JEAS

Journal of Engineering and Applied Sciences 3 (2007) 27 - 30

JOURNAL OF ENGINEERING
AND

Effect of porosity on the shrinkage behaviour of Ukpor and Nsu clays

C. I. Nwoye

Department of Metallurgical and Materials Engineering, Federal University of Technology, Owerri, Imo State, Nigeria

---

Abstract

The paper describes the effect of porosity on the shrinkage behavior of clay, pellets and briquettes of different porosities. The result of the investigation indicates that shrinkage which is a major cause of rupture in fired clay in creased with decrease in porosity. It was also discovered that the porosity of pellet/briquette plays important role in controlling and determining the shrinkage index of the pellet.

Keyword: Effect; Porosity; Shrinkage behaviour; Ukpor and Nsu clays

---

1. Introduction

According to Barsoum (1997), the contents of the basic clay materials are divided into three groups. The first group involves clays containing mainly the mineral kaolinite. The second groups are clays containing mineral rnontmorillorite, while the third group are clays which are intermediate product of disintegration of mica into kaolin. The structure of sinters and pellets may be divided into two parts viz, the mineral and the pores. It is widely accepted that the properties of pellets and sinters are closely related to the mineral constituents (Unal, 1986). Voids volume in packed dispersed powder according to Fumass (1928), depends on the ratio of smallest size (S_s) to largest size (L_s) particle as well as the percentage of constituent monosized particles. He maintained that the smaller the (S_s/L_s) ratio, the more continuous the distribution and the lower the void volume of the system. Arisa (1997) stressed that the behaviour of ceramic product are very dependent on their composition, grain size, grain distribution, structure of grain and pores. Nwoye (2003) also posited that the grain size and grain distribution of the clays have significant effect on their physical and technological properties (binding ability, shrinkage and plasticity).

Pores are usually deleterious to the strength of ceramics not only because they reduce cross-sectioned area over which the load is applied but more importantly act as stress concentrators (Barsoum,1976).

Viewey and Larrly (1978) in an intensive study of the relationship between particle size and linear drying shrinkage revealed that there is no visible relationship existing between them. They also stated that the finer the particle size, the lesser the apparent porosity and the greater the bulk density.

Singer and Singer (1963) pointed out that on heating dried clay, water is given off with time and a hard porous piece forms. They also stated that a swollen appearance might occur during the release of some gases; but overall shrinkage must occur when vitrification set in leading to a strong dense piece. Drying according to Reed, (1988) occurs in three stages; increasing rate, constant and decreasing rate. He pointed out that during the increasing rate, evaporation rate is higher than evaporating surface hence more water is lost. At constant rate, the evaporation rate and evaporation surface are constant. Shrinkage occurs at this stage. Keey (1978) suggested that at this stage, free water is removed between the particles and the interparticle separation decreases, resulting to shrinkage. During the decreasing rate, particles make contacts as water is removed, which causes shrinkage to cease.

Reed (1988) described firing as having three stages through which it proceeds; preliminary reactions which include binder burnout, elimination of gaseous product of decomposition and oxidation, sintering as well as cooling which may include thermal and chemical annealing.

Several works have been done on shrinkage of clay (Singer and Singer, 1963; Reed, 1988; Keey, 1978; Barsoum, 1997) but none of them investigated the effect of porosity on the swelling and shrinkage behaviour of clay. Porosity is known to influence the swelling and shrinkage behaviour of clay sinters and pellets. Chemical composition of the pellet, pelletisation parameters and firing conditions have been found to affect the shrinkage of clay pellets Nwoye (2003). He posited that the rate of chemical reaction is very much dependent on the gas-solid contact area, which is mostly governed by the porosity of the pellet. He also stated that the shrinkage of clay is probably due to volume change resulting from evacuation of water from the voids, reduction of the size of the pores as well as decrease in the interparticle separation.The present work is an attempt to investigate the effect of porosity on the shrinkage behaviour of Ukpor and Nsu clay.

2. Materials and methods

All the clays used were collected in lumps from deposits. The lumps of the clays were allowed to dry in air for four days. Each of these clay samples were crushed and sized to a fine particle size of less than 100µm using an assembly of sieves and test sieve shaker. Each sample was manually homogenized separately in a mixing drum using 2% starch as binder. Samples were thoroughly mixed with water (6% of the total weight of dry (materials).

The mixture was then rolled into balls is a pelletizing drum. The porosities of these samples (green pellets) were determined by the following equation.

$$\xi = 1 - P_b/P_t \tag{1}$$

Where P_b is the bulk density of the sample ie the ratio of the sample weight to volume, g/cm^3. The sample volume was calculated with a micrometer putting into consideration the sizes measure. $P_t = 1/(X_c/P_c + X_s/P_s)$ is the total density of the sample, g/cm^3, p_c and p_s are true densities of clay and starch particles respectively measured by grinding the particles to - 45µm (80%) and using a density bottle and kerosene (ASTMC -188 - 44), X_c and X_s are the proportions of clay and starch respectively. On the foregoing, G_c and G_s will refer to the particle sizes of clay and starch respectively.

