

BENEFICIATION OF THANA BULLAH KHAN AND JANGSHAHI SAND FOR GLASS-MAKING

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In the Thana Bullah Khan and Jangshahi deposits, the main impurity is that of iron which is present in the form of heavier black particles and the yellow to reddish coating over the grains. These have been significantly reduced by washing the samples over shaking tables and afterwards by treating with sodium acid oxalate and ferrous sulphate solution (Adam's process). The processed sand is suitable for the manufacture of colourless glass.

Introduction

The glass-making factories situated in Hyderabad and Karachi regions use silica sand deposits of Daud Khel and Hazara district which are about 1000 miles from the factories, whereas huge deposits of sand are available at Thana Bulla Khan and Jangshahi which are about 50 miles both from Karachi and Hyderabad. The sand from these nearby deposits is not suitable for making colourless glass because it contains high percentage of iron which cannot be removed by simple water-washing as in the case of the sand from Daud Khel and Hazara district. Any successful attempt towards the purification of Thana Bulla Khan and Jangshahi sand would greatly benefit the glass industry of the southern part of West Pakistan provided the cost of the processed sand remains lower than the cost of the Daud Khel and Hazara sand at Karachi and Hyderabad. Since major part of the glass industry is situated in the southern region, this work would be of great overall significance to the glass industry in the country.

With these considerations, the work on the improvement of Thana Bulla Khan and Jangshahi sand was undertaken in these Laboratories. Two representative samples of this sand from different locations in Thana Bulla Khan area were collected through two glass manufacturers in Karachi.

Experimental

Sampling was done by the usual method of coning and quartering.¹

When a few grains of the sample of sand were seen under a microscope through ordinary and polarized light, sample 1 showed a number of colourless grains without any coating, and a few with reddish and yellowish coating. Other im-

purities appeared to be very insignificant except for the presence of distinct tiny black particles. Sample 2 had more uniform yellow coating but lesser black particles. The black particles in both the samples were not attracted by a strong magnet when rotated in a portion of the sample. They were also not carbonaceous as they remained unaffected when ignited strongly for an hr. On washing with water, the black particles collected at the bottom of the container beneath the sand particles. They were separated completely by bromoform in which they settled at the bottom of the container. The specific gravity of these particles was 2.81 as against 2.74 for the whole sample. The percentage of black particles separated by bromoform in Samples 1 and 2 was 0.501% and 0.381%, respectively.

Determination of Iron Content.—Colorimetric² method was used for the determination of iron content of the original and treated samples. E.E.L. colorimeter was employed which was calibrated with standard solution of Fe_2O_3 . Two calibration curves to give percentage ranges of 0.001–0.009 and 0.01–0.09% of Fe_2O_3 were drawn against percentage transmission as shown in Fig. 1.

The percentage transmission of the required solution was noted from colorimeter using red filter and Fe_2O_3 was seen directly against that particular transmission from these curves.

Chemical Analysis.—Chemical analysis of original and treated fractions was done according to B.S.S.No. 2975 (1958).³ Only SiO_2 , R_2O_3 , Fe_2O_3 , CaO and MgO were determined.

Grain Size Determination.—100 g each of the original samples were sieved through B.S. test sieves No. 18, 25, 36, 44, 60, 72, 85, 100 and 120, in a mechanical sieve shaking machine for 15 min. The residue left over each sieve was weighed. The results are represented graphically as per-

centage cumulative against sieve apertures in microns. (Fig. 1).

Purification of Sand by Washing with Water.—500 g of the ungraded sand was washed with 3 l of tap water in a china dish till the supernatant layer was clear of any suspension. The loss in weight on washing was 1.5%. The amount of Fe_2O_3 removed by water-washing and grading is given in Table 1.

Purification of Sand by Adam's Process.—Adam⁴ removed the iron coating of the sand grains by treating the samples with a solution of sodium acid oxalate containing some $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$. This has been commercially exploited⁵ for a long time in England. The solution was prepared by taking 0.5 g sodium oxalate, 3 ml 1:1 H_2SO_4 and 0.1 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ in 100 ml water.

