

## Assessment of Utilizing Gypsum and Clays in Manufacturing Thermal Insulators.



\* Mazin Y. Tamar-Agha, \*\* Suzan N. D. Mohamed

\*Dept. of Earth Sciences, College of Science, University of Baghdad, Baghdad /Iraq.

\*\*The State Company of the Geological Survey and Mining, Baghdad / Iraq.

### Abstract

Utilization of gypsum, clay and marl from the Fatha Formation (Middle Miocene) in Darbandi Bazian – Sulaimani Government were assessed for manufacturing thermal insulators. The raw materials were analyzed mineralogically, chemically and physically. They were then crushed and ground to less than 125  $\mu\text{m}$ . Ten batches were prepared by mixing 70/30, 60/40, 50/50, 40/60 and 30/70 weight % ratio of gypsum to clay or marl. 210 test samples were formed by semi-dry pressing, oven dried at 105°C and fired at 1000, 1050 and 1100 °C with a rise of 100 °C/h and one hour soaking time. The fired briquettes were tested later on to assess their suitability as thermal insulator. The results of physico-mechanical tests showed that the linear shrinkage is 0.13 to 3.75 %, apparent porosity is 30.8 to 41.4 %, water absorption 17.4 to 28.0 %, bulk density is 1.48 to 1.78  $\text{g}/\text{cm}^3$ , compressive strength is 100.1 to 438.5  $\text{kg}/\text{cm}^2$  and modulus of rupture is 31.5 to 76.5  $\text{kg}/\text{cm}^2$ . Meanwhile, four samples were selected to investigate their thermal conductivity (k) and found that it ranges between 0.3 and 0.4  $\text{w}/\text{m} \cdot ^\circ\text{C}$ , which reveals that they are good insulators.

**Keywords:** Thermal insulators, structural bricks, ceramics, gypsum.

### Introduction

Construction materials used for the main residential areas in northern Iraq changed drastically during the last five decades from the traditional building stones to concrete blocks. Other parts of the country maintain their reliance on the traditional common bricks, which were used for several millennia. Thermal conductivity, represented as “k” factor, of concrete block is known to be at least twice as much as that of building stones [1]. In contrast, the climate in the region encourages utilization of building materials with higher thermal insulating properties rather than higher thermal conductivity. This issue encouraged researchers to strive for alternatives. Some workers suggested the use of common brick made out of Neogene and Paleogene clays instead of agricultural soils [2, 3] for

the fact that soil conservation is essential to agriculture. Al-Nuaimi (2000) used various mixtures of gypsum, phosphogypsum or plaster of Paris with kaolinitic clays in the preparation of thermal insulators [4].

This work attempts to prepare construction materials with higher thermal - insulation properties from raw materials widely available in the region, namely gypsum and mudrocks. The briquettes produced in this attempt were tested for their physico-mechanical and thermal insulation properties.

### Methods

Chemical, mineralogical and physical properties of the new materials were first determined. The major constituents were analyzed as oxides by various methods in the laboratories of the

\*E-mail: mtamaragha@yahoo.com

\*\* Cited from her Ph.D. Thesis

MgO, Na<sub>2</sub>O and K<sub>2</sub>O were analyzed by AAS only, CaO was analyzed by AAS and titration with EDTA, Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> were analyzed by UV-spectrophotometer and titration with EDTA, SiO<sub>2</sub> and SO<sub>3</sub> by gravimetric methods and finally L.O.I. (loss on ignition) by calculating the difference in weight of dried samples (105°C) and heated samples (1000°C for one hour). Mineralogy was studied by X-ray diffractometry (XRD) using Philips PW1130 with Cu Kα radiation. Grain size analysis of the clay and marl were executed by hydrometer method (ASTM C775-79). Atterberg's Limits were measured by the methods specified in ASTM standard specification D424-59.

The raw materials were crushed and ground in porcelain mill to particle size less than 125 μm and homogenized. Ten mixes were prepared consisting of 70/30, 60/40, 50/50, 40/60 and 30/70 (weight %) of gypsum/clay or marl, respectively (Table 1). The mixes represent two groups according to their content of the plastic raw materials, i.e. whether clay or marl. Group 1 consists of gypsum and clay and Group 2 consists of gypsum and marl. 150 test tiles (8x4x1 cm) and 60 test cylinders (3x6 cm) were formed by semi-dry pressing method using 8% moisture content and pressure 250 kg/cm<sup>2</sup>. The test samples were oven dried at 105 °C for 24 hours then divided into three groups, the first fired at 1000 °C, the second fired at 1050 °C and the third fired at 1100 °C. The rise of temperature was 100 °C/hour and the soaking time was one hour.

Properties of the fired briquettes were assessed by several methods. XRD was used to investigate the phase transformation after heating. The color was checked using Munsell Color Chart. Linear shrinkage of the briquettes was measured following the procedure specified by ASTM C326-76 whereas other sintering properties were measured using ASTM C373-72. Compressive strength and modulus of rupture of the test samples were measured following the procedures in ASTM C133-81. Thermal conductivity of briquettes of four mixtures was measured using Lee's disc procedure (each measurement was repeated twice). For comparison, another briquette was formed consisting of clay only, to represent common brick, and treated in the same procedure as above and fired at 1100°C with the same firing program and its thermal conductivity was tested similarly.

#### Raw Materials

Three raw materials are used in the present study, namely gypsum, clay and marl. All the raw materials are collected from the Fatha Formation (Middle Miocene) near Darbandi Bazian-Sulaimani Governorate (Fig. 1). These rock types are present in the Fatha Formation nested in cyclic pattern (Fig. 2) and are extensively exposed. Each complete cycle consists of greenish grey marl, limestone, gypsum and reddish brown mudstone (in ascending order). The Neogene sequence, represented by the Fatha, Injana, Mukdadiya and Bai Hassan Formations (in ascending order), are especially well exposed on the southwestern limb of Bazian Anticline.

