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A Simple Method for Evaluating Pore Surface Mineralogy

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ABSTRACT

Majority of mineralogical studies directed at hydrocarbon producing formations are conventional studies that treat the formation as a bulk entity. Focusing on pore surface mineralogy, which is the identification of the elemental composition of the pore surface, seems to be a more realistic approach, since fluids in the formation come into direct contact with these elements on the surface. Rock-fluid properties such as relative permeability, wettability, capillary pressure and certain rock properties, are influenced by pore surface mineralogy. Hence, characterization of pore surface mineralogy will enhance understanding of the interaction between fluid and the porous medium.

This paper discusses Multiple Voltage Scanning Electron Microscopy as a novel method for characterization of pore surface mineralogy. Multiple Voltage Scanning Electron Microscopy, a new method that has

References and illustrations at the end of paper.

successfully been used to study the surface of particles, has been implemented to identify the elemental composition of pore surface.

Results of the investigations of pore surface mineralogy for a reservoir in West Virginia is presented. This reservoir has experienced high injection pressures during a waterflooding project. The concentration of clays on the pore surface can be a possible explanation for high injection pressure.

This paper will try to direct the attention of scientists and researchers to this issue and emphasize the importance of pore surface mineralogy and its effects on rock-fluid properties.

INTRODUCTION

Identification of the mineralogical makeup of the hydrocarbon producing rocks is an old and common practice during the reservoir characterization process. Thin section analysis, in which a very thin slice of the rock is cut and

looked at, under a microscope, is usually used to identify the presence of different minerals in a reservoir rock. Although a necessary step, thin section analysis is able to identify only the presence of minerals. This method could be referred to as bulk mineralogy of the reservoir rock since the relative position of different minerals with respect to the flowing fluids are not determined. From a reservoir engineering point of view, it is of utmost importance to identify not only the minerals but their relative position in the rock. For example, a clay mineral that is present in a reservoir rock plays no important role in fluid flow characteristics as long as it is confined by other minerals and does not come into direct contact with the fluids flowing through the rock. On the other hand, a clay mineral may significantly affect the permeability and relative permeability of a rock during a water injection process if it is located on the surface of pores and becomes exposed to the injected water.

Hence, pore surface mineralogy, which is defined as identification of elemental composition, as well as the mineralogical makeup of the surface of the pores, is an important piece of information for reservoir engineers, especially when dealing with enhanced recovery processes in which non-native fluids are introduced in the porous medium.

In 1983, Ramirez, et.al.¹ presented the result of their work on identification of surface composition of Berea sandstone. In their study, Ramirez, et.al. used Auger Electron Spectroscopy (AES). AES is a surface analysis tool. It can penetrate the top few Angstrom of the surface. If the tool and expertise to work with it are available, it is an excellent device for surface analysis as shown by Ramirez, et.al. In this study, a new method is introduced to achieve similar results. This method could be

employed in the absence of an Auger Electron Spectroscopy, and shows some advantages over AES method.

NEW METHOD

In the method introduced in this paper, Scanning Electron Microscopy - Energy Dispersive X-Ray (SEM-EDX) analysis is used to identify the elemental composition of pore surface in hydrocarbon bearing rocks. This method of pore surface analysis uses the technique introduced by Wallace et.al.² in 1990 to analyze the respirable-sized dust particles from coal mines. Wallace and his colleagues at the Division of Respiratory Disease Studies at the U.S. National Institute for Occupational Safety and Health, developed this technique while researching the discrepancy in pattern of lung disease in coal miners in different parts of the State of Pennsylvania. They found that the number of cases of lung disease is inversely proportional to the amount of clay on the surface of the silicon dust particles. In other words, the higher the amount of clay coating on the silicon dust particles, the lesser the number of lung disease cases. Wallace used different incident electron energy levels of a scanning electron microscope to look at shallower depth of particles under investigation.

As far as reservoir characterization is concerned, this technique can be used to look at the surface of the rock particles, which is the parts of the rock grains that forms the walls of the pores. By identifying the minerals that are concentrated on the surface of the pores, reservoir engineers will be able to predict the possible interactions between the rock and the injected fluids.

MECHANISM

Figure 1 is the schematic diagram of the process by which Scanning Electron Microscopy identifies elemental composition of particles. In this process, an incident electron beam penetrates the sample and loses energy in inelastic collisions, some of which excite X-ray emissions characteristic of the element present. Within any layer at a given depth in the particle, the intensity of an X-ray line stimulated by inelastic electron collision with a specific element is proportional to: (a) the concentration of the target atom; (b) the intensity of the electron beam in the layer; (c) the cross-section for ionization of the target atom shell by those electrons; and (d) the fluorescence yield, which is the probability that the ionization will result in that X-ray emission.² As the incident electron energy reduces, the penetration of the electron beam also reduces. This allows the analysis of surface of the particles rather than the entire particle.

