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ASSESSMENT OF PHYSICAL PROPERTIES OF SOME SELECTED CLAY DEPOSITS IN JIGAWA STATE FOR REFRACTORY APPLICATION

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ABSTRACT
The physical properties of clay deposits located in Mailolo in Hadejia Local Government Area, Gwaram in Ringim Local Government Area and Firimo in Birnin Kudu Local Government Area of Jigawa State were assessed for use as refractory materials. Jigawa State is blessed with abundant deposits of clay, which is mostly used for the production of potteries, mud houses and structures. Samples of clays were collected from the deposits and test specimens were prepared for evaluating their physical properties to determine whether they will be suitable for refractory applications. The results of the assessment showed that Firimo, Mailolo and Gwaram possesses cold crushing strength values of 44.6 kg/cm^2, 281.13 kg/cm^2 and 223.6 kg/cm^2 ; bulk strength values of 1.93 g/cm^3, 1.73 g/cm^3 and 1.41 g/cm^3; moisture content values of 2.20% , 3.70% and 4.30%; percentage clay content values of 32.83%, 64.66% and 83.12% ; water absorption values of 3.00% ,4.00% and 5.00% and refractoriness values of 1250^{0}C, 1200^{0}C and 1200^{0}C respectively. Due to their low refractoriness, thermal shock resistance and apparent porosity the clays will only be suitable for low temperature refractory applications such as insulation, bakery oven lining, patching materials, slag pot and ladles lining etc.

Keywords: *Refractory Material, Clay, Cold Crushing Strength, Refractoriness, Bulk Density*

1. INTRODUCTION

Nigeria is highly in need of refractory linings for industrial applications such as melting of materials, heat treatment, power generation to mention but a few (Abaa *et al*, 1989 and Borode *et al*, 2000) . Data bank on some clay deposits in Nigeria showed that Nigeria has a sizeable deposits of clay that may be used for production of refractories (NMDC, 2007). Clay is a general name for minerals containing small plate like crystals, which have been formed from the decomposition of feldspar in igneous rocks, such as granite by the action of air and water over long periods (Heud, 1982).

A major product of this decomposition is the clay mineral kaolinite, a hydrated aluminum silicate. Clay formed from the parent rock may be deposited at its place of origin, and this is known as residual or primary clay, or it may be transported by water and redeposit at some distance and this is known as sedimentary or secondary clay (Grimshaw, 1976, William and Callister, 1978, Keller, 1968). Clay composed of small plate in the crystals that are found because of decomposition of feldspar in igneous rocks (Norton, 1982, John, 1974).

In metallurgical, chemical and ceramic products manufacture, high temperature operation is involved for processing ores and other raw materials. As such, equipment used for treatment of these materials must sustain the operating temperature and other working conditions such as corrosive, erosive and load conditions. Refractories are, therefore, the classes of materials, which withstand high temperatures, resist the action of corrosive liquids and dust-laden currents of hot gasses, etc. (Chseti, 1986, Misra, 1975, Norton, 1982).

Refractories are materials that have the ability of with – standing high temperature without melting or decomposing and the capacity to remain un-reactive and inert when exposed to severe environments. In addition, the ability to provide thermal insulation is often an important consideration. Refractory material is marketed in a variety of forms, but bricks are the most common. Uses include furnace lining for metals refining, glass manufacturing, metallurgical heat treatment and power generation. Refractory materials must also resist the action of molten metals, slogs, glasses, abrasive particles and hot gasses. Refractory materials are classified as acidic, basic and neutral. Acidic refractories are rich in silica, an acidic oxide while basic refractories are composed mainly of magnesite, dolomite and lime. There are many neutral refractories which group includes alumina, fireclays, chrome, carbon and other pure metal oxides such as zirconia (Musickant, 1991).

