



Saturation Procedures of Soluble Rocks (Emphasizing on Gypsum-Anhydrite Rocks)

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پذیرش مقاله: ۹۹/۰۱/۳۱

دریافت مقاله: ۹۸/۰۴/۲۰

Abstract

To identify natural behavior of rocks under the similar moisture conditions, two saturation methods of under atmospheric pressure conditions or vacuum pressure are usually used. In the method of vacuum pressure, the values of vacuum pressure P_{vac} as well as the time of applying this pressure T_{vac} are two effective parameters. In this study, the saturation method of rock, especially soluble ones such as gypsum and anhydrite, have been studied. The results of the experiments were analyzed in terms of sensitivity for four parameters of dry vacuum pressure $P_{vac(dry)}$, wet vacuum pressure $P_{vac(wet)}$ and the time of applying each one ($T_{vac(wet)}$, $T_{vac(dry)}$). The results showed that by increasing $P_{vac(wet)}$, the percentage of water absorption has been increased and the samples generally gain more than 95% of their increased weight after 4 days of applying wet vacuum and they can be practically assumed to be saturated. Applying dry vacuum $P_{vac(dry)}$ before wet vacuum $P_{vac(wet)}$, increases the weight of most samples after applying $P_{vac(wet)}$. In case of gypsum-anhydrite rocks, the experiment showed that applying dry pressure equivalent to -0.5 atmospheres (atm) and to 5 hours ($P_{vac(wet)} = -0.5 \text{ atm}$, $T_{vac(dry)} = 5\text{h}$) propose ideal results.

Keywords: *Wet and dry vacuum pressures, Sensitivity analysis, Rock moistur, Gachsaran formation*

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1. Introduction

The analysis of rock properties in the laboratory usually includes measuring engineering properties of rock under atmospheric conditions or any other conditions. Due to the variability of the natural moisture of the rocks in the engineering project sites and need to know the mechanical and strength properties of rocks in the certain stabilized conditions, civil and engineering tests of rocks are usually conducted in dry and saturated conditions, unless for environmental reasons, testing at a certain moisture level is desired. For drying rock samples, oven and for saturation, different methods such as submerging the sample in water in atmospheric condition or vacuum pressure are usually used. In atmospheric pressure conditions, no particular time has been proposed for different types of rocks and the application of this method in many of weak and soluble rocks can change them greatly (such as swelling, dissolution, slaking and physical erosion). As the result, the accuracy of the results is error-prone because of changing the physical properties of samples. In the method of vacuum pressure also, the value of vacuum pressure (P_{vac}) as well as the time of applying this pressure (T_{vac}) are two effective parameters.

In order to determine the water saturation and porosity of rock samples, some standards have been proposed. In the standard of ISRM (1977), to determine the volume of rock pores, vacuum saturation has been introduced to them. In this method, the rock sample is dried in the temperature of 105 ± 3 °C for 24 hours and for saturation, the pressure of less than 800 PAs has been recommended. To measure the porosity of concert in standard ASTM-C1585-13, 2013 and ASTM-C642, 2013, three sample saturation techniques of cool-water saturation (CWS), boiling-water saturation (BWS) and vacuum saturation (VAS) have been proposed. Water absorption experiment in atmospheric pressure or the cold saturation indicates that how much

weight the material gain after being immersed in water till saturation point (at least 48 hours) (as a percentage of dry weight) (BS EN 13755, 2008). Water immersion method is also used to determine the saturation coefficient, which determined as percentage of filling porous volume after 24 hours of immersion. Saturation coefficient is used as an experimental guide for durability of masonry constructional rocks (BRE Digest 420, 1997; Ross and Butlin, 1989).

In the application of the vacuum saturation method, there are two different approaches in the experiments; a) using dry vacuum pressure or $P_{vac(dry)}$ on the sample before immersing it in water, b) using wet vacuum pressure or $P_{vac(wet)}$ while immersing a sample in water. In the case of interfering time in the two above-mentioned factors, the two parameters of T_{vac} $T_{vac(dry)}$ and $T_{vac(wet)}$ will also be important in performing any researches through vacuum saturation method. To predict the durability of freezing-wetting of stabilized materials, Dempsey and Thompson (1973) used $P_{vac(dry)} = 24$ in Hg (about 0.8 atm) and $T_{vac(dry)} = 30$ min for vacuum saturation of samples. The reason of using $P_{vac(dry)}$ was announced, reducing the pressure in stabilizing soil samples as much as possible. Then, they immersed the samples in water for 1 hour. Prout and Hoff (1991) investigated the variations of absorbed water based on $P_{vac(dry)}$ and $T_{vac(dry)}$ and $T_{vac(wet)}$ with $P_{vac(wet)} = 1$ atm for clay bricks. He considered the water content of the samples after 5 hours of pumping and 5 days of immersion in water, as a full saturation nominal value, and found that 5 hours of using the vacuum pump and 10 minutes immersion in water or 6 minutes of pumping and then immersion for 15 minutes leads to complete saturation of the samples. He also mentioned that using very long times of immersion (~2 months) only leads to a partial increase in the mass of bricks. This increase for high porous bricks was less than 0.25% and for low porosity bricks less than 1%.

