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CHARACTERIZATION OF HEAT-INSULATING CERAMIC FIBER RAW MATERIAL FOR INSULATION

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Abstract

New environmentally-friendly heat insulation materials, which are made from local raw materials can serve as an alternative to heat insulation materials already existing in the market, using clay as a raw material in its production, but dolomite is used as bio-solubility, thus obtaining material with the necessary heat and acoustic insulation qualities, as well as fire-resistance and chemical stability in an aggressive environment and providing to the development of industries in Bangladesh. Bangladesh is a developing country in the region of South Asia. Day by day its industrial growth is increasing very fast. Natural gas is used in the furnace region of all industries. It is a matter of disappointment to us that all industries never use proper insulation to save natural gas and the environment. For saving our natural gas, all industries must maintain proper furnace insulation not only for carbon emission but also for cost cost-effectiveness) of product price. Like many insulators, ceramic fiber is a key material for industrial furnace insulation. For continuous ceramic fiber fabrication, raw materials resources are important factors. In Bangladesh, raw materials are also available due to geological position. But clay raw materials are not only used as traditional raw materials but also used as heat-insulating materials. For developing ceramic fiber-related insulation in our county, extensive study and characterization need for selecting the right clay raw materials. Characterization methods like XRF, XRD, and DSC-TG and sieve analysis give deep information about locally available clay raw materials.

Keywords: Ceramic Fiber, XRF, FESEM-EDX, XRD, and DSC-TG

1. Introduction

Ceramic fiber is a man-made mineral fiber that contains generally alumina, silica, and other oxides. The main source of alumina and silica is kaolin clay. Clay is composed mainly of silica, alumina, and water, frequently with appreciable quantities of iron, alkalis, and alkali earth [1]. Clay materials are normally formed by two structural units that are involved in the atomic lattices. One unit consists of closely packed oxygen and hydroxyls in which aluminum, iron, and magnesium atoms are embedded in an octahedral combination so that they are equidistant from six oxygen or hydroxyls. The second unit is built of silica tetrahedrons. The silica tetrahedrons are arranged to form a hexagonal network that is repeated indefinitely to form the sheet of composition, $Si_4O_6(OH)_4$ [2].

Ceramic fiber made from local kaolin clay may gain better properties than other refractory materials by modification of composition. Because of its exceptionally low thermal conductivity, ceramic fiber dissipates little heat from the furnace and has remarkable energy-saving properties. Ceramic fibers are largely divided into vitreous alumina-silica ceramic fiber whose service temperature is below 1250℃ and crystalline alumina ceramic fiber whose service temperature is above 1250℃ [3].

The main advantage of ceramic fiber is fibrous lightweight refractory, with lightweight, high-temperature resistance, good thermal stability, low thermal conductivity, specific heat, and small mechanical shock resistance and other advantages. Ceramic fiber is also resistant to the erosion of aluminum zinc and other non-ferrous molten metal, and it has excellent high-temperature strength. It also has excellent resistance to acid, oil, and water corrosion [4]. Also, we understand the use of fossil energy increases greenhouse gas emissions, which accelerates global warming and causes climate change, and suffers our country's people through natural calamities. Ceramic fiber bulk density is much lower than other refractory materials, so it has small heat storage, which can greatly reduce energy loss. Another ceramic fiber is commonly called super wool. The typical chemical composition of super wool consists mainly of SiO_2, CaO, and MgO, whereas vitreous ceramic fiber consists mainly of Al_2O_3 and SiO_2 [5].

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2. Materials and Methodology

In this work, clay is used as a principal ingredient for targeting ceramic fiber production. The name Durgapur clay, Hobiganj clay is white clay quality and is now mainly used by the ceramic, paper, and rubber industries of the country. Generally, Bangladeshi clay occurs abundantly in some localities of the districts of Netrokona and Habiganj in Bangladesh. These regions are mostly sediment areas. The clay is deposited in different riverside areas. The predominant color of the Bahubal formation is reddish yellow. Generally, the clay is light greyish white to bluish white with light yellow and slight soap to feel, massive and soft to medium hard. That clay is collected in a necessary amount and prepared for the beneficiation process. A beneficiation process is needed for removing the free silica and compounds like iron oxide, and titanium oxide. A study was made on the raw materials beneficiation of two different deposits in Bangladesh. The aim of the present research is the investigation of the beneficiation and properties of clay and its thermal behavior. After investigating the main properties of clay bio-soluble ceramic fiber raw materials are prepared.