Some researches on clays showed that Nigerian clays possess properties that are suitable for refractory applications. Atanda et al (2012), investigated clay from deposits in Awo and Ipetumodu areas of Osun State and found out that the clays can be used for the manufacture of refractory bricks of heat treatment furnaces. (Atanda et al, 2012). Kankara clay was investigated as a choice of appropriate local refractory material for furnace lining, the results obtained showed that Kankara fireclay has the required properties for producing grooved bricks for lining of laboratory electric resistance Furnace (Agboola and Abubakre, 2009). Physio-chemical and mineralogical studies of clay samples obtained from the confluence of River Niger and Mimi River in Lokoja for industrial applications were investigated and the Researchers found out that the clays have limited industrial potentials they are only suitable for making building blocks, traditional ceramic pots and insulation bricks (Lawal and Abdullahi). The physical properties of some clay deposits from six locations in Edo, Delta and Borno States were investigated for furnace brick lining (refractory) production, the study revealed that Shuwari clay in Borno State can find application in structural engineering works such as building or burnt bricks, Pulka clay also in Borno State is suitable as construction building materials (bricks) and can be used· in earth dams and water canals. Ijetu clay in Edo State will perform excellently as a general purpose brick for use in reheating furnaces, open-hearth furnace, soaking pits, boilers, kilns etc. Eruemukohwarien clay in Delta State will find application in reheating furnaces, open-hearth furnace doors and checkers, soaking pits, kilns, boilers etc. (Inegbenebor, 2002).

2. MATERIALS AND METHODS

The methods used for evaluating the physical properties of the selected clay deposits include the collection of clay samples from the selected areas and conducting tests to find out the physical properties of the clay samples (Onyemaobi, 2004, Ovri, 1997, Nnuka and Agbo, 2000, Hassan and Adewara, 1993, Yami and Mahdi, 2008) . Samples of clay from the selected locations were collected and prepared for the physical properties evaluation in order to determine the followings: cold crushing strength, apparent porosity, bulk density, shrinkage (volume stability), moisture content, water absorption, clay content, thermal shock resistant and refractoriness.

2.1 Cold crushing strength test determines the maximum load that the sample clay can withstand before failure**.** The apparatus used were electric furnace and hydraulic press. The test pieces were prepared in form of cubes of sizes of 76.2 mm. The pieces were fired in a furnace at 1100^0C for 6 hrs. They were then cooled to room temperature. A compression test was conducted by applying load on the pieces axially at a uniform rate until failure of the pieces occurred.

2.2 Apparent porosity test determines the percentage of apparent porosity of the clay. Porosity is the percentage relationship between the volume of the pore space and the total volume of the sample. Here the volume of liquid or water that the sample can absorb was measured. The pores were broken down by grounding the sample followed by placing 100g in a flask containing 250cm^3 of xylene and the specific gravity was measured. Twelve samples of 25 mm x 25 mm x 25 mm were cut, four from each sample location and each specimen was freed from all loosely adhering particles. They were dried between the temperature range of 105^0C and 110^0C to a constant weight. They were then placed in distilled water and boiled for two hours. During this period, the specimens were kept constantly under water and were not allowed to touch the heated bottom of the vessel. After boiling, the test specimens were cooled to the room temperature by running cold water onto the vessel. The suspended weight of specimen was taken keeping them suspended in water after which were then taken one by one, wiped, dried and weighed in air.

2.3 Bulk density test . Samples measuring 60 mm x 60 mm x 15 mm blocks were prepared and dried in air for 24 hours, after which they were oven dried at 110^0C for 12 hrs and cooled in a desiccators. They were weighed to obtain the dried weight, after which the specimens were transferred into a beaker and heated for 30 minutes to release the trapped air. The specimens were then placed in a distilled water to be boiled for another two hours. They were suspended in water in the heated vessel and then allowed to cool by running cold water into the vessel to obtain the suspended weight. Each sample was brought out and quickly wiped gently to remove excess moisture on the surface to obtain the saturated weight. These three values obtained were used to calculate the bulk density of each clay from the following formula (Misra, 1975)

$$B = \frac{D}{V} \text{ (g/cm}^3\text{)} \quad [1]$$

$$\text{or } B = D \big/ W\text{-}S \text{ (g/cm}^3\text{)} \quad [2]$$

where, B – Bulk density (g/cm^3); D – Dried weight of sample (g); V – Volume of sample (cm^3); W – Saturated weight of sample (g) and S – Suspended weight of sample (g)

