*Civil and Architectural Engineering*

# Effect of bentonite addition on some properties of porcelain

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## ABSTRACT

**P**orcelain is one of the most important ceramic materials with a wide range of traditional and technical applications. Since most mixtures of porcelain have a high sintering temperature, bentonite has been added in this research to improve the characteristics of sintering and burning. The porcelain mixture consisted of the following Iraqi raw materials: 30% wt kaolin, 30 wt% non-plastic clay (grog), 10% wt sodium feldspar, 10 wt% potassium feldspar and 20 wt% flint. After the mechanical mixing process and transfer the powder mixture to the slurry by adding distilled water, then different weight percentage of the sodium bentonite(0, 2.5, 5, 7.5 and 10) wt% was added. The specimens were prepared by using the solid casting method, and after the drying process, the specimens were burned at1100 $^{o}$C. The results of x-ray diffraction showed that bentonite reduced the crystallization of the main ceramic phases (mullite, quartz), which stimulates the appearance of amorphous glass phases. Also, the loss of mass on ignition increased when the addition of bentonite from 5.66% to 8.2%. There was also a great convergence between the granules of porcelain when adding bentonite and thus increase the shrinkage of the dimensions from 9.33% to 12.37 %. This led to increasing the bulk density from 1.97 g/cm$^3$ to 2.67 g/cm$^3$ at firing temperature 1100$^{o}$C, and the porosity was decreased from 17.1% to 1.44%. Diametrical strength and flexural strength (bending) increased with bentonite (14.88 to 34.46MPa), (6.2 to 8.65 MPa), respectively.

**Keywords**: Solid casting, Porcelain, Sodium silicate, Bentonite, Diametrical Strength.

## تأثير أضافة البنتونايت على بعض الخصائص للبورسلين

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### الخلاصة

يعتبر البورسلين من أهم المواد السيراميكية التي تتميز بمدى واسع من التطبيقات التقليدية والتقنية .ولأن معظم خلطات البورسلين ذات درجات حرارية مرتفعة فقد تم في هذا البحث أضافة البنتونايت لتحسين خصائص التلبيد والحرق.تألفت خلطة البورسلين من الخامات العراقية التالية (wt% 30 كاؤلين و wt% 30 طين محروق و wt% 10 فلدسبار الصوديوم و

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wt% 10 فلدسبار البوتاسيوم و wt% 20  فلنت) وبعد اجراء عملية الخلط الميكانيكي وتحويل المساحيق الى  عالق بأضافة
الماء المقطر تم أضافة نسب مختلفة من البنتونايت الصوديومي ( 0,2.5,5,7.5,10wt% ) وتمت عمليه تشكيل العينات بطريقة
الصب الصلب.وحرقت العينات في درجة حرارة C° 1100. أما نتائج حيود الاشعة السينية فقد اوضحت ان البنتونايت قلل من
تبلور الاطوار السيراميكية الرئيسية ( المولايت والكوارتز) , حيث ان الكوارتز يعمل على تحفيز ظهور الاطوار الزجاجية
العشوائية . كذلك ويسبب الفقدان الكبير في الكتلة للبنتونايت فقد ازداد الفقدان في الكتلة عند اضافة البنتونايت من % 5.66 الى
% 8.2 . كذلك حصل تقارب كبير بين حبيبات البورسلين عند اضافة البنتونايت وبالتالي زيادة الانكماش في الابعاد من %
9.33 الى 12.37% وانعكس ذلك على زيادة الكثافة من 1.97g/cm$^3$ الى 2.67g/cm$^3$ عند الحرق بدرجة حرارة C° 1100
المسامية فقد تناقصت بشكل واضح وكبير من 17.1% الى % 1.44.اما مقاومةالكسرالمحوري والانحناء   ازدادت مع
زيادة نسبة البنتونايت ( 14.88-34.46 MPa)و(8.65 Mpa - 6.2) على التوالي.
**الكلمات الرئيسية:** الصب الصلب, بورسلين ,سليكات الصوديوم, بنتونايت , مقاومة الكسر المحوري.

## 1. INTRODUCTION

Traditional ceramics are usually based on clay and silica. There is sometimes a tendency to equate traditional ceramics with low technology, however, advanced manufacturing techniques are often used. Competition among producers has caused processing to become more efficient and cost-effective. Complex tooling and machinery is often used and may be coupled with computer-assisted process control, **Barry** and **Norton, 2007.**

Slip casting is one of the traditional techniques to make a pottery and widely used. This technique uses an aqueous slurry, also known as slip, of ceramic powder. The slip is poured into a plaster of Paris ($CaSO_4.2H_2O$) mold. As the water from slurry begins to move out by capillary action, a thick mass builds along the mold wall. When sufficient product thickness is built, the rest of the slurry is poured out (drain casting). It is also possible to continue to pour more slurry in to form a solid piece (solid casting). The mold is then opened and the part is removed. The steps in slip casting are (1) slip is poured into mold cavity, (2) water is absorbed into plaster mold to form a firm layer, (3) excess slip is poured out, and (4) part is removed from the mold and trimmed. It is used to make teapots, vases, art objects, and other hollow-ware products. In solid casting, it is used to produce solid products and an adequate time is allowed for the entire body to become firm. The mold must be periodically resupplied with additional slip to account for shrinkage because of absorbed water, **Norazlina, 2015.**

