INTRODUCTION – The petroleum industry processes crude oil from many sources to make a range of finished petroleum products. One case study is presented herein of the analysis of oil product extracted from tar sand deposits for processing. A sample of this tar in the form of froth with high bitumen content was analyzed using the Canty Vector and microscope camera to view water emulsion droplets with sizes as small as one micron. The analysis was done for images from both the Vertical microscope viewing emulsion between two glass slide plates and the Canty High Pressure Flow Cell coupled to the re-circulating pressure pots for a many pass view of the emulsion. The flow cell was also fed from a single pot with dip tube discharge inside a pressured enclosure for a one time look at emulsion. A similar analysis could be done for other crude oil sources.

GOAL – The goal for this project was to measure particle size distribution of water droplets in the process emulsion for sizes from 1 micron to over 100 microns. Process conditions for this measurement are temperature up to 140 C and pressure up to 200 kPa.

SAMPLE PREPARATION – The heavy tar-like sample materials contain bitumen, water and solids. In Canty lab, the sample was warmed for a while, then naphtha was added as a solvent, and mixed to form a uniform emulsion of all components. The emulsion was then allowed to settle out the heavy solids and the top portion used to study the process of water extraction. There are several methods to induce the small water droplets to coalesce into bigger and bigger droplets, and the desire is to reduce the time required for this coalescence to occur. The classic method is to use high-G centrifuge devices. Any method used to enhance coalesce requires monitoring the emulsion droplet size to carefully control the water extraction process.

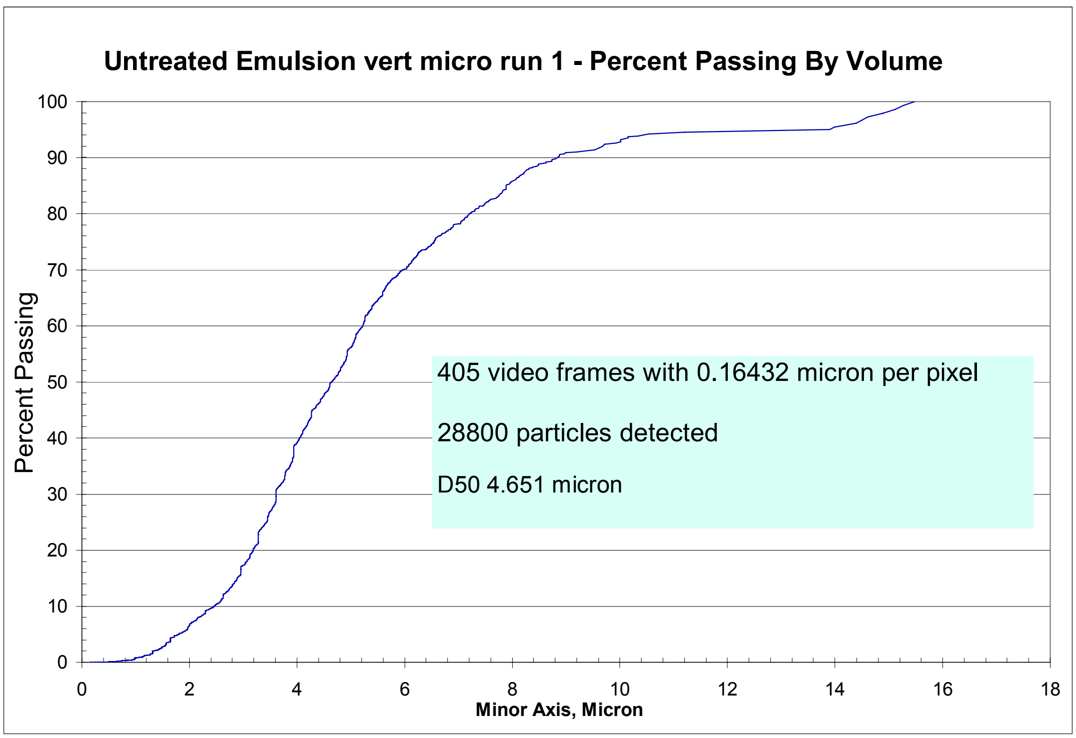
SAMPLE VIEWING –

1. Preliminary Analysis - Vertical Microscope
   The emulsion was extracted from the top, center, or bottom of the sample container after a known settling time and one or two drops placed on a clean glass slide. The drops were covered by a second glass slide to produce a thin emulsion layer and immediately placed between the microscope lens and a light source for viewing. A flow is seen as the material equalizes between the glass slides. (see images group 1)

2. Primary Analysis – Canty Flow Cell Microscope
   The prepared sample was then introduced to the Canty Flow cell for simulation of on-line analysis. The Canty Flow Cell is equipped with a re-circulating system that allows continuous flow of a sample through the unit for viewing and analysis.
   To optimize the process view, the cell gap was adjusted to115 microns. The sample reservoirs included a heater jacket to keep the vessel wall between 185 and 195 degrees F.

