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The Effects of Different Water Types on the Dissolution, Physical Appearance, and Compressive Strength of Gypsum Rock

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ABSTRACT

Dissolution of gypsum rock in water is significant, which may result in hydrocarbon reservoir formation and evaporate deposits. However, the complexity of the gypsum dissolution process is still of interest because of its uncleanness that requires more critical analysis. The objectives of this experimental study are emphasis on the dissolution characteristics of gypsum rock under room temperature and by various types of water; namely: deionized, tap, fresh, acidic, well, and normal rainwatre. In addition, the influences of dissolution on gypsum rock's mechanical and physical characteristics. Gypsum rock was obtained from Agjalar area, in the southwest of Sulaymaniyah city, Northern Iraq. Experimental results show that well water is the most effective type to dissolve gypsum and cause significant reductions in the mechanical characteristics of gypsum rock.

Keywords: Water Type, Dissolution Characteristics, Physical Appearance, Compressive Strength, Gypsum Rock.

تأثير أنواع المياه المختلفة على الانحلال والمظهر الفيزيائي والقوة الانضغاطية لصخور الجبس

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الخلاصة

يعتبر انحلال الصخور الجبسية في الماء أمرًا مهمًا، مما يؤدي إلى تكوين احتياطي هيدروكربوني ورواسب متبخرة. ومع ذلك، فإن تعقيد عملية إذابة الجبسوم لا يزال موضع اهتمام بسبب عدم وضوحه الذي يتطلب المزيد من التحليل النقدي. تتمثل أهداف هذه الدراسة التجريبية في التركيز على خصائص انحلال الصخور الجبسية تحت درجة حرارة الغرفة وبواسطة أنواع مختلفة من الماء. وهي: الماء منزوع الأيونات، ماء الصنبور، الماء العذب، الماء الحامضي، مياه الابار، وماء الأمطار العادية. بالإضافة إلى تأثير الذوبان في الخصائص الميكانيكية والفيزيائية للصخور الجبسية. تم الحصول على الصخور الجبسية من منطقة أغجلار، جنوب غرب مدينة السليمانية، شمال العراق. أظهرت النتائج التجريبية أن مياه الآبار هي النوع الأكثر فاعلية في إذابة الجبسوم والتسبب في انخفاض كبير في الخصائص الميكانيكية للصخور الجبسية.

الكلمات الرئيسية: نوع الماء ، الذوبان ، المظهر الفيزيائي ، قوة الضغط ، صخور الجبس .

1. INTRODUCTION

In sedimentary stratums, gypsum exists in a combination with other minerals such as sulfur, dolomite, halite, anhydrite, which rely on water's salinity and temperature (**Blattler, 2020**). Gypsum consists of little crystals that vary from small to coarse crystal grains. Also, gypsum is vastly available with shale and limestone in interstratified layers (**Testa and Lugli, 2000**).

It is essential to study gypsum rock's mechanical characteristics because of it dissolves rapidly, a hundred times faster than limestone and slower than halite by around a thousand times. When gypsum rock close to a river, it easily dissolves by a rate of 1.0 m per year due to 1.0 m/sec rate of water flow that across the face of it. Therefore, it poses a threat to any development that encounters it (**Sijiang et al., 2020**). Weak realization of the dissolution of gypsum rock might lead to safety troubles like collapse, roof fall, etc. Gypsum dissolution is hazardous for dams due to water can leak and cause dissolution in their foundations. The dissolution amount can be accelerated quickly; little primarily flows can rapidly be as main seepage, which may threaten the safety of the structures (**Salih, 2003; Salih, 2013; Gutiérrez et al., 2015; Salih et al., 2015; Al-Hadidi and AL-Maamori, 2019; Al-zubaidy et al., 2022).**

Gypsum dissolution issues can be affected by water supply. Extraction from gypsum strata rocks underground can make too much hard water, speed dissolution process, and cause extensive drop (**Sun et al., 2018**). Therefore, precautions are necessary to stop gypsum and associated layers pollution. This is due to the pollutants can be transmitted by the beds of gypsum as fast as river water. In addition, bad agricultural works can be in charge of severe problems in the creation of gypsum layer contamination (**Milanović et al., 2019**).

