

United States Patent

Chemtob et al.

[15] 3,635,338

[45] Jan. 18, 1972

[54] **REAGENT FLOTATION OF BORAX FROM SALT MIXTURES AT LOW TEMPERATURES**

[72] Inventors: **Elie M. Chemtob**, Claremont; **William R. White**, Alta Loma, both of Calif.

[73] Assignee: **Occidental Petroleum Corporation**

[22] Filed: **Aug. 6, 1969**

[21] Appl. No.: **848,093**

[52] U.S. Cl. **209/11, 209/166**

[51] Int. Cl. **B03b 1/00, B03d 1/02**

[58] Field of Search **209/166; 23/121**

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Primary Examiner—Frank W. Lutter

Assistant Examiner—Robert Halper

Attorney—Christie, Parker & Hale

[57]

ABSTRACT

Sulfonated fatty acids and their salts are shown to be effective reagents for the low-temperature flotation of borax from a mixture of salt crystals, particularly a borax-Glauber's salt-natron salt complex.

5 Claims, No Drawings

REAGENT FLOTATION OF BORAX FROM SALT MIXTURES AT LOW TEMPERATURES

BACKGROUND OF THE INVENTION

Complex salt brines, as found in saline mineral bodies such as Searles Lake, are most difficult to separate into their salable components. At low temperatures in order of 20° to -20° C., sodium salts, including the borax salts, may be crystallized free of potassium values. The principal cooling harvest is a Glauber's salt (mirabilite)-natron-borax complex which is stable at temperatures below about 20° C. While fractional crystallization is a feasible method for separating this complex into salable components, fractional crystallization is expensive and of low efficiency. Flotation separations are more economical. However, little was known concerning the flotation separation of salts at temperatures essential to the preservation of the natron and mirabilite salt crystal structures.

SUMMARY OF THE INVENTION

It has now been found that sulfonated fatty acids, their salts or mixtures thereof are unusually effective reagents for the flotation separation of borax from salt mixtures, particularly a mirabilite-natron-borax salt complex, at low temperatures.

DESCRIPTION

According to the present invention sulfonated fatty acids, their salts, or mixtures thereof, are provided as reagents for the flotation separation of borax from salt mixtures at low temperatures.

The sulfonated fatty acids and salts used in practice of this invention are obtained by sulfonation of unsaturated straight chain fatty acids containing from about eight to about 22 or more carbon atoms in the chain. Illustrative, but nowise limiting the fatty acids which may be sulfonated for use in the practice of this invention, are caprylic, lauric, myristic, palmitic, stearic, oleic, linoleic, linolenic, arachidic, behenic, tall oil and like acids. Sulfonated fatty acids and sulfonated fatty acid salts having from about 18 to about 22 carbon atoms in their chain are preferred.

The sulfonated fatty acids used in the practice of this invention are readily obtained by procedures well known in the art and generally involve the reaction of the fatty acid under mild conditions with common sulfonating agents such as concentrated sulfuric acid, fuming sulfuric acid, sulfur trioxide, alkali disulfates, pyrosulfates, chlorosulfonic acid and the like.

The salts are obtained by similarly conventional means involving neutralization of the sulfonated fatty acid with common basic neutralizers such as sodium hydroxide and potassium hydroxide with sodium hydroxide being preferred. Fairly conventional flotation apparatus, such as Denver and Wemco flotation systems, and conventional techniques may be conveniently used in conjunction with the use of sulfonated fatty acids, their salts or mixtures thereof as a reagent for the flotation of borax from salt mixtures in an aqueous media. As indicated above, where the salt mixture contains natron and mirabilite, the flotation separation must be carried out at temperatures heretofore unexperienced in the flotation art. Accordingly, the flotation equipment used must be provided with means, such as conventional refrigeration equipment, to maintain the system at a temperature at which the natron and mirabilite salts will substantially retain their respective crystal identity. More particularly, this cooling equipment must be capable of maintaining the system at a temperature of about 20° C. or less, preferably from about 15° C. down to about -15° C. or less.

