



## **Study the physical properties of refractory mortar contains kaolin-metakolin-fire brick powder-SiC**

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**Abstract :** The research aims to use one of the raw materials available in Iraq, namely kaolin Duekhla, which is available in the western desert in Iraq, and to know and diagnose the effect of some additives on the physical properties such as open porosity, bulk density and linear shrinkage of firing. The samples are composed of a mixture of basic material and some additives. The main mixture consists of 70% kaolin and 30% metakaolin. The first type of additives is commercial powder silicon carbide(SiC), and the second type of additives is the fiery brick obtained from the waste of lining of the kilns where it was converted into powder. The samples were prepared according to the specified ratios with addition Up to 40% (some samples containing sic only, and others contain fire brick powder only and other samples containing both together. The samples were sintered at 1100 °C and 1500°C. The properties studied were open porosity, bulk density and linear firing shrinkage.

**Keywords:** refractory mortar, kaolin, metakolin, fire brick powder, SiC.

### **Introduction**

Refractory materials are made to be chemically and physically not likely to change significantly or to deteriorate suddenly at high temperatures (above 500°C) and are used in different types of high temperature vessels such as linings for furnaces, kilns, converters, reactors, ladles. Depending on the working and application conditions, refractories must have certain properties, for example, they must have a certain amount of resistance to thermal shocks, be chemically inert and / or have a specific value of thermal conductivity and thermal expansion coefficient (1).

Refractories are one type of ceramic material that has the potential to maintain its physical and chemical properties when used at high temperature. Because of its unique properties it can be suitable for the requirements of production factories such as metal casting plants, The primary task of these heat-resistant materials in metal casting is to isolate the product of high temperature from the external environment or as a container to place the dissolved product. Therefore, knowledge of the properties of heat-resistant materials is necessary in the metal casting industry(2).

Refractories are mostly used in basic metal industries. In the steel-making process, the molten iron from the blast furnace is purified from the impurities including C, S, P, Mn, etc(3). One of the basic conditions that refractories should be characterized by refractory slag. In the aluminum metal industry, the requirements of the thermal materials are completely different from those of the thermal materials used in the steel industry, for example, in the process of refining the aluminum, although the temperature used is lower compared with the heat in the steel industry, but there is an important problem is the penetration in refractories. Hence, the refractory should be choosing so that it has a non wetting characteristic to molten aluminum(4).

Clay and its minerals are important industrial raw materials. They are used in many industrial and scientific applications. They are used in many industrial applications, for example, ceramics, papermaking, painting, petroleum industry, catalysis etc(5). Its industrial use area is closely related to its structure, composition and other physical properties. Diagnosing and knowing these properties helps in determining the best exploit, and eventually may open up new areas of application (6).many studies deals with physical properties of kaolin for example Adindu C. Iyasara1 et al(7) study the suitability of using local kaolin (Nsu clay) and Nsu clay grog to enhance efficiency (reduce shrinkage, improve abrasion and reduce porosity) in the production of dense refractory bricks they found that as the percentage of grog increased, the linear shrinkage and the bulk density decreased, the optimal apparent porosity (20.22 %) and cold crushing strength (61.77 MPa) values were obtained in 30 % grog size. Dunia K. Al-Nasrawy(8) investigate the physical and mechanical properties based on  $\alpha$ -SiC powder with different percentage addition of Duekhla raw kaolin, samples were sintered at 1400 °C and the apparent density and apparent porosity decreasing with increasing kaolin content. Other studies concern with silicone carbide for example, Wan Nur Azrina Binti Wan Muhammad et al(9) studied, the effect of different amount and particle size of SiC susceptor on the heating rate and properties of sintered magnesium prepared by microwave sintering has been investigated. It was found that SiC susceptor would increase the heating rate during sintering process of magnesium. Job AjalaAmkpa et al (10) investigated The mechanical, chemical and physical property of Alkali fireclay for its appropriateness for refractory application. they found that the optimum sintering temperature was at 1200 °C; the optimum values of porosity was 24.52 %, the optimal value for compressive strength was 15.37 MPa, firing shrinkage was 8.9 % and 1.8 g/cm<sup>3</sup> was for bulk density. The current study aims to study the physical properties of refractory mortar by using Iraqi kaolin, metakaolin, additive from the commercial powder SiC and powder from the milling of the waste of the lining of the thermal ovens(fire brick powder).

