International Journal of Earth, Energy and Environmental Sciences
ISSN: 2517-942X
Vol:7, No:11, 2013

Evaluation of Droplet Sizes from Video Images for Metal Working Fluids

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***Abstract*—**Metal working fluids were used in the preparation of oil in water emulsions. The size of oil droplets were evaluated by using the analysis of video images taken from the zeta potential measurements. The evaluated size distributions for emulsions were also tested by microscopic analysis. In addition, emulsion stabilities were discussed depending on electrolyte concentration and pH. The results showed that the stability of oil emulsions was strongly related to pH and the concentration of $CaCl_2$. However, the same dependency was not observed for NaCl.

***Keywords*—**Droplet size distribution, emulsion stability, o/w emulsions, video images.

I. Introduction

EMULSIONS are colloidal systems composed of droplets of one liquid dispersed in another liquid. They form the basis of a wide variety of natural and manufactured materials including foods, pharmaceuticals, petrochemicals and cosmetics [1]. It is known that the emulsion properties such as rheology, stability, texture, appearance, and taste are mainly dependent upon droplet size distribution [2]. In addition, the size of droplets in an emulsion has a strong impact on its stability (gravitational separation, flocculation, and coalescence), its optical properties (color), and its rheology (viscosity) [3].

Microscopy, light-scattering, ultrasonic techniques have been used in the literature for the analysis of droplet size measurement in emulsions [4], [5]. On the other hand, the number of work on the application of video images to analyze size distributions is rare. Novales et al. [6] present a video imaging instrument for rapid and non-intrusive monitoring of the destabilization of dispersed systems.

Emulsions are unstable systems and tend to break down with time in order to minimize the interfacial area between the aqueous and the oil phase. Stability of emulsions is often related to zeta potential, i.e., the difference in potential between the surface of the tightly bound layer of ions on the oil droplet surface and the electro-neutral region of the solution [7]. The electrical charge on the droplet interface influences its interactions with other charged molecules, as well as its stability to aggregation. The zeta potential is much easier to measure than the electrical potential or surface charge density, and therefore droplet charges are usually characterized in terms of zeta potentials [3].

Metal working fluids (MWF) or cutting fluids are usually composed of a mineral oil (40-80%), a surfactant, and additives. Emulsions of metal working fluids are widely used in metal forming, metal cutting, and in the galvanic industry where cooling, lubrication and rust control are important in operations. Two types of metal working fluids were used in the preparation of oil emulsions and the size of oil droplets have been predicted by analyzing video images. Then the stability of emulsions was discussed based on the results of zeta potential measurements and size distribution evaluations. In addition, the effects of electrolyte types and concentrations on the oil droplet size distribution have been investigated in the present study.

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This work was supported by the Scientific and Technical Research Council of Turkey (Project No: 111Y233).

II. Material And Methods

A. Preparation of MWF Emulsions

Oil emulsions have been prepared by using two commercial MWFs: TapmaticDual Action Plus 1 (EAL) and Taptamic Natural (FAM). EAL can be used for all metals except aluminum and FAM can be used for all metals. The chemical contents of EAL and FAM are presented in Table I. Both MWF forms emulsions when they are mixed by water.The oil emulsions were prepared by mixing EAL or FAM to tap water at a volume percentage of 2; then the mixture was stirred mechanically for 30 minutes. Sodium chloride (NaCl) and calcium chloride ($CaCl_2$) were selected as electrolytes and the studied concentrations were 0.01, 0.03, 0.05, 0.07 and 0.09 M.

The pH of emulsion was around 8 after mixing EAL and FAM. The emulsion pH values were adjusted either by using hydrochloric acid or sodium hydroxide.

TABLE I
Chemical Compositions of Metalworking Fluids

Trade Name	Code	Composition
Taptamic Dual Action Plus 1	EAL	Chlorinated alkane (40%-60%) Chlorinated paraffin (20%-40%) Not soluble in water
Taptamic Natural	FAM	Alkyl esters Biodegradable Soluble in cold water

B. Zeta Potential Measurements

Malvern Zetasizer-4, which calculates zeta potential depending on electrophoretic mobility, was used for the measurements. During the measurement, a sample of emulsion was placed into the measurement cell and an electrical field was applied to the electrodes. This causes any charged colloid in the emulsion to move towards the oppositely charged electrode at a velocity that depends on the magnitude of their charge. The colloid movement was recorded to a computer by taking the signals from the Unitron FSB-4X stereoscopic microscope. It features 20X wide field eyepieces in combination with a 4.0X paired objective. Overall magnification is 80X.This microscope is very adequate for colloids as small as 1.5 microns in size. Electrophoretic mobility of an oil droplet was obtained by tracking one oil droplet by pressing the mouse and holding it down while the droplets moves across the grid. Then the zeta potential is calculated by using Smouclowski equation.

