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Effect of exposure on material response of a swelling elastomer

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ABSTRACT

Purpose: This paper reports some results from a comparative study of the behavior of fresh and exposed samples of an EPDM-type water-swelling elastomer.

Design/methodology/approach: Experiments were designed and performed in line with standard ASTM test methods and in consultation with petroleum development engineers. Small test fixtures were designed and fabricated, to be used together with standard testing equipment. Elastomer response was studied for hardness, compression set and tensile set (at different temperatures and for different periods of time), tensile properties (fracture strength and percent elongation), and swelling (gradual thickness and volume change with exposure to saline solution). In the swelling test, unconfined samples and samples mounted on steel plate were tested for a total duration of 1000 hours (roughly 45 days) in salt solutions of different concentrations and at different temperatures.

Findings: Exposed elastomer samples (EPDM1) showed higher hardness than fresh samples (EPDM2). Compression set values of exposed samples was significantly higher than fresh ones. Tensile set values were almost the same for the two sample types after 10-min test, but were higher fort exposed elastomer after longer-duration tests. Stress-strain graphs for both sample sets were almost linear, in contrast to highly nonlinear graphs for usual rubber-type materials. Values of fracture stress and elastic modulus for exposed elastomer were noticeably higher, but percent elongation was lower. Swelling behavior showed a fluctuating trend with increasing swelling time for both elastomers. For same temperature and salinity, fresh elastomer samples yielded much more swelling than exposed samples. All of these observations indicate that such swelling elastomers lose flexibility and swelling capacity when exposed to sun and moisture, etc for extended periods of time.

Practical implications: Results of this study can be used by oilfield engineers to gauge the suitability of these elastomers for downhole applications. Material properties after swelling can be used by designers using FEM or other numerical simulation methods for improvement of elastomer-based sealing and packer design. Comparison of fresh and exposed elastomer samples highlights the significant change in material response due to exposure.

Originality/value: The paper presents a comparison between material properties of fresh and exposed samples of the same water-swelling elastomer. Such a comparative study, highlighting the effect of exposure on material response of an elastomer, has not been carried out before.

Keywords: Engineering polymers; EPDM-type water-swelling elastomer; Material characterization; Hardness; Compression set; Tensile set; Tensile properties; Volume change; Thickness change

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MATERIALS

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1. Introduction

Water-swelling and oil-swelling elastomers are good candidate materials for sealing applications in oil exploration and development work. Solid expandable tubular (SET) technology and swelling elastomers have been successfully used together in solving various problems associated with enhanced oil recovery in the Gulf region and in several other countries around the world. Some of these elastomer applications are related to well completion: the process of finishing a well so that it is ready to produce oil or natural gas [1,2].

With more than 130 successful installations in the past few years, SET has established itself as a workable solution for drilling and re-completion challenges in both conventional and deepwater wells. Some of these successful applications include open hole liner installed in the Gulf of Mexico [3]; open hole liner installed in Asia [3]; cased-hole liner installed in Texas [3]; one of the longest open hole clad and open hole liner installed in Oman [4]; etc.

In SET system the swelling elastomer is used as a joint to both seal and anchor the system. The most common SET applications are expandable open-hole liner OHL [4,5], expandable cased-hole liner CHL [3], expandable open-hole clad OHC [3], and expandable liner hanger ELH [6]. Swelling elastomers are also used as packers: a rubber element vulcanized onto the pipe, used to isolate a specific zone (zonal isolation) of a cased or uncased wellbore since it swells significantly when it is exposed to hydrocarbons or saline water [7]. Design of a swelling packer is affected by various main parameters like life-span, required maximum differential pressure across the packer, swelling time and temperature, and geometry of the pipe, packer and borehole [8].

A detailed study is being conducted by the authors on mechanical profile control in well engineering. The work includes development of different semi-analytical and finite-element based numerical models for simulation of swelling elastomer performance in downhole sealing applications [9,10]. All such models require a reasonably good knowledge of material response under different environmental conditions. Some authors have discussed specific material response of elastomers [11-16]. However, detailed information about elastomer behavior under different field conditions is generally not available. Various experiments were therefore designed and implemented for testing, characterization, and performance evaluation of swelling elastomers. Properties of an EPDM-type water-swelling elastomer were reported in a recent paper [17]. It was discovered during subsequent work that elastomer behavior can significantly change if the material is exposed to the elements. Pipes with elastomers mounted on them (known as packers) were obtained from the pipe yard of a local petroleum firm. Though the elastomer sections were generally covered by protective sheets, the covering was damaged in places, so that the elastomer was exposed to the environment for several months. This paper covers test results and brief analysis for the same water-swelling elastomer, comparing fresh and exposed samples. Exposed samples are identified as EPDM-1 and fresh samples as EPDM-2. Compositional details of the elastomer have been withheld due to confidentiality reasons [21].

