Improvement of Physical and Mechanical Properties of Iraqi Gypsum Using Resole Resin

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ABSTRACT

The objective of the present work is to improve physical and mechanical properties of local gypsum reinforced with Resole resin. The water-gypsum ratio was kept at 45% throughout all these experiments. The prepared composites show a clear improvement in impact, compression strength and modulus of elasticity while, the bending strength is reduced when the Resole resin proportion in gypsum was increased from 2.5 to 10%. The other objective of this study was to evaluate the water absorption of Resole –gypsum composite. From the experimental results it may be concluded that swelling % was reduced when the Resole percentage in gypsum- Resole composite was increased.

Keywords: gypsum, Resole, swelling, mechanical properties.

الخلاصة

الهدف من العمل هو بتحسين الخواص الميكانيكية والفيزيائية للجبس المحلي المعزز براتنج الريسول. نسبة الماء الى الجبس ثبتت على نسبة 45% خلال جميع التجارب. النماذج المتراكبة المحضرة من راتنج(Resole) مع الجبس حصل فيها تحسين ملحوظ في قوة التصادم والانضغاط ومعامل المرونة في حين لوحظ هناك انخفاض في قوة الانحناء عندما تغيرت نسبة الريسول في الجبس من 2.5% الى 10% الهدف الاخر من هذه الدراسة هو تقييم امتصاص الماء من قبل متراكبات الجبس والريسول. من النتائج العملية ظهر بشكل واضح, ان درجة الانتفاخ تقل كلما ازدادت نسبة راتنج الريسول في متراكبات جبس-ريسول.

INTRODUCTION

In the past decades, gypsum (hydrated calcium sulfate ($CaSO_4.2H_2O$))-based material and plasters have became the materials of choice for indoor finishing in Iraq and other countries. Excellent performance, attractive appearance, easy application, and its healthful contribution to living conditions have made gypsum a most popular finishing material for centuries [1–3]. This building material is abundant in Iraq and has high purity. The natural gypsum rock reserves of Iraq are estimated to be about 1.2 billion tons. Availability, the relatively low level of start-up investments, and a favorable market situation, all provide conditions for growth and the profitable industrial production of gypsum-based materials in Iraq [4]. The majority of gypsum-based composite materials (GBCM) can be specified within the following groups [5]: Plasters and renders, adhesives, jointing/filling compounds. Gypsum, serves as the base of a number of products, including plaster of Paris (also known as molding plaster, wall plaster, and finishing plaster), Keen's cement, Parisian cement, and Martin's cement. At present time, gypsum is used only for interior applications as plasterboards, blocks for bathroom walls or as fire safeguards [6,7]. Gypsum-polymer composites have been considered suitable alternatives to neat gypsum due to the improved mechanical properties of the composites [8,9].
There are two basic types of phenolic resin, one is called a two-step novolac and the other is called a resole resin. Phenol formaldehyde resins have the largest volume use of any synthetic adhesive. A resole resin is made by condensing phenol and formaldehyde in the presence of an alkaline catalyst using a higher level of formaldehyde than used with novolac. The reaction is stopped by cooling before the gel point is reached. The adhesive is activated by heating just before use so gelatin

occurs[10,11].

The objective of the present work is the preparation of gypsum reinforced with resole resin. Tensile strength, hardness, bending strength, compression strength and water absorption are determined and compared to the properties of hardened gypsum without any admixtures.

Experimental

The specimens of unmodified and modified gypsum with resole resin were prepared with hydration of calcium sulfate hemihydrate. The resole-gypsum ratio was varied from 2.5 to 10% by weight. The quantity of water: gypsum ratio was adjusted at 45% by weight. After casting, the specimens were cured in laboratory conditions at 30°C and about 25% relative humidity for 5 days.

Resole resin

Preparation of Resole -Phenol Formaldehyde Resin

Resin was prepared by condensation reaction between phenol and formaldehyde under alkaline conditions. In a 500 ml three-necked round bottom flask fitted with condenser, mechanical stirrer and thermometer, 0.5 mol phenol, 1 mol 37% aqueous formaldehyde solution and added 25% ammonia solution. The mixture was refluxed with stirring for 90 min at (50-45) °C . The pH of the reaction mixture was then adjusted to (7-8) by addition of 10% sulfuric acid. Two layers were formed then we separate the aqueous layer from resole by decantation[12].