The flotation separation of borax, according to this invention, can be effectively carried out at slurry solid salt contents up to about 40 percent by weight, or more, although it is preferred for efficiency to carry out the flotation at a salt solid content between about 20 and 30 weight percent, and a particle size of -30 mesh Tyler or less.

The amount of reagent required to float borax from the salt complex is not narrowly critical. It has been found that as little

as 200 grams of the reagent per ton of salt crystal mixture can effectively separate more than 80 percent of the entrained borax at what is known in the art as a first rough separation, which is an economically leachable salt system. Finer separations have been found to yield purer floats.

Generally the amount of reagent used in the practice of this invention is from about 75 to about 500 grams of reagent, preferably from about 100 to about 300 grams, per ton of entrained salt solids depending, in part, on the estimated borax content of the mixture.

While the sulfonated fatty acids and fatty acid salts were established to be effective low-temperature reagents for the flotation separation of borax, it is evident that they will be equally or more effective at higher temperatures where it is sought to separate borax from less temperature-sensitive salt systems. Accordingly, it is contemplated within the scope of this invention to use sulfonated fatty acids and salts thereof as flotation reagents for the separation of borax from other salt systems at higher temperatures.

The following are examples of the flotation separations achieved according to the practice of this invention.

EXAMPLE 1

A harvest of mirabilite, natron and borax salts was obtained by cooling Searles Lake brine to a temperature of -15° C. and holding the brine at this temperature until a major amount of the borax contained in the brine crystallized. Sulfonated tall oil acid was then added to a slurry containing 25 percent salt solids content in a chloride free brine in the amount equivalent to 100 grams per ton of contained salt crystals. The slurry was agitated and aerated at a temperature of -15° C. in a Denver Cell. In this rough flotation of borax there was obtained at 60.0 percent yield of boron in the float, which was found to be 41.5 percent pure borax. This represented nearly a 400 percent beneficiation of borax.

An analysis of this system before and after flotation is shown in table I.

TABLE I

| | Cl | Analysis % | | |
|-------------------------------|--------|---------------------------|-----------------|-------------------------------|
| | | SO ₄ | CO ₂ | B ₂ O ₃ |
| Feed—solid (with entrainment) | 9.49 | 5.20 | 10.26 | 3.67 |
| Liquor | — | 8.0 | 8.28 | 0.99 |
| Float (entrainment free) | — | 0.42 | 12.05 | 16.85 |
| Residue (entrainment free) | — | 11.40 | 12.50 | 1.58 |
| End Liquor | 2.95 | 6.40 | 7.55 | 1.05 |
| | | Composition of Solids (%) | | |
| | | Entrainment Free | | |
| | Ha'ite | Glauber | Natron | Borax |
| Feed—solid (with entrainment) | 14.0 | 20.2 | 56.5 | 10.3 |
| Liquor | — | — | — | — |
| Float (entrainment free) | — | 1.4 | 57.5 | 41.5 |
| Residue (entrainment free) | — | 38.3 | 59.5 | 3.84 |
| End Liquor | — | — | — | — |

EXAMPLE 2

To a harvest of mirabilite, natron and borax salts obtained in the manner set forth in example 1, there was added sulfonated oleic acid in an amount equivalent to 250 grams per ton of contained salt crystals. The slurry was agitated and aerated at a temperature of -15° C. in a Denver Cell. In this rough flotation 75 percent of the boron was extracted at a borax purity of 43.5 percent, which represented close to a 600 percent beneficiation of borax. An analysis of this system before and after flotation is shown in table II.

TABLE II

| | | | | |
|--|--|--|--|--|
| | | | | |
|--|--|--|--|--|

| | Cl | Analysis % | | |
|-------------------------------|-------|-----------------|-----------------|-------------------------------|
| | | SO ₄ | CO ₃ | B ₂ O ₃ |
| Feed—solid (with entrainment) | 10.75 | 5.0 | 8.05 | 2.4 |
| Liquor | — | 7.2 | 9.26 | 1.44 |
| Float (entrainment free) | — | — | 12.7 | 17.7 |
| Residue (entrainment free) | — | 10.55 | 15.6 | 0.97 |
| End Liquor | 2.18 | 6.88 | 8.85 | 1.57 |

| Feed—solid | Halite | Composition of Solids (%) | | |
|----------------------------|--------|---------------------------|--------|-------|
| | | Entrainment Free | | |
| | | Glauber | Natron | Borax |
| (with entrainment) | 14.2 | 18.2 | 60.2 | 7.6 |
| Liquor | — | — | — | — |
| Float (entrainment free) | — | — | 60.5 | 43.5 |
| Residue (entrainment free) | — | 33.6 | 66.5 | 2.6 |
| End Liquor | — | — | — | — |

