BENEFICIATION OF LOCALLY AVAILABLE SAND FOR THE MANUFACTURE OF COLOURLESS GLASS

Md. Abdul Matin

BANGLADESH UNIVERSITY OF ENGINEERING AND TECHNOLOGY
DHAKA, BANGLADESH

AUGUST, 1998

DECLARATION

This is to certify that this research work has been carried out by the author under the supervision of Dr. A. K. M. Bazlur Rashid, Associate Professor, Materials and Metallurgical Engineering Department, BUET, Dhaka, and it has not been submitted elsewhere for the award or degree of any kind.

Coutersigned by

Signature of the Supervisor

Md. Abdul Matin

Signature of the Author

The undersigned examiners appointed by the Committee of Advanced Studies and Research (CASR) hereby recommend to Materials and Metallurgical Engineering (MME) Department of Bangladesh University of Engineering and Technology, Dhaka, the acceptance of the thesis entitled "Beneficiation of Locally Available Sand for the Manufacture of Colourless Glass" submitted by Md. Abdul Matin, in partial fulfilment of the degree of the requirement for the Degree of Master of Science in Engineering (Metallurgical).

1. Rashid 12/8/98
Dr. A. K. M. Bazlur Rashid — Chairman (Supervisor)
Associate Professor
MME Department, BUET, Dhaka.

2. Abdus Salam
Mr. Abdus Salam — Co-Chairman (Co-supervisor)
Industrial Consultants (Glass)
B/4, Zakir Hossain Road, Block-E
Mohammadpur, Dhaka.

3. Rezaul Haque ১২/৮/৯৮
Head — Member (Ex-officio)
MME Department, BUET, Dhaka.

4.
Dr. A. S. W. Kurny — Member
Professor
MME Department, BUET, Dhaka.

5.
Mr. Sanowar Hossain Mondal — Member (External)
Principal Scientific Officer
Glass and Ceramic Division
BCSIR, Dhaka.

ABSTRACT

The investigation of Noapara glass sand deposit of Sylhet district is performed to beneficiate the sand for the manufacture of colourless glass. First of all the physical and mineralogical identification of heavy minerals present in the sand were carried out. The study includes sieve analysis, heavy mineral separation and magnetic separation. Semi-quantitative analyses of the glass sand were performed with the help of XRD and Petrography and the results are reported in sequence. The classified sand is then subjected to water wash and attrition to remove clay and ferruginous coating followed by magnetic separation to eliminate magnetic minerals. The water-washed and magnetically treated sands are then subjected to various chemical treatments in order to reduce the iron level. Finally, a number reagent batch combinations are prepared using water-washed and magnetic treated sand for the forth flotation technique. One of such reagent combinations reduce Fe_2O_3 and Cr_2O_3 levels in the sand to 0.032 and 0.0012 % respectively after froth flotation. This is close to the required chemical composition for the manufacture of colourless glass.

ACKNOWLEDGEMENTS

The author wishes to express his heartiest felicitation and profound indebtness to his thesis supervisor Dr. A. K. M. Bazlur Rashid, Associate Professor, MME Department, BUET, Dhaka for his constant guidance, valuable suggestions, encouragement and benevolent help in carrying out the research work as well as in writing this thesis. The author is grateful and much obliged to Mr. Abdus Salam, the co-supervisor of the project, for his valuable suggestions during the project work as well as in writing this thesis.

The author is also grateful to Professor Md. Mohar Ali (previous head) and Professor A. A. Md. Rezaul Haque, Head, Materials and Metallurgical Engineering Department, BUET, Dhaka for providing him with the laboratory facilities.

The author also express his sincere thanks to Professor Md. Badrul Imam, Chairman, Department of Geology, Dhaka University, and Professor Md. Mujibur Rahman, project co-ordinator, Post-graduate Studies and Research, Environmental Engineering Department, BUET, Dhaka and Mr. Sanowar Hossain Mondal, Principal Scientific Officer, Glass And Ceramic Division, BCSIR for providing him with the laboratory facilities and valuable assistance during the work.

The author expresses his sincere appreciation to Prof. Md. Fazley Elahi, Department of Soil Science, and Md. Anwar Hossain Bhuiyan, Lecturer, Department of Geology, Dhaka University for their valuable suggestions and help during the work. Appreciation also goes to Professor Md. Mohafizul Haque and Professor A. S. W. Kurny and Dr. Md. Fakhrul Islam of MME Dept., BUET, Dhaka for taking care the advancement of the work.

Finally the author wishes to record his gratitude to his parents and well wishers for their continued inspiration throughout of this research work.

The Author
MME Dept., BUET, Dhaka.

CONTENT

Chapter One

1 INTRODUCTION

Silica-sand is the chief raw material for glass making. The quality of a glass depends primarily on the quality of raw materials used. Colorless and other high grade glasses are normally made from sands which do not contain much of the objectionable impurities. The impurities likely to be present in the glass sands are shown in Table 1.1 along with their chemical compositions and specific gravity [1].

The glass maker needs only quartz in the sand so other minerals if present in it are to be regarded as impurities. Impurities that are likely to impart an undesired colour in glass must be reduced to a tolerable limit. Undesired colours produced in the glass are normally due to the presence of objectionable colour bearing ions such as iron, chromium, copper, manganese, etc. in a form or in a such structural environment as to give the visual colouring effect. These colours are of very low order on a chromaticity diagram having faint but very unpleasant effect on eyes. Such undesired colours are usually bluish-green or yellowish-green in the ware resulting mostly from iron contamination.

The color-bearing agents usually present in the following three forms with silica sand [2]:

1. Silica sand is frequently associated with a certain amount of clay material which may contain more or less ferruginous matter.

2. There also present a small fraction of heavy minerals containing iron and chromium as discrete minerals (e.g. ilmenite, rutile, tourmaline, magnetite, hornblende, staurolite, and some others).

3. To a lesser or greater extent, compounds of iron and alumina are carried by the silica grains as surface staining i.e. coating on the grains. The grains of silica may assume an overall deep color even after water washing due to the iron staining on the surface.

There are only a few parts of the world where high quality quartz with very low iron content of the order of 0.01 or less is found which can be comfortably used without any beneficiation process to produce optical and crystal grade ware. So the beneficiation of sand to produce colourless glass is common. However, such beneficiation process varies to a wider extent depending on the composition and nature of the deposit. Moreover, for the ease and efficient removal, the physical and mineralogical identifications of each and every impurity mineral are essential.

There are two aspects of the up-gradation of sand. Firstly, the undersized and oversized sand which might lead to inhomogeneity in the glass melt must be removed, and secondly, the colour bearing impurities must be reduced to an acceptable level. Most sands can be improved by washing with water and magnetic separation which remove the silt and iron particles associated with the sand. But this leaves the iron bearing heavy minerals and the iron oxide film on the sand grains unaffected. To remove these, along with other colour bearing minerals, further treatments like froth floatation and chemical treatment of the washed sand are necessary.

Although the recommended impurity levels are not more than 0.03 % Fe_2O_3 and 0.0005 % Cr_2O_3 in the chemical compositions of sand for the manufacture of colourless glass [3], these are found to be in the range of 0.15-0.30 % and 0.001-0.008 % respectively in the deposits of our country [4]. This is one of many reasons for which Bangladesh can not produce colourless glass. The present work deals with the characterisation of the recently founding glass sand deposit at Noapara, Sylhet and its beneficiation with a view to make it suitable for the manufacture of colourless glass.

Chapter Two

2 THEORETICAL BACKGROUND

2.1 SAND AND GLASS MAKING SAND

In Geology the term sand is applied to a mineral substance consisting of loose grains of a definite size. Sand is thus a mineral with a large percentage of grains of diameter lying between 0.1 to 1.0 millimeter [5]. Actually sand is one of the many forms of silica. Silica may be broadly classified as crystalline and non- crystalline silica. These two are again subdivided into many forms that are shown in Fig. 2.1.

Sand for glass manufacturing is essentially a quartz (SiO_2) which constitutes about 12 % of the lithosphere. The many forms of quartz may be divided into three categories :

1. **Vitreous varieties.** Rock crystal (sp.gr. 2.65), amethyst, rose quartz, smoky quartz, yellow quartz, ferruginous quartz, averturine quartz.

2. **Chalcodonic varieties.** Agate, chalcodony, chrysoprase sand, onyx, flint, hornstone.

3. **Jaspery varieties.** Jasper, bloodstone or heliotrope, touchstone, bassanite.

Quartz is a well-known universal constituent mineral of the Salic igneous rocks (granite, rhyolite, syanite, etc). It is much less common in the femic rocks (dorite, basalt, etc) but is found there not infrequently. It is present in nearly all metamorphic rocks irrespective of their origin. It makes up the greater part of the sand stones and occurs in nearly all shales and clays. The Physical and mineralogical properties of quartz [6] are shown in Table 2.1.

The crystal system of quartz is trigonal. Crystals are prismatics. Quartz is only one form of silica, occuring abundantly in nature. The other forms of silica are: tridymite (870-1470^{0}C); cristobalite (1470-1710^{0}C); opal (SiO_2. nH_2O)- amorphous; lechatelierite (silica glass)-amorphous. Fig. 2.2 and 2.3 show the structure of silica and different types of polymorphs of silica respectively.

In the figure, the reconstructive transformation represents an abrupt change in bond angles and in some cases coordination number.

Because of these change old bonds are broken, new bonds are formed. On the other hand, a displasive transformation on the other hand involves coordination changes in the second sphere by distortion of the structure. This type of transformation is faster than the other type.

Normally quartz is α-quartz or rock crystal. This α-quartz gradually transforms to other forms in case of heating at higher temperature. Quartz is stable below the temperature of 870^{0}C and other form are metastable.

$$\begin{tikzcd}
\alpha\text{-quartz} \arrow[r, "573\ ^{0}C"'] & \beta\text{-quartz} \arrow[r, "870\ ^{0}C"'] & \text{Tridymite} \arrow[dl, "1470\ ^{0}C"] \\
\text{Liquid} & \text{Cristobalite} \arrow[l, "1710\ ^{0}C"] &
\end{tikzcd}$$

The specific gravity of quartz, tridymite and cristobalite are 2.66, 2.26 and 2.32 respectively. Every forms of silica becomes soft at a temperature of 1600^{0}C and at the temperature of 2220^{0}C the liquid silica boils. High temperature can often be cooled below the transformation temperature without reverting to the thermodynamic stable form.

Based on coordination of particles three varieties of quartz are mainly recognized. These are:

1. quartzites (hard rock)
2. sandstone (light consolidation)
3. unconsolidated sands.

The difference in the degree of coordination determines the specific processes which are to be applied in the production of glass sand for the glass manufacturer.

Silica sand is the largest constituents (60 - 80% by wt.) of all types of commercial glasses. It is seldom a pure silica. It always contains varying quantities of impurities of which iron oxide is the most undesirable one that imparts objectionable green colour to a glass.

The quality of a sand for good glass manufacturing is determined from the percentage of loss during washing by water, amount of the sand retained between BS 30 and BS 100 mesh sieve and also the iron content. The sands used for making glass consist essentially of quartz grains having size range with about 80% of the grains falling between 420 microns and 120 microns.

2.1.1 Mineralogical Characteristics of the Minerals

A silica sand is always associated with a certain amount of clay mineral which may contain much or little ferruginous matter [7]. There are also present a small fraction of mineral grains denser than sand, the so-called 'heavy mineral' [8] fraction consists usually of particles of iron oxide, ilmenite, rutile, zirconium minerals and sometimes chromite and pyrites. As the name suggests, these minerals are characterized by having a specific gravity considerably in excess of 2.66, the specific gravity of quartz.

The mineralogical characteristics of the minerals generally present in glass sand deposit are given below :

1. **Kyanite.** The diagonostic feature of this mineral is dendritic form. Colourless to yellowish gray, well laminated tabular crystals with flaky cleavage, high relief and extinction angle are observed.

2. **Rutile.** The foxy-red colour is the diagonostic feature. The grains are subrounded with parallel extinction and very high relief.

3. **Staurolite.** Staurolite is typically brown to yellow colour with moderate pleochroism and is represented by irregular surface grains with hackly fracture. It possesses high relief and moderate birefrigence.

4. **Zircon.** The grains are euhedral (angular) to rounded and characterized by "zoning" in the boundary. The crystal form of the mineral is definite. It has parallel extinction and extremely high relief. Inclusions are generally found.

5. **Tremolite.** Grains are colourless to light green and possesses high relief. It shows oblique extinction (10-20) and weak pleochroism.

6. **Ilmenite.** Grains are quite opaque in transmittd light with characteristic steel gray-black colour in reflected light.The grains are irregular, subangular.

7. **Tourmaline.** Grains are green, brown, blue or black. The brown and dark coloured varities are strongly pleochroic. Crystals are prysmatic with high relief and straight extinction.

8. **Monazite.** The shape of the grains is often oval. The grains are colourless to lemon-yellow with a pitted surface

9. **Biotite.** Subhedral, brown, tabular and prismatic crystal with well developed basal planes. Prysmatic grains show almost parallel extinction ($3-5^0$). It shows unequal absorbtion of light under crossed nicols.

10. **Hornblende.** Grains are prismatic, fibrous with bluish green to bottle green colour. The colour may be unevenly distributed in the grains, being most dense in the middle and becoming paler towards the boundaries. It has also characteristic strong pleochroism and oblique extinction ($12-15^0$).

11. **Magnetite.** The very characteristic of this mineral is silver-gray or black crystals. The grains are angular and subrounded.

12. **Hematite.** The grains are irregular and also rounded. The very characteristic of this mineral is reddish-brown colour in reflected light.

13. **Sillimanite.** Grains are usually colourless, prismatic with irregular terminations, fibrous and sometimes vertically striated having straight extinction.

14. **Actinolite.** Usually grains are yellowish-green with moderate to weak pleochroism and extinction angles varies from 8^0 to 20^0 .

15. **Garnet.** Grains are irregular, isotropic with red and sometimes emerald green in colour. Inclusions are commonly found in them.

Of all these impurities the most objectionable ones are the iron bearing minerals ; next in line are the chromium bearing minerals (to glass makers).

2.2 SAND FOR COLOURLESS GLASS

The sands used for making glass are mainly composed of silica, the content of which is usually over 60% and in the high class glass sands more than 99%. Colourless and other high grade glasses are normally made from sands which do not contain much of the objectionable impurities. The major colour-bearing impurities of glass sand are iron oxide, chromium oxide and titanium oxide.

Great difficulties are encountered by the glass manufacturer due to the presence of these colour bearing impurities even when present in minute quantities. In the production of colourless glass, the usual practice is the addition of another colouring agent to produce a complementary colour to mask the undesired colour caused by a certain colour bearing impurity. But when the impurity content in sands exceeds a certain limit it is difficult to mask the objectionable colour by using a decolourizing agent.

2.2.1 Colouring Agents

It is mentioned earlier that the major colour-bearing impurities of glass sand are iron oxide, chromium oxide and titanium oxide. In the following sections, the nature of main two impurities are described in details.

2.2.1.1 Iron oxide

Iron oxide is the principal colour bearing imputities in glass sand. A number of iron oxides is found in glass sand:

1. Ferrous oxide (FeO)
2. Ferric oxide (Fe_2O_3)
3. Magnetite (Fe_3O_4)

The presence of ferric iron and ferrous iron produce yellow and bluish green colour respectively in glass. Usually the colour resulted for iron oxide in glass due to the combined effects of ferrous and ferric oxides and so the resultant colour in glass is the midway between yellow and bluish green.

$$Fe_2O_3 = 2FeO + O$$

The above reaction is carried to the right at high temperature or by the presence of a reducing agents and by the acidity (SiO_2) contents of the glass.

As the melting proceeds there is a tendency for iron to pass into ferrous state, and this tendency can be offset somewhat by the introduction of arsenic oxide or antimony oxide. Ferric oxide conditions predominate if oxidising agents such as nitre etc. are added to the batch. Iron is always present as an impurity in raw materials and so invariably glass is coloured as green.

2.2.1.2 Chromium oxide

Two forms of oxides are usually found in sand deposits. These are:

1. **Chromium trioxide (CrO_3).** This is an acidic oxide and has an orange red colour. With alkalies it forms chromates and dichromates, such as yellow coloured sodium chromate and orange coloured sodium dichromate. The basic melts favour the chromate condition. In common silica glasses CrO_3 breaks down to Cr_2O_3 which is green in colour and whose solution in the glass gives an emerald green colour.

$$2\,CrO_3 = Cr_2O_3 + 3\,O$$

2. **Chromium anhydride (Cr_2O_3).** It is a basic oxide and this or any of the chromium salts produces green colour in the sand.

2.2.2 Chemical Composition of Sand for Making Different types of Glasses

The sand used for making glasses must comply with the compositions specified by BS: 2975-1988 [9]. It should be noted that for making optical glasses, crystal glasses, flint glasses, and specially colourless glasses the colour bearing agents i.e. iron, chromium and titanium oxide must be in the tolerable range and the minimum amount of silica must be present as stated in Table 2.2.

2.2.3 Terminology and Class Intervals for Grade Scales

The size of the sand particles is an important factor for glass making. Particle size is defined by a grade scale which is a systematic division of a continuous range of sizes into classes or grades in which the intervals have some constant relationship to one another [10]. Sedimentologists usually plot the data with grain size as the abscissa and percentage as the ordinate. The standard practice is to plot grain size decreasing from left to right. A linear scale is of little use as the distribution of sizes within the range say of 1-2 mm is of far greater significance to the sedimentologist than the range 101-102 mm. Two main types of grade scale are used [11,12]:

1. **Geometric** (commonly referred to as the Wentworth scale). Here each grade differs from its predecessor by a constant ratio of 1/2 and is assigned a specific name to identify the particles.

2. **Arithmetic** (phi scale of Krumbein). This is a logarithmic expression of the Wentworth scale and commonly used by sedimentologists. The Phi scale is defined as

$$\varphi = -\log_2 d$$

where *d* is the diameter of the particle in mm. Particle sizes greater and less than 1 mm are equivalent to negative and positive values of φ respectively. The φ grade scale has several advantages in that it deals with whole numbers rather than fractions, being logarithmic it increases the symmetry of the size distribution curve, and it also simplifies the measurement of grain sizes when computing statistical parameters. The percentages are determined from the mechanical analysis data which will be presented in a form which varies according to the technique used in the analysis, i.e. weight of grains (sieves), number of grains in different size classes (Zeiss Analyser) etc.

According to the American Standard Specifications as published in the journals of the American Ceramic Society [13], the most desirable sands for glass making are those containing largest proportion of particle sizes between BS 40 and BS 80 screen mesh i.e. those passing through about 0.42 mm opening and retained on 0.18 mm sieve opening. Grains larger than BS 16 mesh (1.2 mm screen opening) and those smaller than 120 mesh (0.125 mm screen opening) are stated to be undesirable for glass manufacture. Restricting the sizes of sand grains to a relatively narrow range results in a more homogeneous melt. The glass maker is interested not only in a high degree of purity of the quartz but he is also particular about the grain size of the individual grains. The American Ceramic Society and the National Bureau of Standards recommend the terminology and class intervals for grade scales of sands which is shown in Table 2.3.