Gypsum

Gypsum used as a main matrix in this project was calcium sulfate hemihydrate gypsum ($CaSO_4.2H_2O$), which was obtained from local market in Baghdad.

$$CaSO_4.\tfrac{1}{2}H_2O + 1\tfrac{1}{2}H_2O \rightarrow CaSO_4.2H_2O$$

Mechanical Properties Tests

Impact Strength

Impact resistance is a measure of the ability of a material or structure to withstand certain level of the application of a sudden load without failure. In charpy method for impact measurement the specimen is notched sometime un-notched [13,14]. In this method, a free swinging pendulum with a round-up mount is used as an impactor. The Charpy impact strength of un-notched specimens (G) in kilo joules per square meter is given by [13]:

$$G = \frac{U}{X.Y} \times 10^3$$

Where U is impact energy in Jules, absorbed by the test specimen, X is the width of specimen in (mm) and Y is the thickness in (mm) of the test specimen. The samples were prepared according to ISO-179.

Bending Strength

Bending strength can be defined as the resistance of material to bending or the ultimate load to be tolerated by the specimen without failure. Bending strength was determined by using 5 *100 mm specimen tested under three-point loading on a span 10cm [15].From three-point test, modulus of elasticity (E) can be evaluated [16]. The test was carried out according ASTM-D790 test.

$$E = \frac{MgL^3}{48IS}$$

Where

M is the load applied at the specimen in gram.

g is acceleration of gravity.

L is the distance between the two loaded points.
S the bending of specimen.
I is the engineering bending moment.Which can be determined by the following relation:

$$I=\frac{bd^3}{12}$$

Where b is the width of specimen
d is the thickness of specimen

Compression Strength
Compression test or crushing strength is the maximum stress that a rigid material withstand under longitudinal compression.Compression strength is the measured as a force per unit area of initial cross-section (50mm), and is listed as MPa.

$$\text{Compressive}=\frac{\text{Load}}{\text{Cross sectional area of sample bfore deformation}}$$

When the resin matrix has a high cross-link density, the polymer becomes rigid with a high value of compressive strength [17]. The samples preparation according ASTM-D695 Method.

Modulus of Elasticity
Three point flexural test was used to evaluate the elastic bending modulus of samples (E). The mechanical tests were determined by using the following relation [18]. The specimens were determined according ASTM-D648 test.

$$E=\frac{gL^3}{48I}$$

Where g is 9.81 m/Sec^2
L is the distance between the two loaded points.
I is the engineering bending moment.

Swelling test
The gypsum specimens were dried to constant weight and immersed in deionized water at room temperature. The gypsum specimens were periodically weighted after removing excess water from the surface of samples with a filter paper. Swelling was calculated from the following relation:

$$\text{Degree of Swelling (\%)}=\frac{W_t - W_o}{W_t}\times 100$$

Where, W_t is the weight of swollen gypsum specimen at time t and W_o is the initial weight of dry specimen.

RESULTS & DISCUSSION

Swelling properties of gypsum
The water absorption by gypsum materials is of a major concern as building material, because the absorption of water by gypsum materials cause serious damage to its mechanical properties [19].
The swelling of unmodified gypsum and gypsum-Resole composite were done through the immersion of these materials in water for different time intervals (from 15 to 195 minutes). Table 1 and fig.1,2 is clearly reveal the decrease in degree of swelling of gypsum-Resole composite, when the Resole resin percentage in gypsum matrix varied from 2.5-10% in comparison with unmodified gypsum. The decreases in water absorption with increase of percentage of Resole polymer may be attributed to the decrease in porosity of gypsum structure. Inherently, the solid structure of solidified gypsum, which, created by hydration of hemihydrated gypsum is porous and the porosity increases with increase in water:gypsum ratio[19]. On the other hand, the hydrophobicity of Resole

resin when dry may be another reason for the decrease in the degree of swelling in gypsum-Resole composite.

