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S. Kelebek[a]; S. Yoruk[a]; G. W. Smith[b]
[a] Department of Mining Engineering, Goodwin Hall, Queen's University, Kingston, Canada [b] Department of Mining and Metallurgical Engineering, University Street, McGill University, Montreal, Canada

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WETTING BEHAVIOR OF MOLYBDENITE AND TALC IN LIGNOSULPHONATE/MIBC SOLUTIONS AND THEIR SEPARATION BY FLOTATION

S. Kelebek,[1,*] S. Yoruk,[1] and G. W. Smith[2]

[1]Queen's University, Department of Mining Engineering, Goodwin Hall, Kingston, Ontario, Canada
[2]McGill University, Department of Mining and Metallurgical Engineering, 3610 University Street, Montreal, Quebec, Canada

ABSTRACT

Flotation characteristics of molybdenite and talc are controlled by surface properties of their cleavage planes. Because the inherent hydrophobicity is common to the cleavage planes of both of these minerals, their separation by flotation is relatively more difficult compared to other mineral separation systems. Wettabilities of molybdenite and talc samples in sodium lignosulphonate/MIBC solutions have been investigated using both cleavage and disc specimens at a pH of about 7.2. The variation of adhesion tension, $\gamma_{lv} \cos\theta$ with interfacial tension, γ_{lv} was found to be linear. The slope value, β for each case has been utilized as an indicator of relative adsorption densities. This approach has indicated that the adsorption density of sodium lignosulphonate at the solid/liquid interface of molybdenite face was significantly larger than that of talc by

*Corresponding author. E-mail: kelebek-s@mine.queensu.ca

a factor of 2.7. The difference in adsorption densities/wettability has been exploited to demonstrate highly efficient separation of these minerals in this surfactant system. The results of current investigation are consistent with the industrial practice of talc-molybdenite separation and provide some insight into the mechanism of depression behavior of molydenite.

Key Words: Molybdenite depression; Talk flotation; Lignosulphonate; Wettability; Adhesion tension; Adsorption

INTRODUCTION

Molybdenite (MoS_2), or "Moly" as it is sometimes called, represents the primary source for molybdenum. Moly is useful as a lubricant, especially at high temperatures where grease-based lubricants would decompose. The metal itself is incorporated into a variety of industrial applications such as a filament material in electronic and electrical devices and a promoter in catalytic refining of petroleum. It is also used as a critical alloying element to provide heat-resistant and corrosion-resistant properties for a number of alloys used in nuclear energy applications and for missile and aircraft parts. Talc ($Mg_3Si_4O_{10}(OH)_2$) is the primary source for talcum powder, which is a common household item. It also has various uses as a filler material and extender in paint, plastic, roofing, and flooring industries.

Molybdenite and talc can be obtained as a by-product from mineral processing of various base metal ores. Although these minerals are different by their chemical nature, being a sulphide and silicate mineral respectively, they have similar surface properties and natural flotation behavior. This has an important implication in processes involving these minerals. In the flotation concentrates of some complex sulphide ores, for example, nickel-copper ores, talc can be the main nonsulphide contaminant lowering the concentrate grade. In the processing of such ores, use of polymeric depressants like carboxymethylcellulose may become inevitable for separation of talc in order to avoid smelter penalties (1,2). Gomes and Oliveira (3) reported that the carboxymethylcellulose depression of talc from sulphide ores can be enhanced by addition of aluminium chloride.

In the treatment of molybdenite ores, the separation of molybdenite from other ore constituents is largely based on exploiting its natural floatability. Depending on the separation process requirements, this natural floatability may either be enhanced by using hydrocarbons in a so-called emulsion flotation (4) or decreased to a minimum level using hydrophilic macromelecular compounds such as dextrin. The influence of dextrin on various surface properties and the depression of molybdenite has been studied (5).

In some orebodies, molybdenite and talc occur together. Porphyry type of molybdenum and copper-molybdenum sulphide ores represent the most typical

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example of such ore deposits that are exploited commercially. Some details of mineralogy may be found elsewhere (6). The processing of these talcose molybdenite ores presents serious separation problems because of the coflotation properties of the two minerals. Mathieu and Bruce (7) evaluated various treatment methods for the separation of talc and molybdenite. The methods included electrostatic separation, hydroseparation (elutriation column) and flotation. Flotation tests were carried out with depressants that included aluminium sulphate, lignin sulphonates and other hydrophilic polymers, which are commercially available under various trade names. The efficiency of flotation separation with various depressants with the exception of lignin sulphonates was primarily affected by the ratio of talc and molybdenite in the feed to be treated.