2.2.4 Different Techniques of Size Analysis

The materials are generally treated in different ways to furnish the required size analysis investigation. As sand is the only materials used in this research work it is size analysed by sieve analysis technique. The different techniques [14] generally used depending on the size range of the particles are shown in Table 2.4.

2.3 GLASS SAND DEPOSITS OF BANGLADESH

Glass sand deposits have been found at Balijuri in Jamalpur, at Chauddhagram in Comilla, at Nazirhat in Chittagong, at Kulaura and Shahjibazar in Sylhet district and in Sylhet town hillocks. According to the reports of the Geological Survey of Bagladesh [15,16] the reserves are 0.40 million tons in Chauddhagram, 0.25 million tons in Nazirhat, 0.3 million tons in Sylhet town, 15.50 million tons in Kulaura, 0.20 million tons in Balizuri, 1.45 million tons in Shahjibazar deposits. Commercial exploitation has been found possible in the above mentioned first four areas but other areas viz. Balizuri, Durgapur, Shahjibazar, Bijoypur etc. are not systematically investigated and tested yet.

Another glass sand deposit was recently found at Noapara of Sylhet district. This sand has been extensively used by a sheet glass manufacturing factory of MEB group of industries at Rupganj, Narayangonj.

The glass sand deposits of Bangladesh were found to contain colour bearing impurities in the range of 0.15 - 0.30 % for iron oxide, 0.001 - 0.008 % for chromium oxide which are well above the required limits set by the standard BS: 2975-1988. So Bangladesh is unable to produce any kind of colourless glass or glassware and, for this reason, beneficiation of local sand is mandatory possible to produce colourless glassware.

2.4 SAND BENEFICIATION PROCESSES

There are two aspects of the up-gradation of sand. First, the undersized and oversized sand which might lead to inhomogeneity in the glass melt must be removed. Second, impurities that are likely to impart an undesired colour in glass must be reduced to a tolerable limit.

The colour-bearing agents usually present in the following three forms with silica sand :

1. Silica sand is frequently associated with a certain amount of clay material which may contain more or less ferruginous matter.

2. There also present a small fraction of heavy minerals containing iron and chromium as discrete minerals (ilmenite, rutile, tourmaline, magnetite, hornblende, staurolite, and some others).

3. To a lesser or greater extent, compounds of iron and alumina are carried by the silica grains as surface staining i.e. coating or the grains. The grains of silica may assume an overall deep colour ever after water washing due to the iron staining on the surface.

The purification of sand may be effected in several ways, according to the nature of the sand and the amount of purification necessary. Most sands can be improved by water washing which removes the silt associated with the sand and the clayey iron containing minerals. But this leaves the tenacious iron oxide film on the grains of sand and the dark-coloured heavy minerals unaffected. To low down these colour bearing minerals, froth floatation and chemical separation processes are necessary. The following sections describe briefly the methods commonly applied to beneficiate sand deposits.

2.4.1 Washing

Washing consists of applying water to the material in such a manner as either to remove undesirable constituents or to remove the sand and the impurities behind.

In washing, the clay and other small particles are separated from the remainder in accordance with (a) their weight, and (h) their size In the former method, the larger particles settle out of suspension whilst the clay, etc. is carried off by the wash-water, and in the latter method the larger particles are retained by screens, through which the water and smaller particles escape.

In both of these methods, the water not only removes the clay and silt, but also a considerable proportion of any carbonaceous matter present; some ferruginous matter may also be removed, though the separation of iron films on grains of sand is much more difficult.

There are various methods [17-18] of applying the water in order that it may carry off the small particles of clay and other impurities. These are accomplished with the following washers: Trough washer, Log washer, Mud-wheel, Green away washer, Wash mill, Drum washer, Elutriator, Lewistown washer etc.

The efficiency of all these types of sand washer depends on (1) the extent to which the clay etc. is separated from the sand and is suspended in the wash water; (2) the speed of the current, which determine the size of the particles which will be carried away and, therefore, controls the amount of impurity left in the sand and the amount of sand carried away and left ; and (3) the sizes of the smallest particles of the sand which it is desired to retain, as this limits the speed at which the water can be allowed to flow through the trough.

Trough washer consists in putting the sand in a long trough having a v-shaped iron section, through which a constant stream of water is kept running. The sand is stirred up by hand using rods, spades when clay is removed and washed away by the stream of water, having the clean sand behind. This is uneconomical because of inefficient use of water and high labour cost.

Log washer uses a mechanical agitating device to ensure a thorough admixture of the sand and water. It consists of an inclined wooden trough and filled with a shaft carrying paddles arranged at an angle so as to mix and convey the material to be treated from the lower end to the upper end of the trough. The shaft revolves at about 12 r.p.m and discharges the washed sand at the top end of the trough, the water traveling in the opposite direction and carrying with it the dirt, clay etc. removed from the sand.

Mud wheel, consisting of a paddle-wheel 5 or 6 feet in diameter, revolves in a trough containing the sand and water. The paddles lift up the sand and allow it to fall again into the water, thus putting the clay and dirt in suspension so that they escape where the water is run off.

In green away washer the sand is fed onto an inclined rotary screen through which it is washed by jet of water, any gravel having being separated by the screens. The sand and water fall into a long trough provided with a rotating shaft carrying a large number of blades which turn up the sand and water and gradually transport the sand to the farther end of the trough, whilst the water flows in the opposite direction. On reaching the end of the trough the sand falls through a tank, up which a current of water is rising, into a second mixture similar to the first, through which the sand is conveyed in a direction contrary to that of the water flowing through. Finally, the thoroughly washed sand is discharged into a tank of clean water from which it is rinsed by a bucket elevator with perforated buckets which allow most of the water to escape.

Wash mill consists of a circular tank 6-15 feet in diameter and 2-6 feet deep. In the center is a vertical shaft which is rotated by a motor. Attached to the vertical shaft are several horizontal arms carrying harrows which trial in the sand and water in the mill, and stir them up, and put both sand and clay, etc. in suspension. After a sufficient amount of stirring, the contents of the mill are allowed to remain motionless, thus separating the clayey matter from the sand. The water and clay are then run off through an opening in the side of the mill and afterward the clean sand is removed. The chief drawback of the process is that it is intermittent.

Drum washers may be regarded as covered trough washers; being closed they can be worked at a greater speed and inclined more steeply than an open trough washer. If the drum is to be inclined, the shaft and blades must usually be replaced by a continuous or Archimedes screw. Scholes describes that washing is accomplished by carrying the sand mechanically through an inclined trough against a stream of water. In this washer, the trough is practically square in cross-section and in it revolves an Archimedes screw forcing the sand upstream. From the top of the first of the troughs the wet sand flows down a chute to the bottom of a second washing effect, and the operation is repeated once or twice more the water leaving the first washer carries a heavy load of loam, negetable detritous and undesirable fines. Water leaving the third or fourth effect is practically clear. Careful judgement must be exercised to control the velocity of the stream of the water in order that proper washing may be accomplished without loss of product. Scholes also mentions of a washing device in which a series of paddles with a slightly elliptical reciprocating movement thrust the sand up-stream. This type of washer is very effective, because it keeps the sand suspended as a slurry through most of the operation. There is also less sliding of the sand against steel blades and the bearings of the device are all above water.

Elutriation consists in mixing the particles to be separated with a large volume of water which flows forward at a pre-arranged rate, so as to carry away the smaller particles and leave the larger ones behind. Elutriators are all based on this separating power possessed by a current of water following at a pre-determined rate and their efficiency depends on (1) the accurate regulation of the rate at which water flows, (2) the absence of disturbing currents, and (3) the supply of materials in a state of suspension, each solid particle being definitely separated from the others. This method has shown definite promise in the separation of mica from silica sand. Elutriation may also serve as a method of classifying sands prior to further treatment. In one practice, a series of flat vibrating screen are arranged one above another. The sand is supplied to the uppermost screen and is graded and washed by jets of water which drive it, according to the sizes of the particles, through each screen in turn. The objection to this arrangement is that the sand acts as a filter and retains some of the dirt which would be separated if the sand were fully suspended in water and the dirt removed by flowing water without the use of screens.

Rake and spiral classifiers can also be used for sand washing purposes. The processes described should produce a well sized and clean silica sand product unless the surface contamination is extreme. In these circumstances cold techniques employing the use of acid may well not be vigorous enough and hot acid leaching may be undertaken. However, it must be emphasised that this technique will only be applicable where the grains are surface contaminated. It is employed in several operations world-wide and can, in the right mineralogical circumstances, reduce iron levels by a factor of 10 or more. The process uses sulfuric acid which is mixed with sand in a reaction drum and rotated for a predetermined duration. The iron coating combining with the sulfate leaves the grains clean and largely devoid of coatings [19].

2.4.2 Attrition

J. Dasher and O. C. Ralston of the U. S. Bureau of Mines [20] constructed a device which they called an attrition scrubber to agitate a sand-water pulp in such a way that the grains of sand abrade each other and grind off the film of impurity. This is because of the fact that, coated grains behave as if they were entirely made up of the coating material and thus during magnetic separation are removed along with such impurities. So this method gives help in the direction most needed, that is, in removing obstinate staining from otherwise pure silica grains. It would seen that power consumption is not excessive and the method does not demand the use of chemicals which generally raises difficulties in plant design and sometimes of maintenance of supplies.

2.4.3 Screening

Screening is accomplished by passing materials, either in the dry or wet state, through a series of screens or sieves of increasing fineness thus determining the screen analysis. The screen analysis of a sand is the expression of a very important property, the size distribution. B.S 2975: 1958 lays down the specification for size grading of glass sands and are shown in Table 2.5.

2.4.4 Concentration

In washing, the lighter and smaller particles are removed by the water, but in concentration the particles of higher specific gravity are retained whilst those of lower specific gravity are carried off. In washing, separation is largely based on the size of the particles, but in concentrating, their specific gravity is the important factor.

2.4.4.1 Concentrtation by magnetic means

Concentration by magnetic means [21-25] is often be cheap and efficient. Magnetic separations may be divided into (a) retaining separators, and (b) deflecting separators. In each case the separation depends on the fact that some substances are more readily attracted by a magnet than the others. Ilmenite, staurolite and some tourmaline are strongly magnetic; some tourmaline, zircon and some staurolite are weakly magnetic; while quartz, rutile and kyaniite are non-magnetic. In all magnetic separators it is important:

1. to use sufficient strong magnets-preferably electro-magnets as their intensity is under better control,

2. to allow the material to be separated to be in as close contact as possible with the magnets, as all intervening substances, such as belts, etc. reduces the efficiency of the separation of the smaller particles, and

3. to allow ample time for the desired material to reach and adhere to the magnets.

Retaining separator consists of a trough in which the magnets are in the form of a series of baffles across the trough so as to disturb the water carrying the sand and secure is an effective separation process of the magnetic particles from the non-magnetic ones. The magnetic particles are retained by the magnet. For cleaning, the flow of the materials is stopped and electric current is switched off. The magnetic particles can then be washed down with water and the apparatus started again in less than five minutes.

Deflecting separator, the simpleat ones, consists of a narrow inclined table or trough, down the sides of which are fixed a series of magnets. As the material flows down the trough the readily magnetized particles are deflected and adhere to the magnets. Rotating magnets are also regarded as deflected separators. It consists of a pulley formed of a series of magnets which rotates whilst the material to be treated passes over it. The attracted particles adhere until the drum has reached a pre-arranged part of its revolution, when the electric current which creates the magnet is automatically switched off, and any adherent particles then fall on into a suitable receiver. As the drum continues to revolve, the electric current is applied automatically and the drum again becomes a magnet.

On the other hand electrostatic separation is based on the difference in electrical conductivity of mineral. The principle is that if a stream of material is brought into contact with an electrically charged body, those particles in the stream are good conductor of electricity will become similarly charged and repelled, whilst those which are poor conductor will continue their flow undisturbed. This method is feasible when the specific gravity of the particles is too close to permit a satisfactory separation by gravitational methods. At present this principle is not used to any extent in the sand industry primarily because of its recent development and the relatively high cost. However it is possible to separate fluorite from quartz, rutile from zircon, and both rutile and zircon from beach sand by electrostatic means.

2.4.4.2 Concentration by floatation

A method of separation depending on differences in chemical composition is obviously desirable and Gaudin [26] has pointed out that the technique known as froth floatation does in fact depend on such differences. A mineral which contains the atom or ion not present in a second mineral can, in theory, be separated from it. To understand the principle of the method it is helpful to consider the difference in behavior of water droplets on two glass or metal surfaces, one of which is rather greasy, and the other of which has been thoroughly cleaned. On the first, the dorplets remain separate, like drops of mercury which do not readily coalesce. On the second the water spreads evenly over the whole surface. The first surface is water repellent, the force of cohesion between the water molecule being greater than the force of adhesion between the molecules of water and those in the surface of the solid. The second surface is said to be wettable, and the adhesion of the water to the surface is greater than the cohesion between the water molecules. If two mineral species are to be separated, the first stage in the floatation process is to reproduce on the surfaces of the grains the condition obtaining on the two surfaces already described, i.e the grains of one mineral must be rendered water repellent whlist the grains of the other are left unchanged. This condition is broght about by the addition of a reagent known as collector. Various other reagents may also be required to assist the collector to react with mineral if it is required to float, and to prevent from reacting with the minerals which are not required to float.

The slurry having been conditioned, the mineral particles which have been taken up the collector will have water-repellent surfaces and will tend to collect at any air-water interface which is presented to them. Air in the form of small bubbles is blown through the pulp and the bubbles become attached to the collector-coated grains, so that the latter are carried up through the water to the surface. By the addition of some surface active agent which adequately lowers the surface tension (the collector may act in this way also) a froth is maintained on top of the water, and the particles of mineral collect in this and are thereby prevented from settling back to the slurry; the froth is removed continuously by rotating scrapers. The slurry must be agitated to prevent the solids from falling out the suspension, and it is usual to combine agitation and aeration by employing a slotted impeller through which air is blown. The issuing air is sheared into very fine bubbles between the impeller and the stator which surrounds it.

Table 2.6 shows the different chemicals used in froth floatation technique. All collectors consist of a fairly large hydrocarbon molecule containing single polar group. The polar group anchors the molecules to ions in the surface of the minerals grains, leaving the hydrocarbon part of the molecule turned away from the surface. In effect, the grains are coated with hydrocarbon which, of course, is water repellent. Typical collectors are xanthates, sulphonates, and carboxylic acids for the collection of metallic minerals, nitrogen compounds such as amines, quaternary ammonium compounds and pyridinium derivatives for non-metallic minerals. The two groups generally classified as anionic and cationic collectors respectively.

The larger the hydrocarbon group the more effective is the collectors, the less soluble in water, and this is a disadvantage. A compound be sought between effectiveness and solubility, since an insoluble reagent is difficult to disperse through the pulp. There are, however, notable exceptions to this, for example, oleic acid, which is insoluble although it will spread on water, is used as a collector. It is essential that there should be only one polar group in the molecule; if it has more than one polar group the water-repellent character of the hydrocarbon part of the molecule is reduced or destroyed.

For the collection of iron minerals the petroleum sulphonates have been found quite effective. An acid pulp is neceessary to maintain the pH around 1.5; with a pH value higher than 2.0, the efficiency of collector falls off rapidly, but a reduction below 1.5 does not significantly improve it. The acid probably serves to liberate the sulphonic acids which are present as sodium salts in the commercial reagents. It may also serve to activate the mettalic minerals by cleaning the surfaces of the partcles, and for the same reason it prevents the silica from floating.

The dosage of reagent required is quite small, being of the order of 1 lb per ton of sand in a pulp containing two parts of water to one part of solid. It seems that the sulphonate is strongly adhered, as it is very firmly held once the minerals have been conditioned. Concetrate which has been removed from a floatation machine, repeatedly washed in fresh water and dried, will float again when replaced in water; sand in the range between 100 and 300 mesh float most readily [27].

To summarise: on using floatation as a means of separating certain minerals from the sand, there are three factors to be adjusted relative to one another.

1. The cresol or other froth producing substance
2. The paraffin oil or other froth stabilizer and
3. The acid which reduces the floatability of the sand.

The sand is mixed with about four times its weights of water and agitated by rotating blades. During the agitation the reagents are added and after sufficient mixing and aeration, the frothing liquid is transferred through an aperture in the side of the vessel to another vessel containing still water. Here the bubbles loaded with discrete mineral particles rise and form a froth which is removed, whilst the particles of sand left to the bottom.

2.4.5 Chemical Leaching

The true surface is, in most sands, considerably larger than that would correspond to the size of the grains. A few sands seems to consist of grains which are reasonably free from cracks and capillaries, but other have a large adsoptive capacity. Since contamination may be present on parts of the surface which are not readily accessible to attack by reagents, not all sands can be purified to the extent required for glass making and others may not be capable of economic purification; probably only those sands which are moderately free from surface fissuring can be purified. The dissolution of ferric oxide is a relatively simple matter if the choice of reagents and the quantities which can be used is not limited in any way. A large number of investigations [28-33] have been carried out to study the possible purification by chemical treatment of the natural sand deposits with the aim of improving their properties to make them suitable for use in glass industry. These include leaching the sand with (I) hydrochloric acid, (ii) hydrofluoric acid, (iii) oxalic acid and (iv) sodium hydroxide solutions of different concentrations and applied at various temperatures.

Hot concentrated hydrochloric acid is a satisfactory solvent, but even this drastic treatment must be prolonged to bring about complete solution. For economic reasons, such treatment is out of the question for commercial purification of sands. A cold leaching technique was developed by Segrove for the reduction of iron oxide coatings on the sand grain. The method uses a sodium hydrosulphite, hydrofluoric acid leach, but modern processing techniques now use sulfuric acid in place of hydrofluoric acid.

In absence of a reducing agent higher concentrations of acid are necessary. In the presence of hyposulphite, a concentration of one part in a thousand is satisfactory and stronger solutions only tend to break down the hyposulphite too rapidly. Sulfuric acid brings about some dissolution of ferric oxide under reducing conditions but it is not so effective as hydrofluoric. The reason for this seems to be that iron forms a ferrifluoride complex $(Fe\ F)^{3-}$ which is quite stable. When fresh water is added to wash the soluble ferruginous matter and the chemical liquor out of the sand, there is a rise in pH which tends to cause iron compounds to be re-deposited on the sand grains. This tendency is considerably reduced if the iron has been locked up in the ferrifluoride complex. The technique has been considerably modified in recent years by the adoption of a combined attrition and hydrosulphide acid leach, working in a sigle attrition cell. This development was undertaken in order to overcome the oxidation of the hydrosulphide used in Segrove's technique. However, single stage modification of this work has shown that the combined attrition and cold leaching technique has greatly improved many types of sands [34].

