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Flotation applied to silicate sludge

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FLOTATION APPLIED TO SILICATE SLUDGE.

BY

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AND

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A

THESIS

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Professor of Metallurgy.

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FLOTATION APPLIED TO SILICATE SLUDGE.

Within the past few years great success has been made of the flotation of sulfids, and to a certain extent of carbonates.

Since sulfids are easily recovered by flotation it seems practicable that silicates could be made to float by first sulfidizing them, and then subjecting them to ordinary flotation treatment.

This problem involves the treatment of a silicate sludge obtained from the Joplin district, the sludge will easily pass through a 250 mesh screen. At the present time this sludge is being sent to the tailings pond.

The experimental work of this thesis was done along the line of present flotation patents abstracted from the U. S. patent office bulletins, the abstracts will follow.

Abstracts of Flotation Patents.

807501 Process of Concentrating Ores. Alfred Schwarz. New York, N. Y. assignor to Schwarz Ore Treating Co.

Pheonix, Arizona.

1 The method of treating ores which consists in subjecting a non-sulfid ore to the action of a soluble sulfid to convert the mineral into a sulfid, then treating the mass with a hydrocarbon and finally separating the hydrocarbon with the entrapped metallic constituents of the ore from the tailings.

2 Same as (1) but subjecting ore to action of an alkaline sulfid.

3 Same as (1) but subjecting ore to action of an aqueous solution of potassium or sodium sulfid.

4 Same as (1) but treat resulting metallic sulfid with a melted hydrocarbon solid at normal temperatures.

5 Same as (4) but treat with compound of melted paraffin and resin.

807505 Process of Concentrating Ores. Alfred Schwarz. New York, N. Y. assignor to Schwarz Ore Treating Co. PHoenix, Ariz., A Corporation of Arizona.

1 The process of concentrating ores consisting of mixing the ore with an adhesive agent composed of

a hydrocarbon and sulfur, separating said agent with the entrapped values from the tailings, and recovering the values from the adhesive agent.

2 Same as (1) but ore is mixed out of contact with water with the adhesive agent and sulfur.

3 Same as (1) but hydrocarbon is solid at normal temperatures.

4 Same as (1) but adhesive agent is a resinous hydrocarbon and sulfur.

5 Same as (1) but adhesive agent is composed of a resinous and non-resinous hydrocarbon and sulfur.

6 Same as (1) but adhesive agent is composed of resin, paraffin and sulfur.

7 Same as (1) but adhesive agent is composed of resin and sulfur.

8 Same as (1) but washing the mixture with water.

9 Same as (8(but water is heated.

1140866 R. F. Bacon. U. S. Pat. Office BuLL., May 25/15

1 The method of effecting the separation of oxidized ores from associated gangue, which consists in subjecting the mixture in a finely divided condition, to the action of a soluble sulfid, thereby

effecting a conversion of the oxidized ore into sulfids, and then converting the H_2S present into constituents innocuous to flotation, and finally subjecting the mixture to flotation.

2 Subject to action of a soluble sulfid, thereby effecting a conversion of the oxidized ore into sulfids, the converting the H_2S present into constituents innocuous to flotation, and making the solution faintly acid, finally subjecting mixture to flotation.

3 Same as (2) making solution faintly acid by admission of sulfur dioxid.

1159142 H. B. Howland. U. S. Pat. Office Bull., Nov. 1, 1915.

1 The method of treating metalliferous materials which consists in bringing the metallic constituents into solution and sulfidizing the metallic constituents by means of calcium sulfid and feric sulfate.

2 Method of treating copper-bearing ores which consists in bringing the copper into solution and precipitating copper as copper sulfid by means of calcium sulfid and feric sulfate.

3 Method of sulfidizing metal which consists in bringing together a soltuion of the metal to be

1140865 R. F. Bacon. U. S. Pat. Office Bull., May 25, 1915.

1 The method of effecting separation of sulfid minerals from associated gangue, which consists in subjecting the mixture, in a finely divided condition to the flotation action of colloidal sulfur; substancially as described.

2 The method of effecting the separation of sulfid minerals from associated gangue, which consists in subjecting the mixture, in a finely divided condition, to the flotation action of colloidal sulfur the flotation solution being faintly acid.

3 Method which consists in subjecting the mixture in a finely divided condition, to the flotation action of colloidal sulfur, the flotation solution being produced by reaction there in between a soluble sulfid and sulfur dioxid.