2.1 Bio-soluble ceramic fiber composition

In this research, we are targeting to produce thermal insulating bio-soluble ceramic fiber by mixing kaolin-grade clay and dolomite through conventional blowing methods. The solubility of this ceramic fiber is improved by optimizing the amounts of network-forming oxide SiO_2 that comes from local clay compounds which also reduces the viscosity of the melt. Intermediate oxide Al_2O_3 also comes from local clay near about 20 wt % which shows refractory properties as an insulating material. When the content of alumina Al_2O_3 has increased the viscosity of the melt is simultaneously increased.

At high temperatures, intermediate oxide Al_2O_3 forms aluminum silicate which is insoluble in human body fluid. For bio-solubility, Al_2O_3 content is below 25 wt % of the raw materials. Dolomite compound consists of $CaMg(CO_3)_2$, after calcination CaO and MgO remain in dolomite. The network-modifying oxide CaO comes from dolomite which play an important role in ceramic fiber by increasing solubility. The CaO content is not more than 25 wt % for bio-soluble ceramic fiber materials. Another network-modifying oxide MgO also appears from dolomite which controls the fiber diameter and length during fabrication. The amount of MgO is less than 8 wt %, otherwise, the melt viscosity increases [7].

Thermal properties such as heat resistance are improved by optimizing the amounts of network modifying oxide ratio (CaO and MgO) as 3:1 by wt % addition. P_2O_5 and B_2O_3 are networks former like silicon dioxide. By the addition of some water-soluble P2O5 and B_2O_3, the bio-solubility of ceramic fiber is increased into body fluid. The main sources of P_2O_5 and B_2O_3 are phosphorite mineral $Ca_3(PO_4)_2$ and boric acid. These bio-soluble ceramic fibers impart the ability to withstand temperatures up to 1250ºC [8]. Bio-soluble ceramic fiber raw materials collected from low-cost local clay make ceramic fiber cost-effective in the local market. Oxide which has some effect on ceramic fiber manufacturing is shown below in TABLE 1.

TABLE 1. EFFECT OF OXIDE ON FIBER

Effect	Melting Temp.	Effect on Ceramic Fiber
Al_2O_3	2030°C	Increase viscosity of melt
SiO_2	1710°C	Decrease viscosity of melt
Fe_2O_3	1565°C	Blackish color as an impurity
TiO_2	1840°C	Yellowish color as an impurity
MgO	2800°C	Increase bio-solubility and control
CaO	2570°C	Increase bio-solubility and control
B_2O_3	450°C to 510°C	Increase bio-solubility
P_2O_5	560°C to 570°C	Increase bio-solubility

2.2 Ceramic fiber production

Ceramic fibers are produced by melting a combination of alumina (Al_2O_3) and silica (SiO_2) in approximately equal and near proportions or by melting kaolin clay. Other oxides, such as zirconium dioxide (ZrO_2), boric oxide (B_2O_3), titanium oxide (TiO_2), and chrome oxide (Cr_2O_3) are sometimes added to alter the properties of the resulting fibers [9]. For example, chemical composition is one of the factors that determine the maximum feasible end-use temperature. As for all manmade vitreous fibers, the fiber length, diameter, and bulk density-controllable to some degree by the manufacturing method and chemical composition also affect key physical properties of the refractory ceramic fibers, e.g., the thermal conductivity [10].

The basic composition of refractory ceramic fibers has not changed appreciably since their initial formulation in the 1940s [11], but modifications to the composition such as raising the content of alumina and the addition of CaO, MgO, and other materials create fibers that tolerate higher maximum end-use temperatures. Refractory ceramic fibers were first produced in the USA in the 1940s for the aerospace industry. The commercial importance of refractory ceramic fibers increased during the 1970s when rising energy costs created a strong demand for efficient refractory insulating products [12].

