

Chemical and Sedimentological Characterization of Moulvibazar Silica Deposit of Bangladesh as Standard Sand

Md. Faruk Hossain¹, Md. Ashraful Islam Molla¹, Shah Md. Masum¹, Ashequl Alam Rana¹, A. F. M. Saiful Amin², Raziya Sultana Chowdhury³, Salma Sultana⁴ and Mohammad Mainul Karim^{1,*}

¹Department of Applied Chemistry and Chemical Engineering, University of Dhaka, Dhaka-1000, Bangladesh.

²Department of Civil Engineering, Bangladesh University of Engineering and Technology, Dhaka-1000, Bangladesh.

³Department of Civil Engineering, Ahsanullah University of Science and Technology, Dhaka-1208, Bangladesh.

⁴Nuclear and Radiation Chemistry Division, INST, Bangladesh Atomic Energy Commission, Dhaka, Bangladesh.

^{1,*}Tel.: +88 029661920-73 (7404); fax: +88 028615583

^{1,*}moinulcd@yahoo.com

Abstract-- Standard sand is used for conducting the compressive strength test of hydraulic cement mortars. American society for testing and materials (ASTM) recommends Ottawa Standard Sand (OSS). Bangladesh has quiet a rich sand deposits but in the scenario of not having standard sand, OSS is imported from abroad for using in different tests in Bangladesh. In this work, a very strong effort has been made to standardize the sand for Bangladesh using its own available deposits and thus to eliminate the unwanted expenditure of foreign currencies. Hence, we selected the Moulvibazar silica deposit (situated at the piedmont of Dupi Tila formation) with the maximum probability of having high silica content by studying the geological formation of Bangladesh. OSS contains about 99.7% of silica and has 'rounded' shape which enables non-reactivity of sand with cement constituents during the testing of hydraulic cement mortars. XRD analysis of the sand sample shows that it contains about 96.87% of silica after water wash and 91.17% of silica after acid wash. SEM images were used to measure the roundness of the sand samples. The roundness of the water wash and acid (0.1N HCl) wash sand samples (according to Wadde scale) were found to be 0.28 and 0.27 respectively. Both sand samples fall into 'sub-rounded' class of roundness grade (of Pettijohn gradation). An attempt was taken to observe the change of roundness by mechanical abrasion of the sample. It was observed that the roundness was decreased because of the growth of secondary corners. DSC isotherm analysis was carried out at three different temperatures (30^o, 45^o and 60^o C) to investigate the reactivity of sand samples. Neglecting the unaffected reactivity, instead, considering the high silica content and subrounded shape of the sample, the sand of Moulvibazar deposit could be recommended for using as standard sand for testing hydraulic cement mortars.

Index Term-- Moulvibazar; X-ray diffraction; scanning electron microscopy; differential scanning calorimetry; roundness.

Highlights

High silica sand can be used as standard sand for testing hydraulic cement mortars.

Moulvibazar sand contains 96.87% of silica after water wash and 91.17% of silica after acid wash. Moulvibazar sand grains belong to 'sub-rounded' (0.27-0.28) class of roundness grade.

DSC isotherm analysis was carried out to investigate the reactivity of sand samples.

Acid wash sample react with cement's constituents more intensively than the water wash sample.

1. INTRODUCTION

In the past there was no need to test cements in Bangladesh, because all of the cement was imported from other countries. But in 1995, Hyundai Construction Company started to grind clinker in Bangladesh during the construction of Jamuna Multipurpose Bridge. After that, a large number of cement clinkers grinding industries was established in this country and thus the importance of the testing of cement has been created for the quality assurance. Bangladesh import ASTM recommended Ottawa Standard Sand (OSS) from the USA for testing cement mortar and the strength of concrete as per ASTM procedures.

Ottawa sand consists of rounded grains of clear, colorless quartz. It contains about 99.7% of silica (by mass) (Xenaki and Athanasopoulos, 2002; ASTM C 778-02, 2002) which is free from clay, loam, iron compounds, or other foreign substances. The imported sand costs 810.87 USD plus shipment charge for each 50 Kg of sand. This is a huge amount of currency the country is losing every year due to the lack of standard silica sand. Therefore, an alternative source from local deposit for standard sand production is economically very essential and saves the country from high foreign currency loss as well as taking one step ahead in the development and exploitation of national resources.

