

IMAGE ANALYSIS APPLIED TO THE CHARACTERIZATION OF RADIOACTIVE INORGANIC DEPOSITS

A.P.A. Oliveira¹, R.C.G. Oliveira² and J.F. Oliveira^{1,*}

¹ Department of Metallurgical and Materials Engineering, COPPE – Federal University of Rio de Janeiro, Rio de Janeiro, Brazil

² CENPES, Petrobras Research Centre, Rio de Janeiro, Brazil

*e-mail: oliveira@metalmat.ufrj.br

ABSTRACT

In offshore petroleum exploitation technology, the injection of seawater to maintain the pressure in production wells is a standard practice. This procedure, however, promotes the mixture of formation water and seawater rich in sulphates. Some of these geological formations present a high content of divalent cations and traces of radioactive substances, such as thorium, uranium and radium. As a result, precipitation of inorganic sulphate in the production pipelines and facilities is commonly observed. The presence of radioactive material implies in special procedures related to handling and discharge of these deposits, which lead to high operational costs for the producing company in order to preserve the environment.

The present work is concerned with the characterization of these radioactive inorganic deposits by scanning electron microscope (SEM) applying automated image analysis procedures. It was observed that the deposits samples were composed basically by barium-strontium-rubidium sulphates which were represented in the image by a dark gray color. Very small quantities of bright white grains of uranium-thorium oxides were also identified. These compounds were usually liberated from the sulphates and had an average grain size of 20µm. These results indicated the possibility of separation of these radioactive phases. On the other hand, radium compounds could not be identified by the EDS analysis.

INTRODUCTION

The exploitation of oil in offshore installations produces a significant volume of water derived from the injection process. The treatment of this water is considered one of the most important challenges of the petroliferous industry from either an environmental, technical or economical approach. The legislation establishes discharge limits based on free oils and greases. This type of evaluation is efficient for dispersed

oil, however, it can underestimate dissolved organic and inorganic compounds. It is expected that the amount and the type of dissolved inorganic and organic compounds that are discharged, including radio-chemicals, will also be controlled [Hughes-1994]. So, it is expected that petroliferous industry will optimize established operational procedures and develop new processes to attend more stringent discharge limits.

Traces of radio-chemicals, such as K^{40} , U^{238} , Th^{232} , Ra^{226} and Sr^{90} , can be found among the inorganic components present in the produced water. Radium and strontium can co-precipitate with barium and calcium to form carbonates or sulfates with low specific activity [Hughes-1994, Butler-1964]. The content of radio-chemicals in produced water is not available in details in the literature.

The injection of seawater has been the technique used by Petrobras to maintain the pressure in the offshore producing wells. Some producing formations, such as the Namorado field, contain high levels of divalent cations and traces of radioactive material such as radium, uranium and thorium. The mixture of the producing formation water containing these cations and the injected seawater, rich in sulphates, leads to the formation of inorganic deposits of radioactive material in the production lines and facilities. Special care in the manipulation and discharge of these deposits results in high operational costs for the producing company. In view of the low content of the radioactive material in the inorganic deposits, a process to separate the radioactive material would permit the discharge of non-radioactive inorganic material in a conventional and more economic sanitary backfill, or eventually use this material in industrial applications.

In this context, the present work deals with the first part of the problem, the characterization of the precipitated material, which was carried out by image analysis using scanning electron microscopy.

PRECIPITATES CHARACTERIZATION

The advances in the computer science, software and hardware, the improvements in the X-rays analyzers for energy dispersion (EDS), the digital control of the scanning electron microscope (SEM) and the new backscattered electron detectors (BSE) provide better conditions for the application of the automatic image analysis in the general tailings treatment. In the modern methods of automated analysis, the images of SEM are first digitalized by an interface, converting the sign of the detector into 256 gray levels, where level zero corresponds to the black color and level 255 to the white color. According to the resolution settled, normally 512x512 pixels, each element of the picture in correspondence with each point of the sample receives a gray level, in accordance with the intensity of the sign transferred to the interface [2,3].

The image analysis procedure can be divided in the three following steps:

a) Acquisition and improvement of each global picture.

In a backscattered electron image obtained by SEM, the intensity of the sign emitted to the detector is proportional to the average atomic number of the precipitated material, which permits the observation of distinct gray levels for each precipitate. The discrimination of different compounds will depend on the variation of their chemical composition and the operational conditions of the SEM and the detector, which must be kept unaltered during the image processing. An efficient image analysis depends on some improvements of the image that are possible by the utilization of some algorithms. Typical algorithms are those used for edge enhancing and image equalization.

b) Identification of the precipitated compounds that correspond to the several gray levels.

Each precipitated compound is represented by a characteristic gray level, as a consequence of its average atomic number. The identification of each level is carried by the EDS system by sampling several points of the investigated phase.

c) Segmentation of the global image in individualized images of each precipitated compound.

