Superscripts

g = gas phase value
o = standard state value
s = surface phase value
∞ = value at maximum adsorption limit
$*$ = standard state value at 1 bar total pressure

Subscripts

i, j, k = component i, j, k
m = mixture
n = total number of components in a mixture
1, 2 = adsorbates
3 = vacancy

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The Influence of Mixing on the Antisolvent Induced Agglomeration and Sedimentation of Mineral Matter in Coal Derived Liquids

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There are several ways to remove mineral matter from coal-derived liquids: antisolvent agglomeration and subsequent sedimentation is one. Adding antisolvent to coal liquids causes asphaltenes and/or preasphaltenes in the oil to precipitate. The precipitated asphaltenes then agglomerate the particles, increasing the sedimentation rate. The magnitude of this increase depends on the mixing conditions used to disperse the antisolvent in the oil.

This article discusses the results of sedimentation experiments performed on the antisolvent addition to coal liquids, using an x-ray detection system. The system has the capability of high temperature (up to 315°C) and high pressure (up to 2.75 × 10^6 Pa). The important parameters are the mixing time, the impeller speed, and the antisolvent addition rate. These are studied in detail for the antisolvent Soltrol.

SCOPE

Studied here is the influence of mixing conditions on the antisolvent-induced agglomeration of mineral matter and subsequent settling in coal-derived liquids at 285°C. The commercially available aliphatic hydrocarbon Soltrol-130 is the antisolvent. The variables studied are the antisolvent-to-oil ratio (0.4 to 1.0 by volume), mixing time (2 to 30 min. at a constant mixing speed of 250 rpm), mixing speed (100 to 550 rpm at a constant 30 min.), sequential mixing (mixing at 550 rpm for 2 min. followed by mixing at 250 rpm for various times up to 28 min.) and sequential antisolvent addition (added in two aliquotes, five minutes apart, with the total antisolvent to oil ratio of 0.64 by volume). Previous theories of breakage and agglomerations are extended to model the influence of mixing time and

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sequential mixing. The effect of mixing speed in the case of antisolvent precipitation and agglomeration is compared with steady-state breakage and coalescence models proposed for liquid-liquid systems.

CONCLUSIONS AND SIGNIFICANCE

The initial settling rate is strongly affected by an increase in antisolvent concentration that, in turn, increases the extent of precipitation and particle agglomeration. The settling rate increases from 18 cm/hr to 330 cm/hr as Soltrol, of 130 concentration, goes from 0 to 50% by volume. The impeller speed has more influence than the mixing time on the initial settling rate, for a fixed antisolvent-to-oil ratio, by volume. The breakage stabilized, dispersed model of Shinnar and Church (1960) for liquid-liquid systems is consistent with the effect of mixing speed at a constant mixing time on the initial settling rate. High speed mixing (550 rpm) for a short period of time (2 min) followed by a slow speed mixing (250 rpm) for a long time (28 min) increases the initial settling rate from 107 cm/hr to 200 cm/hr. This result correlates with the theory of agglomeration involving binary collisions during the low speed mixing. Finally, the results indicate that sequential adding of antisolvent to coal-derived liquids is less advantageous than a single rapid addition.

The results of this study will have a direct impact on the processes for removing solids from coal-derived liquids by antisolvent addition. Further, this work has significance for any process in which the particle size produced by antisolvent addition is important.

Particle removal from coal-derived liquids is one of the more important problems to be resolved in the commercial development of coal liquefaction. The resulting fuels should be convenient to handle and meet the ash and sulphur specifications for boiler or gas turbine fuels. In the case of boiler fuels, the mineral matter concentration must be reduced to 0.4 wt. %. It must be removed to less than 0.1 wt. % for gas turbine fuels. The ash content of coal and resulting coal liquids ranges from 3 to 21 wt. %, depending on the process and the amount of solvent recycle involved.

Solvent-Refined Coal (SRC) filter feed obtained from the Solvent Refined Coal Pilot in Wilsonville, Alabama was used in this study. The SRC filter feed contains three parts of recycle solvent for one part of Solvent-Refined Coal. It is believed that the process works because the antisolvent causes materials from solution to precipitate onto the solids, and the solids then agglomerate. These solids settle more rapidly than before agglomeration.

