

IMAGING OF PSEUDO OIL BASE MUD BY ENVIRONMENTAL SCANNING ELECTRON MICROSCOPY

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IMAGERIE DES BOUES DE FORAGE À BASE D'HUILE DE SUBSTITUTION PAR MICROSCOPIE ÉLECTRONIQUE À BALAYAGE AMBIANT

La caractérisation des boues de forage à base d'huile de substitution (POBM) équivaut à définir la relation entre la répartition dimensionnelle des gouttelettes et la stabilité des émulsions. Les microscopes électroniques à balayage (SEM) et à transmission (TEM) classiques ont été utilisés pour étudier ces problèmes. Toutefois, les échantillons hydratés sont d'un traitement difficile à l'aide de ces techniques. Le microscope environnemental électronique à balayage (ESEM ou electroscan) et les techniques cryogéniques ont ouvert la voie à l'étude de spécimens humides et non conducteurs, de liquides et d'émulsions divers.

À froid, l'ESEM nous permet de visualiser des émulsions :

- sans aucune préparation ou enrobage,
- à différentes températures, en régulant la température (entre -180°C et -80°C) et la pression (entre 400 et 1400 kPa) du spécimen.

Le présent document se propose de caractériser les boues à base d'huile de substitution et d'étudier :

- l'homogénéité de la formulation et la procédure de préparation,
- l'influence de la répartition dimensionnelle des gouttelettes sur la stabilité des émulsions.

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Characterizing pseudo oil base muds (POBM) amounts to determining the relationships between the size distribution of droplets and stability of emulsions.

Conventional scanning and transmission electron microscope (SEM and TEM) have been used to investigate these problems. However, hydrated samples are difficult to handle using such techniques. The Environmental Scanning Electron Microscope (ESEM or Electroscan) and the cryogenic techniques have opened the door to the study of wet and non-conductive specimens, and of various liquids or emulsions.

ESEM with the cold stage allows us to directly visualize emulsions:

- without any preparation or coating;
- at different temperatures, by controlling the specimen temperature (between -180°C and -80°C) and pressure (between 400 and 1400 Pa).

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The purpose of this paper is to characterize pseudo oil base muds and to control:

- the formulation homogeneity and preparation procedure;
- the influence of droplet size distribution on the stability of emulsions.

IMAGINERÍA DE LOS LODOS DE PERFORACIÓN A BASE DE ACEITE DE SUSTITUCIÓN POR MICROSCOPIA ELECTRÓNICA DE EXPLORACIÓN MEDIOAMBIENTAL

La caracterización de los lodos de perforación a base de aceite de sustitución (POBM) equivale a definir la relación existente entre la distribución dimensional de las gotas finas y la estabilidad de las emulsiones.

Se han utilizado los microscopios electrónicos de exploración (SEM) y de transmisión (TEM) de tipo convencional para el estudio de estos problemas. No obstante, el procesamiento de las muestras hidratadas resulta difícil por medio de estas técnicas. El microscopio medioambiental electrónico de exploración (ESEM o Electroscan) y las técnicas criogénicas han venido a facilitar el estudio de muestras húmedas y no conductoras, así como de líquidos y emulsiones.

Operando en frío, el ESEM nos permite visualizar las emulsiones :

- sin ninguna preparación o revestimiento,
- según distintas temperaturas, previa regulación de la temperatura (entre -180°C y -80°C) y la presión (entre 400 y 1400 kPa) de la muestra.

El presente documento tiene por propósito caracterizar los lodos de aceite de sustitución y estudiar :

- la homogeneidad de la formulación y el procedimiento de preparación,
- la influencia de la distribución dimensional de las gotas finas sobre la estabilidad de las emulsiones.

INTRODUCTION

This study aims to illustrate the use of cryogenic techniques with the environmental scanning electron microscope (ESEM or Electroscan) in the study of:

- complex emulsions;
- microstructural changes at the preparation stage.

Emulsion selection was based on the sensitivity of materials, with the selected emulsions being excellent candidates for imaging, analysis and measurements by ESEM.