Hydrofluoric and perchloric acids are the most effective in the direct dissolution of ferric oxide. Of the commercially available reducing agents, the titanious salts are comparable in reducing power to hydosulphite, and they have the advantage of being stable in acid solution. In this connection sand is purified by the treatment with relatively strong solutions of hydrofluoric acid and titanous sulphate.

Thinking of the same phenomenon, Adam's [35] developed a process of treating sand with a hot solution containing sulfuric acid, oxalic acid and ferrous sulphate. Examination of this process, however has made such a classification rather difficult and it seems possible that it is the ferrous oxalate which first formed bring about the solution of ferric oxide. The chemical process operated at one of the first plants in the world was aimed at the removal of ferruginous surface coating from the sand grains. It was based on the dissolution of the iron oxide in a warm, acidified oxalate solution in the presence of ferrous sulphate. Leaching with an acid solution under reducing conditions formed the basis of a number of sand beneficiation processes; one of these in which hydrofluoric or sulphuric acid is used with the addition of hydrosulphite ion, as either the sodium or the zinc salt, has been operated at various plants in UK for over 20 years [36].

The use of strong acids in high concentrations and at elevated temperatures has also been reported. A process in which 20 % hydrochloric acid is used for sand leaching at around 100 C and in which the acid is recovered for recycling has been operation in UK, since 1970 [37]. Later on, because of the difficulties experienced with 20 % hydrochloric acid, the plant was converted to using 30 % sulphuric acid.

2.5 SUMMARY OF THE LITERATURE REVIEW

Among the colouring constituents in glassmaking, iron has the most wide-spread influence. The choice of a silica sand for glass making is thus largely governed by its iron content. Magnetic separation has been founded to be useful in sands containing magnetic iron. Naweed and Safdar [38] have recently shown that the non-magnetic iron can be converted to the magnetic form and separately easily. This method, however, employs a temperature of 900-1000^0 C and in its presents form can find application only in cases where high purity silica is required without much consideration of cost. Tabling is a much favoured technique in ore-dressing but it involves a good deal of space and labour [39]. In tabling the separation between two or more minerals depends mainly on the difference in specific gravity between the phases.

Many workers were devoted to remove iron from silica sand. Tscuschner [40] was probably the first worker to suggest the removal of iron from a glass-making sand by treatment with chlorides. He treated a sand containing a high amount of iron oxide with a mixture of salt and sulfuric acid, heated to redness and extracted with water. Rhodin [41] merely heated the sand with sodium chloride. Removal of iron has been attempted by heating with sulfer dioxide and chlorine and methane, by hydrochloric acid gas, and with several chlorine. Bateson and Turner [42] have made an excellent survey of the literature on this problem. The sodium chloride method was never adopted by industry because of the obvious expense on heating. From Bateson and Turner's work it appears that the removal of iron from molten glass by volatilization is more difficult to accomplish than from sand and powdered minerals. Damour and Nadel [43] report a 24 % increase in the removal of iron from glass when sodium chloride is used in the presense of selenium. Hall and Turner [44] have also studied the removal of iron from a soda-lime-silica glass with sodium chloride in the presense of selenium, boric oxide and ammonium sulphate which accelerate the reaction. They claim 3.75, 6.2 % and 17.5 % more reduction respectively as compared to sodium chloride used alone.

Instead of being treated with HCl, sands may be mixed with common salt and heated to redness. This results in the formation of $FeCl_3$

which is volatile and so escapes. Unfortunately salt is no more efficient than HCl but it is sometimes cheaper to use it. Safder [45] tried a similar experiment with a sand from Pezu, Pakistan. He reported that 40.6 % of the iron present in the sand was volatilised in the form of $FeCl_3$ and 68.3 % of the remaining portion was found magnetic after treatment and was therefore magnetically removed. In all 81.2 % iron was removed by this treatment. He further reported that in addition to iron, the volatilization of chromic oxide was also effected.

After simple heat-treatment with sodium chloride, the reduction in the iron content becomes measurable at 750^0 C but proceeds quantitively only at 900^0 C and above. The maximum removal of 40.6 % Fe_2O_3 is observed using 3 % NaCl at 1000^0 C for 15 minutes, at 950^0 C for 25 minutes and at 900^0 C for 60 minutes.

Although the maximum removal of iron by volatilyzation is attained within a relatively short time, the further removal by magnetic separation requires further treatment before the maximum removal is achieved.

The maximum removal of 68.3 % $Fe_2\ O_3$ is achieved by heating at 1000^0 C for 75 minutes. The same degree of removal is observed at 950^0 C for 85 minutes and 900^0 C for 105 minutes. At 850^0 C this degree of removal is not achieved even in 110 minutes. The effect of the heat and magnetic treatment on the Cr_2O_3 content also deserves mention.

He claimed that he was able to eliminate chromium completely by magnetic separation after heat-treatment of the sand. This has proved to be more efficient than that worked out by Bateson, Hall and Turner. The process, nevertheless is uneconomical because of the heat required (temperature about 1000^0 C). What is really needed is something which, say, we can add to the glass batch to volatilize the iron and in this connection chlorides at once occur to the mind. Knapp [46] made some trials on these lines. He tried by addition of different individual chlorides to a lead glass batch melted in amounts of 30 grams at a time. Of those trial $BaCl_2$ was the most promising, though this had little effect when trial was made with a magnesia glass. A mixture of magnesium chloride with barium peroxide, however, when used in such a batch containing an iron bearing sand was effective in reducing the iron content of the glass from 0.15 % Fe_2O_3 to 0.087 % with a reduction of 42 %. Further investigation are necessary before a large scale trial can be made.

The latest and successful process is the modified Segrove Process which involves adoption of a combined attrition and sodium hydrosulfite-sulfuric acid leach. This process may have to be extended by adding froth floatation which uses selective reagents (sulfonates) coupled to frothers under controlled pH conditions and pulp densities. These two techniques will remove iron oxide coatings on the sand grains as well as discrete iron minerals present in the sand thus making it fit for producing colourless glasses.

2.6 SCOPE OF THE PRESENT WORK

There are about ten glass manufacturing industries currently running in Bangladesh. But none of these industries can manufacture colourless glass, one of the most attractive and demanding application of tableware purposes. Colouring impurities already mentioned are present in local sand in a range not suitable for the production of colourless glasses.

So far no work of any kind is reported to be carried out in the glass manufacturing industries to beneficiate the local sand and achieve the percentage of iron oxide and chromium oxide close to the acceptable level.

Like all other industrial sectors of Bangladesh, there exist no R & D facilities in the glass manufacturing industries and, for this reason, all local glasses are found to have an irritating greenish tint and, consequently, have a considerably low selling price.

So if suitable way is developed to reduce colouring impurities from the sand, a new horizon will be opened up to the glass manufacturing industries of Bangladesh. It is obvious that once a process is developed and implemented to produce colourless glasses, Bangladesh will save a lot of foreign currencies which are currently being used to import these products. Glass making is one of a few industrial sectors in Bangladesh where a bulk portion of the raw materials required for production is found locally. So one should have asked for its development and diversified application.

In these connection, a careful examination of our local silica sand will be made to

1. study the amount of coloured impurities present in silica sand deposit of our study area,

2. work towards the eventual development of a systematic and cost-effective sand beneficiation process of local sand which will be suitable for the commercial production of colourless glass.

Chapter Three

3 EXPERIMENTAL

3.1 INTRODUCTION

The various methods applied for the beneficiation of the sand deposit were divided into two stems viz. physical treatments and chemical treatments. The physical methods comprises water washing, bromoform separation, magnetic separation were mainly applied to grade the sand, and the standard chemical methods viz. Adam's process, Sherlock's process, dithionite method,etc. and the froth flotation methods were used to remove the colour bearing impurities from the graded sand

3.2 SAMPLING

Sands usually contain grains of different sizes and specific gravities. On handling, therefore, the different types and sizes of grains tend to segregate and, in order to obtain a portion truely representative of the bulk, sampling is not an easy matter. Special procedure must be followed rigorously when a full examination is to be made. For the preliminary tests, however, the sampling may be done by the method of coning and quartering. The method can be used under both dry and wet conditions. The processes can be described as follows:

3.2.1 Dry Sampling

The whole of the sample dried at 110^0 C shall be gradually but uniformly heaped so as to form a cone, which is then slightly flattened and divided into four equal quadrant parts. Two opposite quarters are reserved, while the other two are well-mixed together, then heaped uniformly into another cone. After flattening it slightly and quartering it, two opposite quarters are again reserved. The other two are well mixed, piled into a cone and quartered. Two opposite quarters are again formed into a cone, and two quarters of this, totalling not less than 150 gm are put together and used if necessary for the grading test on the as-received sand.

3.2.2 Wet Sampling

The remaining quarters are added to all the portions of the sand which have been reserved, and any nodules or aggregates are gently broken down in a larger morter. The whole mass is then mixed and uniformly wetted by the addition of water in the form of a spray until the sand moves freely without balling. The whole of the wetted sand shall then be mixed thoroughly with the hands on a flat, non-absorbant slab or table. By mixing and quartering, the size of the sample shall be reduced to about 1.5 kg, which shall constitute the laboratory sample. The latter shall be spread out in a flat layer about 12.5 mm (0.5 inch) thick. Portions of about 5 gm each shall then be taken from different parts all over the layer, so as to yield a total of not less than 150 gm which is to be employed for the mechanical analysis of the sand. After the nodules have been broken down, portions of about 2 gm each be taken away to yield a sample of about 50 gm which shall be collected to constitute the sample for the complete chemical analysis.

3.3 PHYSICAL TREATMENTS

3.3.1 Water Washing

To remove the ferruginous clay fraction the sand was subjected to water-washing treatment. For this purpose 100 gm of as-received sand was taken in a beaker and mixed with water in the sand-water ratio of 1:2. The mixer was then gently stirred for about two to three minutes and decanted off. This operation was performed in several times until the clear water was found in the mixer. The washed sand was dried in an oven at 110°C and weighed in a balance. The amount of lost sand was determined.

Each 2 gm sample of unwashed and washed sand was heated to bright redness in a silica crucible. After cooling, the colour of the ignited sands was compared with that of the original. Any carbonaceous coating or particles of coal, etc. will have been burned off, and ferruginous matter will have assumed a deeper reddish tint. The photographs of these samples, Figs. 3.1 and 3.2, clearly show the difference between them. The water-washed sand, along with the as-received sand, were finally chemically analysed for its constituents. The results are shown in Table 3.1.

3.3.2 Sieve Analysis

Screening is accomplished by passing the dry sand through a series of screens or sieves of increasing fines to determine the size distribution. Sieve analysis was also performed to remove unwanted coarser and finer sand fractions from the sand. 100 gm each of the as-received and the water washed samples were placed in turns on the top of the sieve shaker having a series of U.S. test sieves with apertures 125, 150, 180, 250, 425, 600 and 710 microns and shaken for 30 minutes. The sands retained after shaking on each sieve and also on the bottom pan were weighed and the percentages of the whole sample determined. The graded results are reported in Tables 3.2 and 3.3. Each sieve fractions was chemically analysed for Fe_2O_3 determination. The results are shown in Table 3.4 and Fig. 3.21.

3.3.3 Attrition

100 gm of water washed sand was taken in a long beaker having capacity of 1000 ml and mixed with water in the ratio of 1:4. The slurry was then stirred by an electro-mechanical stirrer at 500 r.p.m. for about 5 to 10 minutes. The attrited sand was washed with water until the washed water seemed clear. The sand was then dried up and finally chemically analysed for iron and chromium determination.The results are shown in Tables 3.5-3.6 and Fig. 3.23-3.24.

3.3.4 Magnetic Separation

The dried water washed sample was subjected to magnetic separation to remove the ferromagnetic particles using a strong bar magnet. After this operation the weight of magnetic and non-magnetic portions of the sand were determined and finally the non-magnetic portion was chemically analysed for iron and chromium determination. The dried water washed and attrited sand was also treated in similar way. The results are reported in Tables 3.5-3.6. and Fig. 3.23-3.24.

3.3.5 Bromoform Separation

Above 100 gm of dried water-washed sand sample was subjected to this operation at a time. The sample was poured into a wide mouthed separating funnel containing about 100 ml of bromoform (sp. gr. 2.8). The funnel was fitted with a stopper at the bottom. The minerals floated on the bromoform were stirred in for 1 minute and allowed to stand for about 5 minutes. The cycle was repeated three to five times until all the heavy minerals (sp. gr. higher than 2.8) have been settled down on the bottom of the funnel. Heavy minerals along with some quantity of bromoform was then flushed through the stopper into a filter funnel containing filter paper. The bromoform was collected in a beaker, then stored in a bottle for re-used. The filter paper containing minerals was washed properly to make it devoid of bromoform by using acetone and dried up in an oven. The heavy mineral fraction of the sand was then weighed to calculate the percentage of heavy minerals present in the sample. The result is reported in Table 3.7and Fig. 3.22.

Bromoform that contains lighter minerals (sp. gr. less than 2.8) in the wide mouthed separating funnel was then recovered for re-use in the similar manner.

Some amount of heavy minerals was subjected to magnetic separation using bar magnet. The magnetic and non magnetic portions of heavy minerals were accurately weighed. The result thus obtained is shown in Table 3.8.

3.4 MINERALOGICAL INVESTIGATION

3.4.1 Introduction

For the ease of effective removal of colour bearing minerals from the sand deposit, identification of minerals containing the sand is essential. The quantitative and semi-quantitative identification of minerals were carried out with the help X- ray diffractometry and polarising microscope.

3.4.2 Mineralogical Microscopy

3.4.2.1 Grain Slide Petrography.

Slides of as received sand, water washed sand, heavy minerals, magnetics and non-magnetics were prepared. Enough grains of each types were placed on a glass slide containing Canada Balsam and were heated to about 120^0 C to act as a binder. A thin glass cover was then placed over the grains when Canada Balsam acted as a gum. After that the slides were cooled down and washed with acetone. The minerals were identified under the polarizing microscope. Figs. 3.3 to 3.15 show the different minerals identified by this method.

Minerals' frequency was obtained by a line counting method [47] and about 100 mineral grains were counted for each slide. The results obtained from grain counting analysis are shown in Tables 3.9 to 3.11.

Opague grains were counted separately. The opague grains consist mainly of ilmenite, magnetite and hematite. Unidentified grains consist of minerals that have been altered to such an extend that their optical properties are impossible to recognize.

3.4.2.2 Thin Section Slide Petrography

The first step in the manufacture of a thin section [48] involves production of a smooth flat surface impregnated with sand grains suitable for mounting on a glass slide. Canada balsam was properly mixed with sand grains and then was placed over a glass slide just forming roughly a rectangular block making a flat surface by covering with a cover slide.

Then the slide was placed on a flat surface and retained for two days for adhering purposes. The block became hard. Then the block was placed on Precision Thin Section Equipment and gradually cut from the cover slide side with slow feed. During each cut special care was taken for moving the side away handle. When the chip was brought to 1 mm thickness the cutting in this machine was stopped. Then the mineral slice was finely ground on carborundum powder and each time was observed under the microscope. The thin slice was kept uniform in thickness during grinding and the was continued until a thickness of about 0.03 mm was attained.

The thickness of the slide was controlled through the final stage by the microscopic observation of the interference colours given by some known mineral (quartz in our case) in the section when covered with a film of water. As quartz is frequently present in our sample the resulting interference colours were almost entirely white or gray.

When the section was ground to the proper thickness, it was washed free from grinding powder and dried. Fresh balsam was then smeared over the surface of the slice. It was cooked and faced with a cover glass slide. The preparation was then cooled and excess balsam around the edge of the cover glass was dissolved with aceton followed by a wash with kerosene. The thin section was then covered and now ready for petrography. Fig. 3.16-3.17 show the microphotograph of the thin section slide of water washed sand.

3.4.3 X-ray Diffractometry

The XRD procedure [49] was applied to identify the predominant minerals present in the heavy mineral portion of the sand. For this purpose samples were pulverized using an Agate Mortar. The fine powdered grains of each sample were packed tightly into the cavity of a glass slide with random orientation of the powder.

The sample was subjected to the test by using a JEOL-100P X-ray powder diffractometer with Mo (Mn) K_α radiations at 30 kV and 20 mA over an angle of 5 to 50 degrees 2θ with a scanning speed of 2 degrees 2θ per minute and a chart speed of 10 mm per minute. The resultant XRD patterns are shown in Fig. 3.18. From the pattern d spacings were calculated and, by comparing the d-spacings with those of standard d-spacings, the minerals were identified (Table 3.12).

3.4.3.1 Semi- quantitative XRD Analysis

Peak-height ratios (peak height and 1/2 width ratio) of the various minerals were used as semi-quantitative indicators of relative abundance [50]. These ratios were obtained by measuring the heights of the peaks' characteristic of the minerals.

For determining the percentage of minerals it was necessary to use peaks not entirely free from the effects of other minerals. To get relative percentage of the minerals the height of the peaks and the corresponding half width of the peaks were determined. The average percentage of minerals is given in (Table 3.13).

3.5 CHEMICAL TREATMENTS

3.5.1 Standard Chemical Treatments

Usually the chemical treatment of sand is mostly used to produce sands suitable to manufacture high quality special glasses only because it is expensive. A number of standard chemical methods and the froth floatation method were applied to water-washed and magnetically treated sand in the present work and a brief description of each method is given below.

3.5.1.1 Adam's Process

50 gm sample was stirred in a dish at 50° C for 5 minutes with a solution containing 2 gm sodium acid oxalate ($NaHC_2O_4$) and 1 gm ferrous sulphate ($FeSO_4 .7 H_2O$). in 100 ml of water. The sand and the solution was taken in the ratio of 1 : 2. The liquid was then drained away and the treated sand was properly washed with water to remove all soluble salts. Then the sand was dried at 110° C and chemically analysed for iron oxide and chromium oxide determination. The results are shown in Tables 3.14- 3.15 and Fig. 3.25-3.26.

3.5.1.2 Sherlock's Process

The sand was well mixed with a solution containing 60 parts of water, 0.5 part of a 15 % solution of titanium sulphate and 0.5 part of 60% commercial hydrofluoric acid. The slurry (sand : solution) was made in the ratio of 1:4. The treatment was carried out for the duration of about 10 minutes. After treatment, the solution was decanted off. Then the sand was properly washed with water and dried at 110° C and chemically analysed for iron and chromium determination. The results are reported in Tables 3.14 -3.15 and Fig. 3.25-3.26.