## 2.Experimental work

### Raw materials

Iraqi kaolin was used as raw material and commercial materials (fire bricks and silicon carbide), taking into account the purity of each and the distribution and rate of granular diameter as follows:

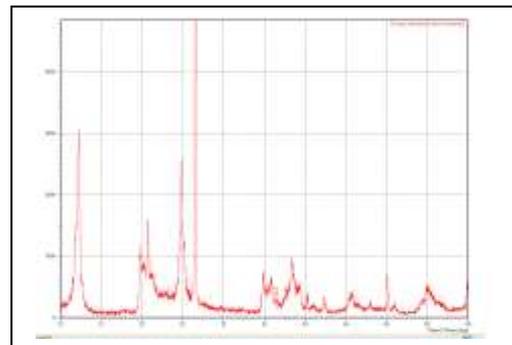
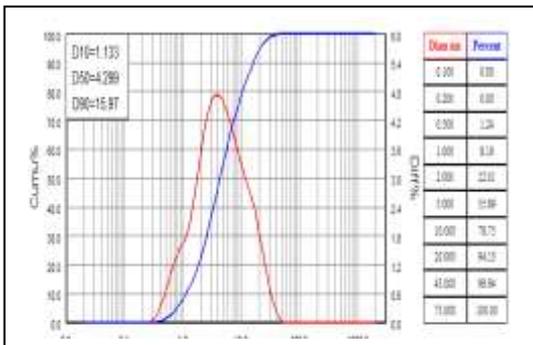
- Kaolin ore is abundant in the Dweikla region in Western Desert, Iraq, where it is laboratory milled and with various milling steps to select the particle size required for the research purposes(4.299  $\mu$ m).The measurements of particle size were achieved by using Bettersize2000 laser particle size analyzer, particle size distribution is given in figure 1. Table (1) shows the chemical analysis of the Iraqi kaolin clay and its XRD pattern is shown in figure2.
- Metakaolin powder with particle 3.227  $\mu$ m and particle size distribution is shown in figure 3 was obtained by firingIraqi kaolin at 800 °C for an hour. The disappearance of the kaolin peaks is a sign of the transformation of kaolin into a randomized powder (metakaolin).
- Silicon carbide powder, a powder of black color, where the powder particle size was69.81  $\mu$ m with the XRD pattern as shown in Figure 4. And particle size distribution is given in figure 5.
- Powder of fire bricks obtained from grinding of residues furnaces lining, particle size was 98.08  $\mu$ m and particle size distribution is given in figure 6.

**Table 1 chemical analysis of Iraqi kaolin.**

%wt( weight percentage )								
L.O.I	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	MgO	CaO	AL <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>
14.6	0.12	1.97	0.03	0.17	0.031	1.2	33.5	49.6

**Table 2** the sample compositing of different additives and sintering temperature.

% wt Matrix(70% kaolin+30% metakaolin)	SiCwt%	B.P wt%	Firing temp
90	10		1100
			1500
90	5	5	1100
			1500
90		10	1100
			1500
80	20		1100
			1500
80	10	10	1100
			1500
80		20	1100
			1500
70	30		1100
			1500
70	15	15	1100
			1500
70		30	1100
			1500
60	40		1100
			1500
60	20	20	1100
			1500
60		40	1100
			1500



**Figure 1.**particle size distribution of Iraqi kaolin .

**Figure 2** X-ray diffraction pattern of Iraqi kaolin

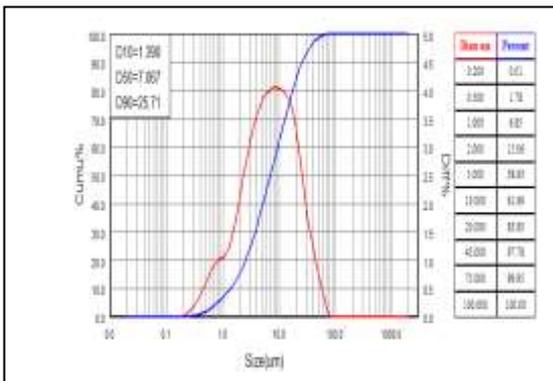


Figure 3 particle size distribution of Iraqi metakaolin.

