Effect of Adding Mgo & Al_2O_3 on Ceramic Body Prepared from Iraqi Activated Bentonite

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Abstract

In this work, Iraqi bentonite clay (Al-Safra bentonite region) has been modified to convert it from calcium bentonite to sodium bentonite, by using Na_2CO_3 at concentrating ratios wt% (5%) under limited conditions of activation, extraction and drying. Ceramic bodies have been prepared from Al-Safra activated clay with Alumina: Magnesia additives, (sodium bentonite 64.39% + alumina 29.13% + magnesia 6.39%). By using forming pressure (100 MPa), the samples have been formed as a disc shape and then treated by using firing temperature (1200 ºC, 1250 ºC, 1300 ºC). The X-ray diffraction results have showed that the exist main phases are: Crestobelite, Mullite, Cordierite and Corundum phase. The most important achieved results are: First Al-Safra activated with the additives (alumina and magnesia) underwent firing temperature at (1300 ºC) without any distortion in the appearance and gave the maximum compression strength to 60.41 MPa , which is higher than of bentonite (as based material) which is about 6.2 MPa. In addition, the best results of the apparent porosity (0.69%), bulk density (2.239 gm/cm^3), and water absorption (4%).

Keywords: MgO, Al_2O_3, Bentonite, Mechanical properties, Thermal properties.

1. Introduction

The field of ceramic science could be classified as traditional and advanced ceramics. Traditional ceramics are those made from naturally occurring materials like clays, porcelain and pottery, characterized by mostly silicate-based porous microstructures non-uniform and multi-phase [1, 2]. Advanced ceramics developed by synthesis, they are man made and can be made from highly refined naturally occurring materials [3].

Cordierite-Mullite ceramics

Cordierite ($2MgO.2Al_2O_3.5SiO_2$) and mullite ($3Al_2O_3.\ 2SiO_2$) represent technically important ceramics which are applicable in a variety of areas. The cordierite ceramic due to its low dielectric constant, high chemical durability, high resistance to thermal shock and have very low thermal expansion coefficient [4, 5]. Mullite is used as structural materials due to its excellent mechanical properties even at high temperatures. By formulating cordierite-mullite is possible to improve the mechanical behavior and thermal properties [6]

Bentonite clay

The name bentonite was suggested initially to the plastic clays. They are composed predominantly of smectite clay minerals, and it is usually sodium and calcium montmorillonite [7].

Ca-bentonite is treated with some inorganic materials like Na_2CO_3 to convert it to the sodium form, because of that Na-bentonite is preferred for most of the industrial applications [8].

2. Materials and methods

The sodumizing modification of Ca-bentonite

Fractions smaller than 38μm of Al-Safra Iraqi Ca-bentonite were used. The chemical analysis of this bentonite is given in table (1). The X-ray diffraction of the original and activated bentonite (figure. 1) were obtained with XRD unit model (7000), target Cu, (λ) =1.5405 ºA, 40 Kv, 30 mA .

Table 1. Chemical analysis of ca-Bentonite sample

Material oxide	*SiO_2*	*Al_2O_3*	*Fe_2O_3*	*CaO*	*Na_2O*	*MgO*	*SO_3*	*L.O.I*
Bentonite (wt%)	*53.08*	*4.52*	*14.74*	*0.93*	*7.56*	*3.60*	*1.63*	*1.11*

Al-Safra bentonite was activated with Na_2CO_3 at the mass ratio (5%) labeled (S5). Bentonite and Na_2CO_3 were add to distilled water until we get a slurry, the slurry was heated and mixed for 60 min by using mechanical stirrer, then cooled with water quickly, left for 24h, they mixed with a suitable amount of Ethanol (1:4 Ethanol: Water), and centrifuged (6000 rpm, 3 min) by using (LABOFUGE-HERAEUSE) , and dried in an oven at 100°C . Then the dried samples were ground by ball mill to reduce the particle size < 53 μm.

