Pakistan J. Sci. Ind. Res., **12**, 62–66 (1969)

# STUDIES ON AN INDIGENOUS FIRE CLAY FOR THE MANUFACTURE OF GLASS TANK BLOCKS

M. Aslam Chaudhary, Ghulam Rasul and M. A. Rana

*Glass and Ceramics Division, P.C.S.I.R. Laboratories, Lahore* 16

(Received July 24, 1968; revised September 19, 1968)

K.D. Fire Clay No. 1 from Mianwali district has been extensively studied. It has been shown that water washing considerably improves this kaolinite type plastic clay for use as a suitable raw material in the manufacture of glass tank blocks. Test bricks made from the washed clay containing 60% grog of the same material are comparable to the imported fire clay blocks. The test bricks contain 42-43% $Al_2O_3$ and 1.1% $Fe_2O_3$. They show only 5% subsidence at 1575°C under a load of 2 kg/cm$^2$ and have a PCE value of 35. Their bulk density and porosity are 2·1 and 18-19% respectively.

Glass industry in Pakistan is expanding rapidly and new factories for the manufacture of more sophisticated products are coming up almost every year. With the expansion of glass industry, demand for glass house refractories is also increasing. These refractories are mostly imported in the country, and glass tank blocks claim a major portion of the foreign exchange required for the import of all refractories. The development and manufacture of glass tank blocks in Pakistan, therefore, would not only save foreign exchange but would also help in further expansion of glass industry in the country.

Although many refractory clays have been discovered in various areas of West Pakistan and although some evaluation work[1,2,2a] has been done on these fire clays, yet no attempt has been made regarding making of suitable glass tank blocks from these clays. Some attempts have, however, been made by the fire brick manufacturers towards making of tank blocks but their products are of low standard. These blocks can not withstand a temperature higher than 1350°C, and they have very poor corrosion resistance resulting in much shorter furnace life. This results in a poor quality of glass produced in furnaces made from local blocks. These inferior tank blocks may be due to lower grade fire clays or due to the defective manufacturing process.

As a first step towards this project, it was, therefore, decided to study thoroughly an abundantly available fire clay and to see if it can be improved by some simple methods. The clay selected for the present investigation is a fire clay from Mianwali district commonly known as K.D. Clay No. 1 which is being used commercially by Pakistani fire bricks manufacturers. It is grey in colour, fine in texture and possesses good plasticity. It was discovered that proper washing treatment improves the clay for its use as a suitable raw material for making glass tank blocks. An exploratory study was undertaken and sample bricks containing 60% grog and 40% washed clay were prepared under carefully controlled drying and firing schedules. Subsequent testing proved that these bricks were comparable to the imported tank blocks.

## Experimental Methods

*Washing of the Clay.*—20 g of the original clay was washed in a 400-ml beaker with 250 ml water. The clay was stirred thoroughly in water and allowed to settle for 2 min. Fine clay particles were separated by decantation and the clay was stirred again and allowed to stand for another 2 min. Finer particles from this portion were also separated after 2 min. This was repeated 4–5 times. All the heavier particles thus collected were further washed 4–5 times by stirring with water and removing the fines by allowing to settle for 2 min every time. The heavier portion was taken in a platinum dish and dried on a water bath. It was weighed after further drying in an air oven at 110°C. The percentage of heavy particles was found to be 25% in the original clay. The finer suspension was allowed to settle overnight and separated from water by decantation and dried on a water bath first and then in an air oven at 110°C. This finer fraction was called the washed clay in these investigations. For a clear understanding of the behaviour of the clay itself from these investigations no dispersing agents were added for washing purposes.

*Chemical Analysis.*—The washed and original clays were chemically analysed by British Standard Methods for the analysis of refractory materials.[3]

*Estimation of Soluble Salts.*[4]—The original clay (5.0 g) refluxed in 250 ml water for 0.5 hr. After allowing 0.5 hr to settle, the clay was filtered and the filtrate was dried at 110°C and weighed.

*Microscopic Analysis.*—A slide of the original clay was prepared and studied under a polarising microscope.

*IR Absorption Analysis.*—IR absorption analysis was conducted with a 'Leitz' double beam instrument No. 105. The sample was prepared by the method described by Hunt and Turner.[5]

*Differential Thermal Analysis.*—D.T.A. of the washed sample was carried out by the usual method using $Al_2O_3$ as inert material.