In the proposed paper by Wilson et al. (1999), in case of vacuum saturation porosity, drying the samples in 105°C, the application of $P_{vac(dry)} = 0.1$ mm Hg (0.03 atm) and $P_{vac(wet)} = 1$ atm on samples has been mentioned. In their paper, $T_{vac(dry)}$ and $T_{vac(wet)}$ aren't particularly mentioned. In RILEM (1994b), in case of absorbing water and porosity of masonry materials, the time of pumping $T_{vac(dry)}$ is unclear, but it is proposed that $T_{vac(wet)} = 1$ hour. In methods of testing saturation using vacuum pressure for natural rocks in RILEM (1994a), applying $T_{vac(dry)} = 3$ hours and $T_{vac(wet)} = 3$ hours and finally 21 hours of immersion in atmospheric pressure conditions has been predicted. In RILEM (1994c), for water absorption in concrete with immersion in vacuum condition, $T_{vac(dry)} = 24$ hours and after 2 hours of immersion in vacuum and then 24 hours of immersion in atmospheric pressure have been considered. In this method, till reaching fixed weight, the material is kept immersed in water. Three recommended methods of ASTM C1585-13 have been compared by Safiuddin and Hearn (2005). The experimental results of this study showed that the vacuum saturation method is generally more efficient than cool-water saturation or boiling-water saturation. Also in the experiment of water absorption through boiling, with 5 hours of boiling, almost 80 to 90% of vacuum saturation can be achieved in the samples (Wilson et al., 1999). In the study, conducted by Li et al. (2015) for determination of concrete porosity using the vacuum saturation technique, temperature of 105°C have been also used for drying samples and $T_{vac(dry)} = 2$ hours, $P_{vac(wet)} = 1$ atm and $T_{vac(wet)} = 24$ hours. Due to low permeability and very low velocity of water in gypsum rocks, reaching water to the center of a rocky core with NX dimensions requires relatively large time. Additionally, Due to the high solubility of such rocks and sometimes their swelling potential, the samples can be deformed a lot during the saturation time. As the result, the ultimate saturated weight that is obtained, because of volume change of the sample, won't be exactly corresponding with

a un-deformed or original sample. So water absorption technique in atmospheric conditions cannot be used reliably in these rocks till reaching a fixed weight.

The conducted studies about saturating evaporative rocks, especially gypsum and anhydrite rocks are very limited. For example, Ali (1979) has mentioned using the method of Hawkes and Mellor (1970). In this method, the samples are first dried in the oven for 24 hours in 70°C (the temperature announced that the gypsum chemical composition isn't changed). Then the samples are saturated applying $P_{vac(dry)} = 0.001$ mm Hg, $T_{vac(dry)} = 3$ hours, $P_{vac(wet)} = 0.001$ mm Hg and $T_{vac(dry)} = 24$ hours. In that study, 100% of samples saturation has been mentioned during 24 hours, but there aren't any confirmatory results for that. This method has been also used by the other researchers such as Mann and Fatt (1960) and Boozer et al. (1962). The major issues with this technique is heating the sample primarily to 70°C, may lead to change of gypsum to hemihydrate.

In order to investigate the solubility and creeping behavior of gypsum-anhydrite rocks of Gachsaran Formation in Iran, four under construction, reservoir dam sites in Iran were visited and several rock block samples were taken. Since, after the end of construction of the dam and impounding of the reservoir, the foundation and abutments will be in the saturation condition, therefore, one of the important conditions in which the mechanical, strength and behavioral properties of the rocks must be determined is saturation conditions. A review of previous literature showed that there aren't many studies or reports about the manner of weak and soluble rocks saturation, even in ASTM and ISRM standards. Therefore, the aim of this study is to investigate the manner of saturating the rocks, especially in case of weak and the soluble ones such as gypsum-anhydrite rocks.

2. Experimental Equations and Calculations

Various phrases are proposed for water content and water saturation in rock samples. Water content (W) or moisture content is defined as the ratio of the mass of water in a sample to the mass of solids in the sample, expressed as a percentage (ASTM D2216, 2019; BRE Digest 420, 1997; Yuen, 2015):

$$W = M_w / M_s \quad (1)$$

Where, M_w is the mass of pore water and M_s is the mass of grains. "Water absorption" (A_w), also known as "absorption" or "total water absorption", is given in ASTM C127-01 (2001) as Eq. 2:

$$A_w = (M_{sat} - M_{dry}) / M_{dry} \times 100 \quad (2)$$

Where, M_{sat} is the mass of saturated-surface-dried test sample in air and M_{dry} is the mass of oven-dry test sample in air. As it was mentioned before, sample saturation is usually done through two atmospheric pressure and vacuum pressure condition technique. Water absorption under atmospheric pressure condition (W_{atm}) has been also called as "freely or unforced water absorption" by Siegesmund and Török (2011). "Water absorption under atmospheric pressure condition" (W_{atm}) is known as the ratio of "freely or unforced water absorption" to the dry mass of the sample as Eq. 3:

$$W_{atm} = (M_{wet} - M_{dry}) / M_{dry} \times 100\% \quad (3)$$

Where, M_{wet} is the mass of the sample immersed in water for 48 hours, and M_{dry} is the mass of the sample. "Water absorbed under vacuum conditions" (W_{vac}), also known as "forced water absorption", has been proposed by Siegesmund and Török (2011) as Eq. 4:

$$W_{vac} = (M_n - M_t) / M_t \quad (4)$$

Where, M_n is the mass of the sample after water forced absorption and M_t is the dry mass of the sample. Degree of saturation has been proposed as Eq. 5 in ASTM - C642 (2013):

$$S_r = V_w / V_v \times 100\% \quad (5)$$

Where, V_w is the volume of the pore water and V_v is the volume of the pores. Saturation coefficient (S_r) has been also stated as the ratio of freely water absorption to force water absorption in percentage (Siegesmund and Török, 2011):

$$S_r = W_{atm} / W_{vac} \times 100\% \quad (6)$$

In Eq. 6, W_{atm} is the water absorption under atmospheric pressure and W_{vac} is the water absorption under vacuum conditions. This equation shows that in the best conditions and spending required time and getting the value of W_{atm} to the value of W_{vac} , the value will be equal to 100%. Based on the above definitions, degree of saturation (S_r), water absorption by weight (A_w), dry density (ρ_{dry}) and effective porosity (ϕ_e) can be related by the following equations:

$$A_w = \rho_w \phi_e / \rho_{dry} \quad (7)$$

$$S_r = W / A_w \quad (8)$$

If the rock is assumed to be fully saturated, i.e. $S_r = 100\%$. By filling all available voids of rock sample, the value of water in the sample can be reached to maximum possible value, $Max(w)$. $Max(w)$ is equivalent to water absorption by weight (A_w), as shown in Eq. 2:

$$Max(w) = A_w \quad (9)$$

$$Max(\rho_{bulk}) = \rho_{sat} = (1 + A_w) \rho_{dry} \quad (10)$$

Therefore, effective porosity ϕ_e can be calculated from Eq. 7. Accordingly, as the value of parameters such as M_{dry} and M_{sat} can be calculated through more reliable methods, rock physical and engineering properties can be estimated with better accuracy.