Conventional scanning and transmission electron microscope (SEM, TEM) were used together with cryogenic techniques to investigate the emulsion characterization. Complex specimen preparations, high vacuum during coating may modify their original characteristics and affect image interpretation (e.g. cracks) due to the artefacts introduced. Using such techniques makes it difficult to handle liquids.

ESEM allows us to:

- observe the pseudo oil base on cryofractured surface (POBM) without coating;
- examine the influence of droplet size distribution on the stability of emulsions, etc.

Pictures of POBM are attached herewith. The effect of $\text{Ca}(\text{OH})_2$ is discussed below.

1 ENVIRONMENTAL SCANNING ELECTRON MICROSCOPE (ESEM)

1.1 Description

ESEM allows visualization of liquids and natural wet samples due to its following characteristics:

- It operates with a differential pumping system which creates a pressure gradient in the electron optical column (Fig. 1). Several chambers separated by a series of small apertures along the beam path allow to keep the electron beam source and the upper column under high vacuum, while the specimen chamber can be under pressure from 2 to 7 kPa.
- A new secondary detection electron system operating at chamber pressure (2 to 7 kPa) was used. Gas environment, temperature and pressure can be set up in the specimen chamber. For example, gas environment can be ambient gas, nitrogen, argon, water vapour, organic solvent vapour, etc. Danilatos [1] developed the formulae governing gas dynamic and beam scattering in air. He showed that the spatial

resolution of unscattered beam fraction can still be used to form an image with a good resolution as the one obtained under high vacuum.

ESEM incorporates the best features of conventional SEM (high resolution, depth of field, electronic signal detection, elementary analysis X-ray detectors) with, in addition, the flexibility and easiness of use of a light microscope. It can be used to visualize and record in real time dynamic processes such as drying, melting, dissolution, crystallization and chemical reaction at temperatures up to 1000°C.

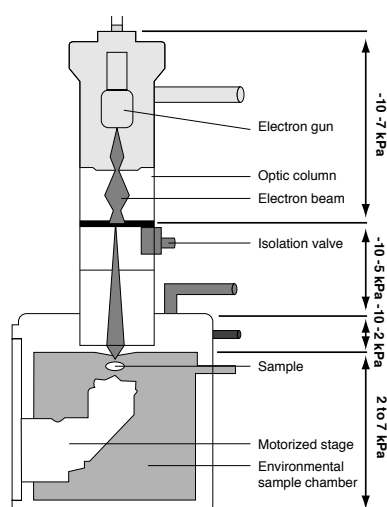


Figure 1

Schematic diagram of the ESEM.

1.2 Environmental secondary detection system

As a conventional SEM, the specimen electron beam interactions produce electronic signals including secondary electrons, backscattered electrons and X-rays. The standard Everhart-Thornley detector cannot be used because of the high voltage requirement on scintillator surface which would create electrical breakdown in gaseous environments. So, ESEM uses the environmental secondary detection system first described by Danilatos [1] and [2]. Gas ionisation is induced by electrical field forcing collision between highly mobile secondary electrons and neutral gas molecules. Successive collisions liberate additional free electrons resulting in the cascading multiplication of secondary signal. The negative charges build up on sample surface (Fig. 2).

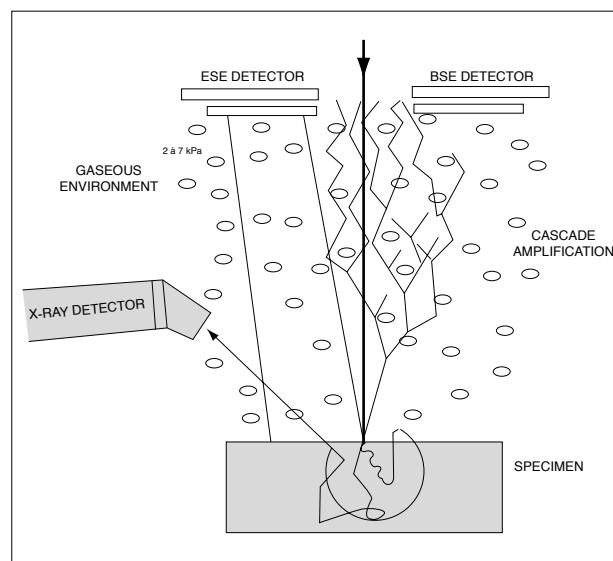


Figure 2

Schematic view of electronic signals in the ESEM chamber.