Cost of 50 kg of Ottawa Standard Sand for testing cement is \$811. Concrete laboratory of Bangladesh University of Engineering and Technology (BUET) tests about 100 bags of cement each month which on average requires 2×100 kg of OSS. Therefore, about 2400 kg or 480 bags of sand is required annually. The cost of the sand used in the concrete laboratory annually is BDT 80×811×480 or BDT 3.11 crore. There are more than 62 cement industries in Bangladesh, if 4 time of the sand, used in the concrete laboratory of BUET, is used at the laboratories of these

industries then the total cost of import is about 15.55 crore of taka per annual. This huge amount of unwanted expenditure of foreign currencies can be eliminated using the standard sand from the local deposits of Bangladesh.

However, most natural sands from riverbeds or seashores are smooth and rounded and are excellent aggregates. It is well known that concrete is more workable when smooth and rounded aggregate is used instead of rough, angular or elongated aggregate. Porosity is also affected by particle shape, with more rounded sands having less pore space than angular sands (Yi *et al.*, 2001; Puri and Mancino, 2002). Furthermore, more angular particle shapes result in higher failure strength in engineering applications.

The objective of the this work is to find out an alternative source of ottawa sand which would be locally available and would comply with standard sand through sampling, mechanical and chemical treatment while the test procedure and other factors kept similar as ASTM specification in testing. For this purpose the Moulvibazar silica deposit (which is situated at the piedmont of Dupi Tila formation) of sand has been chosen by studying the geological formation of Bangladesh. This location has shown the maximum probability of sand having high silica content; moreover, this sand layer is very close to surface.

Early in our studies we had found that the sand under investigation did not satisfy the physical and chemical properties to be used as standard sand. So, to compare the properties of local sand with that of ottawa sand, the sample was thoroughly washed by water and hydrochloric acid separately to remove all the contaminants. To determine the chemical composition, X-ray diffraction analysis was carried out. A portion of the sand sample was mechanically abraded within a Los Angeles Abrasion Machine with specific parameter to improve the shape. Roundness was measured from SEM images of the sand samples using wadell formula (Wadell, 1932):

$$\text{Roundness, } \rho = (\sum r_i / R) / N$$

Where, r_i = individual radii of the corners and edges, R = radius of the maximum inscribed circle; N = number of corners and edges.

Differential scanning calorimetry (DSC) was also carried out to observe the specific heat change in specific temperatures with time to investigate if any kind of reactivity is shown by the local sand.

2. EXPERIMENTAL

2.1. Apparatus

Sand samples were mechanically abraded using Los Angeles Abrasion Machine, constructed from structural steel with welded ½ in (1.27 cm) thick drum wall. Each ball is 46.8 mm in diameter with average mass of 350 to 445 g. Rotation is provided by an electric motor with speed reduction and enclosed chain drive to allow the drum to turn at 30-33 rpm. ASTM E11 standard sieves of four different

mesh sizes 30, 40, 50, and 100 were used. XRD analyses were done in the laboratory of magnetic material division of Bangladesh Atomic Energy Commission (BAEC) using PW 3040-X'Pert PRO Philips diffractometer with maximum input power of 60 kV and 55 mA. Quartz minerals were observed using scanning electron microscope (SEM) (model: JEOL JSM-6490 LA) for surface topographical studies. The grains were mounted on a SEM brass stub. Images were taken at a magnification where 3 or 4 grains of sand can be observed easily. The magnifications were generally varied from 40 to 70 but higher magnification was used for the grains of smaller size. A differential scanning calorimeter (model: DSC-60, SHIMADZU Corp., Japan) was used for thermal analysis. The sample (5g) was taken in an aluminum pan and water was added by a dropper. It was pressed to convert into pellet and the pellet was put into the DSC chamber. It was sealed by pressing a lid on the pan and the resulted sealed pan was put into the DSC chamber. Every sample was treated for an hour in the chamber.