C. Droplet Size Analysis

The droplet diameters were estimated by using the images of emulsions obtained from direct video imaging assembly of Malvern Zetasizer-4. The gray scale video images taken from the zeta potential measurements were processed by writing a MATLAB function. This function looks for places in the image where the intensity changes rapidly, using one of these two criteria: places where the first derivative of the intensity is larger in magnitude than some thresholds and places where the second derivative of the intensity has a zero crossing. The droplets were first identified from the images by applying edge detection analysis [8], which is based on mainly creating color contrast and brightness. Then a histogram was created by counting the number of droplets in a specified area.

The total time for the recorded video images was 60 seconds. Since electrical field was applied during the zeta potential measurements oil droplets were moving. Therefore, the stationary droplets such as air droplets trapped in the measurement cell were identified by the developed MATLAB code using frame difference algorithm and these droplets were disregarded in the size analysis. The gray scale photographs were converted to black and white scales and an example can be seen in Fig. 1. It can be seen that a large droplet on the bottom of the right corner in the original image was removed in the analysis. Six frames of each second from the recorded video sequences were used to obtain the droplet size using the edge detection algorithm. 300 snapshots were taken from a video image in order to obtain the droplet size distribution at a specified experimental condition. The size distribution of droplets was evaluated for each snapshots and the average of these 300 snapshots was presented as the droplet size distribution at the given experimental condition.

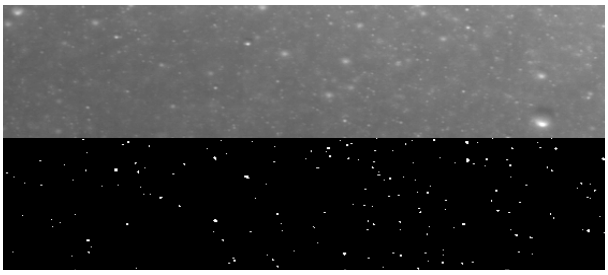

Fig. 1 Droplets identification from zeta potential images

III. Discussions and Results

A. Effects of Electrolyte Concentration on Zeta Potential

Fig. 2 shows the variations in the zeta potentials of EAL and FAM droplets depending on the concentrations of NaCl and $CaCl_2$. It can be seen that these oil emulsions are very stable whatever the concentration of NaCl is. The zeta potential values can reach as high as -70 mV. The surface electrical potential depends on the ionic composition of the surrounding medium due to electrostatic screening effects [3]. The ionic strength of the continuous phase has a strong influence on the zeta potential of the oil droplets, and this parameter controls the electrostatic interactions, thereby influencing the emulsion stability [7]. It is usually expected that addition of inorganic salts reduce the thickness of double layer causing reductions in zeta potential and, therefore, oil droplets can coagulate easier and emulsion becomes more unstable. Even though the zeta potentials of FAM droplets fluctuate it can be postulated that FAM emulsion cannot be destabilized by the addition of NaCl since the zeta potential is on the average of -60 mV. On the other hand, only 0.025 M addition of $CaCl_2$ is enough to get zero zeta potential for EAL emulsion. In the case of FAM emulsions, this value corresponds to 0.08 M $CaCl_2$. These results show that the presence of Ca^{+2} ions affects strongly the stability of EAL and FAM emulsions. According to these results, the addition of $CaCl_2$ can improve separation efficiency for the treatment of wastewaters containing EAL or FAM.

Zeta potential is a strong function of pH as well. Therefore, variations in the zeta potentials of EAL and FAM droplets depending on pH (2, 4, 6, 8 and 10) were also investigated (without addition of electrolytes). When EAL and FAM were mixed water, the emulsion pH was around 8. For both emulsions, a strong dependency of zeta potential to pH was observed. It decreased from 15 mV to -45 mV and -50mV for EAL and FAM, respectively, when pH changes from 2 to 10. These results show that these emulsions are stable in alkaline conditions and can be easily destabilized by the adjustment of pH.