In the next section, we review the various techniques used in antisolvent-induced agglomeration and subsequent settling. A number of variables, such as temperature, type of antisolvent, antisolvent-to-oil ratio, mixing and antisolvent addition rate, influence this process. Mixing plays two important roles, namely, dispersing the antisolvent throughout oil and bringing particles into larger agglomerates. Here, we propose to investigate the mixing parameters which will enhance the agglomeration process, and thereby increase the settling rate.

PREVIOUS WORK

Mixing in Liquid-Liquid Dispersions

When two immiscible liquids are agitated, a dispersion is formed in which continuous breakup and coalescence of drops occurs. After some time, a dynamic equilibrium is established. Various drop properties have been measured and reported. Vermeulen et al. (1955) indirectly measured the average drop size in liquid-liquid and gas-liquid agitated dispersions in baffled cylindrical tanks, with a light transmission technique. They proposed a correlation in which average drop size was proportional to mixing speed, to the negative six-fifths power. Rodger et al. (1956) measured average drop size in liquid-liquid dispersions, using equal volume of both liquids. Their measured average drop size could be corrected with mixing speed to the -0.72 power.

Shinnar and Church (1960) developed predictions for average drop size by using the Kolmogoroff theory of local isotropy. Briefly, it is suggested that at sufficiently high rates of energy input, the small scale turbulent motions are isotropic. Their properties are determined by the energy dissipation rate per unit mass, ϵ which is proportional to the cube of the impeller speed. For breakup as the dominant mechanism they obtained

$$d_{\max} \propto N^{-6/5} \tag{1}$$

They obtained a minimum diameter below which coalescence occurs as

$$d_{\min} \propto N^{-3/4} \tag{2}$$

The above equation is in close agreement with the one reported by Rodger et al. (1956). Equation (1) of Shinnar and Church (1960) corroborates the empirical equation of Vermeulen et al. (1955).

Antisolvent Agglomeration and Sedimentation

Investigators at various laboratories, for example, Conoco Coal Development Corp. (Gorin et al. 1977, Burke 1976), Argonne National Laboratories (Huang and Fischer 1976), Oak Ridge National Laboratories (Rodgers et al. 1977) and C. E. Lummus (Snell and Simone 1976) have studied the phenomena of antisolvent-induced agglomeration and sedimentation of particles in coal-derived liquids.

In the Gorin studies, coal liquid and antisolvent were mixed in a 3.75 L autoclave at a constant impeller speed of 600 rpm or 890 rpm for 40 min. The mixing was stopped and the contents sampled with a travelling probe while it was settling. The experimental conditions were matched to process conditions, although the effect of mixing speed and mixing time was not studied extensively.

Rodgers et al. (1977) studied a variety of antisolvents and flocculating agents. The coal liquid (SRC) and antisolvent were mixed at room temperature, charged into a tube, heated rapidly to the operating temperature (about 10 min.) and maintained for 60 min. The tube was then cooled and sampled from the top for chemical analysis.

They also reported some settling experiments in a modified settler (3.75 cm diameter and 125 cm height). The

TABLE 1. SAMPLE CHARACTERIZATION DATA

(3:1 SRC MONTEREY COAL FROM WILSONVILLE, ALABAMA, 3/10/76).

Viscosity (solids free)	3.4×10^{-3} Pa·s at 120°C 1.9×10^{-3} Pa·s at 175°C
Cresol insolubles	3.49%
Pyridine insolubles	4.25%
Ash	2.37%

antisolvent was injected into a stirred autoclave charged with SRC. The contents were then heated for about 45 min. and transferred to the settler at the same temperature. Samples were withdrawn from six ports at predetermined times for analysis. The effects of mixing conditions were not studied.

Huang and Fischer (1976) investigated additives for the separation of the solids from Synthoil. Oil and additive were shaken at 80°C in a copper tube. The tube was immersed in a heating bath at 200°C for four hours. It was quickly removed, quenched in ice water, and then in liquid nitrogen. The solidification of SRC occurred in 10 min. The copper tube was cut into segments and synthoil removed from each section for chemical analysis. Thermal gradients, chemical measurements, etc. limit this procedure for settling rate measurement. No effect of mixing was investigated.

Burke (1976) used a 2 L autoclave charged with SRC and heated to the operating temperature. Unheated antisolvent was added, mixed at 400 rpm for 30 min., and transferred to the settling chamber. Four samples were taken at predetermined times from sample ports located 6.25 cm below the liquid surface for analysis.