2.2. Sample Preparation

The collected sand sample was at first water washed with agitation in a perforated bowl to remove all the adherent clay from the sample. Some bigger particles such as stone chips, brick chips etc. were removed at the time of washing. Then the sand sample was taken for drying and was designated as water wash (W) sample. Another portion of raw sample was treated with 0.1N hydrochloric acid (acid wash, AW) to reduce metallic compounds. Then sand sample was submerged in the acid solution for 24 hours with manual agitation followed by submerged in water (24 hrs) to neutralize acidic effect. Then it was again mechanically processed.

The third sand sample was mechanically abraded to reduce the angularity of sand grain. 10 kg of sample was taken to the Ball mill machine containing 6 or 10 pieces of balls and the desired number of revolutions (300 or 500) was carried out. Then the resulted sand was thoroughly washed with water and kept in the oven for 24 hours at 110°C for drying fully so that no moisture could present in the sand.

2.3. Grading and proportioning

The dried sand samples were graded according to the ASTM standard #30, #40, #50 and #100 sieves. About 500 g of each sample was taken in the sieve and kept in the shaker machine for about 15 minutes. Then the sand retained in different sieves was kept in different bowls according to sieve number.

3. RESULTS AND DISCUSSION

3.1. X-ray diffraction (XRD) analysis

The technique of XRD was used in this work with the objective to investigate the chemical composition of the

treated sand samples. The overall composition of the sand samples has been given in the Table I.

XRD analysis showed that there are overall 96.87% and 91.17% of quartz content in water wash and acid wash sand samples respectively. In addition, silica content of sample AW could be increased slightly, as there is an indication of the presence of silicon in other forms (Fe_3Si and Al_2SiO_5). However, the reduction in the silica content after acid wash could be because of the dissolution of the silica along with other debris during acid wash.

XRD analysis has also shown that there is about 2.61% of Fe in the sand sample obtained after water wash and that in the acid wash sample is about 2.16%. This indicates the iron contamination was slightly removed after acid wash. Aluminum content of the water wash sand is about 0.15%, but that in the acid wash sample is seen to be 2.81%. It may be predicted that with relative loss of quartz in AW sample, aluminum content become higher in this sample. However this may require further investigation to make a conclusive decision.

TABLE I
OVERALL CHEMICAL COMPOSITION OF THE SAND SAMPLES

| Constituent | Sand W (%) | Sand AW (%) |
|---------------------------|------------|-------------|
| Quartz | 96.87 | 91.17 |
| Fe | 2.61 | 2.16 |
| Al | 0.15 | 2.81 |
| Fe_3Si | - | 3.31 |
| KCl | 0.37 | - |
| Al_2SiO_5 | - | 0.55 |

3.2. Scanning Electron Microscopic (SEM) analysis

In general, with the increase in roundness of sand grain the workability of sand increases, but degree of bond formation with cement decreases. In order to observe the

surface topography of sand grains, scanning electron microscopic analysis was carried out. The SEM images of sand samples obtained after acid wash, and water wash were shown in the Fig. 1 and 2 respectively .

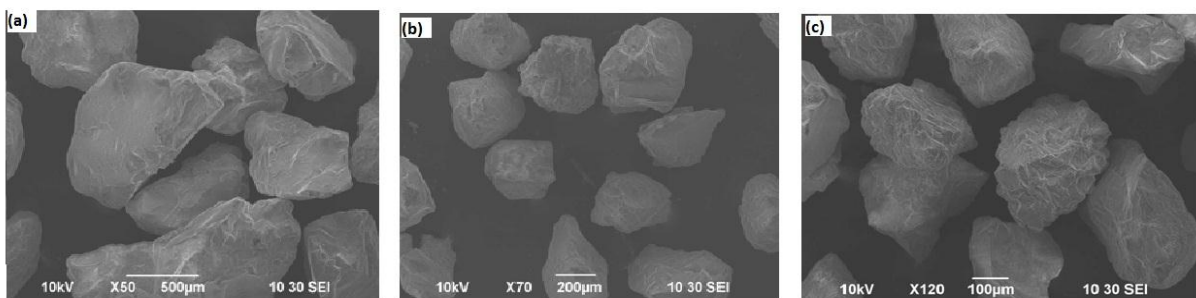


Fig. 1. SEM images of sand sample obtained after acid wash: sand grain retained on the sieve of 30 mesh (a), 50 mesh (b); and 100 mesh (c).

