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APPLICATION OF SCANNING ELECTRON MICROSCOPY TO TAR SANDS EMULSIONS

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APPLICATION OF SCANNING ELECTRON MICROSCOPY TO TAR SANDS EMULSIONS

by

R.J. Mikula*

ABSTRACT

This is a preliminary study demonstrating the potential of scanning electron microscopy in characterizing the emulsions formed during extraction of bitumen from tar sands. The use of a cold stage for the direct observation of the emulsion sample has several advantages over conventional observation of replicas. The sample preparation is less tedious and x-ray analytical capabilities are retained. This paper presents results indicating the utility of the x-ray analysis in determining the composition of the dispersed phase and the electron microscopic observation in relating the morphology to emulsion stability.

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LA MICROSCOPIE À BALAYAGE ÉLECTRONIQUE APPLIQUÉE AUX ÉMULSIONS DE SABLES BITUMINEUX

par

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RÉSUMÉ

Une étude préliminaire servant à démontrer les possibilités qu'offrent la microscopie électronique à balayage pour caractériser les émulsions formées au cours de l'extraction du bitume des sables bitumineux est présentée dans ce rapport. L'emploi d'une phase froide pour l'observation directe des échantillons d'émulsion présente de nombreux avantages par rapport à l'observation classique des mêmes échantillons. La préparation des échantillons est aussi moins fastidieuse, et les capacités de l'analyse aux rayons X sont conservées. De plus, les résultats présentés dans ce rapport indiquent l'utilité d'une part de l'analyse aux rayons X pour déterminer la composition de la phase dispersée et d'autre part, l'utilité des obvervations réalisées à l'aide de la microscopie électronique pour établir un rapport entre la morphologie et la stabilité de l'émulsion.

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CONTENTS

FIGURES

APPLICATION OF SCANNING ELECTRON MICROSCOPY TO TAR SANDS EMULSIONS

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INTRODUCTION

The products of enhanced oil recovery and certain tar/oil sands extraction processes are emulsions and characterisation of their properties is an important step in understanding how they form and how to break them. An economical method of breaking these emulsions would lead to increased production from these petroleum extraction processes.

The present work is a preliminary study to demonstrate the potential of the cold stage technology and the direct observation and characterization of petroleum emulsions in an electron microscope. The important advantage that the direct observation technique has over electron microscopy of replicas (besides speed of sample preparation), is the retention of x-ray analysis capabilities. X-ray analysis may be able to provide definitive evidence of what stabilizes some of the economically important oil and tar sand emulsions. For example, the interactions of the clays present in these samples could be important in determining emulsion stability. The large depth of field in the electron microscope helps in the qualitative (quantitative, when coupled with image analysis) determination of the size distribution of the dispersed phase. Furthermore, the capability of x-ray analysis on the area that is visually observed provides important information regarding the mechanisms of emulsion stabilization.

EXPERIMENTAL DETAILS

A Hitachi X-650 scanning electron microscope with both energy and wavelength dispersive x-ray spectrometers has been combined with an Emscope 2000 cryo-stage. The direct observation of the emulsions involves freezing the sample in a liquid nitrogen slush and keeping it frozen and under vacuum in the cryo-stage. The sample is then fractured and coated following ordinary sample preparation procedures. A cold stage in the microscope then allows direct observation of the sample while retaining x-ray analytical capabilities. Some minor modifications to the sample holders were required in order to maintain the low temperature (frozen condition) of the sample in the scanning electron microscope. The sample holders were made of a simple copper block with a 3mm well drilled to accomodate the sample. The copper block is machined to make good thermal contact with the cold stage inside the electron microscope. Low beam currents and luminosities are used whenever possible in order to minimise radiation damage and local evaporation of the samples. Sublimation and destruction of sample features was found to be negligible for normal observation times, although some sacrifices in image quality had to be made which would not have been necessary with replica samples.

The x-ray detector used in this study is a 30cm Si(Li) Tracor Northern detector coupled to the electron microscope to provide dot map and line scan capabilities. The x-ray spectra were taken with a 20 KeV beam energy for two minutes on a carbon coated sample. The short time was used in order to minimize beam damage while accumulating the x-ray spectra in the spot mode.

RESULTS AND DISCUSSION

Table 1 shows the analysis of the oil and water in the emulsions discussed in this paper, along with their relative stability. The emulsion analysis was done using a Dean-Stark method, which does not total 100 per cent because of losses of some of the more volatile components. Figure 1 shows emulsion E1, a water in oil emulsion that is very stable. The size of the dispersed phase is quite diverse with droplets ranging in size from about 10 microns down to 1 micron and smaller. The majority of the droplets are smaller than 5 microns and this small size is a significant factor in determining the stability of this emulsion. It is not the only factor, however, since Figure 2 also shows a stable emulsion (E2) but with a significantly larger average size for the dispersed water phase.

TABLE 1: Per cent oil and water analysis of the emulsions used in this study.