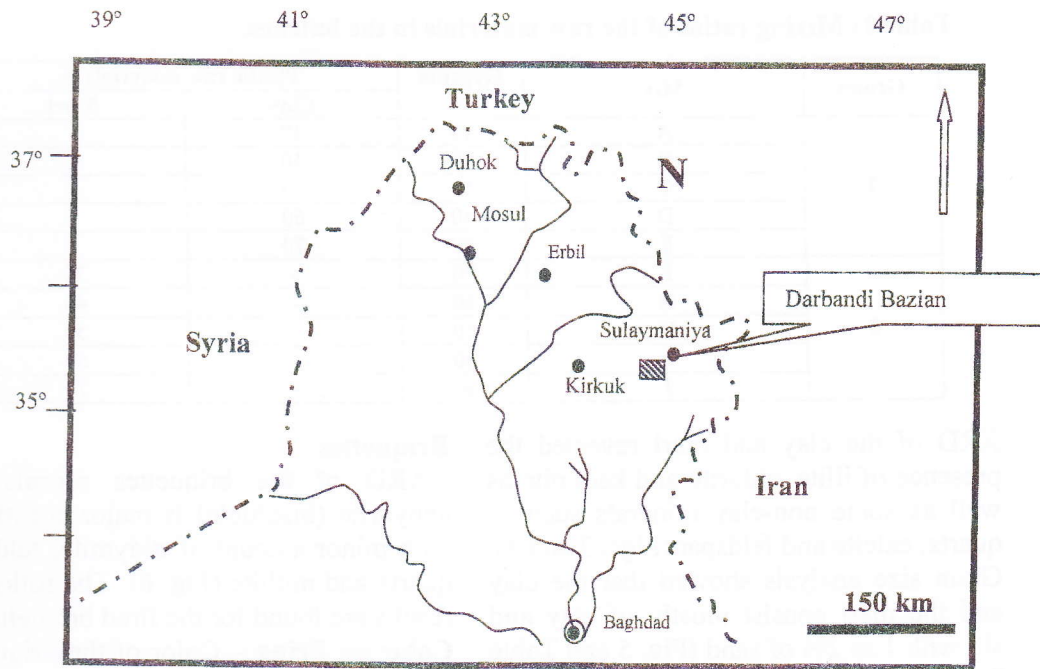


Figure 1: Location map.

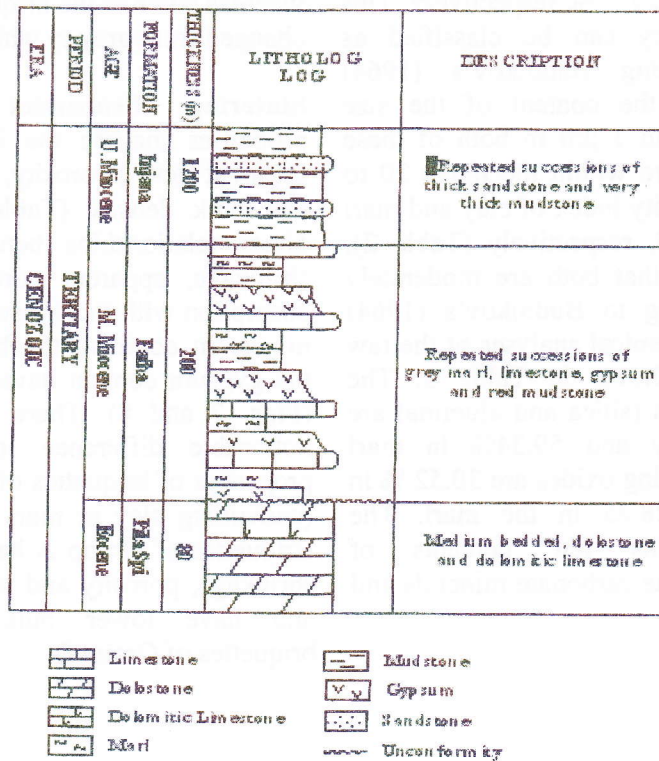


Figure 2: Columnar section of part of the Fatha Formation in Darbandi Bazian area.

Table 1: Mixing ratios of the raw materials in the batches.

Group	Mix	Gypsum %	Plastic raw materials %	
			Clay	Marl
1	A	70	30	-
	B	60	40	-
	C	50	50	-
	D	40	60	-
	E	30	70	-
2	F	70	-	30
	G	60	-	40
	H	50	-	50
	I	40	-	60
	J	30	-	70

XRD of the clay and marl revealed the presence of illite, chlorite and kaolinite as well as some non-clay minerals such as quartz, calcite and feldspar (Figs. 3 and 4). Grain size analysis showed that the clay and the marl consist mostly of clay and silt with 1 to 2% of sand (Fig. 5 and Table 2). In both the marl and the clay, the content of the size fraction finer than 1  $\mu\text{m}$  is 25.7 and 27.7 %, respectively. This means that they can be classified as "dispersed" using Budnikov's (1964) scheme, since the content of the size fraction less than 1  $\mu\text{m}$  in both of these raw materials are within the range 20 to 60% [5]. Plasticity index of clay and marl are 9.5 and 8.9, respectively (Table 2), which indicate that both are moderately plastic according to Budnikov's (1964) scheme. The chemical analyses of the raw materials are shown in Table 3. The refractory oxides (silica and alumina) are 54.83% in clay and 59.34% in marl whereas the fluxing oxides are 30.52 % in the clay and 28.25 in the marl. The gypsum contains few percents of impurities such as carbonate minerals and clay minerals.

### Briquettes

XRD of the briquettes reveals that anhydrite (insoluble) is major constituent with minor amount of tridymite, feldspar, quartz and mullite (Fig. 6). The following results are found for the fired briquettes:

**Color on firing** – Color of the briquettes of Group 1 changed from light brown before firing to light beige after firing whereas color of the briquettes of Group 2 changed from grey to white.

**Sintering Properties** – Sintering properties include the linear shrinkage, apparent (open) porosity, water absorption and bulk density (Table 4). There are direct relationships between the linear shrinkage, apparent porosity and water absorption with the gypsum content in the mixes. In contrast, the bulk density and the gypsum content have inverse relation (Figs. 7 and 8). There is in general a noticeable difference in the sintering properties of briquettes of Groups 1 and 2 (containing clay or marl in their mixes). Briquettes of Group A have higher linear shrinkage, porosity and water absorption, and have lower bulk density than briquettes of Group B.