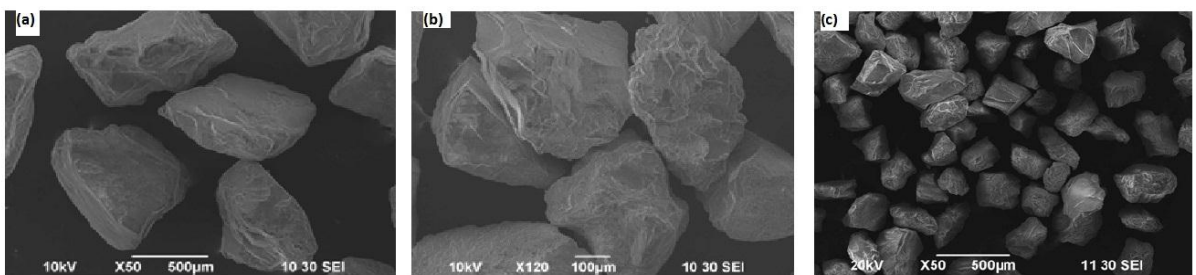


Fig. 2. SEM image of sand sample obtained after water wash: sand grain retained on the sieve of 30 mesh (a), 50 mesh (b); and 100 mesh (c)

The SEM images showed that sand samples are, unlike ottawa standard sand (Vivas, 2006), angular in nature. An attempt was made to increase the roundness of the sand sample by mechanical abrasion varying number of spherical balls and revolution. The images obtained after mechanical abrasion are given in the Figs. 3, 4, and 5 respectively.

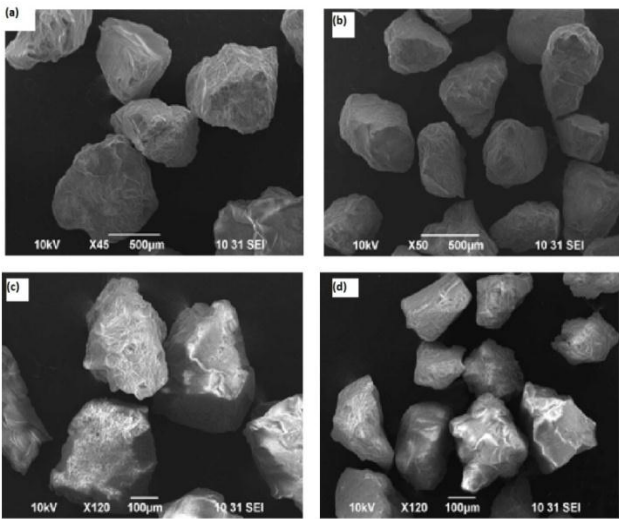


Fig. 3. SEM image of sand sample obtained after mechanical abrasion with 12 spherical balls and 300 revolutions: sand grain retained on the sieve of 30 mesh (a), 40 mesh (b), 50 mesh (c), and 100 mesh (d).

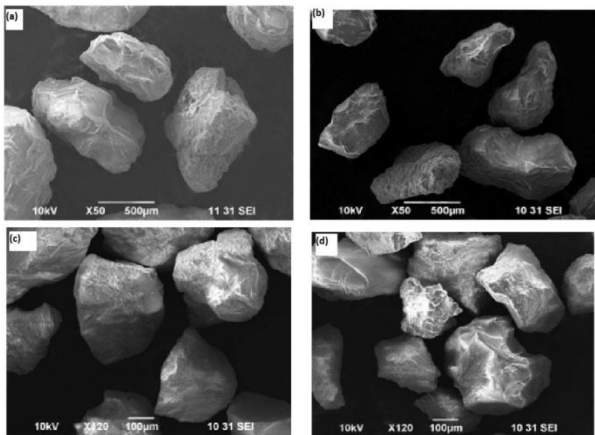


Fig. 4. SEM image of sand sample obtained after mechanical abrasion with 12 spherical balls and 500 revolutions: sand grain retained on the sieve of 30 mesh (a), 40 mesh (b), 50 mesh (c), and 100 mesh (d).