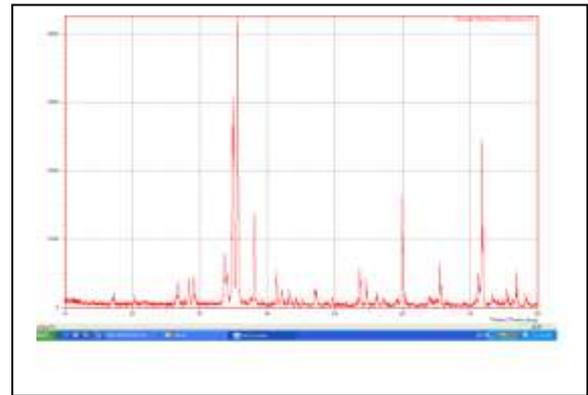


Figure 4 X-ray diffraction pattern of silicon carbide powder.

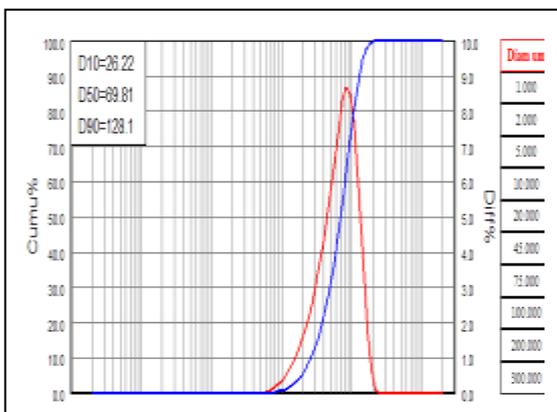


Figure 5 particle size distribution of SiC powder.

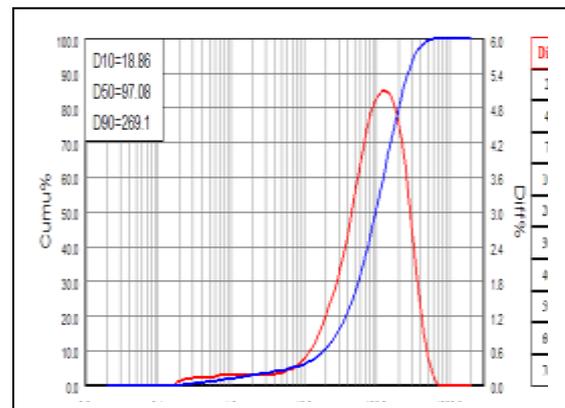


Figure 6 particle size distribution of fire brick powder.

### Sample preparation

Two stages were carried out prior to the preparation of samples:

1. Optimization was performed to select the best quantity of water used to obtain the best systematic samples after drying and free of cracking and the ratio was solid / liquid = 1.5 .
2. Optimization was performed to select the metakaolin content added to kaolin, so that the samples after drying had the largest bulk density and compressive strength and less contraction and the proportion of metakaolin 30% and kaolin 70%. In other words, the basic ingredient matrix consists of 70% kaolin and 30% metakaolin.

SiC and fire brick powder were added to the matrix mixture, as shown in table 2, the ingredients were mixed with a locally made mixer where the amount of the binder was 4 g of sodium silicate, the water was 100 g and the total steel powder was 150 g. The powders are weighed and mixed with a 5-minute manual mixing, then the powders are added to the water, during the movement of the mechanical mixer, after which the mixing continues for 2 hours. After the mixing process, the mixture is placed in oiled wood molds, left for a week and removed from the molds, then placed in the drying oven and gradually raised to 110 ° C and remaining for 24 hours. Samples were fired by electrical furnace at 1100 and 1500 with sintering rate 3 ° /min with maturing time 1 hr.

### Physical Measurement

The bulk density was calculated from the Equation(11):

$$BD = \frac{W_1}{V_1 - V_2},$$

where BD : bulk density, g/cm<sup>3</sup>

W<sub>1</sub>: Dried weight, g;

V<sub>1</sub>: Soaked weight, cm<sup>3</sup>;

V<sub>2</sub>: Suspended weight, cm<sup>3</sup>.

Apparent porosity(AP) was calculated from Equation (11):

$$AP = \frac{W - D}{W - S} \times 100,$$

where AP: Apparent porosity, %; W : Suspended weight, g; D: Dry weight, g;

S – Soaked weight, g.

The firing shrinkage was calculated as percentage of original green length as indicated in the equation :

$$FS = \frac{L_1 - L_2}{L_1} \times 100,$$

Where FS : Firing shrinkage, %; L<sub>1</sub>: Dried green length, cm; L<sub>2</sub>: Fired length, cm.

### 3. Result and discussion

#### Bulk density

Figure 7 shows the effect of sintering temperature and the additive of silicon carbide (SiC) and fire brick powder (BP), where the ratio of the primary mixture 90% (70% kaolin and 30% methacolin), it was found when the sintering temperature increases from 1100 to 1500, bulk density increased due to densification during sintering, increased affinity of particles, reduction of pores that existed in green body, and decreased size of pores during sintering (6).