3.5.1.3 Sodium Hydroxide Process

100 gm, sand was properly wetted with 10 % sodium hydroxide solution and then heated for about 10 minutes at 150 °C. The resulting mass was then stirred with sufficient water and allowed to stand for 5 minutes. The supernatant liquor was drained away. The sand was properly washed with water and dried off. The dried sand

was chemically analysed for iron and chromium content and are reported in Tables 3.14- 3.15 and Fig. 3.25-3.26.

3.5.1.4 Sodium Carbonate Process

100 gm sand was mixed with 10 % sodium carbonate solution. The slurry (sand : solution = 1 : 2) was then boiled for about 10 minutes. The supernatant liquid was decanted off. To this sand 100 mL of hot water was added and boiled for another 10 minutes and the liquid was drained away. Finally the sand was washed with tap water and dried. The dried sand was analysed for iron and chromium content. The results are reported in Tables 3.14- 3.15 and Fig. 3.25-3.26.

3.5.1.5 Iron Sulfate and Sulfuric Acid Combined Process

100 gm sand was mixed with 5 % ferrous sulphate and 15 % sulfuric acid solution in the ratio of 1 : 2 and heated to constant boiling for about 10 minutes. The liquid was then drained away. Then 100 ml of cold water was added to the sand and again boiled for about 5 minutes to wash of the sulfate completely. The supernatant liquor was decanted off. Finally the sand was thoroughly washed with fresh water and dried in an oven at 110 C and chemically analysed for iron and chromium content. The results are shown in Tables 3.14-3.15 and Fig. 3.25-3.26.

3.5.1.6 Hydrochloric Acid Process

100 gm sand was mixed with 10 % hydrochloric acid solution and the slurry was made in the ratio of 1 : 2 and heated to constant boiling for about 10 minutes. The liquid was then drained away. Then 100 ml of cold water was added to the sand and again boiled for about 5 minutes to wash off the chloride completely. The supernatant liquor was decanted off. Finally the sand was thoroughly washed with fresh water and dried in an oven at 110° C and chemically analysed for iron and chromium content. Tables 3.14-3.15 and Fig. 3.25-3.26 report the result of this process.

3.5.1.7 Sulphuric Acid Process

100 gm sand was mixed with 10 % sulphuric acid solution. The slurry was then boiled for about 10 minutes. The supernatant liquid was decanted off. To this sand 100 ml of cold water was added and boiled

for another 10 minutes and the liquid was drained away. Finally the sand was thoroughly washed with tap water and dried. The dried sand was analysed for iron and chromium content. The results are shown in Tables 3.14- 3.15 and Fig. 3.25-3.26.

3.5.1.8 Dithionite Process

100 gm sand was subjected to this treatment. The sand was mixed with 50 mL of 0.3M sodium citrate and 0.5 gm of sodium dithionite. The pH of the solution was maintained at 7.5 using sodium bicarbonate. The slurry was heated at 80° C for about 15 minutes. Then the supernatant liquid was decanted off. Finally the sand was washed thoroughly with fresh water and dried. The dried sand was analysed for iron and chromium content by standard methods. The results are reported in Tables 3.14- 3.15 and Fig. 3.25-3.26.

3.5.1.9 Sulphite Process

100 gm sand was properly mixed with 0.4 % sodium sulphite and taken in a plastic beaker containing water mixed with hydrofluoric acid maintaining pH at 2.7. The slurry was stirred with a wooden rod for about 10 minutes. The supernatant liquid was then decanted off. The sand was thoroughly washed with water and dried in an oven at 110° C and chemically analysed for iron and chromium determination. The results are reported in Tables 3.14- 3.15 and Fig. 3.25-3.26.

3.5.1.10 Treatment With Sodium Chloride

20 gm of sand was well mixed with 0.5 gm of sodium chloride and the mixture was transferred to a platinum crucible and heated at 1100° C for half an hour. It was then cooled, washed with fresh water to remove surplus sodium chloride and other soluble impurities. The sand was dried and was analysed for iron and chromium content. The results are shown in Tables 3.14- 3.15 and Fig. 3.25-3.26.

3.5.2 Froth Floatation

Froth floatation treatment was performed with the help of a froth floatation machine, type-D12, DENVOR, UK. Firstly a sand-water slurry was made in the ratio of 1:2 in a rectangular steel vessel. Each time 1/2 kg to 1 kg sand was treated. The vessel containing the slurry was placed beneath the stirrer and then the stirrer was gradually descended in such a position that the swirling action can be transmitted within the sand. The reagents were added within the slurry and the pH of it was maintained within the required range using H_2SO_4 and Na_2CO_3 . The revolution of the stirrer was fixed within the range of 850-900 r.p.m and the slurry was stirred up for about 5 minutes after which the compressed air was supplied at a pressure of about 35 kPa through the channel of the stirrer to produce bubble. The second stirring action was continued for about 10 to 15 minutes. The black discrete minerals entrapped with bubbles were removed gradually along with the froth. When no such minerals were seen coming with the froth, the operation was stopped and the solution was drained up. Fresh water was then added within the vessel and the sand was rinsed at 500 r.p.m. for another 5 minutes and the solution was decanted off. Finally the sand was washed with fresh water, dried up and analysed for iron and chromium contents.

A total of thirteen batch compositions were prepared for the froth floatation treatment using different chemical reagents (Table 3.16). All but one of these batches have a pH value in the range of 1.5 to 2 (acidic); batch 6 was performed at pH of 10.8 (basic). The results of froth flotation of all the batches are reported in Tables 3.17- 3.18 and Fig.3.27.

3.6 CHEMICAL ANALYSES

3.6.1 Determination of Loss of Ignition

2 gm of the prepared sample was taken into a previously heated and weighed platinum crucible. Let the mass of the sample be M_1. The sample was heated at 925^0 C in a furnace for 15 minutes and then allowed to cool in a desiccator and weighed. The operations of heating, cooling and weighing were repeated until the difference in mass between two successive weighing did not exceed 0.5 mg. The mass of the residue obtained from the last weighing was recorded as M_2. From the two weights, the loss of ignition was calculated and the result was then reported.

3.6.2 Preparation of Standard Solutions

For the determination of oxides of iron, chromium, potassium, magnesium, aluminium, silicon, and calcium standard solutions were prepared for spectrophotometer and atomic absorption spectrometer.

3.6.2.1 Standard Iron Solution

0.4911 gm of di-ammonium iron (II) sulphate hexahydrote was dissolved in water and then 10 ml of hydrochloric acid solution was added and finally diluted to the mark in a 1000 ml one-mark volumetric flask and mix. 1 ml of this solution is equivalent to 0.1 mg of iron (III) oxide. Measured volumes of the dilute standard solution were transferred to separate 100 ml one-mark volumetric flasks to cover the range. Each flask was treated as follows:

The solution was diluted to 40 ml and then 10 ml of tartaric acid solution (100 g/l) was added. To each flask in turn two drops of 4-nitrophenol indicator solution (10 g/l) and ammonia solution (1:1) were added drop by drop and stir until a deep yellow colour was just obtained. The inner walls of the flask were immediately rinsed down and hydrochloric acid solution (1:4) was added drop by drop and stir until the solution just became colourless and two drops were added in excess 2 ml of hydroxyl-ammonium chloride solution (200 g/l) was added, swirled to mix and then added 10 ml of 1,10 phenanthroline hydrate solution (1 g/l). The solution was then diluted to the mark and mixed. The solutions were allowed to stand for 15 min and then the absorbance was measured at 510 nm using 10 mm cells and distilled water as the reference solution. The blank reading was subtracted from the sample reading and a calibration graph was plotted with the absorbance against the mass of iron (III) oxide.

3.6.2.2 Standard Chromium Solution

0.194 gm of potassium dichromate was dissolved in water and diluted to the mark in a 1000 ml one-mark volumetric flask and mixed properly. 1 ml of this solution is equivalent to 100 microgram of chromium (III) oxide. 1 gm of anhydrous sodium carbonate and 0.2 gm of disodium tetraborate were transferred to each of 100 ml beakers and dissolved in 20 ml of water. Aliquot portions of the dilute standard chromium solution was transferred to it to cover the expected range and diluted to 50 ml. 10 ml of sulfuric acid solution, 1 ml of silver nitrate solution and 1 gm of ammonium peroxodisulphate were added to the cold filtrate. Then the solution was boiled gently for 20 min, cooled and transferred to a 100 ml one-mark volumetric flask and diluted to the mark with the stabilized water. 50 ml of the solution was pipeted into a dry 100 ml conical flask containing 2 ml of 1,5-diphenylcarbazide solution (2 g/l), swirled to mix and meseared the absorbance at 540 nm using 10 mm cells with distilled water as the reference solution. The blank value was subtracted and the observed absorbance was plotted against the mass of chromium (III) oxide.

3.6.3 Determination of Silica

2 gm of sample was taken in a platinum crucible to which 15 ml of hydrofluoric acid solution (425 g/l) and 1 ml of sulfuric acid solution (1:1) was added and evaporated to a syrup, taking care to avoid loss by spitting. The crucible was cooled and the sides of the crucible was washed with water. 10 ml of hydrofluoric acid solution (425 g/l) was added and repeated the evaporation to dryness. The residue was heated on a hot plate until fumes of sulfuric acid were no longer evolved and then heated in a furnace at 1100° C for 15 minutes. Then the residue was cooled in a desiccator and weighed. The operations of heating, cooling and weighing were repeated until the difference in mass between two successive weighing was not exceed 0.5 mg. The difference between this mass and that of the original sample was calculated and the percentage of silica was determined.

3.6.4 Determination of Total Iron Content

2 gm of the prepared sample was taken in a platinum crucible having capacity of 30 mm and then moistened with water. 15 ml of hydrofluoric acid (425 g/l) and 1 ml of sulphuric acid (1:1) were added and allowed the mixture to digest on a hot plate until solution is complete. The temperature was increased and evaporated the solution carefully to dryness. The crucible was then removed from the hot plate and allowed to cool. 10 ml of hydrochloric acid solution was added to it , heated to boiling and filtered through an open paper. The filtrate was transferred to a 100 ml one-mark volumetric flask and retained.

The paper was returned to the crucible, dried and burnt off the paper before proceeding with the fusion.

1 gm of anhydrous sodium carbonate and 0.5 gm of disodium tetraborate was added to the dry residue and fused gently over a burner until a clear solution was just obtained. The solution was allowed to cool and then carefully added 10 ml of hydrochloric acid solution (1:1) and boiled to dissolve the melt. The solution was cooled and transferred to the 100 ml one-mark volumetric flask (containing the filtrate from the previous acid treatment). The solution was diluted to the mark and mixed.

Aliquot portions of the soltion were pipetted to separate 100 ml volumetric flasks taking sufficient amount to cover the range. 10 ml of tartaric acid solution (100 g/l) were added to each flask. To each flask in turn two drops of 4-nitrophenol indicator solution (10 g/l) and then ammoia solution (1:1) were added drop by drop and swirl to mix until a deep yellow colour was just obtained. The inner walls of the flask were immediately rinsed down and added hydrocloric acid solution (1:4) drop by drop and with swirling until the solution just became coloureless and then added two drops in excess. Then 2 ml of hydroxylammonium chloride solution (200 g/l) was added, swirled to mix and then added 10 ml of 1,10 phenanthroline hydrate solution (1 g/l). The solution was then diluted to the mark and mixed. The solutions were allowed to stand for 15 min and then the absorbance was measured at 510 nm using 10 mm cells and water as the reference solution.

At the same time a blank solution was prepared following the same procedure but omitting the sample. The blank reading was subtracted from the sample reading and the mass of iron present in the sample solution was determined from the calibration graph and calculated the Fe_2O_3 content of the sample. The result was reported.

3.6.5 Determination of Total Chromium Oxide Content

2 gm of the prepared sample was taken in a platinum crucible having capacity of 30 mm and then moistened with water. 15 ml of hydrofluoric acid (425 g/l) and 1 ml of sulphuric acid solution (1:1)

were added and then allowed the mixture to digest on a hot plate until solution is complete. The temperature was increased and evaporated carefully to dryness. After cooling 1 gm of anhydrous sodium carbonate and 0.5 gm of disodium tetraborate were added and heated at 1000^0 C for 15 minutes. The crucible was cooled and 20 ml of water was added. When the melt was dissolved the liquid was filtered into a small beaker and washed with hot water until the volume was about 70 ml.

10 ml of sulfuric acid solution (196 g/l), 1 ml of silver nitrate solution (25 g/l) and 1 gm of ammonium peroxodisulphate were added to the cold filtrate. Then the solution was boiled gently for 20 min, cooled and transferred to a 100 ml one-mark volumetric flask and diluted to the mark with the stabilized water. 50 ml of the solution was pipetted into a dry 100 ml conical flask containing 2 ml of 1,5-diphenylcarbazide solution, swirled to mix and meseared the absorbance at 540 nm using 10 mm cells and water as the reference solution.

At the same time a blank determination was carried out following the same procedure but omitting the sample. The blank value was subtracted from the calibration graph and the Cr_2O_3 content was calculated using the calibration graph. The result was reported.

3.6.6 Determinations of Calcium, Magnesium, Sodium, Aluminium and Potassium Oxides

1 gm of the prepared sample was transferred to a platinum crucible and moistened with water. 15 ml of hydrofluoric acid solution (452 g/l) , 1 ml of nitric acid solution (994 g/l) and 2 ml of perchloric acid solution (924 g/l) were added and digested on a hot plate until the solution is complete. The temperature was increased and evaporated until perchloric acid fumes ceased. The crucible was cooled, a further 2 ml of perchloric acid solution (924 g/l) was added and then the evaporation was repeated until the residue was almost dry. The solution was allowed to cool. 10 ml of hydrochloric acid (1:4) solution was added and warmed the mixture to produce a clear solution. The solution was then cooled, transferred to a one-mark volumetric flask, diluted to the mark and mixed.

The calcium oxide, magnesium oxide, sodium oxide or potassium oxide content of the sample was then determined by AA-680 Atomic Absorption Spectrometer, Shimadzu, Japan.

Chapter Four

4 RESULTS AND DISCUSSIONS

4.1 INTRODUCTION

The colour bearing agents usually present with the silica sand as: (1) clayey materials which may contain more or less ferruginous matters, (2) heavy particles containing iron and chromium as discrete minerals, and (3) surface stains or coating of iron and aluminium compounds on silica grains. Since iron is the main colour bearing element in the sand deposit, the main emphasis in the present work is given to reduce (remove, if possible) the iron bearing materials. If this can be done successfully, it is expected that the chromium level will also be reduced during the process.

4.2 MINERALOGICAL INVESTIGATION OF SAND DEPOSIT

Both the magnetic and non-magnetic portions of the sand were subjected to qualitative and semi-quantitative analysis by using XRD technique and petrography, respectively. The XRD technique provided only the identification of the major minerals present in the samples. On the other hand, a better identification, both qualitative and quantitative, of minerals in the sample is possible by mineralogical microscopy.

4.2.1 Optical Microscopy

Mineralogical microscopy was employed using both transmitted and reflected light to ascertain the relative percentage of opague and non-opaque minerals of the samples. The identified iron bearing minerals in this study are biotite, hornblende, actinolite, staurolite, magnetite, ilmenite, haematite and tourmaline. The titanium bearing minerals are rutile and ilmenite. The only chromium bearing mineral in the sample is garnet.

Fig. 3.16-3.17 show the microphotoghaph of thin section slide of water washed sand and the major mineral recognised is quartz. This is due to the relative abundance of quartz in the slide.

The petrography of heavy minerals also indicates that the amount of kyanite is the highest (22.60 %) followed by staurolite and tourmaline, although the total percentage of actinolite, tremolite and sillimanite together is the second highest (Table 3.9). The magnetic porion of the heavy minerals contains higher percentage of magnetite (Table 3.10) and its percentage is increased in the finer sieve fractions. The amount of kyanite minerals is highest amongst the nonmagnetic minerals (Table 3.11). Except muscovite and hornblende, the percentage of the non-magnetic minerals are generally increased in the coarsed sieve fractions. Table 3.11 also indicates that the overall percentage of actinolite, tremolite and sillimanite is increased gradually in coarser sieve fractions and hence justified their sorting.

Opaque grains were counted separately. The opague grains consist mainly of ilmenite, magnetite and hematite. Unidentified grains consist of minerals that have been altered to such an extend that their optical properties are impossible to recognize.

4.2.2 X-ray Diffractometry

Fig 3.18 and Table 3.12 show the XRD patterns and the d-spacings of the major minerals identified in the sample respectively. The major minerals identified are kyanite, staurolite, magnetite, tremolite, actinolite, magnetite, garnet, sillimanite, ilmenite and some mica.

The semi-quatatitive XRD analyses of the minerals identified showed that the kyanite content of the heavy mineral is 31.79 % (Table 3.13). On the other hand, the percentages of sillimanite, actinolite and tremolite are 5.93, 7.95 and 14.58 respectively. So the total percentage of these three minerals is 28.46 %. These two percentages are higher than that obtained by petrographical analysis. This is probably due to the inclusion of the peaks of some undetermined minerals in XRD analysis. But the other minerals showed the percentages near to the petrography.

4.3 SEPARATION OF IMPURITIES

The colour bearing minerals are separated partially by froth floatation technique that are confirmed by the micrograph of the petrography of the minerals separated in the froth floatation method. To remove the magnetic minerals fully it is necessary to utilize magnetic separation in line with froth floatation before or after it.

4.3.1 Physical Separation

4.3.1.1 Separation of Clay Minerals

The removal of clay fraction presents no especial difficulty because when water is added to the sand the clay disperses to form a slow settling suspension and separation is affected by taking advantage of the difference in settling rates between the clay particles and the grains of silica. Removal of clayey materials was accompanied by a decrease in the Fe_2O_3 and Cr_2O_3 levels from 0.30 % to 0.12 % and from 0.0067 % to 0.0042 % respectively (Table 3.1).

Inspection of the table 3.19 indicates that the sample contains 4.46% clay and 4.65% moisture. According to British Standard Specifications maximum moisture content of colourless glass should be within the range of 4.5 ± 0.5%. So the moisture content in Noapara sand deposit is within the safe limit.

4.3.1.2 Sieve Analysis

Table 3.2 and Fig. 3.19 show that 21.72% of the as received sand is retained by the 425 no. US sieve and 95.68% by the 150 no. US sieve. This means that the useful sand fraction (the fraction between -425 and +150 micrometers opening) is 73.96% of the original sand. In the other words, 26.04% of the sand goes to waste. Therefore, it is advisable to separate this fraction at the site of the deposits, otherwise it will unnecessarily increase the freight charges and hence the cost of sand.

The results of the mechanical sieving of water-washed samples are reported in Table 3.3 & Fig. 3.20. Close inspection of table shows that 71.34 % of the sample falls within +250 μm grain size which indicates that the sand of Noapara basin is predominantly of medium grade sand and moderately sorted.

