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EFFECT OF CEMENT, LIMESTONE AND HYDRATED LIME ON SETTING TIME AND COMPRESSIVE STRENGTH OF LOCAL GYPSUM

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Abstract: This paper consists of studying the effect of adding different ratios of cement, limestone powder and hydrated lime on setting time and compressive strength of gypsum which available in local market which does not conform to Iraqi Standard Specification (I.Q.S) No.28/1988. The adding materials are low-cost and available in local market and can be used to improve setting time and compressive strength for gypsum which are considered as the most important properties if it used as mortar or in finishing. The adding ratios of cement were (10, 20, 30, 40 and 50) % from gypsum weight, the ratio of (50%) was the best ratio as regarded to setting time and compressive strength. They are (10.33 minutes), (8.04 N/mm^2) respectively. For the limestone powder addition, the ratios were (10, 20, 30, 40, 50 and 60) % from gypsum weight. The results showed that, the ratio of (10%) was the best in compressive strength (6.2 N/mm^2) and ratio of (60%) was the best in setting time. At adding hydrated lime, the ratio of addition (2.5%) gave compressive strength (7.51 N/mm^2) and the ratio of (12.5%) gave setting time (14.15 min.).

Keywords: *gypsum, compressive strength, setting time, cement, limestone, hydrated lime.*

تأثير السمنت، الحجر الجيري والنورة المطفأة على زمن التماسك ومقاومة الانضغاط للجص المحلي

الخلاصة: يتضمن هذا البحث دراسة تأثير اضافة نسب مختلفة من السمنت والحجر الجيري والنورة المطفأة على زمن تماسك و مقاومة انضغاط الجص المتوفر في السوق المحلي لكونه غير مطابق لمتطلبات المواصفة القياسية العراقية (م.ق.ع) رقم (28) لسنة (1988). المواد المضافة ذات كلفة واطئة ومتوفرة في السوق المحلية ويمكن ان تحسن زمن التماسك ومقاومة الانضغاط للجص والتي تعتبر اهم خاصيتين اذا استخدم الجص كمونة او للانهاء. كانت نسب اضافة السمنت هي (10، 20، 30، 40، 50)% من وزن الجص وكانت النسبة 50% هي افضل نسبة من حيث زمن التماسك ومقاومة الانضغاط وهي (10.33 دقيقة) و(8.04 نت/ملم^2) على التوالي. اما الحجر الجيري اضيف بنسب (10، 20، 30، 40، 50، 60)% من وزن الجص واظهرت النتائج ان افضل نسبة (10%) عند فحص مقاومة الانضغاط (6.2 نت/ملم^2) وافضل نسبة (60%) عند فحص زمن التماسك (10 دقيقة). وعند اضافة النورة المطفاة كانت نسبة اضافة (2.5%) من وزن الجص اظهرت نتيجة مقاومة انضغاط (7.51 نت/ملم^2) والنسبة (12.5%) من وزن الجص اظهرت نتيجة زمن التماسك (14.15 دقيقة).

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1. Introduction

Gypsum is sulphate calcium which contains about half molecular of water ($CaSO_4.H_2O$), and it was made from watery aqueous calcium sulphate ($CaSO_4.2H_2O$) when it was burned to about 170 °c and if the temperature rises more than 205 °c, the sulphate will loss all crystallize water and turn to unwater gypsum $CaSO_4$. When gypsum material mixed with water, the unwater gypsum or semi-hydrated gypsum turn to hydrate calcium sulphate [1].

The gypsum production method in Iraq is primary and old where it made by burning gypsum in special (kora) built from solid stones. These ways do not include any control on burning temperature. If the temperature is less than the required, the burning will be incomplete then the resulting gypsum is non effective. If the temperature is high, the gypsum will lose all its water and lose capability of fast setting and also lose its usefulness as a building material [2].

