# The Use of Fatty Chemicals as Flotation Reagents

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## Abstract

Fatty chemicals are widely used as flotation collectors in the mineral-processing industry and, even though this application is quite old, improvements are being made with regard to both application and utilization. Most of the fatty chemicals that are used in flotation are used in the processing of industrial minerals such as phosphate, potash, iron ore, cement rock, and feldspar.

The understanding of the functioning of collectors is not complete, and only recently have concerted efforts been made in researching this area with tools such as electrokinetics, radiotracers, infrared spectroscopy, and adsorption measurements. It appears that a thorough knowledge of the behavior of inorganic solids in aqueous solutions will be required before a full appreciation of collector behavior can be made.

## Introduction

FATTY CHEMICALS play an important role in many industrial processes, but nowhere is their importance greater than in the concentration of industrial minerals. They are used as the principal active ingredient in the flotation processing of phosphate, potash, and feldspar. Glass sand, barite, cement rock, and recently iron ore are also treated by flotation although they do not have the same importance as the first three. The reasons why flotation is important are found in the size of mineral particles that are treated effectively, the selective nature of the process, and the convenience of handling material as slurries.

Although flotation was discovered in the modern concept about the turn of the century, it was not until the 1930's that it was applied to phosphate and potash and that the important use of fatty chemicals began. In 1960 about 200 million tons of ore were treated by flotation in the United States, about one-sixth of which was industrial minerals.

Flotation is a physical separation process which utilizes induced differences in specific gravity to effect separation. An agglomerate of solid and air is formed which will float to the surface of the pulp and can be scraped off the surface. The usual separation in treating mineral deposits is between solids; one solid phase is selected from a mixture of several phases. Obviously it is necessary to liberate the phases from each other prior to the separation. Because there is a maximum size of solid particle that can be floated in a suspension, the ore must be reduced beyond this limiting size. The limit depends on the specific gravity difference between the solid and the liquid and the nature of the reagentized surface. For example, a 20-mesh particle (0.6 mm) is about the largest quartz or phosphate grain that can be floated whereas a 2-mm sylvite or coal granule can be floated. At the other end of the size spectrum there is a minimum-size particle that can be efficiently treated in mechanical subaeration cells. This lower-size limit is about 20 μ.

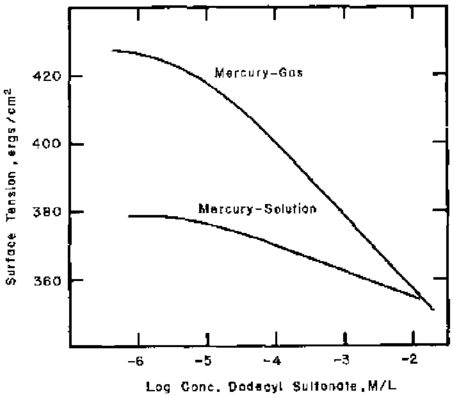

FIG. 1. Change in mercury surface tension caused by adsorption of dodecyl sulfonate.

After the material is liberated and properly sized, it is conditioned with a reagent or reagents that will cause one phase to be wetted by air rather than water. This selective reagentizing is the key to the separation and is the point where the fatty chemicals play their role. To understand something about the important properties of these chemicals in this regard, it is necessary to examine some theoretical aspects of wetting.

## Theoretical Background of Wetting

### Contact Angle

The degree of hydrophobicity of a surface is determined by the magnitude of the contact angle created by the attachment of a gas bubble to a solid when it is immersed in a solution. There are a few naturally hydrophobic solids, a characteristic which is determined by the number and density of broken van der Waals bonds as opposed to ionic bonds at the surface (1), but most minerals are hydrophilic.

To render a naturally hydrophilic mineral hydrophobic, a chemical is added which adsorbs at the solid-liquid interface. These chemicals, which are called collectors, consist of fatty acids, petroleum sulfonates, fatty amines, fatty sulfates, xanthates, and dithiophosphates. All are heteropolar with a hydrocarbon group on one end and an ionizable functional group on the other. The contact angle $\theta$ is measured between the solid and the gas through the liquid phase. A static force balance states that

$$\gamma SG - \gamma SL = \gamma GL \cos\theta$$

where $\gamma SG$, $\gamma SL$, and $\gamma GL$ are the interfacial free energies at the solid-gas, solid-liquid, and gas-liquid interfaces.

