ASSESSMENT OF GYPSUM AND LIMESTONE ROCKS FROM FAT'HA FORMATION (M. MIocene) FOR MAKING WALLBOARD IN THE MAKMUR AREA – NORTHERN IRAQ

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ABSTRACT

Five samples of gypsum and one sample of limestone were collected from an outcrop in Qara Chugh Anticline. The samples of gypsum were analyzed chemically before and after calcination. The purity of the samples of gypsum and limestone was calculated and the (H₂O⁻) content of the calcined samples of gypsum was determined. The physical and mechanical properties of plaster of Paris produced from calcining the samples of gypsum were tested according to the Iraqi and American Standards. A wallboard sample was made using basic materials according to the American Standard. The chemical analyses of the samples of gypsum and plaster of Paris conform to the Iraqi and American Standards. The Fat'ha Formation rocks in the Makhmur area are suitable for making wallboard. The optimum conditions for calcining the studied gypsum rocks to produce plaster of Paris conforming to the standard specifications and suitable for the manufacture of wallboard are 130 °C for 1 hour.

KEYWORDS: Gypsum, Fat'ha Formation, Makhmur Area, Plaster of Paris, Wallboard.

1. INTRODUCTION

Wallboard (also known as plasterboard, drywall, or gypsum board) is a panel made of gypsum plaster pressed between two thick sheets of paper. It is used for making interior walls, floors, ceilings, and facade construction. Wallboard construction became prevalent as a speedier alternative to traditional lath and plaster (Mehta et al., 2013). Wallboard differs from other panel-type building products, such as plywood, hardboard, and fiberboard due to its non-combustible core and paper facers. When joints and fastener heads are covered with a joint compound system, gypsum wallboard creates a continuous surface suitable for most types of interior decoration (Kubba, 2016).

The use of wallboard in the building is more economical than bricks and cement (Natus, 1990). It is also used in the manufacture of fire-resistant doors, furniture, kitchens, and bathrooms due to its high resistance to moisture and fire. The expansion of reconstruction and urban growth in Iraq increases the need to use new building materials that compete with traditional ones in terms of efficiency, low cost, and environmentally friendly. The availability of the numerous geological resources in Iraq encourages their exploitation in the manufacture of such building materials.

Gypsum rocks are widespread in Iraq, especially in the northern regions. Most of them belong to the Fat'ha Formation (Middle Miocene) which is one of the most widespread and economically important formations in Iraq (Buday, 1980). Limestone and gypsum rocks of this formation are used in many industries, such as Portland cement and plaster. It is also used as building and decorative stone.

The current research aims to assess gypsum and limestone rocks from Fat'ha Formation for making wallboards in the Makhmur area - Northern Iraq.

2. GEOLOGY OF THE STUDY AREA

The study area is located about 60 km southwest of Erbil city and about 2.5 km from Makhmur town. Tectonically, it lies within the Hemrin-Makhul Subzone of the Low Folded Zone in the Unstable Shelf according to the tectonic division of Iraq (Buday and Jassim, 1984) (Figure 1). The outcrop from which the samples were collected belongs to the Fat'ha...
Formation at the northeastern limb of Qara Chugh Anticline with coordinates (N 35° 46' 30'') and (E 43° 38' 00''). This site is known as Makhmur Gypsum Quarry. The lithostratigraphic section of this site consists of white to light grey massive gypsum beds of varying thickness, underlain by a well-bedded recrystallized limestone bed of 3 m thick and overlain by a limestone bed of 5 m thick. The total thickness of the gypsum beds in this section is 82 m (Figure 2). Gypsum rocks were used in the production of technical plaster in the Makhmur plant. It is currently used in the manufacture of local plaster calcined by the primitive "Koor" method in several locations near the Dibagah-Makhmur road.