## 2. RAW MATERIALS

## 2.1 Porcelain

Porcelain is the so-called (hard porcelain) which is characterized by high degree of burning, which is between (900°C-1400°C) with the reduction of pressure, has the use of electronic as well as use in the tools of decoration. The other type of porcelain is( soft porcelain) which burns between (1200°C-1300°C) and is used in the manufacture of decorative tools. There is another type or the medical porcelain, **Sladek, 1993.** Porcelain is a material produced from kaolin, quartz and potassium feldspar. Recently, the research of new materials, for example, non-hazardous wastes, which are able to replace traditional fluxing agents without changing the

process or quality of the final products have been realized, **Iqbal, 2008.** Porcelain with excellent technical characteristic was produced which had similar properties to traditional porcelain. The Firing process for porcelain stoneware had a standard temperature between 1200 ℃ and 1300 ℃, **Martin, et al., 2010.**

## 2.2 Kaolin

Pure kaolin has the composition $Al_2O_3.2SiO_2.2H_2O$ and is called *"Kaolinite"*. It is formed by weathering of feldspathic rocks, granites, etc. The Kaolin raw material contains, besides clay mineral "Kaolinite", other oxide fluxes such as $TiO_2$, $MgO$, $Fe_2O_3$, $K_2O$, $Na_2O$, $CaO$, etc., **Kennedy, et al., 2013.** Kaolinite can be distinguished by two layers of silicate sheet structure which consist of an alternate stacking of $[Si_2 O_5]^{-2}$ and $[Al_2 (OH)_4]^{+2}$ layered silicate mineral, in addition to one tetrahedral sheet linked through oxygen atoms to one octahedral sheet of alumina. Conferred to this structure is a lamellate character; favorable to the development of plates, **Damoah and Zhang, 2010, Table 1** shows the chemical analysis of Kaolin.

## 2.3 Feldspar

Feldspar refers to any of several crystalline minerals that consist of aluminum silicate combined with either potassium, sodium, calcium or barium, **Groover, 2010.** Feldspar is the most common rock-forming mineral (about 60% of the earth's crust). Sodium feldspar and potassium feldspar are homogeneous. Feldspar is one of the minerals often used **Rahaman, 2007, Table 2** shows the chemical analysis of sodium feldspar and **Table 3** shows the chemical analysis of Potassium feldspar.

## 2.4 Flint

The flint is the important formula for the silica, which is used in the manufacture of white clay objects and in the porcelain industry. It consists of a small crystal of quartz connected to each other by water molecules and the specific weight is about (2.62) less than quartz. The flint is more effective than quartz and it contributes to giving the white color to the burned objects as a result of combustion of organic matter. It controls the process of thermal expansion of the burned objects, **Rayan and Radford**, **1987 and Worral, 1982, Table 4** shows the chemical analysis of Flint.

## 2.5 Bentonite

Bentonite is a highly plastic clay whose composition is chiefly *"Montmorillonite"* as the main clay mineral. Bentonite have a structure composed by one sheet of octahedral (O) Al placed between two sheets of tetrahedral(T) Si, with substitutions of some tetrahedral Si atoms by Al atoms and/or of octahedral atoms ($Al^{3+}$ or $Mg^{2+}$) substituted by atoms with lower oxidation number, **Andrini, et al., 2016.** The net negative charge of the 2:1 (TOT) layers is balanced by

the exchangeable cations such as $Na^+$ and $Ca^{2+}$ located between the layers and around the edges, **Komadel, 2016**, **Table 5** shows the chemical analysis of Bentonite.

## 3. EXPERIMENTAL PROCEDURE

Preparation of specimens by slip casting technique is used. Fine powders were such as micro feldspar (Na, K), Flint, Kaolin, Nonplastic clay (grog), and Bentonite. Dispersant used is sodium silicate ($Na_2SiO_3$) and sodium carbonate ($Na_2CO_3$) and sodium polyacrylate (darvan7); solvent used is distilled water and molds prepared from Plaster of Paris were used for the casting. The plaster molds were prepared. The mixture is prepared using water and plaster of Paris in the ratio (3:2) such that the negative imprint can be cast over the mold. A glass plate is taken over which the vaseline oil is applied. This vaseline oil acts as a lubricant and does not allow the mold to stick on the glass. Then a hollow cubic made from plywood was used as a container which puts over the glass plate, a piece of rubber takes the form of the specimen (performs) puts over the glass plate at the center of the hollow cubic which helps in uniform drying during the casting process. Vaseline oil is also applied over the performs. Then the prepared mixture was slowly poured into the hollow cubical box to a certain height predefined. The body is kept to be air dried for about (1h). After (1h) the body is removed from the wood box and kept to be air dried (5days). After that, the ceramic suspension is slowly poured into the mold. The ceramic particles will start to settle against the plaster as the water is drawn from the suspension into the plaster mold. The slurry is optimized by studying its properties so as to get a slip which is stable i.e. the suspended particles do not agglomerate or do not settle with changing time. The slip obtained by preparing specimen with different dispersants and different percentage of dispersant. It was found experimentally that sodium silicate ($Na_2SiO_3$), sodium carbonate ($Na_2CO_3$) and sodium polyacrylic (darvan7), when the density of ($Na\text{-}SiO_3$), ($Na\text{-}CO_3$) and (darvan7) solution are (1.25 $g/cm^3$), (1.25 $g/cm^3$) , (400-700 $kg/m^3$). **Table 6** shows the specimen names, the raw materials composition of each specimen.

