

Effect of solid impurity on creep behavior of salt rocks of the Hormoz formation

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ABSTRACT

Salt rocks have one of the most complex behaviors among different rock types due to their creep behavior. Creep in rocks can cause a lot of undesired displacements imposing tremendous rehabilitation and maintenance costs to the projects. Creep depends on many factors such as rock type, stress level, and boundary conditions in order for rock to move freely. Amongst intrinsic factors in the rock type, the impurity of salt samples (either in gas, liquid, and/or solid form) is one of the least studied factors. This study aims to present the influence of impurity on the creep behavior of the salt rocks of the Hormuz series, as the case study. This series is one of the oldest evaporitic deposits in the world resulted in more than 350 salt domes in Iran and other parts of the Middle East. Unfortunately, there has been no comprehensive rock mechanics study on the Hormuz salt rocks so far. In this study, a few recovered cores were obtained and prepared from the exploration boreholes drilled in this formation, and the creep parameters were determined using laboratory tests. Also, the effect of impurity percentage on the creep properties of the Hormuz salt rocks was investigated. Since in salt rock masses the purity percentage is different, impurity affects the creep behavior. The tested samples were categorized into seven different groups, based on the quantity of the impurity, which consists mainly of anhydrite and quartzite. Laboratory tests showed that the uniaxial compressive and tensile strength values increase by increasing the solid impurity in the samples. In contrast, the maximum and instantaneous strains reduce by increasing the percentage of impurities in different stages of the creep test. Increasing the amount of impurity in pure samples led to increasing Burger's parameters. Also, it was observed that obtaining creep parameters from laboratory test results with mathematical approximation method had fewer errors compared to the manual method explained by Goodman. This is worth for the development of underground mining operations in salt structures. Accurate recognition of creep properties might have a considerable impact on the design as well.

Keywords : Creep, Hormuz series, Impurity, laboratory Tests, Salt rock

Nomenclature			
σ	Stress	R	Gas constant
ε	Strain	Z_i	influence of other parameters
T	Temp	K	Bulk module
Q	Activation energy	UCS	Uniaxial compressive strength
η_1	Kelvin viscosity	σ_t	Tensile strength
η_2	Maxwell viscosity	E_s	Elastic module
G_1	Kelvin shear	PM	Personal measurement device
G_2	Maxwell shear	D	Data acquisition system
$(\Delta\varepsilon_1)^2$	Minimum error	DAS	Data acquisition system
		MA	Mathematical approximately method
		M	Mathematical approximately method
T	Time	LSM	Least square method
P	Density	RE	Relative error

form such as clay, quartz, calcite, carnallite, and anhydrite that can change its creep behavior.

The first comprehensive research on the creep phenomena was carried out in 1905. Primary studies had focused on metals. Such studies on rocks have been going on from the late 19th century up until now [3-10]. Rheology, or time dependent behavior, is one of the basic properties of rocks [11]. The creep phenomenon is a time-dependent deformation under constant stress. This behavior is a plastic deformation [3]. For the optimum design of underground structures, creep tests are carried out on rocks. Laboratory tests are the main method of obtaining creep parameters. In the past several decades, extensive laboratory investigations have been carried out for understanding the creep behavior of many kinds of rocks, including salt [12]. Many factors, such as the nature of stress, level of stress, confining pressure, temperature, humidity, and structural factors, affects the creep behavior [6, 13, 14].

Salt rock units exhibit different behaviors according to their impurity content, type, and location [9]. Previous research studies show that increasing the calcium sulfide content of a salt rock by 2% would decrease the creep rate for about 3% [15]. Fabrication and texture are also of other affecting factors on creep. Also, experimental results show that interlayers have an assignable effect on the mechanical parameters of salt rocks, and should be carefully considered numerical simulations [16].