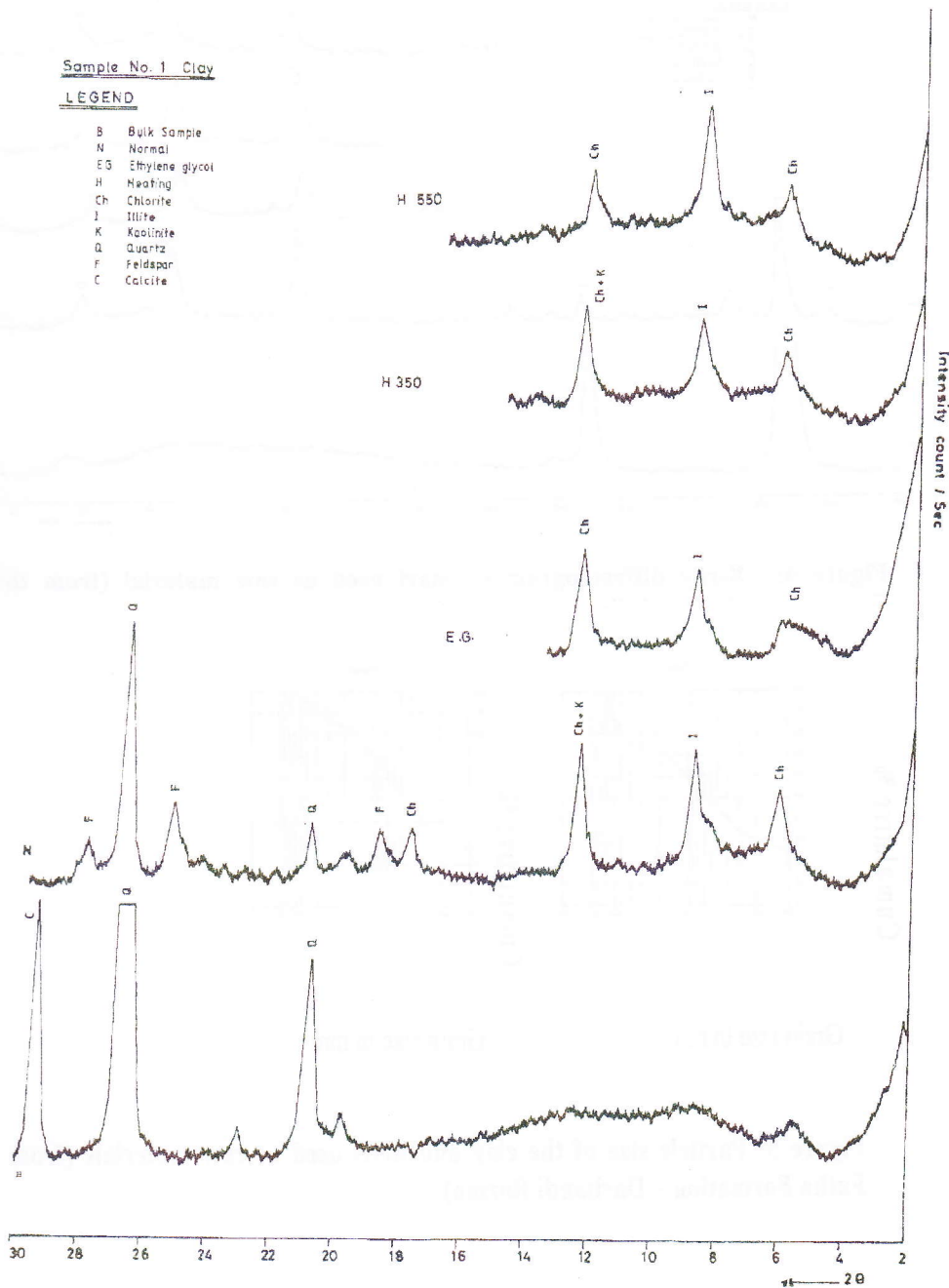


Figure 3: X-ray diffractogram of clay used as raw material (from the Fatha Formation - Darbandi Bazian).

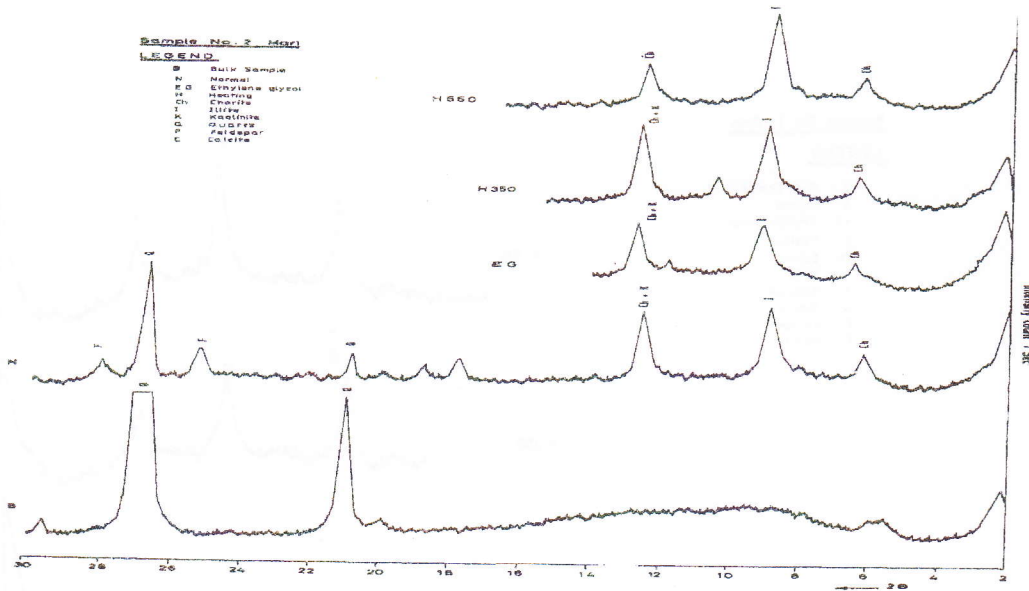


Figure 4: X-ray diffractogram of marl used as raw material (from the Fatha

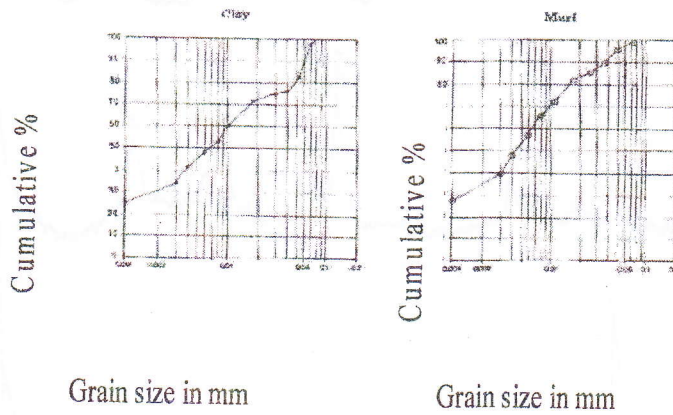


Figure 5: Particle size of the clay and marl used as raw materials (from the Fatha Formation – Darbandi Bazian).