## 4. FIRING PROGRAM

The specimen was fired after drying in a Nabertherm GmbH (more than heat 30-3000ºC), made in Germany, in three steps:

1. The first step: Temperature increased from the room temperature with an average raising in temperature of (15 °C/min) and settled at (450 °C) for (2 hours).
2. The second step: Temperature increased with raising the speed of (15°C/min) from (450 °C) and settled at (900 °C) for (2 hours).
3. The third step: Temperature increased from (900 °C) with raising the speed of (15 °C/min) and settled at (1100 °C) for (2 hours) for the samples ( A, B, C, D, and E).

## 5. MEASUREMENTS

### 5.1 X-ray Diffraction

It has the following specifications and is used as a quality analysis of the specimens before and after firing. Specifications of X-ray apparatus: SHIMADZU, Model: XRD-6000, X-ray Tube: Cu, NF type, Scanning radius: 185mm, Leakage X-Rays: less than 2.5μSv/h at maximum out.

### 5.2 Loss on Ignition (L.O.I)

The mass loss of such specimens was measured using digital balance. This test was determined according to the (ASTM C20 – 00). The was calculated b mass losses using Eq. (1).

$$L.O.I = \frac{m_o - m}{m_o} \times 100\% \qquad (1)$$

### 5.3 Linear Shrinkage

The linear shrinkage of such specimens was measured using digital Vernier Calipe. This test was determined according to the (ASTM C20 – 00). The linear shrinkage was calculated using Eq.(2).

$$L.SH = \frac{L_0 - L_1}{L_0} \times 100\% \qquad (2)$$

### 5.4 Density and Porosity
Bulk density and open porosity are determined by using *Archimedes method* using distilled $H_2O$. The mass of material in the air is divided by its buoyancy (reduction in weight) when suspended in a liquid medium to give a measurement of density, **She, 2002.**
The bulk density of such specimen was measured using the traditional Archimedes method.  In brief, the dry specimen with a mass of (D) was put in a full water beaker and evacuated to $10^{-1}$ bar for (30) minutes to remove the air trapped in the pores, and the sample mass with water (M) was weighed and immersed in water (S). This test was determined according to the (ASTM C373-88), and apparent porosity in the same method measure.

### 5.5 MECHANICAL PROPERTIES

5.5.1 Compressive Strength

(ASTM standard-C773) was used as a standard to test the compressive strength for ceramic specimens, **ASTM C773 – 88, 2011**. The force was applied perpendicular and continuously on the cylindrical specimen by using hydraulic testing compression machine, maximum force (KN).

5.5.2 Diametrical Strength

The diametrical strength (Brazilian) test can be derived by using Eq.(3)

$$(\text{Diametrical strength})\sigma_D = \frac{2F}{\pi \, D \, T} \tag{3}$$

D=12.65 mm

T=9.18 mm

5.5.3 Flexural Strength

Flexural strength was measured in a (3-point bending) test, **Mitchell, 1993,** and the bending strength for the specimens (ASTM C1674 – 11) was calculated using Eq.(4), **ASTM C1674 – 11, 2011.**

$$\text{Flexural strength} = \frac{3FD}{2bd^2} \tag{4}$$

d=14.2 mm, b=25.1 mm, D=50 mm


# 6. RESULT AND DISCUSSION


## 6.1 X-ray Diffraction

From the results of x-ray diffraction analysis, the emergence of mullet phase is observed, which begins to appear in temperatures greater than (1100 °C) in clay materials such as kaolin. However, this grade can decrease in the case of the presence of auxiliary oxides on smelting such as $K_2O$, $Na_2O$, which is found in feldspar and also found in bentonite with iron oxide. In addition to the survival of silicon dioxide in the quartz phase of the temperature of burning (1100°C) for not enlarging this class to turn into other stages such as terminate and cristobalite. It was noticed that the degree of the phases decrease with the addition of bentonite due to the random glass phases consisting of incineration processes which reduce the degree of crystallization, as shown in **Fig.1**

## 6.2 Loss on Ignition

**Fig.2** shows the mass losses increase with increasing of bentonite ratio because the row bentonite has organic impurities and oxides which have a low melting temperature plus that bentonite have crystal water which could possibly affect the mass losses, density and the structure of the samples plus changing in shrinkage as well. The mass losses limit the maximum amount from density. **Table 7** listed the values of loss on ignition with different bentonite additions.

## 6.3 Shrinkage

**Fig.3** shows that the shrinkage increases with the increasing of bentonite ratio, because of the decreasing in space between the ceramic particles which means significantly reduce the size of

the ceramic products. Shrinkage could possibly affect the homogeneous of the dimension of the ceramics products. As the total volume decreases with increasing of bentonite ratio, an increase in density is expected, taking into consideration the changes in mass as a result of changing in firing temp, (mass losses). The values of shrinkage with different bentonite additives are listed in **Table 8**.