Snell and Simone (1976) studied the continuous operation of anti-solvent induced agglomeration and settling. Their pilot plant involved heating the coal liquids and antisolvent to operating temperature and continuously feeding these streams to a thickener. After about 20 hours of continuous operation, the overflow and underflow were collected for chemical analysis.

EXPERIMENTAL

Equipment and Materials

Details of the equipment used in this study can be found in Ondeyka et al. (1977). Briefly, the coal liquid can be heated to the desired temperature in a 300 mL Parr Instrument autoclave. The autoclave can be heated to a maximum temperature of 400°C and withstand up to 1.4×10^7 Pa. The electric motor produces a continuous range of impeller speeds from zero to 1000 rpm.

The sedimentation cell handles pressure up to 2.75×10^6 Pa and temperatures up to 300°C. The temperature is measured at four points on the cell and controlled manually with a powerstat. Thin aluminum (0.15 cm) windows are used for X-rays to penetrate. All valves between the autoclave and the sedimentation chamber have large ports to prevent breakup of agglomerates upon transfer.

We used a Diano Corp. X-ray system, which allows eight exposures per film per experiment. An E-C Corp. densitometer connected with a Seargent-Welch recorder interprets the X-ray film.

Table 1 reports the sample characterization data of the Solvent Refined Coal (SRC) filter feed. The antisolvent is Soltrol-130, a registered trademark of the Phillips Petroleum Co., describing a paraffinic material boiling in the range of 185-207°C.

Procedure

The sedimentation chamber is pressurized with nitrogen and heated to operating temperature. Unheated antisolvent is added to the heated SRC (about 80 mL) in the autoclave, under different mixing conditions. It is then transferred to the sedimentation cell by using a pressure difference between the cell and the autoclave. The sedimentation process is monitored by X-ray photography. The interface heights are located as a function of time and plotted. The slope of the constant-rate period gives the initial settling rate and is reported in cm/hr.

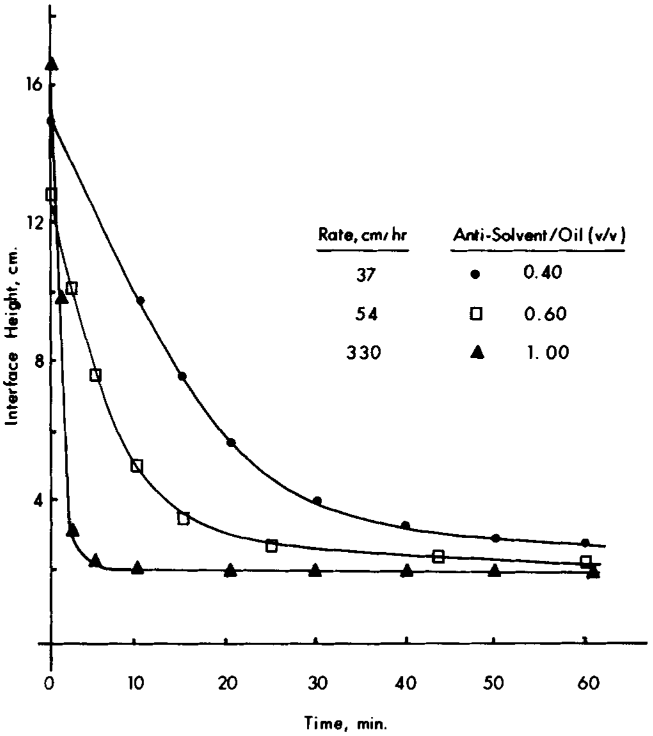

Figure 1. Settling curves for SRC-Soltrol system for various Soltrol doses at 560°K and 30-min. mixing at 550 rpm.

