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COLLECTION IN FLOTATION OF SYLVITE

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2 Claims. (Cl. 209—166)

This invention relates to new and useful reagents as collectors in the flotation of sylvite. More specifically, this invention relates to the use of fatty imidazolines which upon standing under hydrolytic conditions yield fatty aminoamide compounds as collectors in sylvite flotation.

Sylvite (KCl) in its crude ore form is obtained in a mixture with sodium chloride and other miscellaneous impurities. These crude ores are a major source of potassium chloride and are abundantly available in the southwest part of the United States, especially New Mexico. A method employed to separate the potassium chloride and sodium chloride and other impurities is by flotation of potassium chloride particles from sodium chloride particles where the liquid medium is a saturated brine of these ores.

Various reagents are utilized as flotation aids in the above described separation. Among these are fatty amines as shown in Kirby, U.S. Patent No. 2,088,325. These amines are used extensively in the form of their acetate salts. Weiner, U.S. Patent No. 2,329,149, shows the use of the mono-fatty amides of ethylene diamine as a flotation aid.

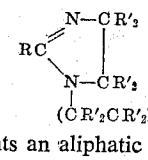
It has now been discovered that certain fatty imidazolines prepared from at least tri-functional polyamines may be utilized to prepare effective flotation aids, i.e., fatty aminoamides containing a plurality of free amine groups by hydrolysis of the imidazoline. This hydrolysis may be carried out either before or after the addition of the reagent to the ore pulp. The hydrolysis of these imidazolines to the corresponding aminoamide is aided by heating the mixture and maintenance of the hydrolysis medium in an alkaline state.

An object of this invention is to teach a new class of collectors which readily disperse in water and brine solutions. For instance, in the use of fatty amines it is necessary to prepare salts such as fatty amine acetates in order to achieve the necessary water dispersibility whereas the reagents encompassed by this invention are dispersible without any modification or alteration.

Another advantage to the use of some of the reagents taught by this invention is their ease of handling since they are much more fluid than other reagents presently employed.

A still further advantage to the reagents as flotation aids is that only a small quantity is necessary to achieve the desired results.

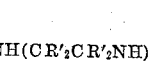
Imidazolines which may be used in accordance with the teachings of this invention have the following structural formula,



$$(CR'_2CR'_2NH)_{x-1}H$$

where R represents an aliphatic hydrocarbon radical containing 7–21 carbon atoms, x is an integer more than 1 and less than 6 and R′ is hydrogen or a lower alkyl radical such as methyl, ethyl, or propyl.

The hydrolysis products of these imidazolines are the corresponding aminoamides which have the fromula



$$RC(=O)-NH(CR'_2CR'_2NH)_xH$$

where R′ and x are as previously defined.

In the above formula R–C corresponds to the aliphatic hydrocarbon radical contained in the naturally occurring fatty acids (RCOOH) containing 8 to 22 carbon atoms. Illustrative acids are oleic, linoleic, linolenic, stearic, lauric, myristic, ricinoleic, undecylenic, behenic and so forth. Vegetable and animal fats, oils and waxes are the common sources of these acids. Illustrative of such sources are castor oil, coconut oil, corn oil, cottonseed oil, horse fat, lard oil, mutton tallow, beef tallow, neat's foot oil, palm oil, peanut oil, rapeseed oil, soya bean oil, sperm oil, whale oil, wool fat, Japan wax, olefin waxes, paraffin waxes, wax tailings, petrolatum, vegetable and animal phosphatidic materials, montan wax, carnauba wax, beeswax, spermacetic, castor oil distillate, ozokerite, tall oil, and the like.

The fatty imidazolines may be prepared by reacting at elevated temperatures fatty acids with polyamines having the general structural formula, $H_2N(CR'_2CR'_2NH)_xH$ where x is an integer where R′ is hydrogen or an alkyl radical containing less than 4 carbon atoms. Illustrative polyamines are diethylene triamine, triethylene tetramine, tetraethylene pentamine, di-1,2-propanetriamine, tri-1,3-propanetetramine, di-1,3-propanetrimaine and the like. R′ is preferably hydrogen.