The sieve analysis also showed that +425 to +250 mm sized sieved fractions of the water-washed sand contain a significantly lower concentration of iron (i.e. 0.06 to 0.08 % Fe_2O_3) (Table 3.4 and Fig. 3.21). On the other hand, -250 to -125 mm sized fractions that constitute the balance sand, contain higher concentration of iron (i.e. 0.18 to 0.42 % Fe_2O_3).

The 70 % portion of the sand containing low concentration of iron is suitable for beneficiation by various techniques which may yield sand with very low concentration of iron and suitable for the manufacture of colourless glasses. On the other hand, the other 30 % portion of the sand can be used to produce coloured glasses.

It is also clear from the sieving of washed sand that only 5.86% improvement of the original as-received sand has occurred. Hence it is advisable firstly to sieve the original sand and then to apply water washing. Two alternate routes could be the wet screening and upward current classification with cyclones techniques to remove the finer fraction.

Table 3.7 and Fig. 3.22 indicate that concentration of heavy minerals (containing colour bearing agents) are higher in the finer sieve fractions hence if the -250 to -125 micrometer opening sieve fractions can be discarded before use, then 0.4553 % of heavy minerals out of 0.875 % can be eliminated, and the remaining portion may not affect the production of clear glass after all ! The amount of water-washed sand remained un-used due to this cut is 28.32 %. It is also clear that 79.05 % of the un-washed sand falls within + 250 micrometer grain size while it is 71.34 % in the case of washed sand. Hence it further established idea to utilize the graded un-washed sand for the subsequent washing and other treatments in order to reduce the processing cost.

4.3.1.3 Bromoform Separation

Each seived fractions was taken for heavy minerals analysis using the method of bromoform separation. The studies indicate that sand fraction greater than 425 μm. diameter are completely devoid of heavy minerals (Table 3.7 and Fig. 3.22). So the content of heavy minerals is appeared to be higher in the finer fractions as compared to the other fractions of the sample. As a rule according to the principles of size, density and sorting action, concentrations of heavy minerals are always higher in the finer fractions [51].

4.3.1.4 Magnetic Separation

The use of mechanical separation processes can lead to a removal of a maximum of only 69.33 % Fe_2O_3 (Table 3.5 and Fig. 3.23) and 76.42 % Cr_2O_3 (Table 3.6 and Fig. 3.24) when a combination of water washing, attrition and magnetic separation is used. Although a significant reduction in iron and chromium bearing materials is made in the mechanical separation processes, this is not enough to produce the specified grade of sand to manufacture colourless glass. About 30.67 % of Fe_2O_3 is still present in the sand either in chemical combination or as heavy mineral fraction which cannot be removed by the above-mentioned mechanical processes. But this sand is still unsatisfactory for the manufacture of colourless glass. From petrography it is clear that the black particles which are removed from the sand by strong magnet are magnetite, ilmenite, and hematite and some iron bearing minerals. The sand at this level of purity are suitable for sheet glass manufacturing.

The magnetically treated sand was subjected to mild acid leaching to make sand suitable for the manufacture of colourless glass. The sand grains with an iron coating were attacked during leaching. Very little volatilization of iron from the dark grains appeared to take place. However the magnetic susceptibility of the dark grains increased considerably. This indicates that magnetic separation thus remove a considerable fraction of iron oxide and chromium oxide both of which are objectionable to the glass-maker.

4.3.2 Chemical Separation

Two separate routes are followed for chemical treatments of the sand: (1) use of different chemical reagents to dissolve colour bearing materials, described simply as chemical treatment processes, and (2) the froth floatation process.

4.3.2.1 Chemical Treatments

Among the various chemical treatment processes used, the sodium chloride process is found to be the most effective. It reduces the Fe_2O and Cr_2O_3 level of water washed and magnetically treated sand from 0.105 % to 0.033 % (Table 3.14 and Fig. 3.25) and from 0.00242 to 0.00071 (Table 3.15 and Fig. 3.26) respectively that are close to the specified level for the colourless glass.

4.3.2.2 Froth Floatation

In froth floatation process discrete minerals are selectively floated and separated from sand grains by using anionic collectors such as sulphonates, xanthates, carboxylic acid etc. Saturated hydrocarbons e.g. paraffin oil are used to stabilize the froth, while sulphuric acid, soda ash, sodium silicate and some other salts are used as depressor or deflocculator.

About 13 trial reagent groups are prepared for the effective separation of iron and chromium bearing minerals in the froth floatation process (Table 3.16 and Fig. 3.27). Among these, T-12 and T-13 reagent groups are found to be the most successful. The iron content has reduced to the level of 0.032 % and 0.034 % receptively which are 89.33 and 88.67 % respectively lower than that of the original Fe_2O_3 level, Table 3.17. The reduction of chromium content is 80 and 75 % respectively and the treated sand contains 0.0012 and 0.0015 % Cr_2O_3 respectively, Table 3.18. So it is seen that the final Fe_2O_3 and Cr_2O_3 levels in the treated sand are very near to the specified colourless glass composition.

However, complete separation is seldom achieved by floatation method and the separation of iron oxide has been proved difficult. Usually the iron minerals in sand deposits are sufficiently small to float readily. But if the metallic minerals be locked into the silica grains then it is impossible to remove them without grinding which would lead to excessive finer fraction. Also, the difficulty arises from the ease with which Fe_2O_3 can be dispersed to form colloidal particles. Iron oxide in this form becomes extremely adsorbtive and takes up large amounts of collector and hence difficult to float due to purely mechanical point of view. Chromium minerals on the other hands float quite readily.

Chapter Five

5 CONCLUSIONS

1. The minerals found in Noyapara glass sand deposit can be grouped into 5 types of associations: (a) stable or ubiquitous minerals (tourmaline, zircon, rutile), (b) metamophic species (staurolite, kyanite), (c) amphibole minerals (hornblende, tremolite), (d) the micas (muscovite and biotite), and (e) discrete minerals (magnetite, ilmenite, hematite, sillimanite, actinolite and garnet).

2. +425 and +250 mm sized sieved fractions constituting 70 % of the total water-washed sand contain significantly low concentration of iron and is suitable for the beneficiation process. On the other hand, -250 to -125 mm sized fractions that constitute the balance sand i.e. 30 %, contain higher concentration of iron and can be used to produce coloured glasses.

3. The concentration of heavy minerals are found to be higher in the finer sieve fractions. Hence if we discard those sand of -250 to -125 sieve fractions, although about 28.66 % of washed sand is lost, we would have left with 71.34% of sands containing only about 0.42 % of total colour bearing minerals.

4. The overall washed sand contains magnetic minerals only about 10 % of total heavy minerals. But the use of mechanical separation processes can remove a maximum of only 69.33 % Fe_2O_3 when a combination of water washing, attrition and magnetic separation is used. This is insufficient to produce the specified grade of sand to manufacture colourless glass.

5. Among the various chemical treatment processes used, the sodium chloride process, is found to be the most effective. It reduces the Fe_2O_3 level of water washed and magnetically separated sand from 0.105 % to 0.033 % which is close to the specified level for colourless glass.

6. The froth floatation treatment of graded, water washed, attriated and magnetically treated sand with an acidic medium with pH 1.8 and in the presence of sodium lauryl sulphonate as a collector and lead nitrate as froth stabilizer was found to give the best result and, hence, could be applied commercially to beneficiate the Noyapara glass sand deposit.

7. Using this specific reagent group, the iron and chromium contents can be reduced to 0.032 % and 0.0012 % respectively which is very near to the specified colourless glass composition.

8. By utilizing of potassium nitrate instead of using poisonous lead nitrate the results were also found good.

Chapter Six

6 SUGGESTIONS FOR FUTURE WORK

The various standards and trial methods employed in beneficiating the sand for the manufacture of colourless glass requires a great deals of research to find out a cost-effective beneficiation method. The author faced a lot of experimental difficulties to carry out some important tests such as froth floatation, chemical analysis employing spectrometric grade reagents with the help of spectrophotometer and atomic absorption spectrometry and also during the petrography. However once the well-equipped instruments are available further studies may be carried out following the guidelines from this work.

1. The one route may be followed in the way that firstly the water washed sand is magnetically treated in wet condition during water washing, then it is treated in froth floatation technique which is followed by mild acid treatment.

2. The other route may be heating the water washed sand with sodium chloride which is in line magnetically treated and then in heated condition (although some amount heat will be lost during going through the route) into the glass making furnace.

3. The another route may be firstly cut the undersize sand before washing then it is water washed followed by magnetic separation and then subjected to froth floatation treatment and finally dried and put to glass making batch.

Bibliography

7 BIBLIOGRAPHY

1. Milner. H. B. Sedimentary Petrography Vol. 1, fourth edition, George Allen and Unwir Ltd. p. 229 (1962).

2. Segrove, H. D., "The production of sand for making colourless glasses," J. Soc. GlassTechnol., Vol. 40, p. 363 (1956).

3. B. S. I., British Standard Specification, "Sand for making colourless glass," B. S. 2975:1958.

4. Personal Communication (1995).

5. Charan, R., Handbook of glass technology., Third edition, p. 19 (1956).

6. Kerr, P. E., Optical Mineralogy, p. 273 (1977).

7. Putrin, A. M. and Naumova, R. A., "Floatation ore-dressing plant for quartz sands," Consultants Bureau, A division of plenum publishing corporation, N. Y., USA, P. 294 (1974).

8. Adams, F. W., British Patent No. 423 053, (1945).

9. B. S. I., British Standard Methods, "Sampling and analysis of glass making sand," B. S., 2975 : 1988.

10. Visher, "Grain size distribution and depositional process," Journal of Sedimentary Petrology, 39, p. 1074 (1969).

11. Wentworth, "Journal of Geology" Vol. 30, p. 377 (1922).

12. Krumbein, "Journal of sedimentary petrology," Vol. 4, p. 65 (1934).

13. Havell, R. F. and Mc-Vay, T. W., "Purification of some Alabama glass sands," J. Am. Cer. Soc., 18429-31 (1939).

14. Griffiths, "Scientific method in the analysis of sedimentary particles," Journal of Sedimentary Petrology, 37, p. 890 (1967).

15. "Glass sand deposit of the Balijuri Area, Sherpur district, Bangladesh," Records of the Geological Survey of Bangladesh, vol. 3, part. 4, p. 10 (1985).

16. "Glass sand deposit of the Balijuri Area, Sherpur district, Bangladesh," Records of the Geological Survey of Bangladesh, vol. 4, part. 5, p. 14 (1986).

17. Schulman, J. H. and Smith, T. D., "Recent Developments in Mineral Dressing", p-393, Inst. Min. Met., London (1953).

18. Komarova, L. A. and Timo Khin, A.. R., "Beneficiation of quartz materials," Glass Ceram., 44 (5-6) 240 (1987).

19. Lavendar, M. D., "Extraction of glass sand and quality control," Glass Yug, p. 45 (1991).

20. Dasher, J. and Ralston, O. C., "New methods of cleaning glass sands," J. Soc. Glass Tech., 29, 268-72 (1945).

21. Uemura, H. and Tabuchi, H., "Purification of silica sand by magnetic separation," Ceramics Japan, 14(4), p. 327 (1979).

22. Deo, P. N., "Studies on reduction of iron in some Indian silica sands for glass making," Proc. Natl. Sem. On Beneficiation of Non-Metallic Minerals, p. 111, December 15-16 (1989).

23. Lawver, J. G. and Hopstock, D. M., "Wet magnetic separation of weakly magnetic minerals," Mineral Sci. and Engg., 6(3) (1974).

24. Murray, H. M., "Beneficiation of industrial minerals using high intensity magnetic separation," Proc. 3rd Indst. Minerals Intl. Cong., Paris (1978).

25. Anon, "High gradient magnetic separation," Mining Mag. P. 449, October (1977).

26. Gaudin, A. M., Floatation : McGraw Hill, New York, USA, p. 4 (1932).

27. Yamamoto, H., "Removal of iron compounds from silica sand by floatation," Ceramics Japan, 14(4), p. 327 (1979).

28. Scheidhanar, G., "Tests on treating sands chemically," Sillikattechnik, 18 (3), p. 76 (1967).

29. Curtain, L. P. and Cranbury, N. J., "The purification of sands," J. Soc. Glass Technol., 24 (A), p. 198 (1940).

30. Sherlock, A., "Some methods of purification of sand for glass making," ibid, 29, p. 268 (1945).

31. Abou-El-Azam, A. and Badry, Kh. M. El., "Suitability of some Egyptian sands for the production of sheet gkass," Cent. Glass Cer. Res. Inst. Bull., 19 (4), P. 103 (1972).

32. Marian, R. B. V. S., Rathor, B. S., Vyas, D. R. and Basu, M. K., "Evaluation of the Surajdewal sand for the glass industry," Trans. Ind. Cer. SOC., 41(4), P. 110 (1982).

33. Das, S. K., "A critical study of the beneficiation of sand from Utkal mines and minerals, Orissa," Ind. Cer., 31(2), p. 25 (1988).

34. Lavender, M. D., " Industrial sand processing and quality control," Glass Intl., p. 49, June (1986).

35. Adam, F. W., "A new process for the removal of iron oxide from silica sands," J. Soc. Glass Tech., 19, p. 118 (1935).

36. Doyle, P. J., Glass Making Today, p. 3, Portcullis Press, Redhill, UK (1979).

37. British Industrial Sand Ltd, British Patent No. 1 233 177 (1971).

38. Islam, Naweedul, and Sofder, M., Pakistan J. Sci. Res., 17, p. 54 (1965).

39. Hill, C. C., Ceramic Age, 60, p. 24 (1952).

40. Tscuschner, Hanbuch der Glasfabrication, p. 64, 1956.

41. Rhodin, J. G. A., British Patent No. 3495.

42. Bateson, H. M., and Turner, W. E.S., J. Soc. Glass Tech., 23, p. 253 (1939).

43. Damour, E, and Nadel, A., Chem. Et. Ind., 28, p. 283 (1949).

44. Hall, R., and Tunner, W. E. S., J. Soc. Glass Tech., 24, p. 41 (1949).

45. Safdar, M., and Islam, N., "Iron removal from glass making sand," Pak. J. Soc. Res., Vol 17, p. 54, Jan (1965).

46. Knapp, O., "The removal of iron from sand," J. Soc. Glass Tech., 17(A), p.141 (1933).

47. Ismail,. M., "Stratigraphical position of Bogra limestone of the platform area of Bangladesh," Proc. 4th Annual Conf. Bang. Geol. Soc., p. 15, Nov, (1978).

48. Rogers, A. F., "Introduction to the study of minerals," 3rd ed, McGraw-Hill Book Company, New York, p. 15 (1937).

49. Cullity, B. D., Elements of x-ray diffraction, 2nd edition, p. 398, (1978).

50. Carver, R. E., Procedures in Sedimentary Petrology, John Wiley and Sons Inc., U.S.A. p. 653, (1971).

51. Sherlock, A., "Some observation on the sampling and testing of sands for making colourless glass," J. Soc. Glass Tech., 25, p. 86 (1941).

Figures

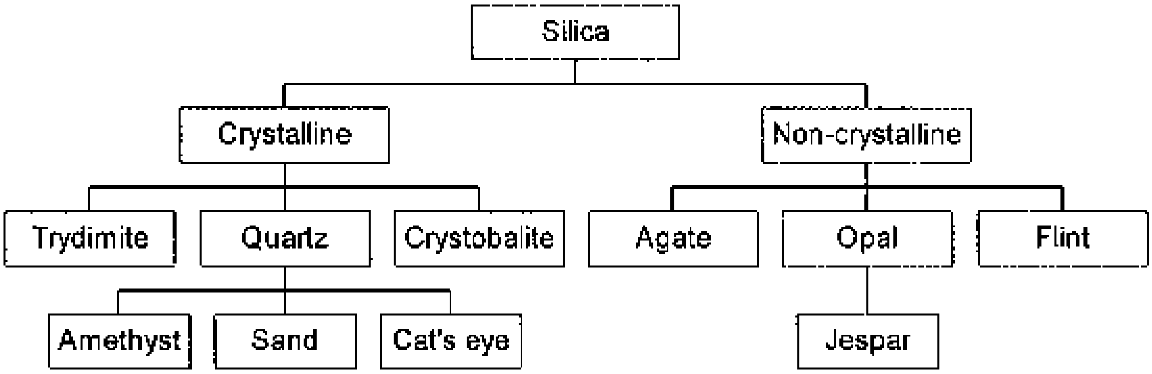
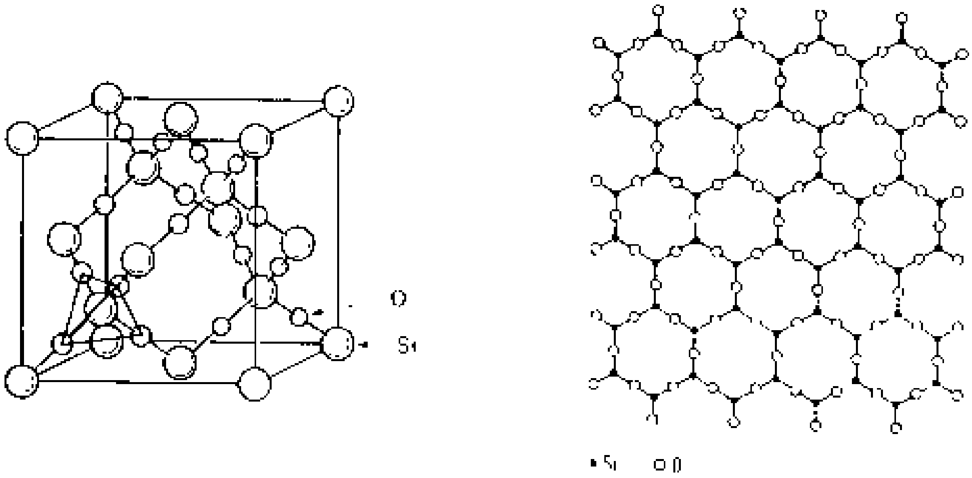


Fig. 2.1 Flowchart showing the various forms of silica.



(a) (b)

Fig. 2.2 (a) The cubic structure of silica; (b) Two-dimensional diagram of an ordered SiO_2 network.