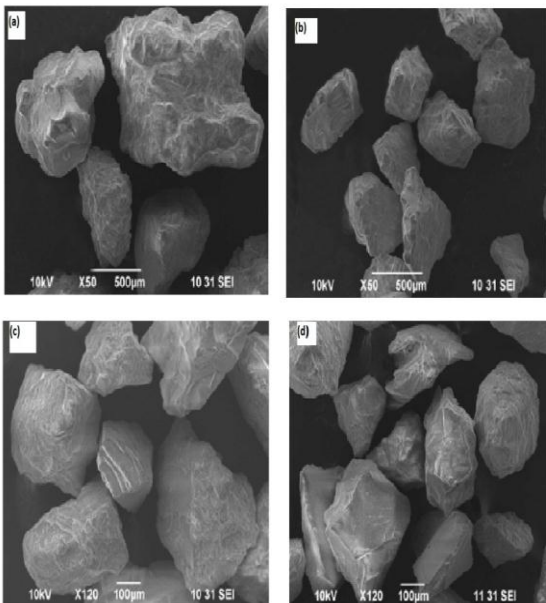


Fig. 5. SEM analysis image of sand sample obtained after mechanical abrasion with 6 spherical balls and 500 revolutions: sand grain retained on the sieve of 30 mesh (a), 40 mesh (b), 50 mesh (c), and 100 mesh (d).

While conducting mechanical abrasion we have observed from some SEM images that there is an increase in roundness because of the reduction in secondary corners and also in some images it seems there might have an increase in secondary corners and thus a decrease in roundness. To qualify the change in roundness of the sand samples obtained after and before mechanical treatment, it is imperative to determine the roundness of the samples in the subsequent section.

3.3. Determination of roundness

Roundness of both ottawa standard sand and the local sand samples were determined using Wadell formula. Results of the analyses and the gradation (Pettijohn, 1949) for different sand samples were shown in the Table III. The roundness of ottawa standard sand is 0.45 and it is classified as rounded sand by Pettijohn gradation. It is in good agreement with others (Ketema, and Whitman, 1959; Ketema *et al.*, 2005). This claim is also congruent to the specification usually labeled on the package of OSS.

Roundness of the local sand samples after water wash and acid wash were found to be 0.28 and 0.27 respectively. Both the samples belong to subrounded class. The roundness values of the sand samples obtained after mechanical abrasion (L, M, and N) are nearly 0.22 and all these samples belong to sub-angular class.

Roundness of the local sand samples indicates that there is a reduction in roundness after mechanical abrasion. SEM images (Figs 3-5) show that a large number of secondary corners has developed after mechanical abrasion. As the number of secondary corners has been increased after mechanical abrasion, more number of smaller circles on the corners has been drawn and consequently the value of roundness has been decreased substantially.

TABLE II
Over all roundness of sand samples and their gradation

| Sample | Sieve Size | Average Roundness (out of 1), ρ (mean \pm S. D.) | Overall Roundness, ρ (mean \pm S. D.) | Roundness Grade |
|---|------------|---|---|-----------------|
| AW | 30 | 0.371 \pm 0.108 | 0.266 \pm 0.188 | Sub-rounded |
| | 50 | 0.209 \pm 0.073 | | |
| | 100 | 0.215 \pm 0.110 | | |
| W | 30 | 0.306 \pm 0.114 | 0.283 \pm 0.106 | Sub-rounded |
| | 50 | 0.315 \pm 0.139 | | |
| | 100 | 0.233 \pm 0.067 | | |
| OSS | - | - | 0.449 \pm 0.178 | Rounded |
| L (10kg sand, 12 spherical balls and 300 revolutions) | 30 | 0.179 \pm 0.015 | 0.215 \pm 0.057 | Sub-angular |
| | 40 | 0.232 \pm 0.058 | | |
| | 50 | 0.178 \pm 0.041 | | |
| | 100 | 0.262 \pm 0.060 | | |
| M (10kg sand, 12 spherical balls and 500 revolutions) | 30 | 0.215 \pm 0.015 | 0.224 \pm 0.054 | Sub-angular |
| | 40 | 0.212 \pm 0.071 | | |
| | 50 | 0.223 \pm 0.040 | | |
| | 100 | 0.242 \pm 0.074 | | |
| N (10kg sand, 6 spherical balls and 500 revolutions) | 30 | 0.222 \pm 0.047 | 0.229 \pm 0.081 | Sub-angular |
| | 40 | 0.196 \pm 0.070 | | |
| | 50 | 0.268 \pm 0.107 | | |
| | 100 | 0.204 \pm 0.085 | | |

