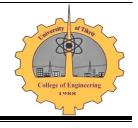


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# Properties of Keene's Cement-Quick Set Locally Product

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#### Abstract

Gypsum has been used as building material over a very long period of time. The production of gypsum plaster in Iraq confined on three types; plaster of Paris, ordinary gypsum and mechanical gypsum. The purpose of this study is to present a proposal to produce another type of gypsum plaster called Keene's cement, it has improvement characterizes than the other types of gypsum. In this study different percentage of alum solution has been used to soaked in gypsum lumps and different recalcinating temperatures also have been used. The results show that 12% of alum and 250 C° recalcinating temperature gave the highest result of compressive strength of Keene's cement product.

Keywords: Gypsum, Keene's cement, Alum.

الخلاصة

استخدم الجص كمادة بنائية منذ فترة طويلة من الزمن، وان انتاج الجص في العراق انحصر في ثلاثة انواع، وهي مسحوق باريس، الجص الاعتيادي (الفني) والجص الميكانيكي. الغاية من هذه الدراسة هو تقديم مقترح لانتاج نوع اخر من الجص يسمى سمنت كين، الذي يمتلك خواص محسنة اكثر من الانواع الاخرى للجص. في هذه الدراسة، استخدمت نسب مختلفة من الشب لتغطيس كتل الجبس فيها، وكذلك استخدمت درجات كلسنة مختلفة. النتائج اظهرت بان 12% من الشب و 250 م° من درجة الكلسنة اعطت اعلى نتيجة لمقاومة انضغاط سمنت كين المنتج.

الكلمات الدالة: الجص، سمنت كين، الشب.

#### Introduction

Gypsum is a useful industrial material found abundantly on the earth's crust. It is inexpensive to mine and process and its calcined products have a wide range of readily controlable properties such as strength, density and setting time [1]. Gypsum belongs to a group that consists of three different forms of hydration. First is to gypsum with two water molecules (CaCO<sub>4</sub>.2H<sub>2</sub>O). Second is hemihydrate (plaster of Paris) with 1/2 molecule of water (CaCO<sub>4</sub>.<sup>1</sup>/<sub>2</sub>H<sub>2</sub>O). Lastly there is anhydrite (insoluble calcium sulfate) with no water of hydration (CaCO<sub>4</sub>). The significance of different amounts of hydration allowed for transformation of gypsum to plaster of Paris, and its' hydration back to gypsum. The

availability and ease of conversion makes gypsum a wonderful building material [2]. CaSO<sub>4</sub>·2H<sub>2</sub>O + heat (170C°) ...... CaSO<sub>4</sub>· $\frac{1}{2}$ H<sub>2</sub>O + 1 $\frac{1}{2}$ H<sub>2</sub>O.

Gypsum plasters such as plaster of Paris, ordinary gypsum plaster and mechanical gypsum plasters have adequate properties in dry interior conditions but are unsuitable for exterior application because of water erosion. Additionally the product has relatively low tensile, flexural and compressive strength.

These three types of gypsum plasters are produced in Iraq in large quantities and have different uses. While there are many types of gypsum plasters are not produced in Iraq such as Keene's cement, Parian cement, Martain's cement, Sirapite cement, Selenetic cement and others, these types of gypsum plasters have good properties and better than plaster of Paris.

Keene's cement can be classified according to its setting time to industrial Keene's cement and industrial Keene's cement-quick set [3].

If gypsum is subjected to a temperature of  $(400 \text{ C}^\circ)$ , it completely dehydrated. When this material is ground and has had a positive catalyst such as alum added to it, it is known as Keene's cement. If this product is mixed with water to form a plaster, it sets and becomes very hard. It is highly resistant to moisture penetration and is used where sanitary conditions or excessive moisture makes it necessary to specify a hard, impervious, smooth surface. About (1 ton) of Keene's cement, mixed with water and applied as a putty coat, will cover 335 to 420 square meter of all surface, with (0.5–1)cm. thick [4].

Iraqi pure gypsum is characterized by its high strength, low density, white finished color and low setting time. Many efforts were made to improve properties of Iraqi gypsum such as [5,6,7] by adding fibers, limestone powder, clinker powder which increase setting time five minutes and indicate little improvement in compressive strength.

A fine grade of Keene's cement can be produced by burning gypsum at a temperature of at least 650 C°, mixing it with a natural catalyst such as one per cent of  $K_2SO_4$  and grinding the mixture to pass about a 150 mesh screen [8].

