

A New Method for Stabilization of Friable and Unconsolidated Core Samples at Well-Site

by

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ABSTRACT

In order to protect friable/unconsolidated whole cores from damage during handling and transport from the rig to the core analysis laboratory, it has become a common practice to inject a stabilizing material in the annulus between the core and the core liner. Current methods are based on a 2-component resin system in order to control hardening time. The usual chemicals contain toxic isocyanates which are able to produce surface active materials in side reactions with drilling muds and polar crude oil components.

A study to investigate the effects of commonly used resins on the wettability of core samples has been undertaken. It is concluded that some chemicals involved may have the ability to alter wettability. However, the health and safety aspects related to these materials are considerable.

A new method, using gypsum, for core protection has been developed. Extensive laboratory testing, measuring its effects on porosity, permeability, wettability and saturation was conducted, but the petrophysical properties appeared unchanged. Furthermore, the gypsum protection does not affect spectral gamma ray recordings. Several successful field tests, under various conditions, have since been performed. The method is operationally simpler than the isocyanate system and does not involve hazardous chemicals. The costs of the chemicals are low, and the pump system is cheaper, simpler and has lower weight compared to the resin system.

The gypsum is either applied as a pseudo two-component or one-component system. Field tests indicate that the one-component system is the best in practical use. Waste disposal problems are eliminated, as only water is involved in the cleaning of equipment. Neither health nor environmental risks are present.

The new, patented method should become industry standard because of its simplicity and both technical and environmental advantages.

INTRODUCTION AND BACKGROUND

General

Coring and laboratory core analysis provide important information for technical and economical evaluation of oil recovery potential. However, the information is dependent on the quality of the analysis and the condition of the core material used in the laboratory. Unconsolidated lithologies

represent a major challenge regarding handling and analysis. Poor treatment and preservation of these cores may lead to erroneous results and misinterpretations.

Several methods have been used for stabilization of unconsolidated and friable sands. Traditionally, unconsolidated core samples were frozen to allow drilling and analyzing of plug samples. Documented evidence proves that freezing of cores may change petrophysical parameters¹ and that the dynamics of freezing are controlled by salinity of formation brine, saturation, rate of freezing, and matrix properties^{2,3}.

However, techniques to avoid freezing of unconsolidated cores have been developed. In the Polymer Resin Injection method, a fast curing polymer resin is injected into the annular space created between the core and the core liner (e.g. inner barrel)⁴. Hence, the core is stabilized and sealed inside the liner. Thus, it can be handled as more consolidated rock material in pieces of one meter length.

The resin injection technique is currently the de facto industry standard for well-site stabilization of friable core sections. So far, it has proven superior to other techniques for maintaining core integrity. Injection of plastic results in less contamination of the core than the previously used waxes⁴. However, it is uncertain whether cores contacted with plastic resin are suitable for tests involving wettability or tests where surface phenomena are important. Even the originators of the Polymer Resin Injection method⁴ recommended that geochemical samples should be taken from the exposed end of core sections prior to plastic injection, and that wettability tests should be performed on one-foot sections of core cut prior to plastic injection and hand-carried to the measurement laboratory without any stabilizing agent in the annular space.

Nevertheless, as resin injection has become wide-spread, these precautions seem to be forgotten, and will at least be very impractical and expensive. In offshore operations, it is not practical to reserve enough core material for hand-carriage to land.

If the resin affects wettability, it would have important consequences since wettability would, in turn, affect most other properties measured in a typical Special Core Analysis (SCAL) programme: i.e. relative permeabilities, capillary pressures and electrical properties. Consequently, this might mean that samples for special core analysis should not be stabilized with this technique thereby complicating the core handling operation additionally. To our knowledge there has been no reported work related to resin injection and the effects on wettability. The chemistry and reactions of isocyanates are described in the Appendix.

Objectives and outline of the present work

The present work was initiated to investigate the effect of isocyanate protection on rock wettability. The experimental program designed incorporated both ideal, "extreme" cases and tests based on actual field situations. The results and the observation of the hazardous nature of the chemicals involved triggered the investigation for alternatives. Thereby, gypsum turned out to be the best candidate. A series of lab. experiments was performed on the new material. Later, the method was successfully tested under differing field conditions. It has now been patented world-wide⁵. In the following, a short description of the wettability investigation is presented. Further, different materials considered for core protection are listed and discussed, and finally laboratory and field tests of the Gypsum Core Protection Method (GypCore™) are discussed.

