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# Formation Damage Caused by Drilling Muds: A Comprehensive Study Using SEM/EDS, GC/MS and Related Instruments

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## Abstract

Formation damage is commonly caused by the invasion of drilling fluid solids and/or the migration/deformation of the matrix clay minerals. Emulsifiers and polymers from the drilling fluid and/or salt crystals re-precipitated from the brine in water-based fluids can also negatively impact the fluid channels of the formation rock.

Scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS) along with gas chromatography/mass spectrometry (GC/MS) and related instruments can offer a comprehensive insight into the nature and impact of the resulting formation damage. In two independent investigations, using core plugs from lab formation damage studies, SEM photos and elemental maps visually show the invasion of barite and carbonate from an oil-based fluid, and the re-precipitated brine crystals from a water-based fluid. GC/MS data positively reveal the presence of emusifiers and polymers in the core samples invaded by the oil-based fluid. This investigation emphasizes the general methodology utilized in detecting the presence and causes of formation damage.

## Introduction

Formation damage can be caused by the invasion of drilling mud material and/or the re-arrangement of the core matrix.

Traditional microscopic study of the core samples involves the preparation of the thin sections; however, critical information is lost during sample preparation. SEM/EDS analysis of the freshly opened surface of the original core samples combines the SEM photos with the related chemical analysis and provides direct evidence if formation damage occurred.

GC/MS testing of the organic extracts of the core samples can detect the presence of mud-related compounds trapped within the core sample.

## Instrumentation and Sample Preparations

It is important to gather the basic information concerning the mud used and how the core sample was treated prior to the SEM/EDS analysis. To minimize the possible disruption of the matrix, the core sample is mechanically opened to expose the fresh cross sections (**Figure 1**). The broken core pieces are examined by the optical microscope first. The selected pieces are placed under the heat lamp to evaporate moisture and volatiles prior to SEM/EDS work.

A low-vacuum SEM can take photos up to 100,000 magnifications. The attached EDS can analyze a selected spot and area, covering elements from boron to uranium. It can perform elemental mapping over the selected area. It is not necessary to coat the sample with a conductive film. The intensity of the key elemental peaks along with the SEM photos are useful in understanding the mineralogy of the core sample and in detecting possible mud invasion (**Figure 2**).

The high-temperature GC column can heat up to 400°C for analyzing semi-volatile compounds. The attached pyrolyzer is used for analyzing semi-volatile and non-volatile organic compounds.

Selected pieces are placed in a capped glass bottle using a methylene chloride/methanol mixture to extract organics compounds for GC/MS analysis.

## Invasion of the Mud Solids

Barite and calcium carbonates are the most common solids found within the clay matrix if invasion of the mud solids occurred. Figure 2 is the EDS display of a core sample from the North Sea. It detected the presence of barite and calcium carbonate 50 mm deep within the core.

## Invasion of the Brine

**Figure 3** presents evidence of the invasion of sodium bromide (NaBr) brine within the core sample. The customer submitted the sample due to the unexpected low value of the return permeability of the core sample after an acid treatment and the production-fluid flush. It is clear that the NaBr brine/salt from the water-based mud was trapped within the pore space and effectively reduced the permeability of the core plug. **Figure 4** shows the depth distribution of the trapped NaBr brine/salt. EDS data show that the bromine content in the core plug was as high as 30% and extended more than 20 mm deep from the mud surface.

## Invasion of the Mud-Related Organic Compounds

The possible presence of organic compounds in the core sample can be detected by performing SEM/EDS analysis of the clayey matrix at different depths of the core sample. The carbon peak normally indicates the presence of organics and/or carbonates in the core sample.

Selected chunks of the core fragments are placed in a glass bottle using a strong solvent mixture to extract the organic material. The GC/MS analysis of the extracts reveals the chemistry of the extracts. **Figure 5** shows the presence of mud-related organics within the core sample. The analysis was done with the pyrolyzer[1] attached to the GC/MS.

## Migration of the Clay Minerals

Careful examination of the SEM photos might help detect the possible migration of the clay matrix. Loosely stacked or dislodged kaolinite crystals are most likely to migrate along with the inter-granular fluids (**Figures 6 and 7**).

