REDUCING KAOLIN SHRINKAGE BY USING KAOLIN GROG

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ABSTRACT

In this work, the suitability of using local Kaolin and Kaolin Grog to reduce shrinkage in clay product like firebrick was experimentally investigated and the optimal ratio of Kaolin grog constituent determined. Ten samples of different compositions were fired at a temperature of 1200ºC. Three of the samples (samples 8, 9, and 10) crumbled during firing. The surviving samples gave the following limits of results:- Total shrinkage: 28%- 9.8%; apparent porosity: 63.6% - 77.13%; water absorption: 47.83% - 60.2%; bulk density: $1.329g/cm^3$ - $1.281g/cm^3$; apparent density: $5.60g/cm^3$ – $3.65g/cm^3$; and compressive strength: 61.5MPa – 52.11MPa. The results showed that the first seven samples had good shrinkage and compressive strength. Mixing ratio of 35:100 (representing weight in grams of water and Kaolin respectively) which represent the critical water content.

الخلاصة

في هذا البحث تم إضافة مادة الكروك إلى طين الكاؤولين المحلي ودراسة تأثيره على التقلص الحاصل في المنتجات الطينية كالطابوق الحراري وتحديد النسبة الأمثل من مادة الكروك المضافة. تم تحضير عشر عينات بنسب مختلفة من مادة الكروك وحرقت بدرجة حرارة 1200م°. ثلاثة من العينات المحضرة وهي(العينة رقم 8، 9، 10) قد انهارت خلال عملية الحرق. النماذج المتبقية قد أعطت حدود النتائج الآتية:- التقلص الكلــي 28%-9.8%؛ المسامية الظاهرية 63.6%-77.13%؛ امتــصاصية المـــاء 47,83%-60,2%؛ الكثافــة الحجمية 1,329غم/سم³-1,281غم/سم³؛ الكثافة الظاهرية 5,6-غم/سم³-3,65غم/سم³؛ مقاومة الانــضغاط (61,5-52,11)ميكاباسكال. أوضحت النتائج بان النماذج السبعة الأولى أعطت تقلــص ومقاومـــة انــضغاط جيدة. نسبة خلط الماء المستخدمة هي 35:100 (وتمثل الوزن بالغرام بالنسبة للطين مع الماء على التــوالي) والتي تمثل نسبة الماء الحرجة بالنسبة لطين الكاؤولين المستخدم.

Keywords:- **Kaolin clay, grog, total shrinkage, compressive strength, physical tests.**
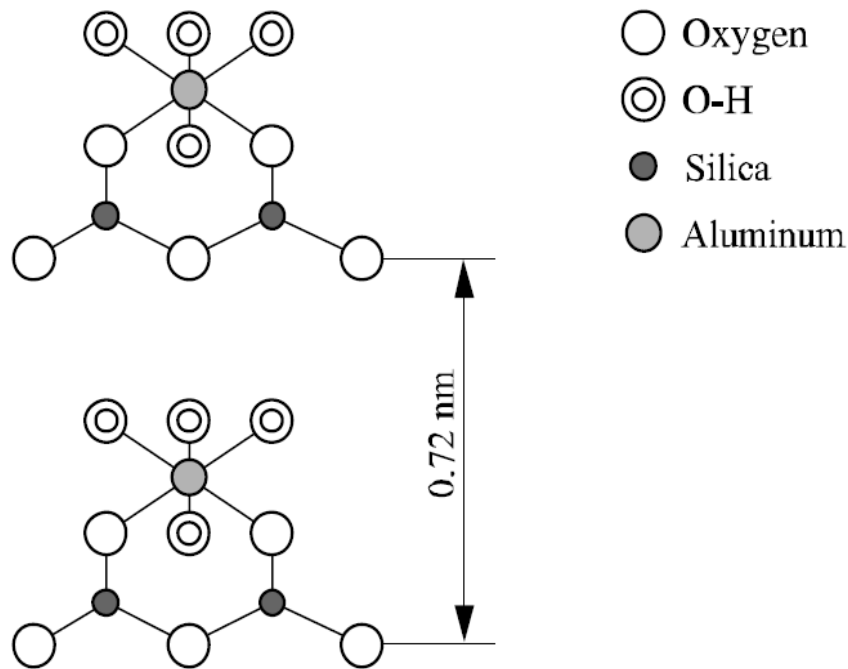
INTRODUCTION

Clays are hydrated silico-aluminous minerals whose structure is made up of a stacking of two types of layers containing, respectively, aluminum in an octahedral environment and silicon in tetrahedral coordination. Their large specific surface (10 to 100 m^2g^{-1}), their plate-like structure and the physicochemical nature of their surface enable clays to form, with water, colloidal suspensions and plastic pastes. This characteristic is largely used during the manufacture of silicate ceramics insofar as it makes it possible to prepare homogenous and stable suspensions, suitable for casting, pastes easy to manipulate and green parts with good mechanical strength. By extension, the term clay is often used to denote all raw materials with proven plastic properties containing at least one argillaceous mineral.

The impurities present in these natural products contribute to a large extent to the coloring of the shard **[F. Bergaya *et al*, 2006].**

Kaolinite, $Si_2Al_2O_5$ $(OH)_4$ or $Al_2O_3,2SiO_2,2H_2O$, is the most common among the argillaceous minerals used in ceramics and is the principal mineral for kaolin clay. A projection of its crystalline structure is represented in Figure (1). It consists of an alternate stacking of $[Si_2O_5]^{-2}$ and $[Al_2(OH)_4]^{+2}$ layers, which confer to it a lamellate character favorable to the development of plates. The degree of crystallinity of the kaolinite present in clays is highly variable. It depends largely on the genesis conditions and the content of impurities introduced into the crystalline lattice[**Carty W.M. and Senapati U., 1998**].

The term kaolin is used to designate the white clays whose principal mineral is kaolinite ($Al_2Si_2O_5(OH)_4$). Its particles are usually hexagonal with diameters ranging from 0.05 to 10 μm (average 0.5μm) since this mineral is a product of the decomposition of feldspars and micas present in pegmatites and micaceous schist, its frequently found together with other minerals such as quarts, sulfides, feldspar, mica and iron and titanium oxides, among others [**J. A. Gonzalez et al, 2007**].

Kaolin is used for different industrial applications due to its physical and chemical properties. Its main used in the paper industry (45 %), refractories and ceramics (31 %), fiber glass (6 %), cement (6 %), rubber and plastic (5 %), paint (3 %) and others (4 %) **[Murray, 2002].**

Fig. (1). Projected representation of the structure of kaolinite

In order to elaborate an efficient technology of construction a better recognition of shrinkage and cracking processes of clays is needed. It has been shown that cracking resulting from shrinkage processes occurs especially if the material is homogenized and close to its saturation point [**Hartge and Horn, 1999**].

The shrinkage process has been divided into normal shrinkage phase and residual shrinking phase [**eg Hartge, 1965; Junkersfeld, 1995; Junkersfeld and Horn, 1997**]. It has been shown by Albiker [**cited in Horn et al., 2001**] that if clay samples were subjected to compaction on the wet branch of the Proctor curve i.e. at moisture content

above the optimum, they showed normal shrinkage properties, while compaction at water contents below the optimum resulted in residual shrinkage behaviour.

Moreover, [**Bauer et al. 2001**] have shown that the shrinkage potential of kaolinite clay was much lower on the dry branch of the Proctor curve. Recently, it has been suggested by [**Horn and Stêpniewski, 2004**] that compacting the soil on the dry branch of the Proctor curve, combined with recognition of hydraulic and mechanical interactions of the mineral substrate, offers a promising method of avoiding the crack formation.

MATERIALS AND METHODS
1-Materials and Samples Preparation:-

The materials used in this work are kaolin, kaolin Grog, and water. Kaolin grog was produced from same kaolin clay used in this research (Grog is clay which has been fired then ground up). Grog can come in many particle sizes, from fine to coarse. It is used to reduce shrinkage in clay bodies. The size of grog used in this research was under mesh 200 (<75 μm) and produced by firing kaolin clay at 1200 $^{\circ}$C for 2 hours then ground by using ball mill and sieved to the required size.

Kaolin clay and kaolin grog which are used in this research have specification shown in **Table (1)** and it was local clay from Iraq, Kaolin grog was prepared from the same Kaolin which mixed with.

