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A STUDY OF GLASS SANDS FROM PIPRI AND FENI AREAS

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A laboratory investigation of beneficiation of two known deposits of glass sand in the Pipri and Feni areas of West & East Pakistan respectively have shown the deposits to be of rather uniform character. The sands were sieved and subjected to water washing to remove the clay stains followed by magnetic separation to eleminate biotite and other magnetic minerals and finally submitted to the Froth flotation and the other chemical treatments in order to remove all the iron bearing minerals. These treatments after Froth flotation gave a product containing 0.065% and 0.037% Fe_2O_3 for sands from Pipri and Feni areas respectively, whereas in the raw sands it was 0.37% and 0.30% respectively.

The glass Industry in Pakistan at present is in developing stage and the prime mover behind its growth is the supply of economical and quality glass sand. There is a lack of quality glass sand supplier and the glass factories therefore, have to do all the beneficiation job right from washing and sieving to magnetic separation and Froth flotation etc. This involves the high transportation cost even for the portion of sand which is discarded after washing and grading. Moreover, beneficiation at a small scale is not efficient and economical.

Glass sand is the major constituent of almost all the commercial glasses containing from 65–75% silica. It is a very important raw material for the glass Industry. High quality and low priced glass sand is therefore, expected to have boosted the development of Pakistan glass industry. In West Pakistan at present, there are about 25 glass factories and their annual demand for glass sand is around 50,000 tons. It is expected that with the establishment of the steel and fiber glass projects, the total requirements of the sand will grow enormously and thus will justify the construction of a dressing and beneficiation plant for sand. The purpose of this investigation was to remove all the objectionable impurities from the sand by means of sieving, washing, magnetic separation, froth flotation and by other chemical treatments and thus to make the sand suitable for making colourless glass.

Deposits of pure sands have been used since long but the supply can not last for long and the means ought to be sought to beneficiate the more bountiful supply of impure sands. The present investigation deals with two samples of sand one from Feni in Chittagong District and the other near Pipri, Landhi, Karachi. Complete survey of these two deposits have not been made but according to the Senior author who personally visited these places, the quantities are in abundance and will last for a long time to come.

In general, specifications[1] for all grades of glass sand are quite similar except iron oxide content, which is quite rigid but varying for different applications. Sand used for making clear container should contain less than 0.06% Fe_2O_3, for flat glass less than 0.10% Fe_2O_3 and for amber container glass less than 0.25%. Silica content should be high, and a minimum of 98% is usually specified, although this specification is not always followed. Other oxides such as Al_2O_3, CaO, MgO, Na_2O and K_2O do not have rigid limits since they are normal constituents of the glass batch, however, the contents of these oxides should be constant (most consumers prefer that the Al_2O_3 content should not exceed 3 to 4%). A physical size range of -20 to $+100$ mesh is usually specified.

Experimental

Physical Treatments

Grading.—The purpose of grading was to remove unwanted coarser and finer sand fractions from the sand. 30 g each of the raw and the washed samples were taken and sieving was done by B.S. test sieves No. 25 (599μ), 60(251μm) 72(211μm) 100(152μm) and 120(125μm). The graphs showing the cumulative percentage versus mesh number are shown in Fig. 1. In order to obtain a large quantity of $-25+120$ mesh fraction, the Raw dry sands A and B were graded. The fraction ($-25+120$) was then used for various physical and chemical treatments. The graded results are shown in Table 1.

Water Washing.—In order to remove the ferroginous clayey fraction, the washing treatment was performed. 500g of raw useful fraction with excess amount of water, was subjected to washing by

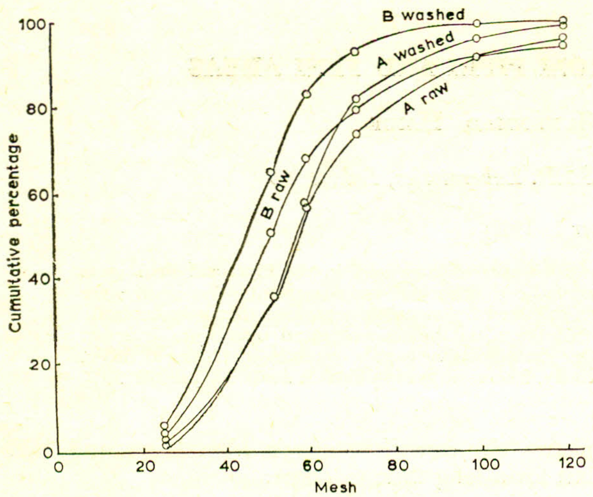

Fig. 1.—Grading of raw and washed sand of samples A and B.

TABLE 1.—GRADING OF SAND A & B.