The industrial practice of the molybdenite-talc separation by flotation involved the use of aluminium sulphate and lignin sulphonates. The former has been utilized in combination with sodium silicate to depress talc for its separation from molybdenite at Cyprus Prima Mining Co. (8). In a different technique adopted by Anaconda Co., the lignin sulphonate has been used to depress molybdenite in a talc preflotation circuit at Twin Buttes moly plant (9). Despite a commercial application, no fundamental study seems to have been reported on the lignosol separation of moly and talc. However, the effect of aluminium and other cations on surface properties and natural floatability of talc was investigated (10). More recently, Kusaka et al. (11) reported additional research work on the effect of aluminium (III) and chromium (III) cations on lyophilicity of talc in the isooctane-water system.

Investigations with talc and molybdenite included liquid/gas interfacial tension as a variable in wetting and flotation (12). Virtually, all of the previous studies in this area featured aqueous methanol solutions to explore the wettability differences of minerals in a wide range of surface tensions, even beyond the practical limits of usual flotation practice. The current work on talc-molybdenite separation arises from recognition of a need to extend these studies to surfactant systems that are more relevant to industrial practice of froth flotation.

EXPERIMENTAL MATERIALS AND PROCEDURES

The samples of molybdenite and talc used in the present investigation originated from Ontario and Quebec, Canada, respectively. The former was a highly crystalline sample received through Ward's Natural Science Establishment, Rochester, New York. The latter came from a talc mine in the province. A 50:50 mixture of the size fraction 80 $\times$ 150 mesh (180 $\times$ 106 μm) was arbitrarily used in flotation tests as an earlier work indicated the talc/moly ratio of the feed as a noncritical variable for the current surfactant system (7).

Contact angle measurements were made following the method developed by Sutherland and Wark (13). The bubble holder consisted of a glass capillary tube with an internal hole diameter of 1.0 mm and pinch valve on a plastic tube

extension for adjustment of the pressure to move the air bubbles of 1.5 ± 0.2 mm in diameter in and out of the capillary. Five to eight bubbles were allowed to attach to the hydrophobic mineral specimen submerged in solution in a glass cell and the stage on which this cell rests was gently tapped for about 15 seconds. To achieve comparability, all bubbles were kept in contact with the surfaces for about 15 min before the angles were read to the nearest degree by means of a built-in protractor. No significant change in the magnitude of the angles was observed during this period. The result of measurement according to this method is considered to approximate an equilibrium value of contact angle. The contact angles of captive bubbles were measured on cleaved specimens as well as disc samples prepared by compacting the flotation feed. The compaction was effected using a hydraulic press operated at a maximum load of 20 tons to produce a stable disc that was about 1.3 cm in diameter. Before and during pressing, the sample compartment in the mold was kept connected to a vacuum pump.

Sodium lignosulphonate, commercially designated as Lignosol-XD, has been provided courtesy of its manufacturer, Reed Ltd. of Quebec City. This reagent is a by-product in the pulp and paper industry and obtained during the delignification of wood using the sulphite treatment. The sample was used as received, without subjecting it to any pretreatment in order to maintain a greater relevancy towards commercial flotation practice. This reagent was used in combination with methyl isobutyl carbinol, MIBC, as the frother. Concentration of MIBC in the solution was kept constant at 10^{-4} M, which provided sufficient frothing activity for flotation. The Lignosol-XD concentration was changed in order to obtain solutions of various surface tension. All experiments were carried out at the natural pH level of these solutions with no adjustment (7.2 ± 0.3).

Small-scale flotation tests were performed using an all-glass cylindrical cell similar in form to that reported by Partridge and Smith (14). The cell had an 18 cm long cylindrical body (1.8 cm in diameter). The flotation feed was floated for 1 min at a gas flow rate of 50 ml/min following a conditioning period of 5 min. The surface tensions were measured at room temperature ($22 \pm 2^{\circ}$C) using the Fisher Autotensiomat (Fisher Scientific, U.S.A.), which employs the ring method. Further details on methods and equipment for contact angle and small-scale flotation tests may be found elsewhere (15–17).