EXPERIMENTAL

Wettability investigation

Wettability effects - oil phase

A simple method was developed for screening the effect of isocyanate derivatives on rock wettability. To generate possible surface-modifying derivatives from crude oil and isocyanate reagents, crude oil from Field A was filtered and then isocyanate reagent was added. After 24 hours the oil was again filtered and cleaned, water-wet 600 mD Berea core samples were saturated with this "modified oil" (MOIL). The Berea plugs were aged in this oil overnight and then displaced with filtered, untreated oil (UOIL). Approx. 20 pore volumes of UOIL were used in order to secure a good displacement of the MOIL.

Another set of Berea samples was saturated with untreated oil (UOIL) for comparison. All samples were placed in Amott-cells and allowed to imbibe synthetic formation water from Field A at ambient conditions. Differences in the imbibition behaviour between cores treated with MOIL and UOIL would indicate that isocyanates react with crude oil which generates permanent wettability changes. Figure 1 shows the imbibition characteristics. The samples treated with MOIL all have a slower imbibition and a lower end-point total imbibition, with the exception of one sample which had an equally high total imbibition as the reference plugs. However, also for this plug the imbibition was slower. It is obvious that the interaction of the MOIL with the rock has changed the surface properties thereby making the rock less water-wet. Moreover, during oil/isocyanates mixing an insoluble sludge remained thereby indicating that the initially soluble isocyanates may create insoluble oil derivatives which, most likely, would precipitate onto the core material.

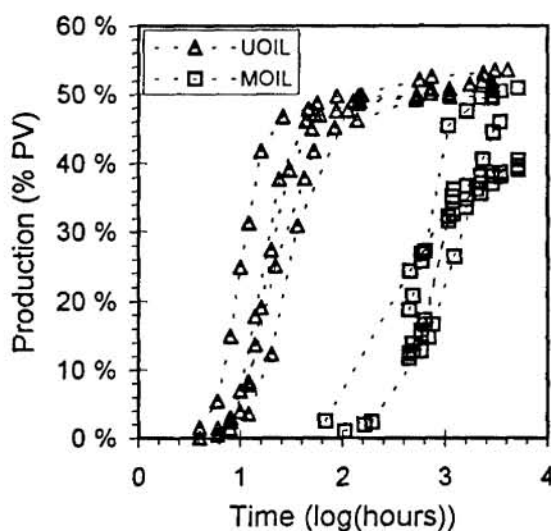


Figure 1 Imbibition characteristics of Berea cores treated with MOIL compared with cores saturated with UOIL

Attempts to measure differences in contact angles using the moving plate technique⁶ and quartz as the solid surface produced inconclusive results (see Table 1). The water phase was synthetic formation water, SFW, (Field A) and modified (MOIL) and untreated crude oil (UOIL), respectively.

Table 1 Contact angles between SFW and modified (MOIL)/unmodified (UOIL) crude oil at 25°C

	θ_A	θ_R
SFW/UOIL	31	17
SFF/MOIL	21	19

θ_A , θ_R = Advancing and receding contact angles after long time equilibration (40 days).

Wettability effects - water phase

Synthetic formation water from Field A was treated with isocyanate monomer and an amine catalyst before being injected into cleaned, water-wet Berea core samples. The water is hereafter denoted "modified water" (MWATER). In parallel, another set of Berea samples was saturated with untreated synthetic formation water (UWATER). All samples were then centrifuged in crude oil to obtain the irreducible water saturation, and placed in imbibition cells for monitoring of imbibition characteristics. Differences in imbibition between samples saturated with MWATER and UWATER could not be

observed. However, this could be due to the experimental set-up itself. During reaction of isocyanates and water it was observed that the glass vessel used for the reaction obtained a hydrophobic coating. Consequently, there may have been little surface active material left in the reacted water. Further testing was, however, not performed.