Ceramic body preparation

For the purpose of this study, the percentage of cordierite-mullite at 70:30 wt% was chosen, which showed a good results in both physical and thermal properties in the case of pure oxides(commercial powders of cordierite and mullite) [9].

The percentage of (SiO_2 57.72 wt%, Al_2O_3 27.34 wt%, MgO 14.92 wt%) which corresponds approximately to the cordierite(10), and the percentages of about ($Al_2O_3$70 wt%, SiO_2 30% wt%) which corresponds to mullite were taken in this study. The mixture percentages after calculation to follow the above requirement were listed in the table (2).

Table 2. Mixture percentage for the sample

Sample of Bentonite	*Bentonite wt%*	*Al_2O_3 wt%*	*MgO wt%*
S5	*64.48*	*29.13*	*6.39*

The samples are formed by mixing their materials by wet method, then drying at 100ºC, followed by crushing to produce an intimate mixing of the raw materials. The crushed samples (4.5 gm for each) were placed in a steel die (of 25 mm in diameter and 3 mm in thickness) and compacted at 100 MPa for one minute. Then sintered by using electric muffle furnace at heating rate 7 ºC/min up to the firing temperature of about (1200, 1250, 1300) ºC, then the samples were left inside the furnace to cool down to room temperature.

Physical measurements

Measurements of apparent porosity, water absorption and bulk density

Archimedes method was used to determine the apparent porosity (A.P), water absorption (W.A) and bulk density (B.D) respectively according to the following equations [10]:

$$A.P(\%) = \frac{W_s - W_d}{W_s - W_{su}} * 100 \qquad W.A(\%) = \frac{W_s - W_d}{W_d} * 100 \qquad (B.D) = \frac{W_d}{W_d - W_{su}}$$

where:

W_d: Weight of the dry sample after sintering (g).

W_{su}: Weight of the sample after immersing it in a distilled water and suspended in air through a balance (g).

W_s: Weight of the sample after immersing it in distilled water for 24hrs (g).

Mechanical measurements

Compression strength

Compression strength was carried out by using the diametrical compression disc test (Brazilian test) where the disc was placed between two surfaces and applied the load until fracture occurs [11], the force obtained at fracture was recorded, then the following equation was applied to calculate the strength of the materials:

$\sigma f = 2p/\pi TD$ where σf: fracture strength (N/mm ^{2}).

p: applied load (N).

D: sample diameter (mm).

T: sample thickness (mm).

Thermal properties

Thermal conductivity

The apparatus used was a modification of the standard Lee's disk method for the measurement of thermal conductivity, whose schematic diagram is represented in figure (2). The sample to be studied is approximately at the same diameter as the copper discs A & B, which are 2.5 cm. The heat is supplied from heating coil inserted between disc B and C with equal diameter after tightening the clamp screw to hold all the discs together; the power to the heater was switched on. The whole assembly was placed in an enclosure to minimize the effects of draughts. The supplied current (I) from D.C supply was constant for all measurements and its value was 0.25 ampere and the D.C voltage (V) was 6 volts, which was constant for all measurements too. The reading of discs temperature was taken every 10-15 minutes, as equilibrium was reached when the temperatures of all parts of the apparatus have been stable; the value for the thermal conductivity (K) of the specimen was calculated from the following eqs. [12].

$$K = e[TA + (2/rd)\ (dA + ds/2)\ TA + (dsTB/rd)]/((TB - TA)ds)$$

$$e = VI/[\pi rd2\ (Tc + TA) + 2\pi rd\ \{dATA + ds\ ((TA + TB)/2 + (dB + TB) + dcTc\}]$$

where TA, TB and Tc: temperature of discs A, B and C respectively in (k).

DA, dB and dc: diameter of disks A, B and C respectively in (m)

rd: sample radius (m)

ds: sample thickness(m)

e : the emitted energy (heat) per unit area per unit time in (W/m^2).