![Location map of the study area](image1.png)

**Fig. (1):** Location map of the study area. Source: Google Earth. August 24, 2020.

![Lithostratigraphic section](image2.png)

**Fig. (2):** Lithostratigraphic section of Fat’ha Formation / Makhmur Gypsum Quarry

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3. MATERIALS AND METHODS

1. Chemical Analysis:

Five samples of gypsum and one sample of limestone were collected from the outcrop. The samples of gypsum were washed, dried, and ground by a tema mill. The resulting powders were sieved with a 150μ sieve and the portion that passed through the sieve was kept in closed boxes for use in chemical analysis. Powders were analyzed chemically before and after calcination. The (CaO %) content was measured volumetrically for each powder by titration of calcium oxide (CaO) solution of the unknown sample with 0.01M EDTA solution using murexide as an indicator. The (SO₄ %) content was measured gravimetrically by barium chloride (BaCl₂) to precipitate sulphate ions (SO₄²⁻) (Vogel, 1961; Abbawai and Hasan, 1990). The (H₂O %) content was measured by heating 1 gm of the powder sample in a muffle oven at 400 °C for 1 hour and calculating the weight loss (Aljubouri, 1972). The insoluble residue (I.R.%), was measured by dissolving 5 gm of the powder sample in 10 ml of 10% HCl and weighing the residue (Vogel, 1961; Ellingboe and Wilson, 1964). The (CaO) and (SO₄) contents were measured in the Geochemistry Laboratory, Department of Geology, University of Mosul, while the (H₂O⁰) and (I.R.) were measured in the Analytical Chemistry Laboratory, Department of Chemistry, University of Mosul using the methods described in (Baddy, 2009).

2. Calculation of Powder Gypsum Samples:

This process is the most important stage in gypsum processing and wallboard manufacturing, where the gypsum is heated in the form of dihydrate containing water chemically bound by about 21% of its weight to convert it into plaster of Paris according to the following equation (Venta, 1997):

\[ \text{CaSO}_4 \cdot 2\text{H}_2\text{O} \overset{130-170 ^{\circ}C}{\rightarrow} \text{CaSO}_4 \cdot 1/2 \text{H}_2\text{O} + 1 \, 1/2 \text{H}_2\text{O} \]

One kg of each powder was calcined in an electrical oven to produce plaster of Paris according to American Standard (ASTM C28/C28-10, 2015) and (Iraqi Standard No.26, 1969). The calcination method is described in (Al-Qaraghooli, 1989). The limestone sample was analyzed by X-ray fluorescence (XRF) (Model MINI PAL 4 CEMENT extends).

3. Physical and Mechanical Examinations of Plaster of Paris:

The compressive strength of plaster of Paris produced from the calcined powders was measured according to the method described in (Aljubouri and Baddy, 2012; Iraqi Standard No.27, 1985) using a compression machine (model ELE HP2 7HB). The (Iraqi Standard No.27, 1985) was followed to measure the water/powder ratio (W/P) and setting time of the plaster of Paris by Vicat Tester (Model No. H-3134) and (Model No. H-3050) respectively. The measurement methods are described in (Baddy, 2009; Younis, 2012).

4. Making a Wallboard Sample:

American Standard (ASTM C36-C36M, 2004) was followed to make a wallboard sample from plaster of Paris in the laboratory at the Department of Geology, University of Mosul. The main components used are a wooden mold (10 × 20 cm) (Figure 3 – A), an iron wire network (9.5 × 19.5 cm), and two pieces of cardboard (10 × 20 cm) (Figure 3 – B).

