

- [54] FLOTATION OF SILICA
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[56] **References Cited**

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[57] **ABSTRACT**

Acid salts of a mixture of normal-alkyl primary amines having 5 to 12 successive chain lengths including both odd and even numbered carbon chains of 8 to 22 carbon atoms, the major proportion of the constituents being near the center of the chain length range, are used as collectors in the flotation of silica from iron ores.

9 Claims, No Drawings

FLOTATION OF SILICA

This invention relates to the concentration of iron minerals by froth flotation using a mixture of water dispersible acid salts of normal-alkyl primary amines having 5 to 12 successive chain lengths of 8 to 22 carbon atoms, the major proportion of the constituents being near the center of the chain length range. Using these flotation agents, we have found it is possible to obtain a higher selectivity of silica and thus produce better recovery of the iron than has been obtained with currently used collecting agents.

In the recovery and concentration of iron minerals from magnetic or non-magnetic iron ores, it is the undesirable silica and miscellaneous cherts and siliceous materials that are floated away. The desired concentrated iron minerals remain. Froth flotation is well known in the art for separating or concentrating minerals from ore. Prior to flotation, the ore is ground, pulped in water, and sometimes, depending upon the nature of the ore being treated, preconditioned with starch or dextrin which is a depressant for the desired iron portion of the ore. In the flotation step, a collector and a frothing agent are added to the ore dispersed in water and air is introduced into the pulp to form a froth, the silica-rich froth being floated away from the desired iron mineral residue, and the step repeated, usually 3 to 5 times.

Considerable effort has been extended to devise more efficient and more economical methods of operation to effect as complete a separation as possible and to produce a higher grade iron concentrate with minimum loss of iron, while consuming a minimal amount of amine collector. Various amines have been used in the past as flotation agents in the separation of silica from iron ore. For example, U.S. Pat. No. 3,404,777 teaches the use of secondary-alkyl primary amines in the separation of silica from iron ore and U.S. Pat. No. 3,363,758 teaches the use of acid salts of primary aliphatic ether amines as silica collectors in the concentration of iron. While many of these collectors have been satisfactorily used, it is always an object to obtain higher grade concentrate and higher recovery of iron minerals.

Accordingly, an object of this invention is to use acid salts of a mixture of normal-alkyl primary amines having 5 to 12 successive chain lengths including both odd and even numbered carbon chains of 8 to 22 carbon atoms, the major proportion of the constituents being near the center of the chain length range, as collectors in the concentration of iron by froth flotation processes. Another object is to use such materials as silica collectors in the froth flotation of iron ore. Another object of this invention is to provide a novel composition of normal-alkyl primary amines to provide highly selective collectors of silica. Other objects and advantages of this invention will become apparent from the following description and claims.

It has now been found that the use of the mixture of the water dispersible acid salts of normal-alkyl primary amines having 5 to 12 successive chain lengths including both odd and even numbered carbon chains of 8 to 22 carbon atoms wherein the major proportion of the constituents are located near the center of the chain length range, effect a marked improvement in silica separation from iron ore. The individual normal-alkyl primary amines may be represented by the formula:

$[R-NH_2]_n^+ [A]^{n-}$ wherein R is an alkyl group having 8 to 22 carbon atoms, A is the salt forming anion of an acid, and n is an integer equal to the valence of said anion. It is not always necessary that the amine be totally neutralized, in some cases 12 to 50 percent neutralization renders the amine water dispersible. It is preferred that the amine be 50 to 100 percent neutralized. Suitable anions include acetate, chloride, bromide, iodide, sulfate, nitrate, borate, carbonate, oxylate, sulfamate, phosphate and salicylate. The acetate salts are preferred.

Typical normal-alkyl groups suitable for R in the above formula include octyl, nonyl, decyl, undecyl, dodecyl, tridecyl, tetradecyl, pentadecyl, hexadecyl, heptadecyl, octadecyl, nonadecyl, eicosyl, heneicosyl and docosyl.

Especially suitable for use in this invention are mixtures of amines having chain lengths C_8 through C_{16} with the major constituents as C_{12} and C_{13} ; chain lengths C_{11} through C_{22} with major constituents as C_{17} , C_{18} and C_{19} ; and chain lengths C_{10} through C_{14} with major constituents as C_{11} and C_{12} .

The amines used in this invention are suitably prepared from synthetic fatty acid mixtures which contain both odd and even numbered successive chain lengths. Synthetic fatty acids are commercially available and are derived from paraffin waxes by air oxidation. The amines are produced from the acids by conventional methods. Such mixtures are different from those previously available from natural sources which only contain substantial amounts of even numbered chain lengths. It appears to be a requirement of this invention that the successive odd and even numbered carbon chains be present.