## Conclusions

The use of SEM/EDS, GC/MS and related instruments can offer a comprehensive study of the nature and impact of possible formation damage. A successeful investigation depends on proper sample preparation, the experience level and knowledge of the analyst, and the basic information about the mud provided by the customers.

## Acknowledgments

The author thanks Steve Blattel, Greg Perez, Steve Almond and Adrienne Silvan to make this paper possible.

## Nomenclature

*NaBr = sodium bromide*

## References

1. T.P. Wampler, ed. *Applied Pyrolysis Handbook*. Marcel Dekker, Inc., New York, 1995.

## Figures

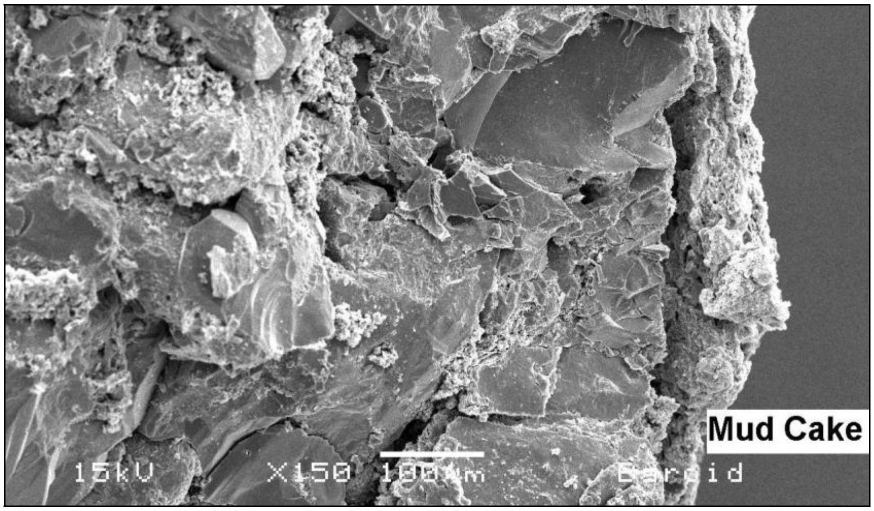


**Figure 1. To minimize the possible disruption of the core matrix, the core sample is mechanically opened to expose the fresh cross sections for direct SEM/EDS analyses.**

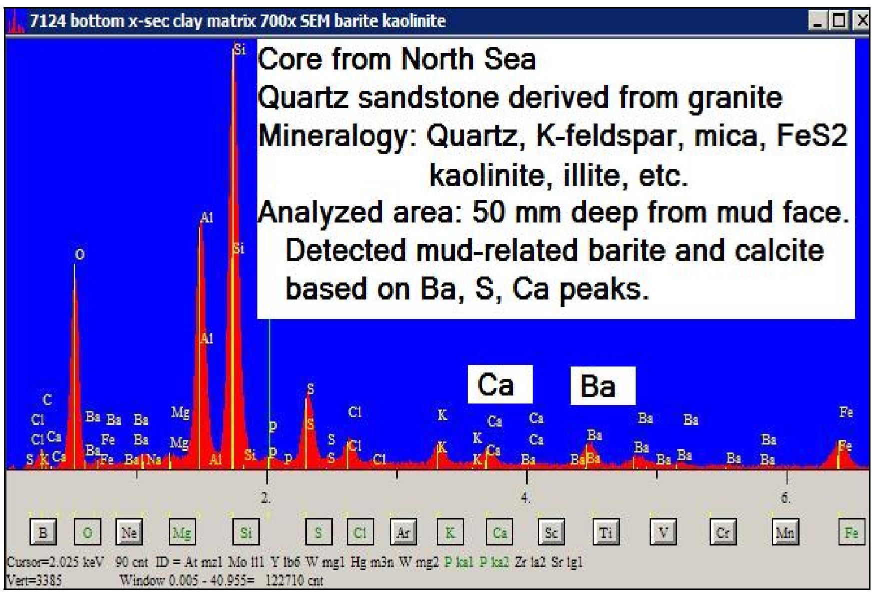


**Figure 2. The SEM/EDS can analyze selected areas and spots for their elemental composition. The presence and the intensity of the key elemental peaks are useful in understanding the mineralogy of the core sample and in detecting possible mud invasion.**