3. Materials and Methods

3.1. Petrography

For samples petrography, three procedures include microscopic of thin sections, XRD and calcimetry have been used. The results of

all three methods were used for choosing the minerals consisting of rocks and their percentages (Table 1). As it is seen in this Table 1, various types of sulfate rock A to K with different mineral composition were used for this study.

The most important minerals, consisting of the studied sample, mentioned in Table 1, include gypsum, anhydrite, calcite, and clay type minerals in the matrix or vein, and sometimes silica mineral. In the determination of minerals percentage, a combination of the results of the mineral frequency analysis through the RIR method of X-Powder software as well as accounting in microscopic sections and calcimetry results were used. The mineralogical composition of the used samples is varied from gypsum close to pure (Type E) to the sample, having more than 80% anhydrite (K), the samples, having about 30% clay (Type D). Also, in Table 1, physical characteristics, including porosity, dry and saturation unit weight and water absorption are presented.

3.2. Sampling and preparing the samples

To study about the effect of $P_{vac(dry)}$, $P_{vac(wet)}$, $T_{vac(dry)}$ and $T_{vac(wet)}$ on the gypsum-anhydrite samples saturation, the rock blocks of Formation outcrops were used. After transferring the blocks to the Engineering Geology and Geotechnical Laboratory of Bu-Ali Sina University, they were cored with the dimension of NX (~54.7 mm). To dry the samples, they were kept in the oven at the temperature of $45 \pm 2^\circ\text{C}$ until drying (fixing weight or achieving a maximum of 0.01 weight loss per day). After drying the samples, the related experiments in this study were conducted on 111 rock core samples. During the experiments, the variations in the weight of the specimens were measured at 4, 8, 12, 24, 48 and 72, 96 hours, and for a number of specimens up to 196 hours. To saturate the samples, ionized distilled water with zero electrical conductivity (Con) and pH of about 5.8 was used. The purpose of this study is to investigate the optimal use of vacuum saturation methods for saturating evaporation rocks with a focus on gypsum and anhydrite rocks, and saturation sensitivity to the four mentioned effective factors.

Table 1. Mineral composition and physical properties of studied sulfate rocks

Rock group	Sampling site code	Rock block code*	Tested sample code**	Sum of tested samples	n(%)	γ_{dry} (gr/cm ³)	γ_{sat} (gr/cm ³)	$A_w(vac)$ (%)	ϕ	Mineral composition (%)				
										Gypsum	Anhydrite	Calcite	Clay	Other minerals
A	KA1	KA1-m	KA1-m-n	15	0.80	2.29	2.30	0.35	0.80	88	4	3	3	2
B	KG7	KG7-m	KG7-m-n	18	2.75	2.26	2.28	1.22	1.75	61.1	3	18.5	15.8	1.6
C	KG9	KG9-m	KG9-m-n	10	2.57	2.25	2.3	1.14	2.57	59.8	2	18.4	19.8	0
D	KG13	KG13-m	KG13-m-n	6	5.37	2.65	2.7	2.03	5.37	53.8	2.8	9.6	29.6	4.2
E	MM1	MM1-m	MM1-m-n	5	2.07	2.32	2.34	0.89	2.07	93	1	0.3	5.7	0
F	MM2	MM2-m	MM2-m-n	7	1.01	2.27	2.28	0.45	1.02	77	2	2.1	18.9	0
G	MG4	MG4-m	MG4-m-n	6	1.49	2.28	2.3	0.66	1.49	55.6	30	12	2.4	0
H	MG5	MG5-m	MG5-m-n	11	2.44	2.64	2.67	0.92	2.44	14	66.7	12.4	5.9	1
I	MG6	MG6-m	MG6-m-n	13	3.28	2.47	2.5	1.34	3.28	13.4	68.8	11	6.8	0
J	MG11	MG11-m	MG11-m-n	17	1.56	2.63	2.65	0.59	1.56	6.2	78.95	8.7	2.95	3.2
K	KG10	KG10-m	KG10-m-n	3	2.56	2.72	2.74	0.77	2.06	2.4	85.1	7.2	2.1	0.2

* For example: KA1-2

** For example: KA1-2-4

3.3. Research methodology

To investigate the effective factors on using methods by other researchers for samples

saturation, the sensitivity of samples saturation was investigated against following factors:

1. The value of $P_{vac(wet)}$ and $T_{vac(wet)}$

2. The value of $P_{vac(dry)}$ and $T_{vac(dry)}$

Because the parameter of $P_{vac(wet)}$ seems to be the most important parameter in sample saturation, so sensitivity against $P_{vac(wet)}$ is first measured and then after determining the best value of $P_{vac(wet)}$, related experiments to sensitivity against $P_{vac(dry)}$ for this value of $P_{vac(wet)}$ were conducted.

3.3.1. The sensitivity analysis of the sample weight to $P_{vac(wet)}$ and $T_{vac(wet)}$

To investigate the effects of $P_{vac(wet)}$ and $T_{vac(wet)}$ on the weight gain of the specimens or in fact the saturation of the specimens, from 6 pressure levels of -0.1, -0.2, -0.3, -0.4, -0.5, -0.6 atmosphere was used. To carry out the saturation experiments, two glass desiccator, a vacuum pump and a negative pressure gauge was used.