2 EXPERIMENTAL DESCRIPTION

2.1 Preparation of formulations

Two fresh emulsions were prepared by mixing salt water (CaCl_2) in a mixture of poly-alpha-olefins (PAO) and surfactant. Then lime was added in sample 1.

TABLE 1

Composition for the formulations called "Lot 2" and "Lot 3"

COMPOSITION	PREPARATION	
	"Lot 2" - Sample 1	"Lot 3" - Sample 2
Oil base	196 cm ³	196 cm ³
Surfactant	8 cm ³	8 cm ³
Water	84 cm ³	84 cm ³
CaCl_2	34 g	34 g
$\text{Ca}(\text{OH})_2$	8 g	

2.2 Experimental Procedure

ESEM operates at various high voltages using the Electron Secondary Detector (ESD). Samples were:

- frozen in nitrogen flush, transferred to the cryogenic system chamber, freeze-fractured under vacuum and placed over the cold stage chamber of ESEM. The cold stage is equipped with an efficient cold trap in the vicinity of sample to preserve it from the formation of ice;

- examined without any coating;
- visualized at various temperatures (between -180°C and -80°C) and pressures (between 400 and 1400 kPa) in an inert nitrogen atmosphere.

3 DISCUSSION

It was the first time we examined the POBM. We have not still arrived at a complete understanding of results. Nevertheless, the observations made on the cryofractured surface allow us to appreciate the homogeneity of the preparation procedure.

Before each examination, the emulsion was mixed to preserve its properties. During mounting and freezing, we did not see any segregation between the different phases in the samples.

Plates 1a, 1b, 4a and 5a illustrate the distribution of droplets. The clusters of inorganic particles are surrounded by a olefin matter. The size distribution of droplets is heterogeneous. With the addition of $\text{Ca}(\text{OH})_2$, the higher magnification images (Plates 3a and 3b) show irregular shapes of particles and voids occupied by brine. Without $\text{Ca}(\text{OH})_2$ the shape of droplets is regular (Plates 5a et 5b).

CONCLUSIONS

As a first approach, the examination of emulsions helped us:

- control the preparation;
- appreciate the homogeneity of formulations;
- suggest the effect of different inorganic components on the stability of POBM.

It is necessary to carry on with this type of experiments to better understand what the images show.

In the future, it will be interesting to work under various conditions to get information on the part played by some parameters (temperature, concentration of constituents) in the stability of muds.

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- 2 Danilatos G.D. (1983), A gaseous Detector Device for an Environmental Scanning Microscope. *Micron and Microscopia Acta*, **14**, 307-319.