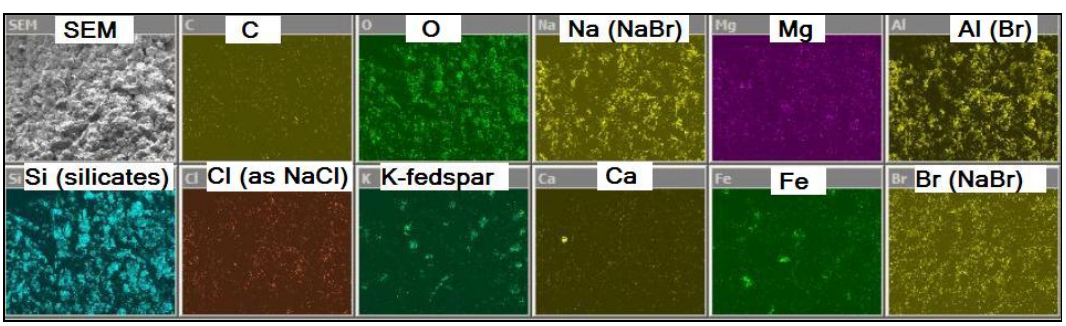


**Figure 3. The elemental maps reveal the widespread invasion of the NaBr brine. The carbon map shows the distribution pattern of the organic compounds within core plug.**

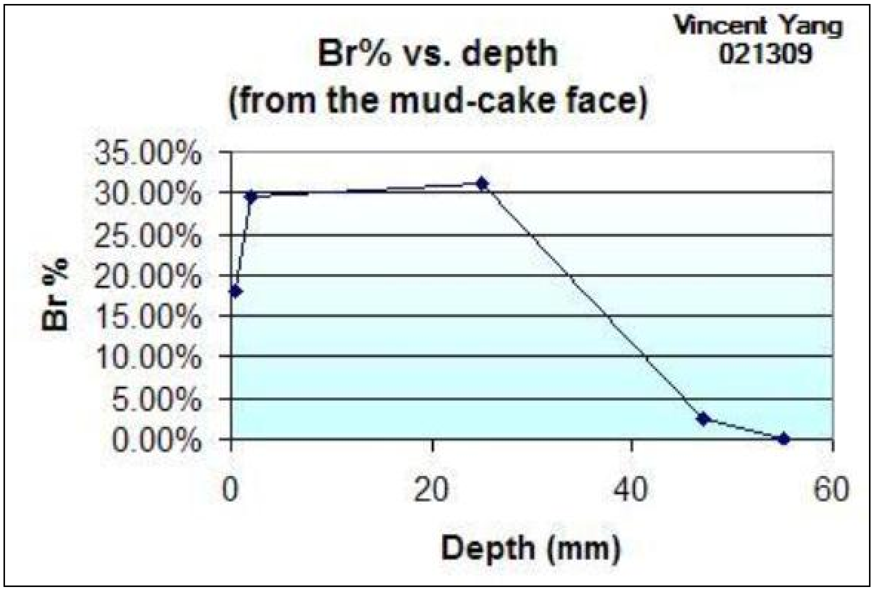


**Figure 4. SEM/EDS semi-quantitative analysis of the Br content at different depths of a customer-provided core plug shows that there is a NaBr-invasion zone approximately 30 mm thick under the mud-cake surface. See Figure 3 for the elemental maps.**