The plaster forming the core of the wallboard sample was prepared and water was mixed with the plaster at a (W/P) ratio of 50 ml/100 gm (Figure 3 – C). A cardboard sheet (veneer) was laid under the wooden mold and the plaster was mixed with enough water to form a smooth plastic mass. After the mass had completely hardened it was mixed with the limestone powder and an additional amount of water to prepare the gypsum slurry. The limestone powder was added at a rate of 0.1% to the mixture as a retarder in order to increase the setting time and allow the formation of the plaster mixture for molding and shaping during the preparation of the wallboard sample (Figure 3 – D). The slurry mass was poured to half the thickness of the wooden mold and the iron wire network was laid to increase the hardness of the prepared sample. The second half of the mold was poured with the slurry mass and the second sheet of veneer was laid to cover the wooden mold (Figure 3 – E). The finished wallboard sample was dried in an electric oven at 35 °C for 1 hour according to American Standard (ASTM C36-C36M, 2004) (Figure 3 – F).

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Fig. (3): Making a wallboard sample. A: Wooden wallboard mold; B: Cardboard and iron wire network; C: Mixing the plaster with water; D: Adding the limestone powder to the mixture; E: The wallboard sample before drying; F: The finished wallboard sample.

4. RESULTS AND DISCUSSION

The chemical analysis showed significant differences in the (CaO%) and (SO₃%) contents (before and after calcination) and there were no significant differences in the (H₂O⁺%) content and (I.R.%). The total content of the main gypsum components (CaO, SO₃, and H₂O) ranged between 97-98% of the weight of the gypsum samples. These results are close to the ideal chemical composition of gypsum (32.57% CaO, 20.93% H₂O, and 46.50% SO₃) and this indicates the high purity of the studied gypsum rocks. The remaining (2–3%) represents the other secondary components (Fe₂O₃, Al₂O₃, and K₂O) which reflect a small amount of detrital impurities that were supplied to the sedimentary basin. According to (Aljubouri and Sulayman, 1996), the first component represents the evaporite minerals while the second component represents the detrital minerals.

Table (1): The chemical analysis of gypsum powder samples before and after calcination

<table>
<thead>
<tr>
<th>Contents</th>
<th>N</th>
<th>Mean</th>
<th>SD</th>
<th>P-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO%</td>
<td>5</td>
<td>Before calcination 32.17</td>
<td>1.2386</td>
<td>0.0393</td>
</tr>
<tr>
<td></td>
<td></td>
<td>After calcination 37.78</td>
<td>0.4374</td>
<td></td>
</tr>
<tr>
<td>SO₃%</td>
<td>5</td>
<td>Before calcination 45.71</td>
<td>0.3980</td>
<td>&lt;0.0000001</td>
</tr>
<tr>
<td></td>
<td></td>
<td>After calcination 53.76</td>
<td>0.1513</td>
<td></td>
</tr>
<tr>
<td>H₂O⁺%</td>
<td>5</td>
<td>Before calcination 19.93</td>
<td>0.1449</td>
<td>0.6609</td>
</tr>
<tr>
<td></td>
<td></td>
<td>After calcination 5.30</td>
<td>0.1782</td>
<td></td>
</tr>
<tr>
<td>I.R.%</td>
<td>5</td>
<td>Before calcination 1.56</td>
<td>0.3129</td>
<td>0.7416</td>
</tr>
<tr>
<td></td>
<td></td>
<td>After calcination 2.12</td>
<td>0.2679</td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>5</td>
<td>Before calcination 99.37</td>
<td>1.0105</td>
<td>0.2327</td>
</tr>
<tr>
<td></td>
<td></td>
<td>After calcination 98.95</td>
<td>0.5687</td>
<td></td>
</tr>
</tbody>
</table>

The purity of gypsum samples was calculated using the following equation (Aljubouri, 1972):

Gypsum (CaSO₄.2 H₂O) (%) = H₂O⁺% × 4.778 = 19.93 × 4.778 = 95.23%

The purity of gypsum rocks varies widely, ranging from 80-95%. This property is important in the manufacture of wallboard which requires high purity gypsum to produce lightweight wallboard (Henkels and Gaynor, 1995). The physical and mechanical properties of pure gypsum can be easily controlled by adding retarders and accelerators compared to impure gypsum.