In many cases (2) the collector has little influence on $\gamma GL$, but it does lead usually to an increase in the contact angle. Under these conditions the quantity ($\gamma SG - \gamma SL$) must have decreased. Both values must have changed in the same direction (2); if $\gamma SL$ decreased, then $\gamma SG$ must have decreased more, or conversely if $\gamma SG$ increased, then $\gamma SL$ must have increased by a greater amount.

### Gibbs Adsorption Equation

Application of the Gibbs adsorption equation (3):

$$d\gamma = -\Sigma \Gamma_i \, d\mu_i$$

where $\gamma$ is the interfacial free energy, $\Gamma_i$ is the adsorption density of component i, and $d\mu_i$ is the change in chemical potential of component i, shows that positive adsorption leads to a decrease in the interfacial tension. It becomes obvious also that adsorption at the gas-solid interface must exceed that at the solid-liquid interface. These predictions were made from the considerations outlined and have been tested in the work of Smolders (4) and Aplan (5) (Figure 1), which show that the predictions are correct. The same arguments which apply to the adsorption of collectors can also be used to explain the action of activators (species which aid in the adsorption of the collector) and depressants (species which prevent the adsorption of the collector). In fact, these arguments suggest that such depressants as starch may function because they are adsorbed only at the solid-liquid interface and not at the gas-solid interface. A large decrease in $\gamma SL$ would result, but $\gamma SG$

would not be affected and the difference $(\gamma SG - \gamma SL)_2 - (\gamma SG - \gamma SL)_1 > 0$, which means that the contact angle would have to decrease.

## Collector Adsorption

The discussion up to now has dealt only with the outward result of collector adsorption and the formation of a contact angle. The means by which the collector is adsorbed at the interface is also of interest, and many attempts have been made to elucidate the mechanism of adsorption.

### Chemisorption

There are two distinct types of adsorption. The first, which is closely related to compound formation (6), is called chemisorption because it is believed to involve a large energy change and because the adsorbed material is held tenaciously at the interface (7,8). Examples of this type of adsorption are xanthates on sulfide minerals and oleates on such minerals as calcite, fluorite, and apatite. Recently (8–10) infrared spectroscopy has been used to study the nature of the bonding between the collector adsorbed on the mineral; Figure 2 shows the infrared spectra for oleate on fluorite. Figure 3a shows the adsorption density of oleate as a funtcion of pH for fluorite. The curve which reaches a maximum at pH about 9 is identified as the chemisorbed oleate by a combination of its infrared adsorption peaks at 6.4 and 6.8 $\mu$ and the fact that it was tenaciously held on the surface.

The relationship between adsorption and concentration is also characteristic of chemisorption, and this is shown in Figure 3b. Similar results have been obtained by using this infrared technique for oleate adsorption on hematite (11). Systems in which the collector is chemisorbed do not provide a large measure of selectivity. For instance, the calcium minerals referred to (calcite, fluorite, and apatite) all chemisorb oleates, and the adsorption goes through a maximum in the pH range from 8 to 10. In order to achieve selectivity in a system involving any two of these minerals, it is necessary to take advantage of slight differences in adsorption density by close control of the collector concentration, to use selective depressants, and even to adjust the temperature.

The identity of the collecting species is still subject to considerable speculation. Quartz in its pure state, for example, cannot be floated by using a long-chain fatty acid. The addition of an agent called an activator will cause the adsorption of oleate and subsequent flotation. Studies (12–14) have shown that, when quartz is activated by such alkaline earth ions as $Ca^{++}$ and $Ba^{++}$, the adsorption density of the activator must exceed that of the collector. In this system deactivation can occur by adding too much oleate. It was shown too that the solubility of the soap need not be exceeded to achieve good flotation.

