

A NOVEL LABORATORY TECHNIQUE TO DETERMINE THE SATURATION STATUS OF SHALE

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Abstract

Drilling for oil and gas is concentrated in sedimentary basins of which 60% of the formations are shales. Worldwide drilling records indicate that most borehole instability occurs while drilling shaley formations.

Study of shale core behavior in the laboratory is the prime method for obtaining information for instability control. However, subsurface shale cores are usually highly altered and thus not suitable for laboratory studies. Results obtained using the saturation technique for one saturated and one unsaturated core are presented. The field handling technique recommended for acquiring and storing shale is also presented.

Introduction

Should a shale experience loss of moisture during retrieval, handling, or storage, its mechanical and physio-chemical properties become seriously altered due to the creation of capillary voids (Forsans, 1994). When such altered shale are placed in water-base drilling mud, the intake of water into the capillaries compresses the air, which can lead to significant damage to the rock mass skeleton.

To avoid the artifacts associated with shale drying during the preservation, handling, and transportation of shale cores, special care is needed. In addition, it is imperative that all shale cores be evaluated for their level of saturation before testing.

This paper describes results obtained using a novel laboratory technique to determine the saturation status of shale cores.

In addition, this paper investigates the effect of restoration of unsaturated (altered) shale to a saturated condition as reflected in shale swelling studies.

Shale Cores Studied

In a previous study (Chenevert, 1996), four shales were taken from storage and investigated for their water saturation level using the technique described herein. All four shales were found to be in an unsaturated condition. This paper focuses on results obtained for the previously studied "Alaska" shale as well as a freshly obtained North Sea shale that was found to be saturated. The composition of the North Sea saturated shale as well as the Alaska shale is given in Table 1. The Alaska shale data are presented for comparison purposes.

It has been shown that many of the physical properties of a shale depend on its water content. Measurements were made on both shales immediately after removing them from their stored environment, referred to herein as their "native" properties. Values for native water content and water activity are listed in Table 2. By definition, water activity equals relative humidity divided by 100.

Shale Handling

Obtaining Oil Field Shale Cores. It has been shown (Chenevert, 1969) that a major driving force for transfer of water between drilling mud and shale is the imbalances of the aqueous chemical potentials (i.e., activities) between the drilling mud and the shale. The process can be eliminated (or at least greatly reduced) by using inhibitive non-aqueous muds as the coring fluid (Chenevert, 1970). Such inhibitive muds were used in the cutting of both the Alaska and the North Sea cores. When the cores reached the surface they were immediately encapsulated so as to prevent moisture transfer out of, or into, the shale from the atmospheric environment.

In the case of the Alaska shale it was sealed using a standard plastic-dip process. One-foot sections of core were dipped several times in a plastic-type coating and hung to dry before being placed into standard cardboard core boxes for shipment.

The plastic-dip process was not used to preserve the North Sea core. In this case, the core was placed inside a three foot section of plastic "coring tube" that had been filled with the pure non-aqueous base fluid used to fabricate the coring fluid. The ends were then sealed with plastic caps and the tubes shipped in standard wooden core boxes.

One major advantage of using the non-aqueous fluid method over the plastic-dip technique is that leakage of the container is easily detected, whereas a small hole in a plastic-dip coating can be overlooked.

Theoretical Consideration

The hypothesis in this study for assessing the saturation level of a shale is based on the assumption that when a shale absorbs moisture the bulk volume change for saturated and unsaturated shales will be different. The bulk volume change for a saturated shale should be directly equal to the volume of water absorbed, whereas the bulk volume change for an unsaturated shale will be less than the volume of water absorbed. Some of the water absorbed by the unsaturated shale will be used to fill void spaces and will not produce a bulk volume change. By comparing such measurements using a graph as shown in Fig. 1, it is a simple matter to determine if the shale was initially saturated. Unsaturated shales fall above the 45 degree line, and saturated shales fall on the line.

The experimental technique developed consists of a three step process: (1) measure the weight and bulk volume of the shale in question, then (2) allow the shale to slowly absorb moisture in a controlled humidity environment, then (3) again measure the weight and bulk volume of the shale.

For this study a typical shale sample had dimensions of 1/2 inch by 1/2 inch by 1 inch, and a weight of about 10 gm. They were cut from a core perpendicular to the bedding planes using mineral oil as a cleaning/cooling fluid in the cutting process. The samples were quickly washed using Hexane to remove all surface oil.