Wettability effects - field cases

Core samples from two fields (A and B) were tested with the objective to compare samples treated with isocyanates at well-site to untreated samples. Due to the number of plugs and the conditions of the core material, no firm conclusion could be drawn.

Wettability effects - overall discussion

The results indicate that components with a strong affinity for mineral surfaces are generated when isocyanate resins contact crude oil components. These new substances are not removed from the mineral surfaces by washing with untreated crude oil, and seem to be able to change the wettability of the Berea core samples from strongly hydrophilic to less hydrophilic.

These substances are difficult to determine, but they are expected to be urea or urethane derivatives of the S,N,O-fraction in the crude oil. A second class of components may arise from reaction between naphthenic acids and isocyanates, while a third class could result from hydrolysis of isocyanate resins by water remaining in the cores after stabilization. In the latter case, health hazardous amines may be produced.

Isocyanates - environmental and health considerations

The uncertainties related to generation, spreading behaviour and insidious health effects of polyaromatic amines creates a workplace hygiene problem when using MDI/PMDI in core stabilization systems (see Appendix for more details about isocyanates).

Slabbing of core material is problematic, as this procedure - and especially dry slabbing, may expose workers to monomeric Isocyanates as well as the parent amines formed upon hydrolysis. The products released may pose dangers of inducing allergic reactions as well as toxic effects related to liver function and oxygen transport in blood. Exposure to such polyaromatic free amines may, in addition, cause cancer.

Core resination is performed under greatly varying conditions, and the involved personnel are not always trained to take the necessary precautions. Overall, handling of dangerous materials should be avoided and therefore, a substitute for the isocyanate resin was sought.

Alternative materials

The ideal substitute for the resins should be non-toxic, non-corrosive, create no effects on wettability and petrophysical properties and possess sufficient chemical and mechanical stability. The curing time should be adjustable and not very sensitive to temperature. Overall, such systems should stabilize cores without use of aggressive chemicals or liberation of dangerous substances during core handling and analytical work. There are several possible systems for stabilizing friable core material. The following candidates were investigated and compared with the MDI/PMDI isocyanate system:

1. Aliphatic or cycloaliphatic isocyanates
2. Acrylates used in dental repair
3. Other acrylates (loctite type)
4. Crosslinked nylons
5. Cyanoacrylates (superglue)

6. Zink - eugenol cements
7. Zink - polyacrylic acid cements
8. Glass ionomers
9. Aluminosilicate cements
10. Zink - phosphate cements
11. Gypsum ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$).

For pure stabilization purposes, all the above-mentioned are suitable. After evaluation of carcinogenic potential, allergy inducing potential, expected influence on wettability, mechanical properties and cost of material storage/transport, gypsum was selected. The material is cheap and easy to prepare, pumping the slurry (Plaster of Paris) is easy, and the hardening time may be adjusted from a few minutes to several (up to eight) hours by addition of various additives.

The Gypsum Core Protection - Laboratory evaluation and field tests

A series of tests were conducted for testing gypsum as a core protection material.

Types of gypsum

Several types of gypsum are available. Dry-burned β -gypsum is the best suited for core protection. Due to the small crystals obtained during dehydration to β -gypsum (formula $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$) it will harden faster than other types. The porous and permeable product goes through a slight volumetric expansion during hardening providing a superior stabilization of the core due to the ability to lock the core to the core barrel.

One- and two-component systems - accelerators and retarders

A mixture of gypsum ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$) and water (H_2O) in a ratio of 60/40 by weight is referred to as *100% mixture with respect to water*. The water needed for the hardening process is only a minor portion, and the result is a product with high porosity (~ 50%) and relatively low density (~ 1.13 g/cm³). The gypsum slurry is completely hardened within approx. half an hour. Different chemicals are available as *retarders* for the hardening of gypsum. The purpose of adding such retarders is to keep the gypsum slurry in the liquid phase for an extended period of time after mixing. Thus, larger batches of fluids can be made and hardening is avoided in the pump and vessel system. The most commonly used retarding polyvalent acid is citric acid and its salts, but other polyvalent acids and their salts may be used as well.

