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Separation of Solids at Liquid–Liquid Interface

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Abstract

The possibility of separation of solids at the interface of two liquid phases by utilizing surface tension to overcome gravity was studied. The proposed theoretical model was verified in several experimental examples.

INTRODUCTION

The process described in this paper is a variant of the conventional flotation technique, consisting in utilization of the surface tension at the interface of two liquid phases to overcome gravity so that one fraction of the processed ore remains suspended at the interface and the other fractions settle. The proposed method is useful with minerals whose high specific gravity rules them out for the conventional sink-float treatment.

Earlier works on this subject include that of Gaudin (*1*), who assumed a cylindrical shape for the solid, and Barnea (*2*), who derived a set of equations for a spherical solid without reference to solid separation application. Further works dealing with the light liquid/solid/heavy liquid system are those of Miller (*3*), Mitzmager and Mizrahi (*4*), and Mellgren and Sherpold (*5*, *6*), but they are concerned more with the deflocculation aspect than with solid separation.

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THEORETICAL BACKGROUND

The system is liquid–solid–liquid; the liquid phases are immiscible, the solid suspended at the interface is assumed to be spherical, and liquid 1 is lighter than liquid 2.

Interrelationship of System Parameters (Fig. 1)

From the scheme in Fig. 1, it is seen that at equilibrium $F_1 + F_2 + F_3 + F_4 = 0$, or more explicitly:

$$[(\gamma_{s1} - \gamma_{s2}) \cos \delta + \gamma_{12} \cos \beta]\, l = g[V_{\text{total}}\rho_s - V_1\rho_1 - V_2\rho_2] \quad (1)$$

Referring to the Young-Dupré equation,

$$\gamma_{s1} - \gamma_{s2} = \gamma_{12} \cos \theta \quad (2)$$

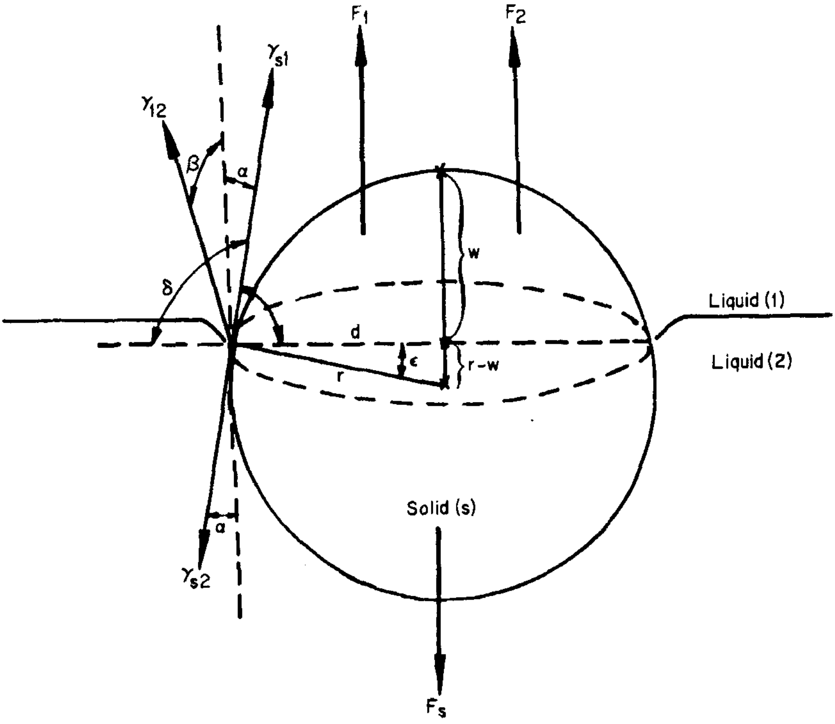

FIG. 1. The solid sphere (s) floating at the liquid–liquid interface.

where θ is the contact angle. Substituting the geometrical values of the three volumes V_1, V_2, and V_{total} in Eq. (1), we arrive at the basic equation for a solid with radius r suspended between two liquid phases and wetted by both:

$$\rho_s = \frac{3}{2}\frac{\gamma_{12}}{gr^2}\sin\delta\,(\sin\delta\cos\theta + \cos\beta) + \tfrac{1}{4}\rho_1(2 + 3\cos\delta - \cos^3\delta) + \tfrac{1}{4}\rho_2(2 - 3\cos\delta + \cos^3\delta) \quad (3)$$