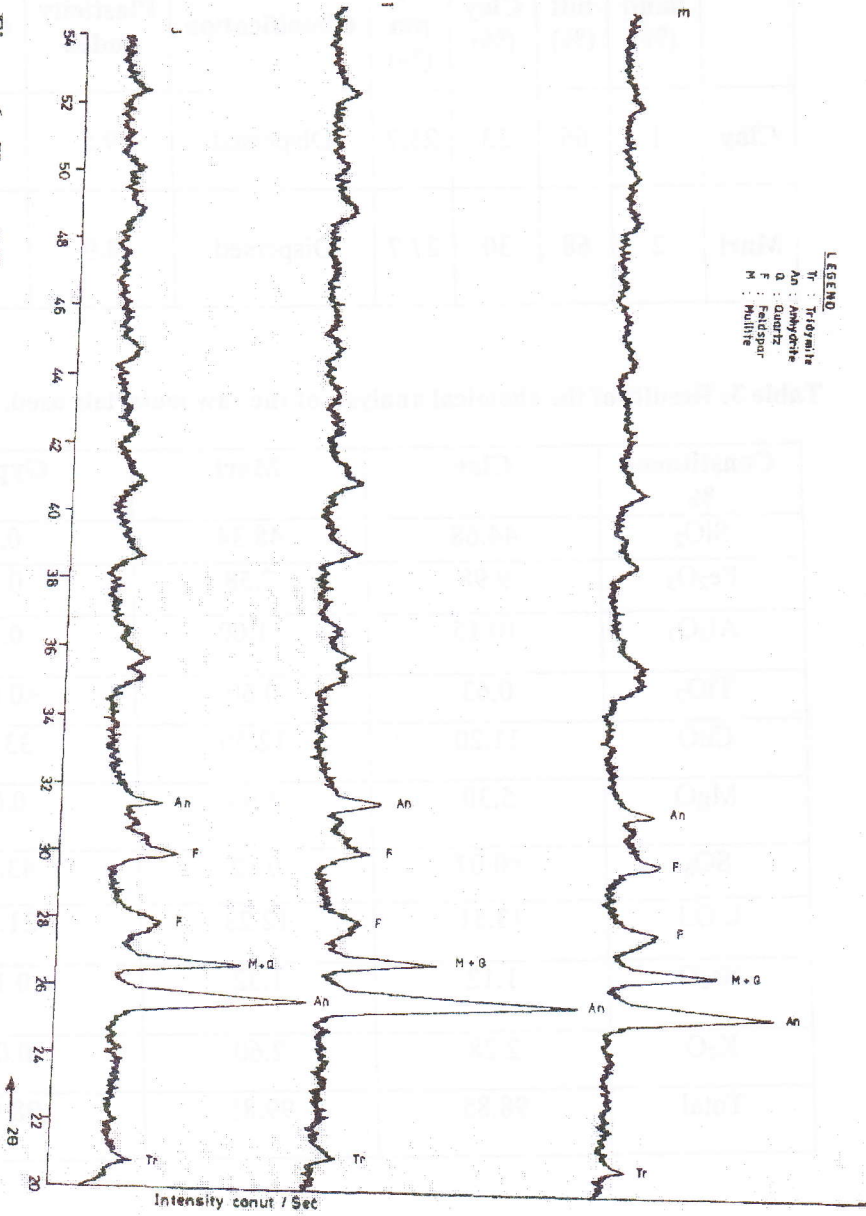
Table 2: Results of particle size analysis and plasticity of the clay and marl with their classification using Budnikov's scheme (1964).

	Particle size				Classification	Plasticity	
	Sand (%)	Silt (%)	Clay (%)	<1 $\mu\text{m}$ (%)		Plasticity index	Classification
Clay	1	66	33	25.7	Dispersed	9.5	Moderately plastic
Marl	2	68	30	27.7	Dispersed	8.9	Moderately plastic

Table 3: Results of the chemical analysis of the raw materials used.

Constituent %	Clay	Marl	Gypsum
SiO <sub>2</sub>	44.68	48.34	0.19
Fe <sub>2</sub> O <sub>3</sub>	9.98	7.58	0.04
Al <sub>2</sub> O <sub>3</sub>	10.15	11.00	0.19
TiO <sub>2</sub>	0.63	0.60	<0.006
CaO	11.20	12.50	33.04
MgO	5.30	3.65	0.08
SO <sub>3</sub>	<0.07	0.12	43.00
L.O.I	13.51	12.23	21.39
Na <sub>2</sub> O	1.12	1.32	0.12
K <sub>2</sub> O	2.28	2.60	0.05
Total	98.85	99.85	98.08

Figure 6: X-ray diffractogram of briquettes J3, I3 and E3, which are fired at 1100 °C<sup>±</sup> Darbandi Bazian.