If required, the effect of a retarder may be eliminated by adding a soluble polyvalent cation (e.g. FeCl_2) to form a stable complex which neutralizes the effect of the retarder. If this is done just prior to injection of the material into the annulus, a rapid set of the gypsum ensures rapid stabilization of the core material.

For practical use when adding accelerators to the system this may be mixed with some of the water to give a *pseudo-two-component-system* where one component is the gypsum slurry with retarder and the other component is water and accelerator. For instance 90% of the water needed in the process may be added to the gypsum powder and 10% to the accelerator. This is done for practical mixing purposes due to the small amounts of accelerators needed.

Hardening time

An extensive program was carried out for testing the hardening time. It is possible to add more water than referred to as the "100% state" in order to make the viscosity lower. Up to 40% extra water could be added without producing free water during hardening. The following parameters were varied:

1. The concentration of water in a one-component system.
2. The concentration of water in a pseudo-two-component system.
3. The concentration of added retarder.
4. The concentration of added accelerator.
5. The temperature while hardening.

It was later found in field tests that the one-component system without accelerator was best for practical reasons. The hardening time was sufficiently rapid even when using a retarding additive alone. The results of these test series are, therefore, only given as conclusions/statements;

1. Adding more water did not influence the gelling time, the hardening time or the time necessary for obtaining full strength. However, adding more than 40% water will produce free water and should thus be avoided.
2. Adding retarders increased the hardening time from 17 minutes (0%) to 119 minutes (0.08% by weight). Figure 2 shows the results of these tests.

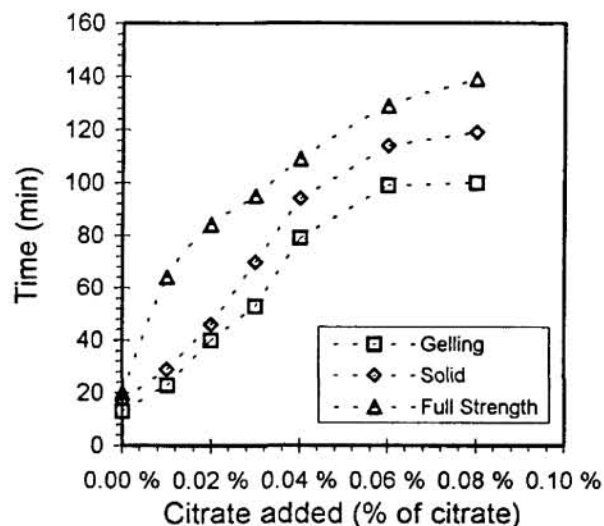


Figure 2 The effect of retarder on hardening time for one-component systems with 100% water

for non-Newtonian fluids. The viscosity is thus dependent on the rotational speed. Figure 3 shows the results at one speed. It is seen that the viscosity can be significantly reduced by adding water (Base case = 100% water).

Colour

The hardened gypsum is white to light yellowish. The colour is ideal for sandstones as it is clearly distinguishable from the rock matrix. If, for some reason, the method is going to be used for limestones or carbonates, water soluble colour additives are available.

Mechanical strength

Gypsum appears to have the necessary mechanical strength to support the core during transportation and storage. The strength of the one-component system (gypsum/water/retarder) and the two-component system (gypsum/water/retarder + accelerator) with increasing water content was measured in ResLab's rock mechanics laboratory. The samples were dried in a heating cabinet (30°C) for several

3. Adding accelerator decreased the hardening time from 70 minutes (system containing 0.04% citric acid) to about 8 minutes.
4. The process is temperature independent in the range of 2°C to 60°C. (Hardening time is constant and approx. 70 minutes in a system containing 0.04% citric acid).

