WAX CORE PRESERVATION – EXTENDED REVIEW OF EXISTING METHODOLOGIES

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ABSTRACT

Core preservation of plugs taken at the rig site or of whole core sections is a common practice during a core handling operation. This is a fundamental step for maintaining the connate fluids saturation in place and preventing the core or plug sample from drying over a long period prior to any core analysis at the laboratory.

A standard procedure for core preservation consists of wrapping the sample with plastic film and aluminum foil before dip coating it. For the purposes of this paper, the term 'wax' will be used to describe the meltable coating used to cover the wrapped core plug or whole core section. The exact composition of the wax may vary in practice but are described concerning these experiments. API recommended practices for core preservation (RP40) [1] provide guidance on the various steps from wrapping to dipping. However, at the well-site, actual procedures differ from the RP40 in terms of wrapping material type and layers and composition and properties of the waxes used. Mishandling can also occur during the operation: trapping of air or water in the wrappings or in the wax or the sample falling into the wax bath. This study reviews the effects of different materials and practices on the efficiency of core fluid preservation over one month period. Results show that wax dipping and cooling are key steps where malpractices can have a major effect on the core fluid content.

INTRODUCTION

According to the API recommended practices [1] (RP 40), one core preservation technique is wax-dipping. This method involves core wrapping with several layers of high quality and non-reactive plastic film (e.g. Saran™), then of heavy-duty aluminum foil. A wire is then tied around the core to make a handle and the core is quickly dipped multiple times in a molten wax. Between each dip, the wax is left to cool down and harden in a way to ensure a constant thickness. The wire handle is removed after the dip coating. However, well-site practices can greatly differ from the RP40 due to: tight processing deadline, limited or no access to appropriate material, or lack of training.

This study focuses on the effect of the alternatives procedures and the malpractices on the core fluid saturation. For this purpose, a desaturation test on wax-coated samples was carried out over a one month period, which typically corresponds to the elapsed time between the sample plugging at the well-site and the hotshot fluids analyses at the laboratory. Results are presented in this paper.

MATERIALS

Rock samples

Type	Size	Formation	Permeability	Porosity	Preparation
54 plugs	1.5”	Sandstone	30 to 1,500mD	17 to 32%	Soxhlet cleaning + Oven drying + Then re-saturation with water.

After re-saturation, the samples porosities contain two phases: water and air. Samples were divided in six test groups according to their properties (Figure 1) and for each group one unpreserved sample was used as a reference for fluid loss.

Preservation media

Plastic film	SARAN® film	basic catering cling film
Aluminum foil	Heavy duty foil	basic catering foil
Wax	Wax 1- low melting point at 80°C, contains paraffin	Wax 2 - high melting point wax at 140°C, no paraffin

METHODS

Plug samples with known water saturations were wax-coated according to different procedures: RP40, alternative practices and malpractices. The plugs were stored with a regular follow-up of their weight, which in some cases reflected an apparent desaturation. After one month, the wax coatings were removed and the final saturations determined.

Preservation procedures

Group 1 was discarded from the tests due to a low initial saturation of the samples.

Group 2 tested the wrapping step (Figure 3) (and water cooling) by comparing samples wrapped with variable layers number of basic material: cling film and light foil against samples wrapped with higher quality material. The next steps were carried out respecting RP40 (except sample 2.5 cooled down by plunging in cold water). Sample 2-1 was used as a reference for fluid loss on a wrapped but non-waxed sample.

Group 3 focused on practices likely to cause air or water trapping within the wrapping or the wax (Figure). These malpractices are rough wrapping of plastic film and foil creating air pockets and cooling of the wax by shaking the sample or plunging it in cold water in order to accelerate the process.

Group 4 tested the wax dipping step with different wax types, viscosities and exposure times to the hot wax material (Figure 7). In this group, samples were submitted to three quick dips in Wax 1 or Wax 2, either very fluid or viscous. In order to simulate a prolonged dip due to a sample fall, four samples were maintained in the wax vat for 1 minute instead of 1 second during the second coating.