2. Experimental work

Procedure for the swelling test was developed in consultation with oilfield and rubber application engineers. All the other tests (hardness, tensile set, compression set, and tensile properties) were conducted in accordance with ASTM standard test procedures [18-20]. Test temperatures and salinity conditions were selected to emulate actual well conditions in different local oilfields: three temperatures (room/ambient, 50°C, and 80°C) and two salt concentrations representing low and high salinities (6.000 ppm or 0.6%; and 200.000 ppm or 20%).

2.1. Hardness test

Hardness of elastomer samples was measured using a Durometer provided with an “A” scale for soft materials and a “D” scale for materials of higher hardness. Test procedure was in line with *ASTM D2240* [18]. Reported hardness values are the average of readings taken at five different locations on each sample at room temperature.

2.2. Compression set test

The test was carried out in a locally designed and fabricated test fixture, following ASTM guidelines. Ability of the elastomer to retain elastic properties after being compressed for specified time period is measured. Disc-type elastomer specimens were compressed by 25% and held for 22-hour and 70-hour durations at three test temperatures: room temperature (~25ºC), 50°C and 80°C. Samples were then removed from the fixture cooled at room temperature for half an hour before measuring the new dimensions. Applicable standard is ASTM D395, method B [19].

2.3. Tensile set test

Another locally designed and fabricated test fixture (in line with ASTM guidelines) was used for the tensile set test. After stretching a ring-type specimen to twice the original size for a specified duration (10 minutes, 10 hours, and 20 hours), it is allowed to recover for the same time. The remaining extension is reported as tensile set. The test is performed at room temperature as outlined in ASTM D412 [20].

2.4. Tensile properties test

This test was conducted to determine the tensile properties (modulus of elasticity, fracture strength, and percent elongation) of elastomer samples. Hook-type grips were fabricated (in accordance with ASTM guidelines) to grip ring-type samples in a tensile testing machine fitted with a small load cell for rubber-type materials. As specified in *ASTM D412* test standard [20], tests were carried out at a loading speed of 500 ± 50 mm. Reported values are average of readings from three tested samples.

2.5. Swelling test

This test was performed to determine the swelling behavior (volume and thickness change) of elastomer samples when immersed in different salt solutions kept at different temperatures for a total time of 1000 hours. Disc samples were used to study the swelling response of free (unconfined) elastomer, while plate samples (elastomer vulcanized onto steel plate) were used to replicate the sealing behavior of elastomer mounted on a pipe. Temperature-resistant sealable glass jars were utilized to maintain constant concentration even at higher temperatures. Thickness and volume of each specimen was measured before swelling, and periodically (every four days) after swelling in different salt solutions at different temperatures. Servo-controlled digital ovens were used for elevated temperature testing. A special apparatus was fabricated, using glass beakers and graduated cylinders, for accurate volume measurements.

2.6. Test parameters

The different experiment design variables are briefly described below.

Material

EPDM-type water-swelling elastomer; samples exposed to the elements for several months (EPDM-1) and fresh samples (EPDM-2).

Sample configuration

- For swelling test: disc samples (25 mm diameter, 6 mm thickness), and plate samples (elastomer vulcanized on 25 mm × 25 mm steel plates);
- For tensile set and tensile properties test: ring samples of 3 mm thickness, 1.5 mm radial width, and inside and outside diameters of 16mm and 19 mm;
- For compression set test: disc samples;
- For hardness test: disc samples.

Test temperature

Room temperature (~25ºC), 50ºC, 80ºC.

Concentration of salt solution

200.000 ppm (or 20%), and 6.000 ppm (or 0.6%).

Testing time

- For swelling test: 1,000 hours (roughly 45 days);
- For tensile set test: 10 min, 10 hours and 20 hours;
- For compression set test: 22 hours and 70 hours.