Viscosity

A gypsum slurry with 100% water appears like "milkshake", a rather viscous fluid. If the space between the core and the core barrel is narrow and pumping is a problem, it may be desirable to lower the viscosity. Viscosity was measured in a one-component system (containing 0.04% citric acid) with a standard viscosimeter

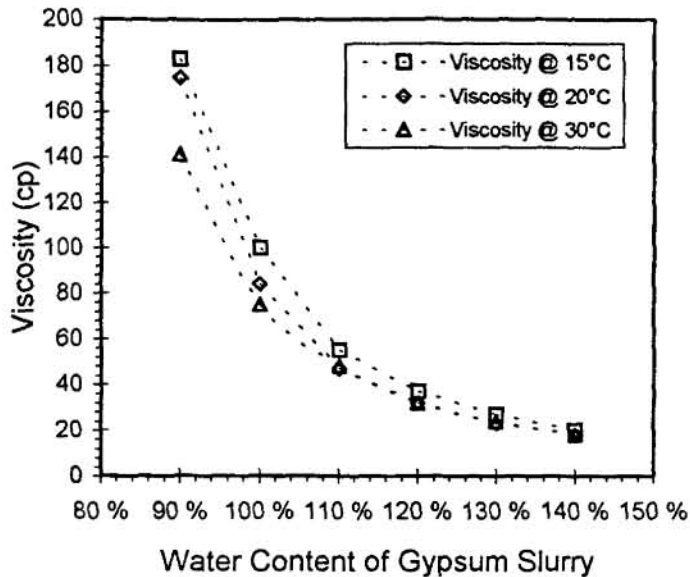


Figure 3 Viscosity of a gypsum slurry (at 300 rpm) vs. temperature and water content

days prior to measurement. Acoustic velocities for the different samples were also measured. The results are shown in Table 2 and indicate;

- One-component systems give higher final strength than two-component systems.
- Increasing contents of water gives higher porosity and lower final strength. The strength was, however, still sufficient.
- Hardened gypsum shows elastic behaviour after failure.
- Acoustic velocity results correlate with strength data.

Table 2 Elastic parameters and strength data

Sample	% Water	System	Conf. Pressure (MPa)	Young's Mod. (GPa)	Poisson's Ratio	Peak Stress (MPa)	Acoust. Vel. (m/s) at 2 MPa Ax. Stress
1A	100	2-comp	2.0	3.1	0.15	12.6	2320
1B	100	1-comp	2.0	4.3	0.29	18.0	2520
3A	110	2-comp	2.0	2.3	0.20	9.1	2060
2B	110	1-comp	2.0	3.4	0.18	14.3	2330
5A	120	2-comp	2.0	1.8	0.16	7.9	1990
3B	120	1-comp	2.0	3.2	0.17	12.3	2300

Core gamma logger response

Two synthetic core samples with diameter of 4" and 1 meter length were made from clean, dry and graded sand with epoxy as binder. After placement in core barrels, gypsum was injected into the annulus between the core barrel and one of the cores and gamma ray response was recorded along the core. The measurements show that the gamma response is slightly attenuated, but this is not a problem as sensitivity of the logger can be increased to negate the effect. See Figure 4.

Invasion depth

A synthetic high permeability (40 Darcy) sandstone was made. The gypsum slurry was injected applying a slight hydrostatic pressure. After slabbing, invasion depths of 1 - 2 mm were found. This is much less than what is seen when using resin systems, and is fully acceptable for later analysis.