EXAMPLE 3

The procedure of example 2 was repeated except that the sulfonated oleic acid concentration was reduced to an amount equivalent to 200 grams per ton of contained salt crystals. The rough flotation extracted 82.5 percent of boron at a borax purity of 61.2 percent, which represented close to a 450 percent beneficiation of borax. An analysis of the salt system before and after flotation is shown in table III.

TABLE III

| | Analysis percent | | | Composition of solids (percent), entrainment free | | | | |
|-------------------------------|------------------|-----------------|-----------------|---|--------|---------|--|-------|
| | Cl | SO ₄ | CO ₃ | B ₄ O ₇ | Halite | Glauber | Tetrahydrate | |
| | | | | | | | Na ₂ CO ₃ ·4H ₂ O | Borax |
| Feed—solid (with entrainment) | 8.25 | 6.8 | 10.75 | 3.88 | 9.5 | 31.5 | 43.5 | 13.2 |
| Liquor | — | 6.8 | 8.68 | 1.88 | — | — | — | — |
| Float (entrainment free) | — | — | 14.95 | 25.1 | — | — | 44.3 | 61.2 |
| Residue (entrainment free) | — | 15.8 | 10.2 | 6.0 | — | 53.1 | 30.2 | 14.8 |
| End Liquor | 1.75 | 6.7 | 8.45 | 1.21 | — | — | — | — |

EXAMPLE 4

The procedure of example 2 was repeated except there was used as the collector sulfonated tall oil acid in an amount equivalent to 300 grams per ton of salt crystals. The rough flotation resulted in a boron yield of 73 percent. The borax purity was 40.8 percent, which represented a beneficiation close to 400 percent. An analysis of the salt system before and after flotation is shown in table IV.

TABLE IV

| | Borax | Natron | Mirabilite | Halite |
|---------|-------|--------|------------|--------|
| Feed | 10.3 | 56.5 | 20.2 | 14.0 |
| Float | 40.8 | 56.9 | 2.4 | — |
| Residue | 3.2 | 46.2 | 50.6 | — |

EXAMPLE 5

The procedure of example 4 was repeated except that sulfonated tall oil acid concentration was increased to an equivalent of 400 grams per ton of salt crystals. This rough extraction resulted in a boron yield of 64 percent, a borax purity in the flotation of 63 percent and a borax beneficiation of about 400 percent. An analysis of the salt system before and after flotation is shown in table V.

TABLE V

| | Borax | Natron | Mirabilite | Halite |
|---------|-------|--------|------------|--------|
| Feed | 13.2 | 43.5 | 31.5 | 9.5 |
| Float | 53.0 | 47.0 | trace | — |
| Residue | 12.1 | 40.0 | 44.8 | — |

What is claimed is:

1. A process for the flotation of borax from a mineral salt grouping containing a natron-mirabilite-borax salt complex which comprises subjecting said salt system to froth flotation to float a major portion of the borax from said complex at a

temperature of from about -15° C. to about 20° C. in an aqueous media using a beneficiating amount of a collector reagent selected from the group consisting of sulfonated straight chain fatty acids containing from about eight to about 22 carbon atoms and salts of a sulfonated straight chain fatty acid containing from about eight to about 22 carbon atoms.

2. A process as claimed in claim 1 in which the reagent contains from about 18 to about 22 carbon atoms in the fatty acid chain.

3. A process as claimed in claim 1 in which the reagent is present in an amount equivalent to from about 75 to about 500 grams per ton of solid crystals treated.

4. A process as claimed in claim 3 in which the reagent is sulfonated oleic acid.

5. A process as claimed in claim 4 in which the reagent is sulfonated tall oil acid.

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