Al-Baghdadi (2010) [3] improved mechanical properties of gypsum mortar by adding two types of plants fibers; reed and a coconuts skin fibers to several number of gypsum mortar. She found that improvements in mechanical properties for gypsum mortar reinforced by these fibers with increasing ratios. The best result of compressive strength was when she used 3% of reeds fibers with length of 40 mm and 4% of coconuts fibers with length of 30 mm.

Raof et al. (2012) [4] studied the effect of polyvinyl acetate, furfural, fumed silica at different rate of addition and two types of fibers (carbon fiber and polypropylene fiber) to the plaster of Paris. The results show that the mixture with 2% polyvinyl acetate and 0.5% carbon fiber is the best one because the flexural strength for gypsum beams increasing about 62.92%. Ali et al. (2015) [5] used waste material like polyethylene terephthalate, sawdust and rubber with plaster of paris. They found that the best additive ratio to produce gypsum board with best physical, mechanical and thermal insulation properties is 5 and 7% of polyethylene terephthalate.

Tesárek et al. (2007) [6] modified gypsum by two different types of secondary raw materials, such as fly ash and milled gypsum. The results show that both compressive strength and bending strength of the gypsum with fly ash and milled gypsum were significantly lower than for the reference mix without fly ash and milled gypsum. One of their explanations of this decrease in strength values is a lower effectiveness of the gypsum hydration process in relation the formation of firm crystalline structure or a bad contact between the binder and the fly ash or milled gypsum. Also, may be because they used lower amount of gypsum, thus higher water/gypsum ratio in the material with fly ash or milled gypsum can be another reason.

Hatim et al. (2007) [7] added gum powder, calcium oxide and ferric oxide to Iraqi plaster. They added these materials as additives with different ratios. They found that gum powder at ratio 0.5% increases setting time from 10.5 min. to 13.2 min. and calcium oxide at ratio of 0.75% increases setting time from 10.5 min. to 16.3 min., but the addition of ferric oxide was not affect the setting time. Also, the results showed that compressive strength decreases from74.47 kg/cm^2 to 70.68 kg/cm^2 at 0.5% of gum powder addition and compressive strength increases to 78.2 kg/cm^2 at 0.75% of calcium oxide addition. So, no important results were obtained when they added ferric oxides.

Al-Ubaidi (2004) [8] used grinded stone, calcium carbonate, hydrated lime, white kaolin, natural sugar, remnants sugar factory (al-mollas) and eggs peel as a natural additives. She used water reducing-retarder as chemical admixture. The results show that the best value for setting time was 47 min. when she used 2.5% from al-mollas. She obtained 28 min. when she used 15% of (hydrated lime +white kaolin). Also, she got 27 min. when she used 15% hydrated lime. The best results of compressive strength gained when she used 2.5% hydrated lime. It increases compressive strength from 10.07 N/mm^2 to 17.7 N/mm^2. Padevet et al. (2011) [9] concluded that the most values of strengths were not changing after 14 days and were the strengths after 28 days. Also, he concluded that compressive strength of mature gypsum is about 2.5 times higher than the strength in bending.

Mohammed (2010) [10] studied effect of elevated temperature and exposure periods on the compressive strength of ordinary gypsum and plaster of Paris. He observed that as temperature increases, the compressive strength decreases for all periods of exposure. He also concluded that when temperature increases to 100°c that leads to slight increase in compressive strength of ordinary gypsum by about 6% as compared with reference value. At 200°c and above, all compressive strength of specimens decreases with the increasing in temperature and period of exposure. No residual strength observed at 1 hour exposure at 900°c. The objective of the present work is to investigate the effects of adding different ratios of cement, limestone and hydrated lime on setting time and compressive strength of local gypsum.

2. Materials

The descriptions of the materials used is shown in the following sections:

2.1. Local Gypsum

The ordinary gypsum was used from Al-Anbar province. The results of chemical analysis and physical properties are shown in "Table 1". As shown in Table 1, the ordinary gypsum used in this study was not conforming to the Iraqi standard specification (I.Q.S) No.28/1988 [11]. The tests were made at the National Center for Construction Laboratories and research.