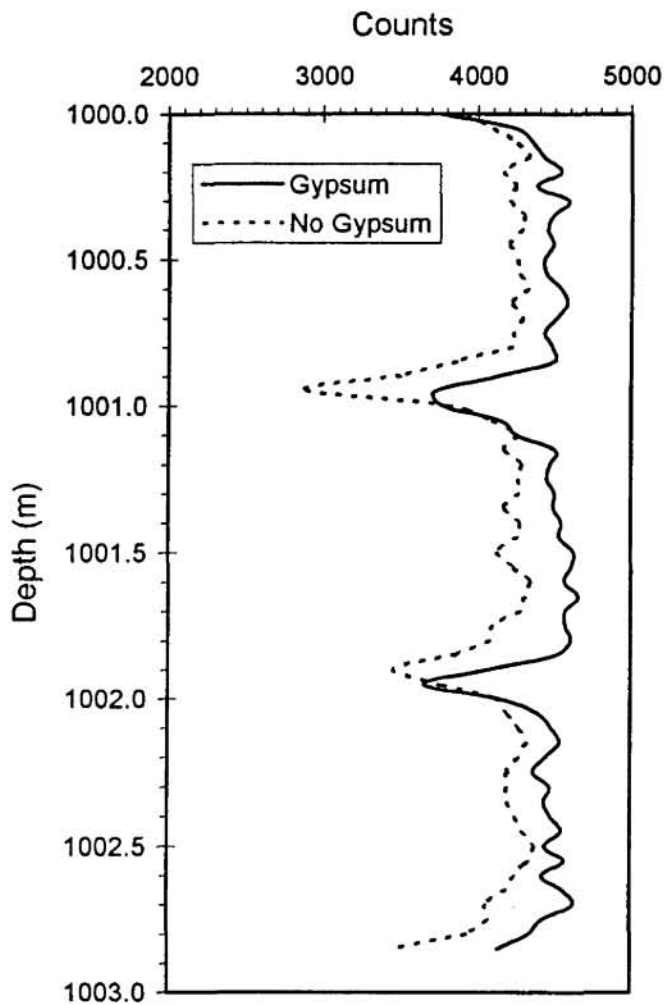


Figure 4 Core gamma logger response on the same core with and without gypsum in the annulus

Porosity/permeability effects

Porosities and permeabilities were measured on high permeable (40 D) and 40% porosity synthetic core material before and after gypsum protection. No differences were measured.

Investigations on environmental surroundings

Gypsum can be used in all types of climate as long as the gypsum powder is maintained dry at all times before use and the temperature is above freezing. In cold climate heated water should be used and the curing performed above 0°C. However, the hardening time itself is not temperature dependent.

Several tests were performed in the laboratory to simulate field conditions. In all cases a synthetic 4" diameter core of 1 meter length was made and gypsum protection was applied when the annulus between core and barrel was filled with crude oil, brine, water-based mud (KCl-mud) and oil-based mud, respectively. In all cases the results were satisfactory and the bound between the core and the barrel was good. The KCl-mud, however, decreased the hardening time somewhat.

Wettability considerations

The pH of gypsum slurries were measured in order to investigate possible chemical reactions between slurry and core fluids. The tests showed that gypsum slurries are near neutral using ordinary tap water in the mixture (pH of tapped water = 9.5, pH of water with 0.04 % citrate mixed with gypsum = 7.8). During the reaction of water and gypsum, no excess water is produced, neither is water or fluids extracted from the core samples. The gypsum contains no surface active material and no "side products" are created during reaction. It is, therefore, not expected that the gypsum in any way will influence wettability of the core samples. The gypsum, once hardened, will create an impermeable barrier to flow so that it will help maintain the conditions of the core samples as they were received at the drill-floor. An investigation of possible wettability effects, however, has been initiated.

Field tests

Six field tests were carried out as the final tests of the method. In the first two tests, the method was compared with the traditional resin injection method, taking every second core meter with the latter method. In the field tests different lithological formations were tested, so were different operational

conditions, such as mud type, water/oil zone, different pump systems, tilt angles during filling etc. A summary is seen in Table 3. Based on the results, the following recommendations were found;

- A peristaltic pump system is recommended due to its simplicity and low weight.
- The one-component system is preferred because it is operationally simpler and give higher final strength and acceptable low hardening time (approx. 70 minutes).
- The core sample should be placed in a near vertical position when filling the gypsum by pumping from the bottom.

Table 3 Summary of Field Tests

<i>Field test no.</i>	<i>Lithology</i>	<i>Type of reservoir/zone</i>	<i>Type of mud</i>	<i>Comment</i>	<i>Result</i>
1, Offshore Norway	Cons. & uncons. sand, shales	Oil & water	KCl/Polymer	Alternating Resin & Gypsum	Fair with both methods
2, Offshore Norway	Shale	Water	KCl/Polymer	Alternating Resin & Gypsum	Fair
3, Offshore Norway	Cons. & uncons. sand, shales	Oil & water	KCl/Polymer		Very good
4, Offshore Norway	Cons. & uncons. sand, carbonate chem. layers, shale	Oil & water	KCl/Polymer		Very good
5, Offshore Norway	Cons. & uncons. sand, carbonate chem. layers, shale	Water	KCl/Polymer		Very good
6, Offshore Italy	Sand Sand, shaly sand	Gas	Water-based Oil-based		Very good

Following the operational procedures developed the gypsum method has given excellent field results, and it has several advantages over the traditional resin injection method. A photograph of slabbed core surfaces after gypsum injection is shown in Figure 5. There is excellent filling (white colour) in the space between the core and the inner barrel, and on close inspection, it is seen that the gypsum neither invades the core, nor fills the small drilling-induced fractures.

