THE DETERMINATION OF THE DISPERSE PHASE REFINEMENT ON WATER IN FUEL EMULSIONS WITH THE USE OF THIN-LAYER EMULSION IMAGES USING A MICROSCOPE AND A MALVERN MASTERSIZER 2000 DEVICE

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Abstract

The paper includes a proposal of methods for testing and evaluation quality and stability of water in fuel emulsion used in combustion tests on marine engine in MAN laboratory test bed. Attempts to satisfy the requirements set forth in Annex VI of the Marpol Convention and Resolution 33EU concerning, among other issues, the reduction of NO_x emission in exhaust gases from marine engines and boilers have resulted in further research on burning of fuel-water emulsions , a fuel considered to reduce the amount of NO_x. The research, within the scope of a project financed by the EU, is performed by the MAN company employing an engine in a test bed of the laboratory in Copenhagen. One of the elements in the research is to indicate a device for making emulsion, such as a static homogenizer developed at the Institute of Technical Marine Power Plant Operation, Maritime University of Szczecin. To obtain reliable results, it was necessary to design the methodology for the examination of emulsion quality. That was basically the objective of the research herein described.

Keywords: *marine fuel, water in fuel emulsion, the examination of emulsion quality*

1. Introduction

Aiming at reducing noxious components of exhausts from ship engines and boilers led to a series of investigations of fuel-water emulsion, i.e. its applications as a fuel reducing NO_x emission. There are two basic theories accounting for the effect of water on the process of water in fuel emulsion combustion in slow speed engines:

- microexplosion theory.
- theory of catalytic role water in fuel emulsion in self-ignition and residual fuel combustion [1].

Dooher, Gondberg, Lippmen, Wraight found from experimental research that for steam contained in a water droplet to undergo micro-explosion, it has to be stronger than fuel surface tension, and the time of water droplet expansion has to be less than fuel expansion time. When this time is longer, steam can penetrate through the droplet surface without its blow-out [1].
The droplet expansion time is described by this equation:

$$\tau_{roz} = \frac{1}{\sqrt{29\Pi}} \cdot \left(\frac{\rho}{\delta}\right)^{\frac{1}{2}} \cdot r_o^{\frac{3}{2}}$$

where:

δ - surface tension [N/m];
ρ - density [kg/m^3]:
r_o - droplet radius [m];

Calculations were made to determine the maximum size of a water droplet in a fuel droplet that causes micro-explosion. This size is 20 μm. The research also implies that emulsions rich in water will burn correctly if smaller diameters of disperse phase particles (water) in fuel are obtained.

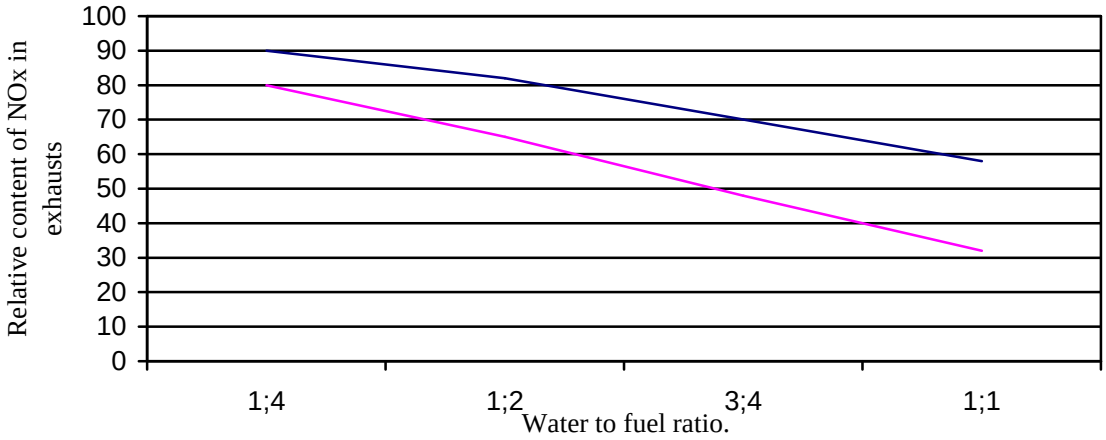

Fig. 1. Effect of water content in a fuel-water emulsion on the amount of NOx in internal combustion engine exhaust gases [2]