Other heavy metal and alkaline earth ions will activate quartz for oleate flotation (15–17), and it has been suggested that an hydroxide complex of the activating ion is the active species. Thus it has been demonstrated that quartz is activated for sulfonate flotation by iron, aluminum, lead, manganese, magnesium, and calcium approximately in the order of the solubility of the hydroxides of these ions.

There are many complicating factors in understanding soap flotation, such as the complex nature of the soap ions and lack of data regarding the solubility of metal soaps. The data that are available were collected recently (18), but they are not very comprehensive.

Much effort (19–28,47) has gone into analyzing the flotation activity of various fatty acids, particularly the 18 carbon acids of differing degree of saturation. There does not exist much agreement between the investigators even though the topic seems straightforward enough. In general, it appears that the saturated fatty acids which form the most insoluble heavy-metal soaps are the best collectors. However getting these insoluble collectors into solution and adsorbed on the interface is difficult and has caused some confusion in experimental work.

One investigation (25) found that the order of efficacy of the 18 carbon fatty acids from stearic to linolenic was opposite, when floating hematite, from that which was obtained when floating calcium activated quartz.

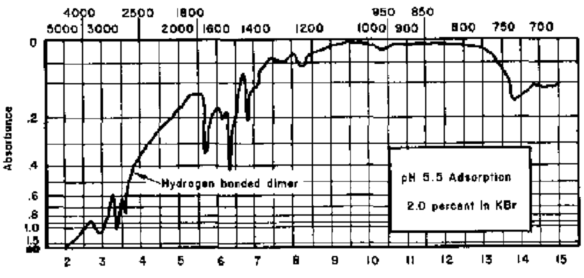

FIG. 2. Infrared spectra of chemisorbed oleate on fluorite.

A variety (29) of rather exotic fatty acids from 14 to 20 carbon were tested for phosphate separation from quartz, and no exciting differences were revealed. In general, most of the results could be rationalized on the basis of the collector solubility or emulsifiability in water and the solubility of the heavy metal soap of the mineral cation.

The choice of collector in any particular industrial application is rather interesting because of the different fatty acids available. Even from the by-products of paper manufacture several products, including soap skimmings, crude tall oil, refined tall oil, light ends, and rosin acids, are available. One of the Florida producers prefers soap skimmings whereas all of the others seem to prefer some form of fatty acid with a little rosin acid. US Patent 3,067,875 advocates a particular mixture of fatty and rosin acids as the most economical mixture.

### Reagent Addition

The problem of making the collector available for adsorption has received some attention both from a theoretical (30,31) and practical (32,33) viewpoint. One recommended procedure is to emulsify the fatty acid either wtih a frother or a sulphonate, or by mechanical agitation. One recipe (34) calls for an emulsion of fatty acid, sulfonate, and fuel oil as the flotation agent. In almost all cases where fatty acids are used, the reagent is conditioned with the mineral slurry at a high pulp density for about 2 to 10 minutes (34b). When amines are used as the collector, the reagentizing step may be omitted, as in quartz flotation from phosphate, or may be about 5 minutes as in potash.

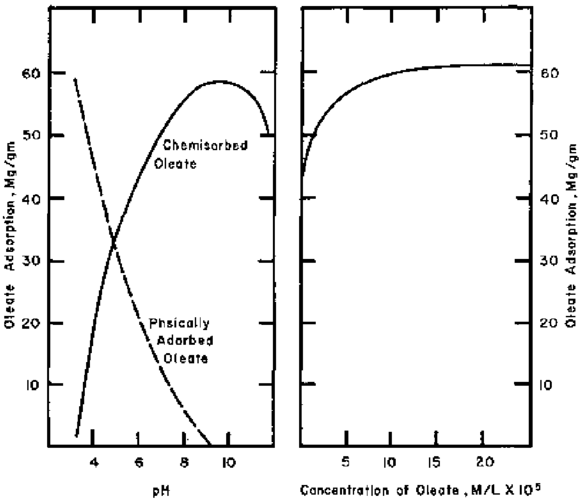

FIG. 3a. Adsorption of oleate on fluorite as a function of pH. 3b. Adsorption of oleate on fluorite as a function of oleate addition.