The process of this invention is carried out under well known techniques of froth flotation. In the case of magnetite ore the natural pH is slightly basic and it is generally not necessary to adjust pH for flotation. When using hematite ore it is usually necessary to adjust the pH to about 9 to 11 by addition of a base such as NaOH. Generally used cell speeds and temperatures according to known flotation techniques are suitable.

The concentration of amine salt used is about 0.01 lb. to 2.0 lbs. per ton of ore, the preferred range being about 0.1 to 0.5 lb. per ton of ore.

The collectors of this invention can be used in conjunction with other conventional chemicals used in froth flotation including activators, frothing agents, depressing agents, dispersing agents and such.

The following Examples further illustrate the advantages of this invention and should be considered as only exemplary of the invention.

EXAMPLE I

Magnetite concentrate ore samples were received with an average of 10 wt. percent water. The ores were then blended and head samples taken. Analysis showed that the ore averaged about 65 wt. percent iron by wet analysis and 8.5 to 10 wt. percent silica by X-ray diffraction. No. sizing studies were undertaken, although the ground ore averaged 80 percent passing 325 mesh. The orebody varied considerably in its gauge composition but is generally reported to hold about equal amounts of silica and iron silicates as the gangue material. The major iron silicate minerals and their silica content are as follows:

Mineral	% SiO ₂
Hypersthene	50.07
Grunerite	54.67
Ferrotremolite	45.73

500 gms. of ore, on a dry basis, was transferred to a 500 gram laboratory float cell, 1,500 rpm, and diluted to 50 wt. percent solids using Chicago tap water (medium hardness) at ambient temperature. The pulp was agitated 2 minutes to re-pulp. The ore was diluted to float solids. With agitation off, one-half of the amount of collector for three floats indicated in Table I in lbs. per ton was added in a 2.5 wt. percent water solution. Then 0.05 lbs. per ton methylisobutyl carbinol frother was added. The cell was turned on and the pulp was conditioned for 15 seconds without aeration. Flotation was carried out for three minutes with sufficient aeration for the froth to reach the lip of the cell. This floated product is known as the first Rougher Froth. The unfloated ore was re-pulped to float solids and using the same procedure, one-fourth of the amount of collector indicated in Table I and 0.025 lbs. per ton of frother were added, followed by conditioning and flotation. This product is known as the second Rougher Froth. The ore is re-pulped to float solids and the second Rougher Froth procedure is duplicated to produce the third Rougher Froth. Both the cell content (the iron concentrate) and the three froth products are separately filtered, dried, weighed and samples are taken for analysis of iron by wet analysis and for silica by X-ray diffraction. Table I shows analysis of second and third floats.

TABLE I

Collector		Flotation Concentrate			
Type	Float	lbs./ton	Recovered	Fe	% SiO ₂
SFA Cut 1 Acetate ⁽¹⁾ (100% neutralized)					
	2	0.15	86.9	69.42	3.11
	3	0.20	73.5	70.82	1.55
SFA Cut 2 Acetate ⁽²⁾ (100% neutralized)					
	2	0.15	86.2	69.51	3.09
	3	0.20	70.1	70.97	1.45
Comparative N-Sec-alkyl C ₁₅₋₂₀ trimethylene diamine acetate (100% neutralized)					
	2	0.12	88.8	68.64	4.41
	3	0.16	75.3	69.99	3.12
N-tridecoxypropyltrimethylene diamine monoacetate (50 % neutralized)					
	2	0.12	74.8	69.7	3.46
	3	0.16	57.2	71.0	2.41
N-Tallow Amine Acetate ⁽³⁾ (100% neutralized)					
	3	0.14	93.2	67.53	6.62
N-Coco Amine Acetate ⁽⁴⁾ (100% neutralized)					
	3	0.14	90.4	67.12	5.54
NOTE: ⁽¹⁾ and ⁽²⁾ SFA Cut 1 and SFA Cut 2 Amine is a mixture of normal-alkyl chain lengths as follows:					

Carbon Chain	SFA Cut 1	Percent	SFA Cut 2
C-8	4.9		
C-10	15.3		0.05
C-11	16.7		0.1
C-12	20.2		0.2
C-13	20.4		1.5
C-14	14.5		6.7
C-15	5.4		12.9
C-16	1.9		20.6
C-17			17.5
C-18	0.7		25.1
C-19			7.7
C-20			2.6
C-21			0.5

