{m}$) yielded statistically significant lower ash separations. Attempts at comparing results using both 4 to 8 μm and 10 to 20 μm diffusers with the standard 40 to 60 μm pore size diffuser were masked by the larger scatter in the data. Statistically significantly smaller separations resulted when the initial ash content was reduced to 2.2%. Raising the initial ash content to 7.6% yielded separations comparable to the standard 4.6% runs. The primary difficulties encountered in the experiments were the instability of the foam and the tendency of particles to stick to the glass column near the top of the foam. Both of these contributed heavily to the large standard deviations in the separations.

A first-order model based on the average $t = 25$ and $t = 35$ min separations for Diffuser B was used to estimate the average separation up to $t = 100$ min. The results compared favorably with actual experimental points for Diffuser G.

Acknowledgments

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