The angle δ is determined by r and W as follows:

$$\cos\delta = -\frac{r - W}{r} \quad (4)$$

A change in ΔW results in a change in $\Delta\delta$:

$$\Delta\delta = \frac{1}{r[W(2r - W)]^{1/2}}\Delta W \quad (5)$$

and also in the forces involved (except that of gravity). Denoting the right-hand expression in Eq. (1) by k, the corresponding change in it is given by

$$\Delta k = \frac{dk}{dW}\Delta W = g\pi W(\rho_2 - \rho_1)(2r - W)\Delta W \quad (6)$$

Maximum Specific Gravity of Suspended Solid

At constant pressure and temperature the system parameters are δ, θ, γ_{12}, r, ρ_1, ρ_2, and ρ_s. The maximum specific gravity ($\rho_{s\,max}$) at which a solid would remain suspended is a function of others. In the example illustrated by Figs. 2–4, the phases are distilled water and tetrabromoethane (TBE), for which $\rho_1 = 1.00$ g/cm^3, $\rho_2 = 2.92$ g/cm^3, and $\gamma_{12} = 38.8$ dynes/cm.

EXPERIMENTAL

The experimental installation was tested for two main functions: (a) determination of the contact angle and (b) separation of solids at the liquid phase interface.

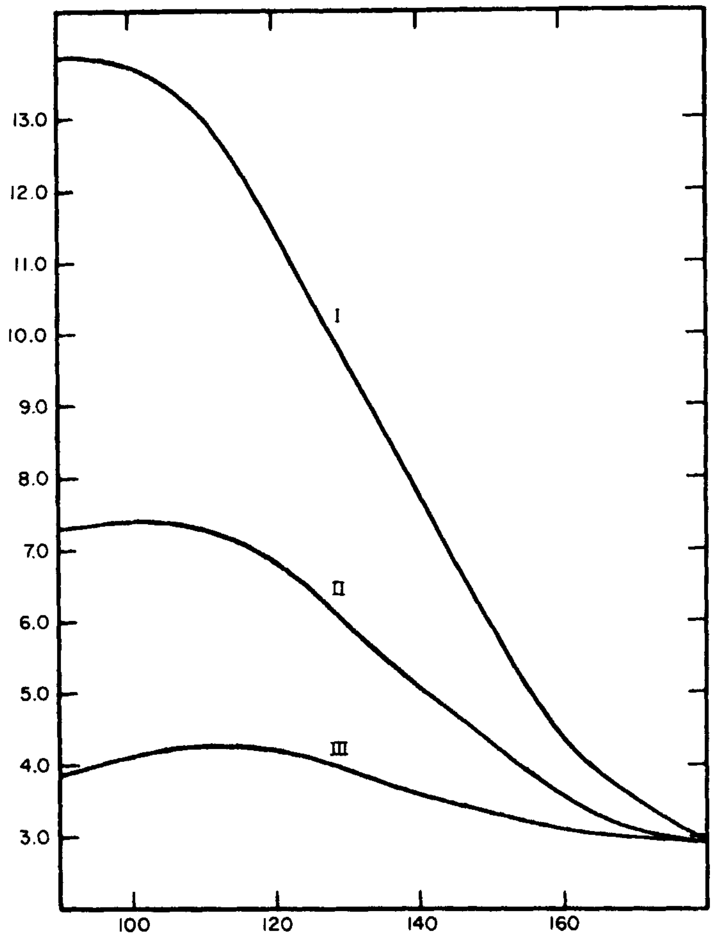

FIG. 2. $\rho_{s\ max}$ vs. δ for different radii in the system TBE–H_2O (distilled). I: r = 0.100 cm; θ = 0°. II: r = 0.150 cm; θ = 0°. III: r = 0.250 cm; θ = 0°. Abscissa: δ (degrees). Ordinate: $\rho_{s\ max}$ (g cm^{-3}).

The contact-angle device (Figs. 5 and 6) comprised a stage on which the measurement cell was mounted, a microscope with all light-absorbing elements removed except the ocular, and a camera. The so-called "tilting method" used consists in rotating the cell with the three-phase system until the interface is horizontal, and the contact angle is recorded on the camera screen (Fig. 7).

The *lighter liquid phase* was distilled water in one part of the experiments, and an aqueous solution (10^{-4} *M*) of dodecyl amine (DA) in another.