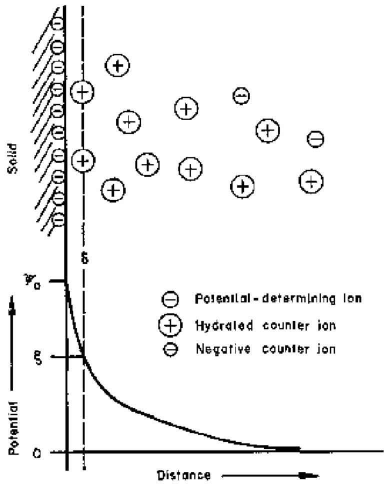

FIG. 4. Schematic representation of a solid-liquid interface.

**Temperature**

The beneficial effects of elevated temperatures have been recognized for many years (25,35–38), but only in the case of fluorspar flotation has it been utilized regularly. More recently (39,40) pulp heating for iron ore flotation has been adopted, and both concentrate grade and recovery have been improved. In most cases the majority of the benefits can be achieved by heating during only the reagentizing step. Since most flotation conditioning, especially that for fatty acids, is done at a high pulp density, the advantages of heating can be had for the minimum cost.

**Physical Adsorption**

The second kind of adsorption of importance in flotation is called physical adsorption because it depends on weaker bonds, is less tenacious, and shows little dependence on the substrate composition. The fatty amines, alkyl sulfonates (short chain), and alkyl sulfates are usually considered to be physically adsorbed. The energy of adsorption is derived from a combination of electrostatic and van der Waals bonds.

Before considering the adsorption, it is necessary to examine the characteristics of solid surfaces in contact with an aqueous solution. It has been recognized for a long time (41) that an electrical charge exists at the solid-liquid interface and that this charge can be varied in both sign and magnitude by adding certain chemicals to the system. A schematic representation of the interface is shown in Figure 4. The solid is visualized as having a fixed, uniformly distributed charge. To maintain electroneutrality an opposite charge of the same magnitude exists in the solution phase immediately adjacent to the solid.

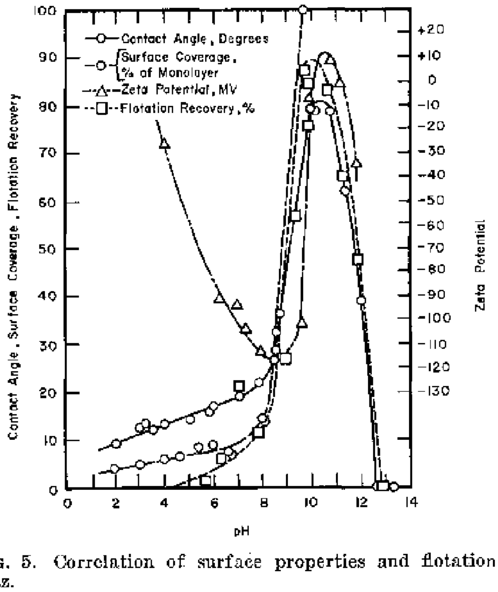

FIG. 5. Correlation of surface properties and flotation for quartz.

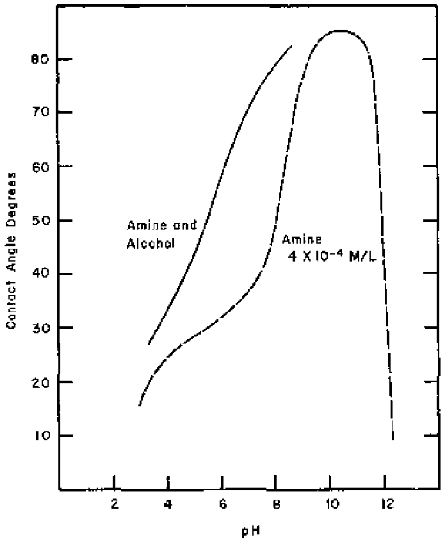

FIG. 6. Increase in contact angle caused by neutral molecule.

This system is what is referred to as "the double layer," and it has been described theoretically (41) and attempts have been made to use the theory of the diffuse double layer to explain the action of various chemicals in flotation (42–45).