2.2.1 Blowing method.

Ceramic fibers are produced by melting a combination of alumina and silica in approximately near equal proportions at temperatures up to 2000°C or in the USA by melting kaolin clay together with several trace ingredients. The molten mixture is made into fiber either by blowing air or stream onto the molten material flowing from an orifice at the bottom of the melting furnace (the blowing process; Figure 1). The fibers are either collected directly as bulk fiber or further processed into a blanket by a needling process. Although ceramic fibers are sold in a variety of forms, all start with the production of either bulk or blanket material, termed „primary refractory ceramic fiber production" [13-14]. The processing of ceramic fibers begins with the fiber in either bulk or blanket form. Bulk material may be used directly but is usually used as a feedstock for other processes.

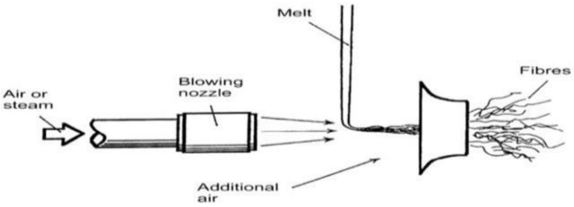

Fig. 1: Ceramic Fiber Production Process

3. Result and Discussion

In this research, the methods used for the identification and quantification of natural and beneficiated clay for ceramic fiber composition include X-ray fluorescence analysis (XRF), X-ray diffraction (XRD), and differential thermal analysis (DSC-TG). X-ray fluorescence analysis gives detail about the chemical composition of raw materials. After studying the chemical composition of selected clay, necessary oxides are added for ceramic fiber composition if modification is needed. X-ray diffraction leads to an understanding of the structural characteristics of the clay mineral. Chemical compositions of Durgapur and Hobiganj clay collected from different areas of Netrokona and Hobiganj district were carried out using X-ray Fluorescence Spectrometer clay samples subjected to various tests. Thermal behavior and weight loss of clay samples are studied by (DSC-TG) apparatus.

A. Optical Microscopy

The theoretical resolution of optical microscopy is about 0.2 μm. The length scale of clay particles is below this value thereby rendering limited information from optical microscopy of clays. The color of clay was found to be whitish. A tint of iron-rich mineral has shown to exist along with this clay.

B. X-ray Fluorescence Analysis

To determine the percentage of oxides present, X-ray fluorescence (XRF) was employed as a widely used versatile tool for elemental and chemical analysis of clays. Basically, XRF emits characteristic secondary x-rays from a material that has been excited by bombarding with high-energy x-rays. The table shows the composition (major) of (Hobi-07) clay. The clay has been found to be Al_2O_3 rich than other natural clays obtained from different areas.

TABLE 2: COMPOSITIONAL ANALYSIS OF CLAY

Clay Type	SiO_2	Al_2O_3	Fe_2O_3	TiO_2
Hobi-07	67.62	23.04	2.99	1.36

The clay also contains a small amount of CaO (0.14-0.21), MgO (0.36-0.45), ZrO_2 (0.26-0.27), P_2O_5 (0.07-0.12) and K_2O (1.60-2.20). The presence of iron and titanium oxide-bearing materials may impair the usefulness of clay.

C. X-ray Diffraction Analysis

To quantitatively analyze minerals, present in natural clays, X-ray diffraction (XRD) is an indispensable tool. The high-intensity X-ray beam (PAN ANALYTICAL EMPYREAN) was diffracted on clays within a scanning range (2µ) from 10 to 700 with CuKα radiation [15]. Fig. 2 presents the XRD pattern of natural clay. Kaolinite and quartz are found to be major mineral constituents of clay. Rutile was detected as the main titanium-bearing impurity along with a small amount of Illite. Fig. 3 shows the XRD analysis of clay samples. In the clay sample, the intensity of silica or quartz is highest which means a high amount of silica is present in these two samples. The hobi-07 sample has a low quartz amount. The Hobi-07 clay sample has more Al_2O_3 rich than other natural clay. The obtained two strong reflections are well matched with the standard prominent basal reflections at 12.390 and 24.930. The peaks were found to be sharp representing well-crystalline kaolinite. To determine the quantitative percentage of these minerals Rietveld refinement was conducted using HIGH SCORE PLUS Software [16-17]. Table III shows the quantitative mineralogical analysis of major minerals. In addition to this list, calcium sodium aluminum silicate and try magnesium dioxide dihydroxide were also detected as 4.6 % and 0.7 % respectively.