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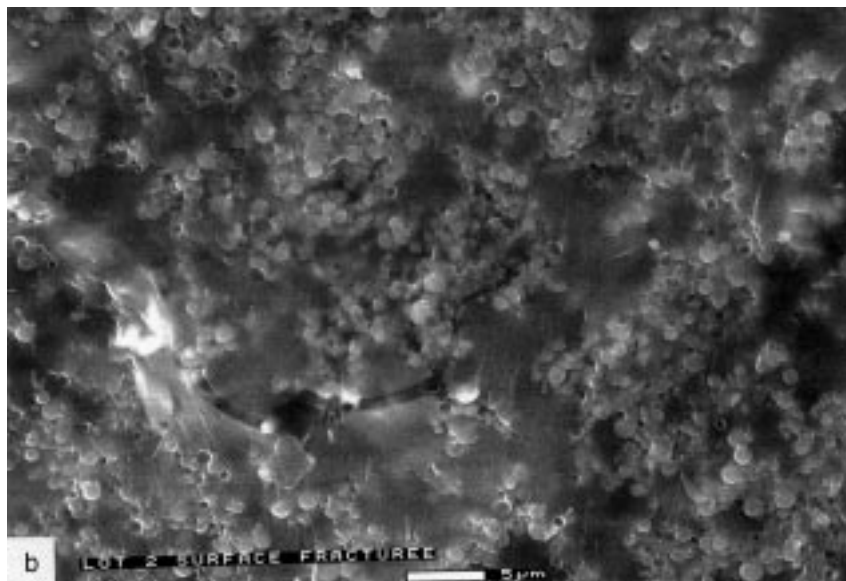
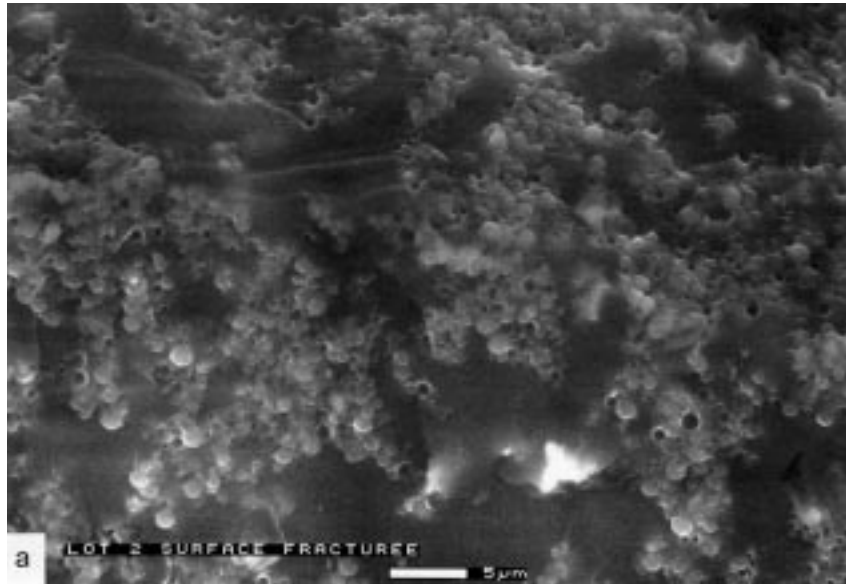


Plate 1

Low-temperature fractures of sample 1. The sample was uncoated and photographed at -150°C . The dispersion of particles is heterogeneous ($G = \times 2000$).