RESULTS AND DISCUSSION

Wettability

The variation in wettability of molybdenite and talc samples in aqueous solutions of lignosulphonate and MIBC is shown in Figure 1 and Figure 2, respectively. The MIBC concentration of the solutions was fixed at 10^{-4} M. There is some scatter of data points. However, for both disc and cleavage samples, a linear trend

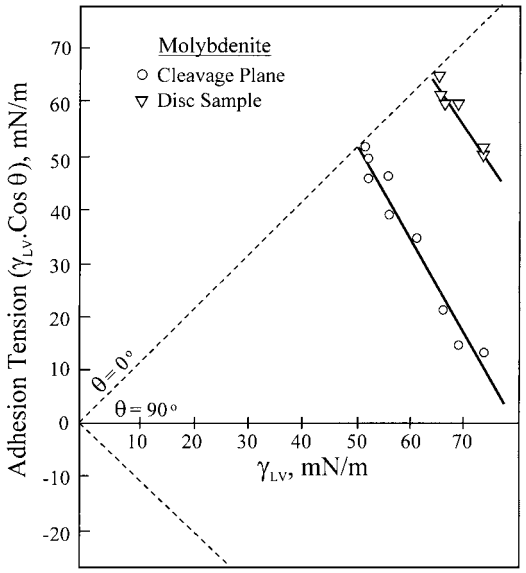

Figure 1. Adhesion tension diagram for molybdenite in aqueous lignosulphonate and MIBC solutions.

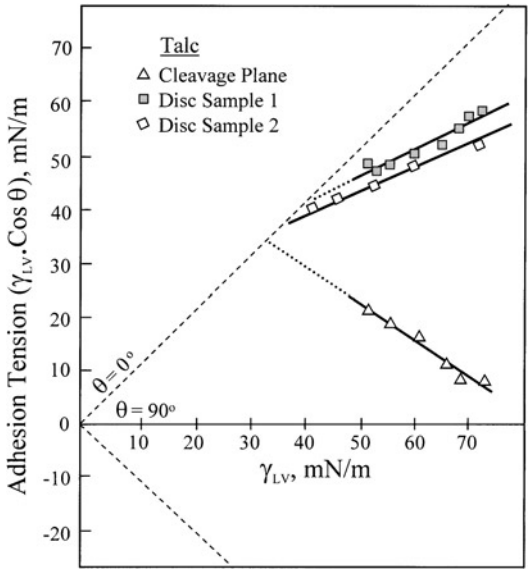

Figure 2. Adhesion tension diagram for talc in aqueous lignosulphonate and MIBC solutions (cleavage plane and disk sample 1) and methanol solutions (disk sample 2).

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between the adhesion tension and surface tension is apparent. In these diagrams, the wettability line is characterized by its intercept (γ_c) and slope (β) values. The former is known as critical surface tension of wetting, which refers to the interfacial tension γ_{lv}, below which the surfactant solution wets a hydrophobic solid completely, thus preventing contact of an air bubble with it (i.e., contact angle $\theta = 0$). Such a diagram is useful because it can at least allow a conceptual link between γ_c and γ_{cf}, critical surface tension of floatability. The latter term was proposed by Hornsby and Leja (18) in view of dynamic nature of the flotation process. As such, the solid particles can be nonfloatable even when their surfaces are not completely wetted. Thus, γ_{cf} involves a finite contact angle to account for dynamic conditions in flotation such as adhesion of a particle to a bubble and particle-bubble aggregate stability. On the wettability diagram, a line of finite contact angle passing through the origin can be drawn as a locus that represents a critical contact angle for each mineral sample (17).