## 6.4 Bulk density and porosity

The density increases with the increasing of bentonite ratio for different firing temperatures (900ºC, 1100ºC), as shown in **Fig.4**. The main reason for increasing density is decreasing the melting temp. For the Bentonite component which works on the increasing the bonding with each other because of the glass phase which is formed in general. The density for firing temperature (1100 ºC) is more than (900 ºC). The density becomes constant for the added ratio more than 7.5wt %. For the apparent porosity, **Fig.5** shows the porosity decreases when the bentonite ratio increases because of the glass phase which exists from firing bentonite between the grain boundaries and sealing the porosity in general. The porosity decreases significantly when the firing temperature raises from 900 ºC to 1100 ºC. The porosity affects significantly all the mechanical and thermal properties and limits their application. **Table 9** listed the bulk density values with different bentonite additives, and **Table 10** shows the porosity with different bentonite additions.

## 6.5 Diametrical strength

The change of the diametrical strength with the increase in bentonite ratio where the resistance fracture is very weak when burring models at a temperature of (900ºC), which means not to get a complete burn and the transformation of the phase of the material used, as shown in Fig.6. As for increasing the temperature of (1100ºC), an increase in the resistance of the fracture is noticed due to the appearance of the glass phase of bentonite to lower the temperature of the fusion and penetration between the granules of porcelain powder and thus increase by (100%) when adding (10wt %) bentonite to the mixture of porcelain to reach (35MPa). These values are considered high for traditional ceramic materials. **Table 11** listed the diametrical strength of specimens with different bentonite additions.

## 6.6 Flexural strength

**Fig.7** explains the strength of the fracture variation with the addition of bentonite. This is consistent with the result of the other mechanical properties, which are significantly affected by the porosity. The porosity decreased significantly with the increase of bentonite, this means that the fracture resistance will increase. Generally, the values of fracture strength ranged from (6 to 9 MPa). The growth of microscopic cracks dramatically increases the speed of fracture. The larger the material is, the greater the growth of the microscopic cracks during the process of mechanical loading. Therefore, the material that possesses a large curvature resistance should be

resistant to the growth of microcracks and can achieve this the presence of grog in the mixture where works to impede the growth of microscopic cracks and thus delay the process of fracture. Show us high mechanical properties. **Table 12** shows the values of flexural strength with a different weight percentage of bentonite.

## 7. CONCLUSIONS

1. Bentonite additions reduce sintering temperature then increase the density and mechanical properties, as well as the appearance of the glass phase at low temperature.
2. Such use to manufacture porcelain tiles because its specifications are similar to tile specification of ISO 10545.
3. The high density of porcelain with bentonite additions led to increase shrinkage and increase the loss of mass. This represents, the continuation of the process of sintering between granules of porcelain products.
4. The porosity affects significantly on all the mechanical and thermal properties and limits their application.

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**NOMENCLATURE**

b= width, mm.

d=thickness, mm.

D=diameter, mm.

F=max. Applied load, N.

F=force at the fracture point, N.

L=length, mm.

$L_1$=fired length of the test specimen, mm.

$L_0$= plastic length of the test specimen, mm.

$m_1$=mass after firing, g.

$m_0$=mass before firing, g.

T=thickness of the disk, mm.

L.O.I= loss of ignition, wt %.

L.SH= linear shrinkage, wt %.

$\sigma_D$=diametrical strength, MPa

**Table 1**. Chemical analysis of Kaolin.

| Oxide | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | TiO$_2$ | CaO | MgO | Na$_2$O | K$_2$O | Loss on ignition % |
|---|---|---|---|---|---|---|---|---|---|
| % | 47.34 | 36.37 | 0.63 | 2.2 | 0.12 | 0.08 | 0.31 | 0.53 | 12.42 |

**Table 2.** Chemical analysis of sodium feldspar.

| Oxide | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | CaO | Na$_2$O | K$_2$O | TiO$_2$ |
|---|---|---|---|---|---|---|---|
| % | 76.66 | 11.89 | 0.29 | 0.69 | 8.43 | 0.81 | 0.24 |

**Table 3.** Chemical analysis of potassium feldspar.

| Oxide | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | CaO | Na$_2$O | K$_2$O | TiO$_2$ | L.O.I |
|---|---|---|---|---|---|---|---|---|
| % | 65.9 | 18.6 | 0.07 | 0.40 | 2.90 | 11.8 | 0.002 | 0.14 |

**Table 4.** Chemical analysis of Flint.

| SiO$_2$ % | Al$_2$O$_3$ % | Fe$_2$O % | TiO$_2$ % | CaO % | MgO % | Na$_2$O$_3$ % | K$_2$O % | L.O.I % |
|---|---|---|---|---|---|---|---|---|
| 42.92 | 35.92 | 0.9 | 1.9 | 3.47 | 1.46 | 0.1 | 0.15 | 13.9 |

**Table 5.** Chemical analysis of Bentonite.

| Oxide | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | TiO$_2$ | CaO | MgO | Na$_2$O | K$_2$O | Loss on Ignition % |
|---|---|---|---|---|---|---|---|---|---|
| % | 55.31 | 14.62 | 5.71 | 1.17 | 3.43 | 3.31 | 1.11 | 0.72 | 14.62 |