For this purpose, 6 types of different gypsum-anhydride rocks, including 52 NX-sized cores were used to measure the sensitivity of water absorption against mentioned dry and wet vacuum pressures. The samples were first weighed 0.01 g and dried in an oven at 45 ± 2 ° C until they reached a constant weight or a maximum weight loss of 0.01 g/day. Drying samples in this way sometimes took about 1 to 12 months. In

order to select the appropriate temperature for drying of sulfate rocks or gypsum bearing soils in the laboratory, it is first necessary to pay attention to the equilibrium temperature between the gypsum and anhydride ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O} = \text{CaSO}_4 + 2\text{H}_2\text{O}$). This temperature by Van't Hoff et al. (1903), between 63.5 ° C and 66 ° C, Ramsdal and Partridge (1929), Hill (1937) between 38 ° C and 42 ° C, Kelly et al. (1941) 40 ° C, Hardie (1964), 58 ± 2 ° C, Kelly (1960) and Hamad (1985), 46 ° C were determined. But in ASTM D 2216 (1999), since materials containing gypsum (calcium sulfate dihydrate with chemical formula $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) or other compounds containing significant amounts of hydrated water may be slowly

dehydrated at standard drying temperatures (100 ± 3 ° C) and will be converted to a new compound such as calcium sulfate hemihydrate ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$), it is recommended that such materials be dried at 60 ° C and or ins a desiccator at room temperature. However, the ASTM C472-99 (2009) standard specifies 45 ± 3 ° C for evaporation of gypsum free water and the temperature required to determine free water in standards such as ASTM C 471M-01(2001), or IS 1288 (1982) in India, as well as 45 ± 3 ° C. Therefore, in order to avoid the conversion of gypsum to anhydride, 45 ± 2 ° C was used in this study.

The specimens were then placed inside the desiccator, and after adding ionized water and full immersion of all samples, a vacuum pump was used to create vacuum pressure. To study how water absorbs by samples and the process of weight variation of samples, they weighed at times of 2, 3, 4, 5, 6, 7, 8, 12, 24, 48, and 72 hours, and in the case of a vacuum pressure test of -0.5 atm, up to 336 hours. In each step sample weighting step, a conductivity meter was used and the variations of electric conductivity, total dissolved solids (TDS) and salinity (Sal) of water were measured. To maintain the accuracy of the tests, the electrical balance was calibrated at each stage of the weighing of the samples, and the conductivity meter was also calibrated using buffer solution 1413. The bubble exit from the specimens was visible at the maximum at the first and second hours of beginning the experiments.

During the saturation process, three different phenomena, physical erosion of the body of the samples with the separation of particles from them, dissolution of water soluble minerals (in the case of these specimens, gypsum and anhydrite minerals), water absorption by the sample occurs. So, the weight variations of each sample at any given time would be due to the weight gain of the sample by absorption of water ($W_{absorption}$) and the weight loss of the sample by dissolution ($W_{solution}$) and the weight of the particles

separated from the sample ($W_{segregation}$). Therefore, the sample weight at each instant of the test (W_t), as well as its final weight, will follow Eq. 11:

$$W_t = W_{initial} + W_{absorption} - W_{solution} - W_{segregation} \quad (11)$$

To obtain the precise value of $W_{absorption}$, the weight of segregated particles of sample body and the weight of solving minerals in water should be measured independently.

Determining each of mentioned weights individually during each of the measurement steps involves removing the specimens from the desiccator, drying and then weighing them complete drying of the sample and its achievement to a constant weight or maximum of 0.01 g of weight loss per day and returning it to the initial dry conditions, require 3 and sometimes up to 12 months. In this case, carrying out a wet saturation test, it will take many years, which is practically impossible during this research due to time constraints, and usually does not have such an opportunity in practical work. Therefore, in the testing process, it was only possible to determine W_t and analyze the results on the basis of it in the short time.

3.3.2. The Sensitivity analysis of the sample weight to $T_{vac(dry)}$

As it was mentioned, in the method of Hawkes and Mellor (1970), a dry vacuum pressure of 0.001 mm of mercury is recommended to be used at least 3 hours before applying wet vacuum pressure, but for this level of pressure and time they did not provide any reasons. Such information gap can be also seen in many other related studies to other materials. The reason for recommending such initial dry vacuum pressure can be considered as creating an initial negative pressure inside the sample and the ease of water absorption in wet vacuum phase. In this sensitivity analysis, two effective parameters are the values of dry vacuum pressure ($P_{vac(dry)}$) and duration of dry vacuum ($T_{vac(dry)}$). Because in sensitivity analysis to wet vacuum pressure ($P_{vac(wet)}$), it

to obtain W_t , filtering the deposits, deposited on the bottom of the desiccator to measure the value of $W_{segregation}$, determination of the weight of existing salts in water $W_{solution}$ through different methods such as using saturation index curves or evaporating and weighting remained particles at 45 ± 2 ° C. The most important deterrent factor in separating the above mentioned weights, is the time required for drying the rock core samples.

As stated, with regard to the permitted temperature of 45 ± 2 ° C used for oven drying the specimens, each stage of the was seen that $P_{vac(dry)} = -0.5$ atm has the best result so in this sensitivity measurement, $P_{vac(dry)} = -0.5$ atm was considered as fixed parameter and ($T_{vac(dry)}$) as variable parameters. To investigate the effect of $T_{vac(dry)}$ on increasing the weight of the sample or their saturation degree, 4 groups of rock cores, consisting 7 types of gypsum-anhydrite (Types A to G) and total 59 samples were used. To measure the sensitivity of sample weight increase to $T_{vac(dry)}$, the time steps of 0, 5, 10 and 15 hours were used. Since the samples were used for this study were gradually dried and prepared for experiments, so the distribution of samples in different groups for different rock types wasn't equal, so that the most number of sample were tested for $T_{vac(dry)} = 15$ h (17 samples) and the least one for $T_{vac(dry)} = 0$ h (9 samples). Therefore, the mean of results was used in the rock groups for the number of each rock type. In each phase, after finishing the time of applying dry vacuum, ionized water were entered into desiccator and same as paragraph 5.1., wet vacuum pressure was applied and the weight of the samples was measured in the times of 4, 8, 12, 24 and 48 hours after getting their body moisture by a soft cloth. The aim of this phase was only to investigate the effectiveness of $T_{vac(dry)}$ on increasing the weight of samples.