OSS is rounded but the local sand sample is subrounded in nature. Thus use of OSS in testing hydraulic cement mortar would provide good workability, but lower compressive strength. In contrast, the subrounded local sand sample would have comparatively lower workability, but higher compressive strength. If we give more time to agitate during the production of mortar than that needed for OSS, the local sand could be considered as feasible for testing hydraulic cement mortars.

3.4. Differential scanning calorimetric analysis

Isothermal differential Scanning Calorimetric analysis was carried out to investigate the reactivity of the sand samples. The sand is mixed with water to observe if any hydration reaction occurs at three specific temperatures 30^o, 45^o and 60^o C respectively. The growths of heat due to

progressive hydration of sand samples were illustrated in the Figs. 6, 7 and 8. The isothermal differential scanning calorimetric analyses indicate that constituents of the local sand samples are slightly reactive with water. As shown in the Fig. 8 the sand sample obtained after mechanical treatment, although, at 30 °C temperature does not show any reaction with water, but it undergoes hydration reaction at elevated temperature (45 °C and 60 °C). In the Figs. 6 and 7 the reaction of the sand samples with water has been prompted with the increase of temperature.

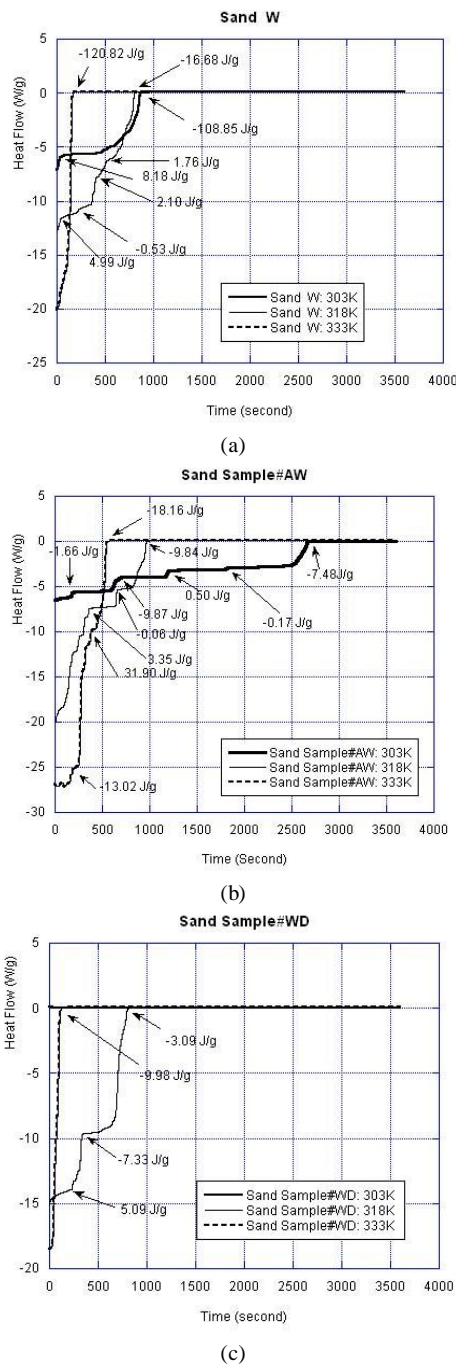


Fig. 6. DSC thermal analysis of (a) water wash sand sample (W), (b) acid wash (0.1 N hydrochloric acid) sand sample (AW), and (c) sand sample obtained after mechanical abrasion (WD) at three different temperatures: 303K (30°C), 318K (45°C), and 333K (60°C).