*Particle Size Distribution of the Clay.*—Particle size distribution of the washed clay was determined by the Anderson pipette method as described by Looms.[6] Sieve analysis of the heavier particles was done by B.S. test sieves.

*Drying and Firing Shrinkage.*—Drying and firing shrinkage was determined by measuring the distance between two parallel lines cut on the freshly prepared test pieces 2.5 in long and 0.75 in in diameter. The test pieces were prepared by extrusion.

*Apparent Porosity.*—Porosity at different firing temperatures was determined,[7] by boiling the test pieces in water for 2 hr and weighing while in water. The test pieces were weighed in air before and after saturation with water. The values were expressed in terms of percentage porosity.

*Making of Test Bricks.*—Lumps of washed clay were placed in saggers and slowly fired in an oil-fired furnace to 1380 ± 20°C. The temperature was raised to this point in 3 days and maintained for a further 24 hr. After cooling, the grog was crushed and graded into different sizes. A few specimen bricks using 40% washed clay and 60% grog were made. The percentage of various particle sizes of the grog used for these specimen is given in the following:

| *B. S. Test Sieve* | |
|---|---|
| −4+8 | 11.0% |
| −8+18 | 22.5% |
| −18+60 | 18.0% |
| −60+120 | 12.5% |
| −120 | 36.0% |

The grog and clay were thoroughly mixed to ensure complete homogeneity. Approximately 6–7% water was added and mixed to obtain a uniform distribution of water and suitable plasticity in the batch. The bricks 4×4×3 in were made by a hand press, and dried at room temperature for 8 days. The drying was further continued in an electric oven at 120–130°C for 3 days. The test bricks were then fired in an electric muffle furnace. The temperature was raised to 1380°C in 4 days. The furnace was shut down after maintaining the highest temperature for 4 hr and the test bricks were taken out on cooling.

*Testing of Test Bricks.*—Properties of the fired bricks like volume and linear shrinkage, bulk density and porosity were determined by standard methods.[7] Cold crushing strength was done on "Avery" 5-ton capacity testing machine.

*Determination of Fusion Point.*[8]—Four test pieces 1½ in long cut from the body of the brick to be tested, were ground on a suitable abrasive wheel to the shape of a pyramid with a triangular base. A test piece and the seger cones from No. 31–36 were mounted around the periphery of a circular sillimanite block. This assembly was placed in a gas fired furnace. The temperature was raised at a steady rate in 1½ to 2 hr to approximately 1450°C, and from then onward the rate of rise of temperature was maintained at 5°C/min. The test was continued till the tip of the test cone bent over level with the base, the block bearing the specimen was then removed from the furnace and the cones were examined when cold.

*Determination of Refractoriness under Load.*[9]—A test piece was cut from the brick, and it was ground properly to ensure that the faces of the test piece were plane and perpendicular to the height. The test piece measured 10.06×4.62×6.0 cm. All these dimensions were measured at four different points by means of a vernier callipers and the mean of all the four measurements was taken. The height of the test piece was 10.06 cm. This was then placed in the furnace on a block of the supporting column of silicon carbide. A constant load of 49.24 kg was applied to the test piece. The furnace temperature was raised at a uniform rate of 10°C/min from 300°C upwards to a temperature of 1475°C. At this temperature oxygen was also used to further raise the temperature until at 1575°C the load applied fell from the supporting column. The percentage subsidence, based on the original length was only 5% for this temperature.

## Results and Discussion

Table 1 shows that there is an improvement in the clay on water washing with a definite decrease in iron content from 1.67% to 0.96%, and increase in alumina content from 32.03% to 36.95% which comes out to 42.37% on fired basis. The amounts of alkalis and soluble salts also decrease on washing.

Microscopic analysis of the original clay shows that the predominant mineral is possibly kaolinite. This portion has a higher refractive index than Canada balsam and also shows birefringence of the first order. The original clay also contains approximately 5% sericite and 5% organic matter. The accessory minerals which were identified are feldspar, quartz, hematite, magnetite, limonite and pyrite.

IR spectrum of the washed K.D. clay (Fig. 1) shows that the clay is of the plastic type. IR absorption curves for kaolinite and plastic clay by Hunt and Wisherd are also given for the sake of comparison[10]. All the absorption bands shown by the standard kaolinite spectrogram are present in the curve for K.D. clay. The intensities of the absorption bands are however, not the same. Absorption bands in the region from 9–11 microns are characteristic of clay minerals of kaolinite type which is evident in this case also.