Group 5 tested the presence of the wire handle as a potential path for fluid loss (and dipping time) for two types of wrapping material and wax (Figure 9). The samples were wrapped and coated according to the RP40, except sample 5-3, dipped for 5 minutes in the vat. After the coating, the handle wire of some samples was not removed.

Group 6 tested the compatibility of a wax to the storage temperature (Figure 1). Samples, prepared with Wax 1 or Wax 2 according to RP40 procedures, were stored at different temperatures simulating ‘hot’ (30 to 40°C), ‘temperate’ (15 to 20°C) or ‘cold‘(below 0°C) environments.

RESULTS AND DISCUSSION

We analysed our results by considering two measures of weight loss: an apparent pore water loss ($\Phi'_{\text{water lost}}$), calculated from the combined weight of wax coated core; and the actual pore water loss ($\Phi_{\text{water lost}}$), measured from the weight of the core at the end of the experiment once the wax was removed. Both are calculated as a percentage of pore volume:

Equation 1: Apparent desaturation

$$\Phi'_{water\ lost} = \frac{m_{0\ coated\ plug} - m_{coated\ plug}}{\rho_w \times V_p} \times 100$$

Equation 2: Actual desaturation

$$\Phi_{water\ lost} = \frac{m_0 - m}{\rho_w \times V_p} \times 100$$

$m_{0\ \text{plug coated}}$: Mass of the coated plug 2 hours after dipping [g]
$m_{\text{plug coated}}$: Mass of the coated plug at the day d [g]
ρ_w: density of water [g/cm^3]
V_p: volume of pore of the sample [cm^3]

m_0: Initial mass of the saturated plug [g]
m: Mass of the plug after preservation [g]

Evolution of the coated plugs weights during time - apparent desaturation

The tests revealed that Wax 2 was not stable over time; it was exuding on the surfaces where it was stored. Hence, for the plugs coated with the Wax 2, the samples weights decrease could not be correlated to a potential fluid desaturation.

Wax 1 did not appear to degrade during the experiment. Therefore, a progressive decrease of the weight for a sample coated with this wax was interpreted as an apparent fluid loss, $\Phi'_{\text{water lost}}$ (Figure 2).

According to Figure 2, most of the samples had an apparent loss of water fluctuating at 0 ±0.1%. The ±0.1 value was interpreted as the measurement error. However six samples showed a progressive desaturation trend with $\Phi'_{\text{water lost}}$ above +0.1%, suggesting fluids exchange with the atmosphere. Five of these samples were submitted to a wax cooling step differing from the RP40: samples 3-3, 3-4 and 3-6 were cooled down carelessly by shaking them, causing air trapping within the wax and heterogeneous wax thicknesses.

The apparent desaturation for the carelessly wrapped samples 3-4 and 3-6 began on day 1 and were higher ($\Phi'_{\text{water lost}}$ reaching 1.8 and 0.5% after 33 days) than for the properly wrapped sample 3-3, which only started after 25 days and reached 0.15% after 32 days. On the sample 3-4, the heterogeneous thickness of the wax left the aluminum clearly visible. Hence, heterogeneous wax layers may create preferential path for fluid migration and cause desaturation, especially if the wrapping is also done carelessly.

Two samples (2-5 and 3-5), for which the Wax 1 was cooled down by plunging them in water, showed an important drop of weight during the first day. This ‘kick-off’ in weight may not reflect the loss of sample fluids but instead a loss of wax material or cooling water might have been trapped in the wax and migrated out of the wax during the first day. Afterwards, sample 2-5 weight stabilised, suggesting the absence of exchange between the plug and the exterior, whereas sample 3-2 showed a progressive weight decrease. This may be interpreted either as ‘water loss’ or also as wax material loss.

Sample 5-3 shows a strong weight decrease trend. This sample remained in the wax vat for 5 minutes and the handle wire was left. There could be water loss via the handle wire.

Comparison of the plug weights before and after preservation - actual desaturation

Actual desaturation $\Phi_{water\ lost}$ values differ, generally higher, from the final apparent desaturation. This can be due to loss of water during the preservation operation and/or by slight loss of sample when removing it from the wrappings and/or from the partial migration of the water to the wrapping film and foil when preserved.