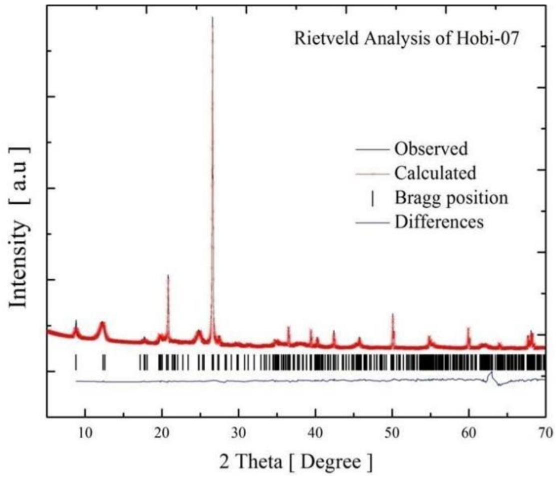

Fig. 2: XRD Pattern of Hobi-07 Clay

TABLE 3: RIETVELT ANALYSIS

Minerals	Wt (%)	Minerals	Wt (%)
Kaolinite	48.07	Illite	11.88
Quartz	37	Muscovite	2.7

5.

D. Secondary Electron Microscopy Analysis

To reveal the approximate size and shape of clay particles, Scanning Electron Microscopy (SEM) can be employed with its ability to obtain a very high resolution and magnified image of individual grains of clay minerals. In this context, Field Emission Scanning Electron Microscopy (FESEM) (Model: JEOL JSM-7600F) was used [19]. Samples were scanned for secondary electrons (SE) mode for morphological contrast and backscattered electrons (BSE) mode for phase contrast [20]. Fig. 3 and 4 show the FESEM micrographs of nanoparticles of natural clay. High-magnified images revealed both plate (Fig. 3) and particle-shaped (Fig. 4) clay particles.

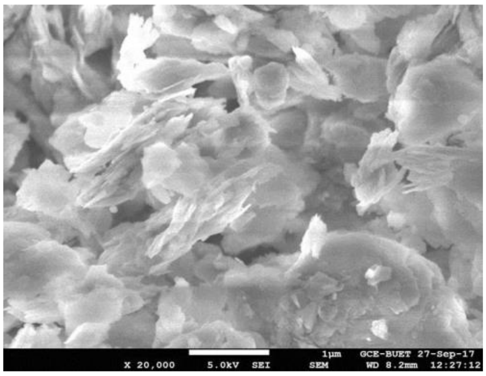

Fig. 3: SEM Micrographs of Natural Clay Showing: Kaolinite Hexagonal Sheet Plates

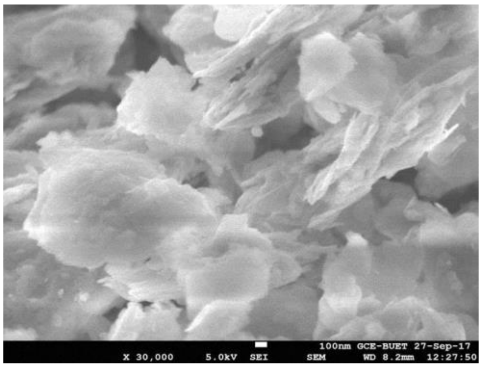

Fig. 4: SEM Micrographs of Natural Clay Showing: Kaolinite Particles Stacking.

E. Energy Dispersive X-ray (EDX) Analysis

Energy Dispersive X-ray (EDX) can provide valuable information on major and minor elements allowing highly reliable compositional identification [20]. Basically, EDX is used for qualitative as well as quantitative (the percentage of the concentration of each element of the sample) analysis. Fig. 3 and Fig. 4 show captured EDX spectra from the clay sample. Evidently, peaks of major elements namely Si and Al with allied oxygen were identified.

