FLOTATION CONCENTRATION OF HALITE

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This invention relates to the use of novel reagents as collectors for the flotation of halite (NaCl). More specifically, the invention relates to the use of reagents known as fatty morpholines as collectors for halite in sylvinite, other water soluble ores, or other partially water soluble ores.

Sylvinite ordinarily is a crude mixture of about ½ sylvite (KCl) and ½ halite (NaCl) with minor amounts of miscellaneous impurities. Sylvinite is a major source of KCl for fertilizer and industrial use and is abundantly available in the Southwestern United States and parts of Canada.

One method employed to separate the sylvite and halite is by flotation of sylvite particles from halite particles where the liquid medium is a saturated brine of the ore. Cationic organic reagents, primarily fatty amines, as shown by Kirby, U.S. Patent No. 2,088,325, are used as collectors for the sylvite.

Another method used to separate the salts is by flotation of halite particles from sylvite particles where the liquid medium is a saturated brine of the ore. Anionic organic reagents, fatty acids, in the presence of lead or bismuth ions are used as collectors for the halite. The methods and reagents taught by the prior art have several disadvantages. The method adopted by industry, involving the anionic collector, required the use of relatively large portions of collecting agents and the addition of lead and bismuth ions. On the other hand, the conversion of a plant to the cationic method from the anionic method requires a reversal of the previous methods of handling the concentrates and tailings. As halite is the major constituent of sylvite ore, the reversal of the methods of handling the concentrates and tailings results in a lowered plant capacity or requires additional investment in equipment to maintain capacity.

An object of this invention, therefore, is to teach a new and improved process of separating KCl from NaCl by concentrating the NaCl in the froth portion of a froth flotation system.

Another object of this invention is to teach a new process of concentrating the NaCl and impurities in the froth portion of the flotation system by use of novel collecting agents which are used in small amounts and which do not necessitate the presence of lead or bismuth ions in the flotation liquor.

A further object of this invention is to teach a new and improved process of concentrating the NaCl and impurities present in sylvinite, in the froth portion of the flotation system.

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Further objects and advantages of this invention will appear as the specification proceeds.

These objects are accomplished by utilizing as a collecting agent in a froth flotation system a novel class of collecting agents.

We have made the unexpected discovery that, although the prior art teaches the use of cationic collection agents to concentrate the KCl in the froth, the addition of relatively small amounts of new cationic collecting agents commonly known as the "fatty morpholines" will result in the NaCl being concentrated in the froth portion of the flotation system.

These new collecting agents have the formula

\[
\begin{align*}
R-N- & \quad \text{where } R \text{ is an aliphatic hydrocarbon group or substituted aliphatic hydrocarbon group containing 8 to 22 carbon atoms.}
\end{align*}
\]

It is not essential that a single morpholine be used as the collecting agent, as mixtures of the morpholines may be utilized in the invention. The mixture of morpholines produced when tallow, hydrogenated tallow and coconut oil fatty acids or other fatty acids, or selected fractions thereof, are converted to the amines and then to the fatty morpholines are particularly well suited for use as collecting agents. In general, fatty morpholines containing 12–18 carbon atoms in the fatty groups are preferred. The mixtures referred to above and containing the whole mixed derivatives of these fatty acids are, for purposes of this invention, referred to hereinafter as tallow, hydrogenated tallow or coco morpholines, respectively.

These morpholines may be prepared by conventional means known to the art. A particularly useful source of morpholines is the process disclosed by Erickson in U.S. Patent 2,694,707, which comprises reacting a fatty amine with 2-chloroethyl ether at temperatures of 50°–200° C.

In practicing this invention with sylvinite, the ore is prepared for treatment in the usual way by grinding or otherwise, reducing the same to liberation size or smaller. While particle size may vary considerably, that which approximates 35 to +48 mesh is considered preferable.

After grinding it is preferable to deslime the ore before adding the frothing, collecting and pH reagents. This step is not absolutely essential, but it is helpful in
reducing the amount of reagents consumed in the flotation process.

The ground, deslimed ore is then mixed with a saturated brine of the ore to produce a pulp of about 25% solids. At this stage, prior to commencing the flotation action, it is convenient to add slime control, collecting, frothing, and pH control reagents to the pulp, preferably in the described order.