4. Resultes and Discussion

4.1. The result of sensitivity analysis of the sample weight to $P_{vac(wet)}$ and $T_{vac(wet)}$

In columns 2 and 3 of Table 2, the results of sensitivity analysis of sample weight gain (W_t) after 24 hours of applying $P_{vac(wet)}$ on the samples obtained from rock blocks (column 1), for $P_{vac(wet)} = -0.2$ atm to $P_{vac(wet)} = -0.5$ atm is presented. The average digits of column 2 (-0.375) show that the weight increase of was obtained at a maximum vacuum of -0.5 atmospheres relative to the specific surface. Such result shows the direct relationship between the sample weight gain (W_t) with $P_{vac(wet)}$. Due to direct relationship between $P_{vac(wet)}$ and increasing of sample weights, the sensitivity of sample weight gain for $P_{vac(wet)} = -0.5$ atm was done in a prolonged time (336 hours). In Fig. 1, the realization of such a process for different tested samples under

samples is generally more in more vacuum pressures. Moreover, if we divide the percentage of weight gain of the samples to the surface area of the samples, the percentage of weight gain of the sample is calculated relative to the specific surface area (increase per square centimeter of the sample surface), except for the blocks of MG11-7. In all tested specimens, the maximum percentage of weight gain of the specimens

different vacuum pressures is shown for rock type I. In Table 3, the results of the tested samples at this wet vacuum pressure are presented for testing duration of 336 hours (14 days) including initial dry weight of each sample W_o and moment weight of sample in different time intervals (W_t), temperature variations, acidity (pH), electrical conductivity, salinity, total dissolved solids during experiments.

Table 2. Maximum sample weight gain percentage per specific surface area at different wet vacuum pressures

Rock Block (1)	Max increase in sample weight (%)		Increase per 1 cm ² surface area %	
	Pressure (atm) (2)	Increase in sample weight (%) (3)	Pressure (atm) (4)	$W_{Increase} / S$ (%) (5)
MG4-9	-0.2	1.17	-0.5	0.0133
MG5-6	-0.5	1.35	-0.5	0.0311
MG6-7	-0.2	0.42	-0.5	0.0050
MG6-8	-0.5	0.71	-0.5	0.0028
MG11-2	-0.4	0.53	-0.4	0.0019
MG11-4	-0.5	0.72	-0.5	0.0026
MG11-7	-0.2	1.29	-0.2	0.0048
KG9-6	-0.5	1.08	-0.5	0.0040
Average	-0.375	0.81	-0.45	0.0082

Table 4 shows that in wet vacuum pressure of -0.5 atm, reaching to $W_{Max(increase)}$ usually requires a time of almost 192 hours (8 days) to 288 hours (12 days) for different samples tested in $P_{vac(wet)} = -0.5$. At this pressure, an average of about 78%, 82%, 95% of the “samples’ weight gain” occurred after 24, 48, 96 hours, respectively. Therefore, the samples were able to obtain about 95% of their maximum gain weight up to 96 hours at a vacuum pressure of -0.5 atm during the studied period (14 days). After reaching the maximum weight, the samples start to lose weight if the experiment continues. In the

equilibrium proposed in Eq. 11, by increasing the time and saturation of the solution, the value of $W_{solution}$ is reduced and tends to zero gradually. By saturating the sample as well as filling its pores, $W_{absorption}$ gradually reaches to its maximum value and gets fixed. Thus, the only factor that can lead to weight loss is the sample, is increasing the value of $W_{segregation}$ due to increasing the physical surface erosion of sample body and, consequently, separating the particles from its outer surfaces and depositing on the bottom of the desiccator

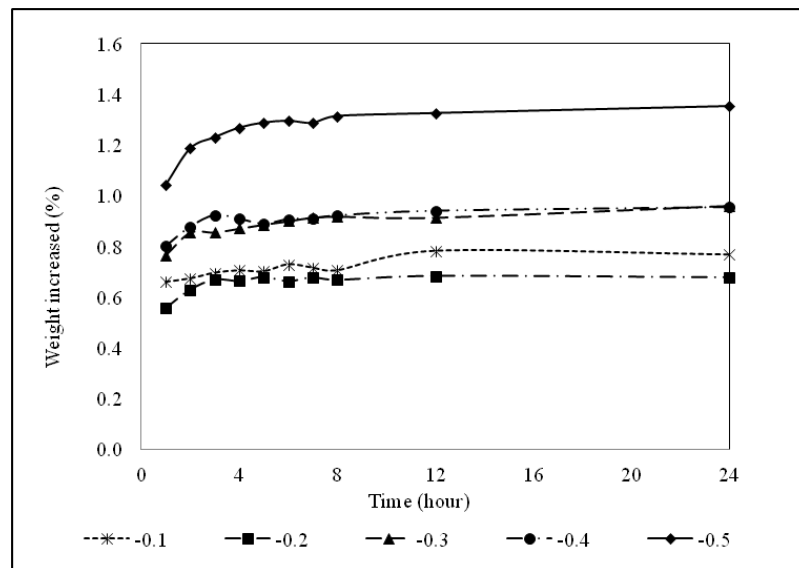


Fig. 1. Time variation graphs for the weight gain percentage of MG5-6 rock block samples at different wet vacuum pressures

In the wet vacuum pressure, by increasing the time, the amount of sediments deposited on the reservoir floor is increased and by increasing the value of $P_{vac(wet)}$, the amount of sediments is increased. This means that there is a direct relationship between the weight of accumulated sediments on the reservoir floor or $W_{segregation}$, and $P_{vac(wet)}$ and $T_{vac(wet)}$ and so by increasing the time from maximum threshold boarder W_{Max} (increase) and after that, continuing particles segregation, the sample loses more weight. Finding a value of $T_{vac(wet)}$, being able to lead the sample weight to its maximum value or in other word cause maximum water absorption in the sample or the percentage of saturation near 100%, it will be a very important subject in saturating soluble samples for experiments in the conditions close to reality of saturation in nature.