Table 1. Chemical and physical properties of gypsum*

Chemical Properties		
Composition	Results%	I.Q.S No.28/1988 [11]
SO_3	36.61	Not less than 40%
CaO	26.55	Not less than 26.7%
MgO	0.05	Not more than 0.25%
H_2O	2.32	Not more than 9%
Loss of Ignition at 230°c	3.08	Not more than 9%
Physical properties		
Test type	Results	I.Q.S No.28/1988 [11]
Fineness	8%	Remained on sieve No.16 Not less than 8%
Setting time	5 min.	8-25 min.
Compressive strength	2.3 N/mm^2	Not less than 3 N/mm^2
Mark	Bulk	

* All tests were made at the National Center for Construction Laboratories and research.

2.2. Cement

Ordinary Portland cement (type I Tasluja-Bazian) which is produced in Iraq by the United Cement Company (UCC) was used in all test specimens. The chemical analysis and physical test results of the cement are given in "Table 2". They conform to the Iraqi specification No. 5/1984**[**12**]**.

Table 2. Chemical and physical properties of cement*

Chemical Properties		
Oxides	Results %	I.Q.S No.5/1985 [12]
CaO	61.19	-
SiO_2	21.44	-
Al_2O_3	4.51	-
Fe_2O_3	3.68	-
MgO	2.31	Maximum 5.0
SO_3	2.7	Maximum 2.8
L.O.I	2.39	Maximum 4.0
I.R	1.18	Maximum 1.5
L.S.F	0.87	0.66-1.02
C_3A	6.06	-
C_3S	42.85	-
C_2S	29.4	-
C_4AF	1.18	-
Physical properties	Results	I.Q.S No.5/1985 [12]
Fineness cm^2/gm (by blaine)	4050	Minimum 2300
Initial setting time (min.)	135	Minimum 45
Final setting time (hr)	3:25	Maximum 10
Compressive strength		
cube (70 mm) (MPa) 3 days	24.4	Minimum 15
7 days	32.3	Minimum 23
28 day	47.2	

*All tests were made at the National Center for Construction Laboratories and research.

2.3. Limestone Powder

Limestone powder from local market is used and the fineness of the grading material is high (9800) cm^2/gm. This powder completely passes the sieve size 0.125 mm. The chemical composition of this limestone is shown in "Table 3".

Table 3. Chemical analysis of limestone powder*

Oxides	Results %
CaO	83.07
SiO_2	1.18
Al_2O_3	0.6
MgO	0.55
SO_3	0.5
L.O.I	12.2

*All tests were made at the National Center for Construction Laboratories and research.

2.4. Hydrated Lime

The hydrated lime from Karbala Company was used. The chemical and physical properties are shown in "Table 4".

Table 4. Chemical and physical properties of hydrated lime*

Chemical Properties		
Chemical compound	Results %	I.Q.S No.807/1988 [13]
CaO+MgO	87.99	Minimum 85
MgO	0.8	Maximum 5
SiO_2, Al_2O_3, Fe_2O_3	4	Maximum 5
IR	0.81	-
CO_2	1.89	Maximum 5
Physical properties	Results	I.Q.S No.807/1988
Fineness remaining on sieve 90 microne	4%	Maximum 10%
Time of hydration (min.)	11	5-15
Temperature of hydration (°c)	75	Minimum 70

*All tests were made at the National Center for Construction Laboratories and research.

3. Mix Design

The ordinary gypsum was mixed with different ratios of ordinary cement (10, 20, 30, 40, and 50) % as a weight ratio from gypsum. The water/gypsum ratio was determined from consistency test. Cement was added by substitution a part of total gypsum weight. Also, limestone powder was added in different ratio in the same way as in the addition of cement. The hydrated lime was added in different ratios (2.5, 7.5, and 12.5) %, but the consistency test was made to get the water/gypsum ratio. Several mixes were done to get optimum water/gypsum ratio.