Results and discussion

Physical properties

Figure (3) and figure (4), illustrate the behaviors of porosity and water absorption for the samples with respect to firing temperature at (1200ºC-1300ºC). The figures showed an increase in porosity and water absorption at firing temperature from 1200ºC to 1250ºC. Then the values were decreased sharply at 1300ºC. This behavior is related to the incomplete reaction between the component of starting materials which leads to reduce the amount of the liquid phase that required to fill the open pores and holes, as shown in the optical microscopic images (figure 6). While when the firing temperature increased to 1300ºC, a sufficient fusion for the ceramic body components has been occurred, this leads to an increase in the glass phase formation that decreases the amount of open pores on the body surface. Figure (5) shows the increasing of the bulk density with respect to the increasing of the firing temperatures for the fired samples. This due to the initiation of crystalline phase mullite ($\rho_{mullite}$= 3.167gm/cm^3) and corundum phase ($\rho_{corundum}$= 4.1gm/cm^3) in the liquid phase, as shown in the related X-ray diffraction patterns (figures 7 to 9).

Mechanical properties

The compression strength increased with increasing the firing temperature to 60.4 MPa at 1300˚ C, which represents the maximum value of the compression strength results, as shown in the figure (10). The results of compression strength for (S5) is higher than (S0, about 6.2 MPa) because of the activation ratio (i.e., the ion exchange for Ca^{+2} by Na ion, where Na_2O considered to be a useful flux at low-fired glazes but in high-fired glazes, CaO may be the principle flux[13]. This means increasing in the fluxing ions ratio. These ions when present, lower the melting point of the system and particular, react with silica at high temperatures (as low at 1000˚C) to form a viscous liquid, which when cooled does not crystallize completely but solidifies to form a glass, which is responsible for the verification and fired strength. [13].

Thermal properties

The thermal conductivity results for Al-Safra activated bentonite (S5) have showed a decrease in its value, as shown in the figure (11). This can be attributed to an increase in the amount of glass phase formation with increase the firing temperature at 1300 ºC (a glass has a low heat conductivity, 1.33 W/m.K) which makes the phonons more diffracted. Moreover, the presence of closed porous inside the ceramic body at heat treatment to 1300˚C, as shown in figure (6). Herewith, the amount of air phase which considered to be as obstructed points to the heat conductivity, influences the thermal conductivity. This means that materials with high porosity are generally good heat insulators [14].

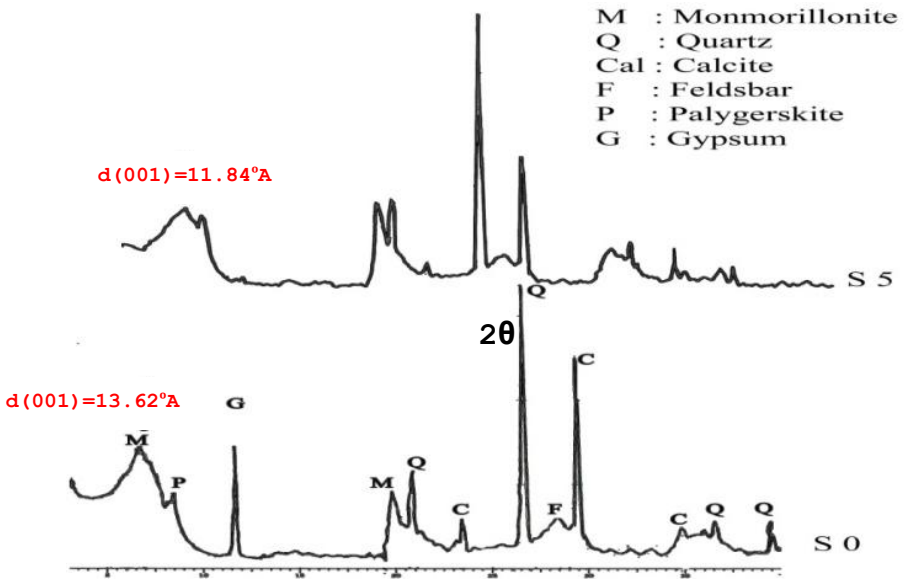

Figure 1: XRD patterns of original and Na_2CO_3 activated bentonite (S0,S5)