1. Introduction

Due to a particular linkage between chlorine and sodium, halite has one of the most complex behaviors among different rocks. Impurity in a salt sample, either in the form of solid, liquid, or gas, makes this behavior even more complex. One of the oldest salt evaporate deposits in the world is the Hormuz series that has generated numerous salt domes in Iran and other parts of the Middle East [1, 2]. These salt domes have different impurity contents. One type of this impurity is in the solid

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Deformable materials, such as salt, have a hardening strain behavior [13]. So far, several models have been introduced for understanding the salt creep behavior in transient and stable deformations, but in general, the deformation rate of salt can be described using the equation below:

$$\dot{\varepsilon} = F(\sigma, T, \varepsilon, Z_i) \exp\left(-\frac{Q}{RT}\right) A \quad (1)$$

Where, A is a constant coefficient, σ and ε represents stress and strain, and T is temperature. Q, R, and Z_i are activation energy, gas constant, and the influence of other parameters such as impurity, respectively.

After conducting the creep test, plastic deformations can be observed. Before the creep test, grains and crystals are square and symmetric, but after imposing a constant amount of pressure, those grains and crystals became distorted. Also, cracked salt rocks sometimes heal and self-recover under creep conditions, as shown in Fig 1. Such behavior makes salt rocks a perfect choice for establishing underground gas storage [7].

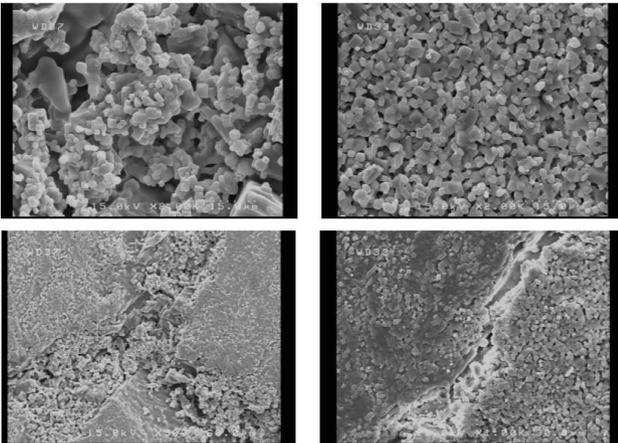


Fig 1. Above: intact salt crystals. Before conducting the creep test (right), and after the creep test (left). Below: bruised salt crystals. Before the creep test (right), and after the creep test (left)[10].

Since the purity percentage in salt masses is different, it is necessary to measure creep and other mechanical parameters of each salt rock sample separately for implementing the design of different projects. This paper investigates the influence of impurity percentage on the creep behavior of salt rocks.

2. Sample characteristics

The Magmam salt dome is one of the largest active salt intrusions in the Hormuz formation. Salt rises from a depth of about 4 km below the sea level to nearly 1.5 km above it, and it spreads over the surrounding lands [2, 17]. For the first time in Iran, several drillings were performed in the Magmam salt dome and returned suitable cores that are observed in Fig 2. To prevent salt dissolution during the drilling and cutting process, saturated brines were used as the drilling coolant liquid. For lab testing, 40 cores were collected with different impurity content from the Hormuz series. These cores were classified into six groups according to their impurity percentage (Fig 3).

The specimens were cylindrical with a diameter of about 6cm and a height of about 12cm, with L/D=2, as suggested in the rock mechanical testing standards. After mechanical testing, the samples were crushed for mineralogical analyses.

3. Laboratory Tests

The samples were prepared for static and creep tests, and finally for mineral analyses. Three samples from each classified groups were selected for each test. Unsuccessful results were eliminated from conclusions.

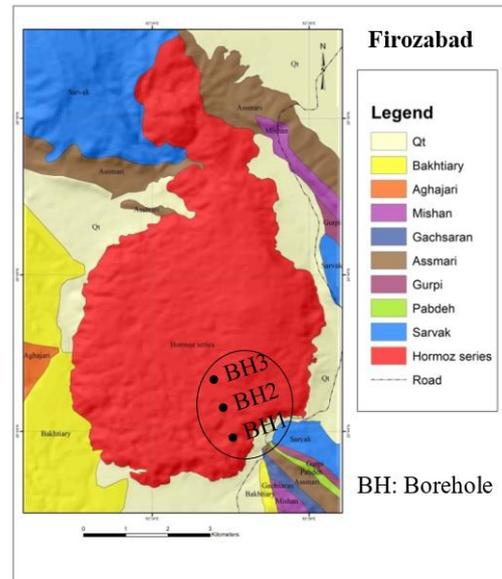


Fig 2. Geologic map of the Magmam salt dome (Firozabad-Fars-Iran) located in the Hormuz series, where borehole drilling was conducted.