A quick-setting grade of Keene's cement can be formed from finely divided calcined gypsum together with a catalyst comprising  $K_2SO_4$  or other salt containing an amount of acid in excess of that necessary for the formation of an acid salt. From 2 to10 % of the product may be added to plaster of Paris to obtain a mixture suitable for use as a surgical cement [9].

#### Aim of the Research

This research was carried out to produce special type of gypsum plaster (Keene's cement-quick set) using different mixing percentages and different calcination temperatures, the second objective was to determine setting time and compressive strength of the produced gypsum (Keene's cement).

## **Experimental Program**

Information was collected about quarries of gypsum rocks which are found in Mosul city, Iraq to investigate the properties of these rocks to found the most purity gypsum rocks, Batnaya quarry as shown in Figure (1) was selected.

Batnaya gypsum quarries: It is laying in the north of Mosul city, Iraq with approximately 20 Km. Generally, it is compound of two gypsum layers containing between them, a layer of green grey marl. Its total thickness about 12 m [10].

#### Raw Material Used Gypsum Rocks

Gypsum rocks from Batnaya quarry was brought, fifteen samples from different depths were taken and tested to find their purity, Table (1) show chemical analysis of all samples taken.

# <u>Alum</u>

It is sulphates of aluminum with chemical formula [AL<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.6H<sub>2</sub>O], is known as alum, it was a combination of an alkali metal, such as sodium, potassium, or ammonium and a trivalent metal, such as aluminum, iron, or chromium. The most common form, potassium aluminum sulfate, or potash alum, is one form that has been used in food processing. Another, sodium aluminum sulfate, is an ingredient in commercially produced baking powder [11], Table (2) show alum properties which is used in this study.

## <u>Lime</u>

Hydrated lime was used as 10% of gypsum weight. Gypsum plaster sets or dries quickly, lime is slow to set so a combination of lime and gypsum plasters sets a medium speed. While setting, gypsum plaster expands slightly and lime contracts slightly. Physical properties of lime shown in Table (3).

## <u>Water</u>

Ordinary potable water was used in this study for mixing purposes.



Fig. 1. Map of Iraq showing the location of Batnaya quarry

Samples	(CaO)%	(SO₃)%	(H₂O)%	(I.R)%**	(CaSO <sub>4</sub> .2H <sub>2</sub> O)%
1	32.07	45.28	20.4	2.25	97.75
2	31.61	46.10	19.4	2.89	97.11
3	31.84	45.79	19.81	2.56	97.44
4	32.30	45.43	19.63	2.64	97.36
5	32.41	45.37	20.2	2.02	97.98
6	32.46	45.11	19.93	2.50	97.50
7	32.15	45.24	20.18	2.43	98.13
8	32.01	45.52	20.6	1.87	98.13
9	32.16	45.78	20.55	1.51	98.49
10	32.31	45.09	20.8	1.80	98.20
11	32.19	45.57	20.3	1.94	98.06
12	32.02	45.83	20.00	2.15	97.85
13	32.38	45.88	20.15	1.59	98.41
14	32.20	45.52	19.79	2.31	97.69
15	31.81	46.09	20.72	1.38	98.62
Average	32.12	45.57	20.16	2.15	97.85

Table 1. Chemical analysis of Batnaya gypsum rock samples\*

\*Test conducted in Geology department/Mosul university, \*\*I.R: Insoluble Residual

Table 2. Alum properties used in this study from the Mishraq company

Compounds by weight	Alum properties	Iraqi standard specification 1640 for alum <sup>[12]</sup>
Al <sub>2</sub> O <sub>3</sub>	17.8%	16.5% as min
Fe <sub>2</sub> O <sub>3</sub>	0.009%	0.01% as max
Insoluble material	0.39%	0.5% as max
PH (1gm/100ml)	3.8	3 as min

Tests	Properties	Hydrated lime	Iraqi standard specification [13]		
1	fineness	7%	The retaining on sieve (90µm) not more than10%		
2	Initial setting time	1.75 hr			
3	Hydrated time	10 min.	5 min. – 15 min.		

Table 3. Physical properties of hydrated lime used

#### **Manufacturing Process**

Steps below and Figures (2) and (3) used to show the processes used to manufacture Keene's cement-quick set.