2.7. Sample preparation

Exposed elastomer material was provided by a local petroleum development firm, already mounted on steel pipes, ready for use as seals. Samples of fresh elastomer were supplied by a rubber development company working closely with the oilfield industry. For the disc and ring samples, some of the material was carefully peeled off from the pipe, and the uneven surface was smoothed out on a surface grinder. Samples were then cut using a special die and punch set. Plate samples were directly cut from the pipe (without removing the elastomer) using saw-cutting and milling machines.

3. Results and discussion

3.1. Hardness

Average hardness value for the exposed elastomer (EPDM1) was 51.3 on the Shore-A scale, while that for fresh samples (EPDM2) was 57.3. This is a significant difference, indicating that hardness of a water-swelling elastomer would increase by exposure to the sun. Increased hardness (or loss of suppleness) should generally result in lower amounts of swelling. Later results would corroborate this conclusion.

3.2. Compression set

Plots of compression set CS (%) against temperature are shown in Fig. 1 for the two test durations of 22 hr and 70 hr. As expected, compression set increases with temperature, the increase being sharper at higher temperatures. Also, as expected, CS curve for the longer test duration is higher than that for the shorter one. This would indicate that if the elastomer is compressed for a longer time at a higher temperature, there would be a large amount of permanent set. Interestingly, CS values for EPDM1 are much higher than those for EPDM2. This implies that the elastomer loses elasticity due to exposure (also indicated by the hardness results), producing higher permanent set due to compression, or relative lack of springback after the release of compressive force.

3.3. Tensile set

Standard test duration for tensile set (TS %) test recommended by ASTM is 10 min. However, 10 hr and 20 hr tests were added for comparison with material data available at some of the rubber manufacturers' sites, and to study the variation pattern more thoroughly. As shown in Fig. 2, room-temperature TS increases with increasing test period. For the 10-min test, exposed and fresh samples yield almost the same TS value. For longer testing times, curve for EPDM1 is higher than EPDM2, and increase in TS with time is also sharper for EPDM1. Higher permanent set under tensile loading again indicates loss of elastic recovery due to exposure.

3.4. Tensile properties

Figure 3 presents results of the tensile properties test for the two elastomers in the form of stress-strain graphs. We know that rubbers and elastomers generally exhibit a nonlinear tensile behavior. It is therefore astonishing to see that the entire stress-strain curve is almost linear for both EPDM1 and EPDM2. This would imply that the special filler materials and cross-linking used to produce swelling elastomers make them behave differently under tension as compared to normal elastomers.