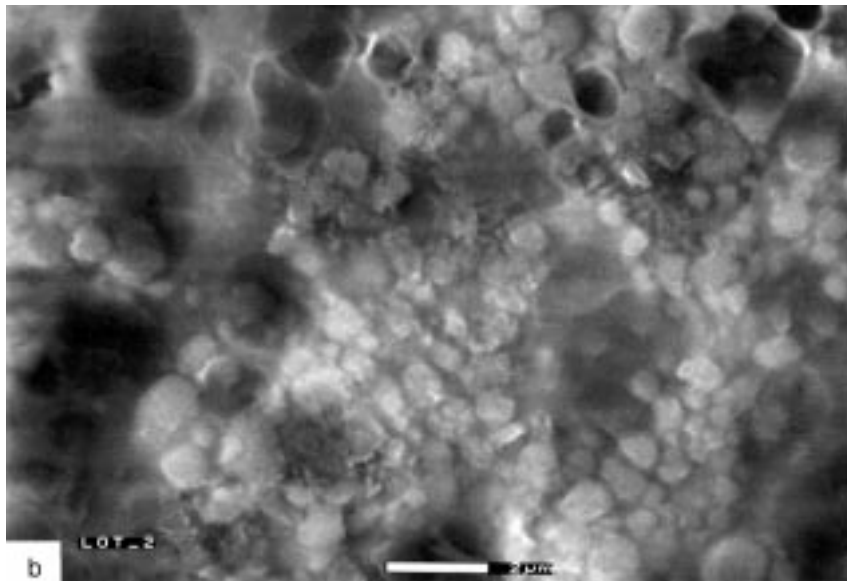
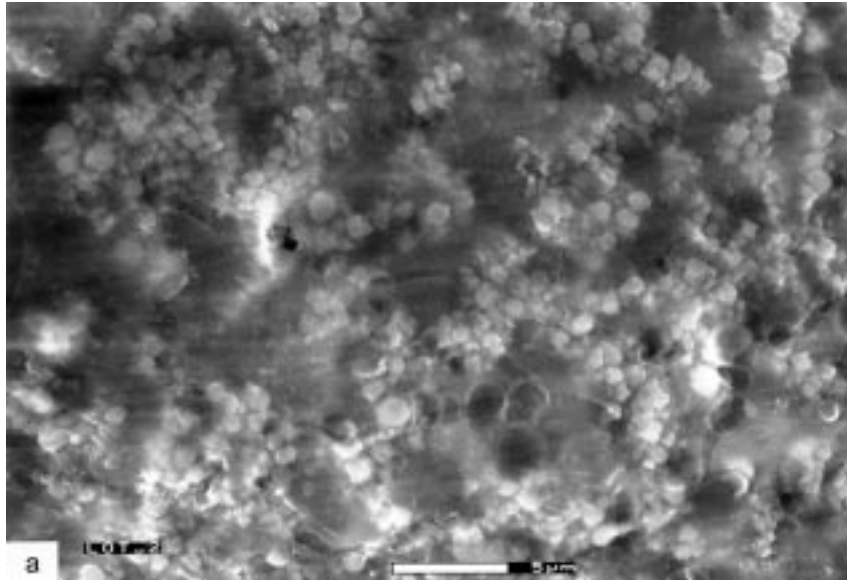


Plate 2

Observation of particles with high magnification. The particles form inorganic clusters surrounded by base oil matrix; a) G = x3000; b) G = x6600.

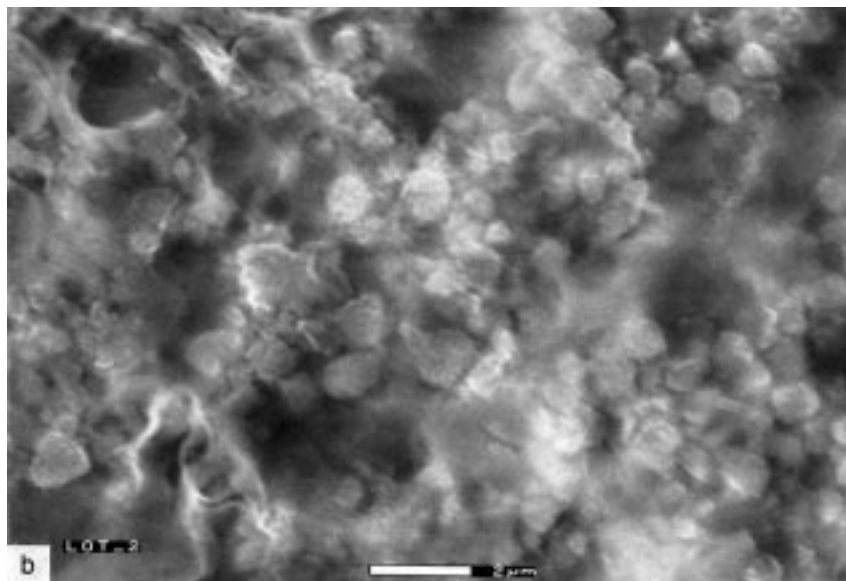
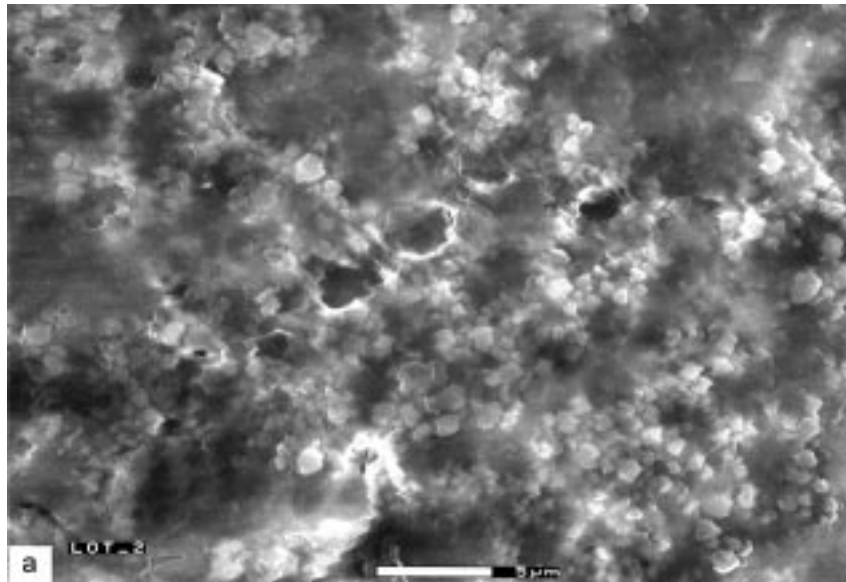