Various studies have been carried out such as a single sample of gypsum rock was subjected to incremental loading tests in natural and water-saturated states, and then the creep properties were evaluated due to water-saturated softening action (**Liu et al., 2020**). After that, the water molecules (in the atmosphere) were believed to be the main reason for the change of gypsum rock's physical and mechanical characteristics with time progress, and the relative humidity highly affected the mechanical characteristics (**Hoxha et al., 2006**). Then, the temperature increase can result in the salt solution concentration increase; the inundation time elongates and weakens the gypsum rocks' mechanical

strength (**Liang et al., 2012; Wei et al., 2020**). The microstructure changes of gypsum crystals in different salt solution concentrations are importantly influenced on its physical and mechanical characteristics (**Meng et al., 2018**).
The significance of this research reveals the simultaneous effects of coexisting reactions of various water types at the static conditions and atmospheric temperature on the solubility of gypsum rock. Dissolved ions concentration in the water category (deionized, tap, fresh, acid, well, and rain) were thoroughly considered in the experiments. Furthermore, evaluation of dissolution impact on the physical and mechanical characteristics of gypsum rocks was also assessed.

2. MATERIALS AND METHODS

2.1 Utilized Gypsum Rock

Aghjalar area (35.641149, 44.962890) locates in Sulaimaniyah governorate, northern Iraq (see **Fig. 1**) contains large gypsum rock layers, and therefore, large blocks of gypsum rock were collected from the gypsum mining areas there. The gypsum block samples were crushed and sieved through sieve 200μm mesh. To recognize the rock's chemical composition that formulated the rock structure, solid figureicles of rock were scanned under X-ray fluorescence (XRF) mechanism (Spectro IQ11). The results are illustrated in **Table1** showing that the SO^3, CaO, and SiO2 carry the largest component percent that is 48%, 46.8%, and 0.6% respectively.

Table 1. Chemical composition of the natural gypsum rock samples.

Chemical Element	Value (%)
SiO_2	0.6
CaO	46.8
SO_3	48
P	Nil
Phosphate	Nil

Figure 1. Agjalar area in Sulaianiyah Governorate, Iraq selected to collect gypsum rock blocks.

2.3 Experimental Work

The experiments were conducted using cubic (5.0cm x 5.0cm x 5.0cm) samples of gypsum rock. Five different types of water with one-liter volume in five containers were separately managed. Three cubes were immersed in each container after taking their weights and checking the salinity of solutions. The cubes were kept in the solutions for one week to know the nature of the reaction that takes place on the external boundary layer of rocks. The mass transfer of rock molecules into the bulk solutions undeniably depends on the environment and quality of solutions. At the end of the week, the cubes were taken out and weighed again after free water drying. The utilized gypsum rock blocks were examined to make certain whether undesired impurities were available or any weaknesses in the rock structure in terms of cracks if found (see **Table 3**).

2.3.1 Dissolution Procedure

Before starting the dissolution process, the gypsum cubes were fully saturated first considering a procedure recommended by the study of **Hawks and Mellor (1970)**. The samples were oven-dried (25°C), then to become evacuated, place the samples inside a vacuum desiccator for three hours. So, during the continuous evacuation process, distilled-water was inserted in the desiccator. Then, the inundated samples for 24 hours were extracted. After that, the scenario of the dissolution process was started. Every three cubes were put in a box filled with one liter of the mentioned water types in **Table 2**. One cycle of dissolution consists of one week, after that the samples were weighed and the water of saturation was replaced by a new fresh liter from same water type. This process has lasted for 15 weeks; then the cubes' dimensions and masses were measured.

Table 2. Chemical composition of the utilized water types.

Water Type	SiO_2	S	Ca	Pb
Normal Rain	116.6	2080	2318	Nil
Acidic	62.6	393.4	682.4	Nil
Tap	102.1	528.4	1087	Nil
Spring	43.5	416.3	683.7	0.12
Well (Rezgari)	134.5	12620	9362	0.2
Well (Tanjaro)	69.0	3479	3629	Nil

Table 3. Physical properties of the utilized gypsum rock.