4 Method which consists in subjecting mixture, in a finely divided condition, to the flotation action of colloidal sulfur, the flotation solution being produced by reaction therein between a soluble sulfid and sulfur dioxid, the sulfur dioxid being admitted in such excess as to make the solution of colloidal sulfur faintly acid.

1098668 Henry B. Hovland. U. S. Pat. Office Bull., June 2, 1914.

Art of Treating Metalliferous Materials.

1 The process which consists in reacting on a substantially dry metalliferous material with a substantially dry gaseous sulfidizing agent at ordinary temperature.

2 Same as above - - absence of exterior heat.

3 The process which consists in reacting on a copper containing material with a sulfidizing agent in the dry at ordinary temperature.

4 Reaction on dry materials with H_2S gas.

5 Reating on substantially dry metalliferous materials with H_2S gas in absence of exterior artificial heat.

1094760 Terry. U. S. Pat. Office Bull., Apr. 28, 1914.

1 A process which consists in the subjection of metalliferous particles of ore existing as carbonates, oxides, chlorides and sulfates, to the action of a hydrogen sulfid gas, then subjecting the resulting product to partial vacuum, then to agitation in the presence of an oil, film, forming substance and recovering the oil coated particles by flotation.

2 A process for preparing non-sulfid ores for flotation methods of concentration which consists in the subjection of metalliferous particles in ore pulp, to the action of hydrogen sulfid gas, subject to vacuum treat as above.

3 A process for the recovery of metalliferous constituents of ores, which consists in reducing the ores to a pulp, adding a soluble metallic salt and precipitating as a sulfid by H_2S to act as nuclei for the formation of sulfid granules and coagulations then subjecting the resulting product to partial vacuum, subject this pulp to flotation.

10022085 James M. Hyde. U. S. Pat. Office Bull., April 2, 1912.

1 The process of concentrating ore pulps comprising a separation treatment which consists in adding to the pulp a material which preferentially coats the valuable particles of the ore.

2 Addition to pulp of an acid precipitant adapted to react upon the ore, allowing a time interval to elapse prior to subjecting the pulp to a separation treatment, then subjecting the pulp

to a separation treatment comprising the steps of adding a material which will preferentially coat the valuable particles of the ore and separating said coated particles as a concentrate by flotation.

970002 Henry Wentworth. U. S. Pat. Office Bull., Sept. 13, 1910.

1 The process of separating the ingredients of comminuted material, which consists in associating with the material a substance chemically reactive upon particles thereof, thereby producing upon the particles affected by the reactive substance superficial coatings of a compound different from the original substance of the particles in respect to film-tension of a liquid, and thereupon separating the differentiated particles by film-tension of said liquid.

PROCEDURE.

DRY SULFIDIZING WITH H_2S.

Experiment A consists of sulfidizing the dry ore with H_2S. The gas was generated in an H_2S generator and allowed to pass over the ore. The ore turned black immediately.

On subjecting the ore to flotation treatment the percentage of the concentrates fell below the percentage of the original ore. This experiment was repeated several times with no satisfactory results.

SULFIDIZING THE WET PULP WITH H_2S GAS.

Experiments B - D and others shown on the data sheets consist of treating the wet pulp with H_2S. The results obtained were similar to those obtained by dry sulfidizing, but the percentage of zinc in the concentrates were about 1 percent better than the original ore.

SULFIDIZING WITH Na_2S_x.

We prepared a 5 percent solution of sodium polysulfid by passing hydrogen sulfid gas into a

5 percent solution of sodium hydroxide. We treated the ore with various amounts of this solution and noted what amount gave the best extraction. We then treated the following ore with the above amount using various oils.

The best results were obtained under the above conditions. We tried several experiments by adding sulfuric acid to the sodium polysulfid solution but the extraction decreased.

TREATING THE ORE BY MEANS OF COLLOIDAL SULPHUR.

We prepared an H_2S solution, placed it in the flotation machine with the ore, and added various amounts of acid to precipitate the sulphur. This was done in order to try to coat the zinc silicate particles with colloidal sulphur and then float them.

Machine Hoover Type.

Experimenter Klepel-Erskine.

ORE: Silicate Sludge. Assay 9.18% Zn.

Missouri School of Mines and Metallurgy

FLOTATION LABORATORY.

OPERATING DATA.

Time 30m.

Amt. H_2O - 3200c c

Amt. ore 800 gms.