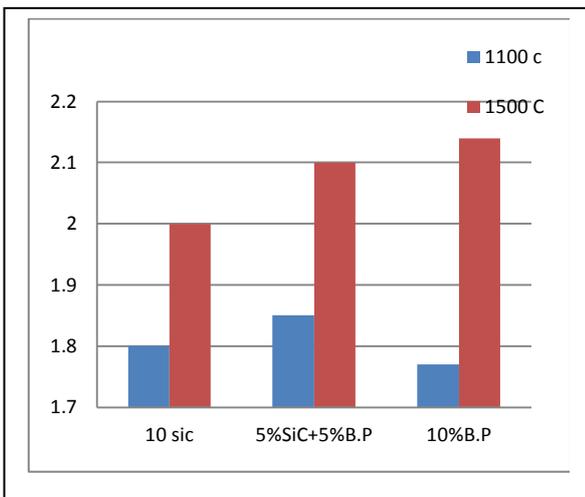
At the 1100° C sintering temperature, by comparing the values of the bulk density in the case of additives from sic and BP separately, the addition of sic was found to have a large bulk density compared to BP. The reason is sic with a granular size smaller than bp, Granularity. However, if both additives were present (5% SiC + 5% B.P), the maximum value of the bulk density was obtained. It can be said that the presence of both additives resulted in obtaining a wide particle distribution of the raw material powder, which in turn led to obtaining the best compact for the particles of green body before the firing . It can be said that the difference between the particle size between SiC and B.P resulted in the best packing of the powder particles leading to better sintering at 1100, since the presence of a wide particle distribution of the raw material powder leads to the best packing of powder grains for the green body(12)

Figure 8 shows the behavior of the bulk density when the ratio of the primary mixture is 80%. In the case of sic or bp or both, at the sintering temperature of 1100, the values were found to be close at 20% sic and 10% sic + 10% , while at 20 % B.P the bulk density was greater.

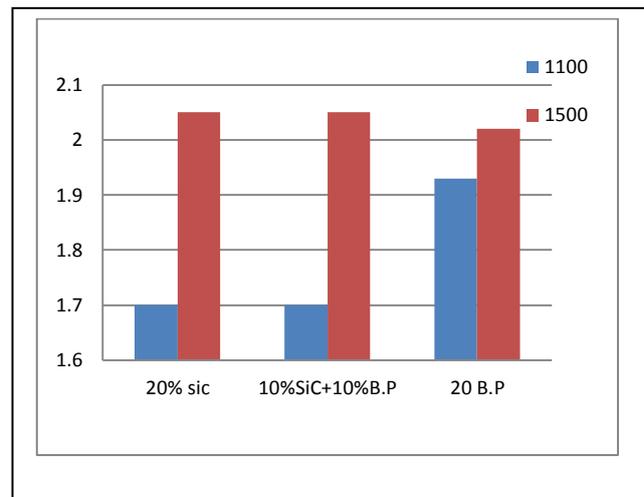
At the sintering temperature 1500°C, with the additives 10% sic + 10% bp, the value of the bulk density was large. This is due to the fact that the sic powder has a narrow particle size distribution and the bp powder with a wide size distribution leading to the best packing of the raw materials leading to an increase the rate of sintering at 1500. For example, Ring and shapell found that the sintering ability of a green body decreases by increasing the width of the particle size distribution and this effect is very clear at the intermediate stage when considering the particle size(13).

Figure 9 shows that, in the case of the primary mixture, 70% (70% K + 30% MK) is increased with the increase in the sintering temperature from 1100 to 1500. With note that at 1100 and with the addition of additives (15% sic + 15% bp), the bulk density is higher than the samples containing sic or bp separately. It can be said that sic and bp have different particle distribution, which causes good packing of powder particles in the green body, giving the highest bulk density after sintering. While the values were close when there were sic alone and B.P alone, the same behavior was found at 1500°C.