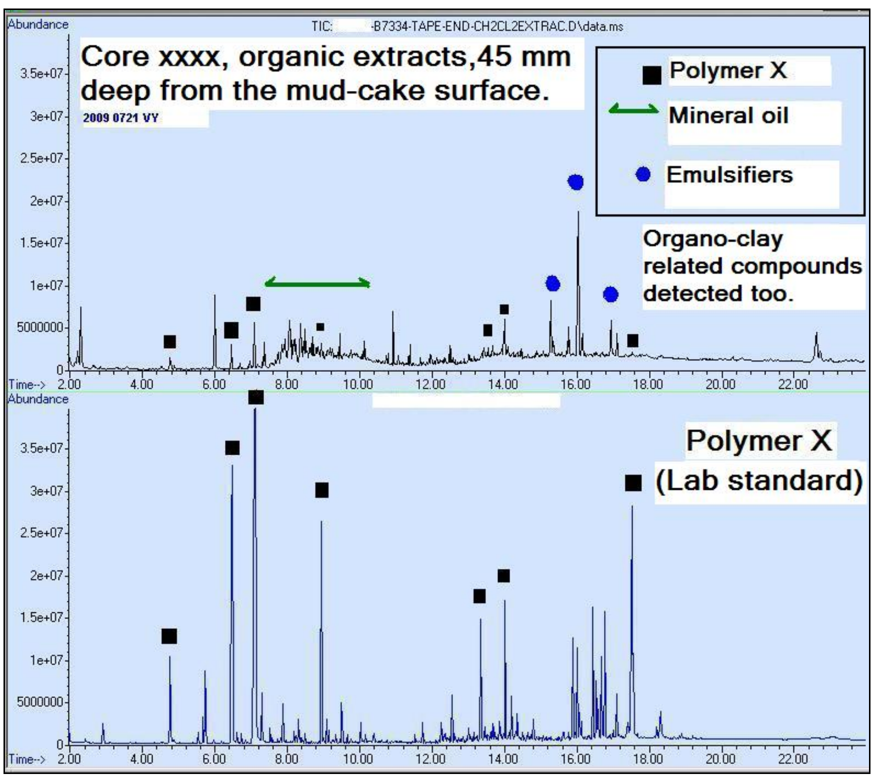


**Figure 5. GC/MS analysis of the organics extracted from a North Sea core sample 45 mm deep from the mud-cake surface. Selected ion masses (not shown) are used to ensure the marked peaks match the lab standards.**