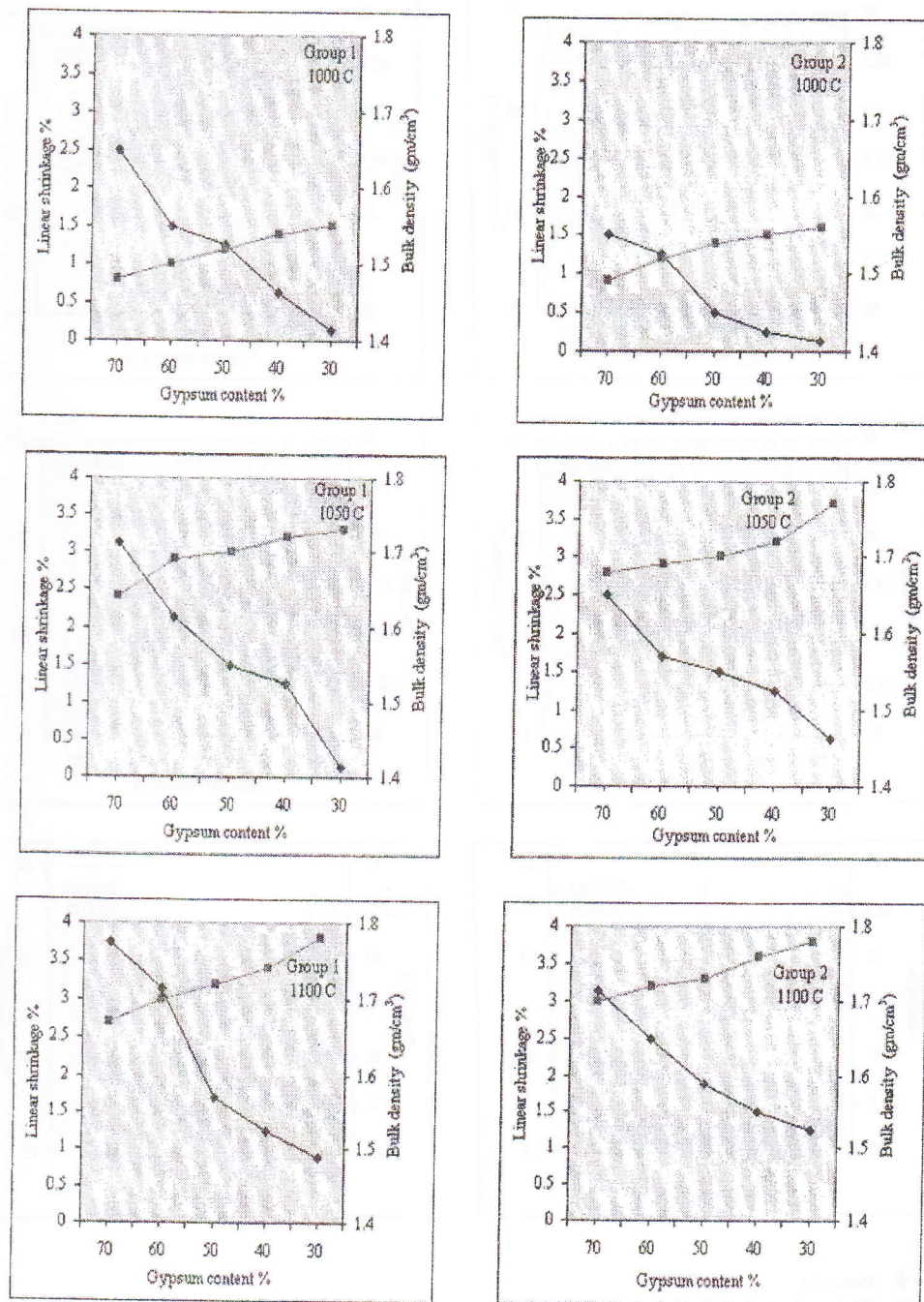
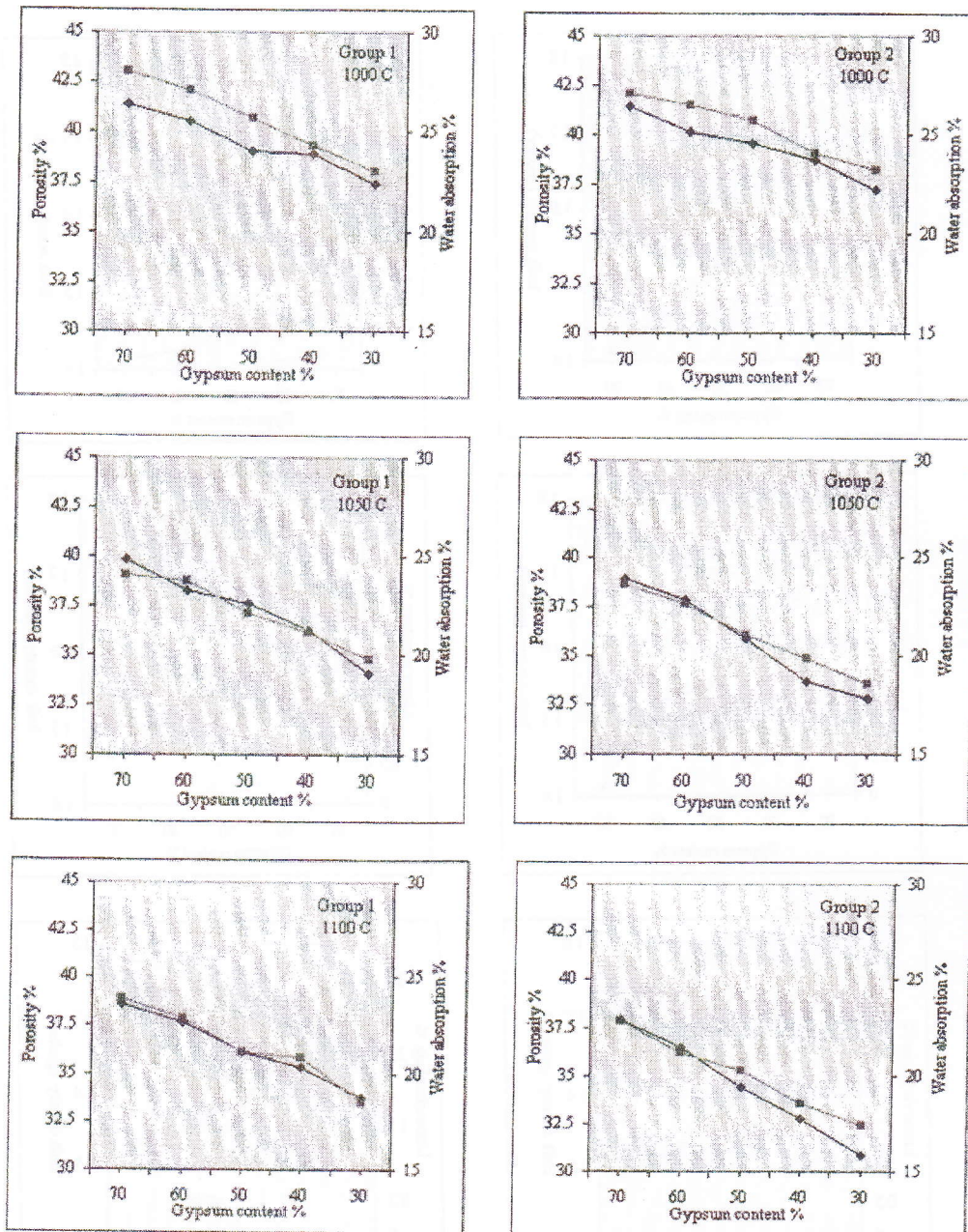


Figure 7: The relationship between linear shrinkage and bulk density vs. gypsum content for briquettes of Groups 1 and 2, which are fired at 1000, 1050 and 1100 °C.