2.4 Shrinkage (volume stability) test. Refractory bricks often shrink in use and do not attain the volume stability at high temperature. Refractory with large amount of stability are the best. The shrinkage or volume stability test was conducted by heating a brick to a higher temperature (about 1200^{o}C) or keeping it at a lower temperature (about 100 ^{o}C) for a long time and then measuring the amount of contraction or expansion undergone by the clays. Apparatus used are sieving mesh**,** hydraulic press and furnace. Test pieces were prepared in the form of standard slabs from 500g of clay powder which was sieved through 125 microns mesh sieve with 50 ml of water addition and compressed in hydraulic press. The blocks were allowed to dry until they were strong enough to remove from the mould without damage. They were then air dried at room temperature for 24 hours and finally dried in an oven at 110^0C overnight. The wet to dry shrinkage was calculated as follows: -

$$\%\text{Wet–to–dry shrinkage} = \frac{W_L - D_L}{D_L} \quad [3]$$

where, W_L is wet length and D_L is the dry length of the sample

The firing shrinkage was calculated from Equation 4

$$S_F = \frac{D_L - F_L}{D_L} \quad [4]$$

where, S_F is the firing shrinkage and F_L is the fired length of sample

2.5 Moisture content test. A 500g of powdered clay sample was placed in a special container of speedy moisture tester that has a calibrated scale attached to it. Powdered calcium carbide (10% of the clay) was added and shaken for about 2 minutes with the calibrated side of the container turned upside down and vice versa while shaking. Acetylene gas was produced proportional to the amount of moisture in the sand because of reaction between the clay and the mixed calcium carbide. The moisture content is read directly from that calibrated scale on the instrument.

2.6 Water absorption test. Equipment used to conduct the test were electric furnace, weighting scale and desiccator. Two specimens from each clay sample were cut and loosely adhering particles were wiped off from them. They were dried at 105⁰C to 110⁰C, placed in weighing bottles and the total weight of each was recorded. The weighed bottles with the samples were then placed into a desiccator containing saturated sodium chloride solution. The desiccator was covered and the content was allowed to stand for 24 hours. The samples were then removed and weighed again. The increase in weight (W) which is proportional to the amount of water absorbed as a percentage of the weight of dry materials was calculated as follows (Heud, 1982).

$$A = \frac{W - D}{D} \times 100 \qquad [5]$$

where, A is the water absorption (%), W is saturated sample weight and D is the dried sample weight

2.7 Clay content test. The apparatus used in this test were electric furnace, weighting scale and clay washing machine. Clay samples were prepared and ground into powdered form and then dried in an oven at 110⁰C. 80 grams of each sample was placed in a beaker and washed with a solution of 790 ml of distilled water mixed with 40 ml of 3% sodium hydroxide solution for 5 minutes in the washing machine. Water was used to wash out the particles that stick to the beaker and allowed to settle for about 10 minutes before siphoning the suspended solution off. This is repeated until the added water is clear. The weight of the minerals that remain multiplied by two will give the American Foundry Society clay percentage.

2.8 Thermal shock resistant test. Apparatus used in the test were furnace, observing lenses and quenching medium. Samples of the clays were prepared and placed for 10 minutes in the furnace set at 1200⁰C. They were removed from furnace and cooled outside the furnace for 10 minutes and the samples were observed for cracks. This process was repeated until cracks were observed on the samples. The number of heating and cooling (cycles) before cracking occurred was recorded and this constitutes the thermal shock resistance.

2.9 Refractoriness test. The apparatus used in the test were oxidizing furnace, grinding machine, sand mixer, molding machine, mesh and weighting scale. Pyrometric Cone Equivalent (PCE) method was used to determine the refractoriness of the clays. Clay samples were ground to a size of 30 mesh sieve and dried. A green binder (alkaline free dextrin) and water were added to the clays and mixed on a glazed ceramic tile until they were moldable. The mixture was then formed into cones with a shape of tetrahedron measuring 8 mm on the sides of the base and 25 mm height in metallic moulds. The moulded samples were dried on a hot plate for 10 – 20 minutes so that they can be handled. The cones were then mounted with their base embedded approximately 3 mm deep in a plague. The samples were arranged around the outer edge of the plague with standard cones in between them. The furnace is heated until the top of the cone bend over and its tip touching the plague surface.