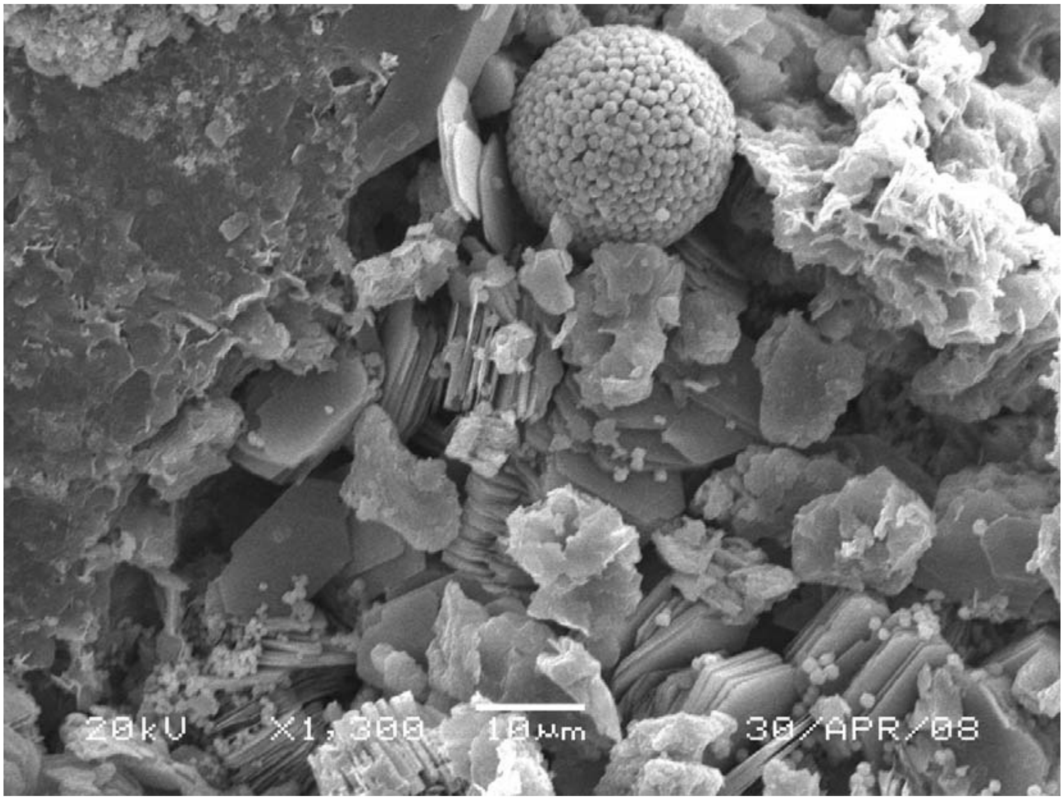


**Figure 6. Loosely stacked kaolinite flakes are ready to migrate along with the inter-granular fluids. Fine illite-mica flakes are glued together to form a small lump by organic material (upper right).**

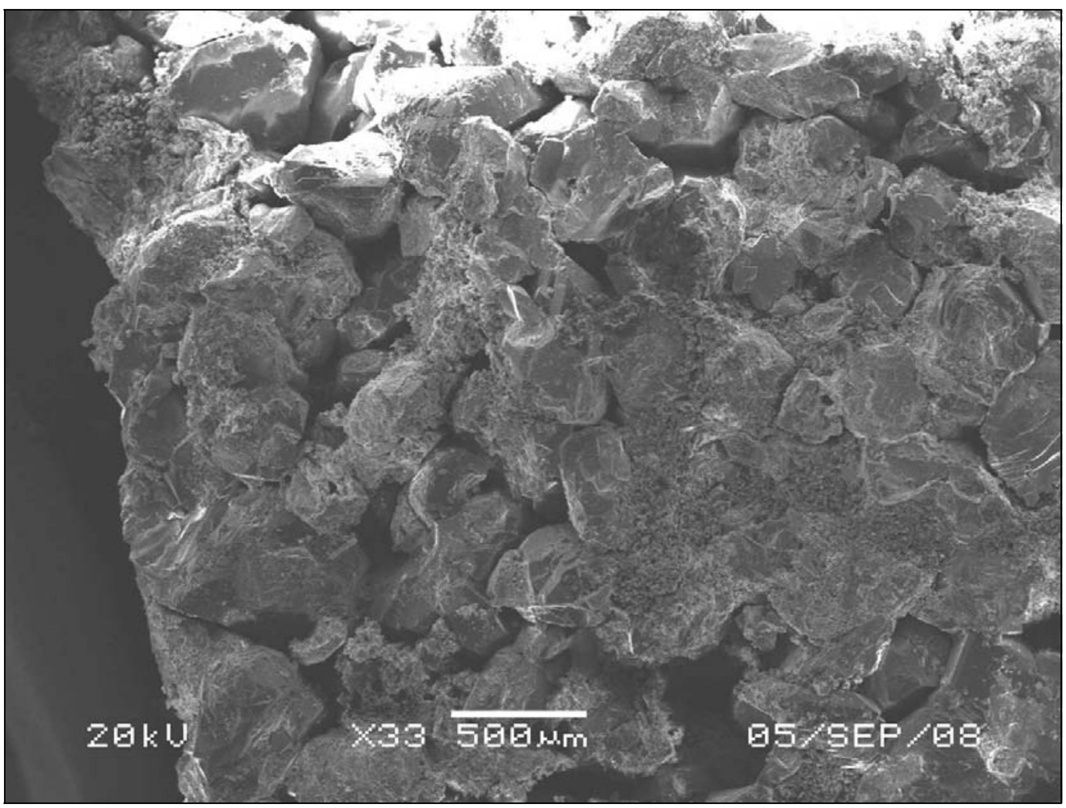


**Figure 7. In a lab-controlled test of a core plug, the return permeability increased dramatically after the high-pressure back flush of a mineral oil. It is possible that the short-distance migration of the clay matrix formed larger clayey lumps (center-right) and at the same time cleared some fluid channels (upper and lower).**