Fig 3. Categorizing salt rock samples based on the impurity content.

3.1. X-ray analysis

In the first stage of investigations, the purity percentage was estimated by measuring the impurity length. The impurity length is the ratio of the apparent impurity length to the total length of the sample observed from the lateral side of the sample. This ratio was obtained by averaging five straight lines at the side surface. Finally, for refining, X-ray analyses were conducted to quantify the parameter more rigorously. This analysis was carried out using a D8- Advance device at the lab temperature (Fig 4). As seen in Fig 5, halite is dominant in all of the samples except sample E. Major impurities in these samples are anhydrite and quartzite, which have been distributed relatively homogeneously throughout the samples. In sample E, calcite is the dominant mineral that has changed the behavior of the sample, considerably. X-ray results are shown in Table 1.



Fig 4. The X-ray device.

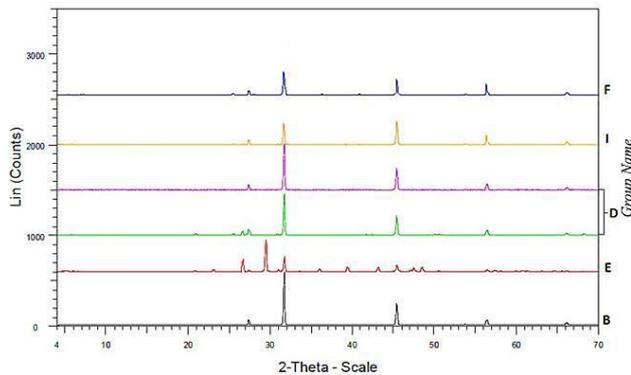


Fig 5. The X-ray graphs of the samples.

3.2. Uniaxial compressive strength and tensile strength tests

A uniaxial compressive strength test was carried out to determine the loading level for the creep test and in order to compare the samples. This test was carried out with an MTS loading machine (Fig 6). With increasing the impurity percentage, the strength and the stiffness of the samples increased.

Since knowing the approximate amount of tensile strength is sufficient in many cases, the Brazilian test was used in the present study as well. The test was carried out using a tensile strength test apparatus on disc samples (Fig 7). Similar to the UCS, the tensile strength also increases slightly as the impurity increases. The results of the uniaxial compressive strength and tensile strength tests for different impurity contents have been shown in Table 1. The amount of obtained elastic parameters for the Hormoz salt rock shows that these parameters are ranging within the reported normal elastic parameters of salt rocks in other parts of the world [18-21].



Fig 6. The uniaxial compressive strength apparatus

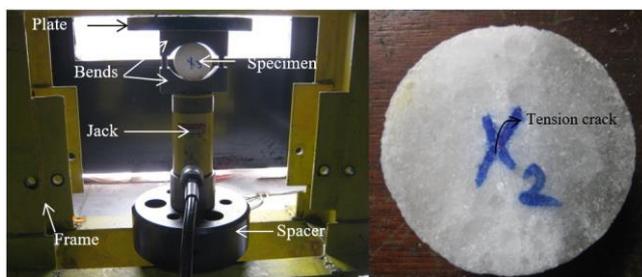


Fig 7. Right: A disc specimen. Left: The tensile strength test apparatus.

4. Creep test

The creep test specimens were prepared with additional accuracy. In the uniaxial creep test apparatus, the primary and secondary creep stages were obtained for each group. The loading chamber in this

apparatus used a 60-ton capacity hydraulic cylinder. A spring system was utilized to prevent load reduction. A 20-ton hydraulic load cell was also used to measure the stress value. Two linear and parallel potentiometers (with an accuracy of 0.002 mm) were used for measuring displacement, for which the results were averaged (Fig 8). Also, a data acquisition system was utilized for reading the data channels. This system stored the data on a computer together with a PMD analog device. It should be noted that all devices were calibrated before the testing process.

Table 1. Mineralogy data and static parameters of selected samples.