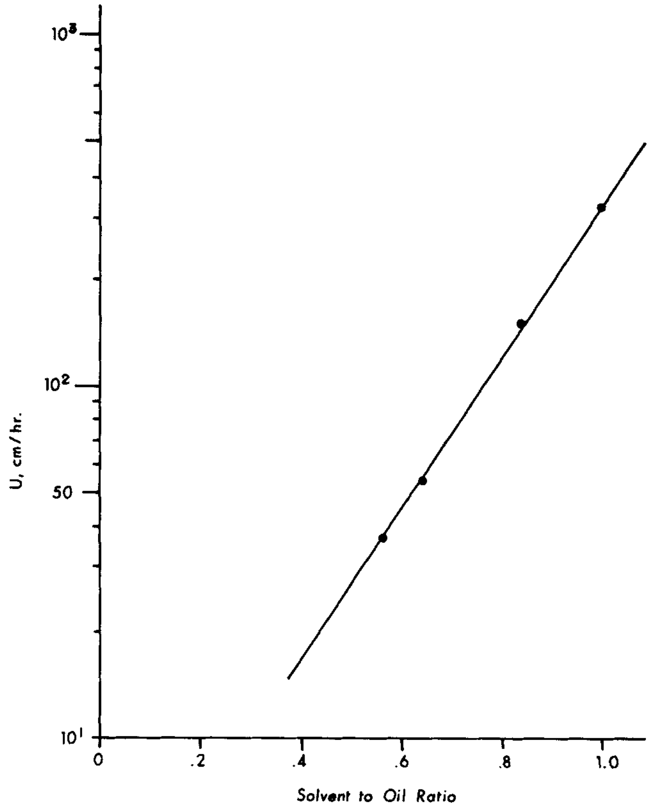

Figure 2. Influence of Soltrol dose on the initial settling rate at 560°K with a 30-min. mixing at 550 rpm.

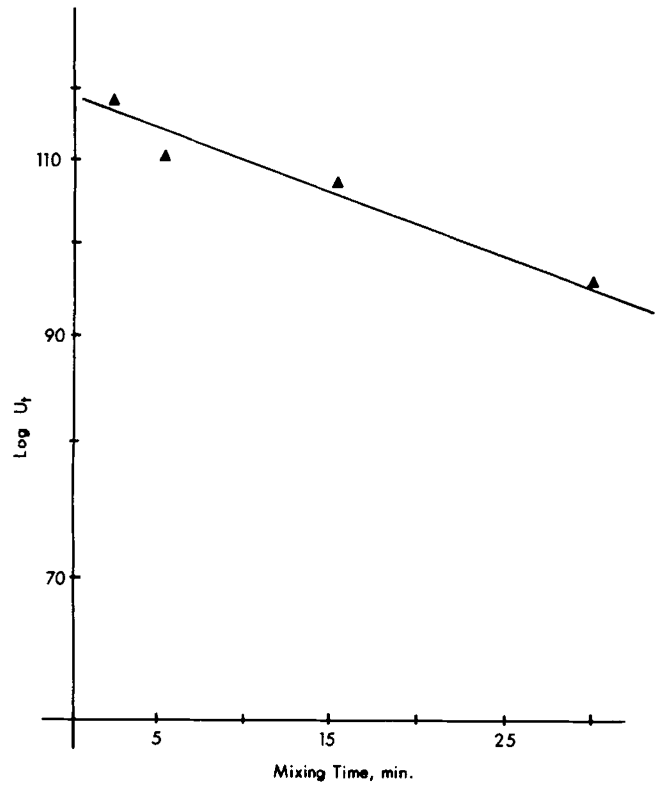

Figure 3. Influence of mixing time on the initial settling rate for SRC-Soltrol system at 560°K and mixing speed of 250 rpm for Soltrol/SRC ratio of 0.64 by volume.

RESULTS AND DISCUSSION

Effect of Antisolvent Concentration

The base for Soltrol addition was chosen as 285°C and 30 min. mixing at 550 rpm. Figure 1 is a plot of interface height versus time, in which the settling rate periods for antisolvent-to-oil ratios of 0.4, 0.64 and 1 are 10 min., 5 min., and 2 min. respectively. The initial settling rate (cm/hr) versus antisolvent-to-oil ratio by volume is shown in Figure 2. Increased antisolvent-to-oil ratio causes more precipitation and agglomeration, thereby increasing the sedimentation rate. An antisolvent-to-oil ratio of 0.64 by volume was chosen for further experimental work; this is in the range used by other investigators.