The segmentation procedure separates in different images the gray ranges that correspond to each compound. Subsequently, each image is transformed into a binary picture, where the selected phase turns into level 255 (white) and the remainder of the image into level zero (black). Several algorithms in this stage are used for the preparation of the image to improve its

evaluation. For instance, it is possible to remove small details without modifying the picture and to use a set of functions that separate the particles eliminating the contact between them. The statistical representation of the results is assured by the automatic change of the image and the quantification of a high number of visual fields. The determination of the composition of the precipitated material is based on the quantification of the areas, its transformation in volume and, finally, percentage by weight taking into consideration the specific weight of each precipitate.

MATERIALS AND METHODS

Precipitates samples were supplied by PETROBRAS according to the following classification: (a) Material 1 – high level incrustation from tubes; (b) Material 2 – low level incrustation from tubes; (c) Material 3 – sludge collected from a gravity separator.

Previously to the characterization step, each sample was submitted, separately, to comminution, homogenization and splitting procedures for the preparation of several samples for image analysis.

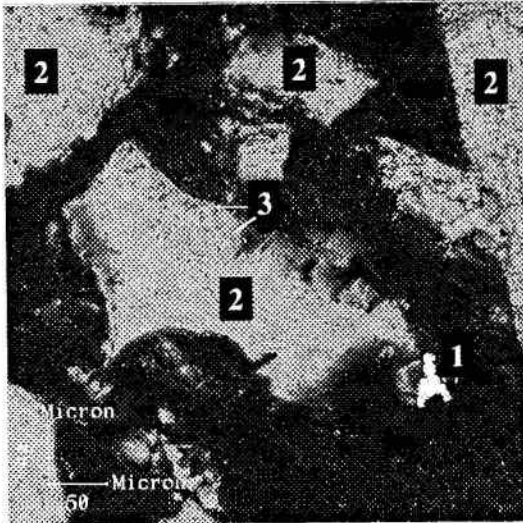
From these samples, briquettes were prepared, polished and metallized. After the metallization procedure, these briquettes were submitted to image analysis in a system composed of a scanning electron microscope (SEM) Zeiss 940-A with secondary and backscattered electron detector, stage motorized for automatic change of the visual field, and a system of microanalysis for dispersive energy (Oxford Link eXL-II). This system was completely controlled by image analysis computer (IBAS-KONTROM).

RESULTS AND DISCUSSIONS

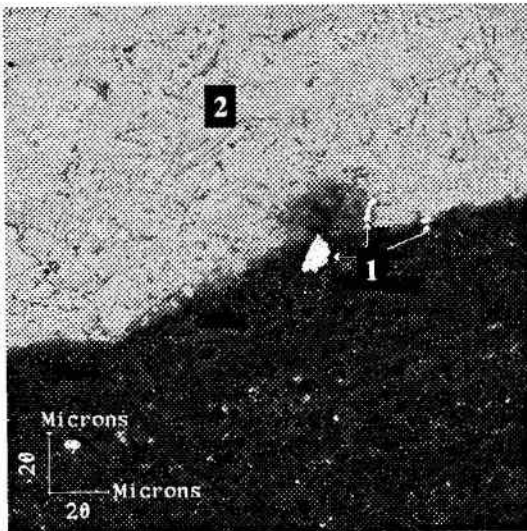
The characterization of Material 1 by EDS analysis showed that the main compounds are co-precipitated sulphate of barium-strontium-rubidium; co-precipitated sulphate/carbonate of barium-strontium-rubidium; quartz and co-precipitated oxide of uranium and thorium. This analysis was not able to detect the presence of co-precipitated radium, previously determined by chemical analysis performed at the IEN (Institute for Nuclear Energy – Brazil). Theoretically, elements from bore (Z=5) to uranium (Z=92) can be detected by EDS analysis, including radium (Z=88). The spectrum lines that can be observed for radium are $M\alpha$ (2,81KeV) and $L\alpha$ (12,32KeV). In both cases, it is necessary a high energy input for achieving radium detection. In this study, even for energy levels as high as

40KeV, it was not possible to identify the presence of radium by the EDS analysis.

The image analysis showed that uranium-thorium oxides appears as white grains in the configuration of backscattered electron detection. Figure 1 depicts two representative micrographies of Material 1. These oxides are presented as free particles or, less frequently, associated to barium sulphate. The size range of this radioactive material is between 7 μ m and 50 μ m.



(a)



(b)

Figure 1 – Micrography of Sample 1 supplied by Petrobras. EDS analysis indicated the presence of: (1) uranium-thorium oxide, (2) barium-strontium-rubidium sulfate, (3) barium-strontium-rubidium sulfate/carbonate.