From a reservoir engineering point of view, where reservoir characterizations are of great importance, such information is very valuable. An advantage that this method has over Auger Electron Spectroscopy is that by using the multiple voltage scanning electron microscopy, one may identify the micro-heterogeneity in the rock. By micro-heterogeneity, it is meant that the heterogeneity in the rock that is detectable only in microscopic scale. For example, the rock particles that form the pore structure may be characterized using high incident electron energies, in which the entire particle is penetrated. In a homogeneous particle, decreasing the incident electron voltage and, in turn, decreasing the penetration level will not reveal any new information. In other words, what is detected in the high electron voltage level is a correct evaluation of the elemental

and, thus, mineralogical composition of the particle (Figure 2).

But, as shown in Figure 3, in a heterogeneous particle (where, for example, there is a clay coating on the particle, or clay building materials are concentrated on the surface of the particle rather than being distributed evenly throughout the particle) information provided by high voltage (which is the Conventional Scanning Electron Microscopy method) can be misleading about the nature of the particle. As shown in Figure 3, in a heterogeneous particle, the percent of aluminum available in the particle increases as the electron energy and, consequently, the depth of penetration decreases. Figure 4, shows the difference between a homogeneous and a heterogeneous particle as they are analyzed by multiple voltage scanning electron microscopy².

Beside the aforementioned advantage of SEM-EDX over AES in reservoir engineering studies, SEM-EDX has few less significant advantages over AES method. It seems that SEM-EDX devices are more accessible in universities, companies and research centers. This may be due to the fact that AES machines are considered as more specialized tools. This further emphasizes the importance of the new method by which pore surface mineralogy becomes possible using a SEM-EDX rather than AES. It is also believed that interpretation of the atomic signatures on SEM-EDX is less involved than that of AES. Another advantage of SEM-EDX over AES seems to be the implementation of the experiments, as far as sample preparation is concerned.

RESULTS

In order to show the capability of the new method introduced in this paper, some samples of a highly heterogeneous formation were analyzed. The formation is Big Injun in West Virginia. A reservoir producing from this formation has exhibit unusual behavior during a waterflood project. One of the unusual behaviors was related to the high water injection pressure. The thin section analysis (bulk mineralogy) does not suggest the presence of clay minerals in amounts that could cause such high injection pressures (observed pressures are more than twice of what they are supposed to be). Some samples from the wells experiencing high injection pressure were taken and analyzed using the method discussed in this paper. The result is presented as the aluminum fraction signals, f_{Al} , (since aluminum is a major clay building material):

$$f_{Al} = \frac{I_{Al}}{I_{Al} + I_{Si}} \quad (1)$$

which is the ratio of measured intensity of aluminum K-alpha X-ray line, I_{Al} , to that of the sum of aluminum and silica.

Therefore a higher aluminum fraction signal indicates the presence of larger amounts of the clay building materials. As can be seen in Figure 5, as the incident electron energy increases the aluminum fraction signal decreases. This means that the particles are heterogeneous in nature and aluminum is unevenly distributed within the particle. Higher aluminum fraction signal at low incident electron beam indicates that majority of the aluminum is concentrated on the surfaces of the particle. This may imply that pore walls contain more

clay that thin section analysis (optical microscopy) is able to reveal. It may be concluded that although a large amount of clay may not be present in this rock, whatever that is present, is concentrated on the surface of the pores. This could in turn mean that clay swelling has created the conditions that require high pressure in the water injection wells. Such conclusions may be helpful to the operators to consider proper remedial actions prior to water injection.

Of course, to draw such final conclusions, one needs to perform a thorough statistical analysis of the reservoir rock using this method. This is because every data point presented here represents only a particle at a certain depth in a particular well. Therefore, a few particles can not represent the state of the heterogeneity of the rock at any specific location. A statistical analysis is essential to determine the number of tests (particles) needed per foot of depth.

To get a better feel of the extent of occurrence of this phenomenon, several particles at different depth from two different wells, which are experiencing high injection pressure, were tested. The results are demonstrated in Figures 6 through 8. The results of the tests for two different incident electron energy level, namely 5 keV (kilo-electron Volts) and 11 keV are shown. It should be noted that the 11 keV electron beam penetrates the entire particle, while 5 keV electron beam only penetrates the surface of the particle. As can be seen from the graphs more aluminum has been concentrated on the surface of the particles in all cases.