**Table 6.** The specimens names, and the composition of raw materials for each specimen.

| Specimens type | Kaolin wt.% | Nonplastic clay Wt.% | Sodium feldspar Wt.% | Potassium feldspar Wt.% | Flint Wt.% | Bentonite Wt.% |
|---|---|---|---|---|---|---|
| A | 30 | 30 | 10 | 10 | 20 | 0 |
| B | 30 | 30 | 10 | 10 | 20 | 2.5 |
| C | 30 | 30 | 10 | 10 | 20 | 5 |
| D | 30 | 30 | 10 | 10 | 20 | 7.5 |
| E | 30 | 30 | 10 | 10 | 20 | 10 |

**Table 7**. Loss on ignition with different bentonite additives.

| Specimen | Bentonite additives Wt % | Mass loses wt % |
|---|---|---|
| A | 0 | 5.66 |
| B | 2.5 | 6.87 |
| C | 5 | 7.05 |
| D | 7.5 | 7.36 |
| E | 10 | 8.2 |

**Table 8**. Shrinkage with different bentonite additives at firing temp. 1100 $^{o}$C.

| Specimen | Bentonite additives Wt % | Shrinkage % |
|---|---|---|
| A | 0 | 9.33 |
| B | 2.5 | 10.07 |
| C | 5 | 11.28 |
| D | 7.5 | 12.27 |
| E | 10 | 12.37 |

**Table 9**. Bulk density with different bentonite additives.

| Specimen | Bentonite additives Wt % | Bulk density (g/cm$^3$) at firing temp. (900 $^{o}$C) | Bulk density (g/cm$^3$) at firing temp. (1100$^{o}$C) |
|---|---|---|---|
| A | 0 | 1.46 | 1.97 |
| B | 2.5 | 1.42 | 1.98 |
| C | 5 | 1.48 | 2.16 |
| D | 7.5 | 1.49 | 2.55 |
| E | 10 | 1.55 | 2.67 |

**Table 10**. Porosity with different bentonite additives.

| Sample | Bentonite additives Wt % | Apparent porosity % at firing temp. (900ºC) | Apparent Porosity % at firing temp.(1100ºC) |
|--------|--------------------------|---------------------------------------------|---------------------------------------------|
| A | 0 | 45.87 | 17.10 |
| B | 2.5 | 43.92 | 10.66 |
| C | 5 | 42.71 | 8.95 |
| D | 7.5 | 42.15 | 5.08 |
| E | 10 | 39.04 | 1.44 |

**Table 11.** Diametric strength

| Specimen | Bentonite additives Wt % | Diametrical Strength(MPa) at firing temp. 900ºC | Diametrical Strength(MPa) at firing temp. 1100ºC |
|----------|--------------------------|-------------------------------------------------|--------------------------------------------------|
| A | 0 | 2.74 | 14.88 |
| B | 2.5 | 3.38 | 17.0 |
| C | 5 | 3.60 | 20.39 |
| D | 7.5 | 3.80 | 27.0 |
| E | 10 | 3.96 | 34.46 |