Physical Properties of Gypsum	Description
Chemical Classification	Sulfate
Color	Clear, colorless, white, gray, yellow, red, brown
Streak	White
Luster	Vitreous, silky, sugary
Diaphaneity	Transparent to translucent
Cleavage	Perfect
Mohs Hardness	2
Specific Gravity	2.3
Diagnostic Properties	Cleavage, specific gravity, low hardness
Chemical Composition	Hydrous calcium sulfate, $CaSO_4{\cdot}2H_2O$
Crystal System	Monoclinic
Uses	Manufacture drywall, plaster, joint compound, and agricultural soil treatment

2.3.3 Compression Testing

Although the superstructure problems may be more significant with creep issues, short-term tests also can be a good indication and useful as well, which is helpful to evaluate the material's responses to quick loading. Uniaxial compression experiments were conducted on the intact and dissolve gypsum cubes. The results were utilized to evaluate gypsum rock's mechanical capability after long-term dissolution. Both standards, ASTM and ISRM, were considered to select the loading rate, which was 0.025 MPa/sec.

3. RESULTS AND DISCUSION

The most fundamental variable that directly impact the solubility of minerals in gypsum rocks is the pH value of water in which rocks are exposed to because when pH value is less than 7, chemical changes occurred to the molecular structure of rocks. Moreover, the water

molecules and carbon dioxide of atmosphere compile with each other to form carbonic acid (H^2CO^3), which finally breaks to hydrogen ions (H^+) and bicarbonate ions (HCO^{3-}**)** **(Bhateria and Jain, 2016)**. The results illustrated that gypsum rock dissolution by aqueous water solutions at the normal conditions of the laboratory increased slightly with a decrease in the pH of the solution.
Water may reach the inner layer of the gypsum rock when it seeps over fissures, factures, and further cavities in gypsum rock structure. Gypsum rock is weakened due to the subsequent reactions. Therefore, the continual inner reactions for a long period of time break down or loosen the bonds holding the rocks together causing dissolution of a bigger figure of the rock and leaves behind void spaces **(Luckner et al., 2017).** However, higher resistance of the rock will slow down the effectiveness of carbonic acid on the rock to reduce the power of the bonds that holding the rocks together.
The release of cation ions of Ca^{+2} from the gypsum rock into the bulk solution is counted as a measurement for the resolved gypsum because calcium molecules are the major component structure of the rock **(Farkas et al., 2018)**. So, the influence of pH solution is larger on dissolution rate within time progress. Gypsum rock dissolution outcomes by each type of water illustrates that the longer period of time rocks immersed in the solutions, the higher disintegration of the boundary layer of the rock exposes to leach will be **(Zaier et al., 2021)**.

3.1 Compressive Strength

Stress-strain relations of unconfined compression tests were presented for gypsum cubes that dissolved under different water types. **Fig. 2** shows the values of UCS (Uniaxial Compressive Strength) for all gypsum cubes by using different water types after 15 weeks of inundation. The largest UCS value is about 15.37 MPa in the intact cube after that the ultimate strength gradually decreases. For example, the UCS values is about 13.98 MPa, 13.46 MPa, 10.45 Mpa, 10.2 MPa, 10.13 MPa, 9.87 MPa, and 9.28 MPa in purified, spring, rain, tap, acidic, groundwater (Tanjero), and groundwater (Rezgari), respectively.
The mechanical characteristics of the tested cubes show different behaviors (see **Fig. 2**). Mostly the cubes showed very little capability at the begging of loading, which is seen in the recorded strains due to a very small loading magnitude. It may be due to the larger dissolution that happened on the surfaces, which made them quite weak to resist the axial loading for a while and then started to show some notable resistances. The dissolution significantly changed the mechanical behavior of the tested cubes. The higher dissolved cubes (by tap and well water) showed a brittle behavior (see **Figs. 2 and 3**). While the lower dissolved cubes (by rain, acidic, and purified water) showed a ductile behavior. It seems that some available ions might cause notable changes inside the gypsum rock structures, which resulted although in lower dissolution, while it caused higher reductions in the mechanical responses for the axial loading, the obtained results were consistent with the finding of **Fengxiang and Mingjiang (1983)**. The achieved outcomes were dissimilar with the results obtained in the study of **Mohammed and Salih (2018)**. Also, gypsum rocks compressive and tensile strength found to be decreased by soaking in water for 20 days **(Karacan and Yilmaz, 2000; Heidari et al., 2012; Salih et al., 2015).**