Mesh No.	Raw samples A	Raw samples B	Washed samples A	Washed samples B
+ 25	2.3%	4.1%	1.5%	4.8%
−25+120	93.0%	90.0%	98.0%	99.0%
−120	4.3%	5.4%	0.2%	0.3%

gentle agitation until the washings were found clear. About 9% of sand A and 6.5% of sand B were lost in this process. The washed sands were then analysed chemically. The results are given in Table 2.

Magnetic Separation.—Washed samples after drying were subjected to magnetic treatment and ferromagnetic particles removed. Approximately 3% of sand A and 2.5% of sand B containing 0.8% and 0.6% Fe_2O_3 respectively were lost in this process.

Froth Flotation.—A series of flotation tests to remove iron bearing minerals such as the heavy mineral particles, the ferromagnetic left-overs, and the diamagnetic particles, were conducted.[2,3] The test was carried out in a cylindrical stainless steel vessel of 8.0 cm diameter. The reagents-mixture (Oleic acid 4 parts and terpeneol 1 part) was first dispersed in water at 45°C. The sand was then poured in and mixed properly. The ratio of mixture to sand was 1 lb to 1 ton and that between sand and water, 1:2. The depth of slury was kept 4.0 cm. The slurry, with sodium silicate (0.5 lb/ton sand), was constantly stirred mechanically for 10 minutes with a wooden rod rotating at an optimum speed (400 rpm), while the air was continuously bubbled from the bottom at a pressure of 5 lb/sq inch The pH was maintained between 7.0–7.5 using dilute solution of H_2SO_4 and NaOH as desired. The function of sodium silicate is to depress silica grains. The heavy particles of iron bearing minerals were entraped in air bubbles and removed alongwith froth formed. The sand was then washed with

TABLE 2.—CHEMICAL ANALYSES OF VARIOUS GRADES OF SANDS.

	Raw sand % A	Raw sand % B	Graded and washed % sand A(−25+120)	Froth floated sand % B	Titenous sulphate processed sand % A
I/L	0.96	0.70	0.42	0.26	0.3
SiO_2	95.40	93.45	92.2	97.10	97.8
Al_2O_3	2.53	4.35	1.67	2.47	1.57
Fe_2O_3	0.37	0.30	0.125	0.037	0.03
CaO	0.58	0 93	0.40	0.19	0.30
MgO	0.18	0 35	0.15	0.10	0.10
Na_2O	Traces	—	Nil	Nil	Nil
K_2O	Traces	—	Nil	Nil	Nil

TABLE 3.—IRON DETERMINATION OF VARIOUS GRADES OF SAMPLE.

	Raw sand %	Graded and unwashed (−120) %	Graded and unwashed (−25+120) %	Graded and unwashed (+25)%	Graded washed (−25+120) %	Magnet treated %	Froth floted %	Adam's process %	Sulphite process %	Titanous sulphate process
A Type	0.37	1.4	0.25	2.5	0.125	0.1	0.065	0.055	0.04	0.03
B Type	0.30	1.27	0.23	2.16	0.124	0.1	0.037	0.05	0.05	0.04

water and chemically analysed. The results are shown in Table 3.

Chemical Treatments

Adam's Process.—A portion of sand after magnet treatment was also subjected to Adam's Process.[4] The sand was treated with a mixture containing 2 parts of sodium acid oxalate and 1 part of crystalline ferrous sulphate in 100 parts of water. Thus the strength of the solution was kept at 2:1:100 respectively. The treatment was carried out at a temperature of 45°C. The process was repeated several times and each time the supernatant liquid was decanted. Finally the sand was washed thoroughly with hot water till no indication for iron in the washing was observed. The sand was then chemically analysed and results given in Table 3.

Sulphite Process.—The sand was mixed with 0.4% of sodium sulphite and treated with water containing HF in a polythene beaker.[5] Sufficient HF was added to bring the pH to 2.7. The whole mass was then thoroughly stirred with a wooden rod rotating at high speed; the amount being sufficient to cover the sand completely. The supernatant liquid was then decanted off. The process was repeated. Finally the sand was washed thoroughly with water and analysed for Fe_2O_3. The results are presented in Table 3.

Titanous Sulphate Process.—This chemical treatment was carried out exactly in the same way as the sulphite process, titanous sulphate being employed instead of sodium sulphite. The obtained results of Fe_2O_3 after this process are given in Table 3.

Glass Melting Tests

Soda-lime-silica glass batches containing raw and beneficiated products were formulated and melted. 3 products, the raw sand, the magnet treated sand and the froth floated sand were evaluated. 100 g batches were formulated to approximate the following chemical composition. 73% SiO_2 and 10% CaO, 0.5 g each of $NaNO_3$ and NaCl were added to each bath to aid in fining. Batches were melted in a gas fired furnace between 1450°C to 1500°C and held at this temperature for 2 hr in a slightly oxidizing atmosphere before pouring. Molten glass was annealed, and evaluated.