The pulp is then placed in a suitable flotation device such as a Fagergren cell or the like, and through use of the novel cationic collecting agents of the present invention a selective flotation is conducted.

The use of the fatty morpholine cationic collecting reagents causes a most unexpected result; the sodium chloride is entrapped in the froth, while the potassium chloride in a substantially pure form is discharged from the flotation system in the liquid portion.

In the practice of this invention the morpholine collecting agent may be used in concentrations ranging from .001 pound to 3 pounds per ton of ore; larger concentrations may be utilized but offer no advantage. We find the preferable range of morpholine concentration is from .4 to .8 pound per ton of ore.

This invention is further illustrated by reference to the following examples in which all parts are expressed as parts by weight and all percentages as percent by weight unless specified otherwise.

EXAMPLE I
Fifty grams of -35 mesh sodium chloride were dispersed in 300 cc. of saturated sodium chloride brine. One cc. of an aqueous 1% solution of coco morpholine hydrochloride was added to, and well mixed with, the dispersion. The mixture was placed in a small flotation cell and the pH of the mixture was adjusted to a pH between 2 and 7 with HCl and NaOH. When air was introduced into the flotation cell, a froth containing the sodium chloride collected at the surface of the cell. The sodium chloride was removed from the froth.

A similar experiment was conducted with dodecyl morpholine hydrochloride and octadecyl morpholine hydrochloride and similar results were obtained.

EXAMPLE II
Fifty grams of -35 mesh potassium chloride were dispersed in 300 cc. of saturated potassium chloride brine. Up to 4 cc. of an aqueous 1% solution of coco morpholine hydrochloride were added to and well mixed with the dispersion. The mixture was placed in a small flotation cell and the pH of the mixture was adjusted to various pH's between 2 and 7 with HCl and KOH. When air was introduced into the flotation cell, no significant amounts of potassium chloride collected at the surface of the cell.

The experiment was repeated with dodecyl morpholine hydrochloride and octadecyl morpholine hydrochloride, and similar results were obtained.

EXAMPLE III
Partially deslimed New Mexico potash ore of -35 mesh and consisting of 65-35% potassium chloride was mixed with a saturated brine of the ore to produce a 25% solids pulp. Various amounts of a solution or dispersion of a fatty morpholine were added to samples of the pulp, to which had been previously added a sufficient quantity of HCl to adjust the pH to 4-5 and an amount of guar equivalent to .3 pound of guar per ton of pulp, as a slime control reagent. The reagent-treated samples were introduced into a flotation cell and the sodium chloride was floated from the potassium chloride. The results are contained in Table I.

The following examples illustrate the preferred embodiment of this invention for separating NaCl and impurities from KCl in ores and salt mixtures containing potassium and sodium chlorides.

Obviously, many variations of the invention as hereinbefore set forth may be made without departing from the spirit and scope thereof and therefore only such limitations should be imposed as are indicated in the appended claims.

We claim:
1. The process of separating sodium chloride from a mixture containing sodium chloride and other water soluble material which comprises subjecting said mixture to a froth flotation treatment in the presence of a collector having the formula

   \[
   \text{R} = \begin{array}{c}
   \text{CH}_2
   \\
   \text{CH}_3
   \end{array}
   \]

   where R is an aliphatic hydrocarbon radical containing from 8–22 carbon atoms.

2. The process of separating potassium chloride from a mixture containing sodium chloride and potassium chloride which comprises subjecting said mixture to a froth flotation treatment in the presence of a collector having the formula

   \[
   \text{R} = \begin{array}{c}
   \text{CH}_2
   \\
   \text{CH}_3
   \end{array}
   \]

   where R is an aliphatic hydrocarbon radical containing from 8–22 carbon atoms.

3. A process as set forth in claim 2, wherein the mixture is formed from sylvite ore and a substantially saturated solution of the soluble ore constituents.
4. A process as set forth in claim 2, wherein coco morpholine is used as the collector.
5. A process as set forth in claim 2, wherein tallow morpholine is used as the collector.
6. A process as set forth in claim 2, wherein dodecyl morpholine is used as the collector.
7. A process as set forth in claim 2, wherein hydrogenated tallow morpholine is used as the collector.

References Cited in the file of this patent

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