This invention may be illustrated further by reference to the following example in which all "parts" are expressed as parts by weight and all "percentages" are expressed as precent by weight unless specified otherwise.

EXAMPLE I

Fifty grams of 150 mesh potassium chloride were dispersed in 300 cc. of saturated potassium chloride brine. One cc. of a 1% basic solution (pH 7.1–12.0) of hydrolyzed imidazoline from soybean fatty acids and diethylene triamine was addde to and well mixed with the salt dispersion . The mixture was placed in a small flotation cell and the pH of the mixture was adjusted to pH 7.0 with HCl and sodium hydroxide. When air was introduced into the flotation cell a froth containing the potassium chloride collected at the surface of the cell and the potassium chloride was removed with the froth.

EXAMPLE II

Deslimed New Mexico potash ore of —35 mesh and consisting of 30–35% potassium chloride was mixed with a saturated brine of the ore to produce a 25% solids pulp. Various amounts of a basic solution of the hydrolyzed fatty imidazoline described in Example I were added to samples of the pulp with constant amounts of pH and slime control reagents. The reagent treated samples were introduced into a flotation cell and the potassium chloride was floated from the sodium chloride. The results of these experiments are shown in the following table:

Table I

Sample Number	Reagent, Lbs./ton	Product	Percent Wt.	Percent NaCl	Percent Recovery KCl	Percent Recovery NaCl
1	0.15	Conc.	16.6	22.5	39	5.5
		tails	83.4		61	94.5
2	0.33	Conc.	28.6	9.4	75	4.1
		tails	71.4		25	95.9
3	0.46	Conc.	32.5	8.6	86	4.3
		tails	67.5		14	95.7
4	0.83	Conc.	35.8	11.6	95	6.2
		tails	64.2		5	93.8
5	1.6	Conc.	41.0	12.0	99	7.6
		tails	59		1	92.4

EXAMPLE III

Deslimed New Mexico potash ore of —35 mesh and consisting of 17.8% potassium chloride was mixed with a saturated brine of the ore to produce a 25% solids pulp. Various amounts of an unhydrolyzed fatty imidazoline

from soybean fatty acids and diethylene triamine were added to samples of the pulp with constant amounts of pH and slime control reagents. The samples were introduced into a flotation cell and attempts at flotation were made. The reagent had no effect except as a frothing agent.

EXAMPLE IV

Deslimed New Mexico potash ore of −35 mesh and consisting of 17.8% potassium chloride was mixed with a saturated brine of the ore to produce a 25% solids pulp. Various amounts of a basic water solution of the hydrolyzed fatty imidazoline described in Example III were added to samples of the same pulp with constant amounts of pH and slime control agents. The reagent treated samples were introduced into a flotation cell and the potassium chloride was floated from the sodium chloride. The results are shown in the following table:

Table II

Sample Number	Reagent, Lbs./ton	Product	Percent Wt.	Percent NaCl	Percent Recovery KCl	Percent Recovery NaCl
6	0.83	Conc.	19.7	21.6	87	4
		tails	80.3		13	96
8	0.36	Conc.	17.9	13.9	86.5	3.0
		tails	82.1		13.5	97
9	0.18	Conc.	17.2	13.4	83.5	2.5
		tails	82.8		16.5	97.5
10	0.09	Conc.	8.4	12.6	41.5	2
		tails	91.6		58.5	98
11	0.04	barren				

EXAMPLE V

Deslimed New Mexico potash ore of −35 mesh and consisting of 17.8% potassium chloride was mixed with a saturated brine of the ore to produce a 25% solids pulp. Various amounts of fatty imidazoline described in Example III which had been hydrolyzed to 73% aminoamide and 27% imidazoline and which had been prepared in acid water solution were added to samples of the pulp with constant amounts of pH and slime control agents. The reagent treated samples were introduced into a flotation cell and the potassium chloride was floated from the sodium chloride. Table III shows the results obtained.