Groups	Salt purity percentage (NaCl) (%)	Uniaxial compressive strength UCS (MPa)	Brazilian tensile strength σ_t (MPa)	Elastic module E_s (GPa)	Impurity content
A	100	28.9	3.41	1.53	Pure salt
B	96	29.2	3.43	1.98	anhydrite
D	75.9	31.2	3.47	3.18	Calcite, quartz, anhydrite
I	53	33.1	3.55	4.8	dolomite, quartz, anhydrite
F	41	33.6	3.68	6.17	carrollite, quartz, anhydrite
E	15.2	39	4.1	13.2	Calcite, quartz, dolomite

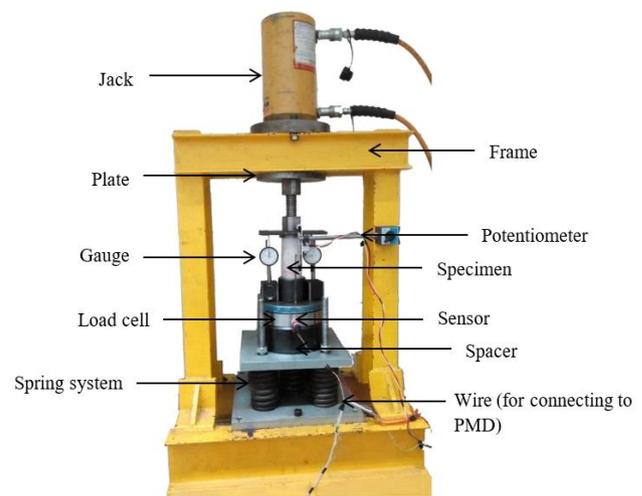


Fig 8. The creep testing apparatus.

The applied stress on the samples was 20.5 MPa (about 50-70 percent of its peak strength), and the testing process usually lasted between 12-15 days. After the loading period, the linear secondary stage of creep was well established, and the tests could be stopped. Fourteen creep tests were carried out on all of the salt rock groups. Seven samples had early failures due to preexisting fractures. Figs 9, 10, and 11 show the creep curves, maximum strain, and strain reduction with an increasing impurity value for all of the six groups, respectively. The maximum strain in these graphs is the amount of strain after reaching the secondary creep stage on the twelfth day. This value was defined as an index to compare the strain of samples in the secondary creep stage based on various impurities. By measuring the temperature and moisture during the tests and keeping them constant, it can be claimed that environmental conditions had not influenced the results. As an extra testing procedure, the G sample creep curve was carried out on salt rocks with undefined impurity contents (about 30 percent). As observed in the creep curves, from top to bottom (groups A, B, D, I, F, and E), increasing the impurity content would decrease the strain rate.

In impure samples, microscopic cracks might have been created in the course of testing due to the non-plastic behavior of impurities. This event happens rarely for pure samples because of the plastic behavior of salt.

As Fig 10 shows, the maximum strain in a similar period is decreased by increasing the impurity in samples. By increasing impurity gradually, plastic behavior is converted into brittle behavior and the strain rate decreases.

A four percent impurity increase in pure samples leads to a decrease in the maximum strain by 15 percent. Also, when impurity increases to

25%, the maximum strain decreases by 50%. Similarly, the maximum strain reduction continues with increasing impurity.

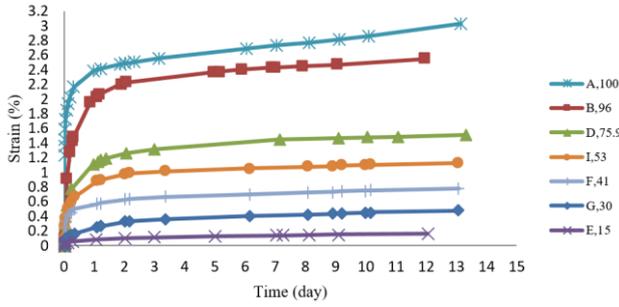


Fig. 9. Strain-time curves of salt rocks with different impurities.