CONCLUSIONS

Pore surface mineralogy provides valuable information to reservoir engineers. This information is of especial importance for design of enhanced recovery processes, where non-native fluids are introduced in to the formation. The interaction of these fluids with the rock is to a large extent a function of minerals and elements present at the wall of the pores. In this paper a simple, new method for characterization of pore surface mineralogy has been introduced. This method is capable of providing information on micro-heterogeneity of the reservoir rock.

Samples from wells that have demonstrated high water injection pressures were examined using the procedure discussed in this paper. The preliminary results showed the concentration of clay building material on the surface of the particles. These results demonstrated the existence of micro-heterogeneity in this formation.

REFERENCES

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2. Wallace, W.E.; Harrison, J.; Keane, M.J.; Bolsaiti, P.; Eppelsheimer, D.; Poston, J.; and Page, S.J.: "Clay Occlusion of Respirable Quartz Particles Detected by Low Voltage Scanning Electron Microscopy - X-Ray Analysis," Ann. Occup. Hyg., Vol. 34, No. 2, pp. 195-204, 1990.

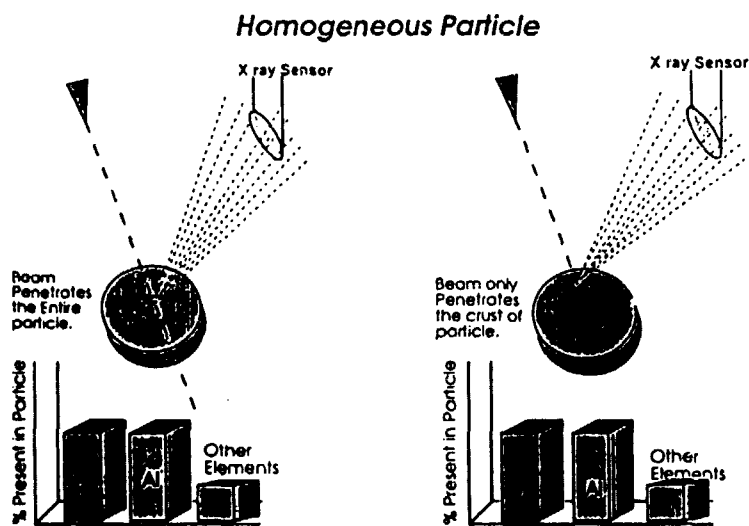


Figure 2. Multiple Voltage SEM on a homogeneous particle.

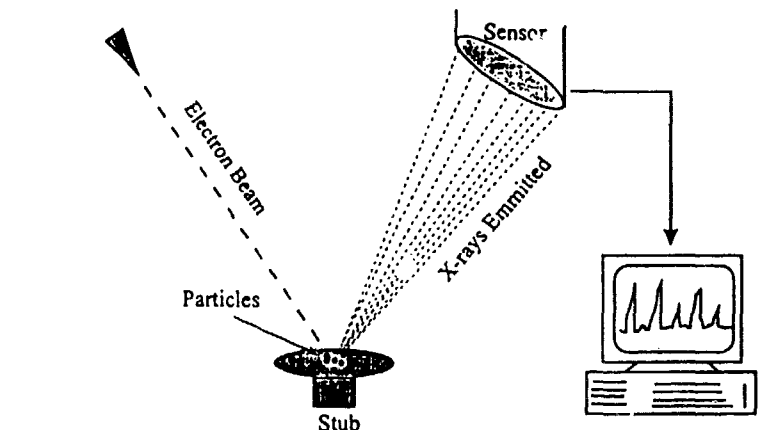


Figure 1. Schematic diagram of a SEM-EDX.

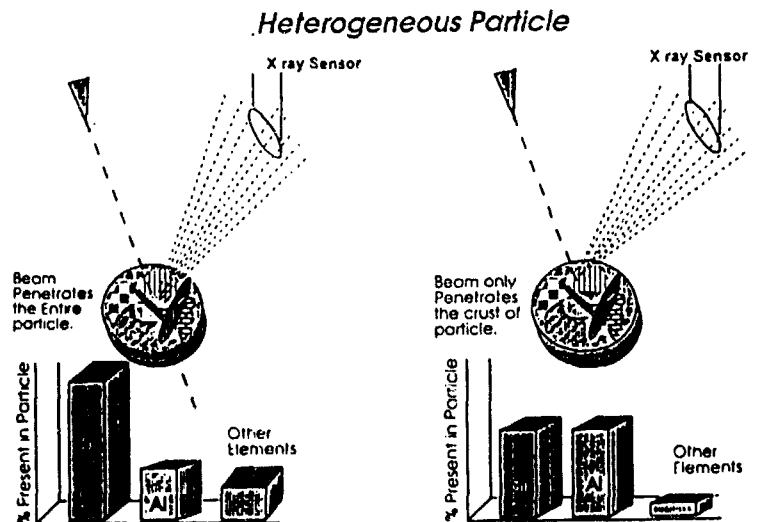


Figure 3. Multiple Voltage SEM on a heterogeneous particle.