4. Preparing of Specimens

The amount of water demandable was measured and put in clean pan. The dry materials were weighted and mixed together. This mixture was strewed by a clean spoon on the water surface in one minute. Then the paste was left to saturate by water for 1 minute. The paste was mixed slowly by using a spatula. After repairing the mixture, the clean and oiled cubic molds (50 mm) were filled immediately by paste in two layers. The molds were uplifted about 1 cm from their ends and demotes to its place, this process was done five times to get rid of any air bubbles. The molds were filled during a period does not exceed 10 minutes from the adding of dry mix materials to the water. Then the upper surface was modified and skimmed as shown in figures (1) and (2).

All specimens were demolded after the setting took place and they were exposed to laboratory environment under polyethylene cover up to the age of 28 days. After that all specimens dried in oven at 45 °c for 3 days before the test till the weight be constant.

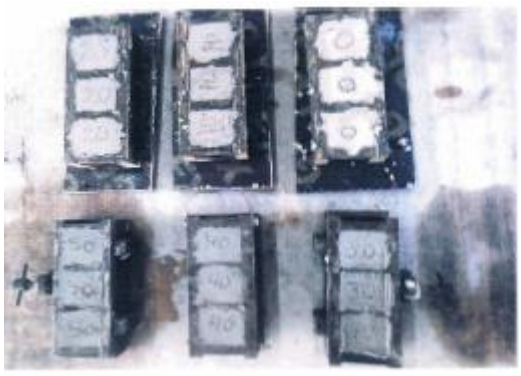

Figure 1. Specimens with cement replacement and reference mix in molds

Figure 2. Specimens with cement replacement ratios and reference mix after demolding.

5. Tests of Specimens

The following tests were done according to I.Q.S. No. 27/1988 [14].

5.1 Consistency Test

This test is carried out to find the percentage of water to be added to the gypsum as a standard consistency. 100 gm minimum of gypsum was added to determine the consistency of the mix. The gypsum dispersed with a known amount of water about 40-50 cm^3 in a mix container for 15 seconds till the water saturated with gypsum. Then it was left for 30 seconds with stirring slowly to get rid of the air bubbles. The mixture was mixed by a clean knife and placed into a mold of 51 mm in height and with inner diameter of 35 mm. this mold was filled completely with paste. The mold was uplifted from the square glass after 60 seconds from adding the material to the water and allow to the paste to separate.

After that the minimum and maximum diameter resulted from material were measured. This process was repeated by changing the amount of water until obtaining the standard spread of to 100±3 mm. Calculate water/gypsum ratio as shown in "equation (1)".

$$\text{Water/gypsum} = \frac{\text{Water } cm^3}{100} * 100 \quad \% \qquad (1)$$

5.2 Setting Time Test

Vicat apparatus was used to determine the setting time by using needle of 1 mm "Figure 3". in this test the water/gypsum ratio of standard percentage was prepared according to consistency test. 200 gm of gypsum have been weighted to get a consistency paste. The mold of Vicat apparatus was filled with the paste, the surface of the mold was equalized by a knife without pushing it.

The needle was dropped until contact with paste surface, and then the needle was allowed to penetrate inside the paste. This process was repeated in different locations from time to time. The setting time is considered as complete if the needle doesn't touch the glassy board and the distance must not less than 12 mm between the edge of the mold and the location of the test.