D.T.A. curve of the washed clay (Fig. 2) shows a pronounced endothermic change at 630°C and an exothermic peak at 980°C. A fire clay of kaolinite type is characterised by a large endothermic effect at 620°C and an exothermic effect at 980°C.[11]

Particle size distribution of a clay is of considerable importance because the physical properties like plasticity, drying shrinkage and modulus of rupture are influenced by the particle size distribution. In this case 75% of the clay is below 5 microns and about 50% is below 1 micron (Fig. 3). The sieve analysis of the heavier particles obtained after water-washing shows that about 80% of the residue is above 120 microns in size which is mostly sand and other minerals (Fig. 4) and can be easily removed.

The shrinkage and porosity curves are shown in Fig. 5. The shrinkage from 150–900°C increases uniformly. It is 0.125% per 100°C rise in temperature in this range. Between 900°C and 1100°C the shrinkage is 0.875% per 100°C rise in temperature. The most critical range in this curve is from 1100–1200°C where the shrinkage is 5.25%. Rapid firing in this temperature range should be avoided. The rate of shrinkage is uniform even in this range. From 1200–1400°C the rate of shrinkage falls sharply to a value of approximately to that at 900–1100°C. The apparent porosity has a high value, i.e. 37.80% at 800°C. It decreases to 35% at 1000°C and thereafter falls rapidly and almost uniformly to approximately 5% at 1325°C. Beyond 1325°C

TABLE 1.—CHEMICAL ANALYSIS OF THE ORIGINAL AND WASHED CLAY.

| Description | Loss on ignition % | $SiO_2$ % | $Al_2O_3$ % | $Fe_2O_3$ % | CaO % | MgO % | $Na_2O + K_2O$ (by difference) | Soluble salts % | Colour |
|---|---|---|---|---|---|---|---|---|---|
| Original clay | 13.23 | 51.20 | 32.03 | 1.67 | 0.74 | 0.23 | 0.90 | 0.97 | Dark grey |
| Washed clay | 12.82 | 48.40 | 36.95 | 0.96 | 0.52 | 0.18 | 0.17 | 0.53 | Light grey |
| Original clay (fired basis) | — | 59.00 | 36.92 | 1.92 | 0.85 | 0.27 | 1.03 | — | Cream |
| Washed clay (fired basis) | — | 55.52 | 42.37 | 1.11 | 0.59 | 0.21 | 0.20 | — | Light brown |

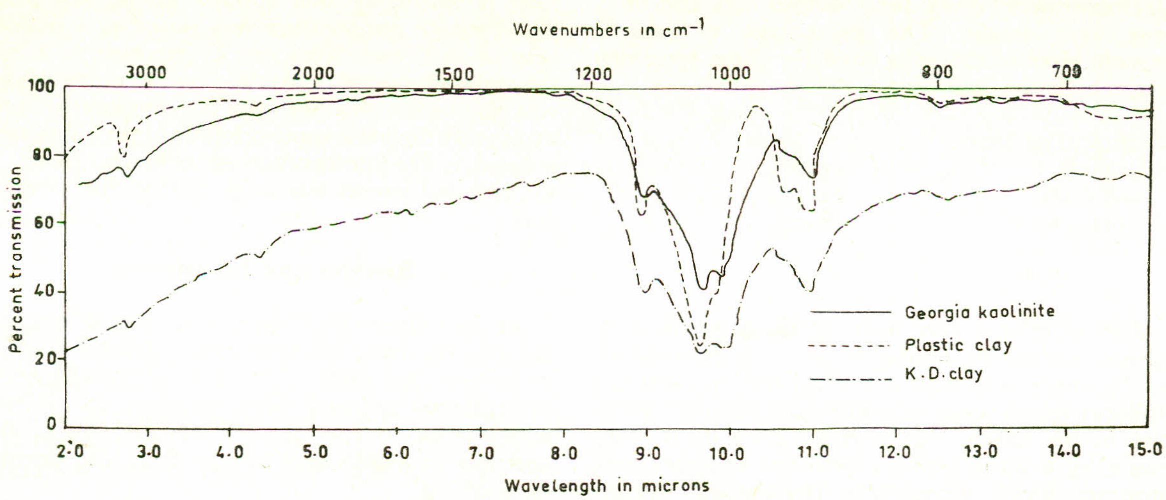


Fig. 1.—Absorption spectra of washed K. D clay, georgia kaolinite and plastic clay.