Table (1) : Degree of swelling for Phenolic resin (Resole)

Sample	0 min	15 min	30 min	45 min	60 min	75 min	90 min	105 min	120min	135 min	150 min	165min	180 min	195 min
Non	2	2.02	2.15	2.21	2.32	2.34	2.45	2.45	2.45	2.45	2.45	2.45	2.45	2.45
Resole 2.5%	2	2.03	2.09	2.15	2.189	2.245	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3
Resole 5%	2	2.03	2.1	2.13	2.17	2.2	2.24	2.24	2.24	2.24	2.24	2.24	2.24	2.24
Resole 7.5%	2	2.01	2.09	2.11	2.16	2.19	2.2	2.2	2.2	2.2	2.2	2.2	2.2	2.2
Resole 10%	2	2.01	2.02	2.09	2.12	2.15	2.18	2.18	2.18	2.18	2.18	2.18	2.18	2.18

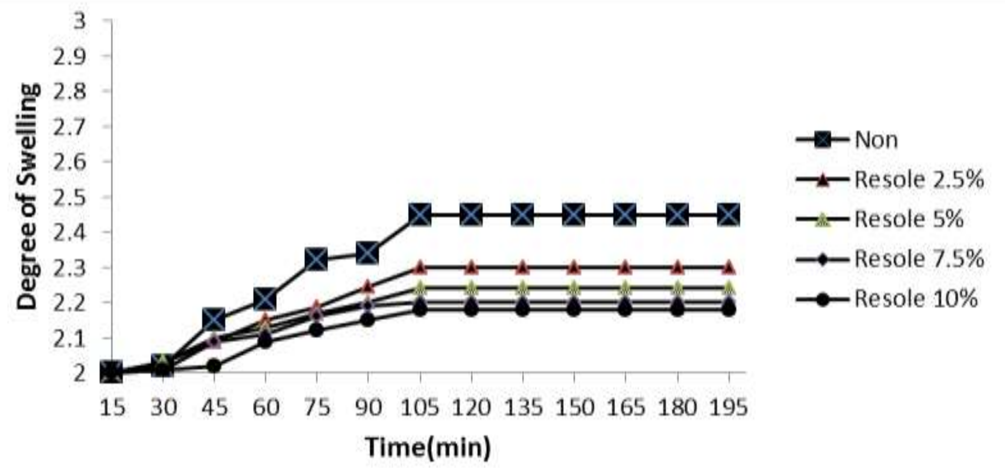

Figure1. Degree of swelling of gypsum and gypsum-Resole composite.

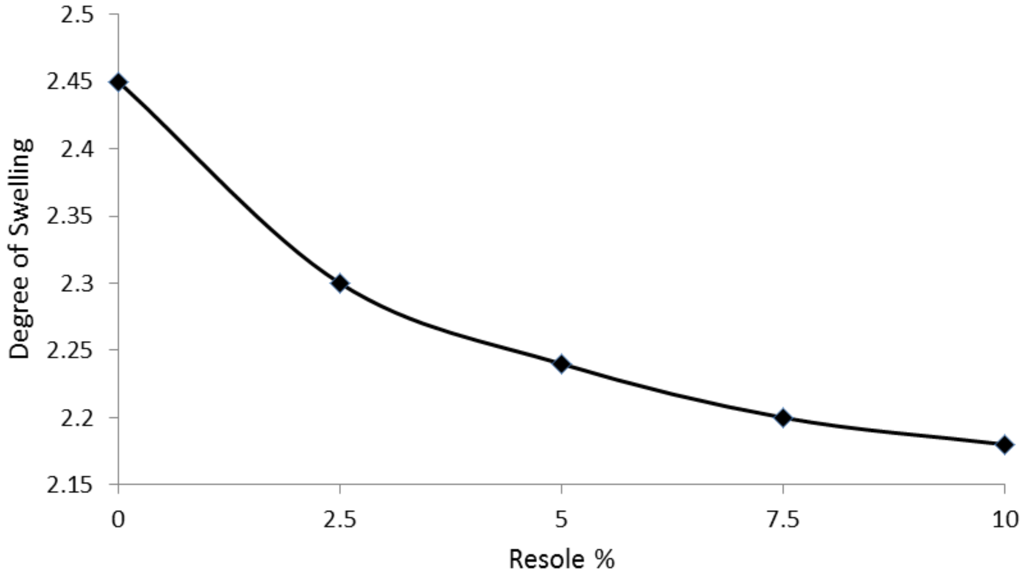

Figure2. Plot of Swelling degree versus the percentage of Resole in gypsum.

Impact strength

When a material or structure is exposed to a sudden force, the material either withstand this load or failed, this behavior is depend on the structure of that material (either rigid or somewhat elastic). The hardened gypsum alone (water:gypsum ratio was 45%) and gypsum Resole composite with Resole resin proportion varied from 2.5-10%, were subjected to different impact forces. It was found that the impact strength is increased when, the Resole resin proportion in gypsum-Resole composite increases figure3 table2. It is clear from table2, that the impact strength increases from 18.73KJ/m-2 for gypsum alone to 36.64KJ/m^{-2} for gypsum-Resole composite with Resole resin percentage is 10%. This increase in impact strength of the composite might be attributed to the ability of the Resole polymer to fillin the voids in the plaster and gives an elastic behavior to the gypsum-Resole composite, whichfinally lead to increase the impact strength of gypsum-Resole composite.