Table III

Sample Number	Reagent, Lbs./ton	Product	Percent Wt.	Percent NaCl	Percent Recovery KCl	Percent Recovery NaCl
12	0.91	Conc.	22.9	20.1	99	2
		tails	77.1		1	98
13	0.33	Conc.	21.5	25.0	90.2	6
		tails	78.5		9.5	94
14	0.17	Conc.	20.2	17.0	94.5	5
		tails	79.1		5.5	95
15	0.09	Conc.	12.1	13.7	58.5	1
		tails	87.9		41.5	99
16	0.04	Conc.	12.9	7.5	67.0	1
		tails	87.1		33.0	99

EXAMPLE VI

Deslimed New Mexico potash ore of −35 mesh and consisting of 17.8% potassium chloride was mixed with a saturated brine of the ore to produce a 25% solids pulp. Various amounts of the fatty imidazoline described in Example III which had been hydrolyzed to 100% aminoamide and which had been prepared in acid water solution were added to samples of the pulp with constant amounts of pH and slime control reagents. The reagent treated samples were introduced into a flotation cell and the potassium chloride was floated from the sodium chloride. The results are shown in the following table:

Table IV

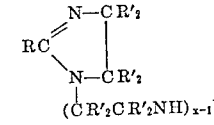
Sample Number	Reagent, Lbs./ton	Product	Percent Wt.	Percent NaCl	Percent Recovery KCl	Percent Recovery NaCl
17	0.91	Conc.	27.4	33	99	12
		tails	72.6		1	88
18	0.31	Conc.	23.4	25.0	98	8
		tails	76.6		2	92
19	0.17	Conc.	20.5	12.0	99	5
		tails	79.5		1	95
20	0.08	Conc.	17.5	16.3	82.5	4
		tails	82.5		17.5	96
21	0.04	Conc.	17.6	13.7	85	5
		tails	82.4		15	95

The flotation aids described in this invention may be employed in the range of 0.01 to 10 lbs. per ton of potash ore.

Many modifications and variations of the invention 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.

Now, therefore, we claim:

1. A process for the concentration of potassium chloride from a crude ore pulp containing potassium chloride particles, sodium chloride particles, and miscellaneous impurities in a saturated brine solution of the crude mixture which comprises adding to said crude ore pulp a substantially completely hydrolyzed product of a fatty imidazoline having the general structural formula:

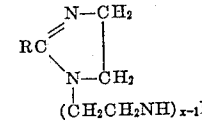


where R is an aliphatic hydrocarbon radical containing 7 to 21 carbon atoms, x is an integer more than 1 and less than 6, and R′ is selected from the group consisting of hydrogen and lower alkyl radicals, separating the treated ore pulp into two fractions one of which is relatively rich in potassium chloride and recovering said enriched potassium chloride fraction.

2. A process for the concentration of potassium chloride from a crude ore pulp containing potassium chloride particles, sodium chloride particles, and miscellaneous impurties in a saturated brine solution of the crude mixture which comprises adding to said crude ore pulp a solution of an amino amide having the formula

$$RCONH(CH_2CH_2NH)_xH$$

where R is an aliphatic hydrocarbon radical containing 7 to 21 carbon atoms and x is an integer more than 1 and less than 6, said aminoamide being the hydrolysis product of a fatty imidazoline having the general structural formula:



where R and x are as defined above, separating the treated ore pulp into two fractions one of which is relatively rich in potassium chloride and recovering said enriched potassium chloride fraction.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,009,575 November 21, 1961

James L. Keen et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 19, after "integer" insert -- more than 1 and less than 6 and --.

Signed and sealed this 17th day of April 1962.

(SEAL)
Attest:

ESTON G. JOHNSON
Attesting Officer

DAVID L. LADD
Commissioner of Patents