According to the group 2 results, the type and number of layers of wrapping material do not affect the efficiency of the core preservation once the plugs are coated with wax (Figure 4). The plugs were well preserved with limited actual fluid loss, averaging 1% of pore volume. However, only wrapping the plug with film and foil (and no wax) is not sufficient to retain fluids: for sample 2-1 the actual desaturation averaged 0.35% per day for an initial saturation of 58% fluid.

Sample 3-2, whose wax was cooled down by plunging it in cold water, gained weight, hence saturation (Figure 6). This suggests a fluid contamination from the cooling water. Sample 3-4 visibly lost fluids, with the actual desaturation greater than 2%, which is consistent with the high apparent desaturation observed during the test: the heterogeneous wax layers were not sufficient to retain fluids.

In the group 4, no big difference was noticed between the samples that were dipped in wax for 1 second each time and those that remained in the wax for 1 minute (Figure 8). However, the wax viscosity seems to be an issue for Wax 1. Higher fluid losses (samples 4-7, 4-8) occur with a viscous wax, which may be due to wax heterogeneities and air trapping.

Sample 5-3 remained in the wax vat for an extended period (5 minutes), causing the cling film to be altered (green patches), high desaturation and water condensation in the film.

The desaturation of the group 6 samples were limited when stored in cold or temperate places (<20°C). However samples stored at higher temperature had higher desaturation (Figure 12), this especially for Wax 2. When opening the packages, foil appeared visibly oxidised (moderately for Wax 1 and strongly for Wax 2, Figure 13). This suggests a transfer of sample fluid to the aluminum foil.

CONCLUSION

It appears that some practices deviating from the RP40 do show a risk of fluid loss, interaction or contamination, while others were not proven to make any difference to short term preservation. The type and number of wrapping layers were not proven to have an effect on the plug fluids. Inversely, the type of wax can be critical in a hot environment. Wax 2, despite having a higher melting point, degraded and did not prevent fluid losses in hot environment. The length of time that the sample was in the wax vat did not show to be an issue up to 1 minute but it clearly was for 5 minutes, where fluid losses and reactions with the wrapping occurred. If a sample falls into the wax vat, retrieval time is critical. Within 1 minute, the fluid saturation can still be considered as fairly reliable. Beyond that the results should be considered as unreliable. The practice of cooling down the wax in cold water may affect the wax's integrity and also result in fluid contamination. This practice, used to prevent wax expansion in case of samples rich in volatile hydrocarbons, should be carried out with great care. A suggestion would be not to cool down the first wax coating with water, but only the next coats and to ensure that the cooling water evaporates or is removed before the next dipping. As showed in this study, heterogeneous wax, air trapping and apparent aluminum foil misapplication are likely to create significant paths for fluid migration. These issues can be solved by using a homogeneous wax, maintained at the correct temperature uniformly in the hot vat and by complying with the RP40 [2] for the cooling down step.

This study focused on the quality of plug fluid preservation, however as good as the preservation may be, the elapsed time between plugging and starting the preservation is a key point in the process, especially for highly permeable samples. Water losses of unpreserved and highly permeable (1000mD) samples reached 15 to 25% of the pore volume within two hours. In order to assure the reliability and representativeness of a plug saturation, it is recommended to put in place at the well-site a report on the dip-coating operation including: elapsed time before preservation, type of wax used, problems encountered with the wax (too viscous, tendency to trap air), and the time for each dipping step.

REFERENCES

1. Recommended Practice: API RP-40, *Chapter 2.5 ‘Dips and coatings’ & Chapter 2.1 ‘Well site core handling procedures and preservation’*, 1998
2. Garcia, J.V, Kirk Petrophysics Ltd; Hurst, A. and Taylor, C. University of Aberdeen, *Laboratory assessment of the efficiency of core preservation techniques*, SCA Calgary, 2007