The *heavier liquid phase* was one of the following: pure tetrabromoethane, pure ethylene dibromide (EDB) (both filtered through chalk before use and stored in a nitrogen atmosphere to prevent reabsorption of atmospheric water vapor), or a mixture of the two.

The *solids* tested were Pyrex glasses and the following minerals: sphalerite, galena, quartz, and fluorite.

Procedure

Series I. Tests confined to a single solid phase (Pyrex glass spheres) with the radius as the only variable.

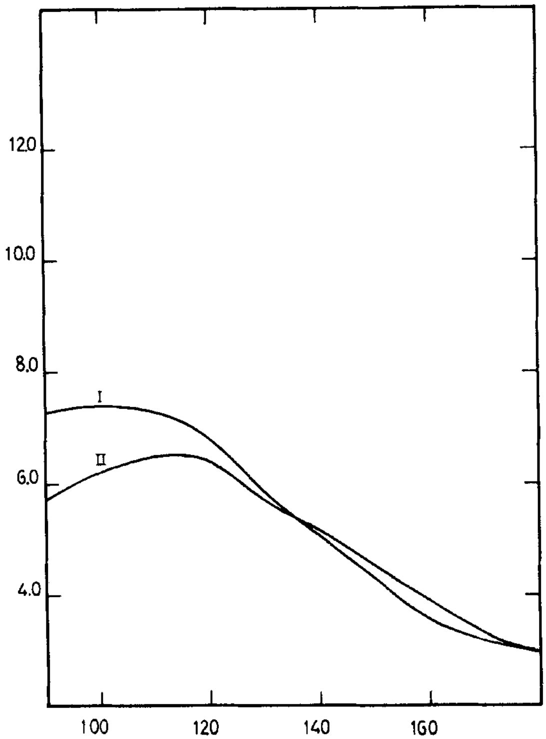

FIG. 3. $\rho_{s\ max}$ vs. δ for different contact angles θ. I: r = 0.150 cm; θ = 45°. II: r = 0.150 cm; θ = 0°. Abscissa: δ (degrees). Ordinate: $\rho_{s\ max}$ (g cm^{-3}).

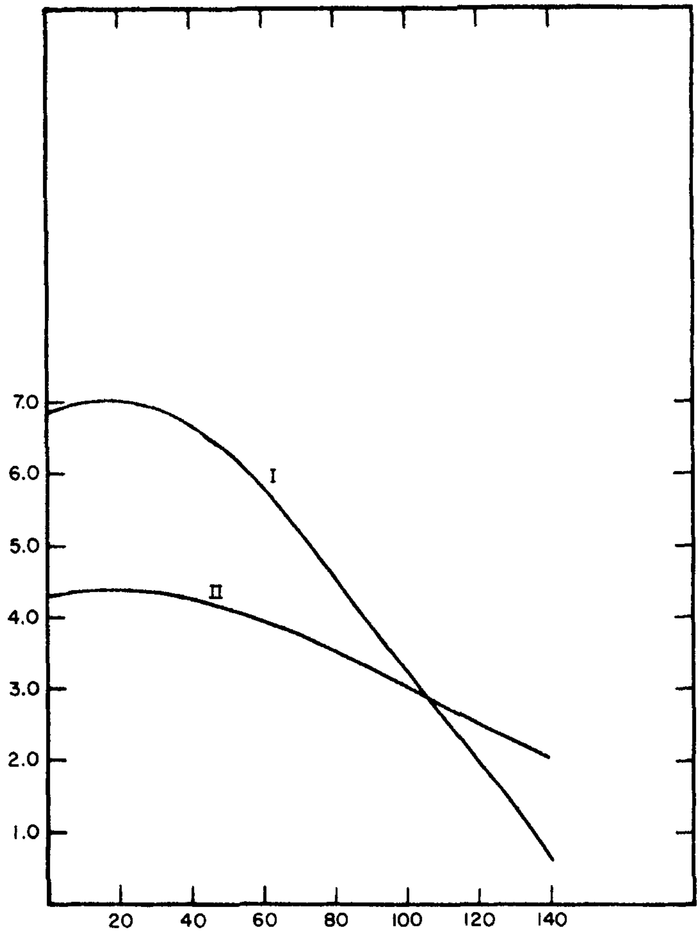

FIG. 4. $\rho_{s\,max}$ vs. contact angle, θ, for different radii of solids. I: $r = 0.150$ cm; $\delta = 120°$. II: $r = 0.250$ cm; $\delta = 120°$. Abscissa: Contact angle, θ (degrees). Ordinate: $\rho_{s\,max}$ (g. cm^{-3}).