Camera configuration for both the Vertical Microscope and Flow Cell Microscope included a 20X objective and a 7 to 1 zoom body. At max zoom, the pixel value for these cameras is 0.16432 micron per pixel for a 105 micron field of view, and for intermediate zoom 0.684 micron per pixel was used for a 400 micron field of view. The zoom adjustment allows measurements of freshly agitated materials with small water droplets and also for the condition where small water droplets have coalesced into bigger drops.

RESULTS – Some typical graphs of size distribution are shown below.



Plot 1, Untreated Emulsion

The percent passing curve is steep where many particles are detected and flattens to near horizontal for size ranges where very little material is detected. The D50 size identifies a mean size where 50% of the detected particle volume is larger and 50% is smaller. For the untreated material with D50 of 4.7 Micron, about 50% of the detected particles on a volume basis are between 2.5 and 5.5 Micron. About 40 % of the particles are larger than 5.5 Micron.

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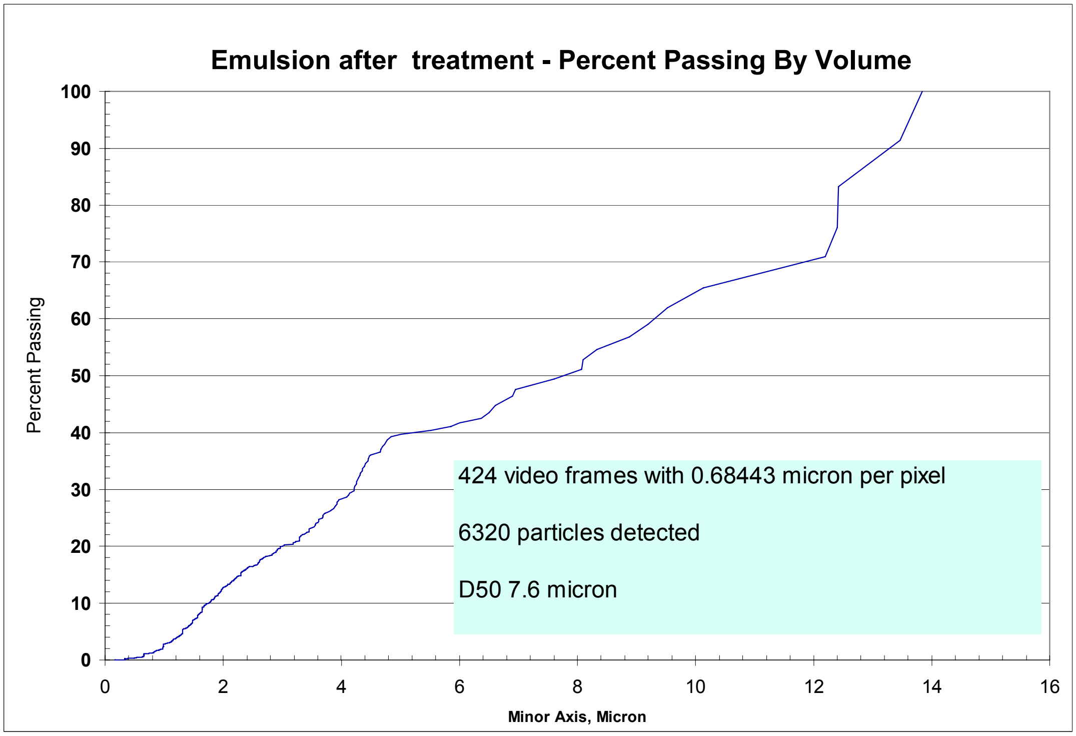

Plot 2, Emulsion After Treatment

The second plot for treated material has a D50 of 7.6 Micron and has a uniform slope out to 14 micron. The treatment has increased the water droplet size. The size distribution as indicated by the slope of the plot has also changed after treatment. The ability to obtain quantitative measurement of the size distribution for the emulsion at process pressures and temperatures before, during, and after the treatment allows users to design treatment protocols and to control treatment process during production.

PROCESS IMAGES – Typical Video Images from the color microscope cameras are shown below.