A quantitative investigation of the weight percent of each compound was performed by image analysis and showed the presence of uranium-thorium oxide and quartz in very low grades, 0.14% and 1.19%, respectively. The image analysis technique is not adequate for quantitative analysis of such low content precipitates because it would be necessary a very high number of fields to obtain reliable results.

Investigations on Material 2 showed that it presents many features similar to Material 1: the same main compounds and the characteristics of uranium-thorium compounds concerning association and size. Figure 2 depicts one representative micrography of Material 2. The quantitative analysis performed showed a lower content of uranium-thorium oxide (0.02%), compared to Material 1. As discussed previously, the quantification of very low content materials by image analysis is not reliable, and so the results obtained for Material 1 and 2 should be looked as semi-quantitative only. However, it can be verified that the uranium-thorium oxides are present in very low levels.

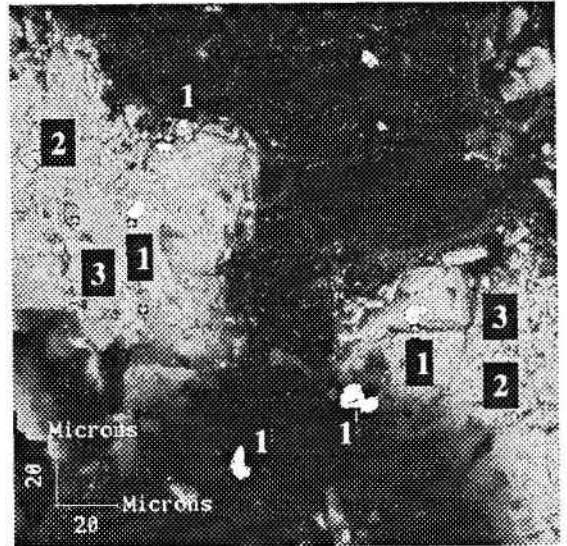


Figure 2 – Micrography of Material 2 supplied by Petrobras. EDS analysis indicated the presence of: (1) uranium-thorium oxide, (2) barium-strontium-rubidium sulphate, (3) barium-strontium-rubidium sulphate/carbonate.

Material 3 was composed of barium sulphate, quartz and iron oxide. No uranium-thorium oxide was found by image analysis. Qualitative radiation analyses performed by IEN reinforced image analysis result by considering this material as non-radioactive. A representative micrography of Material 3 is presented in Figure 3.

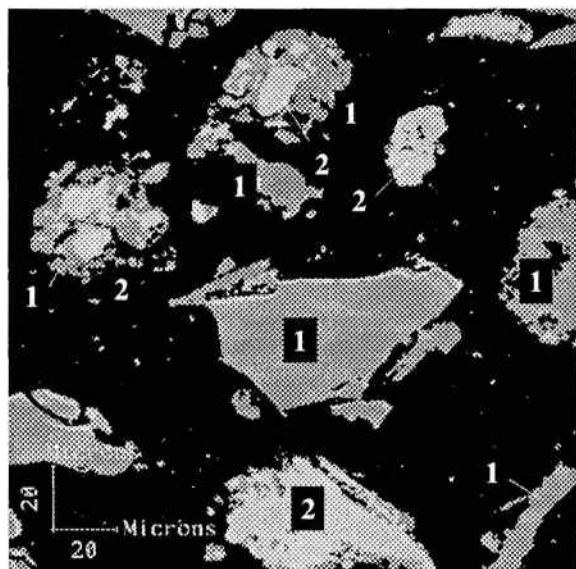


Figure 3 – Micrograph of Material 3 supplied by Petrobras. EDS analysis indicated the presence of: (1) quartz, (2) barium-strontium-rubidium sulphate.

The results obtained in this work indicate that there is a possibility of removing the uranium-thorium oxide by physical or physico-chemical methods. However, as radium is present and probably co-precipitated with barium-strontium-rubidium sulfate, its removal may only be possible by chemical processes. On the other hand, barium sulphate plays an important role in the blocking of radioactivity and its removal would raise significantly the radiation level of the material would mean higher costs for material disposal. An economical approach may define if concentrating the radioactive material leading to the disposal of lower volumes of material with higher level of radioactivity is the most adequate procedure.

CONCLUSIONS

In spite of some limitations observed for the utilization of image analysis in the characterization of the inorganic deposits from the production lines and facilities of PETROBRAS, this method allowed an easy and fast assessment to some important features of the precipitated material, such as composition and constituent associations.

The presence of an oxide phase formed by uranium and thorium co-precipitation partially liberated and in a range size between $7\mu\text{m}$ and $50\mu\text{m}$ indicates the possibility of its removal by physical or physico-chemical method. A technical, economical and environmental evaluation must be made of the benefits of developing such a process.

It is suggested that radium may be present by co-precipitation with barium sulfate or carbonate. The presence of radium was not detected in the EDS analysis. Chemical processes may be the only possibility for the removal of radium from these radioactive inorganic deposits.

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