Series II. Comprising two stages: determination of the contact angle for the solids listed above, and separation tests on pairs of irregular solid particles based on their respective $\rho_{s\,max}$ values. In the latter stage the condition for separation of the solids (fed slowly into the liquid system by gravity) is $\rho_{s\,max\,1} > \rho_2 > \rho_{s\,max\,2}$, with solid 2 remaining suspended at the interface and solid 1 settling.

TABLE 1

Values of Contact Angles in Various Systems

Solid	H_2O(dist)–solid–TBE (degrees)	10^{-4} M DA–solid–TBE (degrees)	H_2O(dist)–solid–EDB (degrees)
Pyrex glass	0	52	0
Quartz	0	52	0
Sphalerite	48	98	37
Fluorite	55	55	60
Galena	20	55	20

Results

The contact angle readings for the H_2O (dist)–solid–TBE, DA (10^{-4} mole liter^{-1} aqueous solution)–solid–TBE (ρ_T = 2.92 g cm^{-3}) and H_2O (dist)–solid–EDB (ρ_T = 2.19 g cm^{-3}) are summarized in Table 1.

The first series of separation tests is summarized in Fig. 8, in which the

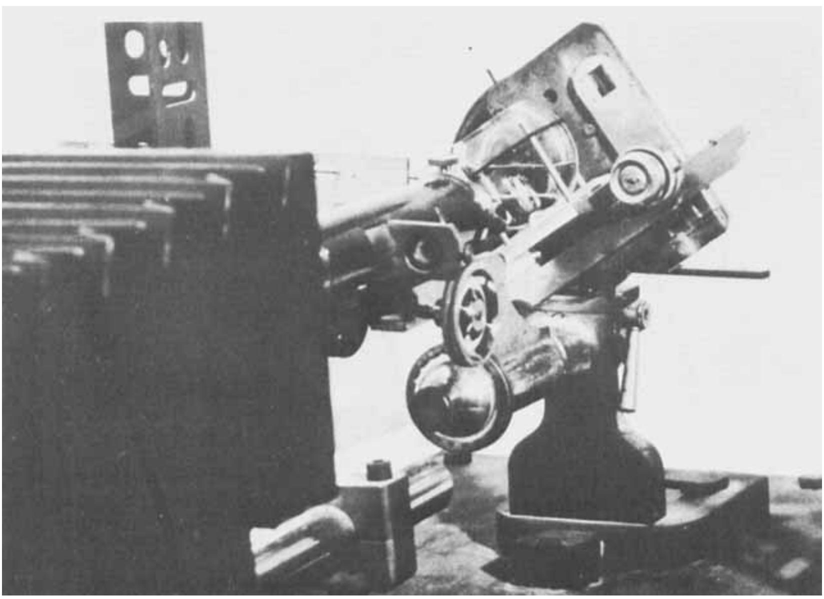

FIG. 5. Contact angle measuring device.

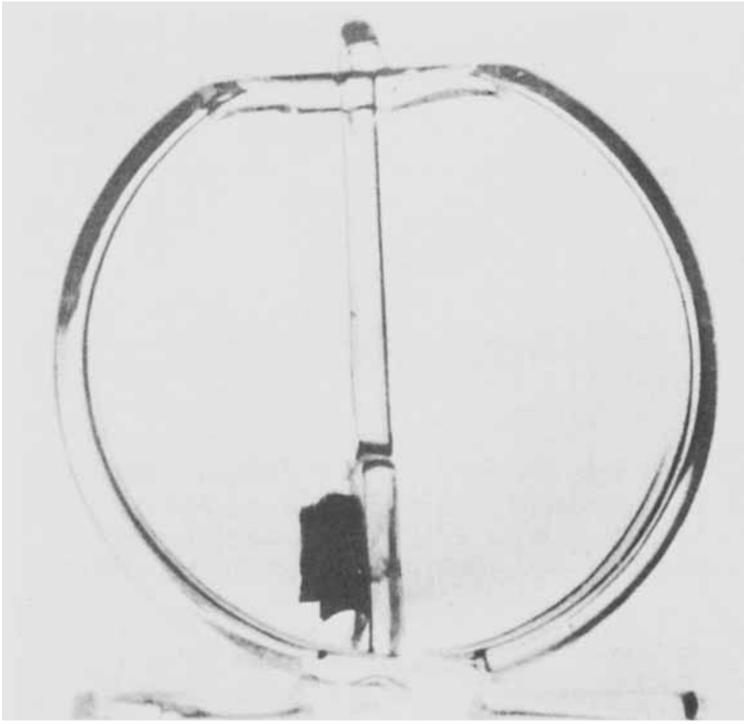

FIG. 6. Contact angle cell (truncated cylinder) with mineral (Galena) mounted.