Effect of Mixing Time at Constant Mixing Speed

Experiments were carried out at 285°C to relate the influence of mixing time on the initial settling rate, holding the mixing speed at 250 rpm and an antisolvent-to-oil ratio of 0.64 by volume. The results in Figure 3 indicate that an increase in mixing time decreases the initial settling rate by decreasing the aggregate size. This suggests that adding antisolvent causes rapid precipitation of asphaltenes and/or preasphaltenes with the formation of solid aggregates. And, subsequent mixing causes slow breakage of these aggregates.

In liquid-liquid systems, the rate laws governing the breakage process can be described as

$$\text{normal drops} \xrightarrow{k_1} \text{broken drops}$$

where k_1 is the rate constant for the first-order breakage process. The average number rate of breakage per unit volume is

$$\frac{dN_p}{dt} = k_1 N_p \qquad (3)$$

The turbulent kinetic energy available to break a drop must be proportional to the energy per unit mass in the turbulent inertial subrange. Thus, for a constant volume fraction of drops

$$KE\alpha\bar{\epsilon}^{2/3} \qquad (4)$$

The average time to decomposition of the drop $(1/k_1)$ is proportional to the time to dissipate the kinetic turbulent energy. That is

$$k_1\alpha\frac{\bar{\epsilon}}{KE} \qquad (5)$$

From Equations (4) and (5)

$$k_1\alpha\bar{\epsilon}^{1/3} \qquad (6)$$

where

$$\bar{\epsilon}\alpha N^3 \qquad (7)$$

combining Equations (6) and (7), the breakage rate expression becomes

$$\frac{dN_p}{dt} = k_2 N N_p \qquad (8)$$

Since mixing speed is proportional to the average shear rate G, (widely used to compare the results from other mixing devices or for scale-up), Equation (8) can be written as

$$\frac{dN_p}{dt} = kGN_p \qquad (9)$$

At constant speed, i.e., at constant G, Equation (9) can be integrated to give

$$\ln\frac{N_{pt}}{N_{p0}} = kGt \qquad (10)$$

Based on the highest initial settling rate obtained for an antisolvent-to-oil ratio of 0.64, the estimated average agglomerate size is about 25×10^{-6}m. The Reynold's number based on this average agglomerate size is about 0.017, well within Stoke's law region. From Stoke's law for hindered settling

$$U_0 = \frac{d_{p0}{}^2 g(\rho_s - \rho_l)}{18\mu_m} f(\phi) \qquad (11)$$

and

$$U_t = \frac{d_{pt}{}^2 g(\rho_s - \rho_l)}{18\mu_m} f(\phi) \qquad (12)$$

where $f(\phi)$ is a function of the volume fraction of suspended solids, ϕ. From Equations (11) and (12)

$$\left(\frac{U_0}{U_t}\right) = \left(\frac{d_{p0}}{d_{pt}}\right)^2 \qquad (13)$$

Computer simulation of floc formation in colloidal systems by Vold (1963) suggests a relation between the number of agglomerates and their size as follows

$$\left(\frac{d_{pt}}{d_{p0}}\right) = \left(\frac{N_{p0}}{N_{pt}}\right)^{0.43} \qquad (14)$$

From Equations (13) and (14), Equation (10) can be written as

$$\ln U_t = -\left(\frac{kG}{0.86}\right)t + \ln U_0 \qquad (15)$$

Equation (15) involves the average shear rate, G, which can be calculated based on the analysis of Nagata (1975) and knowing the geometry of the mixing vessel. The average shear rate is given by