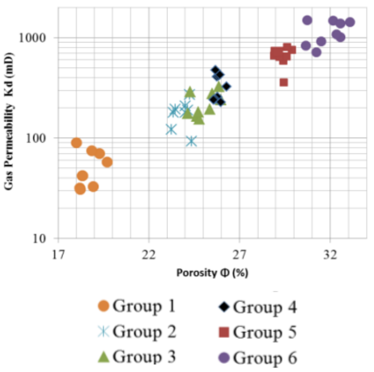

Figure 2: Samples properties

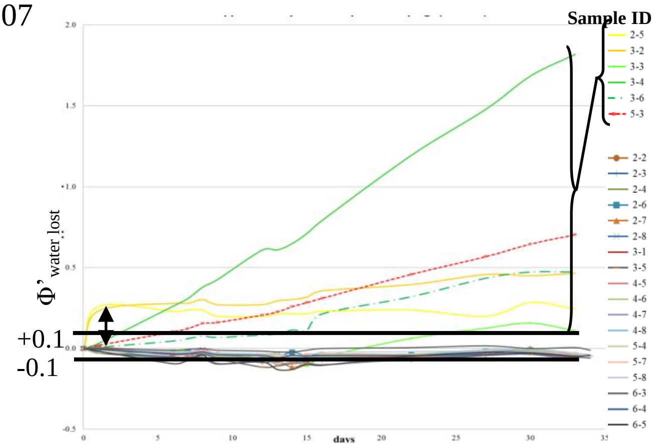

Figure 1: Apparent desaturation of plugs preserved with the wax 1

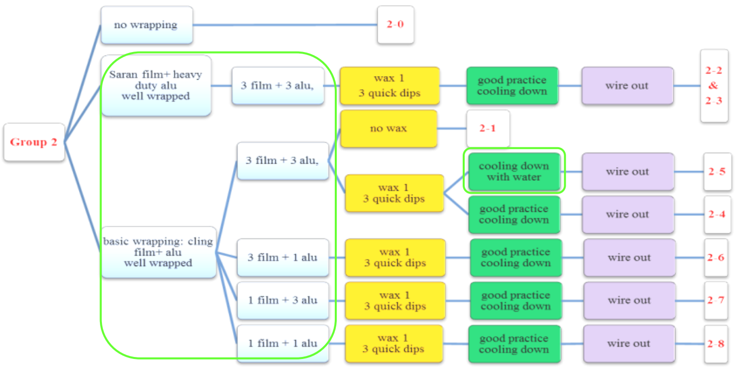

Figure 4: Group 2 procedure – impact of wrapping (and wax cooling)