Speed of machine 1700 RPM

Test No	OIL No.	OIL Amt.	OIL Kind.	REAGENTS Kind	REAGENTS Amt.	FROTH Kind	FROTH Amt.	REMARKS.	RESULTS Wt.	Per Ct.	Wt.	Per Ct.	Wt.	Per Ct.	Per Ct. Ext.
		drops		sol.	c c				gms.	Zn.					
A.	7	7	Flotation	H_2S		good		Dry sulfidizing with H_2S	26	8.6					3.04
B.	7	8	Flotation	H_2S		good		Sulfidizing of pulp with H_2S	42	10.08					5.7
D.	7	5	Flotation	H_2S		good		Dry sulfidizing with H_2S	26	8.25					2.9
	15	2	Pine												
E.	26	5	# 1 Creosot	Na_2S_x	900	Fair		Sulfidizing with Na_2S_x.	69	7.5					7.05
F.	26	7	#1Creosote	Na_2S_x	900	Fair		Neutralized excess alkali with H_2SO_4	26	6.99					2.47

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Section sheet-Form-131-1000 1-16-B

Machine Hoover Type.

Experimenter Klepel-Erskine.

ORE

Missouri School of Mines and Metallurgy

FLOTATION LABORATORY.

OPERATING DATA.

Time 30 Min.

Amt. H_2O - 3200 cc

Amt. ore 800 gms.

Speed of mach. 1700RPM

Test No	OIL			REAGENTS		FROTH		REMARKS.	RESULTS						
	No.	Amt.	Kind.	Kind	Amt.	Kind	Amt.		Wt.	Per Ct.	Wt.	Per Ct.	Wt.	Per Ct.	Per Ct. Ext.
		drops.		Sol.	cc				gms.	Zn.					
	25	5	Flotation.	H_2S sol.	400				86	10.25					12.00
G.	24	1	Pure Pine	HNO_3	5										
-13-	25	5	Flotation	H_2S	300				46	12.14					7.6
H.	24	2	Fuel												
	25	5	Flotation	H_2S	200				59	9.18					7.3
I.	24	2	Fuel	HNO_3	5										
	25	5	Flotation	H_2S	100				41	9.38					5.2
J.	24	2	Fuel	HNO_3	5										
	1	5	Tar	H_2S	1500				49	11.57					7.7
K.				HNO_3	25										

Machine Hoover Type.

Experimenter Klepel-Erskine.

Missouri School of Mines and Metallurgy

FLOTATION LABORATORY.

OPERATING DATA.

Time 30 Min.

Amt. H_2O 3200 cc

Amt. ore 800 gms.

Speed of mach. 1700 RPM

ORE: Silicate Sludge. Assay 9.18%Zn.

Test No	OIL No.	OIL Amt.	OIL Kind.	REAGENTS Kind	REAGENTS Amt.	FROTH Kind	FROTH Amt.	REMARKS.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Per Ct. Ext.
		drops		Sol.	cc				gms.	Zn.					
	1	5	Tar	H_2S	300			1 gm. rosin added	32	12.19					5.3
L.				HNO_3	100										
-14-	23	5	Crude Wood	Na_2S_x	60			Na_2S_x 5% solution	11	11.12					1.6
M.															
	56	6	Creosote	Na_2S_x	20										
N.	22	7	Pine spec.												
	53	4	Eucalyptus												
	24	5	Pure Pine						69.	10.71					10.1
	23	7	Crude Wood	Na_2S_x	20				44	10.76					6.4
O.															

Machine Hoover Type.

Experimenter Klepel-Erskine.

Missouri School of Mines and Metallurgy

FLOTATION LABORATORY.

OPERATING DATA.

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Speed if mach. 1700RPM

ORE: Silicate Sludge. Assay 9.18% Zn.

Test No	OIL No.	OIL Amt.	OIL Kind.	REAGENTS Kind	REAGENTS Amt.	FROTH Kind	FROTH Amt.	REMARKS.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	Per Ct. Ext.
		drops		sol.	cc				gms.	Zn.					
	25	5	Flotation	Na_2S_x	40				14	11.27					2.1
P.															
	24	5	Pure Pine												
-15-	7	2	Flotation	Na_2S_x	15			Janney Mach. 200gmore	14	10.71					8.2
Q.	15	5	Pine												
	15	1	Pine												
R.	7	2	Flotation	Na_2S_x	15			Janney Mach. 200gmore	7	11.02					4.2
	56	3	Creosote												
S.	15	1	Pine	Na_2S_x	15				9	10.92					5.3
	25	4	Flotation	Na_2S_x	15			Janney Mach. 200 Gms.	5	9.59					2.6
T.	15	1	Pine					Ore							

Machine Hoover Type.