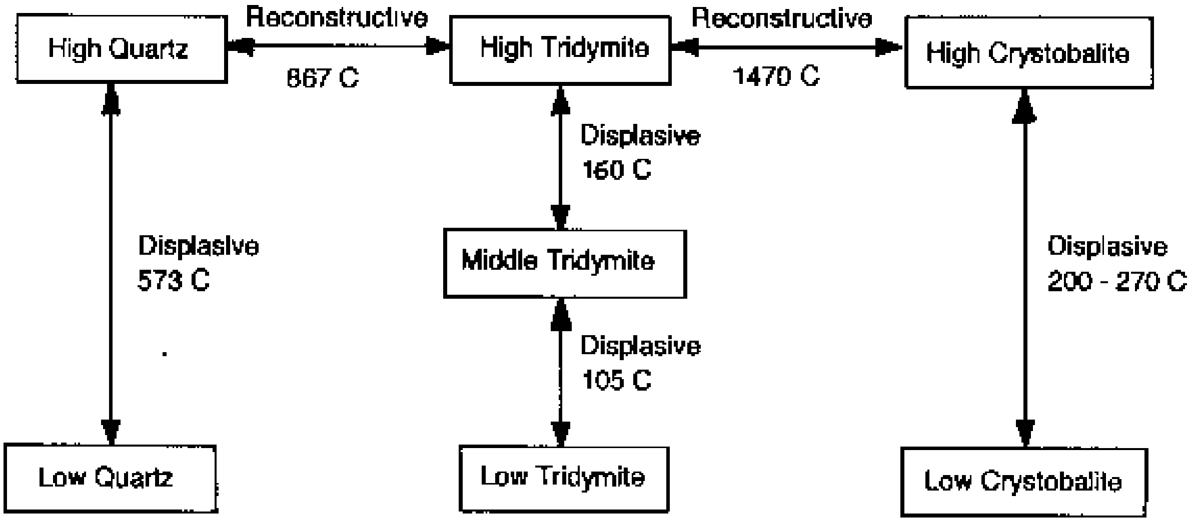


Fig. 2.3 Silica and its polymorphs.

Fig 3.1(a) Photomicrograph of as-received sand showing silica grains. Plane light. (X 100).

Fig 3.1(b) Photomicrograph of water washed sand showing silica grains. Plane light. (X 100).

Fig 3.2(a) Photomicrograph of as-received sand heated to 900 °C for 30 min. showing the presence of reddish tint over the silica grains. Plain light. (X 100).

Fig 3.2(b) Photomicrograph of water washed sand heated at 900 °C for 30 min. showing the presence of reddish tint over the silica grains. Plane light. (X 100).

Fig 3.3 Photomicrograph showing the presence of biotite. Plane light. (X 100).

Fig 3.4 Photomicrograph showing the presence of Muscovite. Plane light. (X 100).

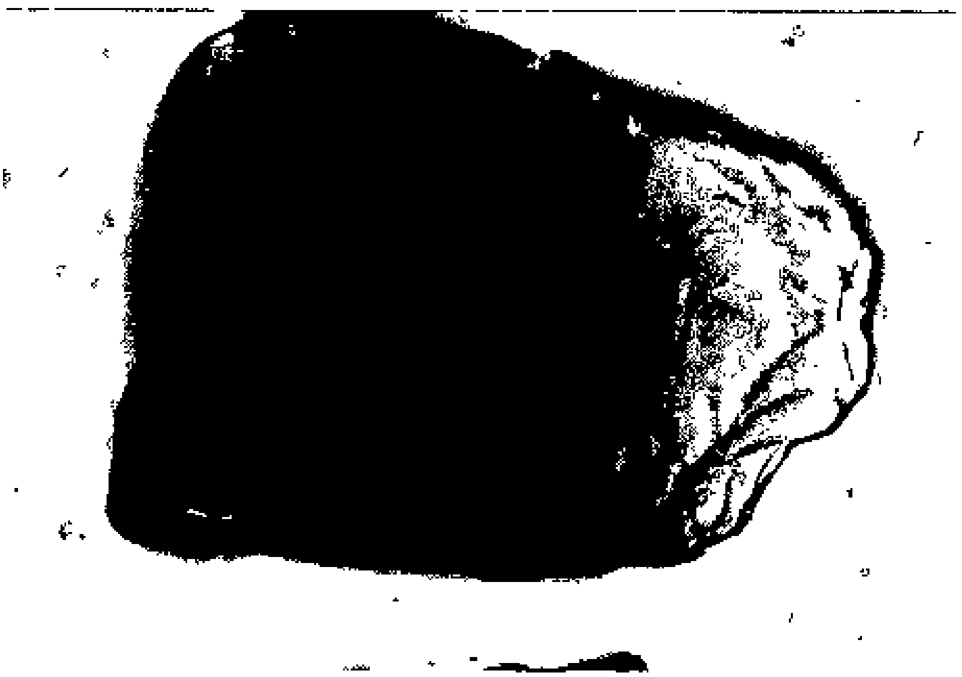


Fig 3.5 Photomicrograph showing the presence of Tourmaline. Plane light. (X 100)

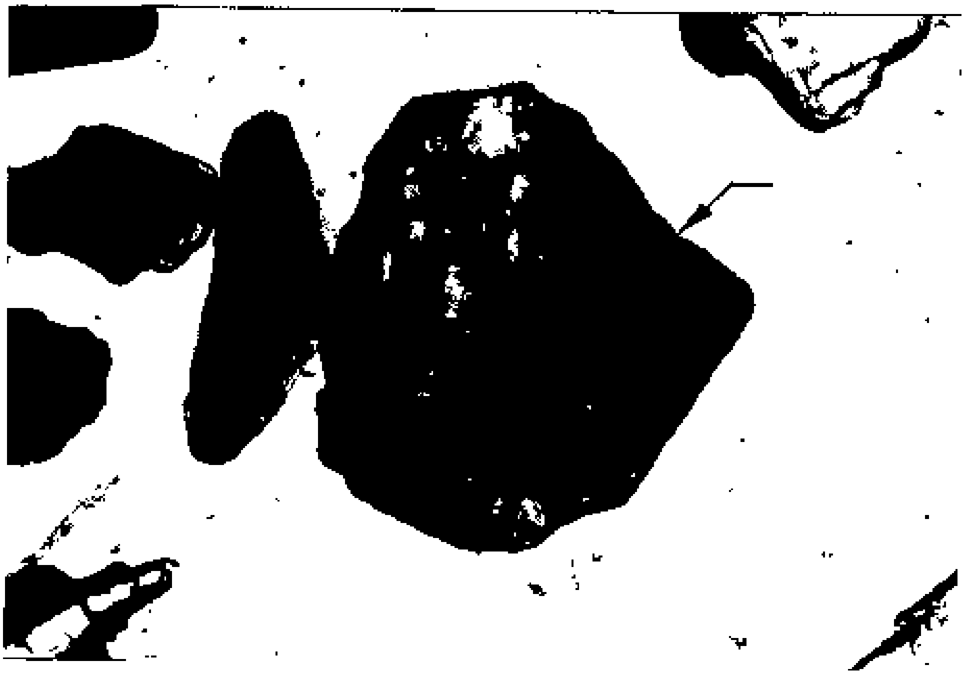


Fig 3.6 Photomicrograph showing the presence of Staurolite. Plane light. (X 100).

Fig 3.7 Photomicrograph showing the presence of Tremolite. Plane light. (X 100).

Fig 3.8 Photomicrograph showing the presence of Garnet. Plane light. (X 100)

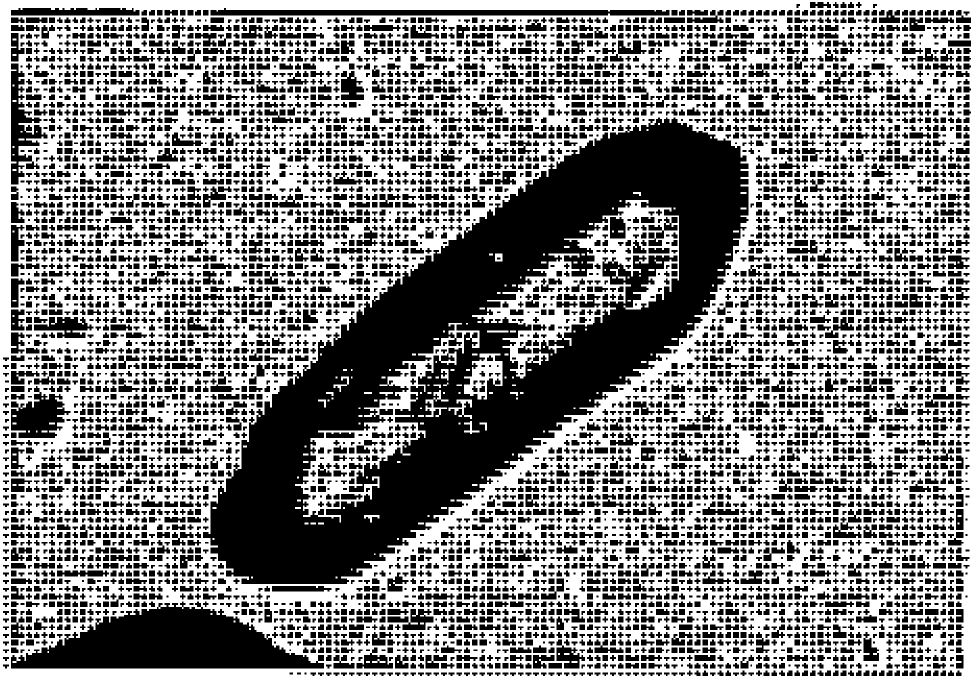


Fig 3.9 **Photomicrograph showing the presence of Zircon. Plane light. (X 100).**

Fig 3.10 **Photomicrograph showing the presence of Kyanite. Plane light. (X 100).**

Fig 3.11 Photomicrograph showing the presence of Rutile.
Plane light. (X 100).

Fig 3.12 Photomicrograph showing the presence of Hornblende.
Plane light. (X 100).

Fig 3.13 Photomicrograph showing the presence of Actinolite. Plane light. (X 100).

Fig 3.14 Photomicrograph showing the presence of Opague minerals along with zircon. Plane light. (X 100).

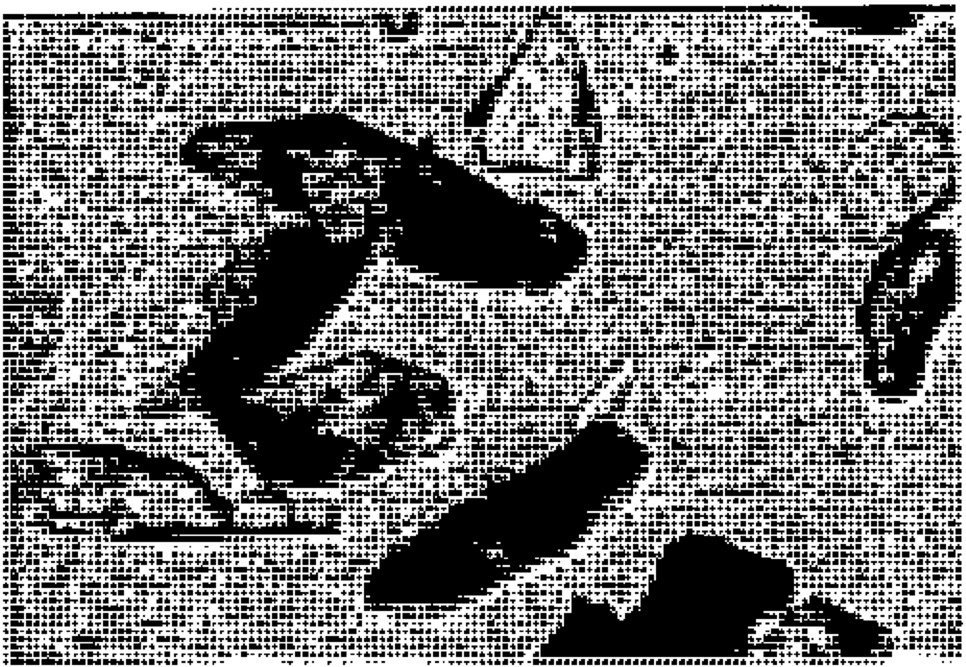


Fig 3.15 Photomicrograph showing the presence of sillimanite. Plane light. (X 100).

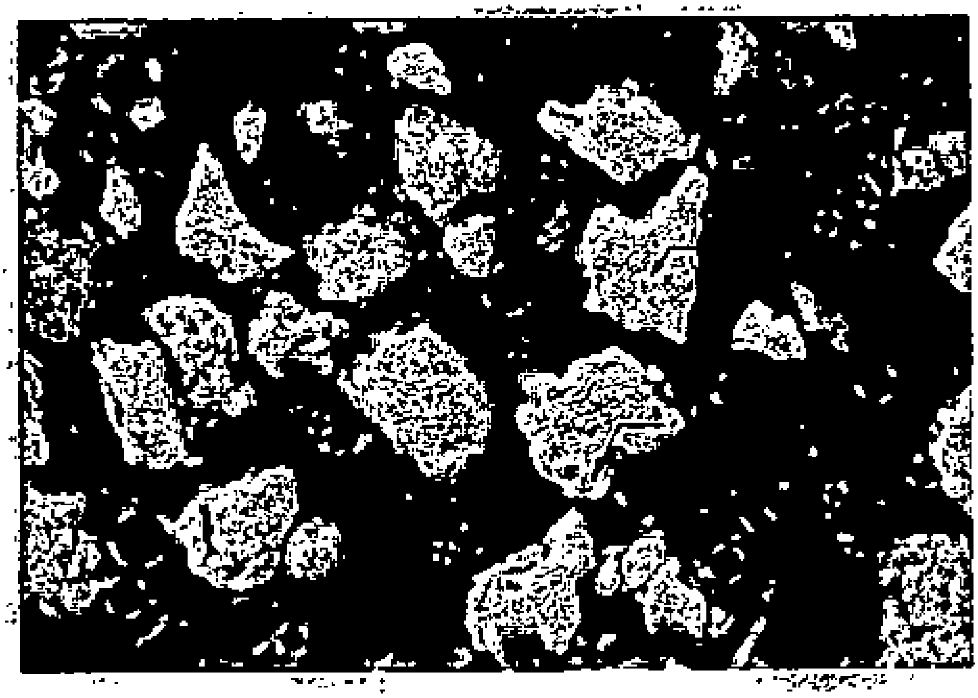


Fig 3.16 Photomicrograph of thin section slide of water washed sand showing mainly quartz grains. Plane light. (X 100).

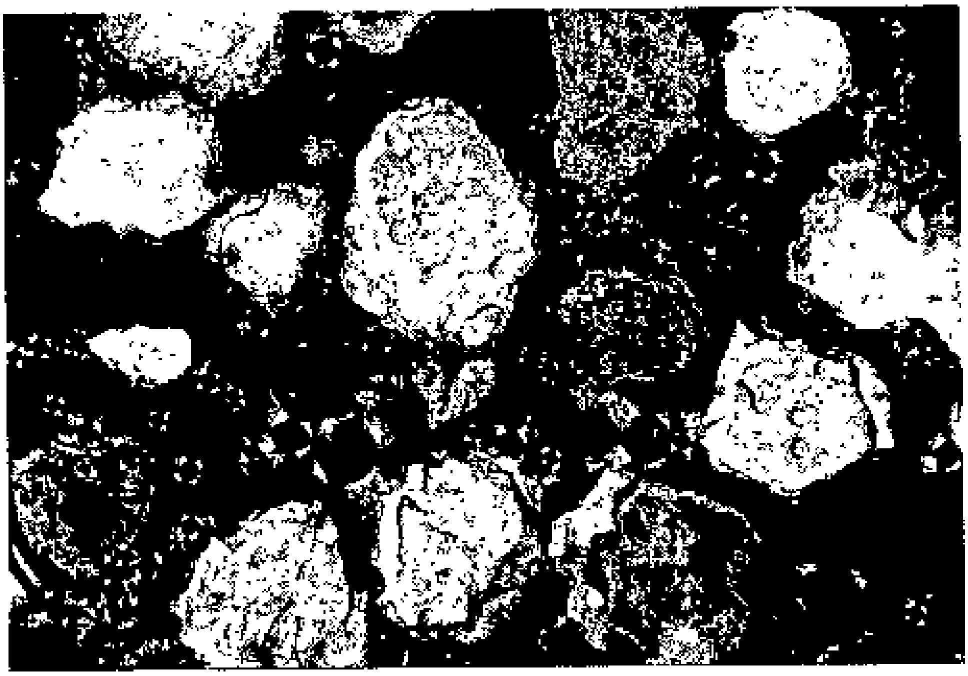


Fig 3.17 Photomicrograph of thin section slide of water washed sand showing quartz along with other impurity minerals. Plane light. (X 100).

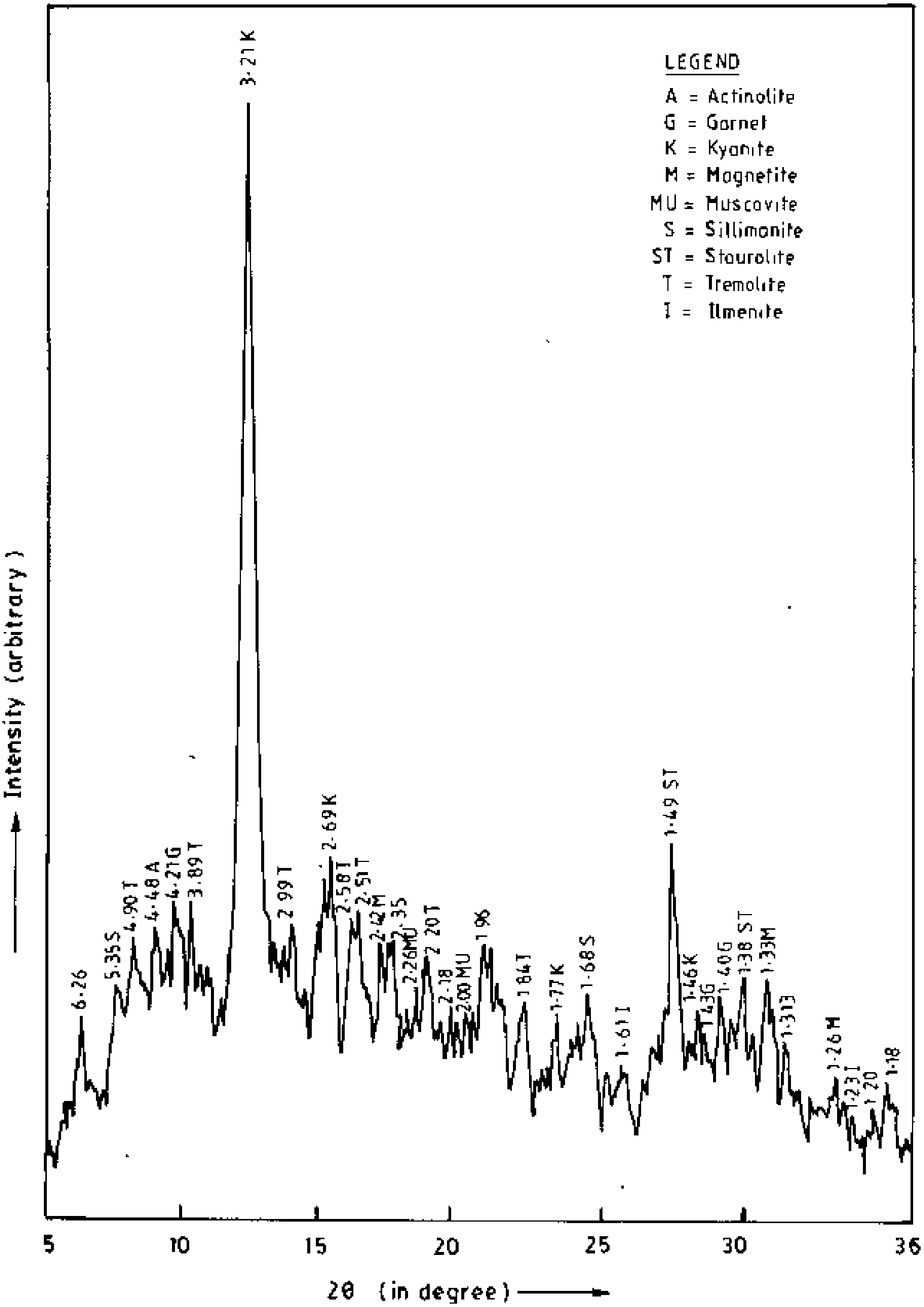


Fig 3.18 X-ray diffraction pattern of heavy minerals in the sand deposit.