Certain research work had been done by this author before joining the research team in Copenhagen where the combustion of fuel-water emulsion was examined in slow speed engines by the MAN company. At the Institute of Technical Marine Power Plant Operation research was done to unequivocally define the emulsion 'quality', understood as the degree of dispersion of water in fuel after homogenization and as the durability of the prepared emulsion, understood as a period in which disperse phase droplets do not increase due to coagulation. The most useful for the burning of emulsion in internal combustion engines are water-in-fuel emulsions, i.e. emulsions in which fuel makes up the liquid phase while water is the disperse phase with most of droplet diameters ranging from 0.5 to 2μ. It was assumed that stability is the time when emulsion maintains its properties making it suitable as a substitute fuel. This time for emulsions made from various fuels is defined experimentally [3]. Because of this, it is important to unequivocally determine the size structure of water droplets (disperse phase) in a fuel-water emulsion used in comparative analysis. To this end an attempt was made to develop a method of evaluating the sizes of the disperse phase (water droplets) of fuel-water emulsion based on microscope images assuring stable conditions for observations of examined emulsions, that is a steady thickness of the observed emulsion layer and stable measurement temperature independent of the time of observation.

2. Microscopic examination of the water in fuel emulsion

Experience from previous tests [4] showed the need for the elimination of convection movements in the examined emulsion caused by heat from illumination of the preparation from the bottom when samples are observed in the transmitted light. Stable observation conditions were obtained using a base plate with a cooled chamber of own design shown on fig.2. Residual fuel with kinematic viscosity of 380 mm^2/s (cSt) was used as the base for producing emulsion. After heating the fuel in an electrical furnace to the temperature of 50-

60°C, it was filtered in a vacuum filter in order to eliminate solid contaminants that might cause coagulation of water into larger droplets. Then, by employing a laboratory homogenizer emulsion specimens were prepared containing 10%, 30%, and 50% of water. Emulsion samples were produced in the homogenizer at the homogenization time of 30 seconds.

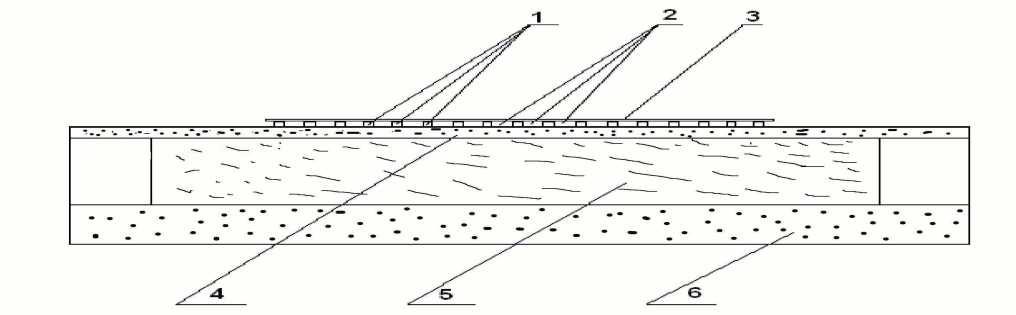

Fig. 2. The design of a base plate with a cooled chamber: 1- distance wires fixing the sample thickness(depending on the plate used, their thickness was, respectively, 0.1 and 0.05 mm); 2- specimen; 3- microscopic cover glass; 4- base glass; 5- chamber cooled by liquid (distilled degassed water at ambient temperature); 6- chambers bottom plate

The equipment used for the research included an Olympus BX50 test bed, a PC computer and Multiscan software, an advanced system of imaging and image analysis created by Computer Scanning Systems.

Emulsion samples were examined on microscopic plates of three types:

- glass Petri dish, where the specimen is put under a microscopic cover glass.
- polycarbonate plate with a cooling chamber allowing to maintain constant temperature of the examined specimen. The design of the base plate assured a steady distance of 0.01 mm between the cover glass and the base plate.
- Polycarbonate plate with a cooling chamber allowing to maintain constant temperature of the examined samples, with a steady distance of 0.05 mm between the cover glass and the base plate with specimen.

The condition of emulsion was observed:

- Immediately after it had been made,
- Three minutes after homogenization,
- Six minutes after homogenization,
- 15- 20 minutes after homogenization of the emulsion.