Table 1. Kaolin Specification

KAOLIN ORE	
GRAIN SIZE	59.0 - 61.0%
KAOLINITE	95.0% min
HARDNESS	3 -4
LOSS ON IGNITION	14.5% max
SPECIFIC GRAVITY	2.6
pH	6 - 8
Al_2O_3	39.0% min
SiO_2	43.5% min
Fe_2O_3	0.7% max
TiO_2	0.6% max
MnO	0.3% max
P_2O_5	0.3% max
Na_2O	0.2% max
CaO	0.3% max
MgO	0.3% max
K_2O	0.2% max
SO_3	0.03% max

 The samples were prepared by hand (moulding was carried out by hand that is, hand moulding). Using ten different weight percent of grog with kaolin as shown in Table(2) and after mixing the clay with water using electrical mixer with the determined weight percentage to make the recipe then filled in the die with slight pressure to make the sample rigid. Dies made from metal and having cylindrical shape and the following dimensions (5cm Length X 3cm Diameter), to make the samples and after we extracting the samples from the mould, specimens were left to dry atmospherically for 24 hr. then dried in a mechanical (controlled humidity) dryer at 110ºC for another 24 hr, after completing the drying process samples were fired at 1200 ºC for 2 hr.

Table 2. Composition of Samples by weight (Total weight = 30g)

Sample code	Kaolin percentage%	Grog percentage%
1	100	0
2	95	5
3	90	10
4	85	15
5	80	20
6	75	25
7	70	30
8	65	35
9	60	40
10	55	45

2-Tests:

2-1- *Bulk density, Apparent density and Apparent porosity Tests*

   The test specimens were dried at 110ºC for 24 hours to ensure total water loss, and later fired up to 1200ºC in an electric furnace for 2 hr. Their fired weights were measured and recorded. Then allowed to cool and then immersed in a beaker of water. Bubbles were observed as the pores in the specimens were filled with water. Their soaked weights were measured and recorded. They were then suspended in a beaker one after the other using a sling and their respective suspended weights were measured and recorded.

   Their respective bulk density, apparent density and apparent porosity were calculated using the formulae [**Chesti A. R., 1986]:**

Bulk Density = D/(W-S) (g/cm^3)   **eq.**   **(1)**

Apparent Density = D/(D-S) (g/cm^3)   **eq.**   **(2)**

Apparent Porosity = (W-D)/(W-S)*100   eq.   (3)

where: D =Weight of fired specimen, S =Weight of fired specimen suspended in water, and W = Weight of soaked specimen suspended in air.

2-2- Water Absorption

The sintered specimens were dried to a constant weight, cooled to room temperature, and then weighted. The tiles were immersed in distilled water and boiled for three hours.
The heating was stopped and the samples were allowed to remain immersed in the water for 24 h. The samples taken out and excess water was removed from their surfaces by wiping with a damp cloth. The samples were again weighted. The water absorption was calculated using the formula:

$$WA = \frac{wf - wi}{wi} \cdot 100$$

eq.   (4)

where WA is the water absorption (%), w_i is the dry mass (g), w_f is the fired mass (g).

2-3- Shrinkage Test

Test specimens from each composition were dried at 110ºC for 24 hours to ensure total water loss. The test specimens were then measured (in terms of dimension) and their values were noted as dry lengths.

The test specimens were also fired in an electric furnace to temperature of 1200ºC. They were allowed to cool. The specimens were weighed and measured. And the fired weight and fired length were recorded. For each sample, three different specimens were tested and the averages of the above parameters were calculated and recorded.

The drying shrinkage, firing shrinkage and the total shrinkage were calculated for each test specimen using the following formula [**Norsker H., 1987**]:

% Avg Drying Shrinkage = (OL-DL)/OL*100   eq.   (5)

%Avg Firing Shrinkage = (DL-FL)/FL*100   eq.   (6)

%Total Shrinkage = (OL-FL)/OL*100   eq.   (7)

where: *OL* means original length; *DL* stands for dry length and *FL* is fired length.

The drying shrinkage indicates to some degree the plasticity of the mixture. A large drying shrinkage means that mixture could absorb much water, which in turn indicates fine mixture particles. The firing shrinkage indicates how fusible the mixture is. A high shrinkage normally means a lower melting point. The total shrinkage of refractory bodies tells how much bigger we should make our moulds [**Norsker H., 1987**].

2-4- Compressive Strength

Compressive strength tests were performed on a standard mechanical machine. Test specimens having length twice its diameter, measuring (20*10) mm for each samples were dried and fired at 1200ºC. Each of them was placed one after the other on the bearing edges of the compression machine apart. Loads were then applied at the middle

of the specimens, uniformly at 1.25 kgf per minute. This test is done according to the ASTM (C 773-88) standard. The loads at which the specimens failed were calculated from the relation [ASTM, 1988]:

$$\sigma = F/A \quad \text{eq.} \tag{8}$$

where σ = the compressive strength (MPa), F = load at fracture (N), A = the cross section area of the specimen(mm^2)

3- RESULTS AND DISCUSSION

The results obtained for the different experiments carried out in this investigation are presented in Tables (3 and 4) and the property trends are discussed below.

Compositions 7 - 10 crumbled during firing. This suggests poor plastic mixture, that is, grog content was too high for clay to bind. Compositions 1 - 6 showed a good level of block properties.

Fig.2 shows that the bulk density of kaolin samples increasing with increasing grog percentage that's because that the grog will occupy bigger size in samples than water and hence it does not absorb water like clay which make density increase with increasing its percentage, while apparent density seems to be decrease with increasing grog percentage as shown in **Fig.3** and the reason behind that is the size of the samples were they increased with increasing grog percentage which made the apparent density decrease.

Fig.4 shows that porosity of kaolin samples decrease as grog percentage increase that's because grog is a fired clay and it absorb small quantity of water which reduce water in samples consequently reduce samples porosity which mainly emanated from excess moisture.

Fig.5 shows that water absorption decrease with increasing grog percentage in clay samples, water absorption related to the porosity of the samples and because grog don't burns out and don't leaves plenty of pores, which leads to low absorption of water by that sample.

Grog is used in pottery and sculpture to add a gritty, rustic texture called "tooth"; it also reduces shrinkage and aids even drying. This prevents defects such as cracking, crow feet patterning, and lamination. The coarse particles open the green clay body to allow gases to escape. It also adds structural strength to hand-built and thrown pottery during shaping although it can diminish fired strength [**Benjamin I. U. et al., 2006**]. **Fig.(6, 7 and 8)** shows the shrinkage of clay samples related to grog percentage which indicate that the shrinkage decrease with increasing grog percentage but that's could be deleterious to other properties such as fired strength as shown in **Fig.9** where compressive strength of clay samples increase with increasing grog percentage until grog percentage reached 20% then decreasing in the clay strength observed above that percentage which means that increasing in grog percentage not always useful for clay but there is limit percentage to get the optimum properties.

4- CONCLUSIONS

Based on the properties of the brick samples tested and analyzed in this study, it can be concluded that:
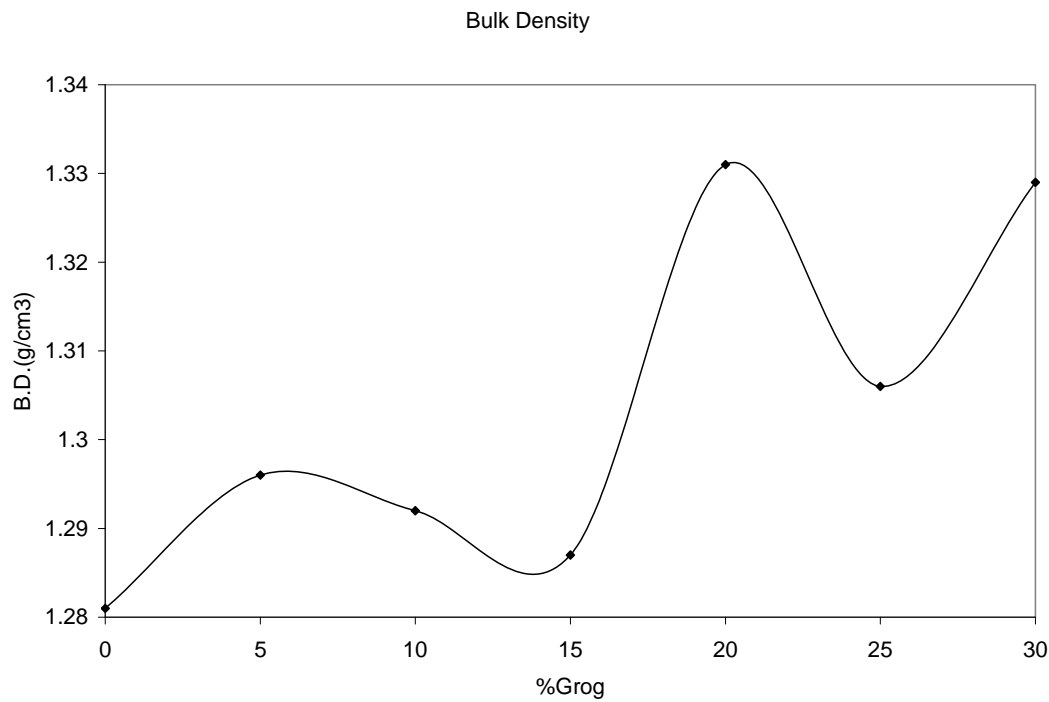
1. The local raw materials – kaolin and kaolin grog - are suitable for the production of building firebricks with good properties.