Weight measurements made before step 1 and after step 2 hydration were easily achieved using a balance sensitive to 0.001 gm. A 220,000g "Sartorius" balance was used in this study.

The bulk volume measurements for steps 1 and 3 are more difficult because of the irregular shape of most shale samples. To achieve satisfactory bulk volume measurements the "Archimedes" immersion technique was used. The shale was attached to the lower hook of the Sartorius balance by a thin thread. Its weight in air and its weight immersed in a fluid were then determined. For this study the fluid used was a mineral oil type "LVT-200," which has a specific gravity of 0.82.

The bulk volume of the shale is determined using the following equation:

$$\text{Bulk Volume, cc} = \frac{\text{Air Weight, gm} - \text{Immersed Weight, gm}}{\text{Density Of Immersion Fluid, gm / cc}}$$

In step 2, different methods were investigated for adding moisture to the shale. It was found that simply immersing the shale in water was unsatisfactory because shale failure (flaking off of particles) often occurred, and the ions within the shale would diffuse out of the shale. A second method, which adds moisture to the shale by subjecting it to a controlled high humidity environment, was then selected.

Controlled humidity environments were achieved by placing saturated salt solution in the base of a desiccator. The shale was then hydrated by placing it in the atmosphere above the solution, sealing the desiccator, applying a vacuum, and then waiting 7 to 10 days for equilibrium to occur.

After hydration step 3 was performed wherein the weight and bulk volumes of the shale were determined following the same procedure as mentioned in step 1.

The salts used in this study, and their humidity control environments, are listed in Table 3.

Results and Discussion

Saturation Studies. Shale saturation tests were reported in an earlier study (Chenevert, 1996) using shale samples that had been stored over a long period of time. All four of the shales had become unsaturated as indicated by the technique described herein. Results obtained for the Alaska shale are presented in Figure 2 as an example of results for an unsaturated shale.

As noted, the sample, which was hydrated in the 84% Relative Humidity environment, absorbed 0.13 cc of water and changed in volume by only 0.10 cc. This result represents an unsaturated condition. Similar results were obtained for samples placed in relative humidity environments of 88.5% and 96%.

For the North Sea shale, testing showed it to be saturated. Fig. 3 shows that the shale absorbed 0.25 cc's of water in a 98% relative environment and had a bulk volume expansion of 0.25 cc's. Similar results were obtained for the shale placed in the 92% relative humidity environment.

Also displayed in Fig. 3 are data taken wherein the native shale was partially dried out in relative humidity environments of 50.5% and 10%. As noted, large quantities of moisture were removed (0.25% and 0.57%) in these environments, and there was very little reduction in bulk volume (0.07% and 0.11%). It is clear that as moisture was removed void spaces were created, and the sample became unsaturated.

Restoration of Saturation

One question that often arises is "can a shale be restored to a saturation condition once it has become unsaturated?" To answer this question shale tests were run by first intentionally altering the moisture content of shale samples by placing them in relative humidity environments of 10%, 50.5%, 92%, and 98%. Next, their moisture and bulk volume changes were measured. They were then restored to their native relative humidity environment of 84%. The final step was to measure their moisture and bulk volume contents. Figures 4 and 5 show results obtained. Note that when the moisture content was slightly altered from 84% to 92% relative humidity, the shales were nearly restored to their original moisture content and bulk volume. However, when the moisture content was highly altered, the shales did not return to their original state.

Swelling Response.

Another question to be answered is, "Do restored shale experience the same swelling behavior as native shale?" To answer this question the restored shale which had undergone the hydration/restoration cycle shown in Figs. 4 and 5 were tested for their swelling behavior when placed in saturated solutions of KCl. A detailed description of the swelling test used can be found in Chenevert (1990).

The swelling response experienced (see Fig. 6) shows that altered shale experienced a higher swelling behavior than native shale, even when they had been restored to their native water activities.

A final study showed how hydrated/dehydrated shale have very different swelling behavior than native shale. As before, moisture was added/removed from native shale using the controlled environment desiccates technique. Fig 7 shows that the addition of moisture to the North Sea shale removed the swelling tendencies and removal of moisture increased swelling tendencies.