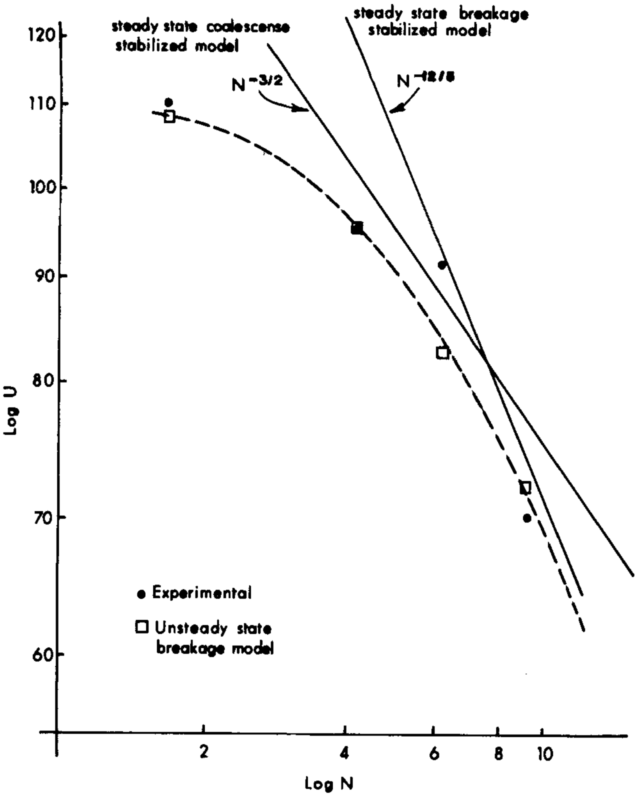

Figure 4. Influence of mixing speed on the initial settling rate for SRC Soltrol system at 560°K and 30-min. mixing for Soltrol/SRC ratio of 0.64 by volume.

$$G = \sqrt{\frac{P}{V\mu_m}}\ \text{sec}^{-1} \tag{16}$$

Power input can be calculated from the following simplified equation

$$P = 42.5\frac{N^2D^3\mu_m}{g_c} \tag{17}$$

From Equations (16) and (17), for $V = 130\text{mL}$, $\mu_m = 1 \times 10^{-3}$ Pa·s and $D = 4$ cm

$$G = 3N \tag{18}$$

From the slope of the line in Figure 3 and the average shear rate G, calculated from Equation (18), the constant appearing in Equation (15) can be evaluated and written as

$$\ln U_t = (-6.0 \times 10^{-4})Gt + \ln U_0 \tag{19}$$

where t is in minutes, and U is in cm/hr. From experimental values, $U_0 = 120$ cm/hr, where U_0 is the initial settling rate at zero mixing time. A generalized equation for the influence of mixing time on the initial settling rate can be written as

$$\ln U_t = (-6.0 \times 10^{-4})Gt + 4.8 \tag{20}$$

For a constant antisolvent-to-oil ratio of 0.64 by volume (i.e., for a constant, ϕ), Equation (20) can be used to predict the initial settling rate as a function of mixing time, knowing the value of G from Equation (18) for various single impeller speed mixing.

Effect of Mixing Speed at Constant Mixing Time

Studies of drop breakup and coalescence were made by Vermeulen et al. (1955), Rodgers (1956), and Shinnar and Church (1960), using emulsions in baffled stirred tanks. According to Shinnar and Church, there is a maximum drop size, $d_{\max}$, below which breakage rate is zero (or very small), which depends on mixing speed (Equation 1). Similarly, there is a minimum drop size, $d_{\min}$, above which the coalescence rate is zero (or very small) depends on mixing speed (Equation 2). The studies carried out in liquid-liquid systems are extended to the present study of particle agglomeration via antisolvent precipitation. Replacing the drop size by mean agglomerate

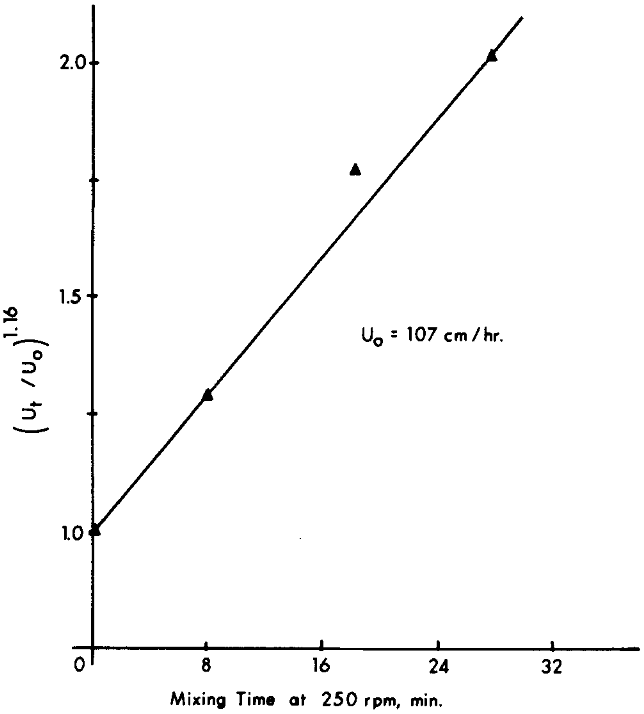

Figure 5. Effect of sequential mixing on the initial settling rate for SRC-Soltrol system at 560°K and 2-min. mixing at 550 rpm, followed by mixing at 250 rpm.