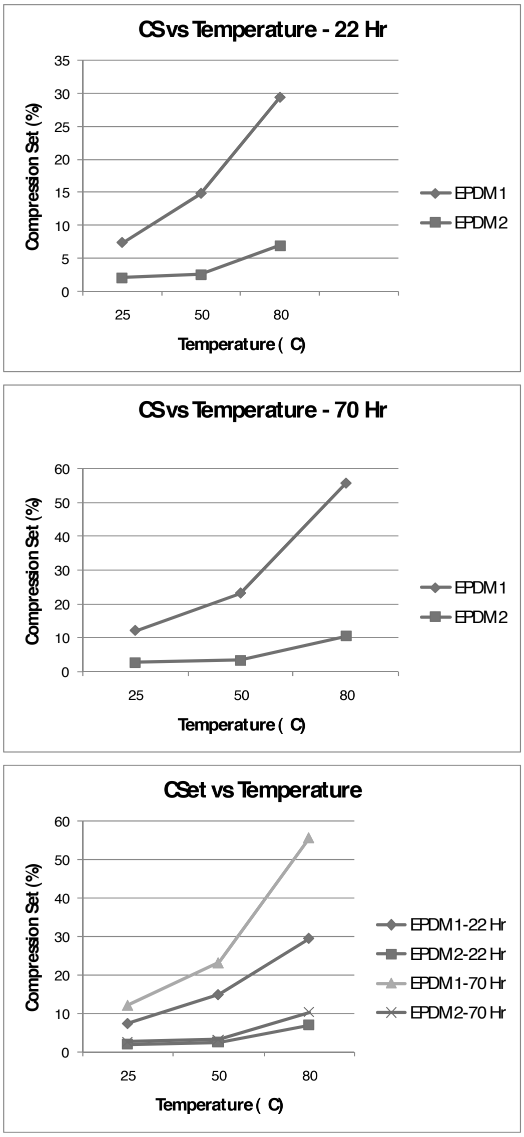

Fig. 1 Variation of compression set with testing time and temperature

Table 1.
Tensile properties of exposed and fresh elastomer

EPDM1 (Exposed)			
Sample #	Sample #	Sample #	Sample #
1	1	1	1
2	2	2	2
3	3	3	3
Average	Average	Average	Average
EPDM2 (Fresh)			
Sample #	Fracture Stress (MPa)	Percent Elongation	Modulus of Elasticity (MPa)
1	10.655	364.900	3.132
2	10.693	342.700	3.235
3	11.920	370.100	3.286
Average	11.089	359.233	3.218

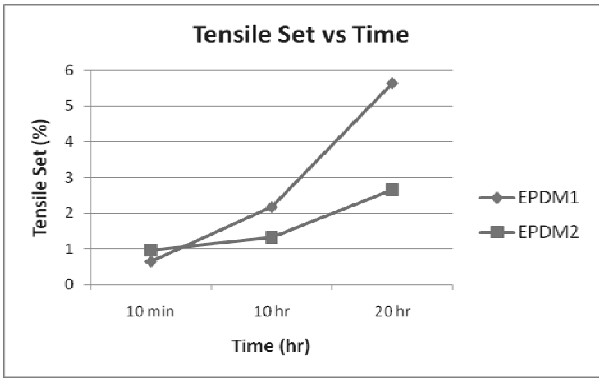

Fig. 2 Variation of room-temperature tensile set with testing time

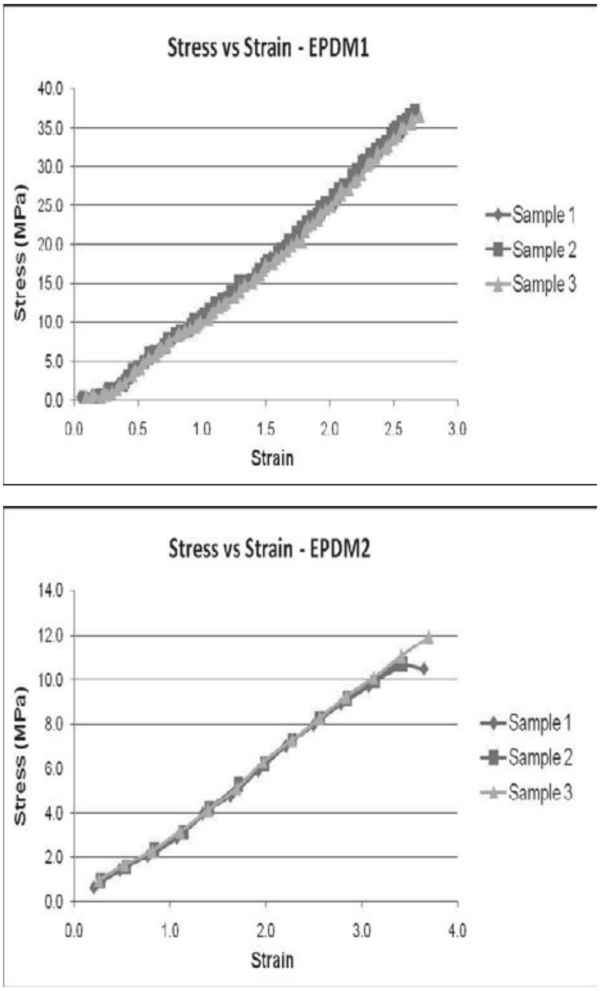

Fig. 3 Stress-strain plots from tensile test of three samples

The fact that curves for the three samples of each elastomer are almost identical, together with the near-linearity of the curves makes it very convenient to calculate tensile properties, especially the elastic modulus (slope). As summarized in Table 1, average fracture stress and elastic modulus for exposed samples are significantly higher than that for fresh ones, while percent elongation shows an opposite trend. This reinforces the previous results; exposure reduces the softness of the elastomer, resulting in lower flexibility (percent elongation) and higher fracture stress.