Conclusions

1. The technique presented herein is a satisfactory method for determining the presence of voids in shales.
2. The controlled humidity desiccator is a convenient method for wetting a sample without loss of materials commonly associated with direct wetting in the presence of water.
3. The technique used for coring, handling, and preserving the North Sea shale was able to maintain the saturation condition of the shale.
4. Restoring altered shale to its native water activity conditions may not necessarily restore their swelling behavior.
5. The swelling potential increases for drier samples but decreases for wetted samples. The different swelling response of the restored and preserved samples indicates some irreversible changes of the shale matrix.

Acknowledgments

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Table 1 Composition of Alaska and North Sea Shale

| Composition (%) | Alaska Shale | North Sea Shale |
|---------------------|--------------|-----------------|
| Quartz | 52 | 9 |
| Plagioclase | 4 | 0 |
| Calcite | | 12 |
| Siderite | 7 | 2 |
| Pyrite | 2 | 2 |
| Feldspar | << 1 | 3 |
| Dolomite | 1 | 1 |
| Smectite | 0 | 6 |
| Illite/ Smectite | 8 | 23 |
| Kaolinite | 10 | 22 |
| Chlorite | 4 | 2 |
| Illite | 12 | 18 |
| Clay (%) | 34 | 71 |

Table 2 - Native moisture content and water activity for Alaska and North Sea shale.

| Shale | Vertical Depth, Ft. | Native Moisture Content, % | Native Water Activity | Native Relative Humidity |
|-----------|---------------------|----------------------------|-----------------------|--------------------------|
| Alaska | 12,800 | 6.0 | 0.70 | 70 % |
| North Sea | 5500 | 7.2 | 0.84 | 84 % |

Table 3 - Saturated salt solutions used for humidity control.

| Saturated Salt Solution | Water Activity | Relative Humidity |
|---|----------------|-------------------|
| K ₂ Cr ₂ O ₇ | 0.98 | 98 |
| KH ₂ PO ₄ | 0.96 | 96 |
| KNO ₃ | 0.92 | 92 |
| ZnSO ₄ .7H ₂ O | 0.885 | 88.5 |
| KBr | 0.84 | 84 |
| Ca(NO ₃) ₂ | 0.505 | 50.5 |
| Zn ₂ | 0.10 | 10 |

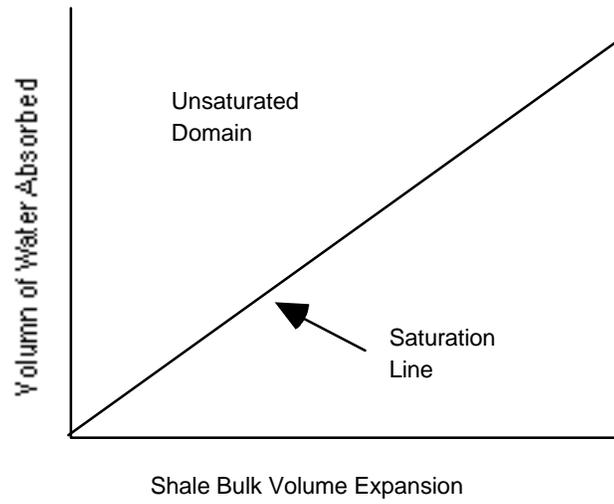


Fig. 1 - Volume-Comparison Method for Displaying Shale Saturation Status

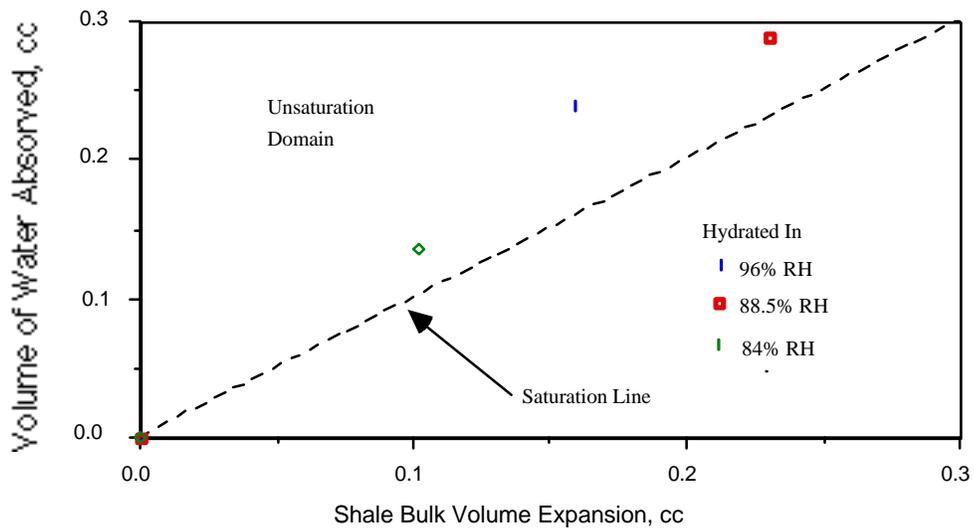


Fig.2 - Volume of Water Absorbed vs. Shale Bulk Volume Expansion for Alaska Shale