3. RESULTS AND DISCUSSION

3.1 Results

Table 1: Results of physical properties of the clays

S/N	Properties parameter	Firimo clay	Mailolo clay	Gwaram clay
1	Cold Crushing Strength (kg/cm^2)	44.6	281.13	223.6
2	Apparent Porosity (%)	26.39	25.58	30.96
3	Bulk Density (g/cm^3)	1.93	1.73	1.41
4	Shrinkage (%)	1.00	4.70	3.00
5	Moisture Content (%)	2.20	3.70	4.30
6	Water Absorption (%)	3.00	4.00	5.00
7	Clay Content (%)	32.83	64.66	83.12
8	Thermal Shock Resistance	5 circles	8 circles	Cracked before test
9	Refractoriness (⁰C)	1250	1200	$\angle$ 1200

3.2 Discussion

3.2.1 ***Cold Crushing Strength*****.** The cold crushing strength of a refractory material represents its strength or show how strong the materials are. Figure 1 shows that Mailolo clay has the highest cold crushing strength while Firimo clay has the lowest value of cold crushing strength, this may be because of Mailolo's clay has small apparent porosity value which indicates that Mailolo clay will have the best abrasion resistance among the three clays. But the three clays have low cold crushing strength since the minimum requirement of cold crushing strength for refractory brick is 15000 kg/cm^2 (Brown, 1998).

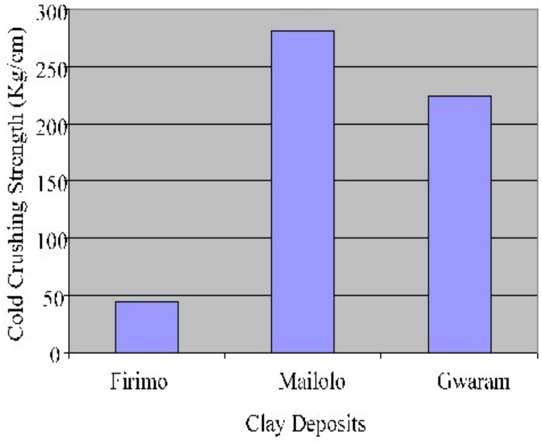

Figure 1: Cold Crushing Strength of the clays

3.2.2 *Apparent Porosity.*

Porosity is the percentage relationship between the volume of the pore space and the total volume of the sample. Porosity is apparent if a volume of liquid absorbed by a sample is noted without considering the sealed pores of the sample. Lower porosity gives greater resistance to slag attack and more sensitiveness to fluctuation in the temperature. It can be seen in Figure 2 that Mailolo clay has the lowest apparent porosity value among the clays which showed that it will have the highest resistance to slag attack and will be more sensitive to temperature fluctuation compared to the other clays. Gwaram and Firimo clays have values that falls within the accepted range of apparent porosity for refractory materials (Misra, 1975)

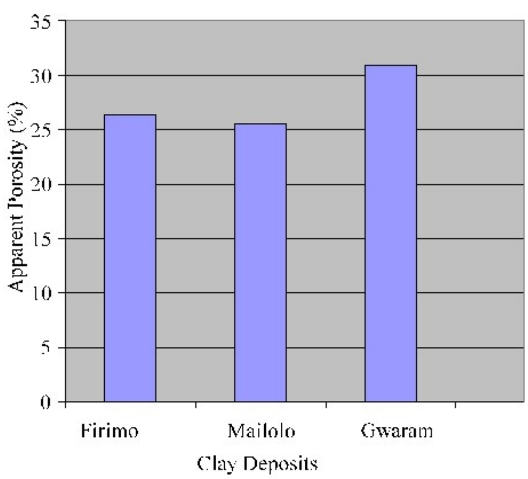

Figure 2: Apparent Porosity of the clays

3.2.3 ***Bulk density***. Figure 3 shows the comparison of the clays' bulk densities. The three clays have bulk density values that falls within the range of 1.73 to 2.90 g/cm^3 required for refractory materials (Misra, 1975). The density of Gwaram clay falls within the range for fire clay which has bulk density of 1.98 g/cm^3 while Mailolo clay's density falls within the range for silica hard fired clay which has bulk density of 1.73 g/cm^3. An increase in bulk density increases the volume stability as well as the resistance to abrasion and slag penetration.