Table (2) :ImpactForce for Phenolic resin (Resole)

Sample	ImpactForce(KJ/m^2)
Non	18.73
Resole2.5%	29
Resole 5%	31.91
Resole 7.5%	36.64
Resole 10%	37.18

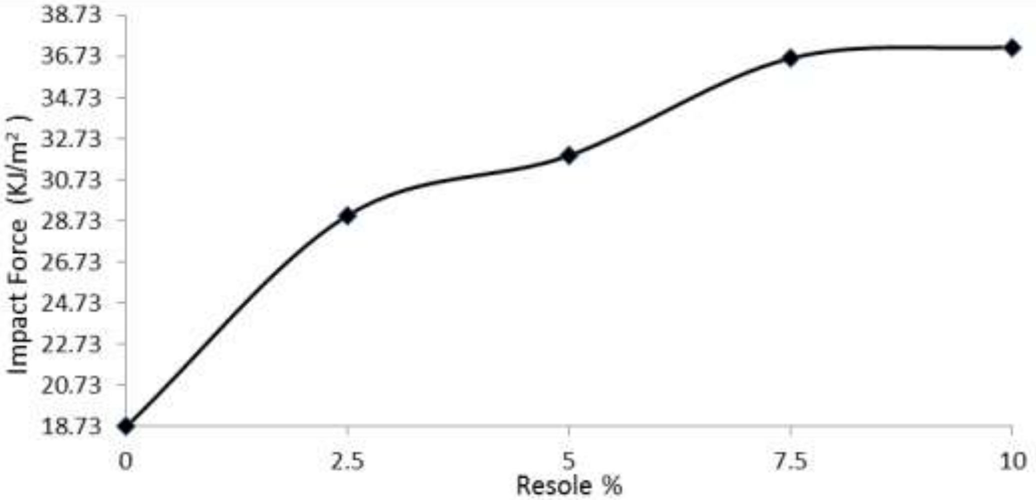

Figure3. Plot of impact strength versus the percentage of Resole in gypsum-Resole composite

Bending and Compression strength

The bending and compression strength are an important factors for evaluation of building materials, such as gypsum. The bending and compression strength of unmodified and modified gypsum with 2.5 to 10% of Resole resin were carried out. It was observed from table3 and fig.4. There was a clear reduction of bending strength with increasing Resole ratio from 2.5 to10% (at constant water:gypsum ratio). In general, the elastic material has low bending strength when the three point test is used for estimation of bending strength accompanied with high modulus of elasticity[20]. Compression strength is the maximum force that applied on a unit area, the increase in compression strength of modified gypsum with Resole is shown in fig.5 and table4, this increase may be due to the decrease of porosity of gypsum structure with the increase of Resole resin proportion. In the case of modified gypsum with Resole the reduction of bending strength accompanied with increase of modulus of elasticity table5 and fig.6, the deflection against mass in bending strength measurement was illustrated in fig.7 and table6. The increase of proportion of Resole in gypsum matrix, although it reduces the voids in the structure of gypsum but, on the other hand, it increases to some extent the elasticity of the product. This behavior leads to decrease of bending strength and increase the elasticity of the modified gypsum with Resole, moreover, the Resole resin has low film strength [19].

Table (3) :Bending strengthfor Phenolic resin (Resole)

Sample	Modulus of Elasticity(MPa)
Non	371
Resole2.5 %	411
Resole 5%	425.4
Resole 7.5%	499
Resole 10%	501.7

Table (4) : Comparison for Phenolic resin (Resole)

Sample	Comparison Force(MPa)
Non	16
Resole2.5 %	21
Resole 5%	23.4
Resole 7.5%	25
Resole 10%	28

Table (5) :Modulus of Elasticity for Phenolic resin (Resole)

Sample	Bending (Kg/mm)
Non	28.5
Resole2.5 %	25.3
Resole 5%	23.5
Resole 7.5%	21.55
Resole 10%	19.0