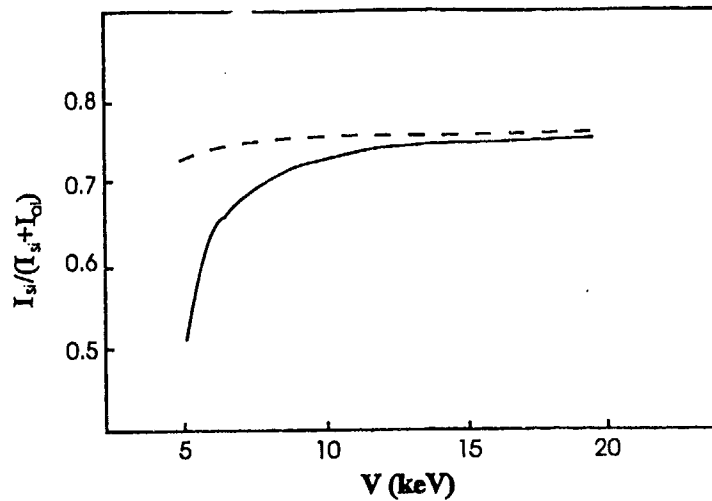


Figure 4. Comparison of silica fraction response of homogenous and heterogeneous particles².

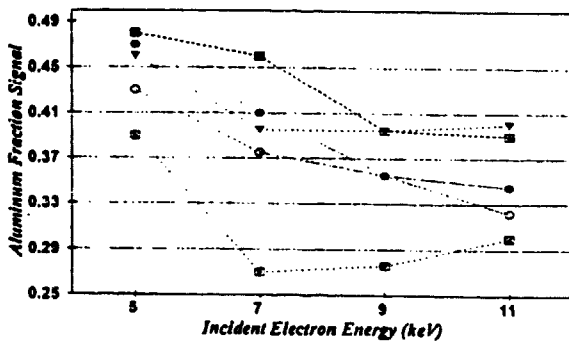


Figure 5. Aluminum fraction signal response for several particles from well #1110 in Big Injune at different incident electron energy.

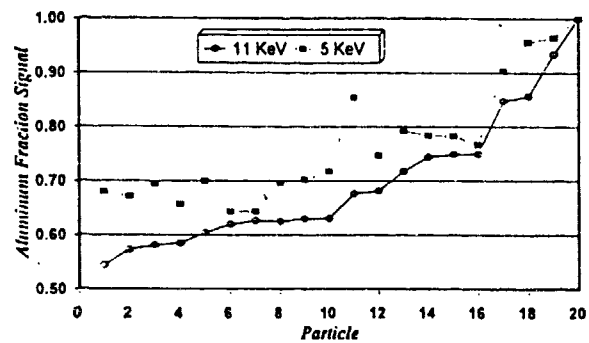


Figure 6. Surface and whole particle analysis for presence of aluminum in the particles, well #1110, 1920 feet depth.

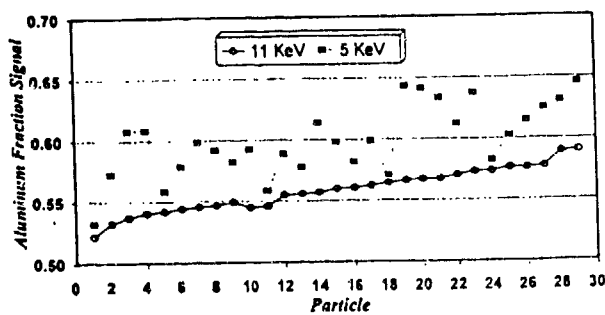


Figure 7. Surface and whole particle analysis for presence of aluminum in the particles, well #1110, 1940 feet depth.

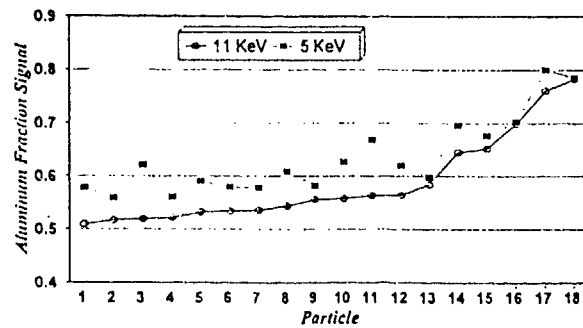


Figure 8. Surface and whole particle analysis for presence of aluminum in the particles, well #1132, 1990 feet depth.