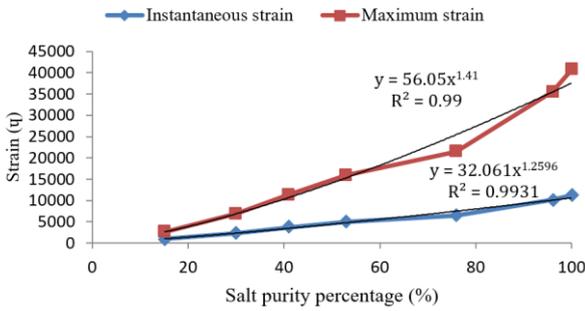


Fig. 10. Instantaneous and maximum strain after a certain period for salt rocks with different impurity contents.

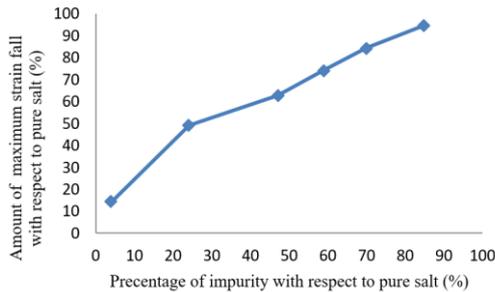


Fig. 11. Percent of strain fall with increasing impurity.

5. Determining viscoelastic constants

The simplest method for determining the viscoelastic constants is the application of the uniaxial compressive creep test on cylindrical samples. The requirement for this test is to apply constant stress, temperature, and moisture values throughout the test that might take days and even months. The constitutive creep equations of salt rocks have a good agreement with Burger’s model [7]. The axial strain with respect to time ($\epsilon_1(t)$) in Burger’s model under constant stress is determined by the following equation [22]:

$$\epsilon_1(t) = \frac{2\sigma_1}{9K} + \frac{\sigma_1}{3G_2} + \frac{\sigma_1}{3G_1} - \frac{\sigma_1}{3G_1} e^{-G_1 t / \eta_1} + \frac{\sigma_1}{3\eta_2} t \quad (2)$$

In this equation, G_1 , G_2 , η_1 , and η_2 are creep parameters. In Burger’s model, the amounts of creep parameters vary at different stress levels. Since the aim of this research was to examine the impurity effect on creep behavior, only one stress level was applied. The details on how to obtain Burger’s creep parameters from a test result are discussed by Goodman [22]. This method is a manual method that is prone to errors due to offsets in the regression line. In order to reduce this error, a mathematical approximation method, such as the least square method (LSM) is more promising. This method adopts values for the coefficients that make the regression model closest to the observations. According

to the strain-time data, the strain varies with time. Therefore, at any time, the creep parameters are changing. If the equations for each time frame are written, they are expressed as below:

$$\begin{aligned} \epsilon_1(t_1) &= \frac{2\sigma_1}{9K} + \frac{\sigma_1}{3G_2} + \frac{\sigma_1}{3G_1} - \frac{\sigma_1}{3G_1} e^{-G_1 t_1 / \eta_1} + \frac{\sigma_1}{3\eta_2} t_1 \\ \epsilon_1(t_2) &= \frac{2\sigma_1}{9K} + \frac{\sigma_1}{3G_2} + \frac{\sigma_1}{3G_1} - \frac{\sigma_1}{3G_1} e^{-G_1 t_2 / \eta_1} + \frac{\sigma_1}{3\eta_2} t_2 \\ \epsilon_1(t_n) &= \frac{2\sigma_1}{9K} + \frac{\sigma_1}{3G_2} + \frac{\sigma_1}{3G_1} - \frac{\sigma_1}{3G_1} e^{-G_1 t_n / \eta_1} + \frac{\sigma_1}{3\eta_2} t_n \end{aligned} \quad (3)$$

In the least-squares method, ϵ_1 is determined at different times according to the initial approximation for unknown parameters. For a specific time, if the difference between calculated and recorded ϵ_1 values (known as $(\Delta\epsilon_1)^2$) is more than a permissible error value, unknown parameters will be changed in a way to minimize this difference. Therefore by matching these two graphs, the estimated creep parameters are obtained with a minimum error. According to the LSM and Goodman’s methods, Burger’s parameters are shown in Table 2 with constant stress and a specified K value for each group. According to the Burger and Goodman methods, creep parameters can be calculated from the second stage of creep when the strain rate (slope of the graph) remains constant. This happened after two weeks in these tests. At this stage, the slope of the curve (strain rate) is constant at any time. Therefore, the amount of Burger’s creep parameters was extracted from the linear part of the creep-time strain curves.