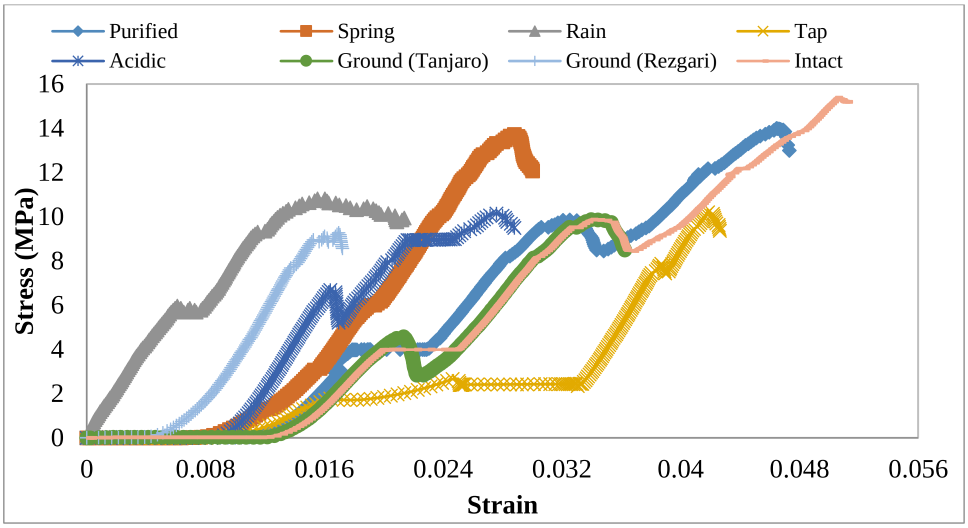

Figure 2. Comparison of different gypsum rock UCS dissolved first by different water types for 15 weeks inundation, and then tested by uniaxial compression machine.

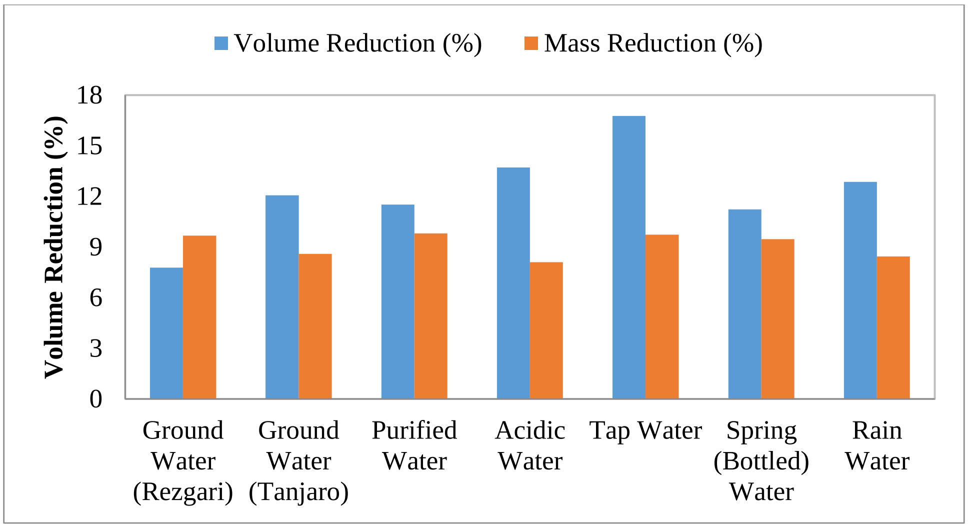

Figure 3. Comparison of different gypsum rock mass and volume reductions dissolved by different water types for 15 weeks inundation.