**Table 12.** Flexural strength

| Specimen | Bentonite additives Wt % | Flexural strength (MPa) |
|----------|--------------------------|-------------------------|
| A | 0 | 6.229 |
| B | 2.5 | 6.553 |
| C | 5 | 7.4 |
| D | 7.5 | 8.083 |
| E | 10 | 8.65 |
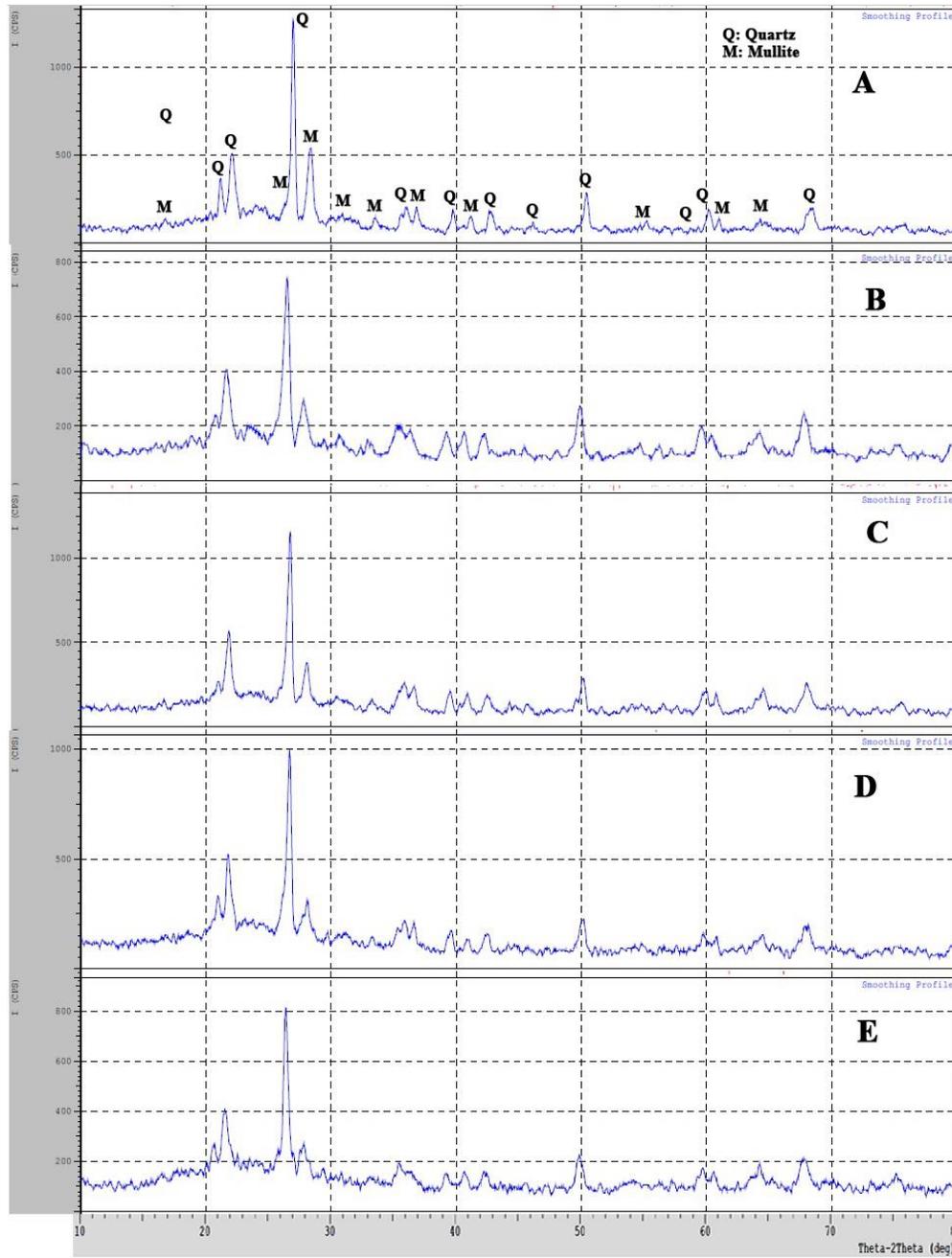
**Figure 1**. X-ray diffraction of porcelain with different bentonite additives,[(a):0 ,(b):2.5 ,(c):5 ,(d):7.5 ,(e):10 wt %].
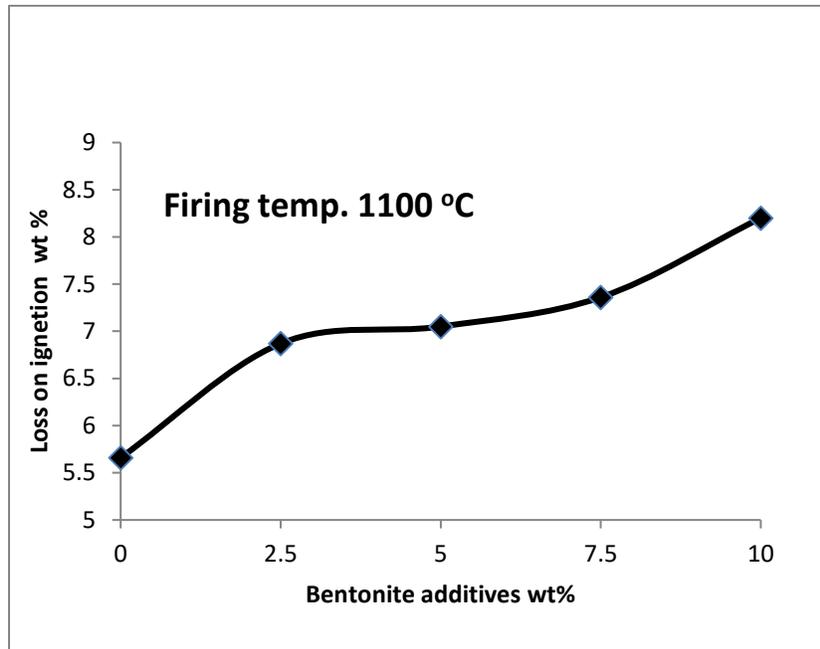
**Figure 2**. Loss on ignition of porcelain with different bentonite additives.
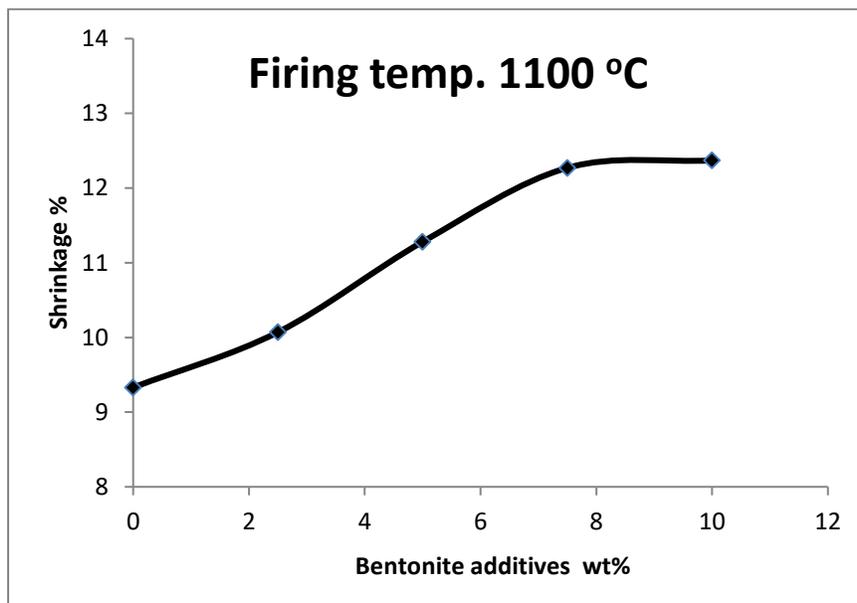