Ottawa standard sand contains about 99.7 % of quartz; quartz does not undergo any hydration reaction during the preparation of hydraulic cement mortar. So, the standard sand is fairly non-reactive with water and the constituents of cement. The local sand sample contains about 96.87% and 91.17% of quartz after water wash and acid wash respectively. The other constituents (like Al and Fe) of the sand sample might present as an active form which undergoes hydration reaction during DSC analysis.

Since the nature of reaction of cement's constituents with aggregate surface is not clearly understood, the

reactivity of the local sand samples cannot be exactly predicted (Bye, 1999). But results shown in the DSC analysis demonstrate that the sand samples unlike Ottawa standard sand would react with the constituents of cement. If the active forms of silica react with the lime, a constituent of cement, some extra bonds could be found which would increase the compressive strength of cement. But if the active components of the local sand react with alkali (NaOH or KOH), it may cause deterioration of the concrete. The concrete could be expanded and cracked because of the formation of alkali silica gel (Lea, 1956).

If we compare the reactive nature of the water wash (Fig. 6) and acid wash sand sample (Fig. 7), it can be observed that the degree of reactivity of the sample increases after acid wash. Thus the acid wash sample may react with cement's constituents more intensively than the water wash sample. Therefore, water wash sample has shown more feasibility than acid wash sample to be used as standard sand. On the other hand mechanically treated sample has shown maximum feasibility to be used as standard sand because of its lower tendency of hydration reaction. In conclusion, the comparative properties of the OSS and the sand sample under investigation can be enlisted into the following tabular form:

TABLE III

Comparison of characteristics between the Ottawa standard sand and sand of Moulvibazar deposits.

| Properties | Ottawa standard sand | Sand of Moulvibazar deposits |
|----------------|----------------------|------------------------------|
| Silica content | 99.7% | 96.7% (after water wash) |
| Roundness | Rounded | Sub-rounded |
| Reactivity | Non-reactive | Slightly reactive |

4. CONCLUSIONS

XRD analysis of the water wash sand under investigation shows the presence of 96.87% of silica in it. On the other hand OSS contains about 99.7 % of silica. Thus a higher percentage of silica content is necessary to replace the OSS. However, the local sand sample contains iron and aluminum contamination. The unwanted active component of the sand may affect the compressive strength of hydraulic cement mortar. Acid wash (with 0.1N HCl acid solution) sample has been considered as the means of removal of the active iron content of the local sand. XRD analysis of the acid wash sand samples shows the presence of 2.16% of iron content. The silica content of the acid wash sand sample was reduced to 91.17%. The reduction in silica content after acid wash could be because of the dissolution of the silica along with the iron from the surface of the sand grain during the treatment of the sample with acid solution.

Roundness of the OSS was calculated (by Wadell scale) to be 0.45 which belongs to the 'Rounded' class. The

values of roundness of the water wash and 0.1N HCl acid wash sand samples were found to be 0.28 and 0.27 respectively. Both the sand samples fall into 'sub-rounded' class of roundness grade (Pettijohn gradation). Roundness obtained after mechanical abrasion of sand samples L, M, and N have been found to be 0.22, 0.22 and 0.23 respectively. These values indicate that they have reduced to sub-angular class. Thus it has been observed that roundness of the sand samples were decreased after mechanical abrasion.

It can be predicted from the DSC analysis that the sand samples unlike ottawa standard sand would react with the constituents of cement. This reaction may increase the strength of cement mortar or it may cause the deterioration of the concrete. As our aim is to use the sand in testing cement, not the production of concrete for long time usage, this reaction would not affect the test results significantly.

Therefore, neglecting the unaffected reactivity of the sand sample, considering the silica content and subrounded shape of the sample, the water wash sand sample could be recommended for using as standard sand of Bangladesh for testing hydraulic cement mortars. Besides, this sand can also find its suitability in glass industries, and water and sewage treatment industries, because of its above mentioned superior qualities.

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