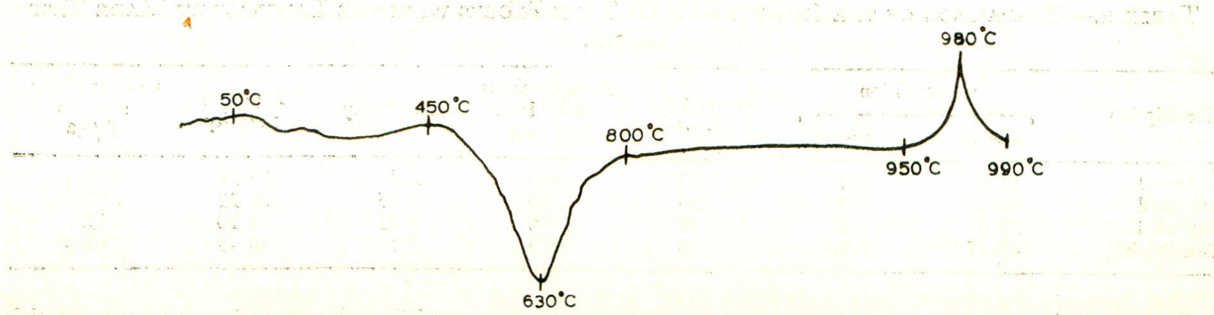


Fig. 2.—Differential thermal curve of K. D washed clay.

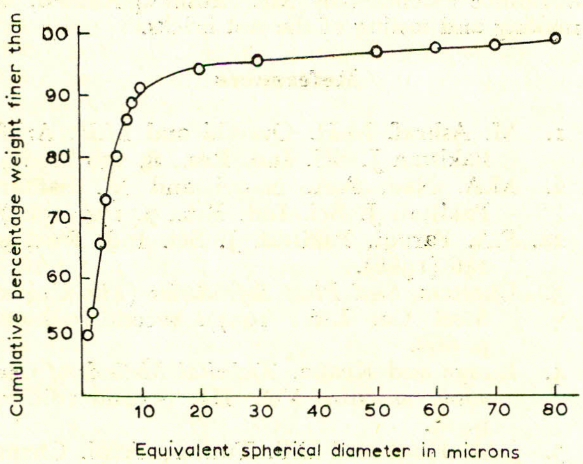


Fig. 3.—Particle size distribution of washed K. D clay.

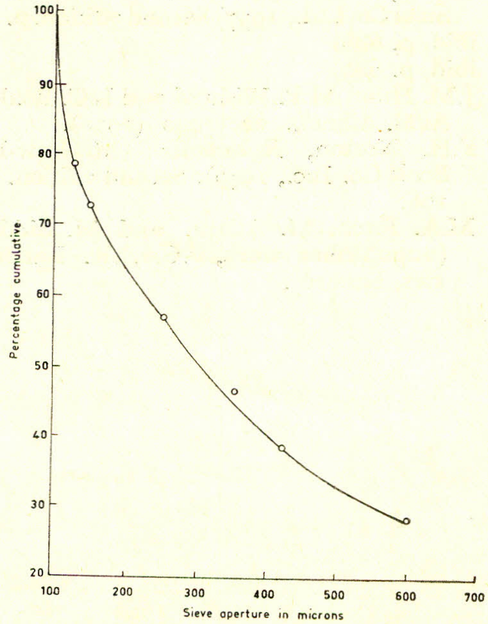


Fig. 4.—Sieve analysis of the heavier residue of K. D clay after washing.

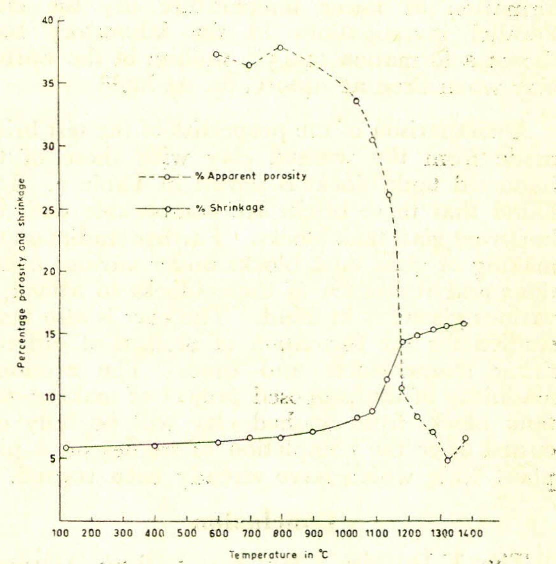


Fig. 5.—Apparent porosity and percentage shrinkage of washed K. D clay.

it again increases very slightly probably due to the development of micro-cracks. This fire clay presents, therefore, a wide range of porosity change which is quite a useful property.