B. Effects of Electrolyte Concentration on Droplet Size Distribution

The video images of oil droplets in the EAL and FAM emulsions show that they are covered a cloudy layer around them (Fig. 3). It is expected that this cloudy layer is formed by the adsorption of emulsifier chemicals in the metalworking fluids. The evaluated droplet diameters from the images include the thickness of the cloudy layer. The smallest droplet diameter that can be measured from the images is 2.5µm because of the sensitivity of the camera. The size distributions of EAL and FAM emulsions obtained from video images are shown in Figs. 4 and 5, respectively. In the graphs, the first column represents the oil droplet size distributions in the emulsions without addition of electrolytes. It is observed that that most of the droplets in EAL and FAM emulsions are smaller than 10µm and there are almost no droplets larger than 60µm. These results are confirmed by microscopic analysis as

ISSN: 2517-942X
Vol:7, No:11, 2013

well. The percentage of oil droplets for EAL in the size range of 0-10µm was increased by 17% and 31% by the addition of 0.01 M NaCl and $CaCl_2$, respectively. The corresponding increases for FAM were 18% and 34%. The percentage of increases or decreases in the droplet dimension ranges of 0-10µm and 10-30µm are presented in Table II depending on for all studied concentration of NaCl and $CaCl_2$. According to these results, the emulsions become closer to the monodisperse emulsions by the addition of electrolytes. The oil droplets do not coagulate by addition of NaCl and $CaCl_2$even though the electrolyte addition causes the decreases in the surface charges of oil droplets in the zeta potential measurements. In addition, the emulsions stabilities were tested with respect to time and no coalescence of oil droplets were observed in a week. It is observed that the addition of electrolytes causes mainly the decreases in the thickness of the cloudy layer around the oil droplets. The cloudy layer thickness is more sensitive to Ca^{+2} ions concentrations. It is expected that Na^{+} and Ca^{+2} ions react with the emulsifier chemicals and create in the reductions in the measured zeta potentials.