The surface potential is governed by the concentration of potential-determining ions and can be calculated from the Nernst equation. The adsorption of the collector, which determines the flotation recovery, is described semiquantatively by the relationship

$$\Gamma = 2rn \exp\left(\frac{-Ze\zeta + \phi}{KT}\right)$$

where r is the radius of the adsorbed ion, n is the number of ions per cm³ in bulk, and Γ is the adsorption density within the Stern plane, ζ is the *zeta* potential that is considered to be the same as $\Psi_0$, φ is a specific adsorption potential caused by van der Waals bonding between the hydrocarbon portion of the collector molecule, K is the Boltzman constant, and T is the absolute temperature.

It has been demonstrated by a number of studies (25, 46–51) that flotation response is directly related to the surface potential. An example of the close correlation of the various measures of interfacial phenomenon is shown in Figure 5 (46). It is clear that as the *zeta* potential, which is a measure of the surface potential, increases, the adsorption of an oppositely charged collector increases. The data are presented as a function of pH because $H^+$ and $OH^-$ have been shown to be potential-determining for this mineral (52).

In fact, these studies all show that these ions are potential-determining for all simple oxides and some other minerals as well. The point at which the surface has zero charge (ZPC) is obviously an important characteristic of the flotation system. Many determinations of the ZPC of minerals have been made, and they were recently collected in an excellent review (53). The concepts of potential-determining ions and surface charge are not limited to oxide systems; in fact, silver iodide was one of the first solids studied extensively (41) and, more recently, attempts have been made to study sulfide minerals by adsorption techniques. Silver sulfide (54) is the only one successfully examined and the same type of behavior was found.

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# *Flotation Reagents . . .*

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The parameter $\phi$ was introduced as a specific adsorption potential to account for adsorption densities that are much greater than predicted by the original equation (55). It has been evaluated for $Ca^{++}$ adsorption on quartz (56) and dodecylamine on quartz (57), silver sulfide (58), and silver iodide (59). It is significant that the values determined for dodecylamine appear to be independent of the substrate and therefore must be characteristic of the dodecylamine.

### Hemimicelles

The observation that some peculiar association took place at a particular solution concentration led Fuerstenau (60) to postulate the existence of hemimicelles. These are regarded as two-dimensional analogs of micelles, and recently (61) it was shown that there is great similarity in the properties of micelles and hemimicelles. The measured value of the parameter $\phi$ was 1 KT (0.6 Kcal) per $CH_2$ group.

The concept of hydrocarbon association at the solid-liquid interface helps to explain many phenomena, including the effect of pH on weakly acidic or weakly basic collectors and the enhanced hydrophobicity that can be achieved by using a nonpolar hydrocarbon along with a collector. Recent studies (62–64) have shown that the addition of neutral molecules to a flotation system produces contact angles greater than any that can be achieved by the collector alone and that this results in an enhanced flotability (Figure 6).

The model proposed to explain these observations envisions a layer of adsorbed collector ions. Because of the charge on the polar end of the adsorbed molecule, there is a limit to the amount of adsorption of ionic species that can occur. However nonpolar molecules can adsorb by van der Waals bonding between the hydrocarbon portions of the molecules and thereby give rise to enhanced hydrophobicity. Because a certain collector adsorption is required before the extender can be adsorbed in any appreciable amount, the effect is selective and only those minerals with which a collector interacts will be changed.

These experiments and this model help to explain the fact, observed in all phosphate and potash plants, that the use of hydrocarbons (extenders) produces both higher grade and higher recovery.