2. The mixing ratio used for sample 5 which was 20% grog gave the best combination of properties such as strength and total shrinkage.

3. Grog percentage above 20% affects the clay properties badly and made samples crumbled during firing as noticed in samples (8, 9 and 10).

4. We can use just kaolin as matrix and reinforced material to make suitable brick for building which effectively reduces the cost of building materials in Iraq.
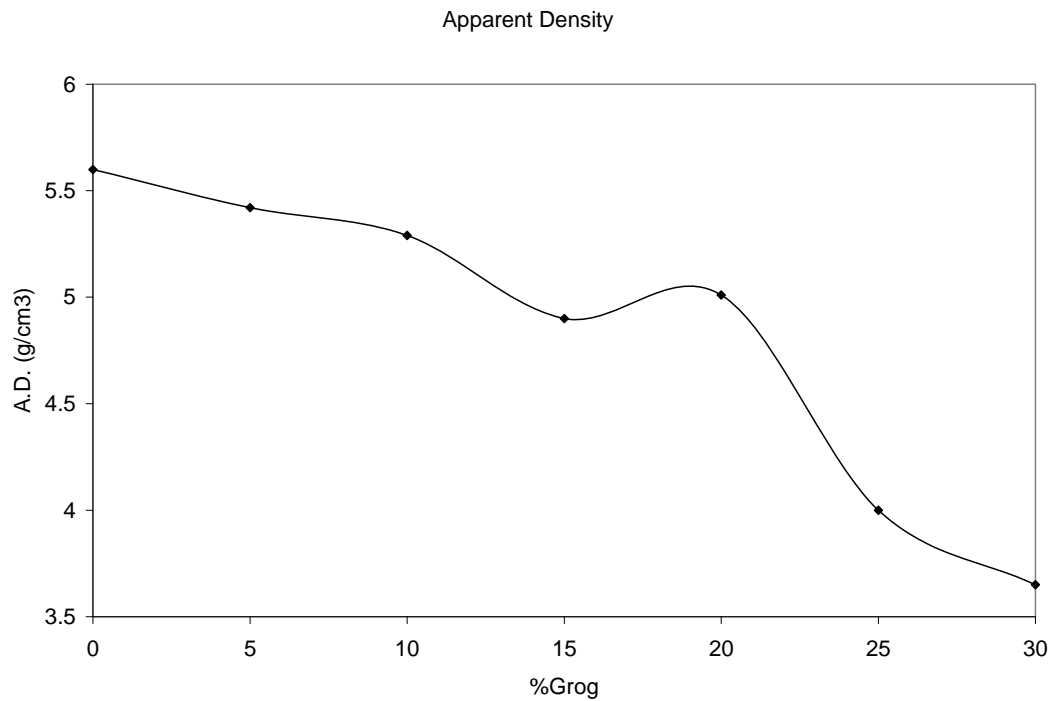
Table 3. *Shrinkage Values*

Sample code	Original length (cm)	Dry length (cm)	Fired length (cm)	Dry shrinkage %	Fired shrinkage %	Total shrinkage %	Temperature (ºC)
1	5	4.3	3.6	14	19.4	28	1200
2	5	4.5	3.82	10	17.80	23.6	1200
3	5	4.775	4.1	4.5	16.46	18	1200
4	5	4.78	4.1	4.4	16.58	18	1200
5	5	4.8	4.285	4.0	12.01	14.3	1200
6	5	4.8	4.333	4.0	10.77	13.34	1200
7	5	4.85	4.51	3	7.53	9.8	1200
8	5	4.88	-	2.4	-	-	1200
9	5	4.91	-	1.8	-	-	1200
10	5	4.9	-	2	-	-	1200

Table 4. *Percentage of Apparent Porosity, Water Absorption, Apparent Density, Bulk Density and Compressive strength*

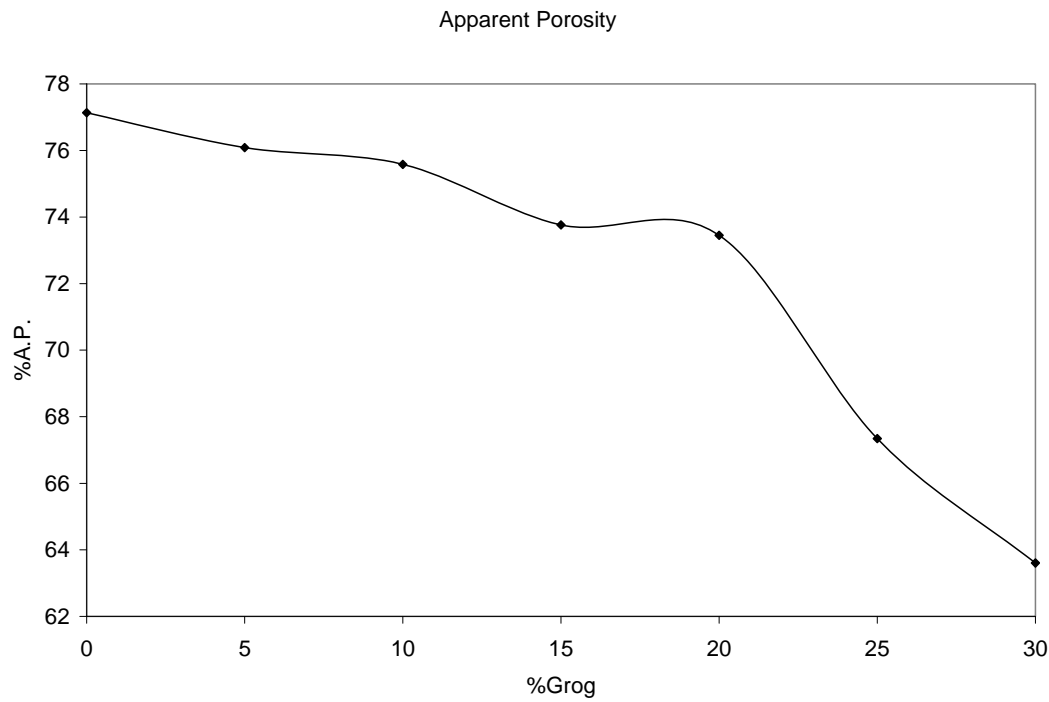| Test specimen code	Fired weight(D) (g)	Suspended weight(S) (g)	Soaked weight(W) (g)	% Apparent porosity	% Water absorption	Apparent density (g/cm^3)	Bulk density (g/cm^3)	Tempt. ºC	Compressive strength(MPa)
1	24.2	19.88	38.77	77.13	60.2	5.60	1.281	1200	52.11
2	23.0	18.76	36.5	76.09	58.69	5.42	1.296	1200	54.80
3	22.02	17.86	34.9	75.58	58.49	5.29	1.292	1200	57.35
4	21.3	16.96	33.5	73.76	57.27	4.90	1.287	1200	60.01
5	19.86	15.9	30.82	73.45	55.18	5.01	1.331	1200	61.5
6	19.2	14.4	29.1	67.34	51.56	4.0	1.306	1200	56.6
7	18.67	13.56	27.6	63.60	47.83	3.65	1.329	1200	53.2
8	-	-	-	-	-	-	-	1200	-
9	-	-	-	-	-	-	-	1200	-
10	-	-	-	-	-	-	-	1200	-