Plate 3

Higher magnification images clearly show particles and voids occupied by droplets of brine;
a) G = x3000; b) G = 6600.

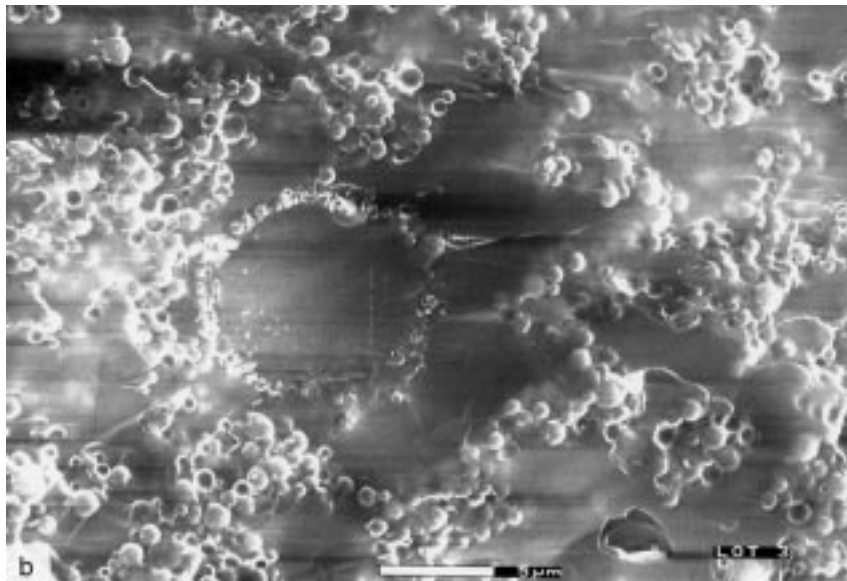
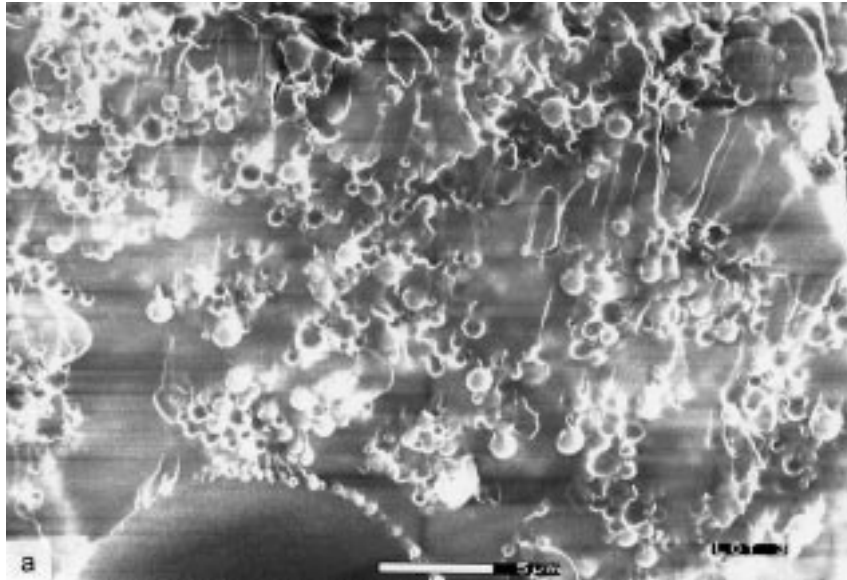