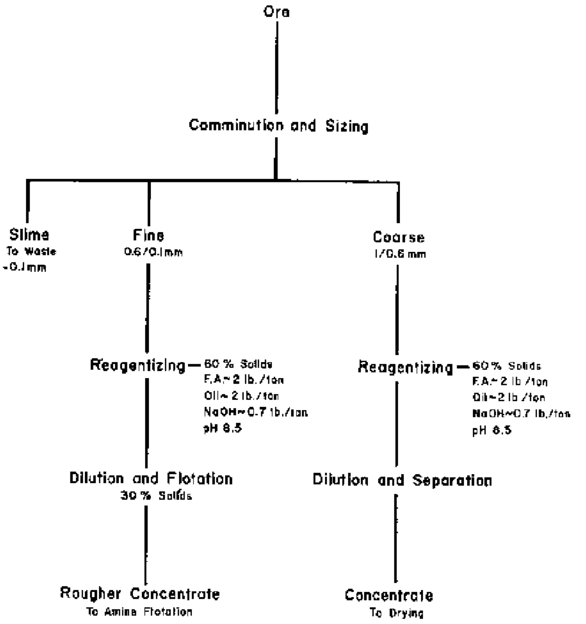

Fig. 7. Phosphate fatty acid flotation.

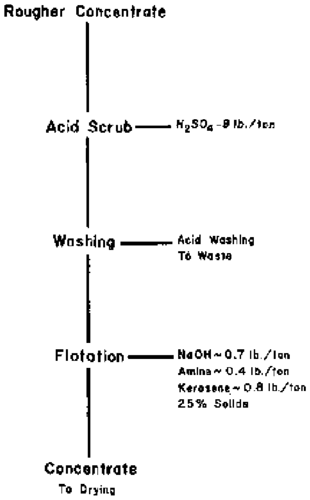

Fig. 8. Phosphate amine flotation.

This discussion has been presented under the topic of physical adsorption, but it appears that the same phenomenon applies to fatty acids (65) and certainly must apply to the interaction of fatty acids and hydrocarbons.

## Slimes

Associated with almost all mineral deposits is a certain amount of clay that results from the weathering of silicate minerals. Because of the clay some very fine particles are generated during liberation and by handling during processing. These fine particles are referred to as slimes and, even though they may be composed of widely different minerals, they are always troublesome in flotation. Several studies have been made to determine how slimes influence flotation (66–69), and it is now clear that these fine particles adsorb on the surface of large particles and hinder the attachment of bubbles. There are two remedies commonly applied. One is to eliminate the slime fraction, as is done in phosphate, potash, and iron ore treatment; the other is to add a reagent such as starch to make the slime size particles hydrophilic and unreactive toward the desired solid. The existence of slimes is important in the utilization of fatty chemicals because those collectors which are physically adsorbed, e.g., amines, sulfonates, and sulfates, are more sensitive to the noxious effects of slime than are the collectors which are chemisorbed, e.g., fatty acids. This is one of the reasons that a straight amine float of quartz is not used in phosphate rock processing. The fatty acid rougher flotation is not as adversely affected as an amine would be by the slime that slips into the feed stream. After rougher flotation and acid scrubbing the slime content is greatly reduced, and amines can be used for the second float.

## Industrial Flotation

The flotation processing of phosphate and potash will serve to illustrate the application of the principles which have been discussed. A description of these and other industrial separation processes can be found in the literature (70).

### Phosphate

The Florida phosphate (71) deposits consist essentially of three minerals, quartz, apatite (the phosphate mineral), and clay. The ore is mined by dragline, slurried with hydraulic giants, and pumped to the processing plant. A separation of the clay is made by desliming the feed slurry in either cyclones or hydroseparators. The lower-

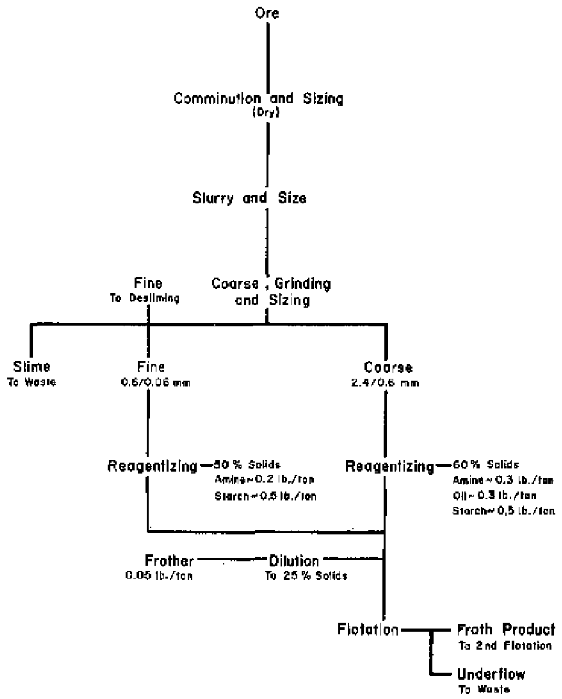

FIG. 9. Potash flotation.