To obtain the value of the total weight of $W_{segregation}$ and $W_{solution}$, after ending each one of conducted experiments with different vacuum pressures, after taking the samples out of desiccator, the remaining water and sediments were poured into the dishes and then kept in the oven until dry at $45 \pm 2^\circ C$. With evaporation of water and the arrival of solid sediment weight with a constant weight

(about 1 month), their weight (W_s) was recorded. Since the weight of obtaining sediment has been the result of solubility and particle segregating of the total used samples surface areas (S_{total}) in each experiment of measuring sensitivity (8 cores in each experiment) to vacuum pressure, so, in order to obtain an idea of the effect of the specific surface area of the samples, the weight of the samples of each experiment was divided into the area of samples and the amount of grams per unit area of total $W_{segregation}$ and $W_{solution}$ was estimated (Table 5).

Since the amount of physical erosion, especially by increasing the experimental time, seems to play a more prominent role in reservoir sediment production than solution, so by increasing the time, a significant part of the sediment will be due to $W_{segregation}$. Figure 2 shows that after 24 hours, the weight of sediment accumulated in the desiccant floor has a relatively linear relationship with used vacuum pressure and by increasing vacuum pressure, the physical erosion of the sample body becomes more severe ($R^2 = 0.96$). Additionally, increasing the amount of sediment weight up to approximately three for vacuum pressure experiment of -0.5 atm (0.006474 g/cm^2 of sample surface area) after 14 days (13.94 g), shows that value of

sediment weight increases by increasing the time of vacuum pressure and has a direct relationship with each other. By drawing the variations of pH, Con, Sal, TDS vs time (Fig. 3), as it was mentioned before, it is observed that the rate of increase of these factors decreases over time and after about 90 to 120 hours the trend is almost linear and reaches relative stability. This means that from this time on, the weight increase of sediments is mainly influenced by the surface physical erosion of the samples, while before it was the result of total dissolution and physical erosion.

4.2. The result of sensitivity analysis of the sample weight to $T_{vac(dry)}$

The results of the weight measurements of the samples as percentages of their weight gain per cm^2 of the exterior area of the samples

after the first 24 hours of the experiment are presented in Fig 4.

The following general results are extracted from the Fig. 4:

- In all the gypsum sample types, the weight of the specimens in the condition of $P_{vac(dry)} = -0.5$ atm and $T_{vac(dry)} = 5h$, was greater than the weight of the samples in the $P_{vac(dry)} = 0$ atm, $T_{vac(dry)} = 5h$ test conditions.
- In 5 types of tested specimens (including A, B, D, E, and G), the weight gain of samples in $P_{vac(dry)} = -0.5$ atm and $T_{vac(dry)} = 10h$ condition was less than $P_{vac(dry)} = -0.5$ atm, $T_{vac(dry)} = 5h$ condition and only in two types of rocks (C, F), the sample weight has increased slightly.
- In most of experimented rock types, the magnitude of the weight gain of samples in $P_{vac(dry)} = -0.5$ atm, $T_{vac(dry)} = 15h$ condition was

Table 3. Sensitivity analysis experiments results in weight gain of specimens at $P_{vac(wet)} = -0.5$ atm

Sample No	Time (hour)										
	0	1	2	3	4	5	6	7	8	12	24
MG4-9-7	818.97	822.78	823.03	823.23	823.28	823.43	823.34	823.24	823.4	823.48	823.76
MG5-6-7	699.66	706.97	707.97	708.28	708.54	708.68	708.73	708.67	708.86	708.94	709.13
MG6-7-8	848.54	849.91	850.01	850.07	850.05	850.13	850.14	850.07	850.04	850.24	850.48
MG6-8-7	726.67	729.8	730.81	731.25	731.55	731.62	731.72	731.73	731.64	731.81	731.86
MG11-2-3	834.18	836.71	836.91	836.97	836.98	837	836.91	836.94	836.83	836.92	836.98
MG11-5-2	859.56	864.1	864.8	865.05	865.14	865.39	865.37	865.44	865.42	865.59	865.71
MG11-7-5	789.42	792.12	792.68	792.87	792.94	792.99	793.06	793.14	793.09	793.16	793.28
KG9-6-7	836.78	843.15	844.46	845.14	845.39	845.45	845.58	845.58	845.59	845.61	845.78
Temp	23.8	23.8	23.4	22.7	22.5	22.5	22.5	22.5	22.5	22.5	22.3
pH	5.7	6.8	7.1	7.2	7.2	7.2	7.1	7.1	7.1	7.1	7.1
Con (μs)	0.01	512	732	900	1068	1132	1259	1330	1381	1538	1828
Sal (ppm)	0.01	255	366	450	534	567	630	667	692	765	907
TDS (ppm)	0.01	339	488	600	711	756	838	886	932	1023	1215
Sample No	Time (hour)										
	48	96	120	144	192	216	240	288	312	336	$W_{Submerge}$
MG4-9-7	824.06	824.82	824.66	824.82	824.9	824.94	824.97	825.04	824.93	824.9	519.7
MG5-6-7	709.36	710.47	710.22	710.29	710.63	710.42	710.53	710.76	710.72	710.52	428.43
MG6-7-8	850.89	852.35	852.29	852.43	852.87	852.85	852.97	853.12	853.1	852.89	545.79
MG6-8-7	731.89	732.71	732.48	732.42	732.78	732.62	732.36	732.86	732.73	732.71	448.98
MG11-2-3	837.05	837.65	837.51	837.59	837.52	837.81	837.81	837.76	837.56	837.77	532.39
MG11-5-2	865.91	866.42	866.35	866.35	866.46	866.45	866.54	866.62	866.52	866.37	549.88
MG11-7-5	793.53	794.07	793.87	793.98	794.08	794.1	794.23	794.12	794.04	794.01	497.37
KG9-6-7	845.82	846.28	846.12	846.11	846.28	846.14	846.18	846.12	845.98	845.99	546.45
Temp	22.2	20.8	22.1	20.7	19	20.2	20.2	20.1	20.1	20.5	
pH	7.1	7.1	7.1	7.1	7.3	7.3	7.3	7.3	7.1	7.1	
Con (μs)	2170	2390	2450	2450	2530	2620	2560	2600	2630	2630	-
Sal (ppm)	1090	1190	1220	1220	1260	1310	1300	1300	1310	1310	
TDS (ppm)	1450	1590	1630	1630	1690	1750	1730	1730	1750	1750	