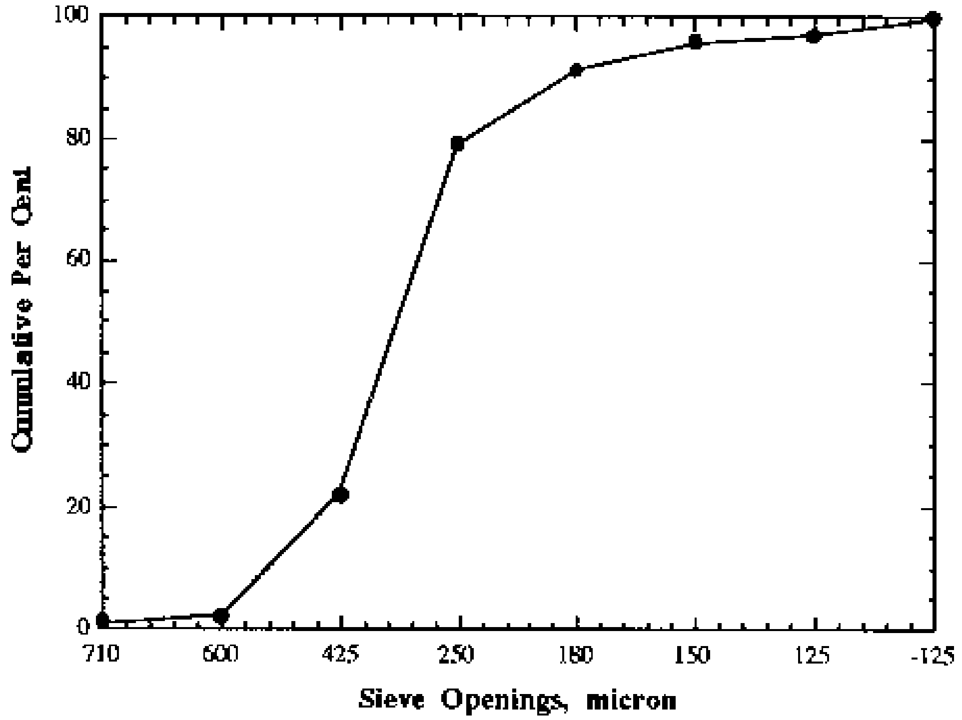


Fig. 3.19 Graph showing the cumulative percentage of sieve fraction of as-received sand.

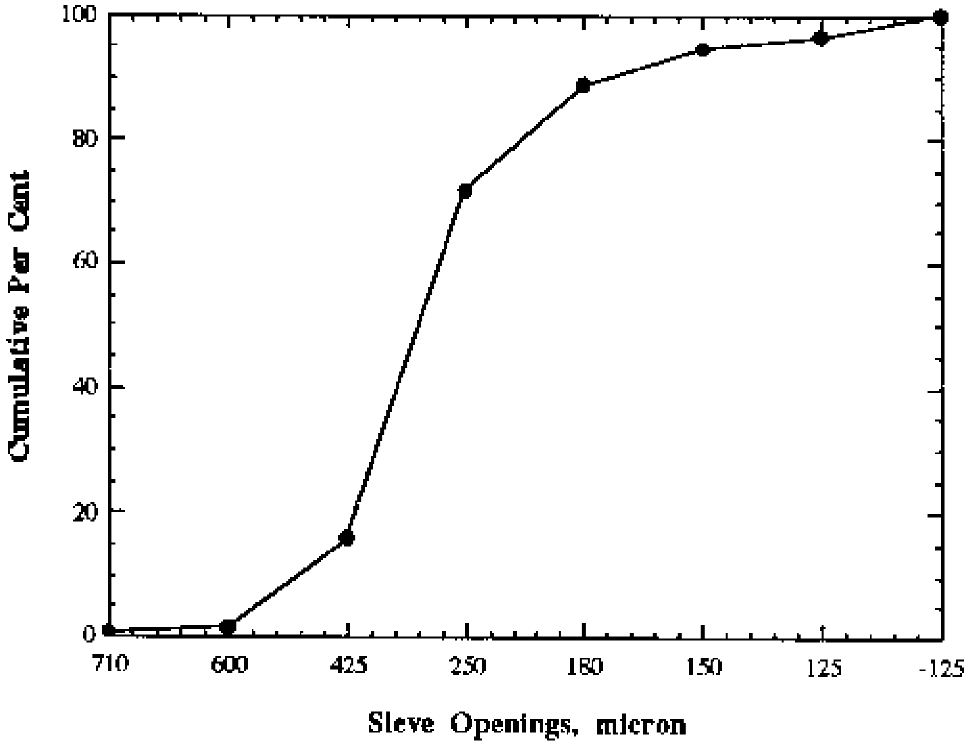


Fig. 3.20 Graph showing the cumulative percentage of sieve fraction of water washed sand.

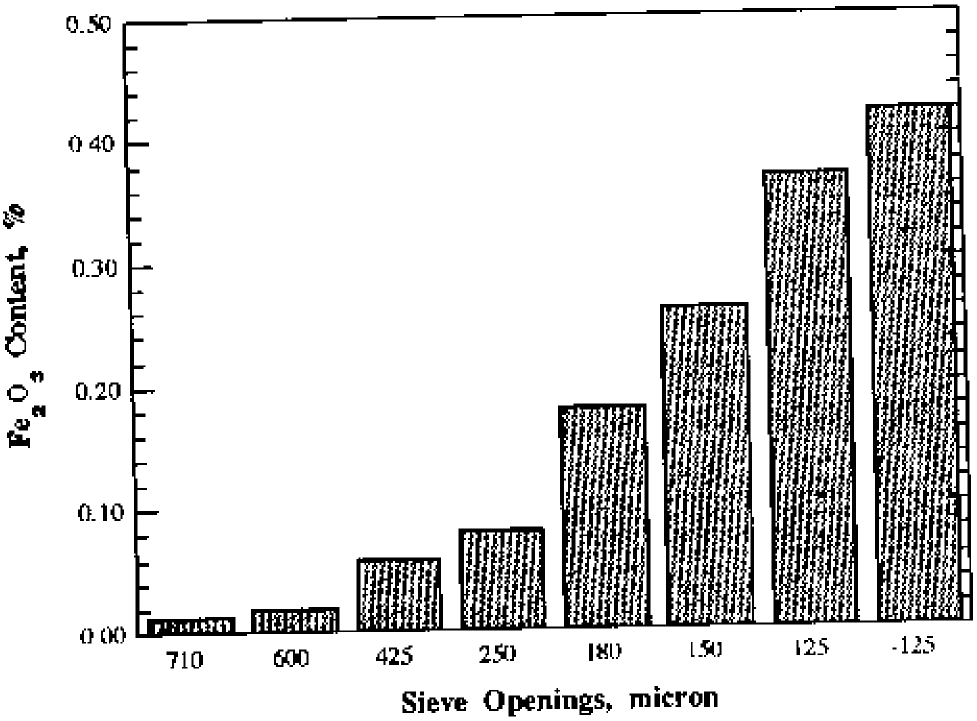


Fig. 3.21 Graph showing the percentage of iron oxide content in each sieve fraction of water washed sand.

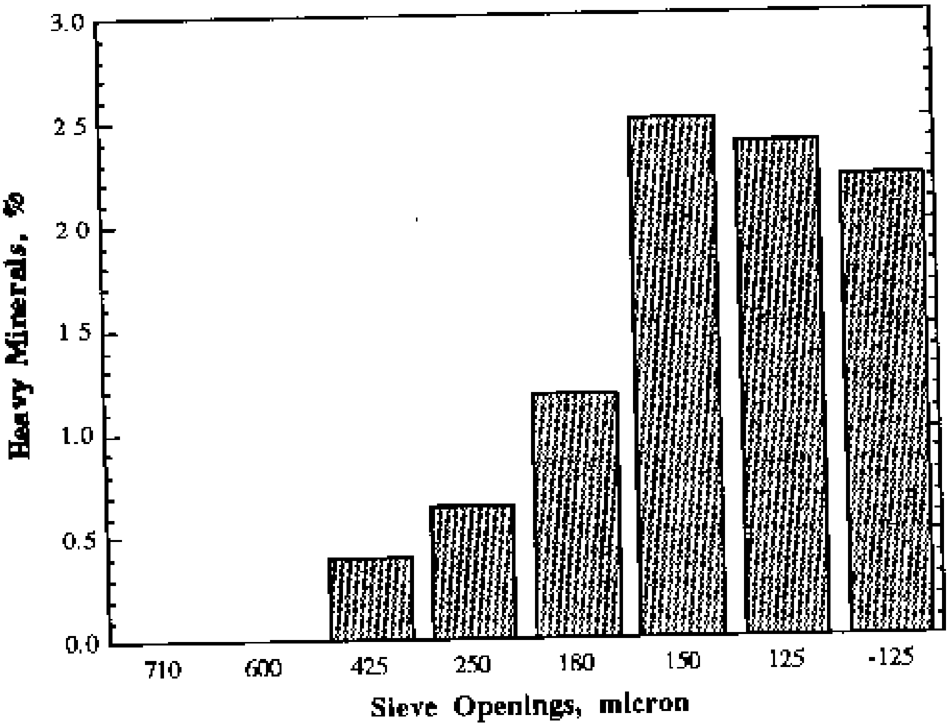


Fig. 3.22 Graph showing the percentage of heavy minerals present in each sieve fraction of water washed sand.

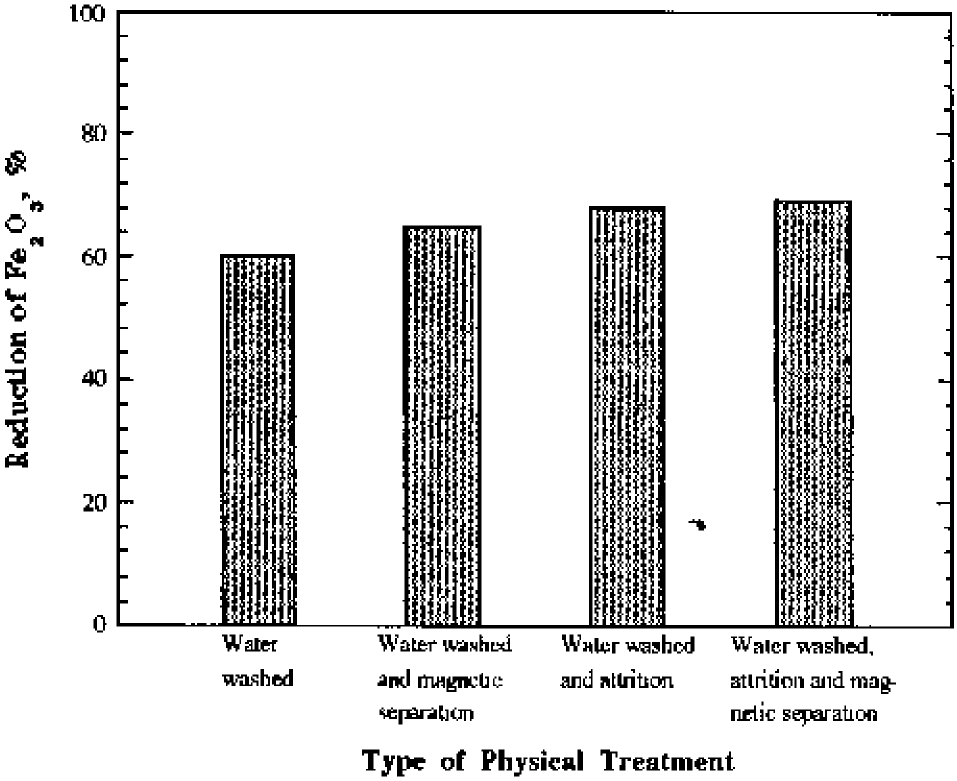


Fig. 3.23 Graph showing the percentage of iron reduction in different physically treated sand.

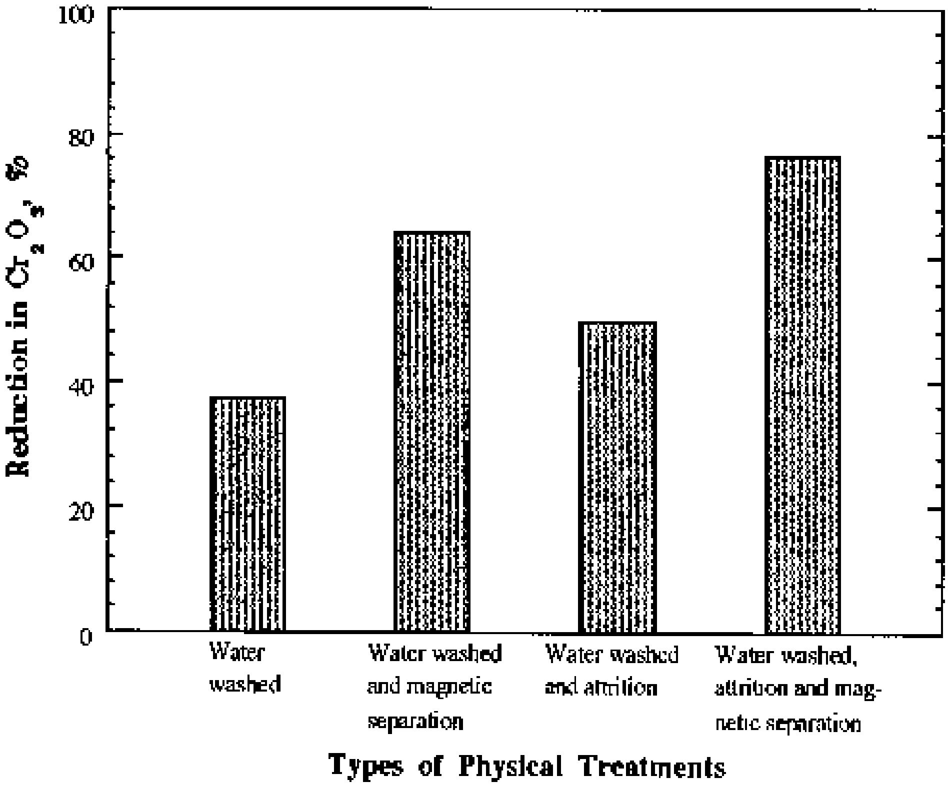


Fig. 3.24 Graph showing the percentage of chromium reduction in different physically treated sand.

Legends

1 Hydrochloric acid process
2 Sulfuric acid wash
3 Sodium hydroxide wash
4 Diothionite method
5 Sulfide process
6 Adam's process
7 Sherlock's process
8 Sodium chloride process
9 Sodium carbonate process
10 Ferrous sulfate and sulfuric acid combined process
11 4 % sodium hydroxide

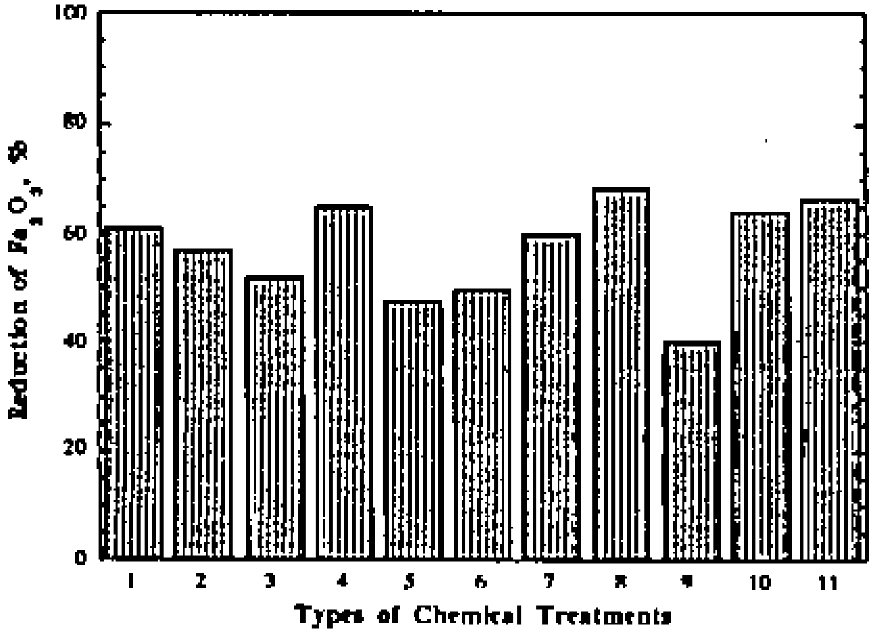


Fig. 3.25 Graph showing the percentage of iron reduction in different chemically treated water washed sands.

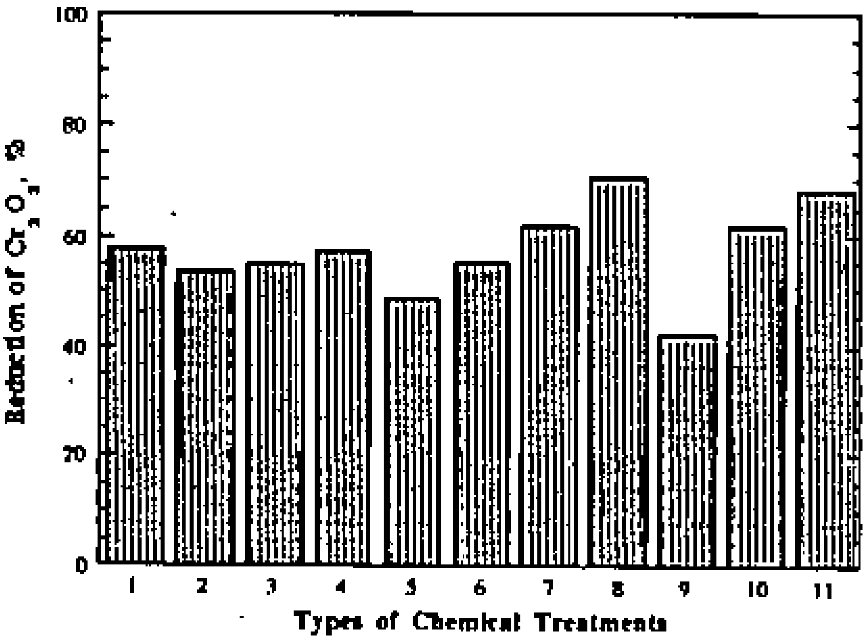


Fig. 3.26 Graph showing the percentage of chromium reduction in different chemically treated water washed sands.