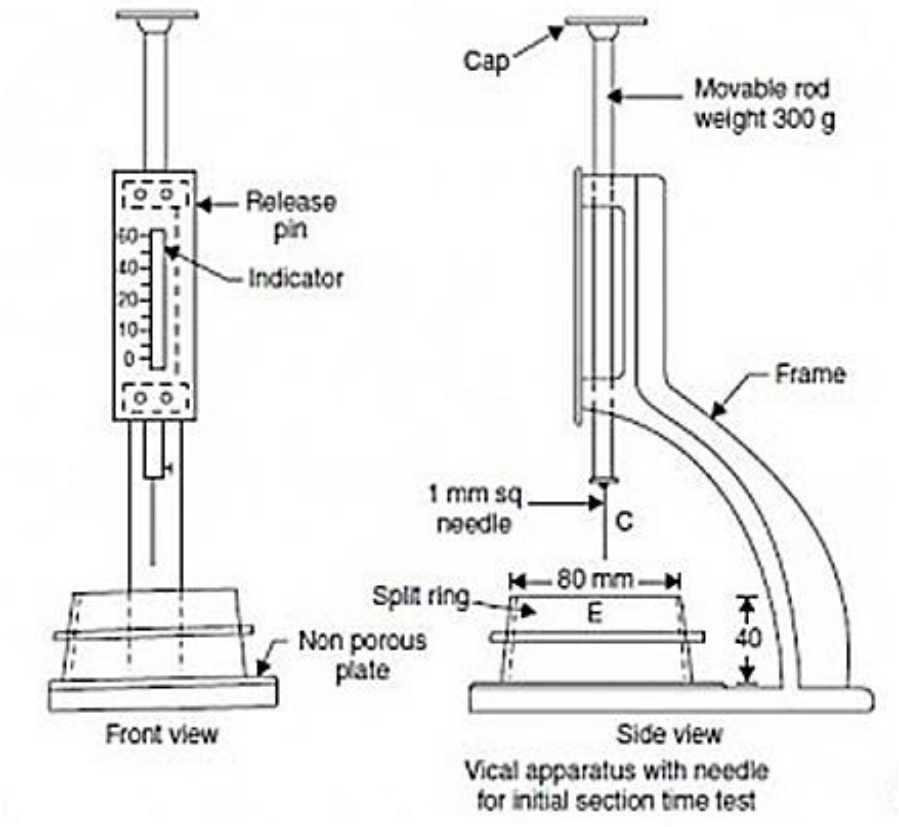

Figure 3 Vicat apparatus

5.3 Compressive Strength Test

The compressive strength test was made by ELE-Auto machine test of 200 kN capacity "Figure 4". The test was done to the samples according to I.Q.S. No.27/1988 [14]. Two steel plate is put in the apparatus and they are oriented as looks so nearer. Then the load was applied as a rate of 2 kN until failure of specimen. The average of 3 results was being taken based on the area of 2500 mm^2.

Figure 4. Compression machine 200 kN

6. Results and Discussions

The results of setting time, compressive strength, increasing in setting time ratio and increasing in compressive strength ratio are shown in "Table 5". Setting time increases when the replacement ratio increases for all additive materials. This is because the additive material disrupts the water arrival to the crystals and delays formation of crystalline mesh which causes hydration.

As shown in "Fig. 5", the setting time for mixes with hydrated lime is the highest and the setting time for mixes with limestone powder is more than the mixes with cement. That is because the setting time for hydrated lime is higher than the setting time for limestone powder. Also, the setting time for limestone powder is higher than setting time for cement.

Table 5. Results of setting time and compressive strength of gypsum

Mix Symbol	Replacement ratio (%)	W/P*	Setting Time (minute)	Compressive Strength (N/mm^2)	Increasing ratio in Setting Time (%)	Increasing ratio in Compressive Strength (%)
RG**	0	0.5	5	2.3	-	-
GC10***	10		6	3.74	20	62.6
GC20	20		6.5	4.21	30	83
GC30	30	0.5	8.1	6.64	62	188.7
GC40	40		9.5	7.57	90	229.1
GC50	50		10.33	8.04	106.6	249.5
GL10****	10		7.8	6.2	56	169.5
GL20	20		8.18	6.0	63.6	160.8
GL30	30	0.5	9.43	5	88.6	117.4
GL40	40		9.85	5	97	117.4
GL50	50		10	4	100	73.9
GL60	60		10	4	100	73.9
GH2.5*****	2.5	0.55	7.43	7.51	48.6	226.5
	7.5	0.60	11.67	5.11	133.4	122.1
GH7.5	12.5	0.65	14.15	3.0	183	30.4
GH12.5						