Bulk Density

Fig (2): Effect of Grog percentage on Bulk Density of Kaolin
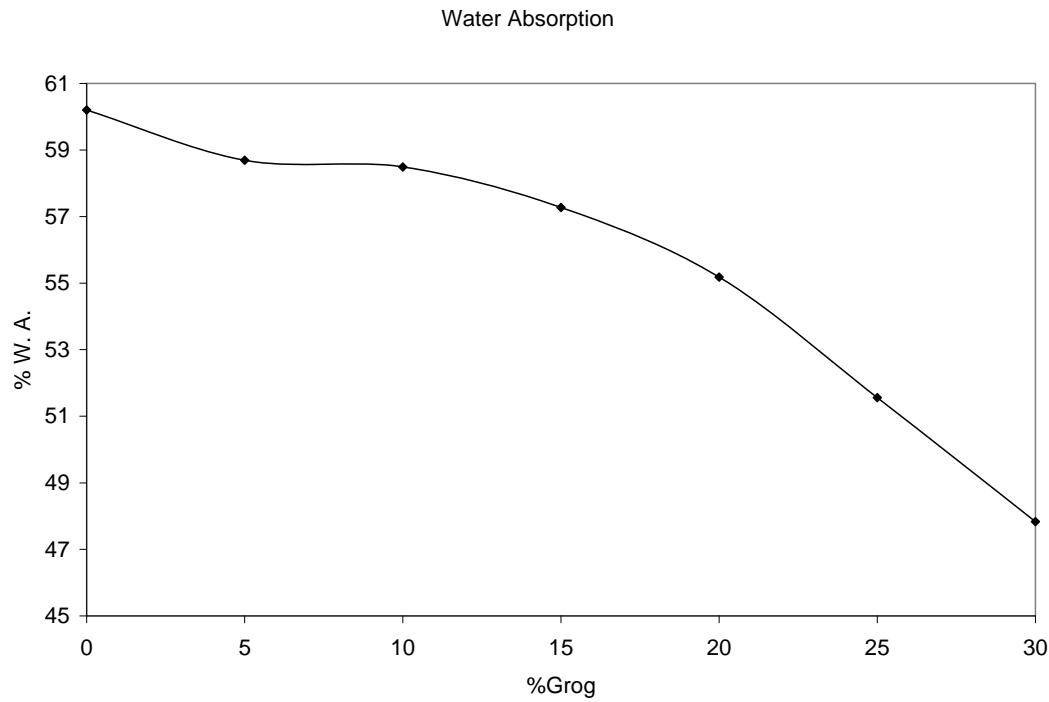
Apparent Density

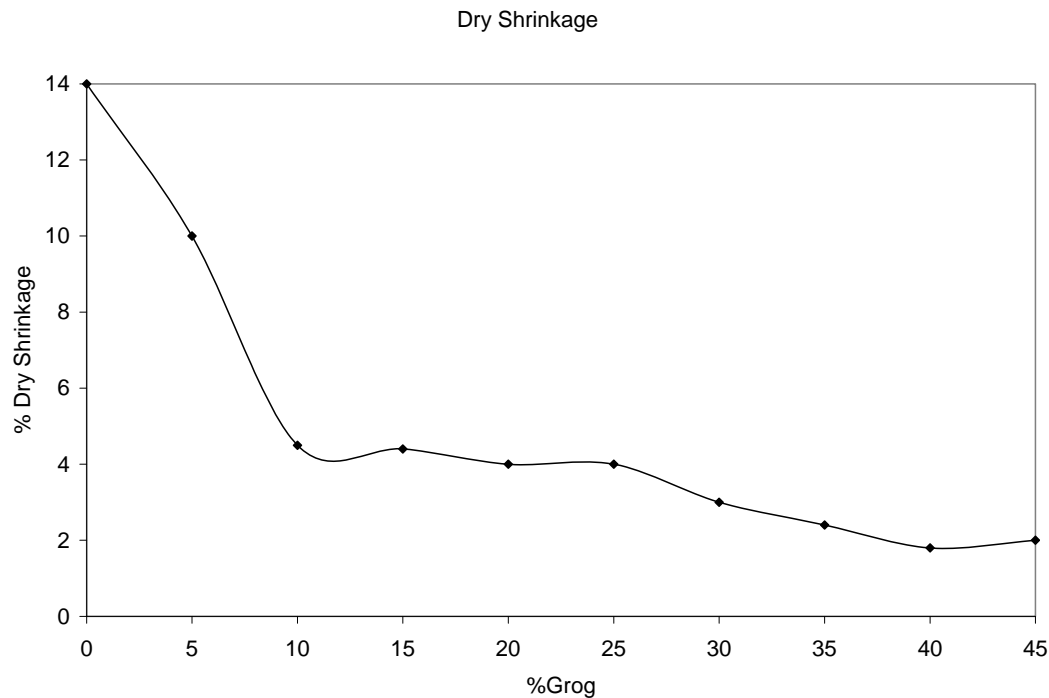**Fig (3):** Effect of Grog percentage on Apparent Density of Kaolin
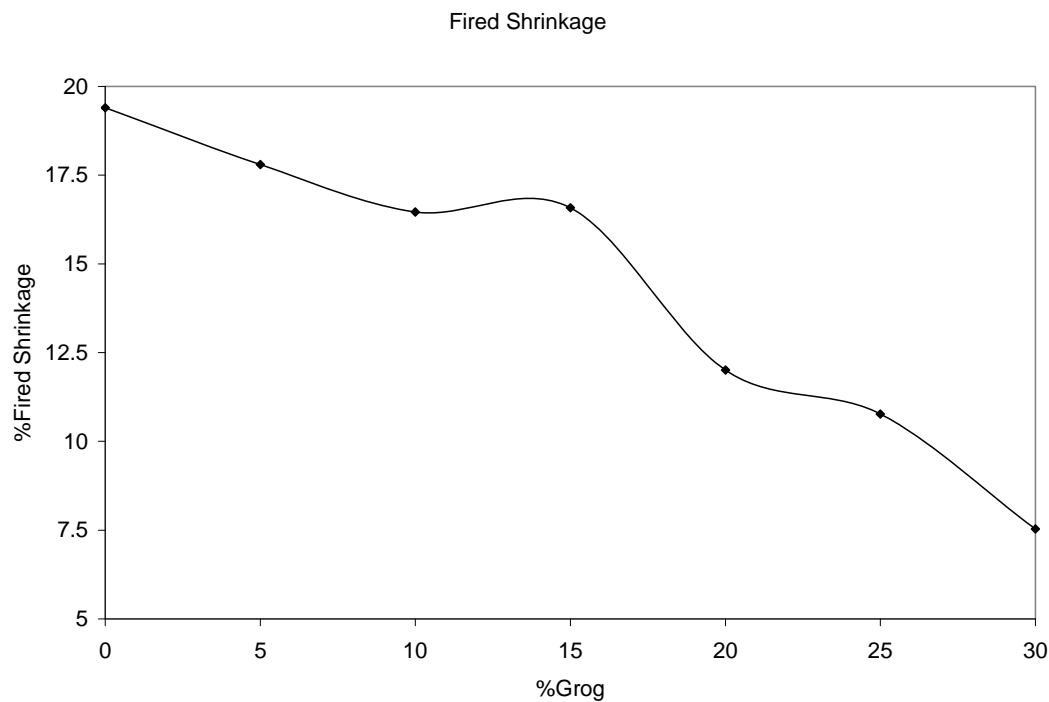
Apparent Porosity