Twenty g of the sample in each case was put into 20 ml of Adam's solution which was warmed to $65^\circ \pm 5^\circ\text{C}$. The reaction was extremely slow at room temperature or lower temperature. Treatment was done at different intervals from 2 to 60 min. The concentration of Adam's solution was then changed by taking 1.0 g sodium oxalate, 6 ml H_2SO_4 and 0.2 g $\text{FeSO}_4 \cdot 7\text{HO}_2$ in 100 ml water, and the treatment was repeated at the same intervals. The results are shown in Table 2. In all these treatments the graded fraction between $-36 + 120$ was taken.

Separation of Black Particles.—For the separation of black particles, the method employed was based on difference of specific gravity of black particles and the bulk sample, as suggested and discussed by Segrove.⁶ A laboratory model of Wilfley's concentration tables manufactured by Mines and Smelter Supply Co., England, was used for this purpose. 500 g each of the original sample was fed in the channel in the form of slurry. By the jerking motion of the deck the black and other heavier particles collected at one corner while the clearer lighter fraction slipped down to the other side of the table. The clear fraction was found to be 92% for sample 1 and 95.3% for sample 2.

Again, 20 g of the fraction free from black particles was subjected to Adam's process and the subsequent iron determinations were conducted. The results are given in Table 3 for sample 1.

Finally overall chemical analysis of original, treated samples and that of the black particles is given in Table 4.

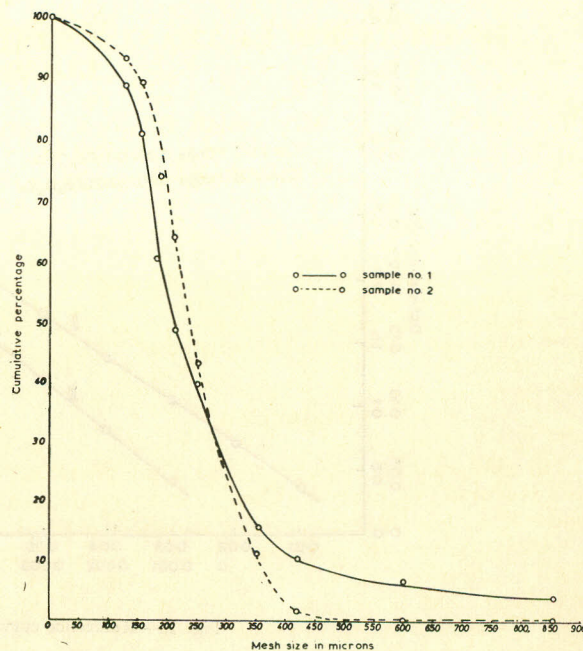


Fig. 1.—Sieve analysis of sample No. 1 and No. 2 showing percentage cumulative against sieve apertures in microns.

TABLE 1.—IRON DETERMINATION OF SAMPLE NO. 1.

Sample	Fe_2O_3 % corrected
Original ungraded	0.3608
Water-washed, ungraded	0.3285
Original graded $-36 + 120$	0.2427
Water-washed graded $-36 + 120$	0.2104

TABLE 2.—EFFECT OF TIME ON REMOVAL OF IRON OF SAMPLE NO. 1.

Time (min)	Dilute Adam's soln	Concd Adam's soln
0	0.2427	0.2427
2	0.1974	0.1269
5	0.1803	0.1041
10	0.1716	0.1036
20	0.1596	0.0858
30	0.1431	0.0793
45	0.1227	0.0784
60	0.0882	0.0760