From these investigations it is evident that this is a kaolinite type clay possessing good plasticity. Chemical composition of the fired clay is similar to that of the imported tank blocks which contain 40–42% alumina. Addition or blending of a plastic or a high alumina clay is, therefore, not necessary if washed clay is used.

The clay possesses fairly uniform shrinkage and a wide range of porosity. Therefore, properties like density, corrosion resistance, dimensional accuracy, thermal shock resistance and heat conductivity can be controlled for the production of not only tank blocks but also other types of alumino-silicate refractories.

TABLE 2.—COMPARISON OF THE IMPORTED GLASS TANK BLOCKS WITH THE LABORATORY MADE TEST BRICKS.

| Description | Composition | | Fusion point (P.C.E.) | Refractoriness under load 2 kg/cm2 | Bulk density | Apparent porosity | Cold crushing strength lb/in² |
|---|---|---|---|---|---|---|---|
| | $Al_2O_3$ | $Fe_2O_3$ | | | | | |
| Imported | 40.0 | 2.0 | 33 | 1480 | 2.1 | 19 21 | 8550 |
| Imported | 52 54 | 1.5 | 36 | 1580 | 2.25 | 20 22 | 8550 |
| Imported | 32.0 | 1.5 | 31 | 1400 | 2.05 | 18 20 | 8550 |
| Test Brick | 42.37 | 1.10 | 35 | 1575 | 2.1 | 18 19 | 15000 |

Presence of small amounts of soluble salts and other fluxes in the clay are advantageous for mullite formation at lower temperature in the clay. Parallel investigations in the laboratory have shown a formation of 45% mullite in the washed clay when fired at 1400°C for 24 hr.[12]

A comparison of the properties of the test bricks made from the washed clay with those of the imported tank blocks is shown in Table 2. It is found that these bricks are comparable with the imported glass tank blocks. Further studies on the making of glass tank blocks under various conditions and resistance of these blocks to attack by various glasses is in hand. The clay is also being studied for the formation of mullite at different firing temperatures and times. The economic feasibility of the proposed project of making glass tank blocks from washed clay will be fully discussed after the completion of studies on a pilot plant scale which have already been started.

## Conclusion

The K.D. Fire Clay No. 1 from Mianwali district is a kaolinite type plastic clay which shows considerable improvement in its composition on water washing. Test bricks made under carefully controlled drying and firing conditions from this clay are comparable to the imported ones. This clay, therefore can be successfully used as a raw material for the manufacture of glass tank blocks.

**Acknowledgement.**—The authors are thankful to Mr. M. Aslam, Director of these Laboratories for providing the facilities to carry out this work. They also acknowledge with thanks the assistance extended by Mr. Mushtaq Ahmed, in making and testing of the test bricks.

## References

1. M. Ashraf. M.H. Qureshi and M.R. Ariff, Pakistan J. Sci. Ind. Res., **8,** 256 (1965).
2. M.A. Naz, A.A. Naqvi and M. Safdar, Pakistan J. Sci. Ind. Res., **7,** 174 (1964).
2a. F.A. Faruqi, Pakistan J. Sci. Ind. Res., **9,** 146 (1966).
3. Chesters, *Steel Plant Refractories* (The United Steel Co. Ltd., 1957). Second edition, p. 668.
4. Lunge and Keane, *Technical Methods of Chemical analysis*, Vol. III. Second edition. p. 15.
5. J.M. Hunt and D.S. Turner, Anal. Chem. **25,** 1169 (1963).
6. G.A. Looms, J. Am. Ceram. Soc., **2,** 393 (1938).
7. *Chesters, Steel Plant Refractories* (The United Steel Co. Ltd., 1957) Second edition. p. 681.
8. ibid, p. 688
9. ibid, p. 689
10. J.M. Hunt, M.P. Wisherd and L.C. Bonham, Anal. Chem., **22,** 1490 (1950).
11. F.H. Norton, *Refractories* (McGraw-Hill Book Co. Inc., 1949). Second edition. p. 176.
12. M.A. Rana, M. Aslam, and M. Siddique (unpublished work, P.C.S.I.R. Laboratories, Lahore).