The average contents of (CaO%), (SO₃%), (H₂O⁺%) and (I.R.%) for the gypsum samples conform to American Standard (ASTM
The (CaO%), (SO₃) and (H₂O+% contents of plaster of Paris in this study are close to those of American Standard (ASTM C28/C28M-10, 2015) and plaster of Paris produced by (Othman, 2002; Aljubouri and Baddy, 2012; Younis, 2012). The (CaO/SO₃) ratio in the plaster in this study was slightly higher than that of the theoretical bassanite (7.00), indicating a small amount of dolomite in the produced plaster. Gypsum and its products usually contain a small amount of dolomite, which is believed to be a product of local dolomitization (Othman, 2002; Aljubouri and Alrawas, 2006; Baddy, 2009; Mohammed, 2010).

The composition of bassanite (CaSO₄.1/2H₂O) in (Table 2) was calculated by multiplying (SO₃%) by (1.8129). This oxide is used in place of (CaO) because the latter is present in greater amounts in dolomite (Aljubouri and Baddy, 2012). (H₂O+) content cannot be used because its quality is controlled by calcination. The oxide content of insoluble residue can be calculated according to (Aljubouri and Alkawaz, 2008) using its chemical analysis as follows:

\[\text{SiO}_2 = \text{I.R.} \times 0.578, \quad \text{Al}_2\text{O}_3 = \text{I.R.} \times 0.1376, \quad \text{Total Fe}_2\text{O}_3 = \text{I.R.} \times 0.1055, \quad \text{Total MgO} = \text{I.R.} \times 0.1789\]

### Table (2): Average contents of plaster of Paris in the current study compared to the commercial plasters and theoretical bassanite (CaSO₄.1/2H₂O)

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ahlia Plaster</td>
<td>Malej Plaster</td>
<td>Rasheed Local Plaster</td>
<td>Madae’n Local Plaster</td>
</tr>
<tr>
<td>CaO</td>
<td>37.78</td>
<td>37.62</td>
<td>37.84</td>
<td>38.26</td>
</tr>
<tr>
<td>SO₃</td>
<td>53.75</td>
<td>53.93</td>
<td>54.29</td>
<td>53.17</td>
</tr>
<tr>
<td>H₂O+%</td>
<td>5.30</td>
<td>7.20</td>
<td>6.70</td>
<td>6.45</td>
</tr>
<tr>
<td>* SiO₂</td>
<td>1.22</td>
<td>0.50</td>
<td>0.45</td>
<td>1.26</td>
</tr>
<tr>
<td>* Al₂O₃</td>
<td>0.29</td>
<td>0.12</td>
<td>0.10</td>
<td>0.30</td>
</tr>
<tr>
<td>* Total Fe₂O₃</td>
<td>0.22</td>
<td>0.09</td>
<td>0.08</td>
<td>0.23</td>
</tr>
<tr>
<td>* Total MgO</td>
<td>0.38</td>
<td>0.15</td>
<td>0.14</td>
<td>0.29</td>
</tr>
<tr>
<td>Total</td>
<td>98.93</td>
<td>99.61</td>
<td>99.60</td>
<td>99.14</td>
</tr>
<tr>
<td>CaO/ SO₃</td>
<td>0.7029</td>
<td>0.7017</td>
<td>0.700</td>
<td>0.7042</td>
</tr>
<tr>
<td># CaSO₄. 1/2H₂O</td>
<td>97.44</td>
<td>97.77</td>
<td>98.42</td>
<td>96.39</td>
</tr>
<tr>
<td>I.R.</td>
<td>2.11</td>
<td>0.86</td>
<td>0.77</td>
<td>2.18</td>
</tr>
</tbody>
</table>

* Calculated from insoluble residue (I.R.); # CaSO₄.1/2 H₂O = SO₃% × 1.8129

Table (3) shows the chemical analysis of the limestone sample, which can be classified as highly pure by its CaCO₃ content. This encourages the use of this limestone as a retarder to reduce the setting time of plaster of Paris during the manufacture of wallboard.