3.2 Macro and Microscopic Analysis

It is clearly visible that the cubes really have some impurities inside their structures (see **Fig. 5/Fig. B),** which means that these places might differ in their behaviors compare to gypsum salt subjecting to inundation and loading. In addition, the microscopic figureicle sizes of the utilized cubes are variable, which mainly starts from 203.78 nm and larger figureicles are in micro-meter sizes (see **Fig. 4**). Different sizes can cause different percentages of dissolution due to the availability of various gypsum surfaces for contacting inundation water.

Figs. C and D in **Fig. 4** show visibly and evidence that the microscopic figureicle sizes were decreased compared to the microscopic figureicle sizes of the intact gypsum samples shown in **Figs. A and B**. In general, the cube's surfaces are more exhibited to dissolution and especially the corners, which mean that the dissolution took place externally (see **Fig. 5/Fig. B**). This may belong to the interfere of the existed ions shown that are present in the quality of tap water to make ion exchange with the surface of the cubes, for instance, ion interchange (calcium ion with decrease in magnesium ion) indicates ion interchange on the surfaces of calcite rock **(Nowrouzi et al., 2019; Abbasi and Khamehchi, 2021)**. Furthermore, the amount of oxygen contents in tap water is adequate to enhance reaction on the surface of rocks where strength bond of molecules is not strong enough. Therefore, a thin figure from the boundary layer of the cubes can be disintegrated and transferred into the bulk water **(Manaka and Takeda, 2016).**

Fig. 5/Fig. B also revealed that some surface trenches were generated in places of impurities, which might be a figure of the mass reduction of the cubes and not exactly the dissolution of gypsum salt. In some way, the shapes of the microscopic figureicles also changed after inundation for 15 weeks, which is evidence of dissolution and shows that the dissolution starts externally and may penetrate, if possible, to the internal figure. The internal dissolution can largely play a great role in weakening the cubes capability to resist loading (see **Fig. 2**) as the external dissolution might do, while in smaller effect.

Similarly, the microscopic images of **Figs. E and D/Fig. 4** achieved the similar cubes states after dissolution by rain water. The rain water contains adequate amount of dissolved carbon dioxide which made its pH lowered causing a bit acid that has power to dissolute small figureicles easily. However, the floc figureicles require more acidity to disintegrate their molecules **(Bogan et al., 2009; Boyd, 2020).**

Fig. 5 presents the microscopic image (**Fig. A**) for the dissolved gypsum cubes after 15 weeks of inundation by acidic water. It is clearly noticed that the diameter of microscopic figureicle sizes resulted from the immersion of the cubes in acid water is smaller than in others as shown in **Fig. 4**. This confirmed that when acidic water interacts with the surface of gypsum rocks in the solutions, a chemical reaction happens. Clay and soluble salts are formed in the reaction which their diameter sizes are tiny. These dissolve in the water easily. Furthermore, the power of hydrogen concentration is helpful to reduce the figureicle size of disintegrated figureicles formed from the reactions **(Hurowitz et al., 2006).**

Fig. 4/Fig. B presents the dissolved gypsum cubes after 15 weeks of inundation by two types of well water from Rezgari and Tanjaro, Sulaimaniyah city, Northern Iraq. Similarly, the dissolution evidence is clearly visible in the dissolved gypsum cubes. The capability of the cubes to resist loading (**Fig. 2**) that dissolved by well waters is significant and much larger than the dissolution that happened due to other water types. This confirmed that the characteristics and chemical composition of well water in the tests is much more powerful to enhance the chemical reaction on the rock surface **(Bahraminejad et al., 2019).** The well waters caused the highest reductions in the values of UCS for the same inundation periods.