*Water to Powder (gypsum+powder), **Reference mix without any addition, ***gypsum with 10% cement mixture, **** gypsum with 10% limestone powder mixture, ***** gypsum with 2.5% hydrated lime mixture

In the mix of 50% replacement of cement (GC50) the value of setting time is the highest (10.33 minutes) with increasing ratio of (106.6 %) as shown in "Fig.6". Also, in the mix GL50 (50% replacement of limestone) the highest value of setting time is (10 minutes) with increasing ratio of (100%). The mix GH12.5 (12.5% replacement of hydrated lime) gives highest setting time (14.15 minutes) with increasing ratio (183%). All mixes with additive material are made gypsum conforms to I.Q.S No.28/1988 [11].

The relationship between replacement ratio and compressive strength are shown in "Fig. 6" and the relationship between replacement and increasing ratio of compressive strength are shown in "Fig.7". The compressive strength is affected primarily by water/gypsum ratio (consistency), type of additive material, percentage of additive material and mix type which leaves pores in the matrix when water evaporates. Compressive strength was tested for reference mix and all mixes at 28 days.

It is clear from "Table 5", Fig. (7) and (8), the GC50 which contains (50%) cement has the greatest compressive strength (8.04 N/mm^2) with increasing ratio of (249.5%). This is because that the compressive strength of cement is higher than that for gypsum and the cement filled the pores. All other mixes contain cement (10%-40%) have greater compressive strength than the reference mix (3.74-7.57 N/mm^2) with increasing ratio of (62.6%-220.1%).

It is clear from "Table 5" and "Fig. (7) And (8) that replacing 10% of gypsum by limestone powder (mix GL10) increases compressive strength from 2.3 N/mm^2 for reference mix RG to 6.2 N/mm^2 (169.6% increase) because the limestone powder filled the pores in the matrix due to its high fineness. However, increasing replacement ratio more than 10% gives lower compressive strength than GL10 but still higher than RG because high replacement ratios delay the formation of crystalline mesh.

Results also show that replacing 2.5% of gypsum by hydrated lime (mix GH2.5) increases compressive strength from 2.3 N/mm^2 for reference mix RG to 7.51 N/mm^2 (226.5% increase) because of the high fineness of hydrated lime which works as filling material to the pores in addition to the probability of carbonation. However, increasing replacement ratio more than 2.5% gives lower compressive strength than GH2.5 but still higher than RG because high replacement ratios delay the formation of crystalline mesh.