was greater than that of $P_{vac(dry)} = -0.5$ atm, $T_{vac(dry)} = 10$ h, but in comparison with $P_{vac(dry)} = -0.5$ atm, $T_{vac(dry)} = 5$ h, the changes have been both as a little increase or decrease. Generally, the application of initial dry vacuum pressure ($P_{vac(dry)}$) has a positive effect on the saturation of the studied rock samples and increases the more weight in the sample compared to when it isn't applied. Also, $T_{vac(dry)} = 5$ h was an appropriate, effective time for increasing the weight of samples better (meaning more water absorption).

5. Conclusion

In conducting research on how the factors affecting the saturation of samples through vacuum saturation method, the effect of 4 effective parameters on the results, include dry vacuum pressure $P_{vac(dry)}$, wet vacuum pressure $P_{vac(wet)}$, and the duration of applying each one ($T_{vac(dry)}$ and $T_{vac(wet)}$) on 111

gypsum-anhydrite rock core samples were measured in terms of sensitivity. The aim of this study has been to find the best wet and dry vacuum pressure as well as the best time of applying each pressure for gypsum-anhydrite rocks. The results of conducting experiments confirmed that: 1. By increasing the value of $P_{vac(wet)}$ to -0.5 atm, the weight of samples is increased. It means that the best sample saturation has occurred in this pressure. Although, a test with more than this pressure was carried out for a limited time, but it showed that by increasing wet pressure more, the weight of samples will rapidly decrease after an increase in the initial hours. This could be due to the accelerated increase in the weight gain of the detached particles from the sample, relative to the weight of absorbed water in the sample.

Table 4. Percentage of the maximum gain weight of the specimens in the sensitivity analysis experiments at $P_{vac(wet)} = -0.5$ atm

(%Weight increase for suction wet pressure -0.5 atm ($W_t - W_0 / W_{Max(increase)}$))											
Sample No	Time (hour)										
	0	1	2	3	4	5	6	7	8	12	24
MG4-9-7	0	62.8	66.9	70.2	71.0	73.5	72.0	70.3	73.0	74.3	78.9
MG5-6-7	0	65.9	74.9	77.7	80.0	81.3	81.7	81.2	82.9	83.6	85.3
MG6-7-8	0	29.9	32.1	33.4	33.0	34.7	34.9	33.4	32.8	37.1	42.4
MG6-8-7	0	50.6	66.9	74.0	78.8	80.0	81.6	81.7	80.3	83.0	83.8
MG11-2-3	0	69.7	75.2	76.9	77.1	77.7	75.2	76.0	73.0	75.5	77.1
MG11-5-2	0	64.3	74.2	77.8	79.0	82.6	82.3	83.3	83.0	85.4	87.1
MG11-7-5	0	56.1	67.8	71.7	73.2	74.2	75.7	77.3	76.3	77.8	80.2
KG9-6-7	0	67.1	80.8	88.0	90.6	91.3	92.6	92.6	92.7	92.9	94.7
Sample No	Time (hour)										
	48	96	120	144	192	216	240	288	312	336	$W_{increased}$ (g)
MG4-9-7	83.9	96.4	93.7	96.4	97.7	98.4	98.8	100.0	98.2	97.7	6.07
MG5-6-7	87.4	97.4	95.1	95.8	98.8	96.9	97.9	100.0	99.6	97.8	11.1
MG6-7-8	51.3	83.2	81.9	84.9	94.5	94.1	96.7	100.0	99.6	95.0	4.58
MG6-8-7	84.3	97.6	93.9	92.9	98.7	96.1	91.9	100.0	97.9	97.6	6.19
MG11-2-3	79.1	95.6	91.7	93.9	92.0	100.0	100.0	98.6	93.1	98.9	3.63
MG11-5-2	89.9	97.2	96.2	96.2	97.7	97.6	98.9	100.0	98.6	96.5	7.06
MG11-7-5	85.4	96.7	92.5	94.8	96.9	97.3	100.0	97.7	96.0	95.4	4.81
KG9-6-7	95.2	97.2	98.3	98.2	100.0	98.5	98.9	98.3	96.8	96.9	9.5

Table 5. Oven dried weight of desiccator bottom deposits in experiments with $P_{vac(wet)} = -0.1$ to -0.5 atm