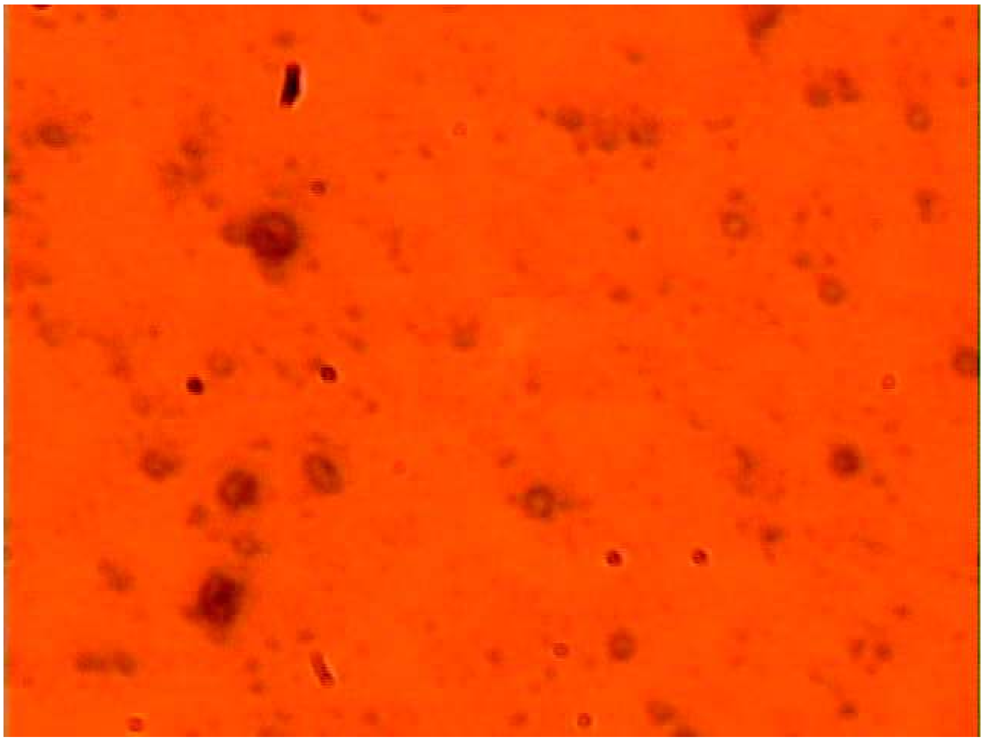

Image 1, Vertical Micro, 105 micron field of view, static sample after 25 minutes

Emulsion containing water droplets seen as dark gray circles with lighter gray centers against orange background. Non circular droplets are clumps of two or more droplets beginning the coalesce process.

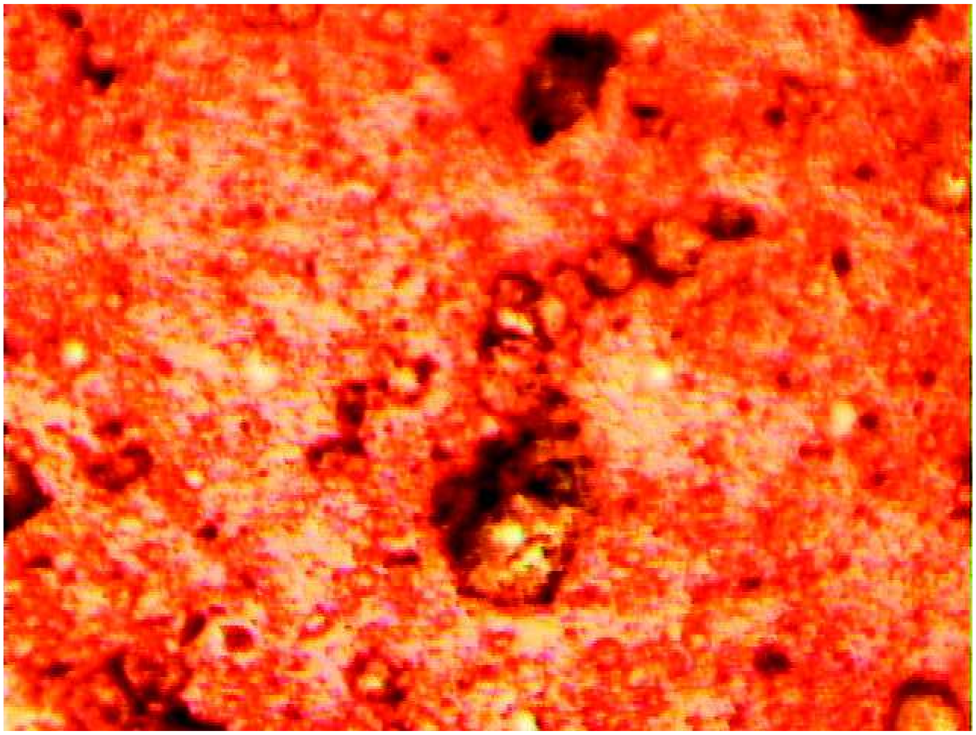

Image 2 , Flow cell, 400 micron field of view, showing solvent induced coalescence

Lower magnification image shows larger water droplets and clumps of droplets.

CONCLUSIONS – Video images give visual verification of the numerical data provided and user has good confidence in the measurement process. The ability to measure the size distribution for the emulsion at process pressures and temperatures enables the user to design and verify processing protocol for petroleum processing. The production process can also be monitored and controlled by installing an on-line system.