Plate 4

Observation of the fractured surface of sample 2 clearly shows particles and voids. The dispersion of particles is heterogeneous and forms clusters; a) G = x3000; b) G = x3000.

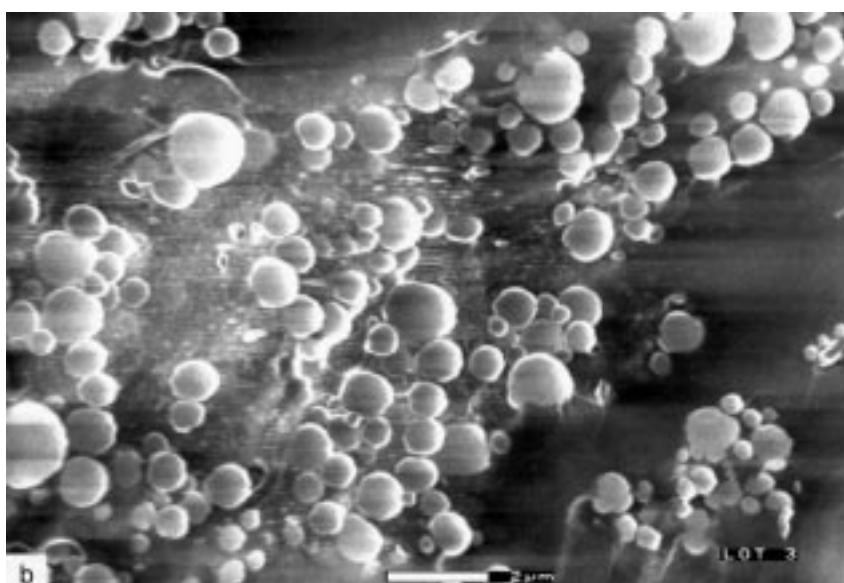
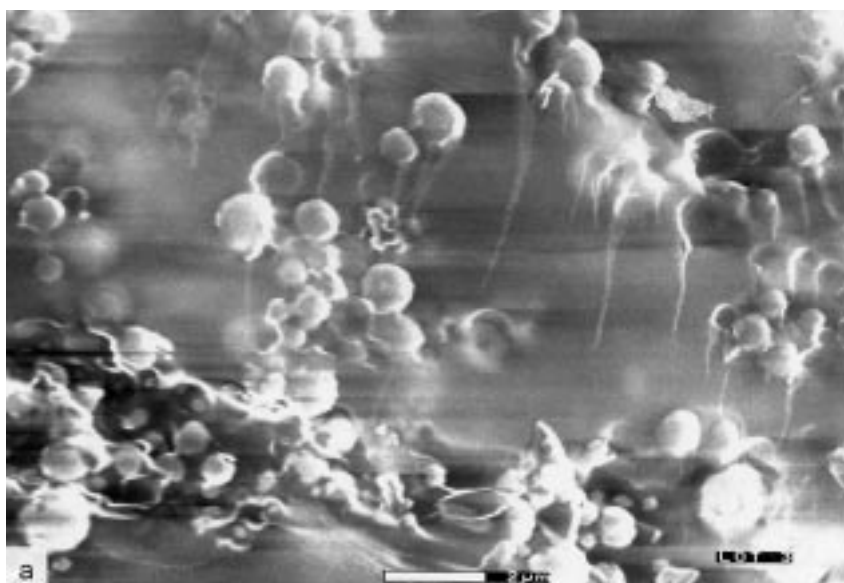


Plate 5

High magnification ESEM micrographs reveal the regular shape of particles with no voids;
a) G = x6600; b) G = x6600.