$P_{vac(wet)}$ (atm)	Sum of samples	S_{total} (cm ²)	W_s (g)	W_s/S_{total}
-0.1	8	2,199.84	3.98	0.0018
-0.2	8	2,172.12	4.31	0.0020
-0.3	8	2,171.34	5.39	0.0025
-0.4	8	2,172.19	5.81	0.0027
-0.5	8	2,153.24	13.94	0.0065

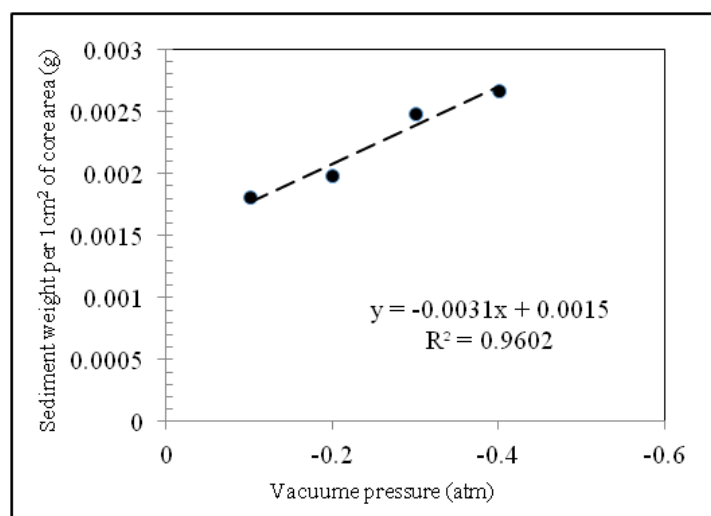


Fig. 2. Oven dried desiccator bottom sediment weights of sensitivity analysis experiments with $P_{vac(wet)} = -0.1$ to -0.4 atm.

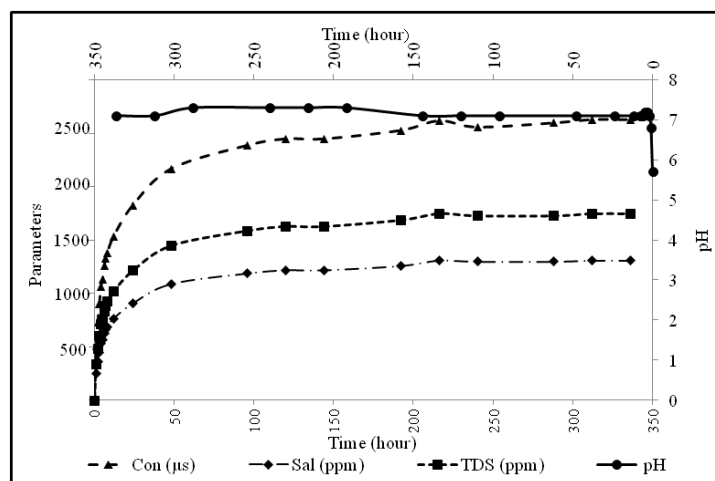


Fig. 3 The Con, Sal, TDS, pH variations during the sensitivity analysis experiment with $P_{vac(wet)} = -0.5$ atm

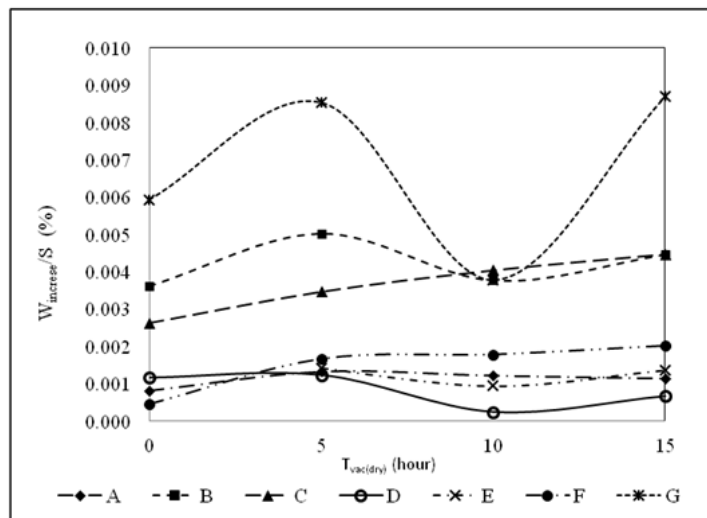


Fig. 4 Weight gain percentage of samples (after 24 h) per specific surface area of the samples relative to dry vacuum pressure, in the sensitivity analysis experiments with $T_{vac(wet)} = -0.4$ atm for different type of rocks

Therefore, $P_{vac(wet)}$ can have a critical value for different soluble and weak rocks, determination of which should be done through conducting similar experiments. Passing this value leads to lack of ideal results. In this study, under the tested vacuum pressure range, the $P_{vac(wet)} = -0.5$ atm was appropriate for saturation of gypsum and anhydrite rocks. This is much higher than the amount set by Hawkes and Mellor (1970). 2. There is a direct and positive relationship between $P_{vac(wet)}$ and $T_{vac(wet)}$ with the weight of deposit sediments on the reservoir floor (samples physical erosion).

3. In a determined $P_{vac(wet)}$, as time passes, the variations of pH, Con, Sal and TDS values gradually get closer to the stability and become linear roughly after 48 to 72 hours. This means saturating of water by cations and

anions and gradual decrease of sample solution. Under more stable conditions, the sample weight is a function of the increase due to the absorbed water in the sample and weight loss due to physical erosion.

4. Applying initial dry vacuum pressure $P_{vac(dry)}$ positively affects saturation of gypsum rocks and $T_{vac(dry)} = 5$ h is an appropriate time for better increase of sample weight. This time consists with experienced times by other researchers about other constructional materials and also is close to the time of Hawkes and Mellor (1970).

Acknowledgements

This research was funded by a grant to authors by the vice-president research office of Bu-Ali Sina University, Iran, that is gratefully acknowledged.

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