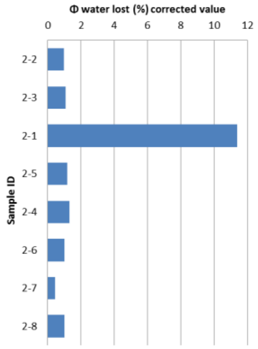

Figure 3: Plugs actual desaturation -group 2

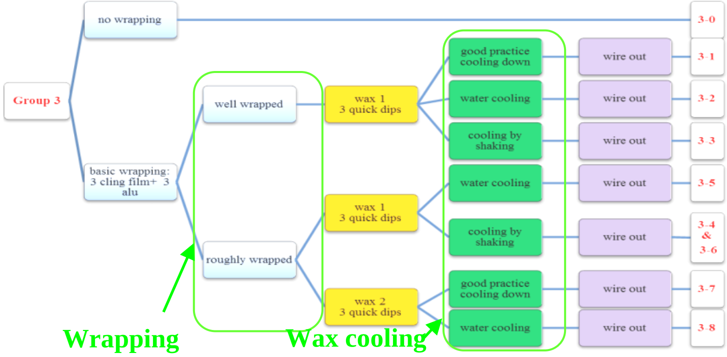

Figure 5: Group 3 procedure - trapping of air/water in wrapping and in wax

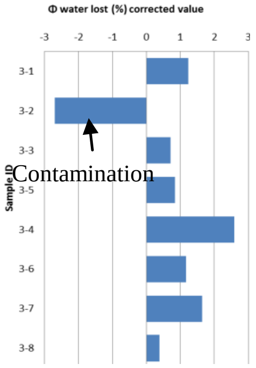

Figure 6: Plugs actual desaturation –group 3

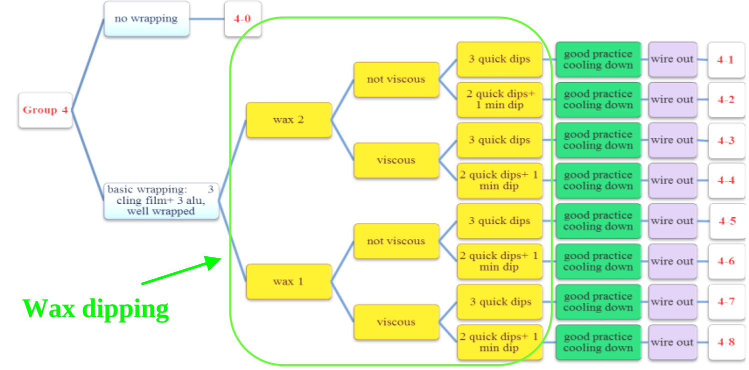

Figure 6: Group 4 procedure- wax type, viscosity and dipping time

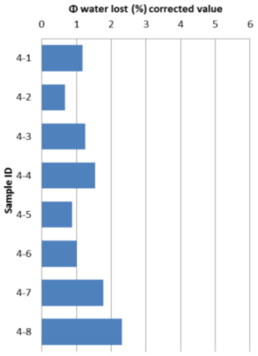

Figure 5: Plugs actual desaturation – group 4

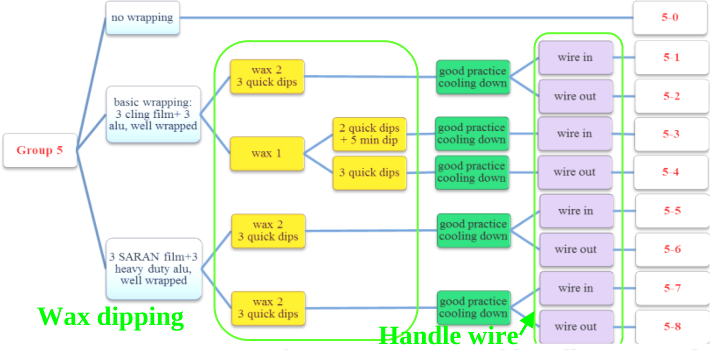

Figure 9: Group 5 procedure – wax type and handle wire in the wax

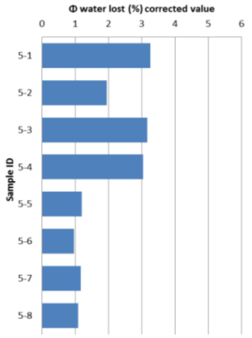

Figure 10: Plugs actual desaturation – group 5

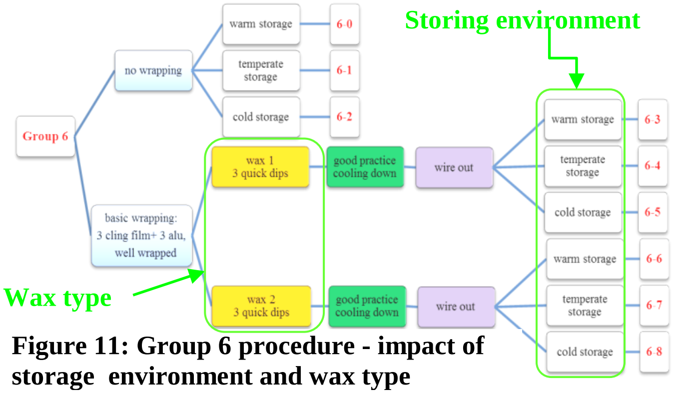

Figure 11: Group 6 procedure - impact of storage environment and wax type

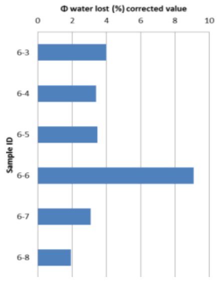

Figure 12: Plugs actual desaturation – group 6

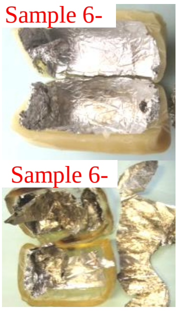

Figure 13: foil oxidation