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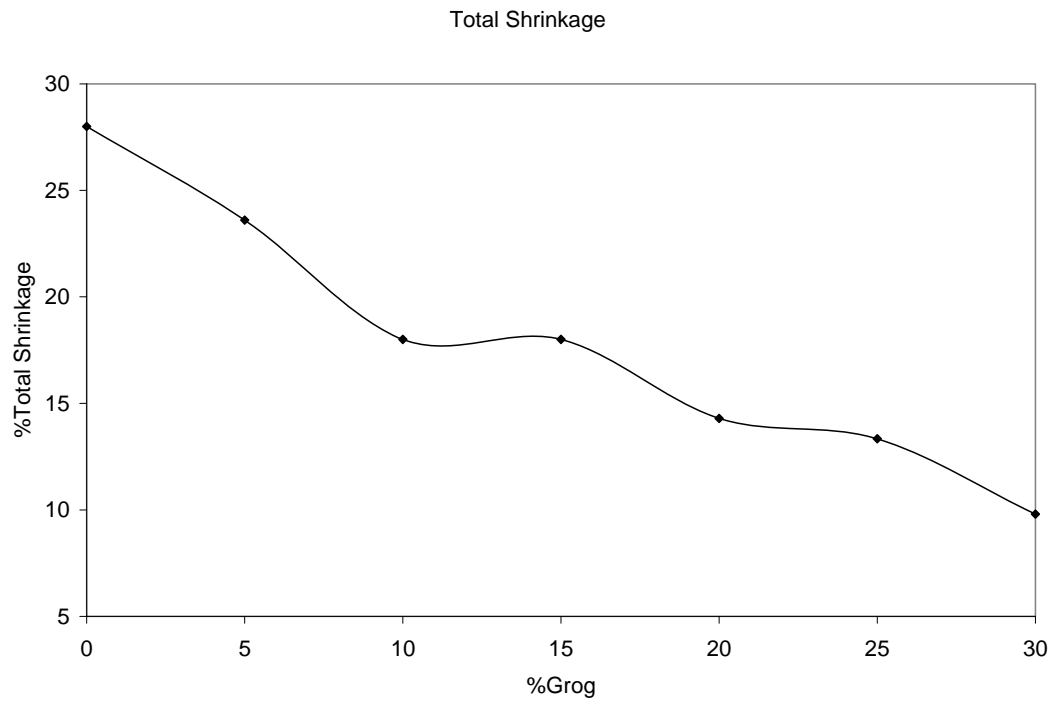
Water Absorption

Fig (5): Effect of Grog percentage on Water Absorption of Kaolin
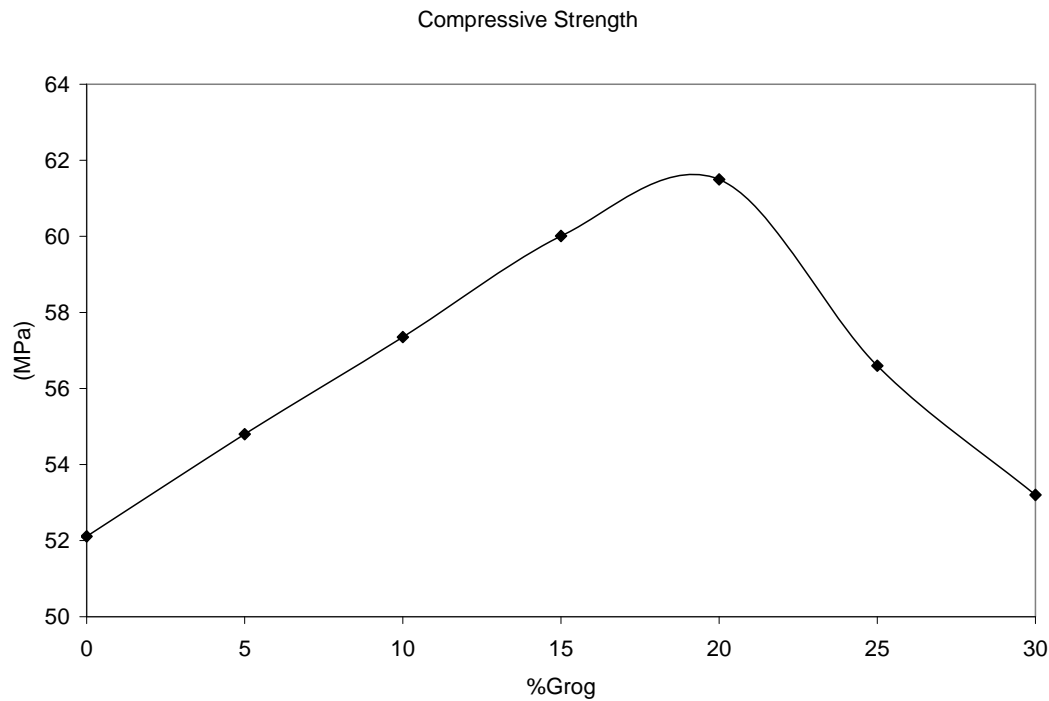
Dry Shrinkage

Fig (6): Effect of Grog percentage on Dry Shrinkage of Kaolin

Fired Shrinkage

Fig (7): Effect of Grog percentage on Fired Shrinkage of Kaolin

Total Shrinkage

Fig (8): Effect of Grog percentage on Total Shrinkage of Kaolin

Compressive Strength

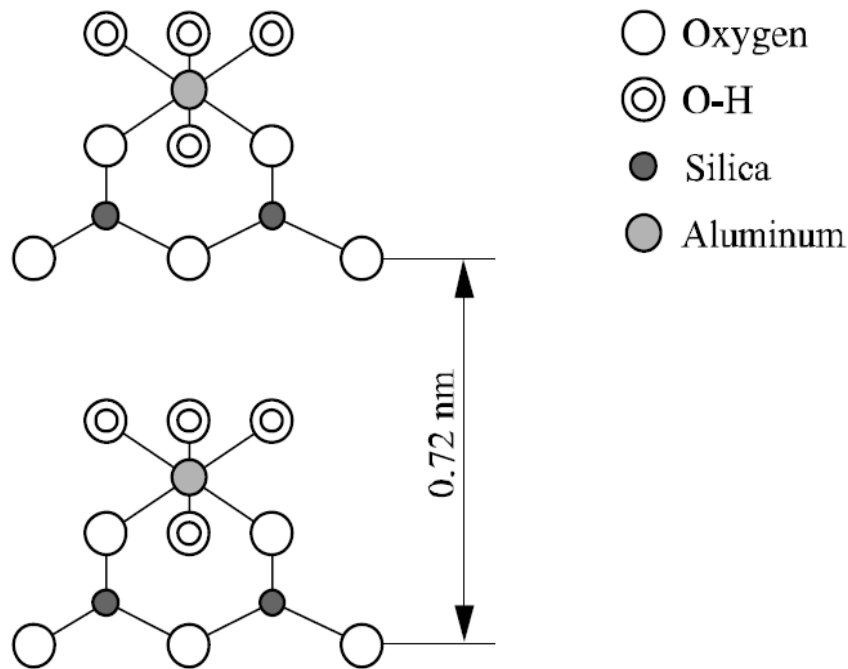**Fig (9):** Effect of Grog percentage on Compressive Strength of Kaolin

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2-2- Water Absorption

The sintered specimens were dried to a constant weight, cooled to room temperature, and then weighted. The tiles were immersed in distilled water and boiled for three hours.
The heating was stopped and the samples were allowed to remain immersed in the water for 24 h. The samples taken out and excess water was removed from their surfaces by wiping with a damp cloth. The samples were again weighted. The water absorption was calculated using the formula:

$$WA = \frac{wf - wi}{wi} \cdot 100$$

                                                 eq.   **(4)**

where WA is the water absorption (%), w_i is the dry mass (g), w_f is the fired mass (g).

2-3- Shrinkage Test

Test specimens from each composition were dried at 110ºC for 24 hours to ensure total water loss. The test specimens were then measured (in terms of dimension) and their values were noted as dry lengths.

The test specimens were also fired in an electric furnace to temperature of 1200ºC. They were allowed to cool. The specimens were weighed and measured. And the fired weight and fired length were recorded. For each sample, three different specimens were tested and the averages of the above parameters were calculated and recorded.

The drying shrinkage, firing shrinkage and the total shrinkage were calculated for each test specimen using the following formula [**Norsker H., 1987**]:

     % Avg Drying Shrinkage = (OL-DL)/OL*100   eq.   **(5)**

     %Avg Firing Shrinkage = (DL-FL)/FL*100   eq.   **(6)**

        %Total Shrinkage = (OL-FL)/OL*100   eq.   **(7)**

where: *OL* means original length; *DL* stands for dry length and *FL* is fired length.

The drying shrinkage indicates to some degree the plasticity of the mixture. A large drying shrinkage means that mixture could absorb much water, which in turn indicates fine mixture particles. The firing shrinkage indicates how fusible the mixture is. A high shrinkage normally means a lower melting point. The total shrinkage of refractory bodies tells how much bigger we should make our moulds [**Norsker H., 1987**].