- 1. Washing the gypsum rocks.
- 2. Crushing the gypsum rocks to small lumps.
- 3. Sieve analysis of small lumps (passing sieve 22.4mm, retaining on sieve 9.5mm) in order to obtain a uniform calcination of all lumps in the same time.
- 4. Calcination of crushed gypsum rocks (small lumps) by 170 C° [14], for 2 hours.
- Soaking the calcined gypsum rocks (small lumps) in alum solution (2, 4, 6, 8, 10, 12, 24, 36, 48, 60)% as shown in Figure (2), for

24 hrs, the purpose from this process is to enter the aluminum element through the structure of gypsum.

- Drying gypsum lumps in an oven by105 C° for 24 hrs, to evaporate of water at less time enable and boiling lumps in alum solution.
- Recalcining of dried gypsum rocks (small lumps) in a furnace by (200, 250, 300, 350, 400) C° as shown in Figure (3), for 2hrs, to dry off more than 1.5 water part presence in gypsum structure.
- Grinding this lumps to powder by (100% passing from 425µm and 85% passed from 150µm sieve).

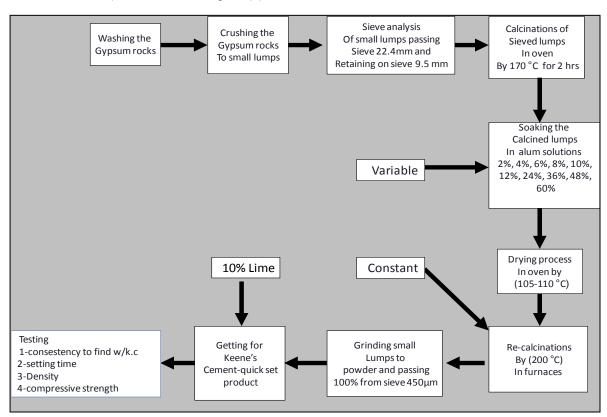


Fig. 2. Step 1 (Flow chart 1); During this step, many of alum solution percents are used (2%, 4%, 6%, 8%, 10%, 12%, 24%, 36%, 48%, 60%) to find the optimum percentage of alum solution

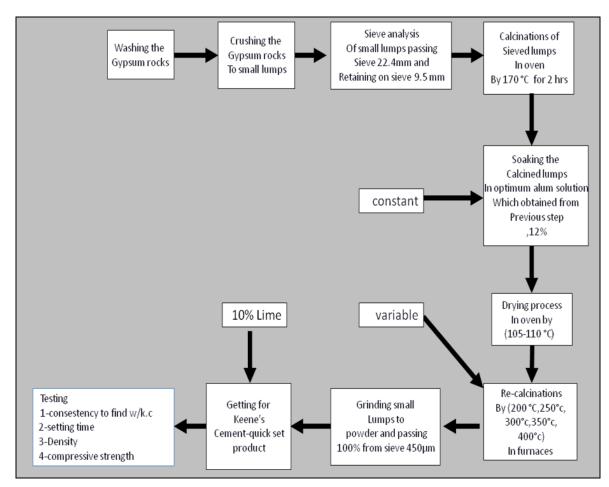


Fig. 3. Step 2 (Flow chart 2); After optimum alum solution percent found from step one, the step two used to find the optimum recalcining temperature. (200 C°, 250 C°, 300 C°, 350 C°, 400 C°) recalcining temperatures are used

## Tests Conducted Normal Consistency

Normal consistency test was determined for Keene's cement-quick set according to ASTM C187 [15]. The results shown in Table (4).

#### Setting Time

This test was done for Keene's cement according to ASTM C 472/99 <sup>[16]</sup>. The results shown in table (4).

#### Density

This test used to determine the density of Keene's cement-quick set according to ASTM C 472/99 [16]. The results shown in Table (4).

#### **Compressive Strength**

The compressive strength test was determined according to ASTM C 472/99 [16], 50 mm cubes were tested using compression testing machine at loading rate 15 Mpa per minute (0.25 Mpa per second). The average of three cubes was adopted.