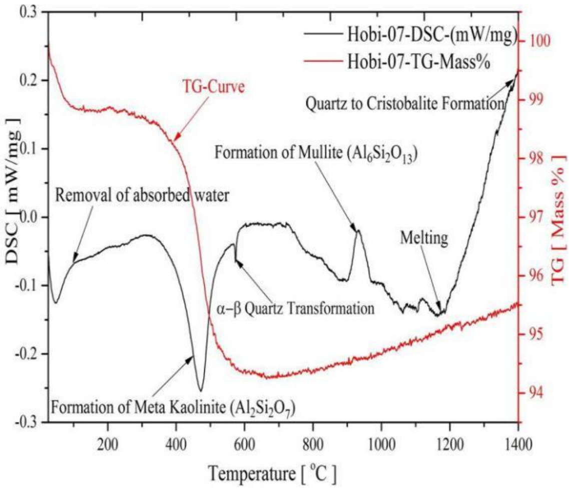

Fig. 5: DSC and TG Curves of Hobi-07 Clay

Table 4 shows the summary of the elemental analysis of clay in atomic (and mass) percentage. Al and Si contents were found to be higher in conformity with the compositional analysis of XRF (Table 4). Except for detected minor elements namely Mg(K), K(K), Ti(K), Cr(K), and Mn(K) concentration of major elements are only presented. In analyzed clay, the ratio of Al to Si is lower around half. In addition, some minor elements namely Ti, Ca, Mg, Mn, Fe, K, Fe and Cr are also identified in the clay sample.

TABLE 4: EDX ANALYSIS OF NATURAL CLAY

Clay Type	O(K)	Al(K)	Si(K)	Fe(K)
Hobi-07	23.37	21.47	49.35	1.90
Area-1	33.55	21.16	41.47	0.77
Hobi-07	28.79	26.41	37.47	2.30
Area-2	40.04	22.99	32.67	1.50

F. FTIR Analysis

The structural differences of clay can be ascertained by vibration spectroscopic investigations yielding useful information about hydration characteristics, interlayer cations, and moisture content in clays. The band at 533 cm^{-1} shows deformation vibration of Si–O and Si–O–Al whereas the band at 469 cm^{-1} indicates the presence of amorphous silica. Bands observed at 780-798 cm^{-1} are due to Si-O-Si inter tetrahedral bridging bonds in SiO_2. Bands at 1620 and 2642 cm^{-1} could be assigned to the H-O-H bending of water, which is observed in almost all the natural hydrous silicates like illite minerals. The absorption bands observed at 1813 cm^{-1} is related to carbonate and at around 2358 cm^{-1} related to calcite. The bands between 3450 and 3670 cm^{-1} is attributed to the OH stretching mode [20]. The band at 3620 cm^{-1} may be ascribed to the inner hydroxyls and the other three characteristic bands are generally ascribed to vibrations of the external hydroxyls.

G. Differential Scanning Calorimetric (DSC) Analysis

DSC-TG analysis measures both heat flow and weight changes in a material as a function of temperature or time in a controlled atmosphere. The complementary information obtained allows investigation of the exothermic and endothermic effects of reactions, such as carbon oxidation and dehydroxylation respectively.

Fig. 5: DSC and TG Curves of Hobi-07 Clay

Differential thermal analysis is an important method to detect reactions due to the dehydroxylation of clay minerals, decomposition of carbonates, loss of combined water, loss of sulfur, and decomposition of organic matter. Fig. 9 shows DSC and TG curves of clay analyzed at room temperature to 1400°C depicting the effect of energy changes (endothermic or exothermic reactions) and weight changes in the sample. A first endothermic reaction was observed at a temperature below 100°C indicating desorption of surface H_2O (e.g., H_2O on exterior surfaces) and dehydration (e.g., interlayer H_2O) at low temperatures. Kaolinite shows a weight loss starting at just above 400°C due to dehydroxylation, extending to about 650°C (TG-curve of Fig. 5).

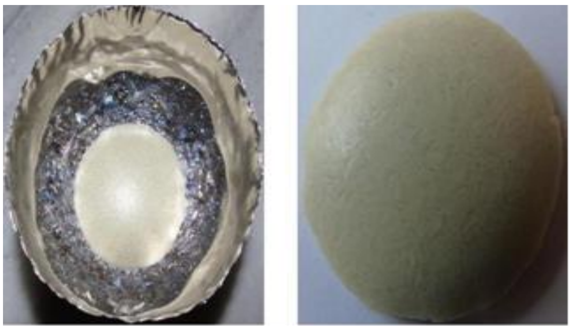

Fig. 6: Ceramic Fiber Raw Material after Melting at 1400°C

A significantly high endothermic peak observed at 456°C is related to the formation of meta-kaolinite ($Al_2Si_2O_7$). Quartz transformation has been shown to take place at 573°C. Next, an exothermic peak that appeared at 1000°C is related to the formation of the mullite ($Al_6Si_2O_{13}$) phase. An allotropic phase transition from quartz to cristobalite was detected by a small exothermic peak at 1200°C.