Table 2. Creep parameters of the six rock groups.

Groups	Parameters Method/unit	ρ	σ_d	K	G_1	η_1	G_2	η_2
		Kg/m ³	MPa	GPa	GPa	GPa·Day	GPa	GPa·Day
A	LSM	2149	20.5	2.55	0.285	0.04	0.73	14
	Goodman				0.281	0.036	0.665	13
B	LSM	2161	20.5	3	0.315	0.095	0.8	19
	Goodman				0.327	0.14	0.8	22
D	LSM	2218	20.5	3.78	0.555	0.17	1.28	30
	Goodman				0.55	0.3	1.15	30
I	LSM	2270	20.5	4.21	0.71	0.24	1.78	55
	Goodman				0.73	0.34	1.74	60
F	LSM	2300	20.5	5.14	1.115	0.25	2.42	60
	Goodman				1.05	0.35	2.4	55
E	LSM	2488	20.5	10	7.2	3.8	9.1	115
	Goodman				7.6	5	9.25	121

Table 2 compares the creep parameters for samples obtained from both methods. Also, the real and estimated strain-time diagrams are shown in Fig 12. As illustrated in these figures, in all samples, the creep curve obtained from the least square method has a better match with the real data. To compare the relative error of both methods, the error value relative to the actual data on the linear part of the curves has been shown in Table 3. Although both error values are acceptable, the least square method has a lower error value.

Table 3. Comparing the errors for the least square and Goodman methods.

Groups	Method	Relative error (%)
A	LSM	0.62
	Goodman	3.18
B	LSM	0.1
	Goodman	3.52
D	LSM	0.49
	Goodman	2.3
I	LSM	0.58
	Goodman	2.94
F	LSM	0.99
	Goodman	2.66
E	LSM	0.72
	Goodman	3.96

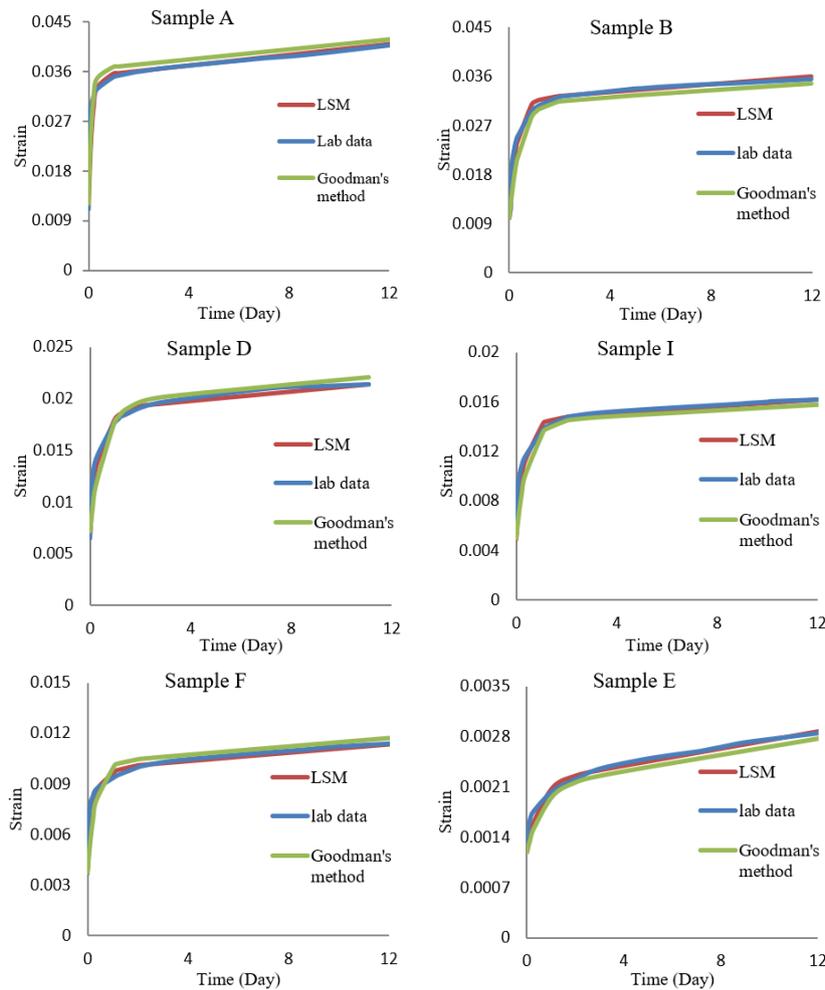


Fig. 12. Comparison of time strain curves of both methods with the actual data.