2-4- Compressive Strength

Compressive strength tests were performed on a standard mechanical machine. Test specimens having length twice its diameter, measuring (20*10) mm for each samples were dried and fired at 1200ºC. Each of them was placed one after the other on the bearing edges of the compression machine apart. Loads were then applied at the middle

of the specimens, uniformly at 1.25 kgf per minute. This test is done according to the ASTM (C 773-88) standard. The loads at which the specimens failed were calculated from the relation [ASTM, 1988]:

$$\sigma = F/A \quad \text{eq.} \tag{8}$$

where σ = the compressive strength (MPa), F = load at fracture (N), A = the cross section area of the specimen(mm^2)

3- RESULTS AND DISCUSSION

The results obtained for the different experiments carried out in this investigation are presented in Tables (3 and 4) and the property trends are discussed below.

Compositions 7 - 10 crumbled during firing. This suggests poor plastic mixture, that is, grog content was too high for clay to bind. Compositions 1 - 6 showed a good level of block properties.

Fig.2 shows that the bulk density of kaolin samples increasing with increasing grog percentage that's because that the grog will occupy bigger size in samples than water and hence it does not absorb water like clay which make density increase with increasing its percentage, while apparent density seems to be decrease with increasing grog percentage as shown in **Fig.3** and the reason behind that is the size of the samples were they increased with increasing grog percentage which made the apparent density decrease.

Fig.4 shows that porosity of kaolin samples decrease as grog percentage increase that's because grog is a fired clay and it absorb small quantity of water which reduce water in samples consequently reduce samples porosity which mainly emanated from excess moisture.

Fig.5 shows that water absorption decrease with increasing grog percentage in clay samples, water absorption related to the porosity of the samples and because grog don't burns out and don't leaves plenty of pores, which leads to low absorption of water by that sample.

Grog is used in pottery and sculpture to add a gritty, rustic texture called "tooth"; it also reduces shrinkage and aids even drying. This prevents defects such as cracking, crow feet patterning, and lamination. The coarse particles open the green clay body to allow gases to escape. It also adds structural strength to hand-built and thrown pottery during shaping although it can diminish fired strength [**Benjamin I. U. et al., 2006**]. **Fig.(6, 7 and 8)** shows the shrinkage of clay samples related to grog percentage which indicate that the shrinkage decrease with increasing grog percentage but that's could be deleterious to other properties such as fired strength as shown in **Fig.9** where compressive strength of clay samples increase with increasing grog percentage until grog percentage reached 20% then decreasing in the clay strength observed above that percentage which means that increasing in grog percentage not always useful for clay but there is limit percentage to get the optimum properties.

4- CONCLUSIONS

Based on the properties of the brick samples tested and analyzed in this study, it can be concluded that:
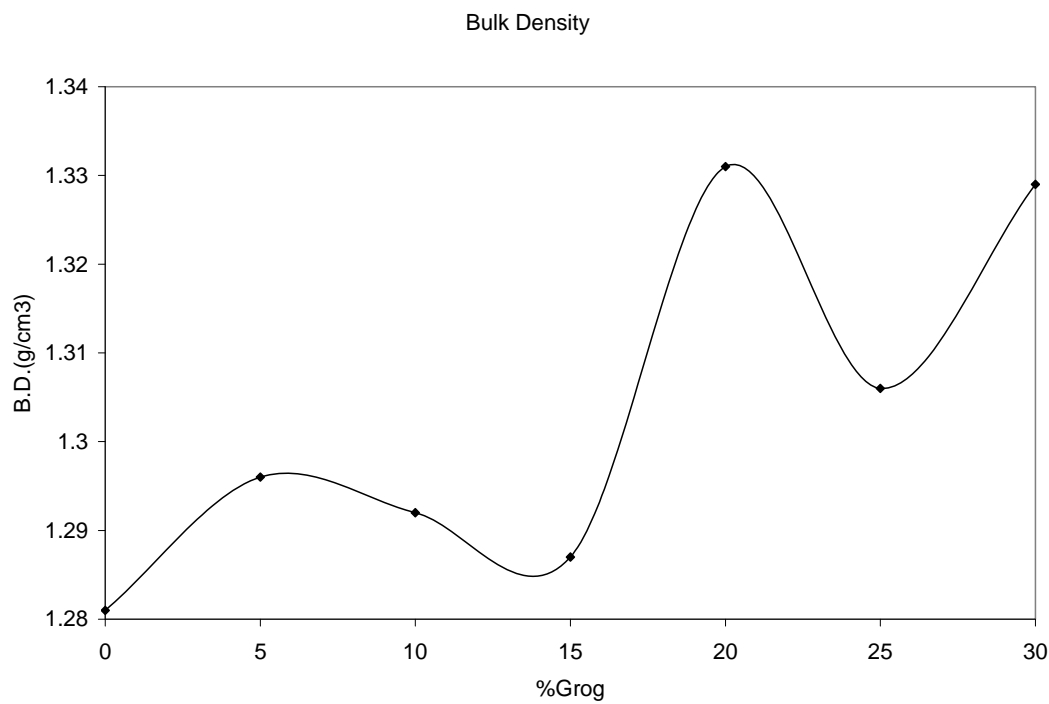
1. The local raw materials – kaolin and kaolin grog - are suitable for the production of building firebricks with good properties.

2. The mixing ratio used for sample 5 which was 20% grog gave the best combination of properties such as strength and total shrinkage.

3. Grog percentage above 20% affects the clay properties badly and made samples crumbled during firing as noticed in samples (8, 9 and 10).

4. We can use just kaolin as matrix and reinforced material to make suitable brick for building which effectively reduces the cost of building materials in Iraq.
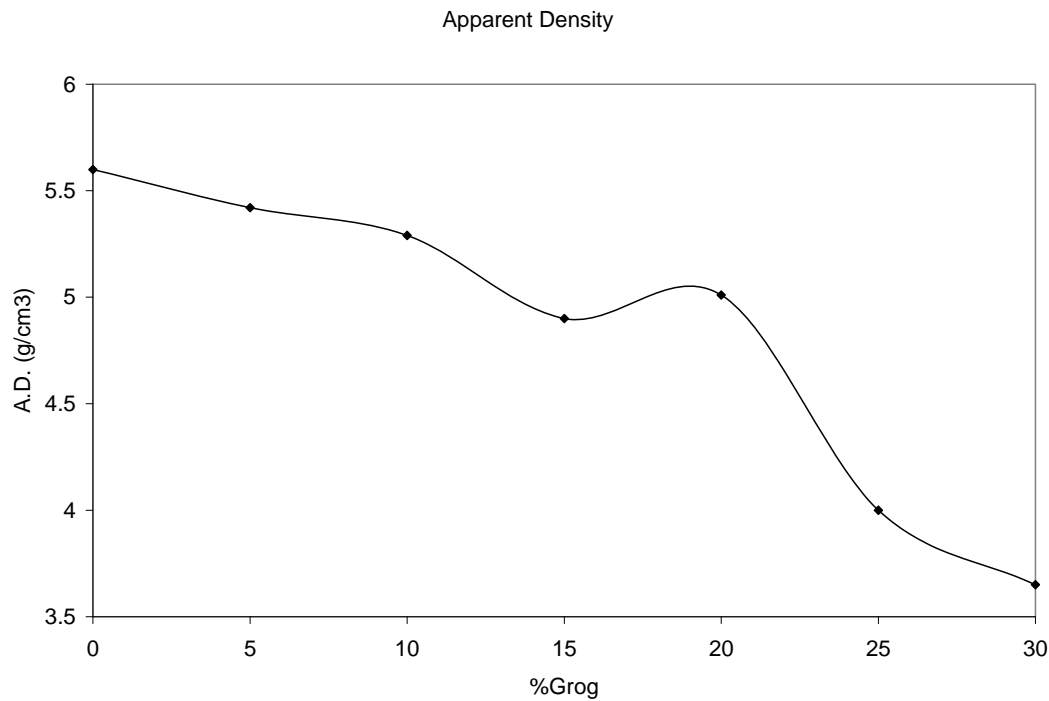
Table 3. *Shrinkage Values*

Sample code	Original length (cm)	Dry length (cm)	Fired length (cm)	Dry shrinkage %	Fired shrinkage %	Total shrinkage %	Temperature (ºC)
1	5	4.3	3.6	14	19.4	28	1200
2	5	4.5	3.82	10	17.80	23.6	1200
3	5	4.775	4.1	4.5	16.46	18	1200
4	5	4.78	4.1	4.4	16.58	18	1200
5	5	4.8	4.285	4.0	12.01	14.3	1200
6	5	4.8	4.333	4.0	10.77	13.34	1200
7	5	4.85	4.51	3	7.53	9.8	1200
8	5	4.88	-	2.4	-	-	1200
9	5	4.91	-	1.8	-	-	1200
10	5	4.9	-	2	-	-	1200