Table 1 shows the values of γ_c and β for each mineral sample tested. The former is 51 mN/m and 64 mN/m for the cleavage plane and disk specimen of molybdenite, respectively. The corresponding β values are -1.84 and -1.5. In a previous study with molybdenite (17) in methanol solutions, the values of γ_c for the cleavage plane and disk specimen were found to be 29 mN/m and 42 mN/m, respectively, and the corresponding β values were -0.43 and 0.34. The γ_c values obtained in the lignosulphonate solutions are larger by 22 mN/m than those obtained in aqueous methanol solutions. The larger γ_c and more negative β values indicate that the wettability of molybdenite by lignosulphonate solutions is substantially greater than that by methanol solutions. Such a difference is expected on the basis of structural features of the two molecules. Methanol is the simplest monohydric alcohol, which may be viewed as the methyl (CH_{3-}) derivative of water. Lignosulphonate is a sulphonation product of lignin, and it has a complex macromolecular chemistry. The exact structure of neither lignin nor lignosulphonates is known. Figure 3 shows some of the characteristic features in a suggested structural model (19). The main frame of the macromolecule is made up of benzene rings, which are hydrophobic in nature. On this basis, hydrophobic bonding can be expected to operate as a specific mode of interaction between an

Table 1. Wetting Parameters Obtained for Talc and Molybdenite Samples in Aqueous Solutions of Sodium Lignosulphonate/MIBC

Mineral Sample	γ_c (mN/m)	β
Talc cleavage plane	33.5	−0.69
Talc disc	42	0.50
Molybdenite cleavage plane	51	−1.84
Molybdenite disc	64	−1.50

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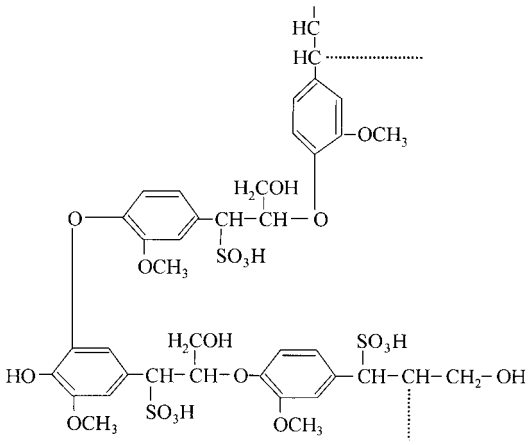

Figure 3. Main structural features in a simplified model for lignosulphonate.

inherently hydrophobic mineral and lignosulphonates. However, there are a number of hydrophilic groups, which are capable of interacting through hydrogen bonding. Depending on its molecular weight a lignosulphonate molecule may have as many as 70 sulphonate groups (20) in addition to a significant amount of phenolic hydroxyl and carboxylic groups.The presence of such hydrogen bonding groups in the molecule accounts for a more frequent mode of interaction in the adsorption of this reagent on many hydrophilic solids such as clays and calcium carbonate (21). Because molybdenite is known to lack a hydrogen-bonding capability on its cleavage surface, the adsorption of lignosulphonate is more likely to proceed through the hydrophobic bonding mechanism. Le Bell (20) also concluded that hydrophobic bonding was a dominant mechanism for the lignosulphonate-latex system.

Under the experimental conditions used, this mechanism does not effectively apply to the talc cleavage surface. An indication of this is that the wetting parameters obtained in the lignosulphonate solutions (Table 1, Fig. 2) do not significantly differ from those obtained in methyl alcohol solutions (15). This implies that surface activity of lignosulphonate on talc in aqueous solution is nearly as weak as that of methyl alcohol.

Relative Adsorption Densities

The slope of the adhesion tension line is related to adsorption by the following equation (22).

$$\beta = \mathrm{d}(\gamma_{\mathrm{lv}} \cos\theta)/\mathrm{d}\gamma_{\mathrm{lv}} = (\Gamma_{\mathrm{sv}} - \Gamma_{\mathrm{sl}})/\Gamma_{\mathrm{lv}} \quad (1)$$

where Γ_{sv}, Γ_{sl}, and Γ_{lv} are the relative adsorption densities of the solute at the three interfaces, solid, liquid, and vapor. A previous analysis of the adsorption densities for the system aqueous methanol solution/graphite (graphon) provided an empirical support for the relation expressed in Equation (1) (16). Lignosulphonates are, in general, large molecules with a molecular mass, for example, of 20,000 g/mole. Ignoring the activity of MIBC in the vapour phase (at a concentration of 10^{-4} M in the liquid phase), Γ_{sv} may be assumed to be negligible. This allows simplification to Equation (1) and leads to

$$\beta = -\Gamma_{sl}/\Gamma_{lv} \tag{2}$$

Considering the β values for the cleavage plane (mp) and disc (md) samples of molybdenite in Table 1, it will be seen that the adsorption density at the cleavage plane is greater than that at the disc sample.