In addition, integrating bio-soluble materials into a project can help to reduce the environmental impacts associated with the extraction, transport, processing, fabrication, installation, reuse, recycling, and disposal of these industry source materials. All the materials used for fabrication must not have harmed the environment, polluted air or water, or cause damage to the earth, its inhabitants, and its ecosystems during the manufacturing process, and during use or disposal

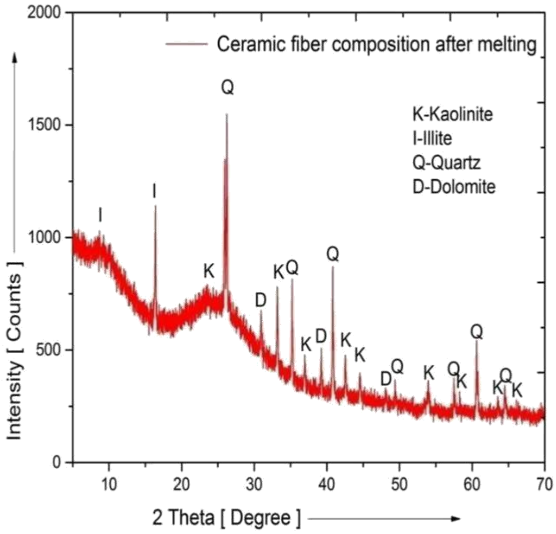

Fig. 7: XRD Pattern of Ceramic Fiber Raw Material after Melting

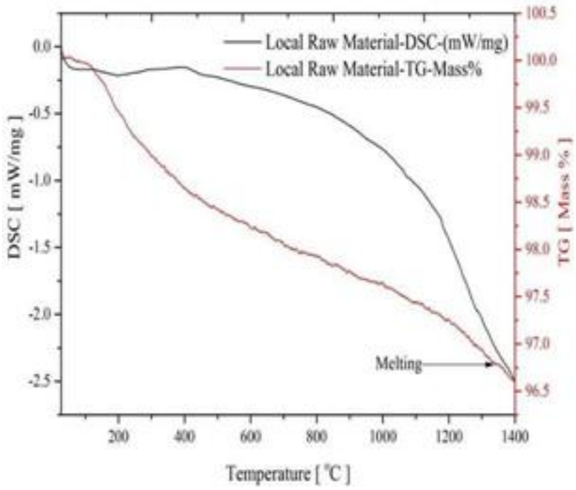

Fig. 8: DSC and TG Pattern of Ceramic Fibre Raw Material after Melting

after their end of life. Materials should be non-toxic and contribute to good indoor air quality. Using environmentally friendly materials is the best way to build an eco-friendly material.

Fig. 7 presents the XRD pattern of Ceramic Fiber Raw Material after Melting. Kaolinite, dolomite, and quartz are found to be major mineral constituents of clay. Rutile was detected as the main titanium-bearing impurity along with a small amount of Illite. Fig. 8 shows the DSC and TG properties of sintered raw material for a better description.

4. Conclusions

The following criteria can be used to identify sustainable materials: (1) Local availability of materials. (2) Embodied energy of materials. (3) Percentage of recycled/waste materials used. (4) Rapidly renewable materials. (5) Contribution to the Energy Efficiency. (6) Recyclability of materials. (7) Durability. (8) Environmental Impact. From XRD, chemical, and DSC analysis of Hobi-07 clay, it is concluded that Hobi-07 clay contains silica and alumina as major constituents, and iron, calcium, potassium, and magnesium oxide are present in minor quantities. Kaolinite, quartz, and illite are present as major phases in Hobi-07 clay. From various properties, it is apparent that Hobi-07 clay may be used as an impotent raw material in ceramic fiber industries in Bangladesh to produce various ceramic fiber-related products and has the prospect of lowering the dependency on imported ceramic insulating materials. Hobi-07 washed clay appears to be the best in quality among all the types of Bangladeshi clay.

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