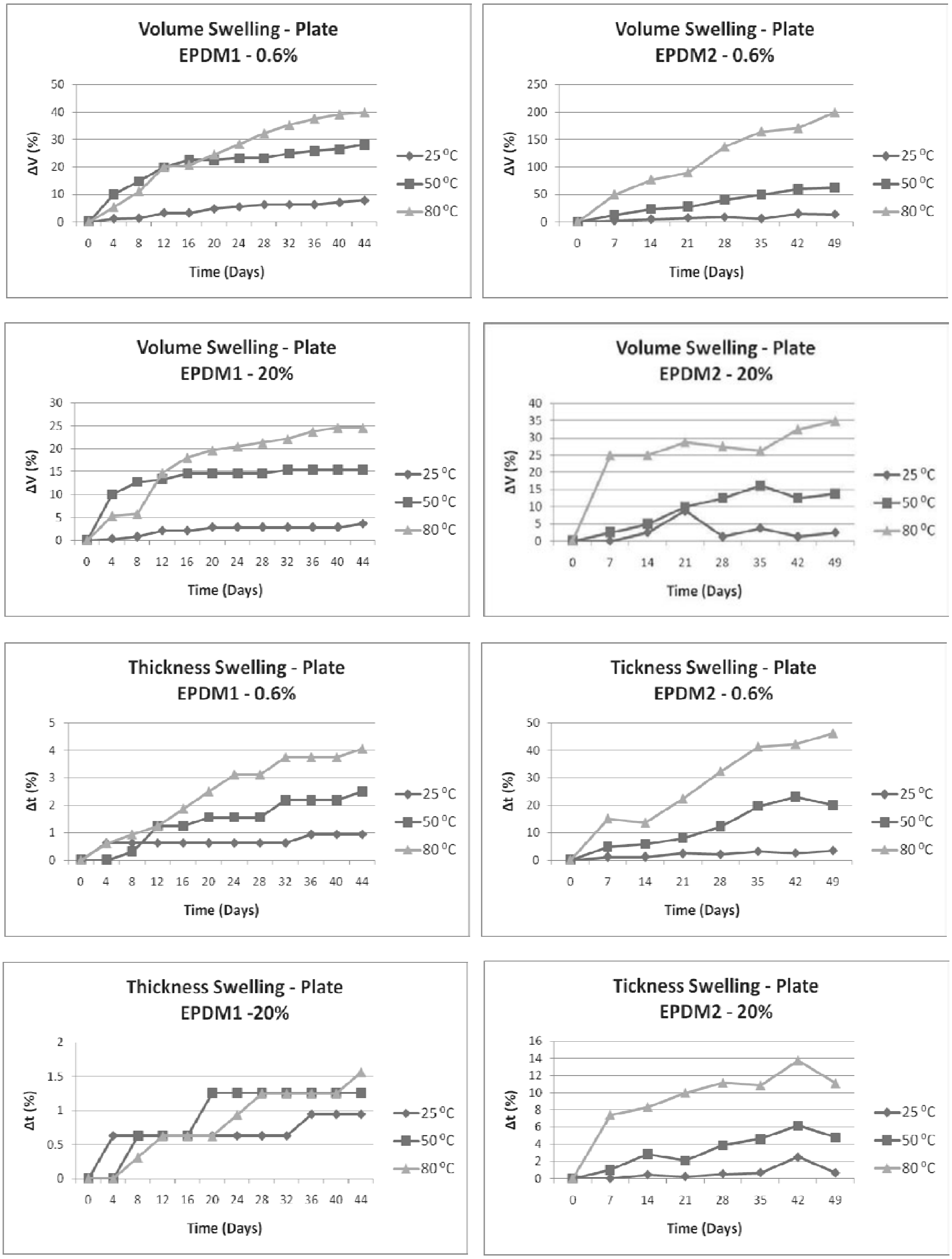

Fig. 4. Volume and thickness swelling for plate samples under different concentrations and temperatures