Similar to previous paragraphs, the dissolution evidence is clearly visible in the other dissolved gypsum cubes. The cubes capability to resist loading (see **Fig. 2**) that dissolved by de-ionized (purified) water is smallest one. The main reason is that deionized water is free from any ions or foreign materials that restrict diffusion of carbon dioxide from the atmosphere into water to escalate acidity of water environment that surrounds the rock **(Zheng et al., 2020).** The dissolution that happened by deionized (purified) water was significantly the highest compare to the dissolution values by the other water types, which is evidence that the availability of various ions in the water of inundation causes significant reductions in the mechanical capability of gypsum rock **(Salih, 2013; Salih et al., 2015; Salih and Mohammed, 2017; Mohammed and Salih, 2018).**

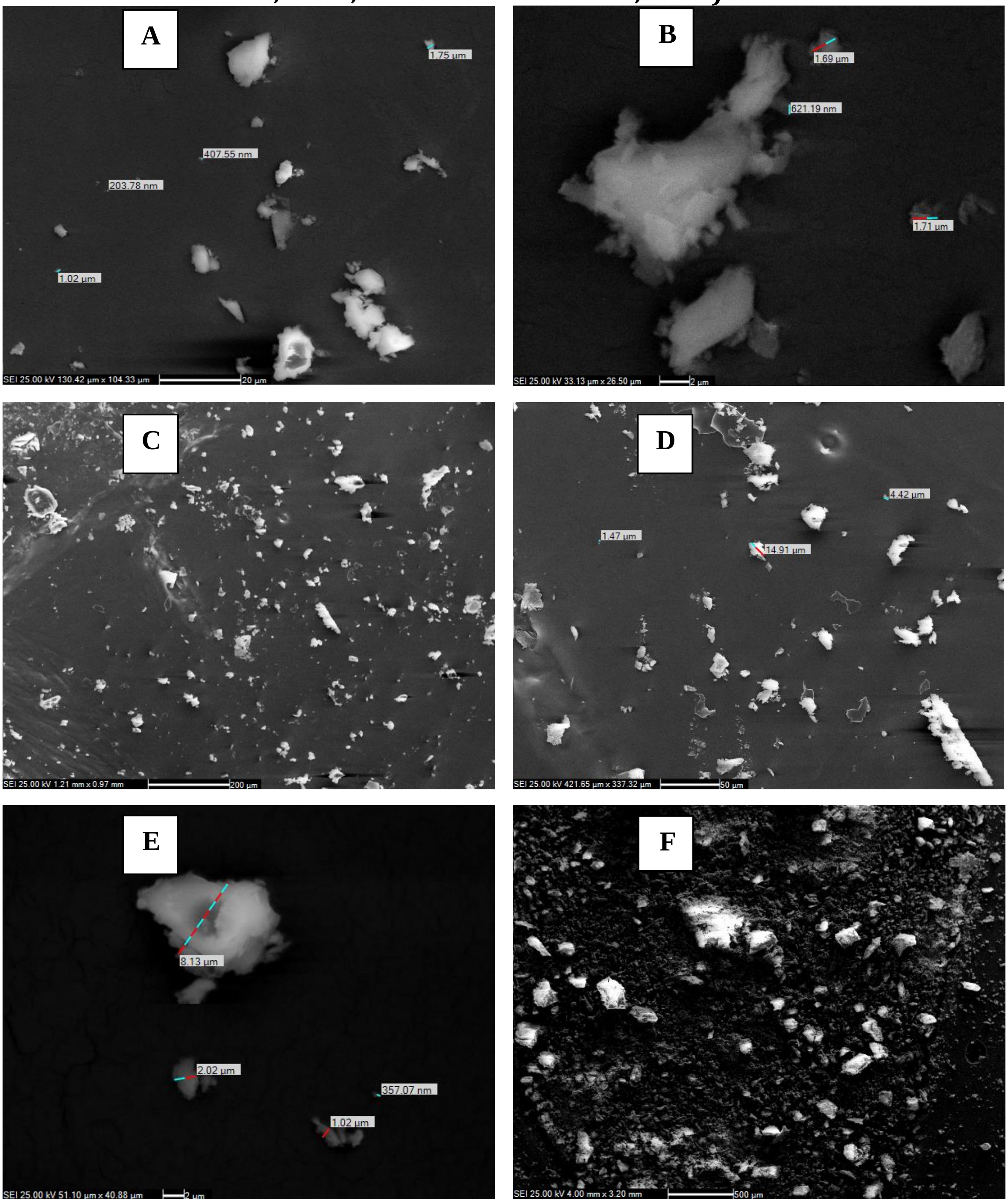

Figure 4. Microscopic images of the natural gypsum rock contain the available particles dimensions. In plates A and B, natural cubes are shown. The cubes that dissolved for 15

weeks inundation by tap water shown in C and D plates. The cubes that dissolved by rainwater shown in plates E and F.

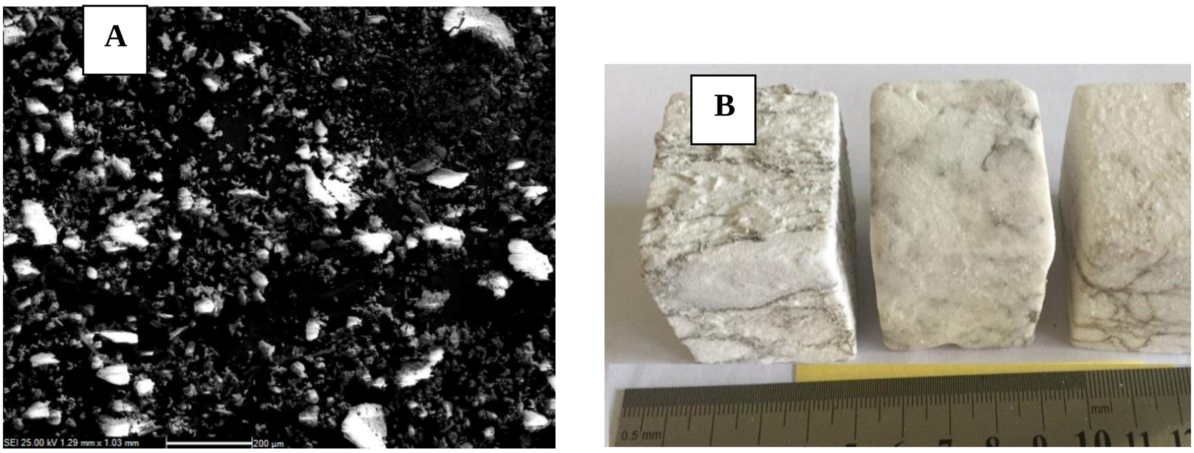

Figure 5. Microscopic image of gypsum rock dissolved by acidic water after 15 weeks inundation (plate A). Plate B presents the dissolved gypsum cubes due to 15 weeks inundation by well water.

4. CONCLUSIONS

The outcomes of this study show that the risk displayed by such rock is not only the development of large cavities. It is a gradual weakening of the rock mass itself. As conclusions, the following points can be drawn:

1. Experimental results show that the water type has a great role in dissolving gypsum salt. The water purity is not the only factor to cause that; the associated ions may either decrease or increase the progressive dissolution of gypsum salt.
2. Chemical ions associated with the water have an effective role in the mechanical behavior of gypsum rock, which can change the rock behavior from ductile to brittle.
3. Evidence shows that gypsum rock surfaces are more prone to dissolution. However, two more factors can be also responsible; associated ions in inundation water and existing surface cracks that can be filled with impurities.
4. The recorded strain of gypsum rock within the begging of loading is clearly related to the dissolution of the surfaces. The maximum resistance and final achieved strain were effectively related to the internal structure deterioration that happen in the rock samples.
5. The dissolution process utilizing various existing water types is significantly varied within the responses of gypsum rock. Tap water caused highest volume reduction (16.7%), while ground water of Rezgari caused the lowest volume reduction (7.8%). Purified water caused the highest mass reduction (9.8%), while acidic water caused the lowest mass reduction (8.1%).

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