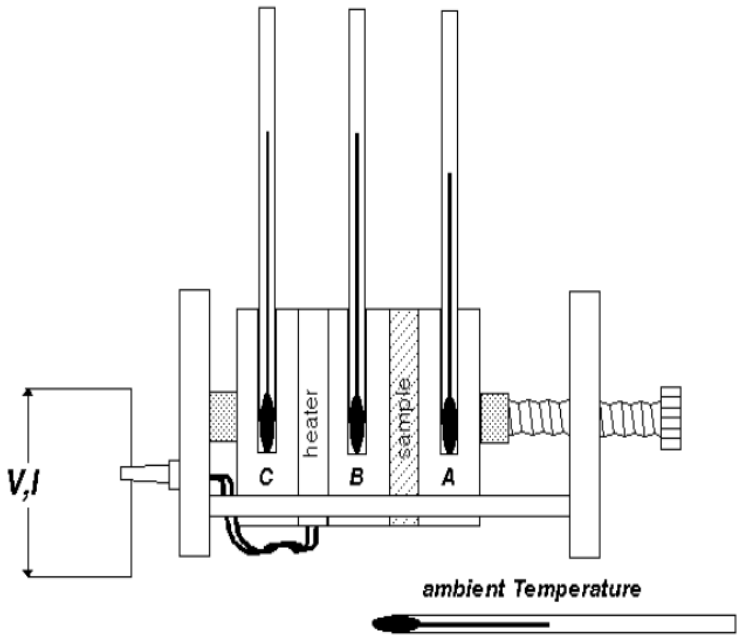

Figure 2.Lees' Disk Apparatus (schematic) [12] .