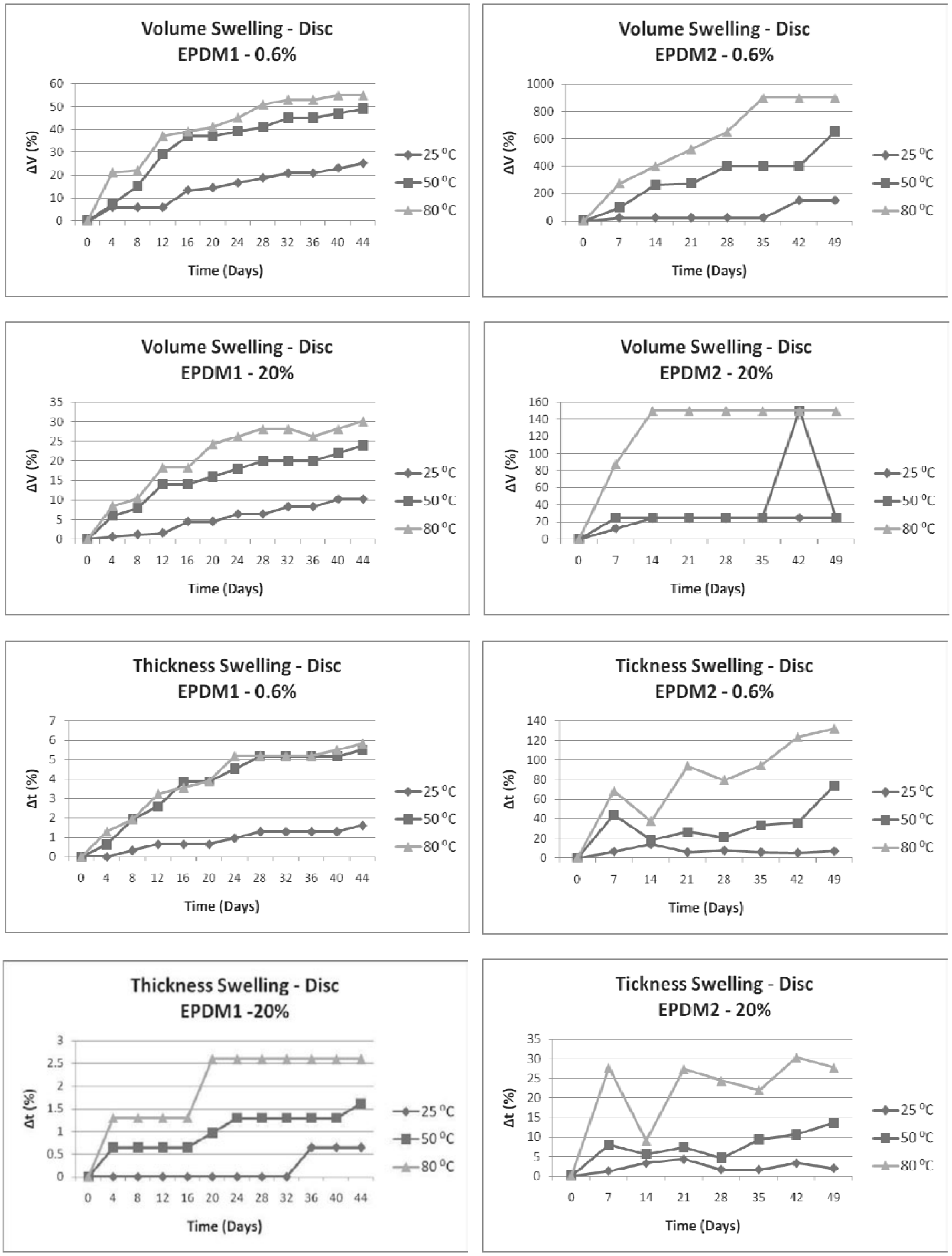

Fig. 5. Volume and thickness swelling for disc samples under different concentrations and temperatures

3.5. Swelling

Being a water-swelling elastomer, the most crucial test was perhaps determination of the swelling response when the elastomer is immersed in saline water at different temperatures. In the following graphs, amount of swelling (volume change ΔV% and thickness change Δt %) is plotted against time for different sample types and test conditions.

Plate Samples

Figure 4 shows the variation of swelling (percent change of volume or thickness) with time for plate samples. It is clear from all of the graphs that more swelling occurs when samples are kept under water for longer duration, as expected. It is noticeable, however, that the increase in volume (or thickness) with time does not progress smoothly, but happens in a fluctuating manner. Swelling increases, then remains constant for some time, then increases again, and even decreases a bit at times. It is known that salt is one of the constitutive materials in a swelling elastomer. For elastomer samples submerged in salt solutions, some salt may seep into the elastomer (together with absorbed water), adding to the salt content of the elastomer. Conversely, some salt may also break away from the elastomer and go into the salt solution. Swelling and change in salt content may also modify the cross-link chains in the elastomer material. A combination of these two ongoing phenomena (buildup/reduction of salt content, and changes in cross-link structure) may cause fluctuations in the swelling response with time.