$$(\Gamma_{sl})^{mp} = 1.84 \times \Gamma_{lv} \tag{3}$$

$$(\Gamma_{sl})^{md} = 1.50 \times \Gamma_{lv} \tag{4}$$

The latter sample has a mosaic surface structure and is considerably less hydrophobic (15). Because these samples share the same surfactant system, from Equations (3) and (4) one can get

$$(\Gamma_{sl})^{mp} = 1.2 \times (\Gamma_{sl})^{md} \tag{5}$$

The greater adsorption density on the cleavage sample suggests that hydrophobic bonding is the favored mechanism for adsorption of lignosulphonate on molybdenite. However, because the ratio is not greatly above 1, the hydrogen bonding mechanism is still relevant for polar regions of the disk sample.

The same analysis may be applied to compare the wetting and adsorption characteristics of molybdenite and talc. The following relation holds for the cleavage plane of the talc specimen.

$$(\Gamma_{sl})^{tp} = 0.69 \times \Gamma_{lv} \tag{6}$$

From Equation (3) and Equation (6)

$$(\Gamma_{sl})^{mp} = 2.7 \times (\Gamma_{sl})^{tp} \tag{7}$$

It is notable that the relative adsorption density of lignosulphonate is greater on molybdenite than that on talc by a factor of 2.7. Because the flotation characteristics of these two minerals are mainly controlled by the surface properties of their cleavage planes, one would expect molybdenite to be preferentially depressed in flotation.

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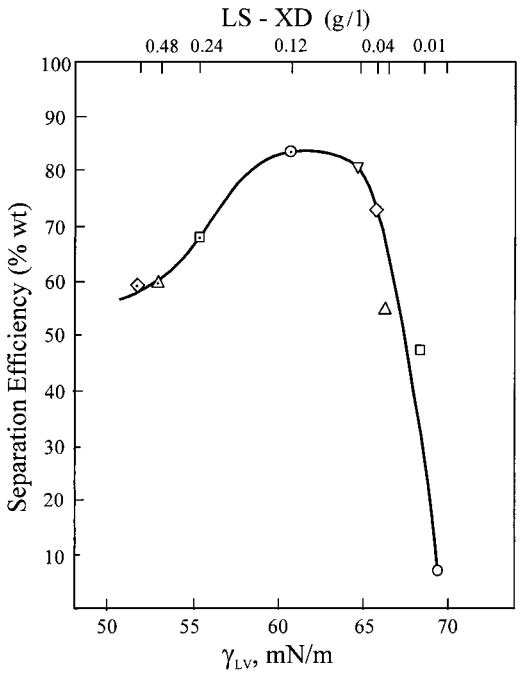

Figure 4. Separation efficiency of talc from molybdenite from their binary mixtures (50:50) in lignosulphonate/MIBC solutions of various surface tension.

Separation by Flotation

The separability of talc from molybdenite by flotation in solutions of lignosulphonate and MIBC is illustrated in Figure 4. For each surface tension value, a separation efficiency (23) has been calculated by subtracting the recovery of talc into the froth product from that of molybdenite. Alternatively, the flotation selectivity may be examined by plotting the talc recovery against the molybdenite recovery, which is shown in Figure 5. This type of representation is convenient, as the results can be simultaneously related to various levels of separation efficiencies, which are indicated by parallel lines. In these tests, the MIBC concentration was kept at the same constant level as in the case of wettability tests. The pH level of flotation medium for the lignosulphonate concentration range (0–0.50 g/l) was 7.2 ± 0.3.