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Table (3): The chemical analysis of the limestone sample

<table>
<thead>
<tr>
<th>Oxide</th>
<th>SiO$_2$</th>
<th>Al$_2$O$_3$</th>
<th>Fe$_2$O$_3$</th>
<th>FeO</th>
<th>CaO</th>
<th>MgO</th>
<th>Na$_2$O</th>
<th>K$_2$O</th>
<th>SO$_3$</th>
<th>CaCO$_3$</th>
</tr>
</thead>
<tbody>
<tr>
<td>(%)</td>
<td>3.33</td>
<td>1.91</td>
<td>0.89</td>
<td>0.80</td>
<td>53.50</td>
<td>0.32</td>
<td>0.39</td>
<td>0.18</td>
<td>0.62</td>
<td>95.23</td>
</tr>
</tbody>
</table>

The calcination experiments of the powder gypsum samples showed that the optimum calcination conditions are 130 °C for 1 hour, giving a value of (H$_2$O$^+$ = 6.40%) similar to that of bassanite (H$_2$O$^+$ = 6.20%) as shown in Table 4.

Table 4: The (H$_2$O$^+$ %) content of the calcined gypsum samples compared to the theoretical content of bassanite (6.20%) using different temperatures with a constant calcination time of 1 hour

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Temperature (°C)</th>
<th>H$_2$O$^+$ %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>110</td>
<td>8.9</td>
</tr>
<tr>
<td>2</td>
<td>120</td>
<td>7.2</td>
</tr>
<tr>
<td>3</td>
<td>130</td>
<td>6.4</td>
</tr>
<tr>
<td>4</td>
<td>140</td>
<td>5.8</td>
</tr>
<tr>
<td>5</td>
<td>150</td>
<td>5.3</td>
</tr>
</tbody>
</table>

Table (5) shows the physical and mechanical tests of plaster of Paris in the present study. These results are consistent with (Iraqi Standard No. 28, 1988) and (American Standard ASTM C472-99, 2014), as well as the results obtained by (Aljubouri and Alrawas, 2009).

Table 5: Physical and mechanical tests of plaster of Paris in the present study compared to the technical plaster in (Aljubouri and Alrawas, 2009), (Iraqi Standard No. 28, 1988) and American Standard (ASTM No. C472-99, 2014)

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Compressive strength (MPa)</td>
<td>18</td>
<td>20</td>
<td>Not less than 3</td>
<td>Not more than 50</td>
</tr>
<tr>
<td>Water/powder ratio (W/P) (ml/100 gm)</td>
<td>50</td>
<td>40 – 50</td>
<td>-----</td>
<td>-----</td>
</tr>
<tr>
<td>Setting time (min.)</td>
<td>9</td>
<td>6 – 8</td>
<td>8 – 25</td>
<td>5 – 15</td>
</tr>
<tr>
<td>Fineness (particle size) (%)</td>
<td>-250, 100%</td>
<td>-250, 100%</td>
<td>100% passing from sieve No. 16 (1.18 mm)</td>
<td>-315 (µ),+80 (µ)</td>
</tr>
</tbody>
</table>

The compressive strength of plaster of Paris in this study is slightly less than that of (Aljubouri and Alrawas, 2009) because the (W/P) ratio of plaster of Paris in this study is higher than that in the other study as the relationship between (W/P) ratio and compressive strength is inverse (Noort, 2002; Abdulla, 2006). The setting time of plaster of Paris in this study is greater than that of (Aljubouri and Alrawas, 2009) as the relationship between this property and the (W/P) ratio is proportional (McCabe, 1985).