Table 4. *Percentage of Apparent Porosity, Water Absorption, Apparent Density, Bulk Density and Compressive strength*
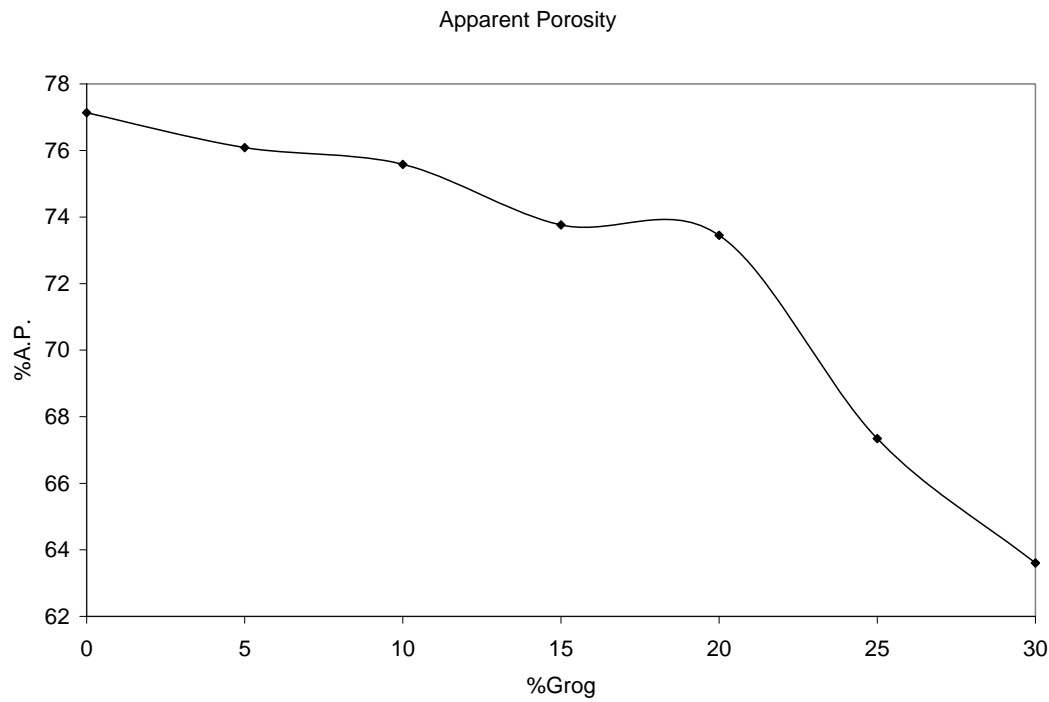
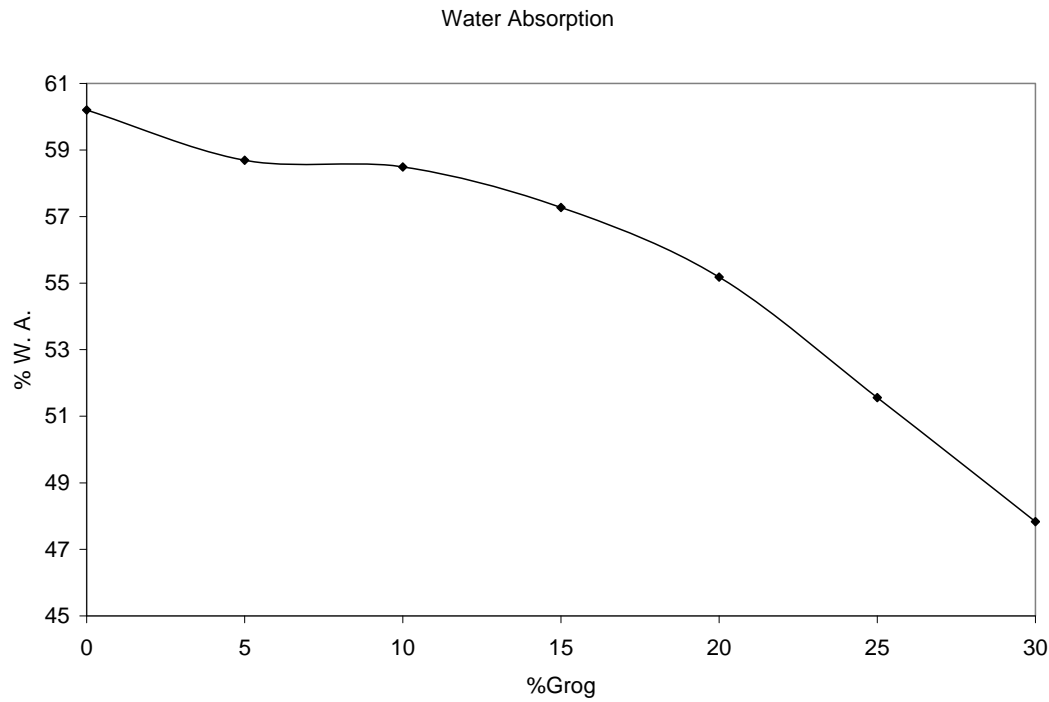| Test specimen code	Fired weight(D) (g)	Suspended weight(S) (g)	Soaked weight(W) (g)	% Apparent porosity	% Water absorption	Apparent density (g/cm^3)	Bulk density (g/cm^3)	Tempt. ºC	Compressive strength(MPa)
1	24.2	19.88	38.77	77.13	60.2	5.60	1.281	1200	52.11
2	23.0	18.76	36.5	76.09	58.69	5.42	1.296	1200	54.80
3	22.02	17.86	34.9	75.58	58.49	5.29	1.292	1200	57.35
4	21.3	16.96	33.5	73.76	57.27	4.90	1.287	1200	60.01
5	19.86	15.9	30.82	73.45	55.18	5.01	1.331	1200	61.5
6	19.2	14.4	29.1	67.34	51.56	4.0	1.306	1200	56.6
7	18.67	13.56	27.6	63.60	47.83	3.65	1.329	1200	53.2
8	-	-	-	-	-	-	-	1200	-
9	-	-	-	-	-	-	-	1200	-
10	-	-	-	-	-	-	-	1200	-

Bulk Density

Fig (2): Effect of Grog percentage on Bulk Density of Kaolin
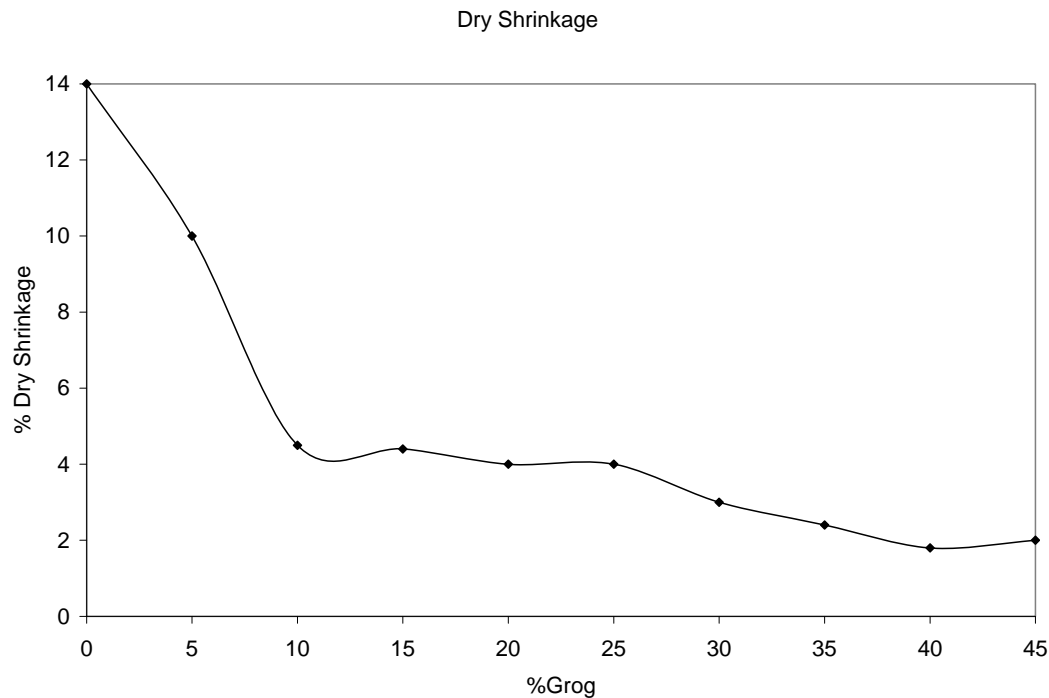
Apparent Density