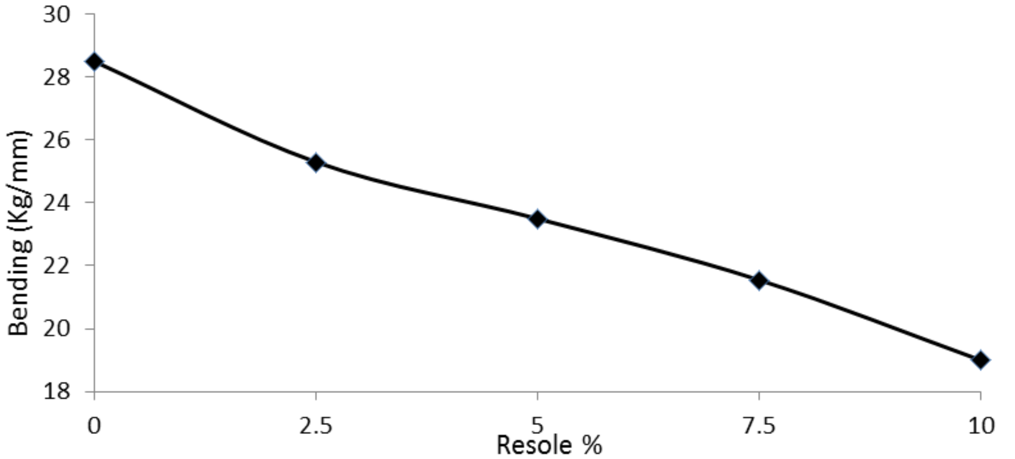

Figure4. Plot of bending strength VS. percentage of Resole in modified and unmodified gypsum.

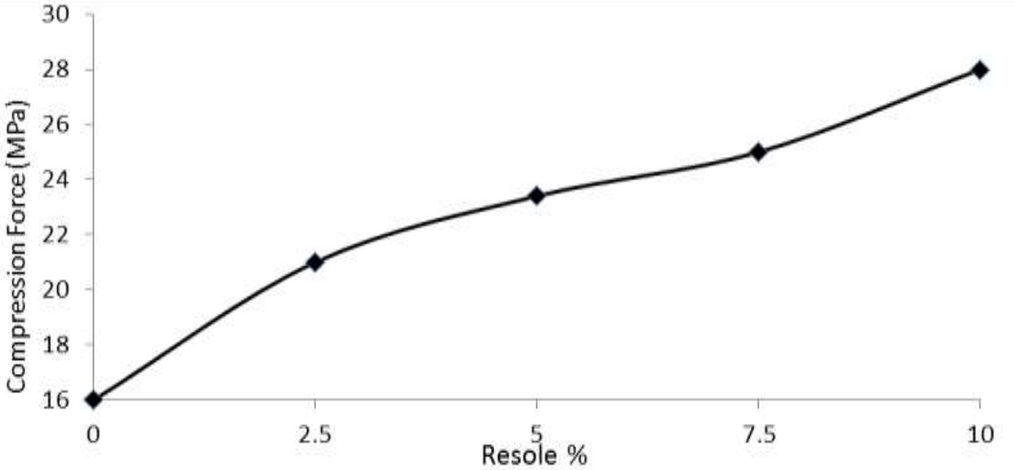

Figure5. Plot of compression strength versus the percentage of Resole in gypsum.

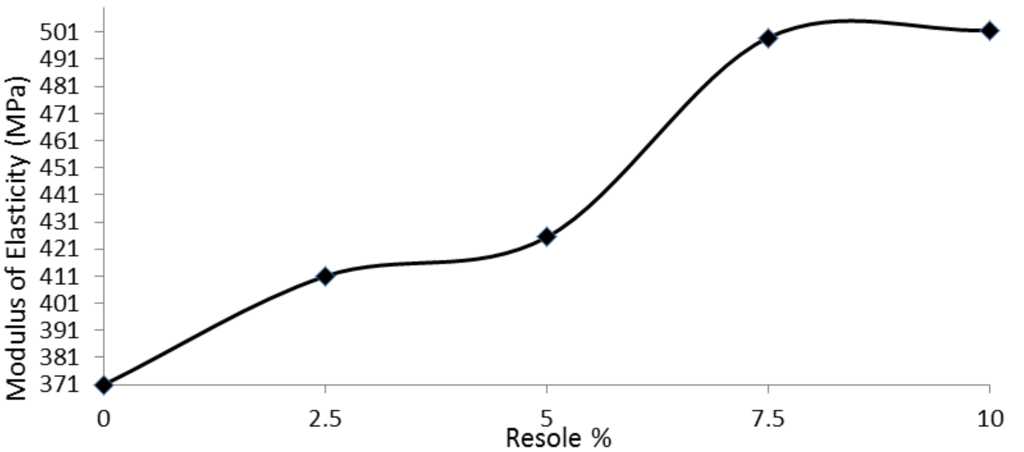

Figure6. Modulus of elasticity versus Resole percentage in modified and unmodified gypsum.