$\rho_{s\,max}$ values of the Pyrex spheres (determined from Eq. 3) in a H_2O (dist)–EDB system are plotted against the radius r. The limiting radius for suspension at the interface is seen to be 0.273 cm.

Figures 9–11 show the separation possibilities of pairs of solids: sphalerite vs. galena, quartz, and fluorite, respectively, with the variously hatched areas indicating the corresponding suspension ranges of the radii. For the *sphalerite–galena* variant, all particles with $r > 0.160$ cm settle and those with $r < 0.115$ cm remain suspended at the interface; if the feed range is between these limits, separation is effected, with the galena settling and the sphalerite suspended. For the *sphalerite–quartz* variant, the range is 0.160–0.251 cm (sphalerite settling, quartz suspended), and for the *sphalerite–fluorite* the range is 0.155–0.275 cm (sphalerite settling, fluorite suspended).

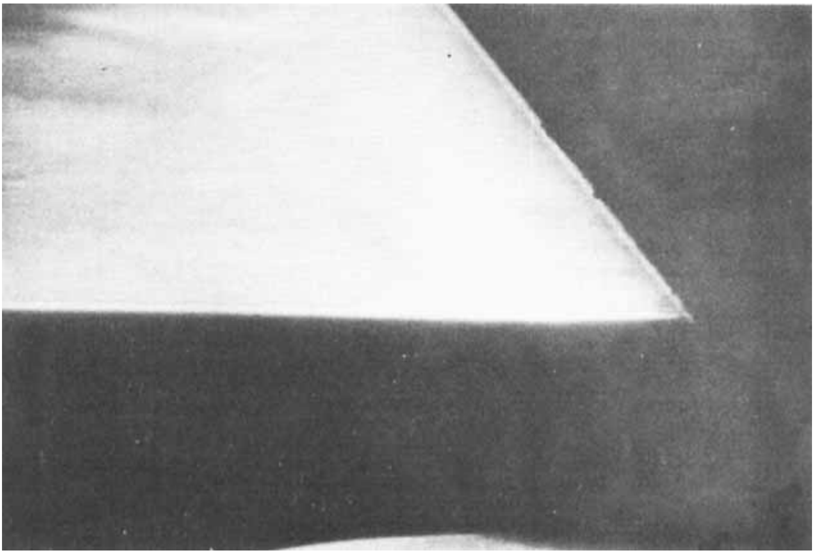

FIG. 7. Contact angle in DA–Pyrex glass–TBE system.

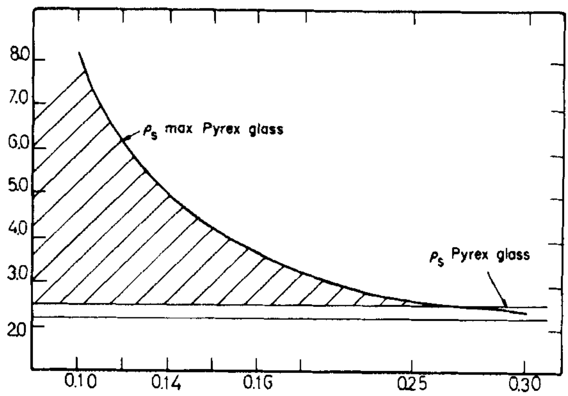

FIG. 8. Pyrex spheres suspended at H_2O (distilled)–EDB interface. Abscissa: Radius of solid (cm). Ordinate: $\rho_{s\,max}$ (g cm^{-3}).