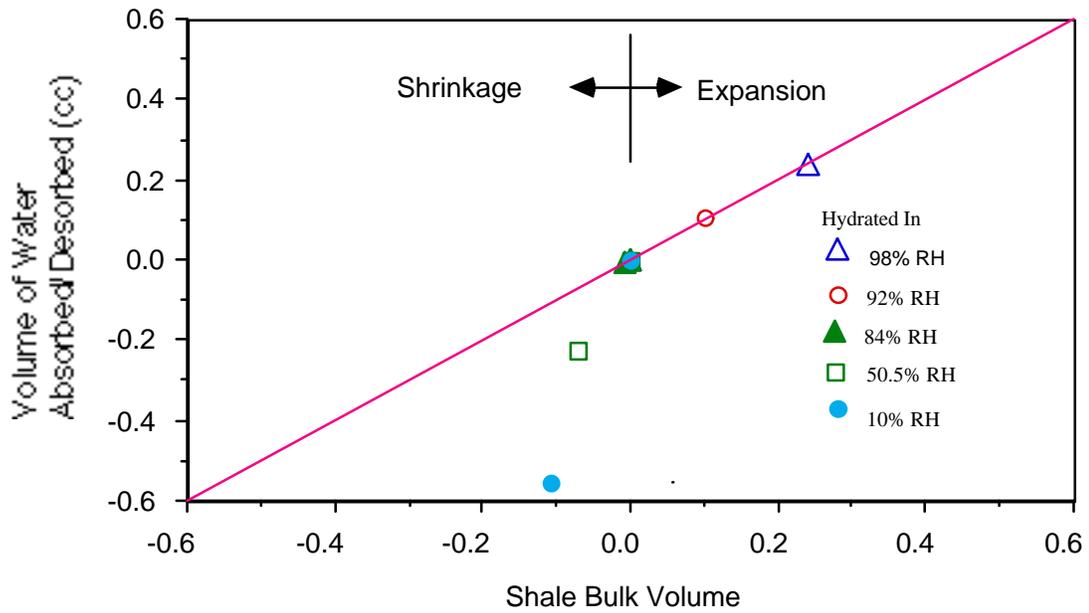


Fig. 3 - Volume of Water Absorbed vs. Bulk Volume Expansion for North Sea Shale

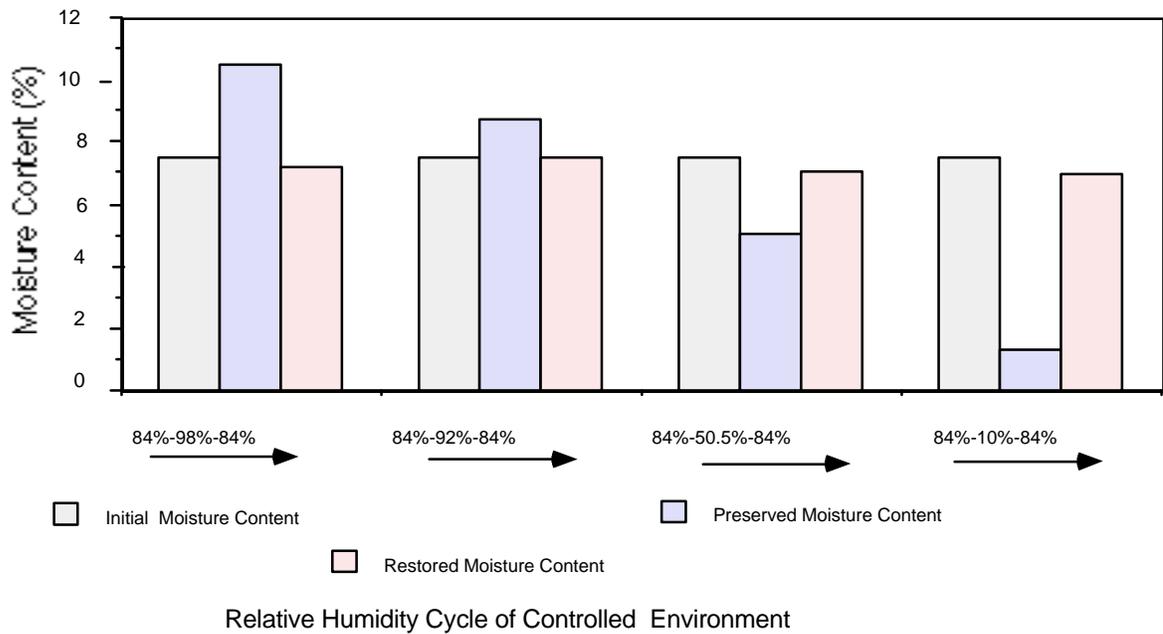