Since the errors in Goodman's manual method are more than that of the mathematical approximation method, the latter's results have been used for the interpretation of creep parameters at the design stage.

Increasing the impurity in salt samples decreases strain in the primary and in secondary stages, which means that viscosity must have increased. According to Table 3, in the first five samples, the obtained creep parameters increased smoothly with increasing impurity, but in the case of E group, because of a jump in impurity, the creep parameters increased significantly (Fig 13).

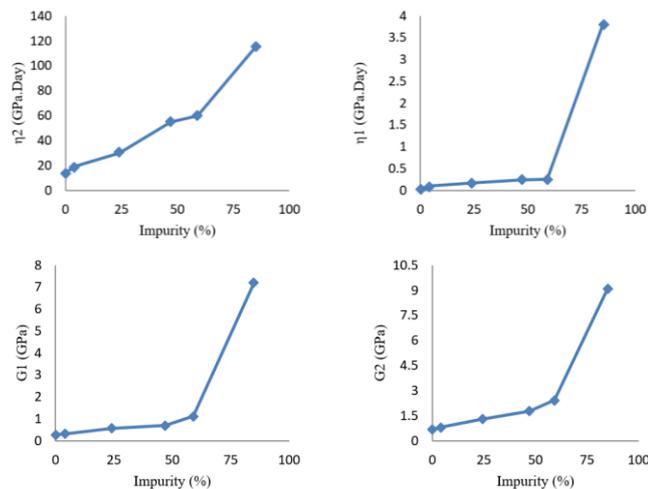


Fig 13. Effect of impurity on creep parameters of salt rock samples.

Increasing the amount of impurity in pure samples leads to increasing η_1 , G_1 , η_2 , and G_2 . As seen in Fig 13, the amount of impurity has more effect on η_1 than the other three factors. This parameter is Kelvin's viscosity that represents the strain rate in the primary stage of creep (slope of the primary creep curve). In other words, increasing impurity considerably decreases the instantaneous strain rate.

6. Conclusions

This was a comprehensive experimental study to investigate the effect of solid impurity on the creep behavior of the Hormuz salt rocks. Uniaxial compression tests, X-Ray measurements, Brazilian disk splitting tests, and uniaxial creep tests were performed on salt rock samples with different impurity contents. According to the results, the following conclusions can be made:

- It is important to consider the impurity content, type, and its distribution form while determining the creep behavior of rocks such as salt. If the formation consists of many layers with a wide range of impurity, it is not safe to assume an average value for the creep parameters; instead, one should take samples from different layers and determine the creep parameters in each group separately.
- The experimental X-ray analysis confirmed that the main impurities were anhydrite and quartz. These were almost homogeneously distributed throughout the sample instead of being localized in distinct bands.

- Since impurity is a unique inclusion within the salt structure, it inhibits interlayer slides; therefore, increasing the impurity content will increase the UCS and σ_1 parameters. Moreover, some impurities (such as quartz) do not have creep behavior; therefore, their presence reduces the overall creep behavior of the samples. It is expected that if the impurity type is changed to the materials with a softer behavior than salt crystals (such as clay minerals), a different behavior might be observed. Investigations on this aspect require more laboratory tests on rocks with such impurity types.
- For the tested samples, the strain rate decreased by increasing impurity. For example, a 4% increase in the impurity content decreased the maximum strain by about 15%. Also, a 25% impurity value would decrease the maximum strain by about 50%.
- It was shown that higher impurity values in salt would increase creep parameters significantly, showing that the sample would behave more viscously.

Obtaining creep parameters from laboratory results with mathematical approximation method would result in higher accuracies than those of Goodman's method. Therefore, the former method is suggested for such cases.

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