The curve in Figure 4 has a maximum separation efficiency of 83% around the surface tension of 60 mN/m. In the alternate representation of the same data (Fig. 5), the maximum separation efficiency is characterized by a drastic change in the slope of flotation selectivity line. Note that the decrease in the separation efficiency

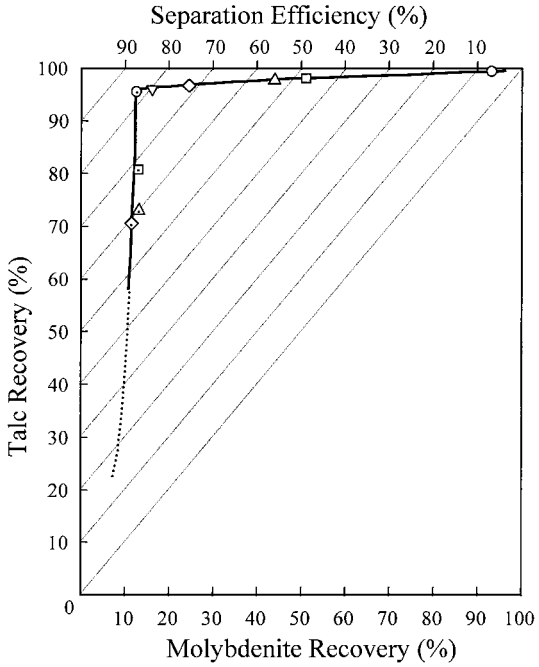

Figure 5. Representation of talc-molybdenite flotation selectivity in relation to their separation efficiency (symbols and raw data are the same as in Fig. 4).

is more pronounced above 65 mN/m than below 60 mN/m. At higher surface tensions both of these minerals become increasingly less wettable. This leads to high but similar floatabilities for both minerals so that the difference in their percent recoveries significantly diminishes with an increase in surface tension. This range represents a nonselective flotation (i.e., bulk flotation) case. At surface tensions lower than 60, where the disc sample of molybdenite is expected to be completely wetted, the face sample of molybdenite becomes increasingly more wettable as its γ_c value of 51 mN/m is approached. Because the γ_c value of the talc face sample is much lower than that of molybdenite, it is still incompletely wetted and highly floatable. The talc-molybdenite separation efficiency drops to about 60% between 50 and 55 mN/m. In this range, talc indicated somewhat lower recoveries at around 75%. However, molybdenite's flotation activity was still noticeable with a recovery of about 15% even in the vicinity of its γ_c. Although part of this recovery may be related to flotation by an "entrapment" mechanism, true flotation of a certain mass fraction also implies that γ_c and γ_{cf} values for the molybdenite cleavage plane are different in this surfactant system. In the case of methanol solutions, these two surface tension values were found to be very close to each other (17). The

nonequality of γ_c and γ_{cf} is not surprising and can be explained by considering the structural differences and molecular sizes of the surfactant components involved. Methanol represents one of the smallest heteropolar molecules. In its aqueous solutions, the surface tension equilibrium is approached very quickly, that is, within a few milliseconds (24,25). Therefore, the case of methanol solutions seems to be more compatible with dynamic conditions of flotation compared to that of sodium lignosulphonate/MIBC solutions. In contrast, surface tension lowering effect induced by large lignosulphonate structures is known to exhibit considerable time lag (21). The establishment of a particular surface tension by this system is very likely to require a longer period than available within the time scale of flotation. For the current case, the observation that some particles of molybdenite are still floatable around its γ_c value, may point out a higher surface tension for the bubbles in flotation than those involved in wettability tests, which are static in nature.

SUMMARY AND CONCLUSIONS

The use of adhesion tension diagrams has been demonstrated to characterize wettabilities of molybdenite and talc samples in sodium lignosulphonate/MIBC solutions. Both cleavage and disc specimens were used for each mineral at a pH of about 7.2. The slope value β for each case has been utilized as an indicator of relative adsorption densities. This approach has indicated that the adsorption density of sodium sulphonate at the solid/liquid interface of molybdenite face was significantly larger than that of talc by a factor of 2.7. It has been inferred from experimental data that hydrophobic bonding is the favored mechanism for adsorption of lignosulphonate. The difference in adsorption densities is the main reason for the observed selectivity in wetting and flotation separation of talc from molybdenite. However, the correlation between wettability and flotation in the lignosol separation of these hydrophobic minerals does not seem to be as clear as in the case of others tested with aqueous methanol solutions. This has been attributed to differences in time-dependent surface properties of surfactant solutions utilized in these tests.

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