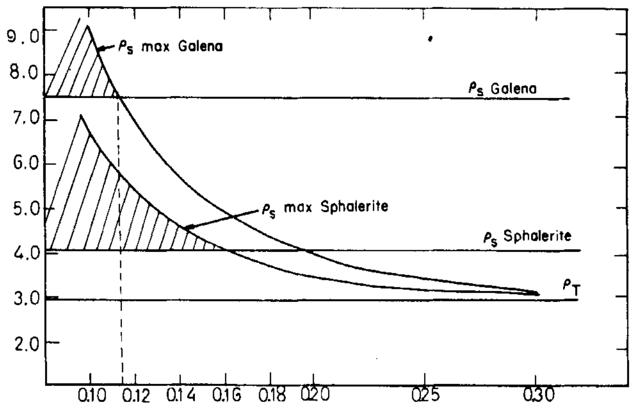

FIG. 9. Separation of galena (sinks) from sphalerite (floats) at the interface of TBE–DA (10^{-4} mole liter^{-1} aq. sol.). Abscissa: Radius of solid (cm). Ordinate: $\rho_{s\,max}$ (g cm^{-3}).

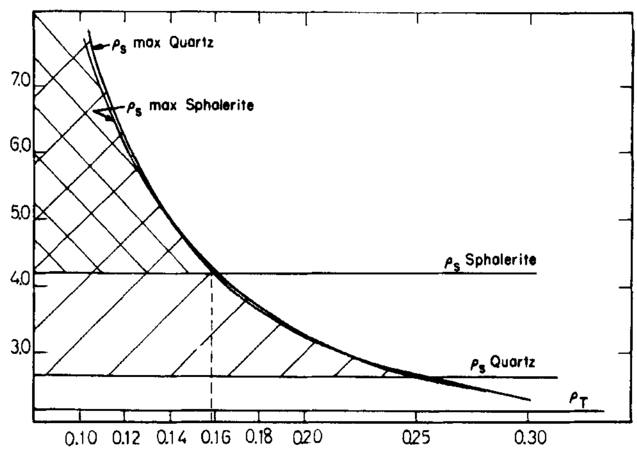

FIG. 10. Separation of sphalerite (sinks) from quartz (floats) at the interface of H_2O (distilled)–EDB. Abscissa: Radius of solid (cm). Ordinate: $\rho_{s\,max}$ (g cm^{-3}).

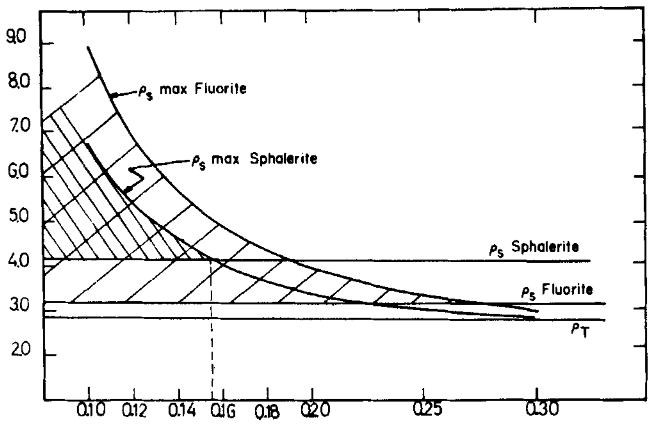

FIG. 11. Separation of sphalerite (sinks) from fluorite (floats) at the interface of TBE–DA (10^{-4} mole liter^{-1} aq. sol.). Abscissa: Radius of solid (cm). Ordinate: $\rho_{s\,max}$ (g cm^{-3}).

CONCLUSIONS

The results of the separation tests are in agreement with the conclusions drawn from the theoretical model and from Figs. 8–11.

The proposed method proved effective under given conditions of particle size and contact angle. In the case of derivation from these conditions, some of the other parameters have to be altered; e.g., the contact angle or interfacial tension (by means of a surfactant).

Equation (3) lends itself to converse use; namely, determination of the interfacial tension of the liquid phases from the suspension radius.

An aspect not yet considered is the behavior of a nonspherical shape, an important practical factor bound to affect the surface energy of the solid.

SYMBOLS

W	altitude of spherical segment in liquid 1
r	radius of sphere
γ_{s1}	surface tension, solid–liquid 1
γ_{s2}	surface tension, solid–liquid 2
γ_{12}	surface tension, liquids
α	angle between vector γ_{s1} and vertical

β	angle between vector γ_{12} and vertical
δ	angle between vector γ_{s1} and plane of common line of three phases
l	circumference of dashed circle
d	radius of dashed circle
V_1	volume of spherical segment in liquid 1
ρ_1	specific gravity of liquid 1
V_2	volume of spherical segment in liquid 2
ρ_2	specific gravity of liquid 2
V_{total}	volume of sphere
ρ_s	specific gravity of solid
$\rho_{s\,max}$	maximum specific gravity at which a solid would remain suspended

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