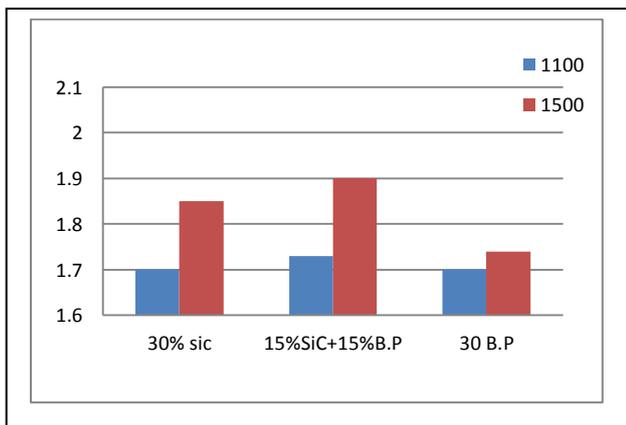
When the mixture is 60%, in figure 10, at sintering temperature 1100° C, the bulk density was greater in case of addition of 40% sic, while at 1500° C the addition of 20% B.P gave the largest value of the bulk density. This is due to the fact that the particle size is large and has a wide distribution, which causes better compacting of the deposits, which increases the densification during firing, represented by high value of bulk density density.



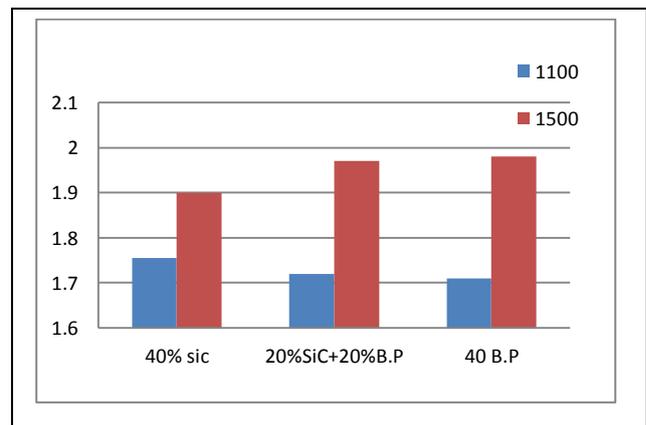
**Fig.7** The effect of the addition of SiC,B.P on the bulk density B.P on the of 90 (Kaaolin+Meyakaolin)



**Fig.8** The effects of the addition of SiC, bulk density of 80 (Kaaolin+Metakaolin)



**Fig.9** The effects of the addition of SiC, B.P on the bulk density of sample 70% (Kaaolin+Metakaolin)



**Fig.10** The effects of the addition of SiC, B.P on the of on the bulk density of sample of 60% (Kaaolin+Metakaolin)

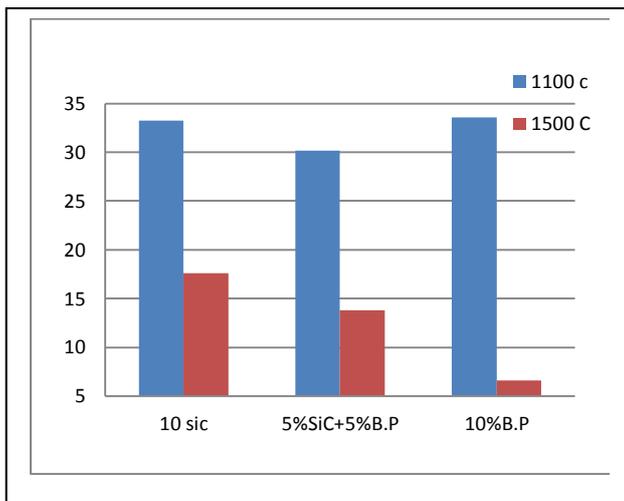
**Open porosity**

From the figures11,12,13 and 14it is noticed that open porosity decreases significantly when increasing the temperature of sintering from 1100 to 1500. This is due to the increase of sintering process and increases the affinity of granules, which reduces the gaps between them with the formation of new phases in the sintered body.

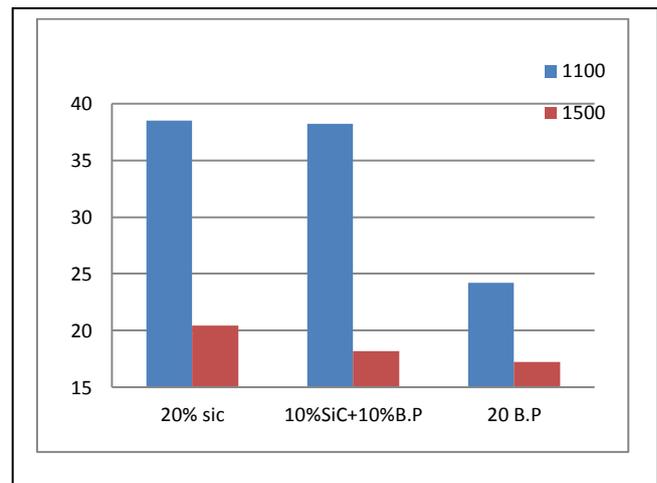
In the case of the ratio of the primary mixture of raw materials 90% and at the sintering temperature 1100 it was observed that the lowest open porosity ratio when both additives are together (5% SiC + 5% BP). Either when the addition of 10% SiC or 10% BP found that open porosity values are close.