		Percent	
(3) Tallow Carbon Chain		Saturated	Unsaturated
C ₁₂		0.1	
C ₁₄		3	0.5
C ₁₆		29	2
C ₁₈		20	44.6
C ₂₀		0.8	
(4) Coco			
C ₈		0.1	
C ₈		5	
C ₁₀		7	
C ₁₂		45	
C ₁₄		17	
C ₁₆		7	
C ₁₈		4	14
C ₂₀			1

EXAMPLE II

Using magnetite ore as described in Example I, flotations were carried out as described in Example I using normal single chain length amine acetates 100 percent neutralized as a flotation agent for three floats for comparison with the mixtures of alkyl chain lengths according to this invention. It can be seen in Table II that the individual chain length amine salts did not produce the very satisfactory results obtained by use of mixtures of the same chain length amine salts shown in Example I.

TABLE II

Collector		Flotation Concentrate			
Type	lbs./ton	Fe Recovered	%Fe	%SiO ₂	
C ₁₀ Acetate	0.16	78.3	50.5	5.27	
C ₁₁ Acetate	0.16	55.3	27.3	4.11	
C ₁₂ Acetate	0.16	41.4	17.5	3.64	
C ₁₃ Acetate	0.16	40.6	16.5	3.50	
C ₁₄ Acetate	0.16	55.5	25.3	3.97	
C ₁₅ Acetate	0.16	79.8	52.8	5.48	
C ₁₆ Acetate	0.16	82.7	52.2	5.41	
C ₁₈ Acetate	0.16	96.5	91.0	8.21	

EXAMPLE III

Hematite ore samples from a Michigan ore body previously magnetically separated from magnetite, were subjected to flotation by the same process as described in Example I, except that 1 lb./ton 60-80 percent soluble British Gum (dextrin) and 2½ lb./ton NaOH to obtain pH greater than 9 were added. Results using the number of concentrates indicated are shown in Table III where the flotation agent is 100 percent neutralized.

TABLE III

		No. of	lbs.	MIBC			
45	Flotation Agent			Frother	Fe		
	Floats	/ton	lbs./ton		Recovery	%Fe	%SiO ₂
	SFA Cut No. 1	Acetate					
	5	0.35	0.22		87.43	60.91	5.20
Comparative							
	N-Sec-alkyl C ₁₁₋₁₅	trimethylene					
	diamine acetate						
50	5	0.35	0.22		83.44	58.87	6.51
	6	0.50	0.22		69.75	61.64	
	N-Coco Amine Acetate						
	5	0.35	0.22		66.22	61.91	
	3	0.21	0.16		77.53	60.31	
	N-Coco hydrogenated Amine						
55	Acetate						
	5	0.35	0.26		78.27	60.70	

While in the foregoing specification this invention has been described in relation to certain preferred embodiments thereof, and many details having been set forth for purpose of illustration, it will be apparent to those skilled in the art that certain of the details described herein can be varied considerably without departing from the basic principles of the invention.

I claim:

1. A froth flotation process for separating silica from iron ore which comprises froth flotation of said ore in an aqueous medium containing a mixture of water dis-

persable acid salts of normal-alkyl primary amines having 5 to 12 successive chain lengths including both odd and even numbered carbon chains of 8 to 22 carbon atoms, the major proportion of the constituents being near the center of the chain length range with at least two of said center chain lengths of said mixture comprising between 12.9 and 25 percent (by weight) each, and, floating the silica from said ore. 10

2. The process of claim 1 wherein said amine salts are used in an amount of 0.01 to 2.0 lbs./ton of ore.

3. The process of claim 2 wherein said amine salts are used in an amount of 0.1 to 0.5 lb./ton of ore.

4. The process of claim 1 wherein said salt is an acetate salts.

5. The process of claim 1 wherein said ore is magnetite.

6. The process of claim 1 wherein said ore is hematite.

tite.

7. The process of claim 1 wherein said amine salts are selected from the group consisting of mixtures of amines having chain lengths C_8 through C_{16} with the major constituents as C_{12} and C_{13} ; chain lengths C_{11} through C_{22} with major constituents as C_{17} , C_{18} and C_{19} ; and chain lengths C_{10} through C_{14} with major constituents as C_{11} and C_{12} .

8. The process of claim 1 wherein said amine salts are selected from the group consisting of mixtures of amines having chain lengths C_8 through C_{16} with the major constituents as C_{12} and C_{13} .

9. The process of claim 1 wherein said amine salts are selected from the group consisting of mixtures of amines having chain lengths C_{11} through C_{22} with major constituents as C_{17} , C_{18} and C_{19} .

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