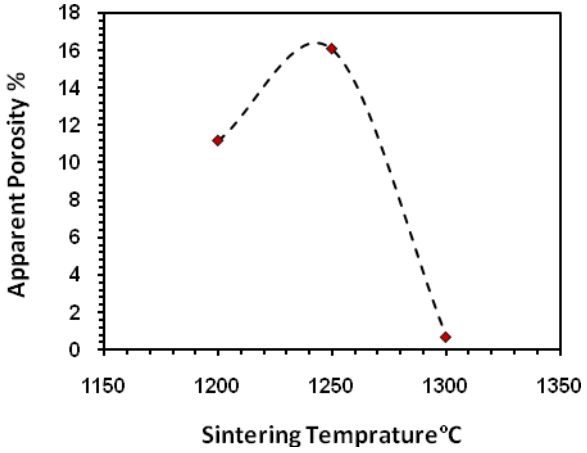

Figure 3: Apparent porosity against the increasing of firing temperature

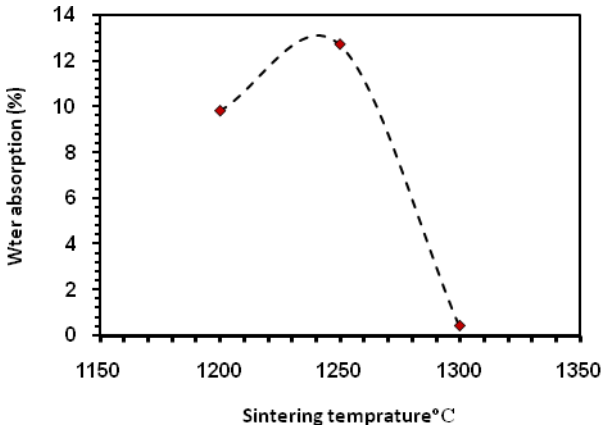

Figure 4: Water absorption against the increasing of firing temperature

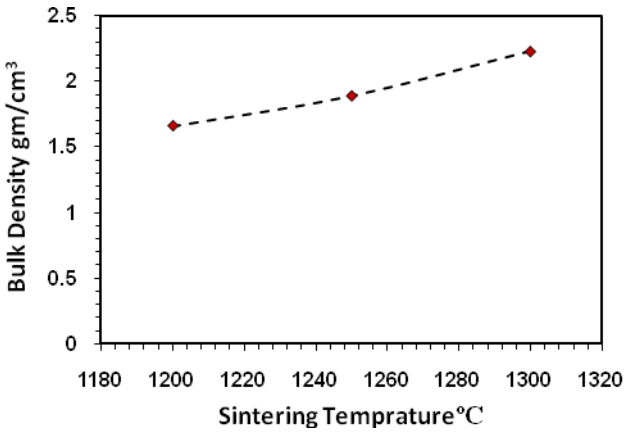

Figure 5: Apparent porosity against the increasing of firing temperature

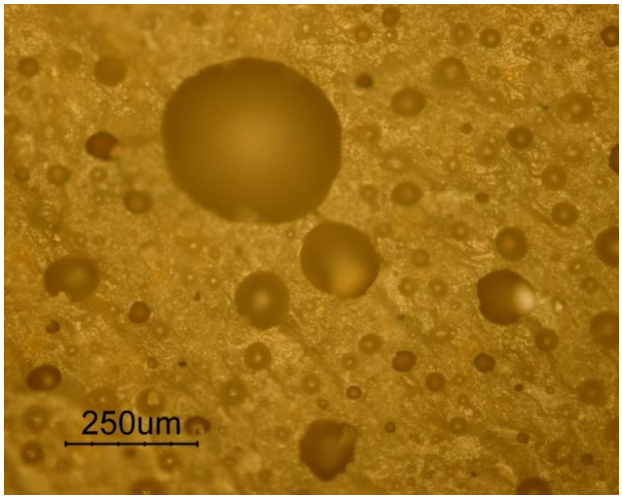

Figure 6: fracture morphology microscope for fired sample at 1300ºC

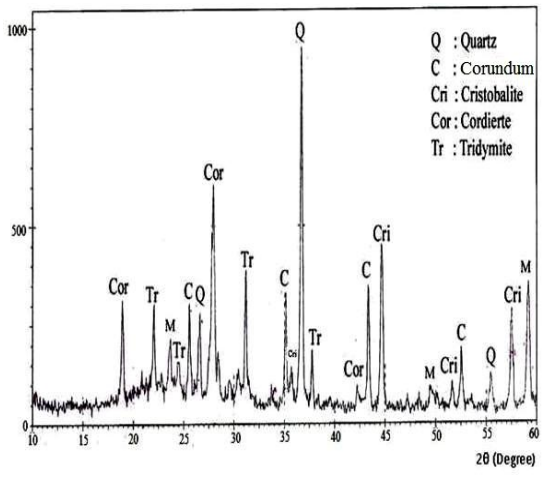

Figure 7: X-ray diff. pattern for the fired sample S5 at 1200ºC

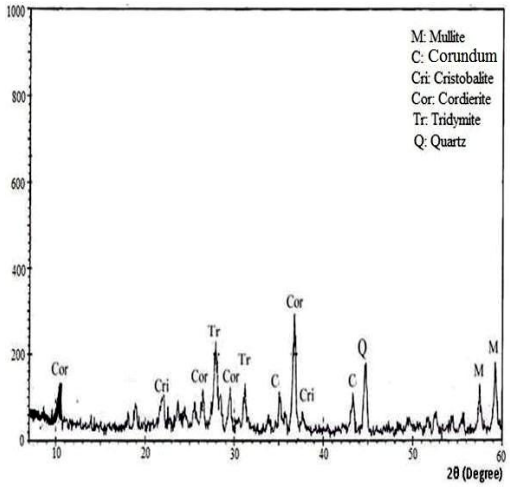

Figure 8: X-ray diff. pattern for the fired sample S5 at 1250ºC

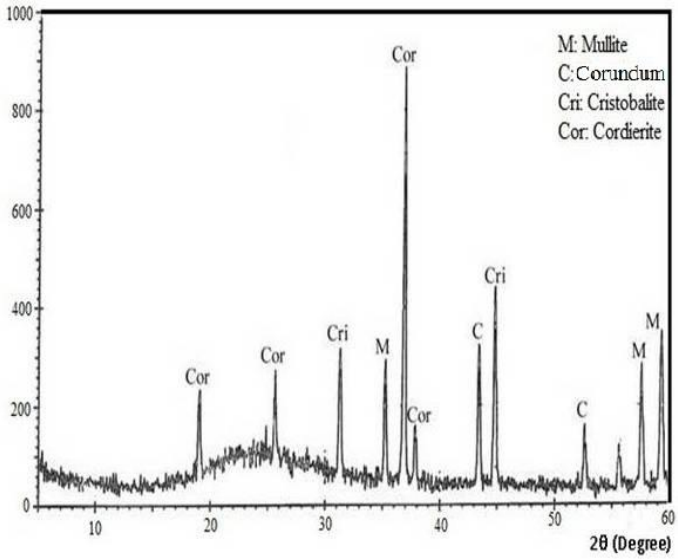

Figure 9: X-ray diff. pattern for the fired sample S5 at 1300ºC