size limit is usually about 0.1 mm, and the top size is limited to 1 mm. The feed is split into two-size fractions, a coarse feed 1 mm/0.6 mm and 0.6/0.1 mm. These two fractions are both treated with reagents in a similar fashion, but the coarse ore is treated on such devices as spirals, tables, belts, or underwater screens whereas the fine fraction is treated by froth flotation. The reagents added are sodium hydroxide to give a pH of 8.5, fatty acid, and an extender. The fatty acid is usually a tall oil fraction. If it is soap skimmings, it is added as a 5% aqueous emulsion whereas crude or refined tall oil would be added directly to the conditioner. It is usually necessary to heat the fatty acids so that they can be pumped and metered readily. The extender is usually a heavy oil fraction or a mixture of Bunker C and kerosene. The mixture is conditioned at about 70% solids for about 3 minutes, then is diluted and sent to the flotation cells. The conditioning step is an important one because the collector reagent must now become attached to the mineral. It is apparent that a high-energy intensity and short time are desirable, the high energy to assure thorough dispersion of the collector and the short time to minimize the generation of undesirable slime by the friable phosphate. The feed to the cells is about 30% BPL, 70% insoluble; the concentrate is about 65% BPL and 25% insoluble. (Figure 7).

This concentrate is scrubbed with sulfuric acid to remove the hydrophobic coating from apatite, then washed by decantation with cyclones. The washed product, which is now slime-free, is diluted to about 25% solids, the pH is adjusted to about 7.5, and amine and kerosene are added to float off the quartz. (Figure 8).

Primary amines have been the principal cationic collector for many years, but recently at least two phosphate producers have been using amidoamines. Since the amides have received little attention from the research community, almost nothing is known about their functioning. Recently *beta* primary amines were introduced and are reported to offer enhanced selectivity over normal primary amines in separating silica from iron oxide (72). The preparation of the amine is (70) important because it usually is given little conditioning time so that it must be in a readily dispersible form to be effective. The kerosene serves two functions; it acts as the extender as well as a froth-control agent. The long-chain fatty acids and amines act as frothers, and on occasions the froth becomes too watery and voluminous. Kerosene addition to a pulp with this kind of froth reduces the froth volume and makes the froth drier.

The underflow from the amine flotation is the final product with assays of 2–3% insoluble and 75–78% BPL. The recovery in both circuits is about 90%.

### Potash

Sylvinite, the principal ore of potash, is a mixture of sylvite (KCl), halite (NaCl), and clay. In some deposits, such as in Carlsbad, some sulfate minerals are present whereas in the Canadian potash mines there is no sulfate but there is some carnallite ($MgCl_2 \cdot KCl \cdot 6H_2O$) (73). Flotation separation of sylvinite ores constituted a major advance in flotation technology because it is carried out in a saturated brine.

The mined ore is comminuted to liberation size by a combination of crushing in impact mills in closed circuit with screens and of grinding with rod mills in closed circuit with classifiers. Ridding the ore of clay, a step called desliming, follows and is one of the most important steps in the processing and one of the most troublesome. The desliming must be done in saturated brine which contains approximately 1 lb of KCl per gallon. The recovery of brine from the slime is economically necessary but physically difficult. There always seems to be some swelling clay (montmorillonite group) which will not compact to high pulp densities in thickeners, a circumstance that leads to brine loss. (Figure 9)

After desliming, the ore is split into two-size fractions: coarse, which is approximately 2.4/0.6 mm, and fine, which is 0.6/0.06 mm. These fractions are treated separately through the reagentizing section.

The coarse is thickened to 60% solids by weight, conditioned with starch to overcome the adverse influence of residual slime, conditioned with amine and extender to render the sylvite hydrophobic, diluted to 25% solids and frother added, then sent to the first stage of flotation.