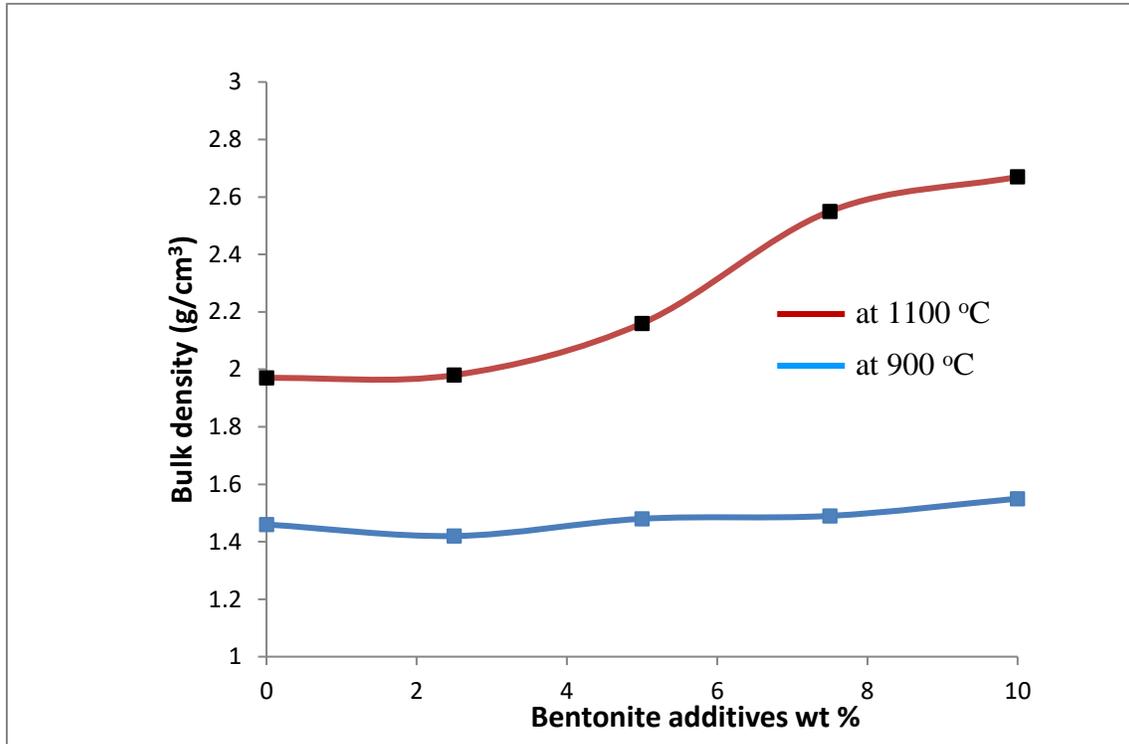**Figure 3.** Shrinkage of porcelain with different bentonite additives.

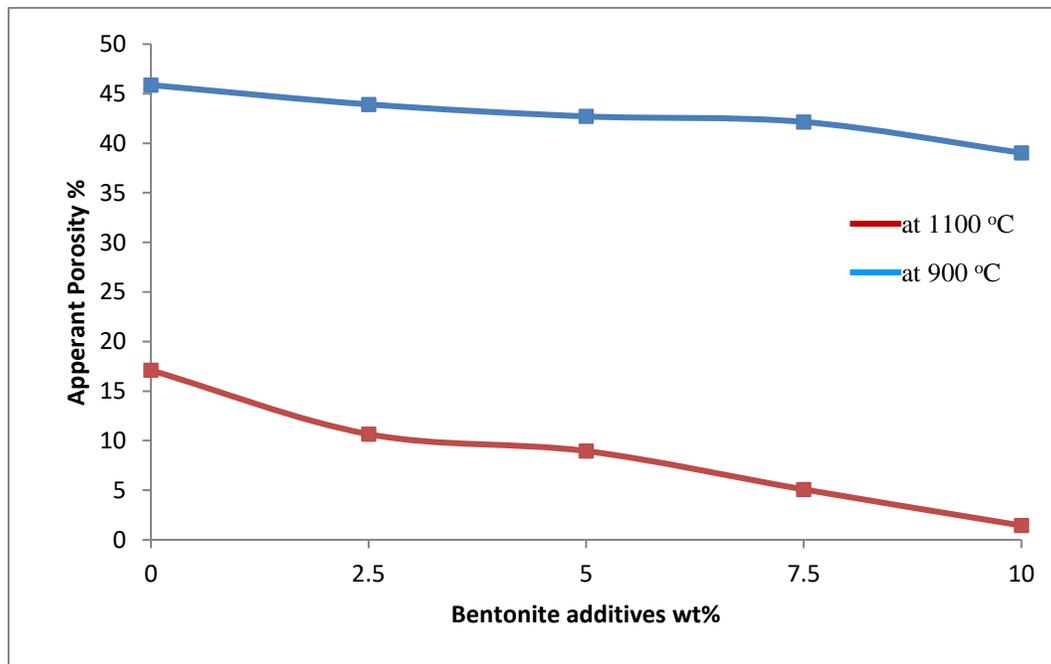**Figure 4.**  The bulk density of porcelain with different bentonite additives