2.1. Moulding

The plastic clays were kneaded using hand to expel any trapped air from the clays. The samples were moulded in rectangular mould of dimension 70mm by30mm and dried under the laboratory temperature condition (25°C) for 18hours after which they were carefully packed in saw-dust to prevent them from cracking and absorbing moisture from the surrounding. These samples were then fired using electric kiln. The samples were charged at lower temperature (125°C), after which the temperature was increased to 1200°C. These samples were fired for 18 hours and then cooled in the furnace for the same time limit. The end of sintering was taken as the moment when the temperature of gases reached maximum.

As it was discovered, that reaction between clay, water and starch particles had already occurred during the heating period, the sintering time which will be referred in the following means the time from the moment the sample was charged into the kiln to the moment the exhaust gas temperature was maximum, the heating period inclusive.

2.2. Pelletizing and Briquetting

In order to determine the effect of porosity on the shrinkage behaviour of the clay pellets and briquettes, two different clay samples from Ukpor and Nsu deposits were taken. These clays were first heated in the laboratory oven to 120°C for one hour to expel any moisture and any other matter present. The particle size of the clay samples was -125µm and the specific surface area was around 1700cm^2/g. The two clay fines were rolled to give 12 to 13mm diameter pellet using 10 - 11% moisture. Since it was difficult to prepare pellets with low porosity (below 20%) cylindrical shaped briquettes (12mm diameter x 14mm height) were made from the clay fines by compaction, and marks 70mm and were made on each sample. For this purpose steel die and punch were used. To also know the effect of shape of the agglomerate, briquettes having higher porosities were made under different compaction pressure: The samples were allowed to dry under the laboratory temperature condition for 18 hours. These briquettes and pellets were fired at 1200°C for 24 hours and the porosity of each pellet and briquette was measured by mercury porosimeter.

The shrinkage behaviors of the pellet was studied using 45mm inner diameter and 1000mm long stainless steel tube having water cooled quartz windows at both ends as the reaction chamber Sharma, et al. (1990).

For determining the shrinkage index, a pellet /briquette of known dimension was kept inside the furnace and a photograph of the pellet was taken with the help of an external light source and the volume of pellet was calculated by projecting the pellet photograph on a screen and measuring the average diameter before and after the shrinkage. The shrinkage index of the pellet during heating was calculated as

$$\text{Shrinkage Index} = 100 \, x \left(\frac{v_i - v_f}{v_i} \right) \tag{2}$$

where v_i = Initial volume of pellet
v_f = volume of pellet after sintering

Also during drying of the pellet; the change in volume has been referred as "Drying Shrinkage" (DS). During preheating of the pellet the change in volume has also been referred as Thermal Shrinkage" (TS). During intense firing (when the temperature was increased rapidly to 1200^0C) the volume of pellet reduces with firing time and the change in volume is referred to as "Firing Shrinkage" (FS), while the maximum value of shrinkage index has been referred as "Maximum Shrinkage" (MS). Hence

$$M_s = D_s + T_s + F_s \qquad (3)$$

The fired shrinkage (f_s) of the briquette is calculated as

$$F_s = \frac{L - L_2}{L} \qquad (4)$$

where F_s = fired shrinkage
L = original length (mm)
L_2 = fired length (mm)

3. Results and discussion

The chemical composition of the raw materials is given in Table 1.

Successful preliminary studies on the effect of initial porosity of raw samples on the pore structure after sintering was carried out as it was widely believed that the pores might arise mainly from the interparticle voids (Kinger, et al, 1976). Samples of different porosities were prepared from Ukpor clay fines of sizes less than 1mm and starch (-1mm). These materials were mixed with water of known quantity, cold pressed and dried under laboratory temperature prior to sintering. As expected, it was discovered that the lower the initial porosity of the raw sample, the lower the final porosity and also the smaller the average pore size.

Table 1
Chemical composition of raw materials

Source:	Al_20_3	Fe_20_3	$T10_2$	MgO	CaO	$S1O_2$	Na_2O	K_2O	Loss of ignition
Model	38.52	0.50	0.16	0.08	0.08	45.44	0.66	0.14	13.60
Ukpor	31.34	0.63	2.43	0.14	0.06	51.43	0.04	0.10	12.04
Nsu	25.61	1.74	1.40	0.78	0.54	59.29	Traces	0.23	9.95

Table 2
Effect of porosity on the shrinkage index for pellets and briquettes of model clay (for 20% porosity)