At the sintering temperature 1500 , the open porosity decreases with the decrease of SiC, and the lowest porosity ratio is found at 10% BP. The presence of SiC is caused by increasing the open porosity ratio at 1500. This can be attributed to the presence of a percentage of iron in its substances which releases oxygen gas during sintering, causing open pores as it emerges from the sample.

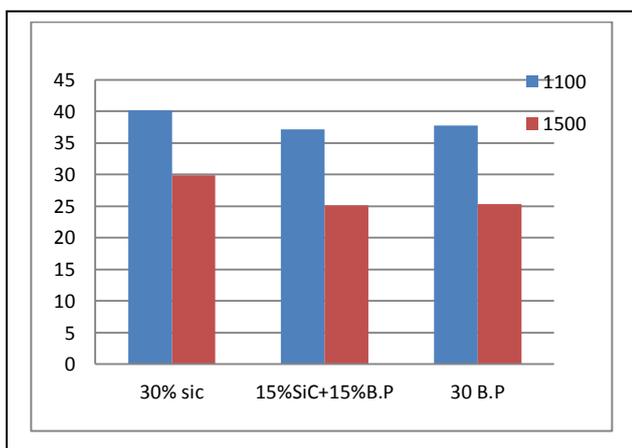
When the percentage of the main mixture was 80%, as seen in fig. 12, it was found the same behavior, where at 1100 ° C, open porosity ratios decreased with a decrease in sic ratio and the lowest open porosity ratio was at 20% B.P.



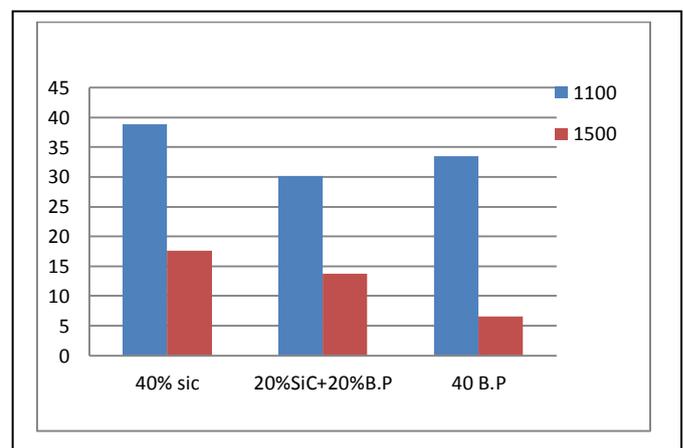
**Fig.11. The effect of the addition of SiC,B.P on the open porosity of sample of 90 (Kaaolin+Mevakaolin)**



**Fig.12. The effect of the addition of SiC,B.P on the open porosity of sample of 80 (Kaaolin+Meyakaolin)**



**Fig.13. The effect of the addition of SiC,B.P on the open porosity of sample of 70 (Kaaolin+Meyakaolin)**



**Fig.14. The effect of the addition of SiC,B.P on the open porosity of sample of 60 (Kaaolin+Meyakaolin)**

In fig.13, when the percentage of additives was 30% (the ratio of the main mixture 70%), and at temperatures of 1100 and 1500, the highest value of open porosity was found at 30% sic. Values were close at 20% B.P and (15% sic + 15% B.P).