5. CONCLUSIONS

The following conclusions were drawn from the current study:
1. The results of the chemical analyses of the gypsum samples and plaster of Paris in this study conform to Iraqi and American Standards.
2. The results of the physical and mechanical examinations of the plaster of Paris produced in this study conform to the Iraqi and American Standards.
3. The gypsum rocks of Fat’ha Formation in the Makhmur area are suitable for use in making
wallboard. The limestone rocks in this area can also be used as a hardening retarder for gypsum plaster in wallboard making.

4. The optimum conditions for calcining the studied gypsum rocks are at a temperature of 130 °C for 1 hour to obtain plaster of Paris conforming to the standards of manufacturing wallboard.

6. REFERENCES

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يوخته

بيان سهل مازب جيسوم و ثبز مازب جيرن جيرن. هانه كونكرن جيثرن جيسوم مازب

يشكينين كيماتي هانه كن بو جيرن جيسوم بردي. ويشني كلاكرن ويازرا بو جيرن جيسوم مازب

دناف جيرن جيسوم مازب كلاكرن هانه بفيك. سحيلتين فيزيائ و ميكانيكي هانه كن بو باريس بلاستر نوؤر مازب كلاكرن جيسوم مازب جيثرن جيسوم مازب دهنجام و ليف تابيه تمليدين عيرافي و نه مربي. سميلهن جيسوم بورد مازب باريس بلاستر هانه دوستكرن بريكا بكارتينا ماددين شرهات و ليف تابيه تمليدين لامريكي بين ستاندر. يشكينين كيماتي بين سملتين جيسوم مازب هن كلاكر و باريس بلاستر وكي تمامتميدين عيرافي و لامريكي ديايرون. هروهسا يشكينين فيزيائ و ميكانيكي بين باريس بلاستر وكي تمامتميدين ستاندر بين عيرافي و لامريكي ديايرون. بيرن جيسوم لدوئين ل دفقر ماخور بين ماتن (باشن) بو دوستكرن بوردن دياوي. باردوخنن باش بو كلاكرن جيسوم مازب بدهستتينانانان باريس بلاستر ل ديف تابيه تمليدين ستاندر و دوختن مان بو دوستكرن بوردن دياوير 130 م. و ردميرى بو هر دمزميرى. 

يوختن كليل: جيسوم، ينكاها فا تحت، دفقرا م مخور، باريس بلاستر، بوردن دياوير.

الخلاصة

جمعت خمس عينات من صخور الجيسوم وعينة واحدة من الحجر الجيري من أحد المكاشف الصخرية ضمن طية قره جوغ المحددة. أجريت التحالل الكيميائي لعينات الجيسوم قبل وبعد الكلسنة وحسب نقاوة صخور الجيسوم غير المكلسة ونيقاوة الحجر الجيري ومحنمو (H2O) لعينات الجيسوم المكلسة. قيست الخواص الفيزيانة والميكانيكية لباريس بلاستر الناتج من كلاسة عينات الجيسوم وحسب المواصفات العراقية والأميركية. حضرت عينة من ألواح الجدار (الجيسوم بورد) من باريس بلاستر باستخدام مواد أساسية وحسب المواصفة القياسية الأميركية. تنتفوق التحالل الكيميائي لعينات الجيسوم غير المكلسة وباريس بلاستر مع المواصفات القياسية العراقية والأميركية. كما تنتفوق الفحوصات الفيزيانة والميكانيكية لباريس بلاستر الناتج مع المواصفات القياسية العراقية والأميركية. إن صخور الجيسوم المكلسة لتكون فتحة في منطقة مخمور صالحة لتصنيع ألاواج الجدار. إن الظروف المثلى للكلسة عينات الجيسوم المدرسة للحصول على باريس بلاستر مطابق للمواصفات القياسية وملائم لتصنيع ألاواج الجدار هي درجة حرارة 130 م لمدة ساعة واحدة.

الكلمات المفتاحية: الجيسوم، تكوين فتحة، منطقة مخمور باريس بلاستر، ألاواج الجدار

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