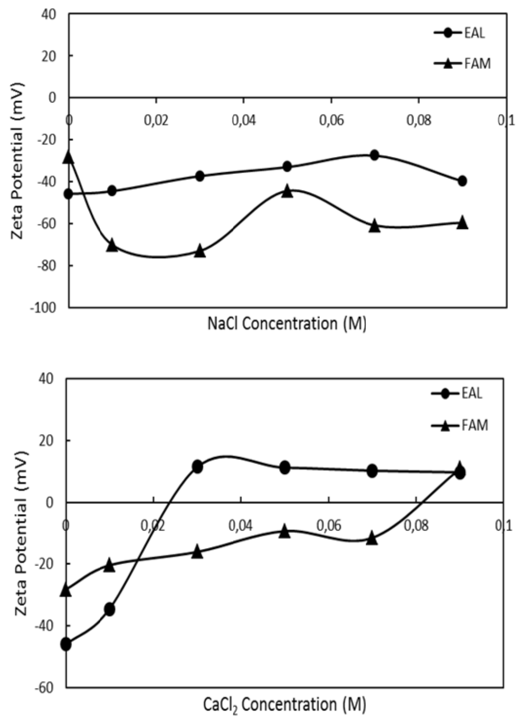

Fig. 2 Effects of electrolytes on zeta potential

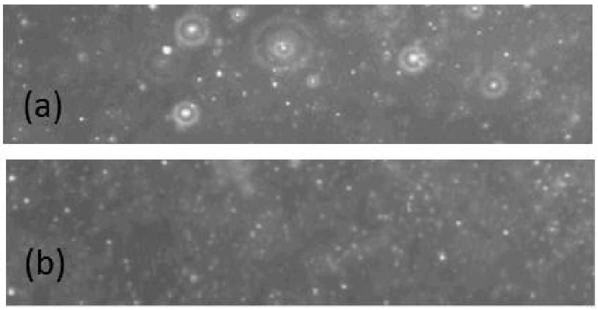

Fig. 3 Thephotographs EAL (a) and FAM (b) emulsions

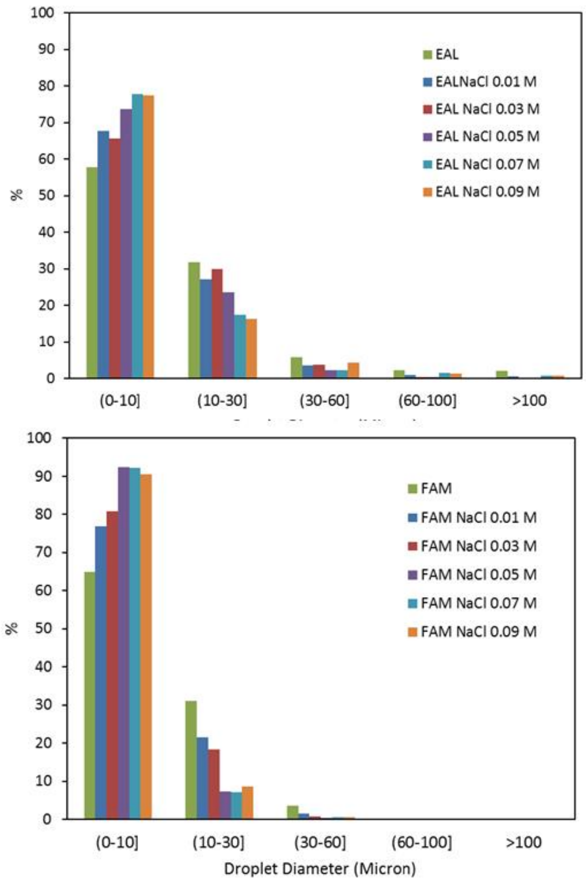

Fig. 4 Effects of sodium chloride addition on size distribution

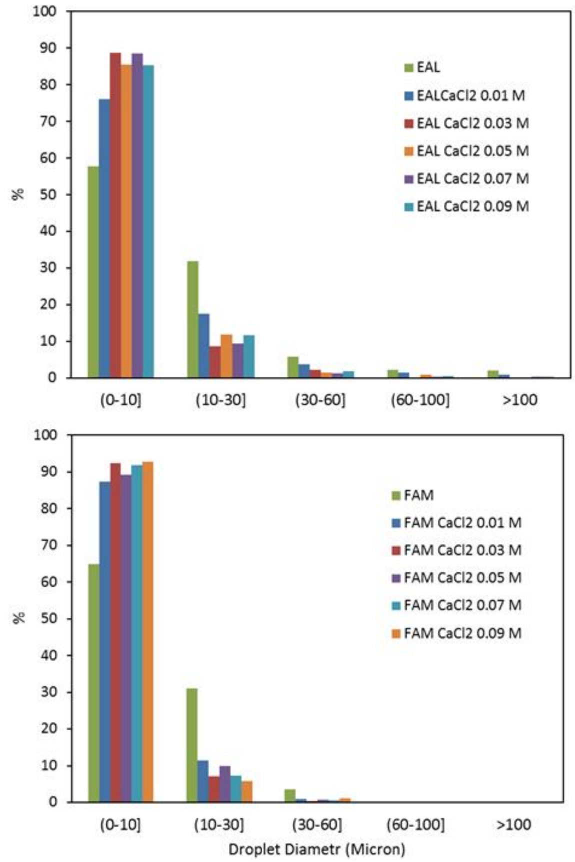

Fig. 5 Effects of calcium chloride addition on size distribution

The effect of pH on the size distributions of EAL and FAM emulsions were also investigated and the results for FAM emulsions are shown in Fig. 6. It can be seen that, similar to the electrolyte addition, monodispersed emulsions are obtained in the strong acidic conditions, i.e., the percentages of droplets diameters smaller than 10µm increases. On the other hand, the droplets diameters show a tendency to increase towards to alkaline conditions. For example, when pH equals 10, the droplets coagulate and almost 20% of oil droplets are larger than 100µm. Similar results were also obtained for EAL emulsions.

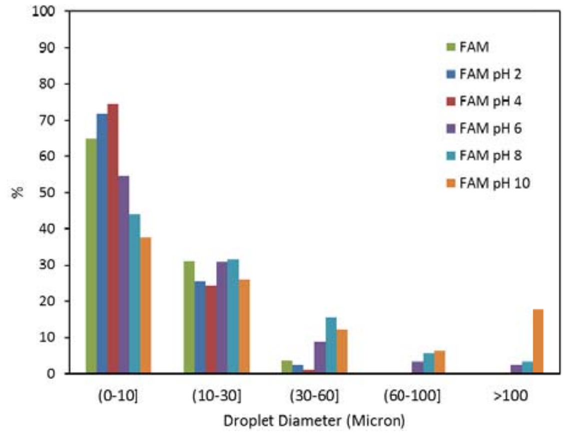

Fig. 6 Effects of pH on size distribution

IV. Conclusion

The droplet size distribution of oil emulsions produced by using metalworking fluids has been studied by analyzing the video images taken during the zeta potential measurements. The emulsions were prepared by using a mechanical mixture and most of oil droplets were smaller than 10µm. The emulsions were stable since the measured zeta potential as high as -70mV. The results show that the metalworking emulsions can be destabilized by the addition of $CaCl_2$. In addition, the droplet size distributions in these emulsions are much more sensitive to pH rather than the electrolyte addition.

TABLE II
Variations in Droplet Sizes (%) Depending on Electrolyte Concentration

	NaCl				$CaCl_2$			
	(0-10 µm]		(10-30 µm]		(0-10 µm]		(10-30 µm]	
Concentration (M)	EAL	FAL	EAL	FAL	EAL	FAL	EAL	FAL
0.01	17	18	-15	-31	31	34	-45	-63
0.03	13	24	-6	-41	53	42	-73	-77
0.05	27	42	-26	-76	48	37	-63	-68
0.07	34	42	-45	-77	53	41	-71	-76
0.09	34	39	-49	-72	48	43	-63	-81

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