◆ Porosity  
■ Water adsorption

Figure 8: The relationship between porosity and water adsorption vs. gypsum content for briquettes of Groups 1 and 2 and, which are fired at 1000, 1050 and 1100 °C.

**Mechanical Properties** – Compressive strength and modulus of rupture of the briquettes ranges from 100 to 438.5 kg<sup>2</sup> and from 31.5 to 76.49 kg/cm<sup>2</sup>, respectively (Table 5). The mechanical properties are inversely proportional with the content of gypsum in the mix (Fig. 9). Briquettes of Group A have higher compressive strength and modulus of rupture than Group B.

**Thermal properties** - Results of the thermal conductivity of the studied samples are presented on Table 6. The thermal conductivity represented as factor "k" ranges from 0.30 to 0.53 W/m.°C. The tested briquettes of Group A showed higher thermal conductivity values (0.41 – 0.53 W/m.°C) than the tested briquettes of Group B (0.3 – 0.38 W/m.°C). Briquette X, consisting of clay only, showed that its thermal conductivity is 0.70 to 0.71 W/m.°C.

### Discussion

The mineral phases present in the briquettes are the result of several transformations occurred during firing. Mackenzie (1972) and Kloss (1974) showed from differential thermal analysis (DTA) and thermal gravimetry (TG and DTG) that anhydrite (insoluble) is the result of heating of the gypsum and its transformation through three major stages [6,7] These results concur with the results of many workers such as Al-Nuaimi (2000) (4). The first stage is the formation of hemihydrate at 120-175 °C (endothermic reaction), with the loss of three quarters of water of crystallization of the gypsum. The second is the formation of anhydrite (soluble) at 200-210 °C (endothermic reaction), accompanied by losing all its water of crystallization. Finally, is the transformation of soluble

anhydrite to insoluble anhydrite at 350-400 °C (exothermic reaction). Tridymite and mullite resulted from crystallization of available silica and/or alumina. Two sources of silica and alumina are available: from the breakdown of the clay minerals and other silicates in the raw materials, during their heating, and from the quartz present in the clay and marl. Color of the briquettes is in general off-white, which is mostly attributed to the dominance of anhydrite. Anhydrite is a white mineral unless it contains some impurities [8-10]. The main coloring impurities present in the raw materials are iron oxides. The brown tint of briquettes of Group 1 can be attributed to the bleaching of ferric oxides by its reaction with the free calcium oxide and silica forming a silicate phase [11-13]. The change of the gray colors of Group 2 is attributed to the oxidation of the organic matter when fired in temperature range between 350 to 900 °C.

Sintering properties are dependent primarily on firing temperature [11,14,15]. Loss on ignition (L.O.I) of the three raw materials is high, being 12.23, 13.51 and 21.39 weight percent in the marl, clay and gypsum respectively. Accordingly, during firing there was considerable loss of gases and vapors, due to dehydration of gypsum, dehydroxylation of the clay mineral and the loss of carbon dioxide of the carbonate. This loss has lead to higher apparent porosity, water absorption, bulk density and linear shrinkage and with further heating the grains sinter leading to denser bodies (as shown by the increase in the bulk density). The progressive increase of gypsum in the mix produced lighter briquettes. Those briquettes fired at 1100°C have bulk density ranging from 1.67 to 1.78 g/cm<sup>3</sup> (Table 4), depending on the amount of gypsum in the mix. Al-

Hakeem (1998) showed that the average bulk density of the briquettes formed of clay or marl of the Fatha Formation, which are comparable to the common bricks, is 1.87, which is considerably heavier [3]. Sintering is believed to be in both, the liquid state (i.e. vitrification), which produced during cooling glass and other phases such as tridymite and mullite, and in the solid state giving rise to further densification [16]. Mechanical properties improve with the increase of firing temperature, which can be attributed to decrease of porosity of the briquettes, and the increase of the densification of their grains. However, the mechanical properties deteriorate with higher gypsum content in the mixes because of the higher production of properties formed by the high loss of water through dehydration of gypsum during firing. Thermal conductivity (k) of the studied samples (Table 6) is low. It is about half (mixes I and J) the conductivity of the common brick (sample X). The conductivity improves (becomes lower) with the increase of the gypsum content in the mix because the gypsum results into higher porosity during firing.

On the other hand, the mechanical properties of the briquettes deteriorate with higher gypsum content in the mixes. Therefore, there should be a balance between the thermal and the mechanical properties of the briquettes.

In addition conductivity of Group B is better than Group A since it has lower k value.

The gypsum content in the mixes of samples fell in Class A is low (30 or 40%) because gypsum content has an inverse relationship with the compressive strength of the briquettes. It is also worth noticing that the firing temperature of all samples in Class A is 1100°C. The briquettes were dipped in distilled water for one month in a beaker (distilled water was added to keep constant level).

The CaO content in the water, which is considered as a measure for the solubility of the anhydrite, was measured before and after dipping of the briquettes. There was no evidence of any solution of the briquettes. However, the anhydrite formed by firing is insoluble. Results of this test complied with those carried out by Al-Nuaimi (2000).

The fired briquettes (30 types: 10 mixes fired at three temperatures) were classified using Iraqi Standard 25-1969 for the manufacture of common brick (Table 7). It was found that six samples fall in Class A, seventeen samples fall in Class B-Rank 1, one sample fall in class B-Rank 2 and six samples fall in Class C-Rank 1.

Table 5: Results of the mechanical properties of the briquettes.

Mix	Firing Temperature (°C)					
	1000		1050		1100	
	Compressive strength (kg/cm <sup>2</sup> )	Modulus of rupture (kg/cm <sup>2</sup> )	Compressive strength (kg/cm <sup>2</sup> )	Modulus of rupture (kg/cm <sup>2</sup> )	Compressive strength (kg/cm <sup>2</sup> )	Modulus of rupture (kg/cm <sup>2</sup> )
A	100.2	34.7	100.2	35.69	115.2	41.30
B	141.4	36.0	141.4	36.10	155.6	49.64
C	155.6	50.1	169.7	51.93	169.0	59.37
D	226.3	56.4	254.6	643.87	297.0	68.87
E	325.3	64.9	325.3	69.07	438.5	76.49
F	100.2	31.5	127.3	34.47	141.4	41.20
G	127.3	32.3	141.4	40.83	169.7	45.69
H	169.7	41.8	183.9	54.19	183.9	55.10
I	198.0	55	198.0	55.23	254.6	58.58
J	311.2	55.2	339.5	59.50	367.8	74.34

Table 6: Results of the thermal conductivity tests.