Maximum swelling (volume or thickness) occurs for both elastomer types at a temperature of 80°C and under 0.6% salt concentration. Obviously, like most of the other materials, elastomers expand more with increasing temperature. Expanded pores allow more water to soak into the material, resulting in higher swelling. It is also natural that an elastomer would swell more in diluted solutions than in higher-concentration solutions, whether swelling happens due to diffusion or due to osmosis.

Volume swelling percentage is evidently much higher than thickness swelling; thickness change represents swelling in only one direction, while volume change corresponds to swelling from all exposed surfaces. Under the same conditions of salt concentration and temperature, fresh samples show substantially higher swelling than exposed ones (200% volume change compared to only 40% for 0.6%-80°C condition, for instance). This is in line with all earlier observations; extended exposure to sun, wind and moisture reduces elastomer flexibility and increases its hardness; harder and less elastic material naturally exhibits reduced amounts of swelling.

Disc Samples

Volume and thickness swelling of disc samples is plotted against swelling-time in Fig. 5. Like plate samples, discs also demonstrate the fluctuating swelling pattern. Also in a similar manner, maximum swelling is observed at the highest temperature and the lowest salt concentration. As before, amount of volume swelling is far higher than thickness swelling. Most importantly, once again, fresh samples undergo noticeably higher swelling than exposed ones (900% volume change compared to only 55% under the 0.6%-80°C condition). Reasons for all of these observations for disc samples are the same as those for plate samples.

If we compare the swelling response of disc samples against plate samples, we notice a huge difference (900% volume change as against 200%, or 130% thickness change in comparison with 48%, under the same salinity and temperature conditions of 0.6% and 80°C). Disc samples are free to swell from all sides, while swelling of plate samples is restricted from one major surface; thus the sizeable difference.

Behaviour of disc samples can be used for reference purposes, to gauge the response of unconfined elastomer. Plate samples replicate real material response, when elastomers vulcanized on pipes are used as sealing elements in the form of packers etc. Also, volume swelling can be used as a base figure to evaluate the total swelling effect. Of more direct interest for downhole applications, however, are the thickness swelling values because they indicate how well a gap between a tubular and a casing (or between a pipe and the formation) will be sealed off.

4. Conclusions

Comparison between material response of fresh and exposed samples of an EPDM-type water-swelling elastomer has been carried out through material characterization experiments, in particular the study of swelling behavior. Experimental strategy was designed for various sets of experiments, including swelling under different saline solutions at different temperatures, for a total swelling time of about 1000 hours. Apart from conventional material testing machines, simple test rigs and jigs/fixtures were designed and fabricated for some of the tests. Swelling test was planned and designed in consultation with drilling engineers and rubber developers. All other tests were performed in accordance standard ASTM test methods.

Shore-A hardness of exposed elastomer samples (EPDM1) was notably higher than that of fresh samples (EPDM2), indicating loss of flexibility with exposure. Compression set was found to increase with increasing temperature and testing time, CS values of exposed samples being significantly higher than fresh ones. Room-temperature tensile set values of the two sample types were almost the same for short-duration test (10 min), but were considerably higher fort exposed elastomer after longer-duration tests. Both compression set and tensile set values suggest that permanent set (or lack of springback) increases with exposure to the elements. Tensile properties test data surprisingly yielded almost linear stress-strain graphs for both sample sets, as against highly nonlinear graphs for most rubber-like materials.

Average values of fracture stress and elastic modulus for the exposed elastomer were clearly higher, while percent elongation was lower, again implying that exposure reduces softness and flexibility of the elastomer. Rather than increasing steadily with time, swelling response showed a fluctuating trend for both elastomers. Higher amount of swelling is generally observed for higher temperatures and lower salt concentrations. Under the same conditions of temperature and salinity, fresh elastomer samples exhibited far more swelling (percent volume or thickness change) than the exposed samples. This strengthens the observation that elastomers lose suppleness if exposed to sun and moisture, etc for extended periods of time. Reported mechanical properties and swelling behavior can be used to qualify elastomers for downhole applications under varying field conditions. These material characterization results are also essential for modeling and simulation of swelling elastomer seals, providing valuable guidelines for seal design. Comparison of fresh and exposed elastomer samples highlights the significant change in material response due to exposure.

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