CONCLUSIONS

- A method for core protection using gypsum has been developed and tested in the field with excellent results. The method is patented world-wide. All chemicals used are non-toxic, and there are no environmental loads involved.
- Gypsum constitutes a perfect seal to the core material, and does not affect petrophysical properties. Neither is it expected to influence wettability.
- The method is simple to perform and the material is cheaper than resin and easily available throughout most parts of the world. The pump system is also commercially available, simple and portable. The reaction is not temperature dependent in the range of 2 - 60°C.

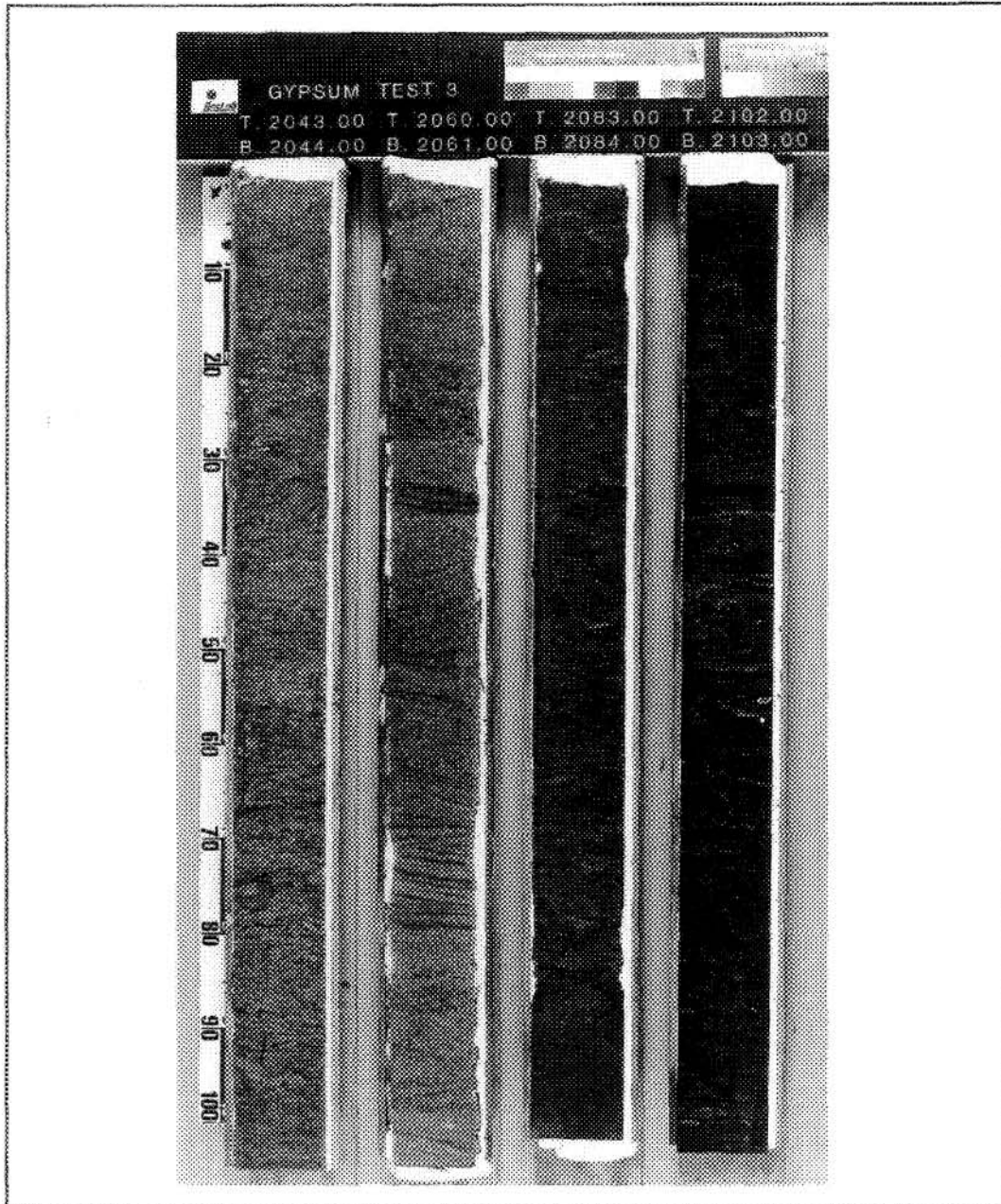


Figure 5 Photographs of slabbed cores protected by gypsum

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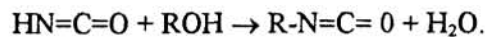
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APPENDIX

Isocyanates, various reaction products and health aspects

Organic isocyanates are esters of isocyanic acid ($\text{HN}=\text{C}=\text{O}$) and alcohols where water (H_2O) has been eliminated to produce the organic ester of isocyanic acid or the isocyanate ($\text{R}-\text{N}=\text{C}=\text{O}$):



The organic isocyanate group ($\text{R}-\text{N}=\text{C}=\text{O}$) is one of the most reactive organic functional groups and hence the chemistry of these compounds is extremely complex and diverse.

An isocyanate group will react with nearly all functional groups present in organic materials. This includes itself and all functional groups containing active hydrogen such as: $-\text{NH}_2$, $=\text{NH}$, $-\text{OH}$, $-\text{SH}$, $-\text{CONH}_2$ or $-\text{COOH}$. Such active hydrogen may be found in water, polar crude oil substances (N,S,O, Naphtenic acids), as well as additives in drilling fluids. Reaction with so-called "active" hydrogen is a general reaction for all isocyanates, either they are of aromatic or of aliphatic type. The active groups mentioned vary widely in reactivity, but amines react faster with isocyanates than any other group.

The isocyanate resin consists of a mixture of MDIⁱ and PMDIⁱⁱ. Hydrolyses of MDI with water results in the formation of carcinogenic and hepatotoxic diaminesⁱⁱⁱ. In optimum conditions, the diamines