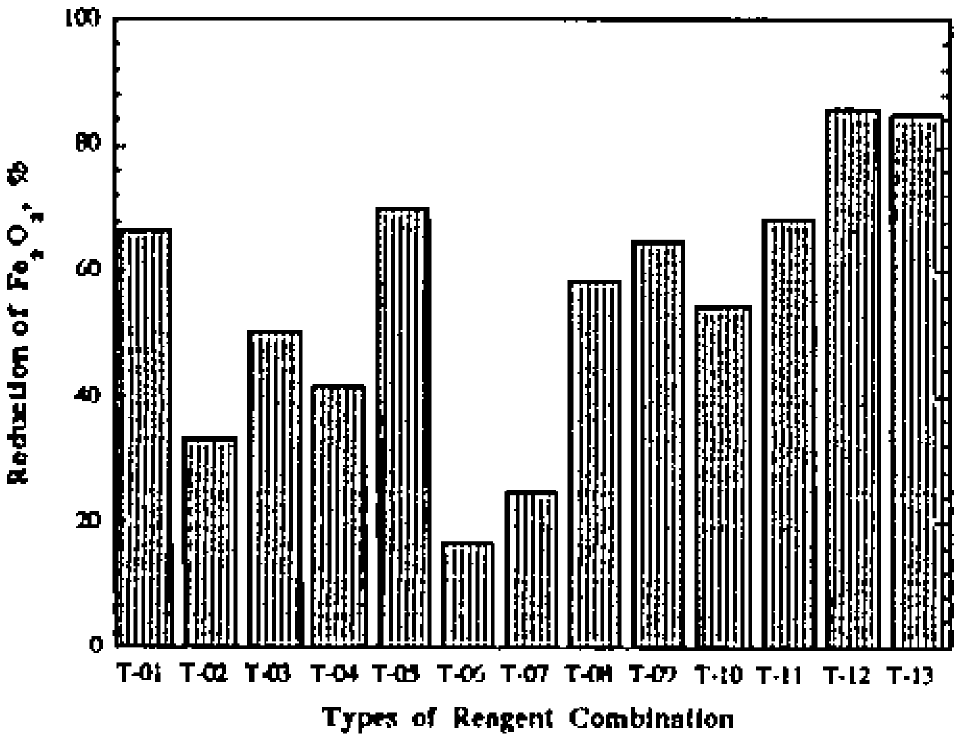


Fig. 3.27 Graph showing the percentage of iron reduction in froth flotation process using different reagent groups.

Tables

Table 1.1 The impurities likely to be present in the sand deposit.

Name of the minerals	Chemical formula	Specific gravity (gms/cc)
Biotite	$K_2O.4(Fe,Mg).2(Al,Fe)_2O.6SiO_2.H_2O$	2.79 - 3.16
Hornblende	$Ca_3Na_2\ (Mg, Fe)_8\ (Al, Fe)_4\ Si_{14}O_{44}\ (OH)_4$	3.00- 3.30
Kyanite	$Al_2O_3.SiO_2$	3.60- 3.68
Actinolite	$2CaO.5(Mg,Fe)O.8SiO_2.H_2O$	3.00 - 3.30
Muscovite	$(K,Na)_2O.3Al_2O_3.\ 6SiO_2.H_2O$	2.76 - 3.00
Rutile	TiO_2	4.18 - 4.25
staurolite	$2FeO.5\ Al_2O_3.4SiO_2.H_2O$	3.65 - 3.77
Tourmaline	$Na_2O.8FeO.8Al_2O_3.4B_2O_5.16SiO_2.5H_2O$	2.98 - 3.20
Tremolite	$2CaO.5MgO.8SiO_2.H_2O$	3.00- 3.30
Magnetite	Fe_3O_4	5.10 - 5.18
Hematite	Fe_2O_3	5.20
Ilmenite	$FeO.TiO_2$	4.50 - 5.00
Zircon	$ZrO_2.SiO_2$	4.50 - 4.70
Garnet	$3RO.R_2O_3.3SiO_2$; R = Ca, Mg, Fe, Mn ; RO= Al_2O_3, Fe_2O_3, Cr_2O_3	3.00 - 4.30
Sillimanite	$Al_2O_3.\ SiO_2$	3.23 - 3.25

Table 2.1 Physical and mineralogical properties of quartz.

Composition	SiO_2
Hardness (Mohs scale)	7
Density (g/cc)	2.66
Colour	Usually colourless or white but can occur in practically any shade
Streak	White
Luster	Vitreous in macrocrystalline varieties, often waxy or dull in cryptocrystalline varieties
Optical properties	Piezoelectric and pyroelectric
Solubility	Hydrofluoric acid

Table 2.2 Limits for chemical composition of sand for making colourless glasses.

Grade of Sand	Type of glasses	SiO_2 content (min.)	Iron oxide content (as Fe_2O_3)	Chromium oxide content (as Cr_2O_3)	Titanium oxide content (as TiO_2)
A	Fine grade optical glassware	99.5	0.008	0.0002	0.03
B	High grade domestic and decorative glassware	99.5	0.013	0.0002	no limit
C	General colourless glassware, including containers.	98.5	0.03	0.0005	no limit

Table 2.3 Terminology and class intervals of sand for grade scales recommended by the American Ceramic Society.

Type	US Standard sieve mesh	Millimeters	Phi (φ) units	Wentworth size class
GRAVEL	-	4096.00	-12.00	Boulder
	-	1024.00	-10.00	
	-	256.00	-8.00	Cobble and Pebble
	-	64.00	-6.00	
	-	16.00	-4.00	
	5	4.00	-2.00	Granule
	6	3.36	-1.75	
	7	2.83	-1.50	
	8	2.38	-1.25	
SAND	10	2.00	-1.00	Very coarse sand
	12	1.68	-0.75	
	14	1.44	-0.50	
	16	1.19	-0.25	
	18	1.00	0.00	Coarse sand
	20	0.84	0.25	
	25	0.71	0.50	
	30	0.59	0.75	
	35	0.50	1.00	Medium sand
	40	0.42	1.25	
	45	0.35	1.50	
	50	0.30	1.75	
	60	0.25	2.00	Fine sand
	70	0.210	2.25	
	80	0.177	2.50	
	100	0.149	2.75	
	120	0.125	3.00	Very fine sand
	140	0.105	3.25	
	170	0.088	3.50	
	200	0.074	3.75	
SILT	230	0.0625	4.00	Coarse silt
	270	0.053	4.25	
	325	0.044	4.50	
	-	0.037	4.75	
	-	0.031	5.00	
	-	0.0156	6.00	Medium silt
	-	0.0078	7.00	Fine silt
	-	0.0039	8.00	Very fine silt
MUD	-	0.0020	9.00	Clay
	-	0.00098	10.00	
	-	0.00049	11.00	
	-	0.00024	12.00	
	-	0.00012	13.00	
	-	0.00006	14.00	

Table 2.4 Range of applicability of different techniques of size analysis

Techniques	Range of applicability (phi units)
Direct measurement	-12.0 to 2.00
Sieving	-4.00 to 4.75
Sedimentation tube	-1.25 to 4.00
Optical microscope	-1.25 to 4.75
Pipette or hydrometer	4.00 to 9.00
Electron microscope	8.00 to higher

Table 2.5 The specification for size grading of glass sands.

Sieve number	Sieve openings (mm)	Cumulative %
Residue on no. 16 sieve	1.60	Nil
Residue on no. 25 sieve	1.02	2 % max
Residue on no. 36 sieve	0.71	10 % max
Material passing no. 100 sieve	0.25	10 % max
Material passing no. 120 sieve	0.21	15 % max

Table 2.6 List of different chemicals used in froth floatation treatment.

Collectors	Froth stabilizers	Aiders	Sand depressors
Soap	Paraffin oil	Sodium hexa-metaphosphate	Sulfuric acid
Amyl alcohol	Lead nitrate		Sodium silicate
Turpentine			Soda ash
Ecalyptus oil			Tannic acid
Camphor			Gelatine
Cresol (ortho, para)			
Phenol			
Oleic acid			
Sodium-lauryl sulphonate			
DDBSA			
Petrolium sulphonate			
Pine oil etc.			

Table 3.1 Chemical analyses of as-received and water washed sand.

Constituents analysed	Per cent of each constituents	
	As-received	Water washed
SiO_2	96.54	97.62
Fe_2O_3	0.30	0.12
Cr_2O_3	0.67×10^{-2}	0.42×10^{-2}
Al_2O_3	1.47	1.16
CaO	0.36	0.24
MgO	0.28	0.18
Na_2O	0.12	0.06
K_2O	0.18	0.08
LOI (loss on ignition)	0.74	0.54

Table 3.2 Sieve analysis of as-received sand deposit.

Sieve openings µm	Amount of sand retained in each sieve fraction (Total weight of sample = 100 gm)	
	gm	Cumulative Per Cent
710	0.95	0.95
600	1.16	2.01
425	19.71	21.72
250	55.33	79.05
180	12.24	91.29
150	4.39	95.68
125	1.20	96.88
-125	2.82	99.70

Table 3.3 Sieve analysis of washed noapara sand deposit.

Sieve openings	Amount of sand retained in each sieve fraction (Total weight of sample = 100 gm)	
µm	gm	Cumulative Per Cent
710	0.60	0.60
600	0.82	1.42
425	14.44	15.86
250	55.77	71.63
180	17.00	88.63
150	5.97	94.60
125	1.85	96.45
-125	3.50	99.95

Table3.4 Percentage of Fe_2O_3 content of individual sieve fraction in water washed sand.

Sieve openings	Wt. of each fractions	Fe_2O_3 content in each sieve fraction	
µm	gm	mg	%
710	0.60	0.07	0.012
600	0.82	0.16	0.020
425	14.44	8.70	0.060
250	55.77	44.60	0.080
180	17.00	30.60	0.180
150	5.97	15.50	0.260
125	1.85	6.83	0.369
-125	3.50	14.70	0.420

Table 3.5 Percentage of Fe_2O_3 in sand after different physical separation processes.

Treatment used	% Fe_2O_3	% Reduction
As-received	0.300	-
Water washed	0.120	60.00
Water wash and Magnetic separation	0.105	65.00
Water wash and Attrition	0.096	68.00
Water wash, Attrition and Magnetic separation	0.092	69.33

Table 3.6 Percentage of Cr_2O_3 in sand after different physical separation processes.

Treatment used	% Cr_2O_3	% Reduction
As-received	0.0067	-
Water washed	0.0042	37.32
Water wash and Magnetic separation	0.00242	63.88
Water wash and Attrition	0.00547	49.85
Water wash, Attrition and Magnetic separation	0.00158	76.42

Table 3.7 Contents of heavy minerals in each sieve fraction of water washed sand.

Sieve fraction µm	Weight of sand taken for each fraction gm	Weight of heavy minerals mg	Heavy minerals in each fractions Per Cent	Heavy minerals in 100 gm washed sample Per Cent
710	0.60	0.0000	0.00	0.00
600	0.82	0.0000	0.00	0.00
425	14.44	0.0582	0.40	0.06
250	55.77	0.3614	0.65	0.36
180	17.00	0.2020	1.17	0.20
150	5.97	0.1318	2.50	0.13
125	1.85	0.0442	2.38	0.04
-125	3.50	0.0773	2.22	0.08

Table 3.8 Percentages of magnetics and non-magnetics in heavy minerals fraction of water washed sand.

Weight of sample	Weight of heavy minerals	Magnetic fraction		Non- magnetic fraction	
		in heavy mineral fraction	in total water washed sand	in heavy mineral fraction	in total water washed sand
gm	gm	Per Cent	Per Cent	Per Cent	Per Cent
20.00	0.175	10.53	0.0921	89.47	0.7828

Table 3.9 Grain counting analysis showing the percentage of each minerals present in the heavy minerals fraction of the sand.

Type of Minerals	Percentage of each Mineral
Biotite	2.80
Kyanite	22.60
Muscovite	2.80
Rutile	1.90
Staurolite	8.60
Tourmaline	5.60
Actinolite + Tremolite + Sillimanite	17.60
Zircon	0.93
Hornblende	3.87
Garnet	3.60
Opaque minerals	29.10
Altered minerals	2.30

Table 3.10 Grain counting analysis showing the percentage of each minerals present in the magnetic fraction of the sand.

Type of minerals	Percentage of each mineral			
	Sieve opening 150 μm	Sieve opening 180 μm	Sieve opening 250 μm	Sieve opening 425 μm
Magnetite	98.17	90.72	87.77	82.08
Ilmenite	1.83	8.25	12.23	15.62
Hematite	0.00	1.03	0.00	2.30

Table 3.11 Grain counting analysis showing the percentage of each minerals present in the non-magnetic fraction of the sand.

Type of minerals	Percentage of each mineral			
	Sieve opening 150 µm	Sieve opening 180 µm	Sieve opening 250 µm	Sieve opening 425 µm
Biotite	2.32	5.02	4.25	4.00
Hornblende	5.82	1.59	1.06	0.00
Kyanite	23.25	15.87	26.23	6.00
Muscovite	4.65	0.00	3.19	2.00
Rutile	0.00	4.50	3.20	4.00
Staurolite	5.81	21.23	5.32	6.00
Garnet	5.25	6.32	1.50	2.50
Tourmaline	8.70	9.55	10.20	6.00
Tremolite + Actinolite + Sillimanite	13.10	26.32	27.56	49.50
Opaque	27.91	7.35	15.26	18.00
Altered minerals	3.18	2.25	2.23	2.00

Table 3.12 Comparison of the observed peak positions and the respective standard peak positions.

Observed peak positions (2θ)	Observed d-spacing Å	Standard d-spacings Å	Plane (hkl)	Type of Minerals
7.62	5.35	5.36	110	Sillimanite
8.32	4.90	4.87	111	Tremolite
9.09	4.48	4.46	021	Actinolite
9.68	4.21	4.24	220	Garnet
10.48	3.89	3.87	131	Tremolite
12.71	3.21	3.18	021, 111	Kyanite
13.65	2.99	3.03	311, 241	Tremolite
15.18	2.69	2.694	211	Kyanite
15.70	2.58	2.590	112	Tremolite
16.28	2.51	2.527	002	Tremolite
16.88	2.42	2.4243	130	Magnetite
18.17	2.26	2.254	200	Muscovite
18.59	2.20	2.181	441	Tremolite
19.80	2.00	2.056	206	Muscovite
22.77	1.84	1.864	460, 191	Tremolite
23.17	1.77	1.764	311, 411	Kyanite
24.42	1.68	1.682	1114	Sillimanite
25.50	1.61	1.6354	018	Ilmenite
27.59	1.49	1.478	2102	Staurolite
28.18	1.46	1.452	350	Kyanite
28.77	1.43	1.432	632	Garnet
29.40	1.40	1.414	822	Garnet
29.84	1.38	1.385	0120	Staurolite
30.99	1.33	1.3277	1010	Magnetite
33.03	1.26	1.2659	622	Magnetite
33.58	1.23	1.2453	306	Ilmenite

Table 3.13 Semi-quantitative analysis of average percentage of heavy minerals by X-ray diffratometry.

Type of mineral	d-spacing	height of the peaks h cm	half width of the peak ½ w cm	h/(½ w)	Average Percentage of minerals
Kyanite	3.21	15.40	0.6875	22.40	31.79
Tremolite	3.89	1.80	0.175	10.28	14.58
Sillimanite	1.68	2.30	0.55	4.18	5.93
Actinolite	4.48	1.40	0.25	5.60	7.95
Garnet	4.21	1.60	0.30	5.33	7.56
Staurolite	1.49	3.85	0.525	7.33	10.40
Ilmenite	1.23	0.80	0.25	3.20	4.54
Magnetite	1.33	1.1	0.15	7.91	10.41
Muscovite	2.00	0.60	0.125	4.80	6.81

Table 3.14 Percentage of Fe_2O_3 content in various chemical treated sand samples.

Treatment used	Fe_2O_3 %	Reduction %
Water wash and Magnetic separation	0.105	-
Hydrochloric acid process	0.040	61.14
Sulphuric acid wash	0.045	57.14
Sodium hydroxide method	0.050	52.23
Dithionite method	0.037	64.76
Sulphite process	0.055	47.61
Adam's process	0.053	49.52
Sherlock's process	0.042	60.00
Sodium chloride process	0.033	68.57
sodium carbonate process	0.063	40.10
Ferrous sulphate and sulfuric acid combined process	0.038	63.80
4 % sodium hydroxide	0.035	66.67

Table 3.15 Percentage of Cr_2O_3 in various chemical treated sand samples.

Treatment used	Cr_2O_3 %	Reduction %
Water wash and Magnetic separation	0.00242	-
Hydrochloric acid process	0.00101	58.13
Sulphuric acid wash	0.00128	53.41
Sodium hydroxide method	0.00108	55.20
Dithionite method	0.00103	57.43
Sulphite process	0.00124	48.78
Adam's process	0.00107	55.62
Sherlock's process	0.00092	62.14
Sodium chloride process	0.00007	70.95
sodium carbonate process	0.00140	42.15
Ferrous sulphate and sulfuric acid combined process	0.00092	61.89
4 % sodium hydroxide	0.00077	68.36

Table 3.17 Percentage of Fe_2O_3 content of sand after froth floatation.

Froth- floatation Types	% of Fe_2O_3	% of reduction
T-1	0.04	66.66
T-2	0.08	33.33
T-3	0.06	50.00
T-4	0.07	41.66
T-5	0.35	70.00
T-6	0.10	16.66
T-7	0.09	25.00
T-8	0.05	58.33
T-9	0.04	65.00
T-10	0.06	54.46
T-11	0.04	68.33
T-12	0.032	86.00
T13	0.034	85.00

Table 3.18 Percentage of Fe_2O_3 and Cr_2O_3 of sand after froth flotation (only the best two).

Treatment used	Fe_2O_3 %	Reduction %	Cr_2O_3 %	Reduction %
Water washing and Magnetic separation	0.105	-	0.0024	-
Froth floatation (Batch-12)	0.032	69.52	0.0012	50.41
Froth floatation (Batch-13)	0.034	67.61	0.0015	38.02

Table 3.19 Moisture content and clay content of the sand

Wt. of sample	After heating above 100°c	After removal of clay	% of clay	% of moisture
100.004	95.353	91.102	4.46	4.65