**Figure5.**  The porosity of porcelain with different bentonite additives.
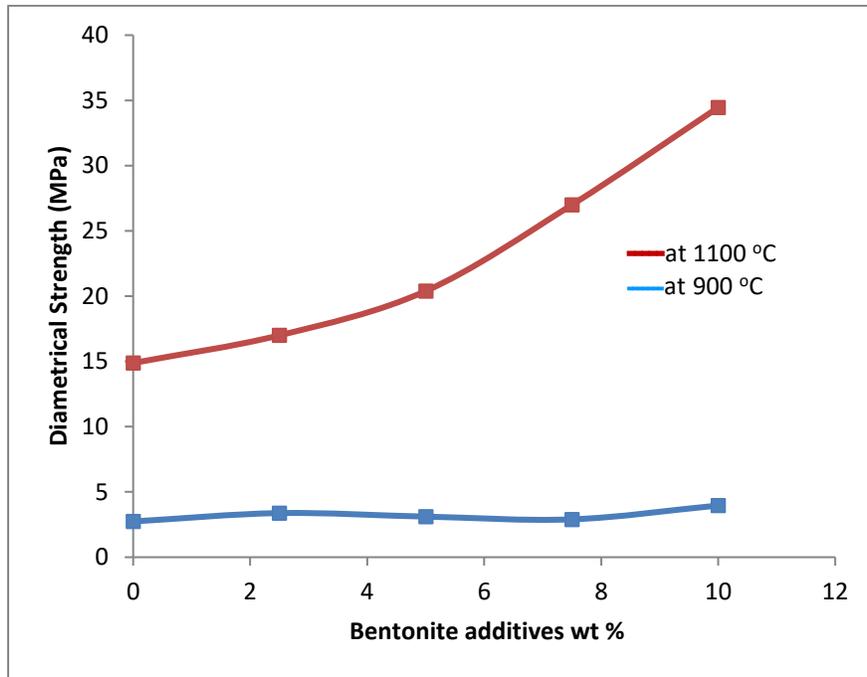
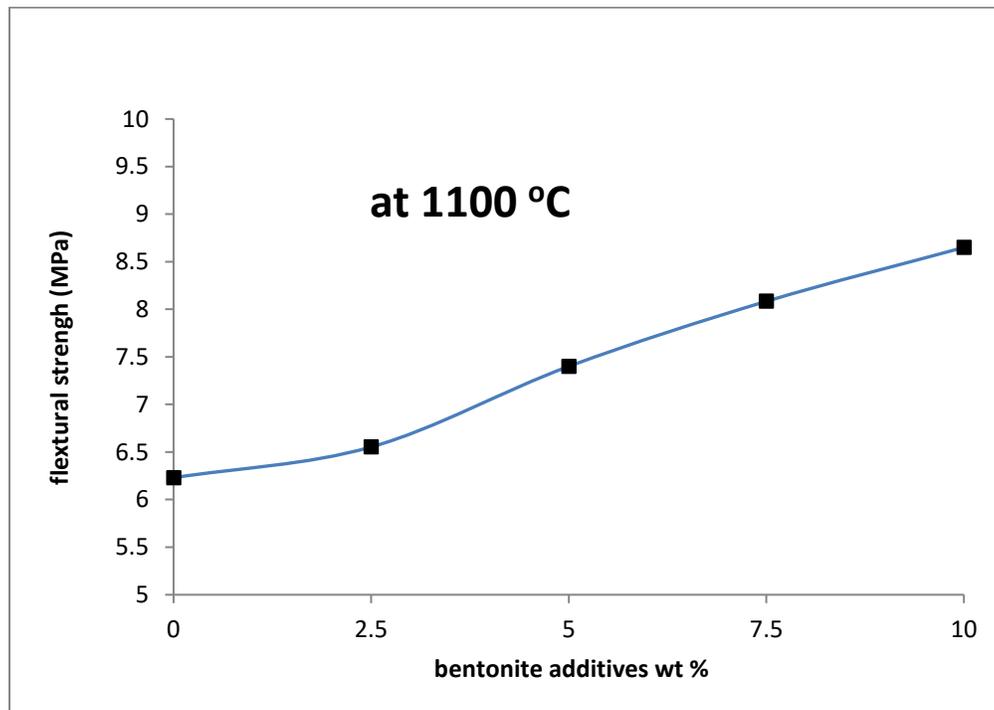**Figure 6.** The diametrical strength of porcelain with different bentonite additives.



**Figure 7.** Flexural strength of porcelain with different bentonite additives.