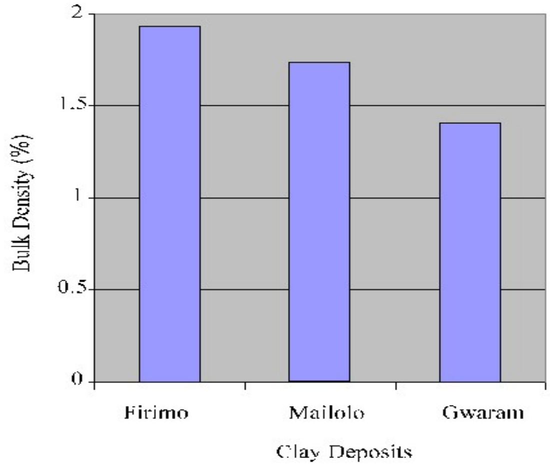

Figure 3: Bulk Density of the clays

3.2.4 ***Firing Shrinkage***. Volume stability (Dry / Firing Shrinkage) was conducted to find out the behavior of the sample clay when exposed to certain changes in temperatures. Refractory materials shrink while in use at high temperature. During the test, it was observed that there was no volume change in the drying shrinkage of the clays but some changes were observed during firing shrinkage (as shown in Figure 4). Mailolo clay shrink by 4.7% due to its low porosity and Firimo clay by 1% while Gwaram clay expanded by 3% instead of contracting which may be due to its high calcium content (1.12% of CaO higher than others). Since refractory with large amount of stability are the best, therefore Mailolo and Firimo clays will be more stable than Gwaram clay.

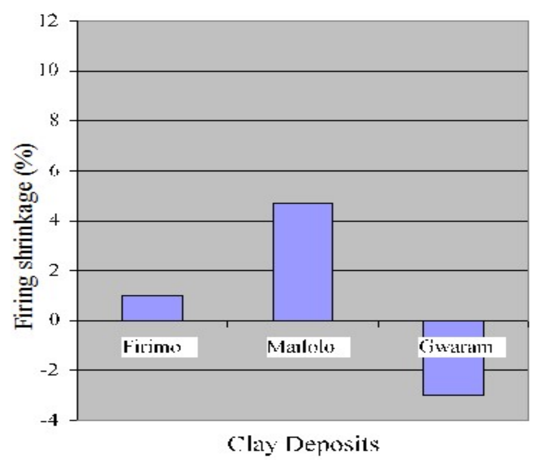

Figure 4: Firing Shrinkage of the clays

3.2.5 ***Moisture Content.*** The assessment of the amount of moisture in the clay was conducted to find out how this moisture can assist in binding of both organic bricks and in organic binder to form the refractory brick. The moisture content increases with increase in clay content, because clay absorbs more water to about four times its own weight. Figure 5 shows that Gwaram clay has the highest moisture content among the clays which indicates that it will have the best binding properties.

Figure 5 Moisture Content in the clays

3.2.6 ***Water Absorption***. Unlike the moisture content, which is the amount of moisture within clay, water absorption is the ability of the material to absorb moisture. However, water absorption is like the moisture content where the percentage of the water absorb depend on the percent of clay content i.e. the higher the clay content, the higher also is the percentage of water being absorbed. It can be seen from Figure 6 that Gwaram clay will absorb more water (5%) than Mailolo (4%) and Firimo (3%) clays.

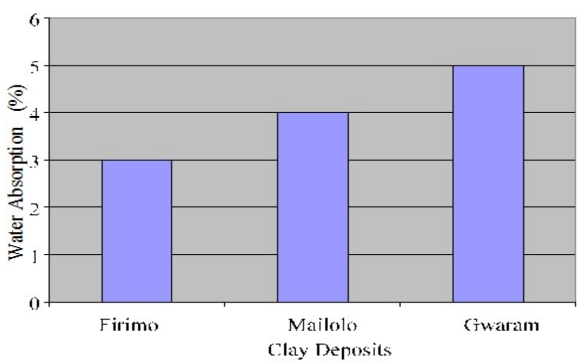

Figure 6: Water Absorption of the clays

3.2.7 ***Clay Content***. Pure clay may be highly refractory, but it may be very weak when heated due to the absence of suitable element, which, if present, would form a verifiable bond to unite the clay particles into a mass of great strength. It can be seen from Figure 7 and Table 8, that Gwaram clay has the more clay content (83.12%) than Mailolo (64.66%) and Firimo (32.83%) clays. It is an indication that Gwaram clay will have good binding properties and strength among the clays.