Mix No.	Recalcining Temperature (constant)	Alum Solution (variabl)	Consistency Water/Keene's	Setting time (min.)		Density (gm/cm <sup>3</sup> )	Compressive Strength at 7-
	(C°)	(%)	Cement ratio	initial	final	(gin/oin )	days (MPa)
1	200	2	0.50	6	10	1.65	11.0
2	200	4	0.48	4:30	9:30	1.60	11.9
3	200	6	0.47	4:30	9:30	1.65	12.3
4	200	8	0.47	5	8:30	1.70	12.7
5	200	10	0.45	5	10	1.75	13.7
6	200	12	0.40	5	10	1.50	14.2
7	200	24	0.47	4:30	8	1.45	08.0
8	200	36	0.5	4:30	9	1.40	03.2
9	200	48	0.5	4:30	10	1.31	03.0
10	200	60	0.5	4:30	9:30	1.25	02.0

**Table 4.** The results obtained from the step 1 (flow chart 1) of manufacturing processes to find optimum alum solution

# Results and Discussion *Gypsum Rocks*

After field investigations for gypsum quarries in Mosul city/Iraq and its surrounding, bringing samples from these quarries, testing the purity of these samples, the Batnaya quarry selected because it contain a high purity gypsum rocks. Table (1) show the results of purity tests of fifteen samples taken from Batnaya quarry, if we are take the average of fifteen samples (CaO+SO<sub>3</sub>+H<sub>2</sub>O), (32.12+45.57+20.62) respectively, we find that the purity is (CaSO<sub>4</sub>.2H<sub>2</sub>O = 97.85%) and the impurities represent (2.15%) only.

#### Setting Time

United States Gypsum Company [3], indicated that the industrial Keene's cementquick set have setting time 10 - 20 minute, so we can classify the result of this study to the type mentioned above.

#### **Compressive Strength**

The strengths of the two steps used to manufacture Keene's cement were shown in Tables (4) and (5). Referring to Table (4), 12% of alum shows the highest (7 days) compressive strength (14.2 MPa), so this percentage was chooses to the second step. Table (5) shows the variation of strength with different calcinating temperatures. Return to the Table (5), (250 C°) temperature of calcinations gave (25 MPa) compressive strength, which is the highest result.

ASTM [17], pointed that (17 MPa) was the minimum strength accept for gypsum Keene's cement, so the result obtained from this work was confirmed with ASTM.

Table (6) shows the gypsum plaster properties according to Iraqi standard specification [18], and the properties of gypsum Keene's cement-quick set produced in this study to compare between them.

Mix	Recalcining Temperature	Alum Solution	Consistency Water/Keene	Setting time (min.)		Density	Compressive Strength at
No.	(variable) (C°)	(%)	s Cement ratio	initial	final	(gm/cm³)	7-days (MPa)
11	200	12	0.40	5	10	1.5	14.2
12	250	12	0.5	10	20	1.48	25
13	300	12	0.5	8:30	20	1.35	21
14	350	12	0.5	9	20	1.35	21.4
15	400	12	0.5	9	20	1.36	23

**Table 5.** The results obtained from the step 2 (flow chart 2) of manufacturing process to find optimum recalcination temperature

No.	Properties	Ordinary gypsum	Plaster of Paris	Mechanical gypsum	Keene's cement produced
1	Fineness (%): The retaining on sieve No.16, not more than	8	0	5	0
2	Setting time (minutes): Not less than Not more than	8 25	8 25	12 20	10 20
3	Compressive strength (MPa): Not less than Obtained	3	5	6	- 25
4	Flexural strength (MPa): Not less than	-	1.5	2	-

**Table 6.** Physical properties of gypsum plasters as Iraqi standard specification <sup>[18]</sup> and Keene's cement produced

# Conclusions

Depending on the results of this investigation, the following conclusions can be drawn:-

- 1- Gypsum Keene's cement plaster can be locally product by using pure gypsum rocks available in north of Iraq.
- 2- 12% of alum solution used to soak in calcinating rocks, gave the highest result of the strength.
- 3- 250 C° degree of calcinations for 2 hours show the best result of compressive strength (25 MPa) and setting time (10 – 20) minutes.
- 4- Gypsum Keene's cement product in this work confirm the ASTM and USGC <sup>[3]</sup> specification type quict set, and gave improvement properties than Iraqi gypsum plasters.

## Recommendations

- 1-There are other solutions such as cream of tartar, petroleum and others can be used instead of alum to soaking gypsum rocks on it.
- 2-Using other recalcination (burning) temperatures more than 400C° such as 500C°, 600C°, 700C°, 800C° and 900C°.
- 3-Adding gypsum retarders to increase setting time but not affect on strength.
- 4-Study dry and wet compressive, flexural and tensile strengths at ages 3, 7, 14, 28 days

and absorption capacity of Keene's cementquick set.

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