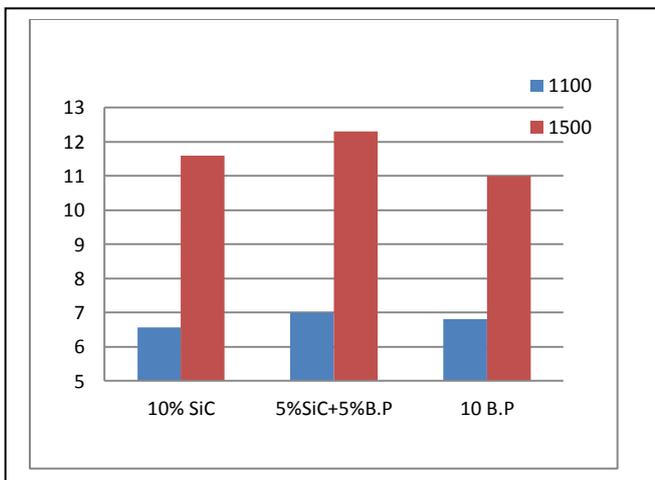
In the case of additives 40%, as shown in fig.14, at the firing temperature of 1100° C, the lowest open porosity ratio was at ( 20% sic + 20% B.P), indicating that the wide distribution of particle size distribution has reduced the emitted gases during sintering, thus reducing open porosity ratio. but samples containing 40 %B.P, at 1500° C, have the lowest percentage of open pores. This is due to the lack of iron in impurities in the powder of the fiery blocks, which means reducing the release of the released oxygen gas during sintering, reducing the open pores at 1500° C.

### Linear firing shrinkage

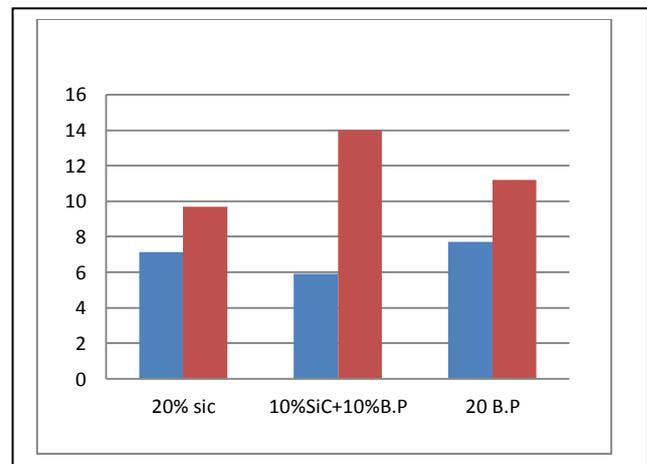
It is noticeable from the results of linear firing shrinkage that shrinkage increases by increasing the sintering temperature from 1100° C to 1500° C, due to the increase in the rate of sintering(densification) and the interactions that cause the decrease of spaces between the components of green body causing the occurrence of shrinkage with increasing firing temperature .Figure 15 shows that the ratio of (5% sic + 5% bp) resulted in the highest linear shrinkage rate at 1100 and 1500. Thus, a large granular distribution due to the presence of both sic and bp together resulted in a large linear firing shrinkage comparison with samples containing sic and B.P separately.

When the percentage of addition of 20%, as shown in the figure16 , at the firing temperature 1100, it was found that the largest linear shrinkage at the rate of 20% bp, due to the large particle size of the powder of fire brick, which leads to the formation of pores between the components of the green body before firing, which in turn increase shrinkage during sintering process. At additive10% sic + 10% bp, there was the least linear shrinkage of burning, which resulted from large particle distribution. At 1500 ° C, the 10% SiC + 10% BP had the largest linear shrinkage of burning.

At the rate of addition (10% sic + 10% bp), the lowest linear shrinkage rate was found, which resulted from the large granular distribution. At 1500 ° C, the ratio (10% sic + 10% B.P) had the largest linear firing shrinkage, which is an indication of a high rate of sintering.



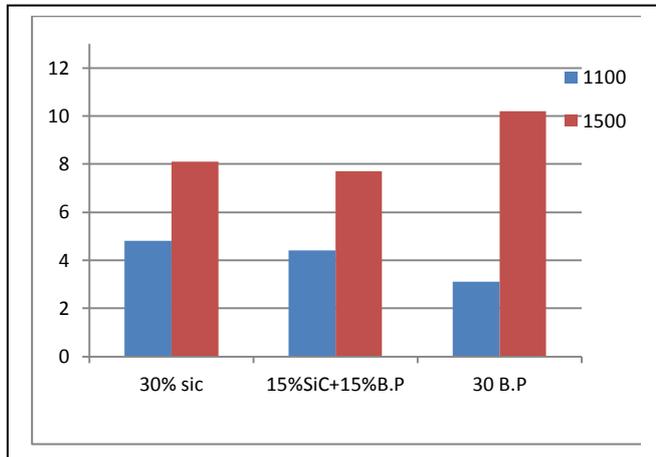
**Fig.15. The effect of the addition of SiC,B.P on the linear firing shrinkage of sample of 90 (Kaolin+Metakaolin).**