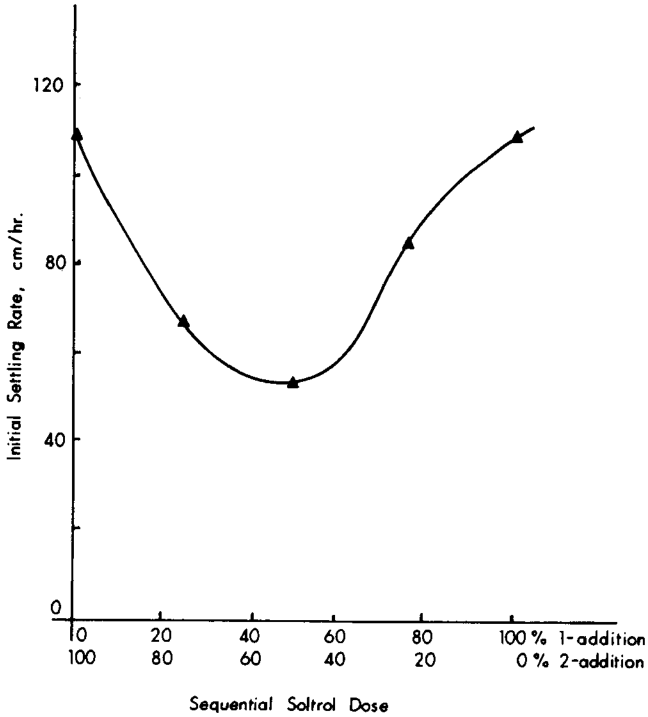

Figure 6. Influence of sequential antisolvent addition on the initial settling rate at 560°K and 10-min. mixing at 250 rpm for total Soltrol/SRC ratio of 0.64 by volume.

size, from Stoke's law, the initial settling rate is related to agglomerate size as

$$U \alpha d^2 \tag{21}$$

Therefore Equations (1) and (2) can be written as

$$U \alpha N^{-12/5} \quad \text{“breakage model”} \tag{22}$$

and

$$U \alpha N^{-3/2} \quad \text{“coalescence model”} \tag{23}$$

Results of the experiments carried out at a constant mixing time of 30 min. for a fixed antisolvent-to-oil ratio of 0.64 by volume, is shown in Figure 4, a plot of "ln U" versus "ln N." The unsteady state Equation (20) is used to calculate U for a constant mixing time of 30 min., knowing the value of G from Equation (18). Comparing experimental initial settling rate data with Equation (20) indicates that the unsteady-state breakage model explains the rate's dependence on mixing speed at a constant mixing time. After some time, this transient model will not be valid, and a steady-state model will apply.

Two different mechanisms are given in the literature for steady-state particle size distributions and hence steady-state sedimentation rates; these are the coalescence stabilized model and the breakage stabilized model. According to the first, a plot of log of sedimentation rate as a function of the log of impeller angular velocity should have a slope of negative three halves. On the other hand, this slope should be negative twelve-fifths for the breakage stabilized model.

Both of these dependencies are plotted on Figure 4. At high impeller speeds, the experimental slope as a function of log impeller speed has exceeded negative three halves and nearly approaches a negative twelve fifths. Thus, the steady-state model which appears appropriate is the breakage stabilized model. This might be expected, since the transient model was dominated by breakage also.

Influence of Sequential Mixing

Further improvement in the mixing might result from using two impeller speeds, sequentially. The first would be high, to promote a rapid dispersion of the antisolvent into the coal-derived liquid. The second mixing would be slow, to increase the agglomerates' size through particle association.

Howarth (1967) performed similar experiments in liquid-liquid systems to study the effect of drop size and impeller speed on coalescence frequency. Using sequential mixing, the impeller speed was first set between 350 and 600 rpm. Final mixing was performed at 210 rpm. During the high speed mixing, a steady mean drop size would be established; lowering the impeller speed would dictate a new maximum stable size. Therefore, no breakup can occur until some drops grow to the maximum stable size by coalescence—the initial rate of increase of mean drop size is determined by coalescence alone.