Material: Degree of firing %	Briquette Shrinkage index %	Material: Degree of firing %	Pellet 1 Shrinkage index %	Material: Degree of firing %	Pellet 2 Shrinkage index %
0	26	0	26	0	26
25	37	25	30	25	28
50	70	50	60	50	54
75	90	75	81	75	64
80	98	80	89	80	73
90	94	90	87	90	70
95	94	95	86	95	68
100	92	100	87	100	68

Table 3
Effect of porosity on the shrinkage index for pellets and briquettes of Ukpor clay (for 20% porosity)

Material: Degree of firing %	Briquette Shrinkage index %	Material: Degree of firing %	Pellet 1 Shrinkage index %	Material: Degree of firing %	Pellet 2 Shrinkage index %
0	26	0	26	0	26
25	37	25	30	25	28
50	70	50	60	50	54
75	90	75	81	75	64
80	98	80	89	80	73
90	94	90	87	90	70
95	94	95	86	95	68
100	92	100	87	100	68

Table 4
Effect of porosity on the shrinkage index for pellets and briquettes of Nsu clay (for 20% porosity)

Material: Degree of firing %	Briquette Shrinkage index %	Material: Degree of firing %	Pellet 1 Shrinkage index %	Material: Degree of firing %	Pellet 2 Shrinkage index %
0	18	0	18	0	18
25	24	25	23	25	22

50	46	50	39	50	36
75	69	75	57	75	50
80	74	80	65	80	58
90	71	90	62	90	52
95	69	95	60	95	48
100	67	100	60	100	48

Table 5
Relationship between porosity and fired shrinkage for model, Ukpor and Nsu clay

Material: Source: Porosity %	Briquette Ukpor Fired shrinkage %	Material: Source: Porosity %	Briquette Nsu Fired shrinkage %	Material: Source: Porosity %	Briquette Model Fired shrinkage %
19.90	11.20	23.67	8.57	24.14	8.40
20.50	10.94	24.16	8.40	24.64	8.23
20.88	10.74	24.40	8.33	24.89	8.16
21.34	10.47	24.55	8.17	25.04	8.01
21.90	10.03	24.68	8.03	25.17	7.87

Also the pores were found to be more closed when porosity was lower. These results are in accordance with common knowledge.

To establish the effect of porosity on the shrinkage behavior of clay pellets and briquettes, fired pellets and briquettes were made from model clay sample relatively free from impurities and two natural clays (Ukpor and Nsu clays) and were subjected to shrinkage test at 1200°C under Argon gas flowing at the rate of 1.54/mm and the results are shown in Tables 2 to 4. From the tables, it is seen that the shrinkage index of pellet/ briquettes increases with increase in degree of firing until an optimum firing is reached after which the shrinkage index drops. In all these values for all the pellets of different porosities, maximum shrinkage is obtained at about 80% firing. The firing shrinkage (F_s) when calculated against porosity values, results into an inverse relationship for briquettes/pellets prepared from the three clay samples as shown in Table 5. The shrinkage values for varying porosities do not seem to be affected by agglomerate geometry or origin of the material but it is dependent on the porosity of the agglomerate.

4. Conclusions

(1) Porosity of pellet/briquette plays important role in controlling and determining the shrinkage index of the pellet.
(2) Shrinkage maintains an inverse relationship with the porosity of the pellet/briquette
(3) The shape of agglomerate has negligible effect on the shrinkage index.

Acknowledgement

The author thanks Christy Nwoye and Osita Iheanacho of Ashmonvaly Research Institute, Port-Harcourt for their assistance during the project. He also glorifies the Almighty God for the thinking cap.

References

Arisa, U. S., 1997. Effect of Initial Particle Size and Size Distribution on Physical and Mechanical Properties of some Nigeria Clays Sintered at 1200^{0}C. A thesis reports p.3,15-18.

Barsoum, M., 1997. Fundamentals of Ceramics. McGraw Hill Inc., Singapore, p. 410-420.

Furnass, C.C., 1928. The Relationship between Specific Voids and Size of Compression In System of Broken Solid of Mixed Size. US Bureau Report Investigation, Vol. 2894, p. 321-340.

Keey, R. B., 1978. Introduction to Industrial Drying Operations. Pergamon Press, Elmsford, New York, p. 132-157.

Nwoye, C. I., 2003. Investigating the Influence of Particle Size And Size Distribution On The Physical And Mechanical Properties Of Ceramic Materials. A Project Report p. 5-8,16-22.

Reed, J., 1988. Principles of Ceramic Processing. Wiley Interscience Publication, Canada, p. 470.

Sharma, T., Gupta, R. C., Prakash, B., 1990. Miner, England.

Singer, F., Singer, S.S., 1963. Industrial Ceramics. University Press, Cambridge, p. 44-70.

Unal, A., (1986): Trans Institute of Metallurgy, section C. 95, 107.

Viewey, F., Larrly, P., 1978. Ceramic Processing before Firing. John - Wiley and Sons, New York p. 80.