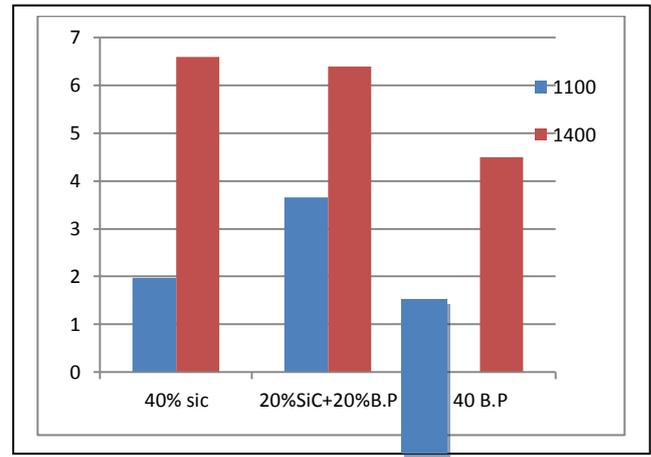
**Fig.16. The effect of the addition of SiC,B.P on the linear firing shrinkage of sample of 80**

Figure 17 shows the linear shrinkage behavior at 30%, and at the sintering temperature of 1100° C with the lowest linear shrinkage rate of 30% B.P. The remaining samples, which contain 30% sic and 15% SiC + 15% B.P), had close values of linear firing shrinkage. At the 1500 sintering temperature, the highest linear shrinkage value was 30% B.P. This is due to the large size of the powder of fire brick and wide width distribution, which causes many pores inside the green body before firing, causing an increase in shrinkage during sintering. In the case of the addition of (15% sic +15% bp), the lowest value of linear shrinkage was found. This is due to the large particle size distribution, which results from the presence of additives together causing the packing of the particles to be well-formed, resulting in a minimum shrinkage of firing temperature 1500° C .

In the case of 40% additives, at 1500° C sintering temperature, the highest linear shrinkage rate was found at 40% sic and conversely, when added 40 % of fire brick powder . In other words, linear shrinkage decreases with decreasing of sic . The reason is that the presence of SiC slows down the sintering process(14), and by 40% this effect is significant at 1500 ° C. At the firing temperature of 1100° C, the lowest value for linear shrinkage was found at 40% bp, while the shrinkage values were approximately equal in the addition of 40% bp and (20% sic + 20% bp). Thus, at the 1100 °C, the effect was clear for the small particle size and narrow distribution of the sic powder granularity, which caused the small amount of linear shrinkage.



**Fig.17. The effect of the addition of SiC,B.P on the linear firing shrinkage of sample of 70 (Kaolin+Metakaolin).**



**Fig.18. The effect of the addition of SiC,B.P on the linear firing shrinkage of sample of 60 (Kaolin+Metakaolin).**

#### 4.Conclusion

1. At the 1100 sintering temperature, the addition of sic was found to have a large bulk density density compared to BP and if both additives were present (5% SiC + 5% B.P), the maximum value of the bulk density was obtained.
2. when the ratio of the primary mixture is 80%,at 1100 °C, the values were found to be close at 20% sic and 10% SiC + 10%B.P,while at 20 % B.P the bulk density was greater.
3. At the sintering temperature 1500°C, with the additives 10% SiC + 10% B.P, the value of the bulk density was larger.
4. In the case of the primary mixture, 70%, at 1100° C and with the addition of additives (15% SiC + 15% B.P), the bulk density is higher than the samples containing SiCor B.P separately.
5. When the mixture is 60%, in , at sintering temperature 1100° C, the bulk density was greater in case of addition of 40% sic, while at 1500 the addition of 20% B.P gave the largest value of the bulk density.
6. At the sintering temperature 1500 ° C, the open porosity decreases with the decrease of SiC, and the lowest porosity ratio is found at 10% BP.
7. When the percentage of additives was 30% (the ratio of the main mixture 70%), and at temperatures of 1100 and 1500, the highest value of open porosity was found at 30% SiC.
8. the ratio of (5% SiC + 5% B.P) resulted in the highest linear shrinkage rate at 1100 and 1500° C.
9. At 1100° C, the ratio of addition (10% SiC + 10% B.P), the lowest linear shrinkage rate was found,. At 1500 ° C, the ratio (10% SiC + 10% B.P) had the largest linear firing shrinkage.
10. In the case of 40% additives, at 1500° C sintering temperature, the highest linear shrinkage rate was found at 40% SiC and conversely

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