Group	Mix	K (W/m °C)	K (W/m °C)
A	D	0.41	0.47
	E	0.47	0.53
B	I	0.30	0.35
	J	0.33	0.38
	X	0.70	0.71

Table 7: Classification of the briquettes according to the Iraqi Standard 25 – 1969.

Brick Class	Rank	Minimum compressive strength (kg/cm <sup>2</sup> )	Maximum water absorption (%)	Sample number
A	-	180	20	E <sub>2</sub> , E <sub>3</sub> , I <sub>2</sub> , I <sub>3</sub> , J <sub>2</sub> & J <sub>3</sub>
B	1	110	25	A <sub>3</sub> , B <sub>2</sub> , B <sub>3</sub> , C <sub>2</sub> , C <sub>3</sub> , D <sub>1</sub> , D <sub>2</sub> , D <sub>3</sub> , E <sub>1</sub> , F <sub>2</sub> , F <sub>3</sub> , G <sub>2</sub> , G <sub>3</sub> , H <sub>2</sub> , H <sub>3</sub> , I <sub>1</sub> & J <sub>1</sub>
	2	90	25	C <sub>1</sub>
C	1	90	28	A <sub>1</sub> , A <sub>2</sub> , B <sub>1</sub> , F <sub>1</sub> , G <sub>1</sub> & H <sub>1</sub>
	2	60	-	-

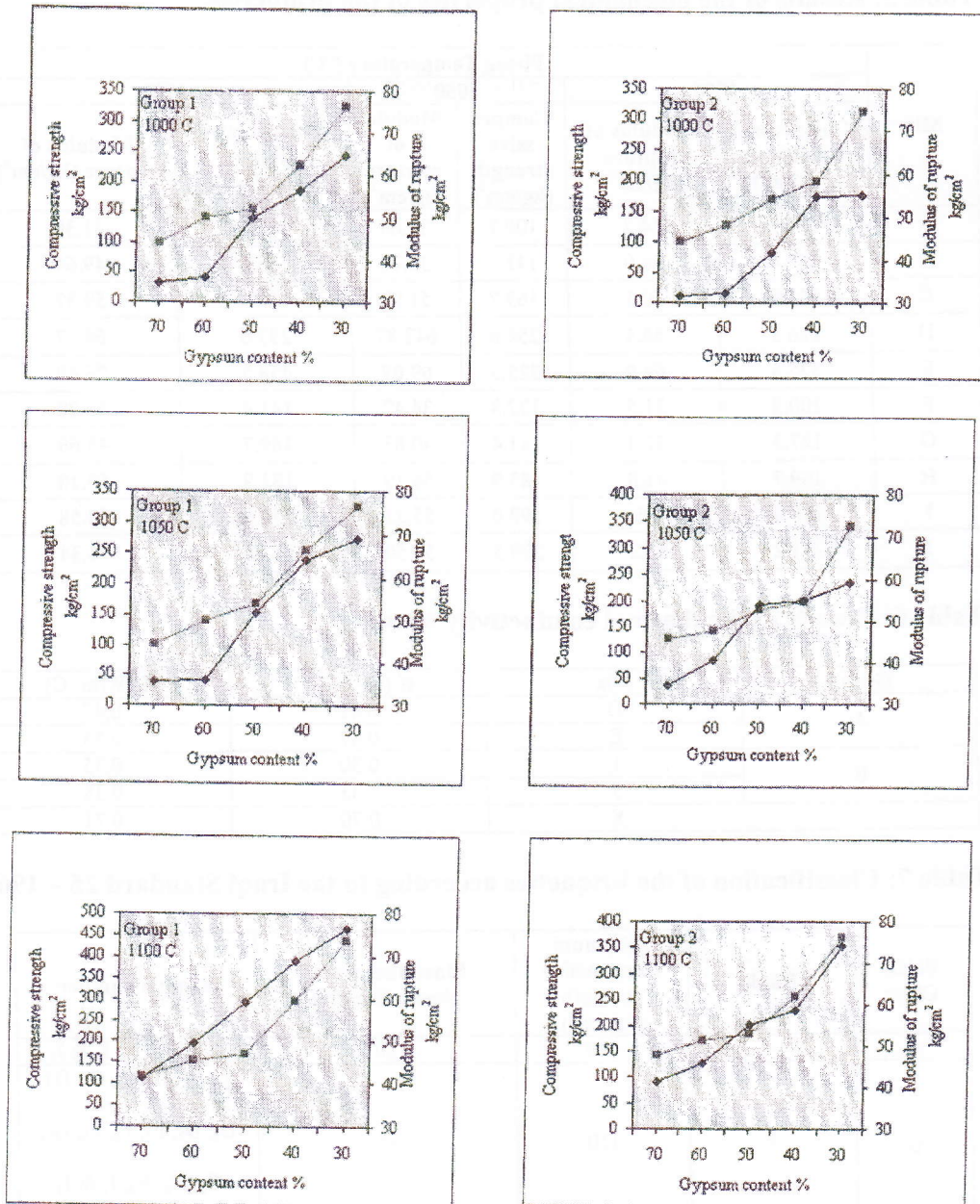


Figure 9: The relationship between compressive strength and modulus of rupture vs. gypsum content for briquettes of Groups 1 and 2, which are fired at 1000, 1050 and 1100 °C.

◆ Compressive strength    ■ Modulus of rupture

## Conclusions

Thermal insulator can be manufactured from gypsum and clay or marl of the Fatha Formation (Middle Miocene) from Darbandi Bazian- Sulaimani Governorate, and can be utilized in construction and building, especially indoors. The tested briquettes showed good thermal insulation (thermal conductivity -  $k$  is 0.30 to 0.53  $\text{w/m}^\circ\text{C}$ ), reasonable bulk density (1.48 to 1.78  $\text{g/cm}^3$ , i.e. lower than the common brick) and good compressive strength (100 – 438.5  $\text{kg/cm}^2$ ). Compressive strength of the briquettes varies inversely with thermal insulation and gypsum content in the mixes. The higher content of the plastic material (clay and marl) in the mixes produced after firing briquettes having higher compressive strength and thus can be classified stronger but their insulating abilities are reduced. In addition,

briquettes with marl, as plastic materials, are generally better thermal insulators than those with clay but they have slightly weaker briquettes (slightly lower compressive strength and modulus of rupture). The briquettes prepared accorded with the specifications of the Iraqi Standard 25-1969 for the common bricks and fell in Classes A, B and C.