Fig. 4 - Gain/Loss Moisture Content for Native-Altered-Preserved North Sea Shale.

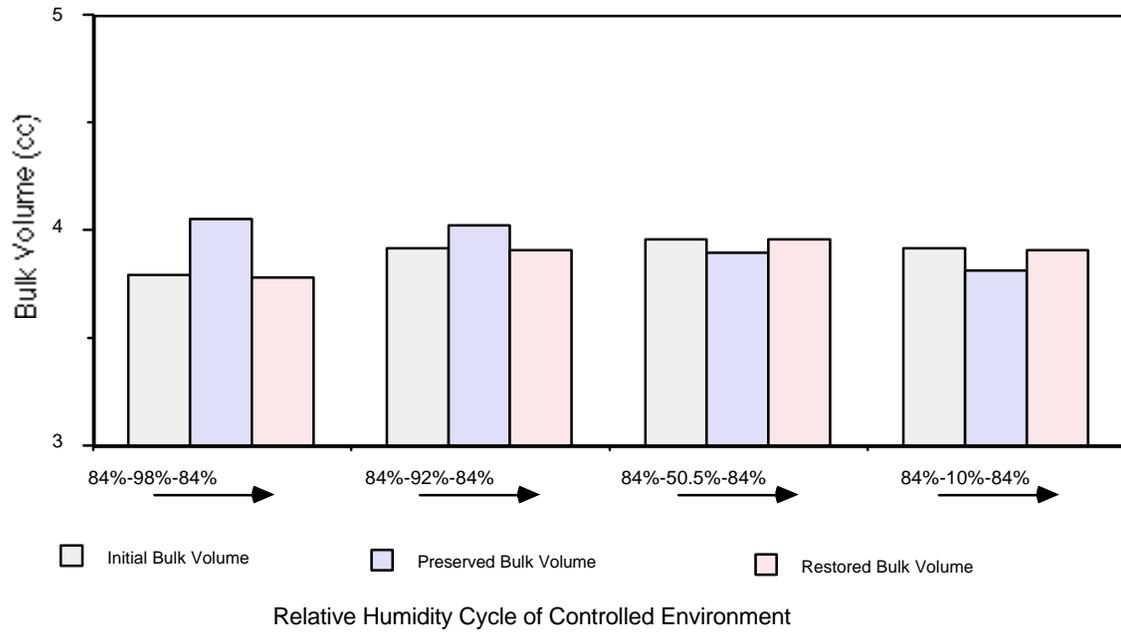


Fig. 5 - Gain/Loss in Bulk Volume for Native-Altered-Preserved North Sea Shale.