Experimenter Klepel-Erskine.

ORE: Silicate Sludge. Assay 9.18% zn.

Missouri School of Mines and Metallurgy

FLOTATION LABORATORY.

OPERATING DATA.

Time 30 Min.

Amt. H_2O 3200 cc

Amt. Ore 800 gms.

Speed of mach. 1700 RPM

Test No	OIL No.	OIL Amt.	OIL Kind.	REAGENTS Kind	REAGENTS Amt.	FROTH Kind	FROTH Amt.	REMARKS.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Per Ct. Ext.
		drops		sol.	cc				gms.	Zn.					
	21	5	Wood Turp.	Na_2S_x	15				7	10.97					4.2
U.	45	5	Rosin Oil												
-16-	26	5	Creosote	Na_2S_x	60				21.	11.58					3.3
W.	53	1	Eucalyptus												
	37	3	China Wood												
	15	2	Pine												
	37	3	China Wood	Na_2S_x					12.	10.76					3.2
	15	3	Pine												
X.	2cc	Soap Sol.													
	56	5		Na_2S_x					12.	11.78					1.9
Y.	21	3	Crude wood turp.												

Machine Hoover Type.

Experimenter Klepel-Erskine

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FLOTATION LABORATORY.

OPERATING DATA.

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Test No	OIL No.	OIL Amt.	OIL Kind.	REAGENTS Kind	REAGENTS Amt.	FROTH Kind	FROTH Amt.	REMARKS.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Per Ct. Ext.
		drops		Sol.	cc				gms.	zn					
	6	5	Flotation	Na_2S_x	60				10.	10.92					1.5
Z.	55	5	Cresilic acid												
-17-	39	6	Menhaden	Na_2S_x	60				8	8					.9
100	15	6	Pine												
	22	4	Pine spec.												
	53	4	Eucalyptus												
	42	10		Na_2S_x											
101.	60	5	Menhaden						32.	10.76					4.7
	15	5	Pine												
	16	5	Refined Tar												

Machine Hoover Type.

Experimenter Klepel-Erskine.

ORE: Silicate Sludge. Assay 9.18% zn.

Missouri School of Mines and Metallurgy

FLOTATION LABORATORY.

OPERATING DATA.

Time 30 Min.

Amt. H_2O 3200 cc

Amt. Ore 800 gms.

Speed of mach. 1700 RPM

Test No	OIL No.	OIL Amt.	OIL Kind.	REAGENTS Kind	REAGENTS Amt.	FROTH Kind	FROTH Amt.	REMARKS.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Per Ct. Ext.
		drops		Sol.	cc				gms.	zn					
102.		6	Oleic acid.	Na_2S_x	60				8.	9.90					1.1
-18-															
102a	28	2	Crude Tar					102 2 drops No. 28 oil	6.	10.15					.9
102b	16	5	Tar					102 a 2 drops No. 16 oil	24	13.88					8.3
103		12	Oleic acid	Na_2S_x	60				6.	7.96					1.04
	16	6	RefinedTar	Na_2S_x	100				18.	5.71					2.24
104.		12	Oleic acid												

Machine Hoover Type.

Experimenter Klepel-Erskine

Missouri School of Mines and Metallurgy

FLOTATION LABORATORY.

OPERATING DATA.

ORE: Silicate Sludge

Assay 9.18% zn.

Time 30 Min.

Amt. H_2O 3200 cc

Amt. Ore 800 gms.

Speed of mach. 1700 RPM

Test No	OIL No.	OIL Amt.	OIL Kind.	REAGENTS Kind	REAGENTS Amt.	FROTH Kind	FROTH Amt.	REMARKS.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Wt.	RESULTS Per Ct.	RESULTS Per Ct. Ext.
		drops		sol.	cc				gms.	zn.					
108.		35	Fels Naphtha.						8.	9.08					1.58
109.		30	Fels Naphtha.	Na_2S_x	40				23.	7.96					3.9
110.		20	Fels Naphtha.	Na_2S_x	20				11.	10.61					2.5
111.		20		Na_2S_x	20				5.	7.55					.81

CONCLUSION:

Taking into consideration that our work was carried out on a small scale, and since the concentrates obtained showed an increase of 3 to 4 percent zinc, it seems as tho by some special means it would be possible to float silicates.