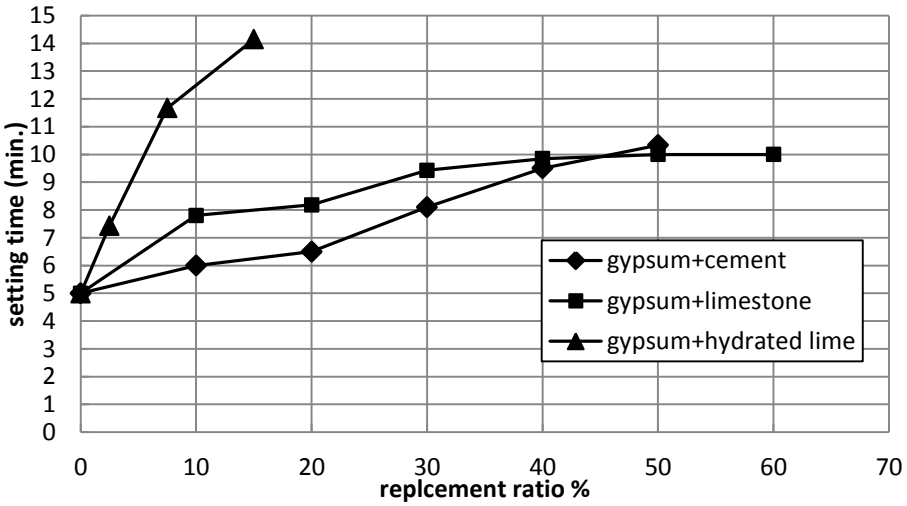

Figure 5. Relationship between replacement ratio and setting time

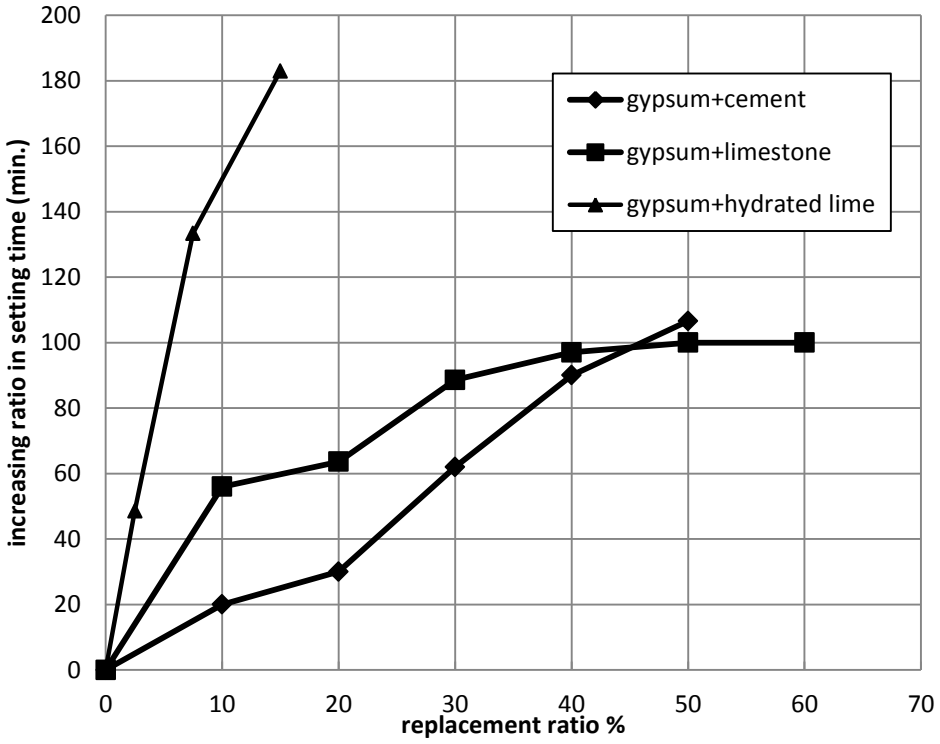

Figure 6. Relationship between replacement ratio and increasing ratio in setting time