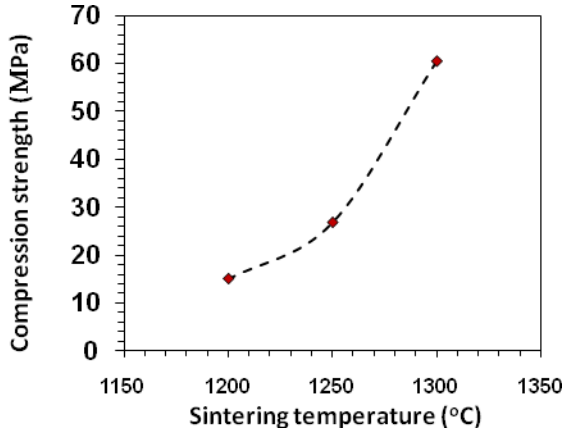

Figure 10: Compression strength against the increasing of firing temperature

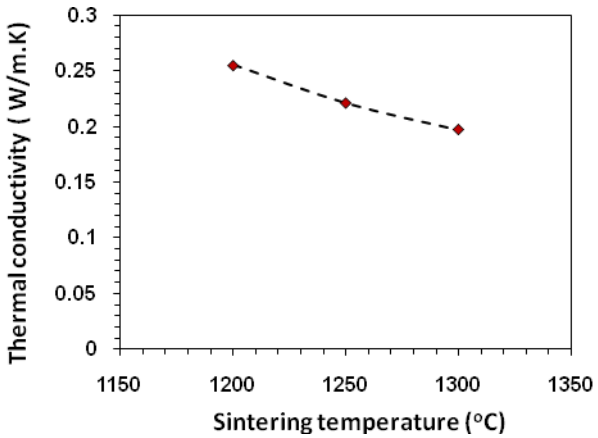

Figure 11: Thermal conductivity against the increasing of firing temperature

Conclusion

Ceramic body prepared from activated bentonite with addition of Alumina and Magnesia gives an optimum value for the compression strength and thermal, physical characterization compared with industrial insulating materials. The treated bentonite (under activation process and Al_2O_3 , MgO addition) gives the bentonite to withstand to high temperatures, reaches up to 1300 ºC for Al-Safra bentonite (which is about 1200 ºC without addition).

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