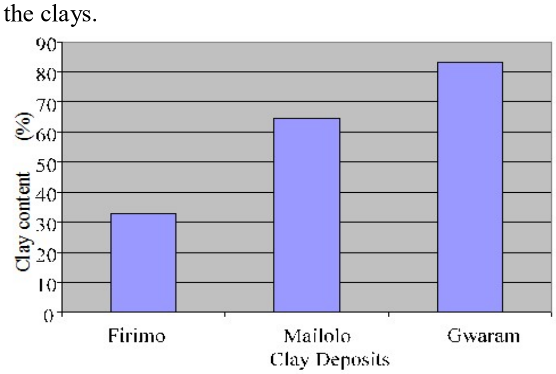

Figure 7: Clay Content of the clays

3.2.8 **Thermal Shock Resistance**. Refractory bricks are expected to withstand different changes in temperature such as heating at elevated temperatures and cooling at low temperature. The test was conducted to investigate the behavior of these clays when subjected to these conditions. As it can be seen from Table 10 and Figure 8, Mailolo clay resist more shock (8 circle) than Firimo (5 circle), but Gwaram clay could not even withstand the test temperatures. Since the number of cycles undergone by clay without cracking is the measure of its thermal shock resistance, Mailolo clay will have the highest thermal shock resistance among the clays.

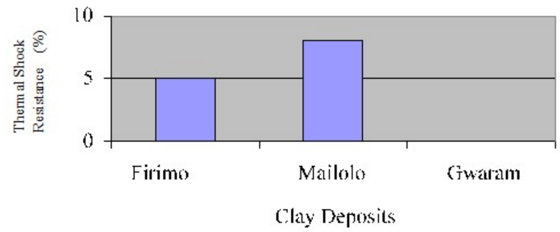

Figure 8 Thermal Shock Resistant

3.2.9 **Refractoriness.** A material is said to be a refractory if it has a high value of heat resistance that fall within the approximated value [range between 1600 – 1770^{0}C) that is internationally accepted for some refractory bricks (Misra, 1975)]. The result obtained in Table 9 and Figure 9, showed that none of the clay has a value that falls within this range, this indicates that none of them can be used as refractory bricks. Since they all have low refractoriness values, they can be used only for low temperature applications.

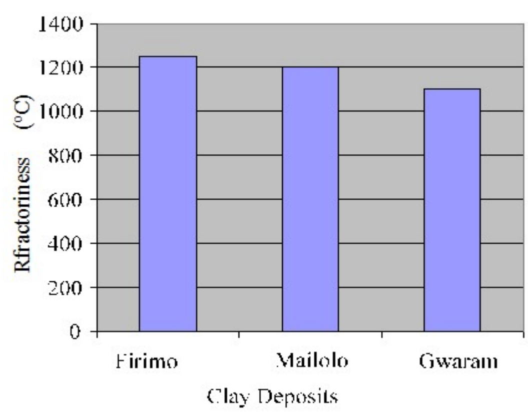

Figure 9: Refractoriness of the clays

4. CONCLUSION

The physical properties of clay deposits in Mailolo in Hadejia LGA, Gwaram in Ringim LGA and Firimo in Birnin Kudu LGA all in Jigawa State were evaluated for use as refractory materials. The results showed that the clays have low cold crushing strength values (44.6Kg/cm, 281.13 Kg/cm^3 and 223.6Kg/cm^3) which is less than the minimum requirement of 1500Kg/cm^2 for firebricks. The clays possess the required apparent porosity values of 20-30% for firebricks. But none of their bulk densities meet the required bulk density of 2.3g/cm^3 for refractory clays. The clay showed no volume changes when dried under 110^0C, but some changes were noted when fired dried. Firimo and Mailolo clays shrink by 1 and 4.7% respectively, while Gwaram clay expand by 3% when heated meaning that Mailolo and Firimo clays will be more stable than Gwaram clay when exposed to temperature fluctuation. The thermal shock resistance of the clays falls between 0 to 8 circles, which is less than the minimum of 20 circles required for firebricks which limits their usage. None among the three clays have the standard requirement of not less than 1500^0C for refractoriness of fireclay, therefore, they will be limited to lower temperature applications.

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