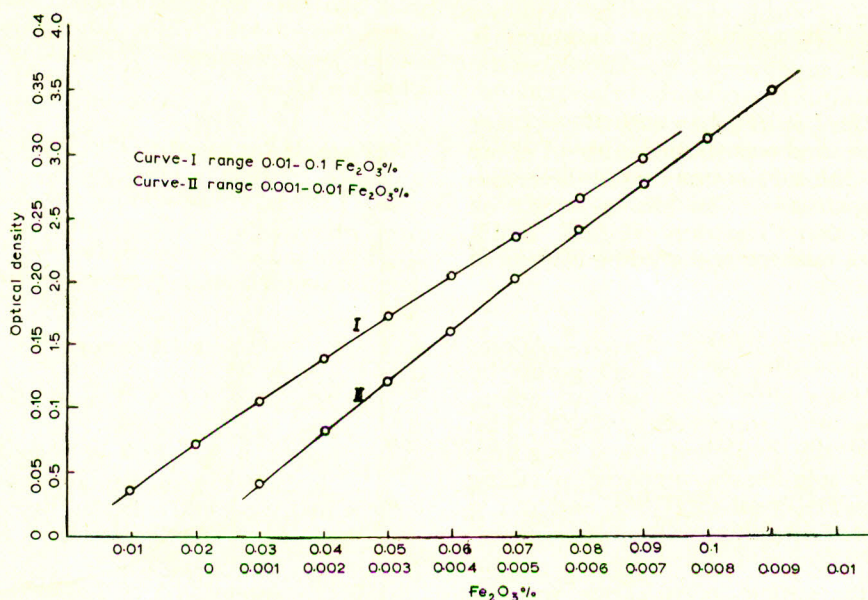
Fig. 2.—Reference curves for the determination of Fe₂O₃.

TABLE 3.—IRON DETERMINATION AFTER TREATMENT WITH ADAM'S WILFLEY'S TABLES SOLUTION (SAMPLE 1).

(colorimeter error +0.0041)

Sample	Time	Fe ₂ O ₃ %
Wilfley's tables treated (ungraded)	0	0.0965
Wilfley's tables treated (graded -36+120)	0	0.0952
" "	5	0.0460
" "	10	0.0361
" "	20	0.0359
" "	30	0.0359
" "	45	0.0358
" "	60	0.0348

Discussion

Preliminary experiments on water-washing and grading of the sand have shown that the sand contains very little fraction of clayey material and fines. The objectionable and predominant impurity is that of iron which is present in two forms. One is the coating on the larger grains and the other is in the presence of heavy black particles. The grading alone has reduced the iron content to the extent of 31%, but even after grading and washing, the content of iron is too high to use this sand for good quality glass-ware. Therefore, other methods had to be tried to further reduce the content of iron.

Among the many processes attempted, the Adam's process has proved to be more successful in bringing down the iron percentage of the sand. As it is evident from Table 2, time of contact and concentration of the original Adam's solution affect the rate of removal of coating.

TABLE 4.—CHEMICAL ANALYSIS.

Sample	SiO ₂ %	Fe ₂ O ₃ %	Al ₂ O ₃ %	CaO%	MgO%	I/L%	Total
1 Black particles	.. 31.88	47.78	15.84	1.17	0.70	2.58	100.04
Original sample	.. 96.68	0.36	2.17	0.14	0.12	0.55	100.02
Wilfley's tables treated	.. 98.19	0.09	1.27	0.13	0.11	0.26	100.05
Treated with Wilfley's tables and Adam's process	.. 98.51	0.03	1.13	0.11	0.9	0.14	100.01
2 Original sample	.. 97.62	0.134	1.99	0.12	traces	0.27	100.13
Wilfley's tables treated	.. 98.17	0.074	1.48	0.11	traces	0.26	100.09
Treated with Wilfley's tables and Adam's process	.. 98.45	0.029	1.19	0.09	traces	0.26	100.02

The separation of black particles by Wilfley's tables followed by removal of iron coating with Adam's solution brings down the iron content of the sand to acceptable limits for colourless glass making. Approximately 90% of iron has been removed in 15 min at the initial concentration of the Adam's solution. Hence the iron content of sand has been reduced from 0.36-0.030 and 0.134%-0.02% for sample 1 and sample 2, respectively by separation with Wilfley's tables and afterwards treatment with Adam's solution.

From the results of analysis in Table 4, the nature of black particles appears to be a complex of iron, silica and alumina. The percentage of Fe_2O_3 in black particles is 47%. Therefore, the Fe_2O_3 present due to black particles in original sample is 0.235%. It means that out of total iron, about 66% is due to the impurity of black particles. This signifies the importance of separating these particles prior to treatment with Adam's solution.

Conclusion

In the Thanah Bullah Khan and Jangshahi sand, the coating on the grains and heavier black particles are two main impurities. Separation of

black particles by concentration tables and subsequent treatment with Adam's solution have made the sand suitable for making high quality colourless glass.

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