The fine fraction is thickened to about 50% solids by weight, conditioned with starch, then amine (extender is omitted) diluted, and sent to the first stage of flotation. For simplicity in the operation and for minimizing control problems the coarse and fine fractions may be combined before they are sent to the first stage of flotation. The froth product from the first or rougher stage of flotation contains an appreciable amount of fine halite, which is trapped in the froth.

The selectivity of amines for sylvite over halite is virtually perfect and is the subject of several theories (74–77). The amines used are distilled high-purity, produced from tall oil or animal fats with some degree of hydrogenation. The required degree of unsaturation (iodine value) depends on the temperature of the brine. Higher temperatures require a greater degree of saturation to maintain recovery at the same reagent addition. Amidoamines do not float sylvite. The rougher concentrate is treated in a second stage of flotation, called a cleaner, in order to drop out the occluded halite and to produce salable-grade concentrate.

There is a marked difference between phosphate and potash with respect to final product specifications because sylvite is a chemical compound with a unique composition whereas apatite is subject to a great deal of solid substi-

TABLE I
Consumption of fatty chemicals ($\times 10^{-6}$ lb/yr)

| | | F.A. | Amine | Oil |
|---|---|---|---|---|
| Phosphate | | 96.0 | 6.7 | 100 |
| Potash | (current) | ...... | 4.1 | 2 |
| | (announced) | | 2.0 | 1 |
| Iron ore | | 1.0 | 0.7 | 1 |
| | | 1.4[a] | ..... | ..... |
| Quartz, feldspar | | 3.0 | 2.0 | ..... |
| Total | | 101.0 | 15.5 | 104 |

[a] Petroleum sulfonate.

in the crystal lattice and consequently has a variety nical formulae.

### Problems

There are several problems common to phosphate and potash flotation, one of which is the flotation of coarse particles. There must be static equilibrium among the forces which hold a bubble and a particle together and, because the disruptive forces increase rapidly with size, there is a maximum size which can be floated. These disruptive forces increase as the density difference between the solid and the suspending liquid increases. The forces of adhesion increase as the contact angle increases, and that is why it is necessary to know something about wetting.

Another common problem is the identification of the proper extender characteristics. The advantages of using an extender are clear, but how to identify the best extender is not known.

The slime problem is, of course, vexing in both treatment schemes. At the present time the best treatment is to eliminate the fine material as thoroughly as possible. Starches and similar compounds called "blinders" are helpful, but their functioning is not thoroughly understood.

### Magnitude of the Industry

The consumption statistics of fatty acids, amines, and oil that are used as collectors in the flotation of phosphate, potash, and iron ore are given in Table I. If these figures are anywhere near correct, then phosphate processing consumes most of the fatty acids used for flotation. About 45% of the amines used in flotation are used in phosphate production also. The only other large consumer of flotation reagents is the potash industry, which currently consumes about 27% of all flotation amines. Despite the size of the iron industry only a small portion of the iron produced is treated by flotation and a correspondingly small portion of the total collectors is used.

The greatest increase in the consumption of flotation collectors of the fatty chemical group is likely to come from the expanding potash industry in Canada. A tremendous increase in capacity has been announced for the next five years, and if it is all brought on stream, it will nearly double the present capacity. The iron industry approximates a sleeping giant with respect to flotation, but it is unlikely that significantly large increases in flotation will occur within the next five years.

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# Plastics to Fight Corrosion

Recognizing the importance of reinforced plastics as a corrosion fighter, the Reinforced Plastics Division of the Society of the Plastics Industry has scheduled two sessions on Corrosion Resistance, one dealing with a new standard for the industry and the other with recent technical advances.

Write: Charles Condit, Secretary, The Society of the Plastics Industry, Inc., 250 Park Avenue, New York City, N. Y. 10017. Request a copy of the program of the 23rd Annual Conference, Shoreham Hotel, Washington, D. C., Feb. 6–9, 1968.

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## • *Obituaries*

L. J. Leonard (1956), Durkee Famous Foods, died August 8, 1967. He had been employed by Durkee Famous Foods, Chicago.

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