Table (6) Bending for Phenolic resin (Resole)

Mass(kg)	mm) Deflection *10^{-2})				
	Non	Resole 2.5%	Resole 5%	Resole 7.5%	Resole 10%
100	2.25	1.35	1.2	2.05	0.9
200	5.5	3.4	3.6	3.4	2.65
300	8.0	5.0	5.55	5.35	4.55
400	11.0	7.35	8.5	7.05	6.0
500	13.0	10.85	10.60	8.85	8.35
600	15.5	13.6	13.2	11.05	10.5
700	18.0	16.55	15.9	13.75	12.1
800	21.5	19.2	18.55	16.55	14.5
900	25.0	22.5	20.85	18.85	16.55
1000	28.5	25.3	23.5	21.55	19.0

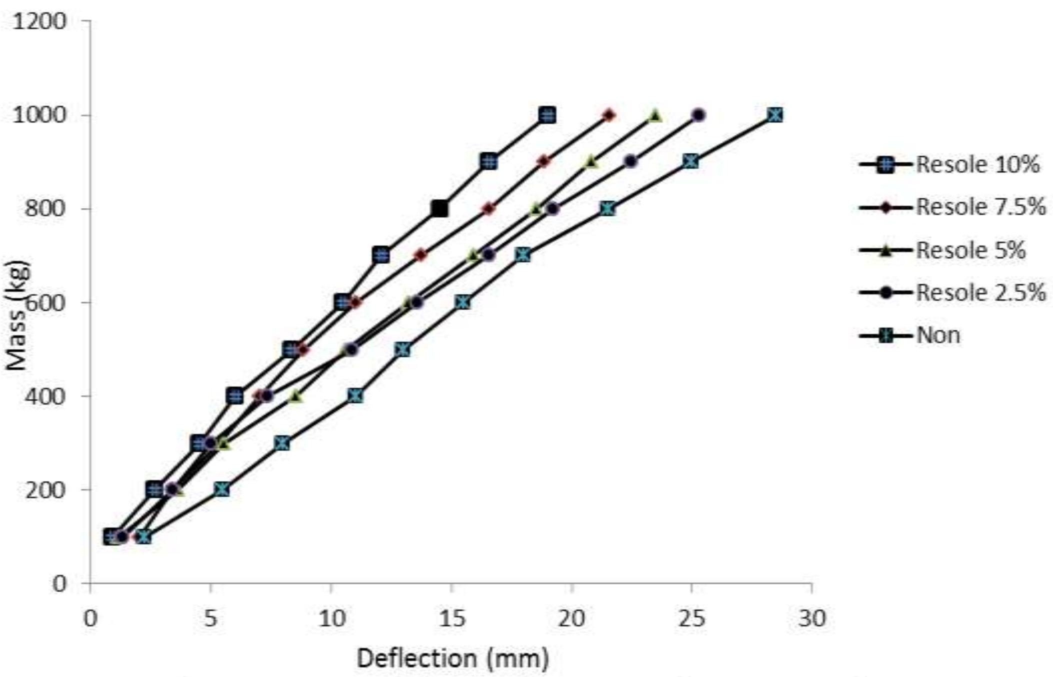

Figure7. Plot mass versus deflection of gypsum and gypsum-Resole composite

Conclusion

Gypsum as building material was brought from local market and modified with Resole polymer. The Resole resin was added with water needed for hydration of $CaSO_4.½H_2O$ during molding of the matrix. The Resole content in gypsum was varied from 2.5 to 10%. The physical and mechanical properties of the produced composite were evaluated. Water absorption by the composite was reduced with increase of Resole polymer percentage. The impact, Compression and modulus of elasticity was increased with Resole increase, while the bending strength was decreased with increase of Resole resin percentage. In brief, the water absorption is a major concern for the gypsum as building material, reduction of this property is a prime objective, So, this work still in progress in order to find a new effective and cheap material to reduce the water absorption of gypsum and increase the mechanical properties.

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