Fig (3): Effect of Grog percentage on Apparent Density of Kaolin
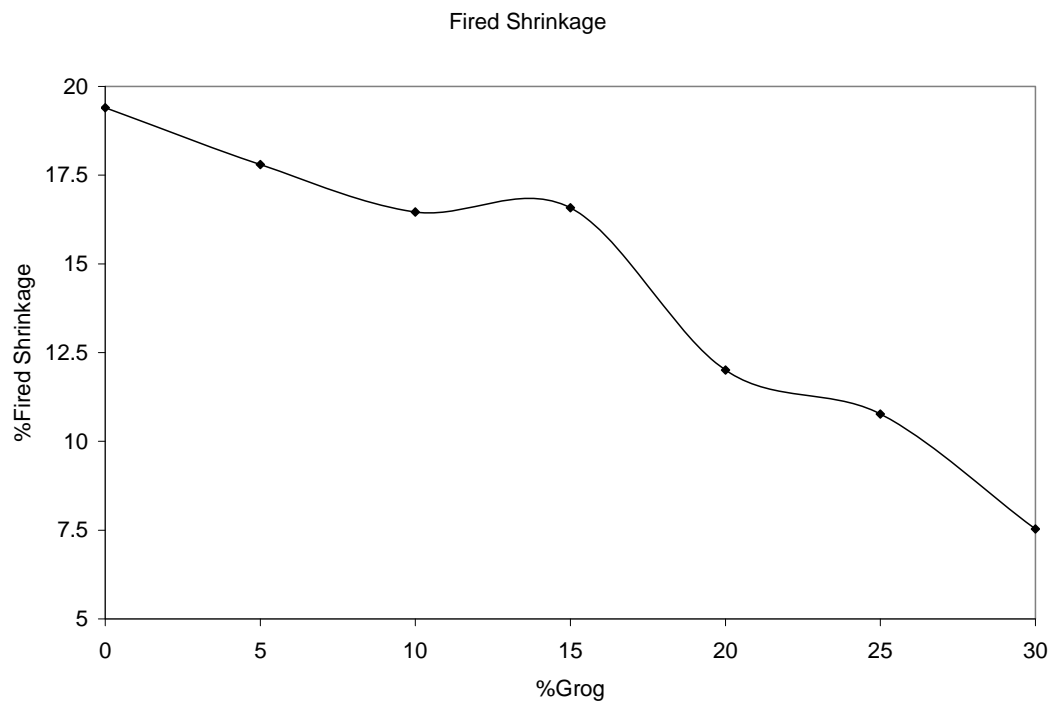
Apparent Porosity

Fig (4): Effect of Grog percentage on Apparent Porosity of Kaolin
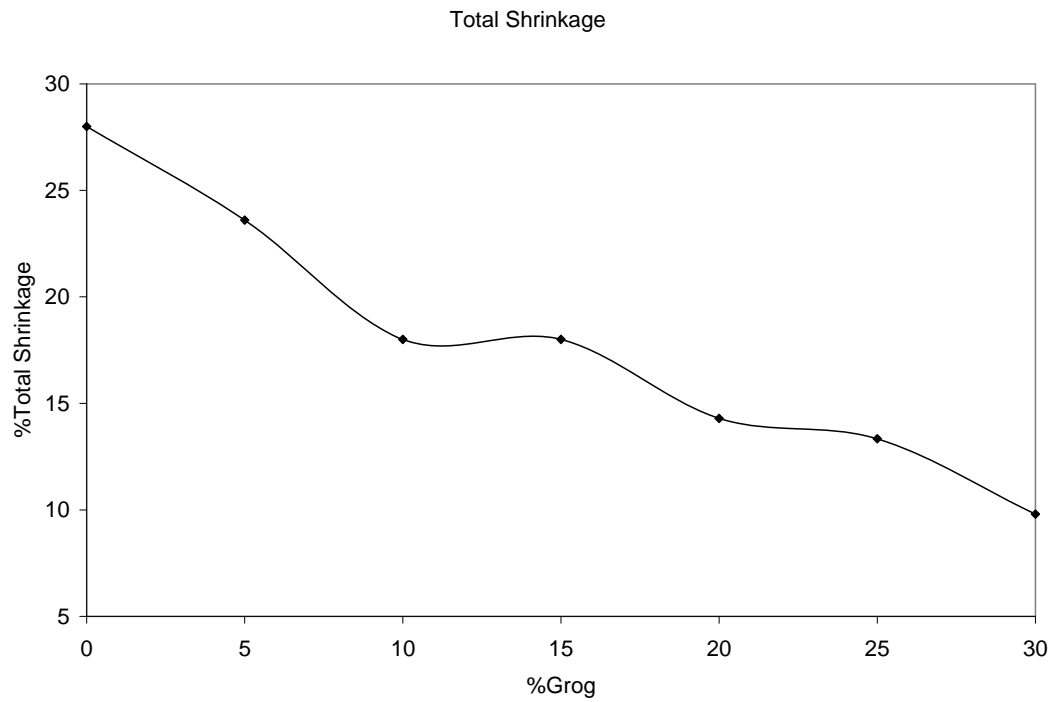
Water Absorption

Fig (5): Effect of Grog percentage on Water Absorption of Kaolin
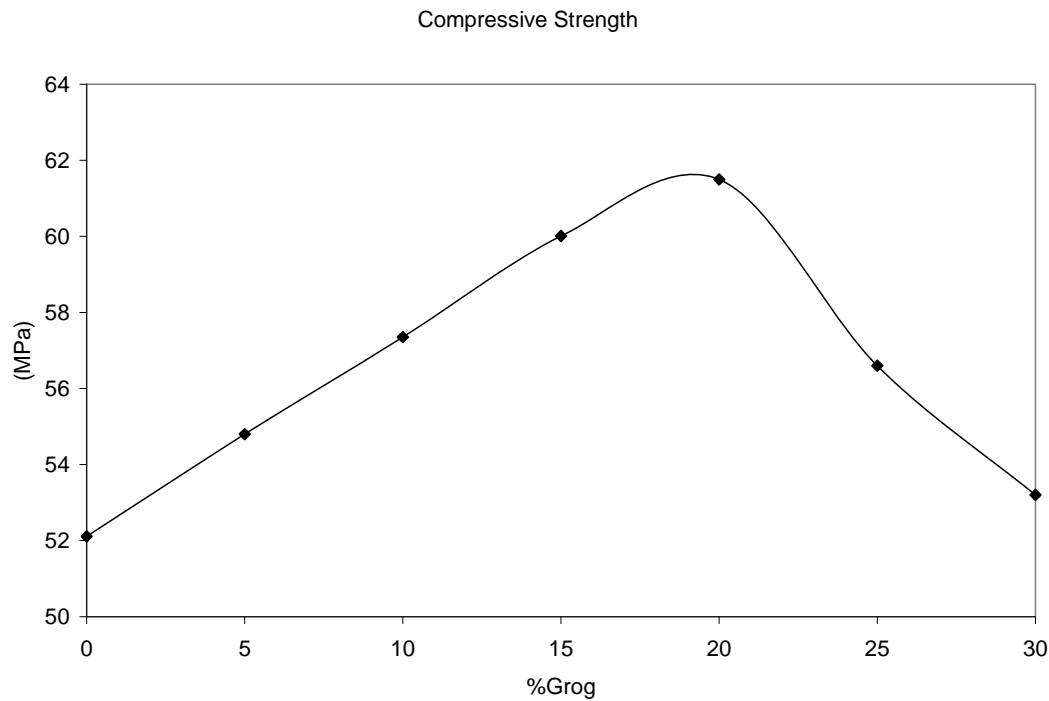
Dry Shrinkage

Fig (6): Effect of Grog percentage on Dry Shrinkage of Kaolin

Fired Shrinkage

Fig (7): Effect of Grog percentage on Fired Shrinkage of Kaolin

Total Shrinkage

Fig (8): Effect of Grog percentage on Total Shrinkage of Kaolin

Compressive Strength

Fig (9): Effect of Grog percentage on Compressive Strength of Kaolin

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