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### هه‌سه‌نگاندنی به‌کارهێنانی جبسوم و خۆن له دروست کردنی نه‌گه‌یه‌نه‌ری گهرمی دا.

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سوزان نوری محمد، کۆمپانیایی گشتی پشکنین و کانزاکان، بغداد / عێراق

#### پوخته

به‌کارهێنانی گه‌چ و قوور و مارل خه‌ملێنراوه له پیکهاته‌ی هه‌تسه (مایوسینی ناوه‌ند) له دهریه‌ندی بازیان - له ناوچه‌ی سلێمانی بۆ دروستکردنی نه‌گه‌یه‌نه‌ری گهرمی. ماده‌ه‌کان کانزاییانه وه کیمیاییانه وه فیزیاییانه شیبته‌نگراوه. پاشان نه‌مانه هارپاون و وردکراون بۆ بچوکتر له ۱۲۵ مایکرومیتەر. ده بر ناماده‌گراوه له تیکه‌ته‌ی گه‌چ بۆ قوور پان مارل به‌رێژیه‌ی کێشی سه‌دی ۲۰/۷۰، ۴۰/۶۰، ۵۰/۵۰، ۶۰/۴۰ و ۷۰/۳۰. وه پاشان ۲۱۰ نمونه‌ی تاقیکردنه‌وه دروستکراوه به‌ په‌ستاوتنی شیوه‌ی وشک، وشککراونه‌ته‌وه له فرندا به‌ په‌ی گهرمی ۱۰۵ س<sup>۰</sup> وه سوتینراون تا ۱۰۰۰، ۱۰۵۰ وه ۱۱۰۰ س<sup>۰</sup> به‌ به‌رزکردنه‌وه‌ی ۱۰۰ س<sup>۰</sup> له هه‌ر کاتر مێریکدا وه کساتی خوساندنی یه‌ک کاتر مێر. دواتر نمونه‌ی سوتناوه‌کان نرخاندنی گونجاویتیان تاقیکراوه‌ته‌وه وه‌ک نه‌گه‌یه‌نه‌ری گهرمی به‌ره‌مه‌ی تاقیکردنه‌وه فیزیکی میکانیکی به‌هه‌ر گه‌یه‌یه‌کان پێشانی داوه که چونه‌وه‌یه‌کی رێک ۰،۱۳ بۆ ۲،۷۵٪، کونه‌ روونه‌کان ۳۰،۸ بۆ ۴۱،۴٪، هه‌تمزینی ناو ۱۷،۴ بۆ ۲۸،۰٪، بارسته‌چری ۱،۴۸ بۆ ۱،۷۸ گم/سم<sup>۳</sup>، هێزی په‌ستاوتن ۱۰۰،۱ بۆ ۴۲۸،۵ گم/سم<sup>۲</sup>، وه هاوکۆلکه‌ی پێچران ۲۱،۵ بۆ ۷۶،۵ گم/سم<sup>۵</sup>. له‌هه‌مانکاتدا چوار نمونه‌ی هه‌نێزێراوه بۆ پشکنینی توانای گه‌یه‌یه‌ندی گهرمی (k) وه دۆزراونه‌ته‌وه که سنووری نیوان ۰،۲ و ۰،۴ وات/م. س<sup>۰</sup>، دهریان خستوه که نه‌مانه نه‌گه‌یه‌نه‌ریکی باشن.

## تقييم استخدامات الجبس والاطيان في صناعة العوازل الحرارية.

مازن يوسف تمرأخا، قسم علوم الأرض، كلية العلوم، جامعة بغداد / العراق

سوزان نوري محمد، الشركة العامة للمسح الجيولوجي والتعدين، بغداد / العراق

### الخلاصة

تم تقييم الجبسوم والاطيان والمارل الموجودة في تكوين الفتحة ( المايوسين الاوسط ) في منطقة دربند بازيان - محافظة السليمانية لغرض استخدامها في صناعة العوازل الحرارية. تم تحليل المواد الخام مهدنيا وكيميائيا و فيزيائيا. بعد ذلك تم سحق وطحن العينات الى احجام اقل من ١٢٥ مايكرون ومن ثمة تهيئة عشرة خلطات بنسب خلط تراوحت بين ٣٠/٧٠، ٤٠/٦٠، ٥٠/٥٠، و ٦٠/٤٠ و ٧٠/٣٠ كنسب مئوية من وزن الجبسوم الى الاطيان او المارل. تم تشكيل ٢١٠ عينة فحص بالضغط شبه الجاف و تجفيفها بالفرن تحت ١٥٥ درجة مئوية ومن ثم حرقها بالفرن تحت ١٠٥٠، ١٠٠٠، ١١٠٠ درجة مئوية مع رفع درجة حرارتها بمعدل ١٠٠ درجة مئوية / ساعة في زمن انضاج قدره ساعة واحدة. ومن ثم فحصت العينات المحروقة لغرض تقييمها كعوازل حرارية. وقد أظهرت نتائج الفحوصات الميكانيكية-الفيزيائية أن الانكماش الخطي تراوح بين ٠,١٣ الى ٢,٧٥٪ و المسامية الظاهرية بين ٣٠,٨ و ٤١,٤٪، وامتصاص الماء بين ١٧,٤ و ٢٨,٠٪ و الكثافة الكلية تراوحت بين ١,٤٨ و ١,٧٨ غرام/سم<sup>٣</sup> و المقاومة الإنضغاطية بين ١٠٠,١ و ٤٣٨,٥ كغم/سم<sup>٢</sup> و معامل الكسر بين ٣١,٥ الى ٧٦,٥ كغم/سم<sup>٢</sup>. اما العينات الاربعة التي تم اختبارها لدراسة الموصلية الحرارية (k) فقد وجد ان قيمتها تتراوح بين ٠,٤ الى ٠,٤ واط/م لكل درجة مئوية مؤكدة كونها جيدة لاغراض العزل الحراري.

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