Our experiments were performed at 285°C with Soltrol/oil ratio of 0.64 by volume. The first high speed mixing was always performed at 550 rpm for two min. After this, the stirrer speed was slowed down to 250 rpm, for various periods of time. Results are shown in Figure 5, which suggests that the first mixing at 550 rpm disperses the antisolvent throughout the system, and the second mixing at 250 rpm promotes agglomeration.

Modeling of Unsteady State Sequential Mixing

Based on the above discussion of sequential mixing, and under the assumptions that the agglomeration rate is independent of particle size and all collisions are binary, the second order agglomeration process can be described as

$$\frac{-dN_p}{dt} = aN_p^2 \tag{24}$$

The solution of Equation (24) is of the form

$$\left(\frac{N_{p_2}}{N_{p_t}} \right) = aN_{p_2}t + 1 \tag{25}$$

Here, let N_{p_2} = total number of particles at the end of two-minute mixing at 550 rpm. N_{p_t} = total number of particles at the end of t min. of mixing at 250 rpm. From Equations (13) and (14) we get

$$\left(\frac{N_{p_2}}{N_{p_t}} \right) = \left(\frac{U_t}{U_2} \right)^{1.16} \tag{26}$$

Inspection of Equations (25) and (26) indicate that

$$\left(\frac{U_t}{U_2} \right)^{1.16} = aN_{p_2}t + 1 \tag{27}$$

Here, U_t = initial settling rate at the end of t min. of mixing at 250 rpm, and U_2 = initial settling rate at the end of two-min. mixing at 550 rpm. Equation (27) is shown in Figure 5 as a plot of $(U_t/U_2)^{1.16}$ versus mixing time at 250 rpm. The experimental data compare favorably with this theoretical interpretation.

Effect of Sequential Antisolvent Addition

The influence of sequentially adding antisolvent was investigated with Soltrol at 285°C and mixed at 250 rpm for 10 min. Results are shown in Figure 6.

Two aliquots ranging from 25 to 100% of the antisolvent to be added were mixed into the SRC at a 5 min. interval. The total Soltrol/oil ratio for the two increments was 0.64 by volume. The Soltrol-oil was mixed five min. after each addition. In all cases, initial settling rates decreased when the Soltrol was added in aliquots. The initial settling rate is dominated by the first dose of antisolvent and the second increment produces no agglomeration—and thus no increase in the initial settling rate. These results indicate the advantage of introducing the antisolvent in one dose to the coal liquid system.

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NOTATION

a = agglomeration rate constant
d = drop size
d_p = particle diameter
G = average shear rate
K, k_1, k_2 = constants
N = impeller speed
N_p = total number of particles
P = power input to impeller
t = time
U = initial settling rate
V = volume of liquid in the autoclave

Greek Letters

μ_m = viscosity of anti-solvent-oil mixture
ρ_s = density of solid
ρ_l = density of liquid

ϕ = volume fraction of suspended solids
$\bar{\epsilon}$ = energy dissipated per unit mass

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Calculation of the Governing Equations for a Seriated Unequal Velocity, Equal Temperature Two-Phase Continuum

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The governing equations describing the flow of an unequal phase velocity, equal phase temperature (UVET) seriated two-phase continuum as derived by Solbrig and Hughes (1978) are solved in this article, in one space dimension. A simple implicit iterative solution procedure is developed to numerically evaluate the five non-linear coupled field equations. Analytical solutions are developed, against which the computer code results are compared. The prototype UVET code results are compared to the equivalent equal phase velocity, equal phase temperature (EVET) code results for the same problem. Major phenomena such as phase flow reversal, countercurrent flow and flooding-like behavior are predicted.

SCOPE

Our objectives are to develop a solution procedure for an unequal velocity, equal phase temperature seriated continuum, to compare the computed results against analytical solutions, and to predict *a priori*, major physical phenomena using the theory. The predictions of unequal velocity phase separation calculated here are of use in many aspects of energy analysis, including vertical pneumatic conveying, fluidized beds, and nuclear safety. The theoretical bases for the model were developed by Solbrig and Hughes (1978).

The theory requires separate continuity and momentum equations for each phase and a mixture energy equation. A seriated continuum is one which represents phase interaction expressions by differences of velocity, for example, rather than gradients. The model has the potential to describe far more phenomena than the equal velocity or gas dynamics model usually used.

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