Emulsion	Per cent Oil+Solids	Per cent Water
E1 (stable)	~27.6 + ~4.1	~63.0
E2 (stable)	~71 + ~.26	~21.8

Figures 3a and 3b show x-ray spectra of emulsion E1 scanned over a dispersed water droplet and over the continuous oil phase respectively. The large increase in the chlorine x-ray intensity relative to the sulfur (in Figure 3) indicates a concentration of chloride (or decrease of sulfur) in the water phase relative to the continuous oil phase. The chloride component is still clearly evident in the continuous oil phase because the oil phase is interspersed with many of the smaller water "droplets" which contribute to the chlorine x-ray peak. The identification of the water as the dispersed phase via the x-ray studies is interesting because the large amount of water in this emulsion (Table 1) might lead one to conclude that it is an oil in water emulsion rather than water in oil. Most of the other peaks are not identified, although silicon and aluminum (from the clay component) are clearly evident.

Preliminary x-ray work has suggested that the clay components are concentrated at the interface of the emulsion droplets although further studies are required to definitely establish this. However, determining the presence of clays at the interface does not necessarily mean that they are the only reason for emulsion stability.

CONCLUSIONS

The direct observation and x-ray analysis of oil-water emulsions from tar/oil sands recovery has clear advantages over other methods, both in the information obtained and the ease of sample preparation, all that remains is to realise its potential. The utility of the technique for characterising emulsions has been shown, with the potential for x-ray analysis of the elements present in the various phases, as well as at the interface. Work is continuing on the study of relationships between emulsion stability, size distribution of the dispersed phase, and the chemical nature of the phases and the interface.

The ultimate goal is to directly establish the nature of the interface and to determine the mechanisms of stabilization of these economically important emulsions.

FIGURE 1: Emulsion E1 (gold coated)

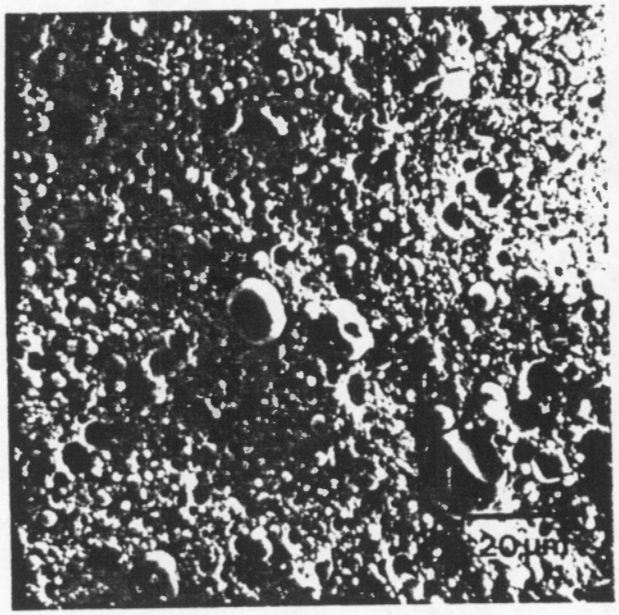


FIGURE 2: Emulsion E2 (gold coated)

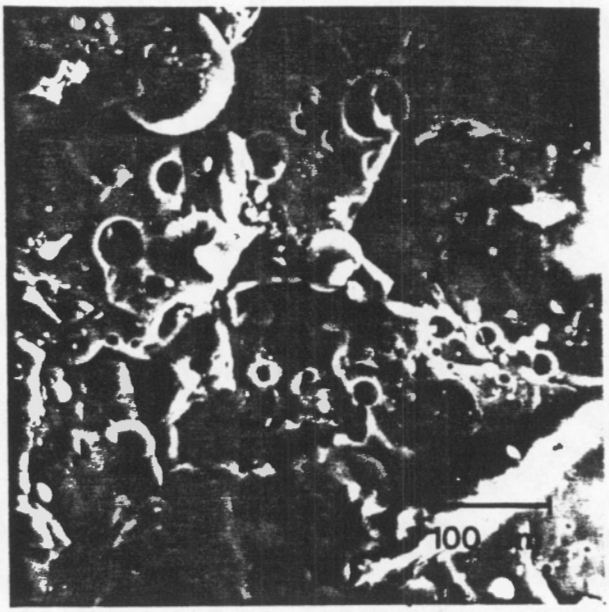
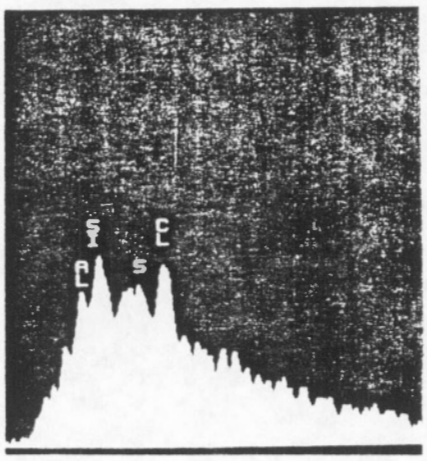


FIGURE 3: X-ray spectra of emulsion E1.

Dispersed Water Phase



Continuous Oil Phase