

# UNITED STATES PATENT OFFICE

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## PURIFICATION OF INDUSTRIAL SANDS

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The present invention relates to an improved process of purifying industrial sands, and particularly glass sands, by froth-flotation.

Such sands generally contain ferruginous impurities in the form of iron-bearing and iron-stained minerals which are detrimental in glass-making. Although such ferruginous impurities exist in the sands in a very small quantity, it is essential that they be reduced to very low limits. That, however, presents difficulties owing to the varying nature of the ferruginous impurities found in industrial sands.

The present invention may be considered an improvement upon the process covered by the patent to Tartaron and Duke, No. 2,283,295, owned by the assignee of the present application, in which process a thick aqueous pulp of glass sands is conditioned with cooperating agents consisting of a substantially insoluble and unsaponifiable oil, a fatty acid compound having a hydrocarbon radical of at least 12 carbon atoms, and an alkali; after which said conditioned pulp is diluted and subjected to froth-flotation to float off the ferruginous impurities from the silica, leaving the residue as the purified glass sands.

The invention herein described is the result of the discovery that the removal of ferruginous impurities from sands may be even more advantageously accomplished, if the treatment of sands, according to the process above mentioned, is followed by froth-flotation treatment of the residue of that process in the presence of cooperating agents consisting of a mineral acid and a cationic-active nitrogenous compound proportioned to collect ferruginous impurities in preference to quartz, usually with a suitable frother; thereby producing a froth containing ferruginous impurities which is discarded, leaving the residue as the purified glass sands. The second froth-flotation treatment of the residue of the first froth-flotation treatment, after conditioning said residue with the cooperating agents mentioned, results in removing a large proportion of the ferruginous impurities which were left in the residue of the first froth-flotation treatment, and without unduly decreasing the weight of the final product which is the residue of the second froth-flotation treatment. While it is unnecessary to theorize about the reasons for the results attained by the successive treatments in the manner described, it is believed that the success of the second treatment after conditioning with the agents mentioned is due partly to the effect of the agents used in the first treatment on the residue of that treatment. At any rate, the two

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conditionings with the agents mentioned, and the successive froth-flotation treatments after those conditionings, cooperate to produce surprisingly good results.

5 In the first treatment, as described in the afore-said patent, the insoluble and unsaponifiable oil may be (for example) fuel oil, lubricating oil, Diesel oil, etc.; the fatty acid compound may be (for example) a crude fatty acid compound derived from fish oil, or the product commercially known as "Varlacoid" which is a mixture of fatty and resin acids; and the alkali may be (for ex-  
10 ample) sodium hydroxide, calcium hydroxide, sodium carbonate, sodium silicate, etc.

15 In the second treatment, the mineral acid may be (for example) sulfuric acid. The cationic-active nitrogenous compound may be (for example) the higher aliphatic amines and their salts with water-soluble acids, the condensation products of amino-alcohols with high-molecular-weight fatty acids and their salts with water-soluble acids, the higher alkyl-O-substituted isoureas and their salts with water-soluble acids, the higher aliphatic quaternary ammonium bases and their salts with water-soluble acids, the higher alkyl pyridinium salts of water-soluble acids, the higher alkyl quinolinium salts of water-soluble acids, etc. Among the cationic-active nitrogenous compounds which we have found satisfactory are aliphatic amines containing from  
20 7 to 19 carbon atoms in the alkyl group; for example, the commercial product sold by E. I. du Pont de Nemours & Co. as "D. P. 243," which is understood to be a 50% aqueous paste of technical laurylamine hydrochloride understood to contain approximately 18% octylamine hydrochloride, 10% decylamine hydrochloride, 55% laurylamine hydrochloride, 17% myristyl amine hydrochloride, and traces of secondary and tertiary amines. Another example of said cationic-active nitrogenous compounds is the commercial product sold by Armour and Company as "Am. Ac. 1180-C" which is understood to consist of approximately 73% mono-octadecylamine acetate, about 24% mono-heptadecylamine acetate, together with small quantities of secondary and tertiary amine acetates. A suitable frother, such as pine oil, is preferably used in one or both  
25 treatments, along with the other agents; and conditioning for the first treatment is best conducted in a thick pulp. We have also discovered that in some cases considerably improved results may be obtained by employing in the second treatment of the process, along with the mineral acid and the cationic-active nitrogenous compound, a

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salt of a trivalent metal such as aluminum chloride.