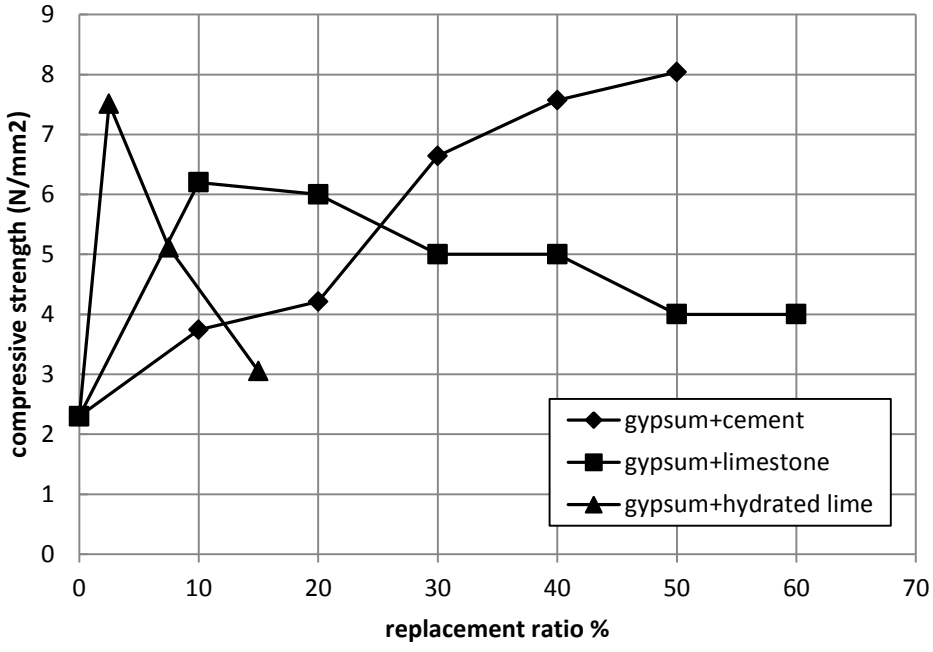

Figure 7. Relationship between replacement ratio and compressive strength

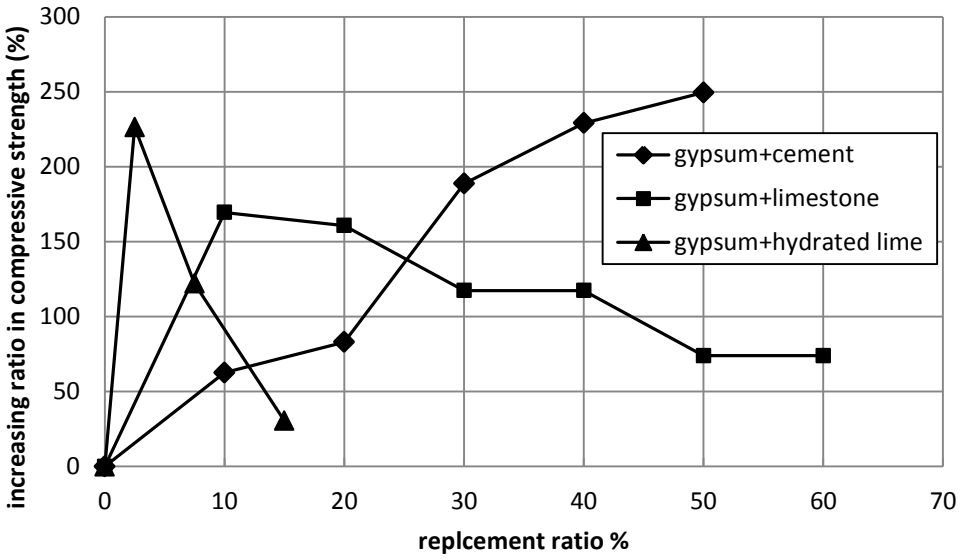

Figure 8. Relationship between replacement ratio and increasing ratio in compressive strength

7. Conclusions

Based on the results of the experimental work performed in this paper the following conclusions can be drawn:

1. The gypsum available in local market is not conforming to I.Q.S No.28/1988 and using cement, limestone powder and hydrated lime as additives overcomes this problem.
2. Using cement to replace 10%-50% of gypsum increases setting time by 20%-106.6% and compressive strength by 62.6%-249.5%, respectively. The best results correspond to the replacement ratio of 50%.
3. Using limestone to replace 10%-60% of gypsum increases setting time by 56%-100% and compressive strength by 169.5%-73.9% respectively. The best results correspond to the replacement ratio of 50% for setting time and 10% for compressive strength.
4. Using hydrated lime to replace 2.5%-12.5% of gypsum increases setting time by 48.6%-183% and compressive strength by 226.5%-30%, respectively. The best results correspond to the replacement ratio of 12.5% setting time and 2.5% for compressive strength.
5. Using replacement ratios more than 10% for limestone powder and 2.5% for hydrated lime does not further increase compressive strength because higher percentages of these additives delay the formation of crystalline mesh and negatively affect compressive strength.

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