Results and Discussion

Consumers want the highest possible pure glass sand at the lowest possible price. Specifications require[1] the glass sand to be 98% silica sized between 20 and 100 mesh as coarse particles are difficult to melt and finer particles tend to produce seeds in the glass and create dust control problems. In practice the consumers usually require grains of the sand to be approximately of uniform size and all of them should pass through a 25 mesh sieve and be not smaller than 120 mesh sieve. Similarly angular grains are preferred to the rounded ones as the former provide weak points for the heat to attack. Particles size distribution of both the sands A and B is reasonably narrow. For the most part, 93.5% of the sand grains of sample A are between 25 and 120 mesh. The same after washing gives grains 98% between 25 and 120mesh. In case of sample B the results are 90% and 99% (Table 1) respectively. As far as particle size distribution is concerned both the sands are almost identical. Distribution curves plotted from the sieve analysis (Fig. 1) formed bell shaped curves centred around the 50–100 mesh fraction. Average grain size of the raw and the washed useful fraction (−25+120) of A and B has been calculated to be approximately 70.4%, 76.8% and 74.0%, 88.9% respectively. This indicates that sand B is slightly better than A as far as average grain size is concerned. It may also be observed from Table 1 that the bulk of the iron is present either in the coarse +25 or in the fine −120 mesh fraction. From the iron point of view as well, the fraction −(25+120) is desirable.

It may be seen (Table 1) that washed fraction (−25+120) of sands A and B contains iron 0.125 and 0.124% respectively. Thus there is a reduction of 50% and 58% of Fe_2O_3 in samples A and B respectively. The washed and graded samples when passed through permanent magnets gave a further reduction in iron, this time about 20% reduction has been obtained. The magnet treated samples of sand A and B are then put to flotation tests in order to remove the iron bearing minerals from the quartz grains. Flotation tests revealed that in case of sand A, the Fe_2O_3 content has been reduced to 0.065% a reduction of about 35%. In case of sample B a remarkable reduction has been observed; the Fe_2O_3 has been reduced from 0.1% to 0.037%, a reduction of about 2/3 (63%). Thus it seems that the froth flotation process is very suitable for sand B. The explanation lies in the fact that it contains some lime feldspar as shown in Table 2. (Al_2O_3 being 4.35) with which the iron is associated as well as a good part of iron seems to be present in the traces of iron bearing minerals such as biotite, garnet and hornblende etc. The removal of most of the above feldspar and other iron bearing impurities, in fact, resulted in the drastic cut in Fe_2O_3 content.

In order to find out the suitability of the froth flotation process and the other chemical processes the magnet treated sands were the starting point for each of the experiments. Treatment with acid to remove surface iron staining from glass sands is more common in Europe than in the U.S.A. In the present study only 3 chemical methods namely Adams process, sulphite process and the titanium sulphate process have been tried. It may be seen (Table 3) that the magnet treated sample A gives 0.055%, 0.05% and 0.04% Fe_2O_3 respectively with a reduction of 45%, 60% and 70% respectively. In case of sample B the iron content is reduced to 0.05, 0.05 and 0.04% respectively giving a reduction of 50%, 50% and 60% respectively. It may be observed that in case of sand A from Pepri the chemical treatments are beneficial as far as the reduction in iron content is concerned. But in the case of sand B from Feni the chemical treatments seem to be ineffective, on the other hand the froth floatation treatment is more effective in reducing the iron content. The main reason for such a behaviour may be due to the fact that in sample A there is an appreciable amount of iron stains which has been removed by acid leaching and thus resulting in the reduction of total iron where as in the case of sample B the amount of iron stain removed by acid leaching was not worth while because of the small amount occuring as stains. These facts have also been confirmed by the microscopic examinations. In general, results indicated that HF was the most effective leaching agent and that the addition of a reductant and an iron collector may greatly increase the removal of Fe_2O_3.

Melting characteristics and glass appearance in all the 6 samples were good. No stones or flaws were noted; the glass had a high lustre, colour ranged from light greenish blue through very faint greenish blue to clear white as iron oxide content decreased. Only the glass, containing raw sands appeared to have too much colour (approximately 0.37 and 0.3% Fe_2O_3).

Conclusions

Water washing, grading, high intensity magnetic separation followed by froth flotation and acid leaching appeared to be the best method for lowering the iron content of the sands. Of the original sand A containing 0.37% Fe_2O_3 the iron was removed by magnetic separation and a product obtained with 0.1% Fe_2O_3. Acid leaching further reduced the iron content from 0.1% to 0.03% Fe_2O_3. In case of sample B froth floation seems to be more effective than acid leaching which has reduced the iron from 0.1% (magnet treated) to .037%, a reduction of 63%. A combination of magnetic separation, Flotation and acid leaching produced concentrates containing 0.03% to .037% Fe_2O_3. It is finally concluded that both the sands aftert reatment are suitable for making colourless and high quality glass.