The invention will be understood from the following examples of the practice thereof on glass sands containing substantial amounts of ferruginous impurities.

## EXAMPLE I

The crude industrial sands treated were of a fineness to pass through a 20 mesh screen. These sands were agitated in water at about 50% solids for about three minutes for the purpose of disintegrating the clay, and the mixture was then deslimed. The substantially deslimed feed thus obtained was conditioned for about two minutes in an aqueous pulp at about 70% solids with the reagents listed below under column (1). The thus conditioned pulp was then diluted and subjected to froth-flotation treatment in a flotation cell, and the first froth product was removed and discarded. Then the reagents listed below in column (2) were added to the residue of the first flotation treatment and were distributed in the pulp by agitation for about one minute; after which the pulp was again subjected to froth-flotation treatment and the second froth product was removed and discarded. The residue was the final product of purified sands. The reagents used, in pounds per ton of feed, were as follows:

(1) First treatment	(2) Second treatment
Caustic soda..... 0.4	Sulfuric acid..... 5.5
Fuel oil..... 1.7	"D. P. 243"..... 0.3
"Varlacoid"..... 0.3	Pine oil..... 0.2
Pine oil..... 0.2	

The metallurgical results of the two treatments were as follows:

Product	Percent weight	Percent Fe <sub>2</sub> O <sub>3</sub>	Percent Fe <sub>2</sub> O <sub>3</sub> recovered
Feed.....	100.0	0.087	100.0
First froth product.....	22.7	.17	43.7
Second froth product.....	9.6	.20	21.8
Final product.....	67.7	.044	34.5

It will be noted that the ferruginous impurities in the sands have been reduced from .087% to .044%, that is to about 35% of the original amount; and that the weight of the final product was 67.7% of the weight of the feed.

## EXAMPLE II

In this example, the procedures were exactly the same as those in Example I; and the reagents used were the same in kind and quantity, except that "Am. Ac. 1180-C" in the amount of 0.07 lb. per ton of feed was used instead of "D. P. 243." In this case the metallurgical results of the two treatments were as follows:

Product	Percent weight	Percent Fe <sub>2</sub> O <sub>3</sub>	Percent Fe <sub>2</sub> O <sub>3</sub> recovered
Feed.....	100.0	0.088	100.0
First froth product.....	17.6	.21	42.1
Second froth product.....	10.9	.18	22.7
Final product.....	71.5	.043	35.2

It will be seen that the ferruginous impurities in the sands were again reduced to about 35% of the original amount; and that the weight of the final product was somewhat higher, namely, 71.5% of the weight of the feed.

## EXAMPLE III

In this example, the industrial sands were obtained from P. J. Weisel, Inc., of Corona, Cali-

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fornia, and were treated as received, without screening or desliming. The percentage of ferruginous impurities in these sands is much higher than in the sands of Examples I and II; and, in addition to the reagents used in Example I, aluminum chloride (AlCl<sub>3</sub>) was used in the second treatment of this Example III.

The feed was first conditioned for about two minutes in an aqueous pulp at about 71% solids with the reagents listed below in column (1); after which the conditioned pulp was subjected to froth-flotation treatment, and the froth was removed and discarded. The residue of this treatment was conditioned for about a quarter of a minute with the reagents listed below in column (2); and was then subjected to the second froth-flotation treatment, and the resulting froth was removed and discarded. The residue was the final product of purified sands. The reagents used, in pounds per ton of feed, were as follows:

(1) First treatment	(2) Second treatment
Caustic soda..... 0.5	Sulfuric acid (H <sub>2</sub> SO <sub>4</sub> )..... 2.5
Fuel oil..... 2.3	"D. P. 243"..... 0.6
"Varlacoid"..... 0.8	Aluminum chloride (AlCl <sub>3</sub> )..... 0.8
	Pine oil..... 0.2

The metallurgical results of the two treatments were as follows:

Product	Percent weight	Percent Fe <sub>2</sub> O <sub>3</sub>	Percent Fe <sub>2</sub> O <sub>3</sub> recovered
Feed.....	100.0	0.232	100.0
First froth product.....	6.0	1.95	64.7
Second froth product.....	5.3	0.93	21.1
Final product.....	88.7	0.037	14.2

The excellent nature of the results in this Example III will be evident from the facts that the ferruginous impurities in the sands have been reduced from 0.232% to 0.037%, that is, to about 16% of the original amount; and that the weight of the final product is 88.7% of the weight of the feed.

What is claimed is:

1. The process of removing ferruginous impurities from industrial sands which consists in subjecting such sands to a first froth-flotation treatment in the presence of a fatty acid compound having a hydrocarbon radical of at least 12 carbon atoms, an alkali and a mineral oil, and removing and discarding the froth; and subjecting the pulp residue of the aforesaid first froth-flotation treatment to a second froth-flotation treatment in the presence of cooperating agents consisting of less than about six pounds of a mineral acid and less than about one pound of a cationic-active nitrogenous compound containing an alkyl group which contains from 7 to 19 carbon atoms, both per ton of solids in the feed, and removing and discarding the froth, leaving the residue as the purified sands.

2. The process of claim 1 in which the cationic-active nitrogenous compound is an aliphatic amine whose alkyl group contains from 7 to 19 carbon atoms.

3. The process of claim 1 in which a frother is used in addition to the other reagents.

4. The process of removing ferruginous impurities from industrial sands which consists in subjecting such sands to a first froth-flotation treatment in the presence of a fatty acid compound having a hydrocarbon radical of at least 12 carbon atoms, an alkali and a mineral oil, and removing and discarding the froth; and subjecting the pulp residue of the aforesaid froth-

flotation treatment to a second froth-flotation treatment in the presence of cooperating agents consisting of less than about six pounds of a mineral acid and less than about one pound of a cationic-active nitrogenous compound containing an alkyl group which contains from 7 to 19 carbon atoms, both per ton of solids in the feed, and aluminum chloride, and removing and discarding the froth, leaving the residue as the purified sands.

5. The process of claim 4, in which the cationic-active compound is an aliphatic amine whose alkyl group contains from 7 to 19 carbon atoms.

6. The process of removing ferruginous impurities from industrial sands which consists in conditioning a thick aqueous pulp of such sands with a fatty acid compound having a hydrocarbon radical of at least 12 carbon atoms, an alkali, and a mineral oil; diluting such conditioned pulp, subjecting it to a first froth-flotation treatment, and removing and discarding the froth; and subjecting the pulp residue of the aforesaid first froth-flotation treatment to a second froth-flotation treatment in the presence of cooperating agents consisting of less than about six pounds of a mineral acid and less than about

one pound of a cationic-active nitrogenous compound containing an alkyl group which contains from 7 to 19 carbon atoms, both per ton of solids in the feed, and removing and discarding the froth, leaving the residue as the purified sands.

7. The process of removing ferruginous impurities from industrial sands which consists in conditioning a thick aqueous pulp of such sands with a fatty acid compound having a hydrocarbon radical of at least 12 carbon atoms, an alkali, and a mineral oil; diluting such conditioned pulp, subjecting it to a first froth-flotation treatment, and removing and discarding the froth; and treating the pulp residue of the aforesaid froth-flotation treatment with cooperating agents consisting of less than about six pounds of a mineral acid and less than about one pound of a cationic-active nitrogenous compound containing an alkyl group which contains from 7 to 19 carbon atoms, both per ton of solids in the feed, and aluminum chloride; and subjecting that treated pulp residue to a froth-flotation treatment, and removing and discarding the froth, leaving the residue as the purified sands.

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