ⁱ MDI = Diphenylmethane 4,4'-di-isocyanate and Diphenylmethane 2,4'-di-isocyanate.

ⁱⁱ PMDI = Poly-MDI.

ⁱⁱⁱ Di-amines i.e. 4,4'-diaminodiphenylmethane and 2,4'-diaminodiphenylmethane.

would further react with excess isocyanate to form a stable and harmless product. However, especially in the presence of - often used - alkaline drilling mud, the reaction may end prematurely and leave unreacted diamines within the core material. If absorbed by the human body, the diamines (or their metabolites) may intercalate between pairs of DNA and introduce errors in copying the DNA/RNA base sequences. This is a potential carcinogenic process that also may lead to birth defects. The amines are, however, not easily absorbed through intact skin.

Crude oils contain active hydrogen through carboxylic and phenolic oxygen (-COOH, -OH), primary and secondary amine nitrogen (-NH₂, =NH) and thiophenol- (-SH) groups. In extreme cases, asphaltenes may contain tertiary carbon atoms that are activated by aromatic substitutes so that the remaining carbon-hydrogen bond must be considered an active hydrogen (R₁R₂R₃C-H). The reaction between organic isocyanates and primary or secondary amines is usually the fastest and most favoured.

Of these two amine classes, only secondary amines are usually present in any practical amounts as heteroatomic ring structures in the asphaltenic crude material. The main type of structure is the secondary amine group in pyrrole rings as represented in pyrrole, indole and carbazole derivatives of the S,N,O-fraction in crude oil. In sour crudes, the amount of hydrosulphide (H₂S) and thiophenol (R-SH) groups may be so high that they will compete successfully for any isocyanates present in spite of lower reactivity than the amines.

Recommended precautions

Although most of the recommended precautions are trivial and already required for other reasons, they are worthwhile reiterating:

- When handling resinated core material, disposable gloves and protective clothing - only used once between washing - should be worn.
- Before consumption of food, workers should wash their hands very thoroughly.
- During wet slabbing, face shield should be worn; during dry slabbing, fitted dust masks should also be worn. Dry slabbing should, however, be avoided.