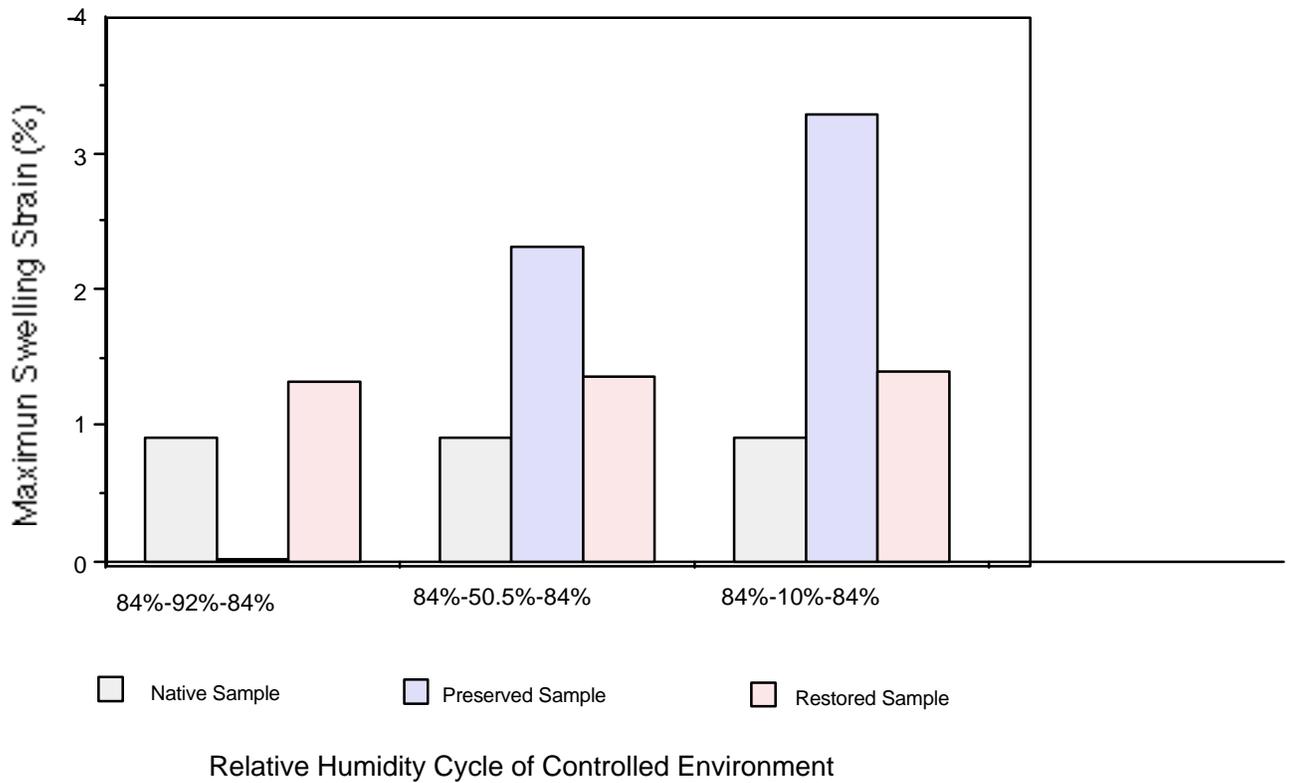


Fig. 6 - Maximum Swelling for Native-Altered-Preserved North Sea Shale

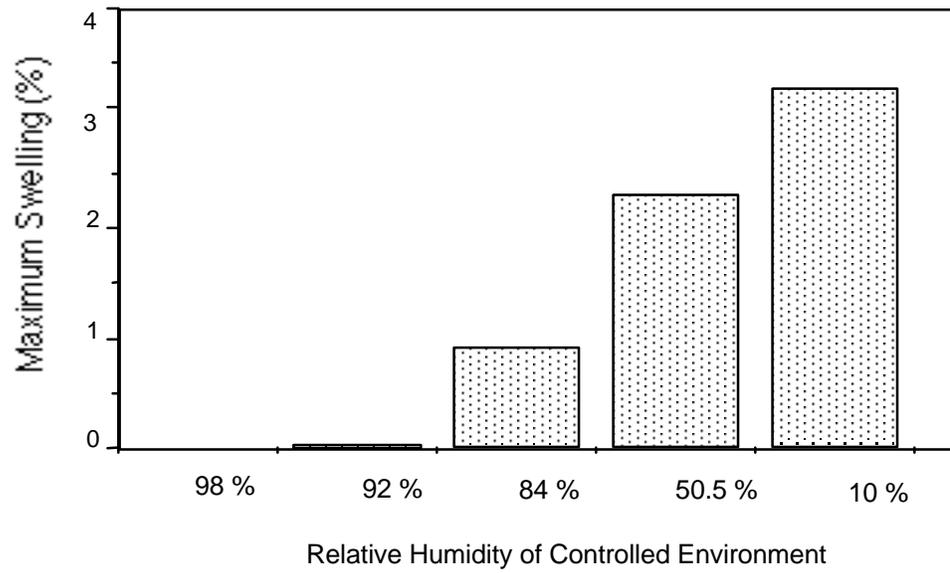


Fig. 7 - Maximum Swelling of North Sea Shale After Alteration in Various Environments