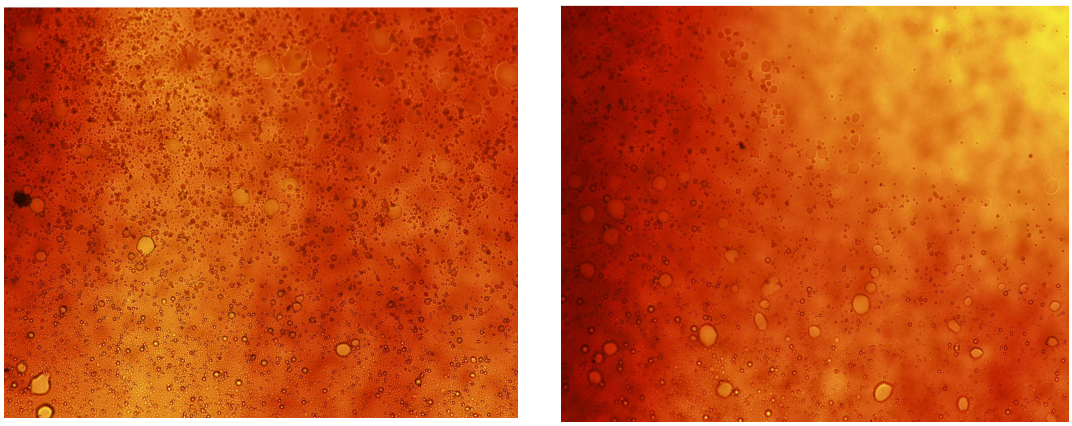

Fig.3. Examples of images of the emulsion made from fuel containing 10% of water, immediately after its homogenization. The measurement performed on a plate with a 'cooling chamber' with distance wire diameter 0.01mm

The created sample was observed with a microscope at 40X magnification. The image was digitally recorded by a camera fitted in the microscope tube. Four photographs were taken at randomly chosen places of each sample. The next step was counting the number of water droplets in the emulsion. After counting the droplets and grouping them in certain size ranges using Multiscan software, the results were gathered in a final report.

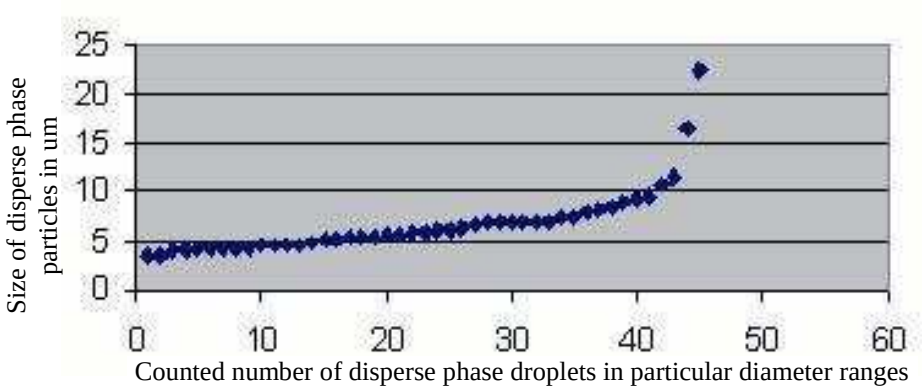

Fig.4. Number of disperse phase particles in emulsion containing 30% water immediately after homogenization, determined on the basis of observed particles

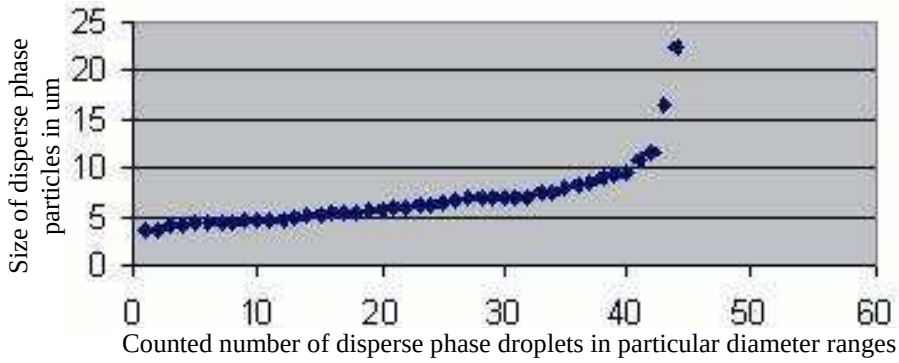

Fig.5 . Number of disperse phase particles in emulsion containing 30% water six minutes after homogenization, determined on the basis of observed particles

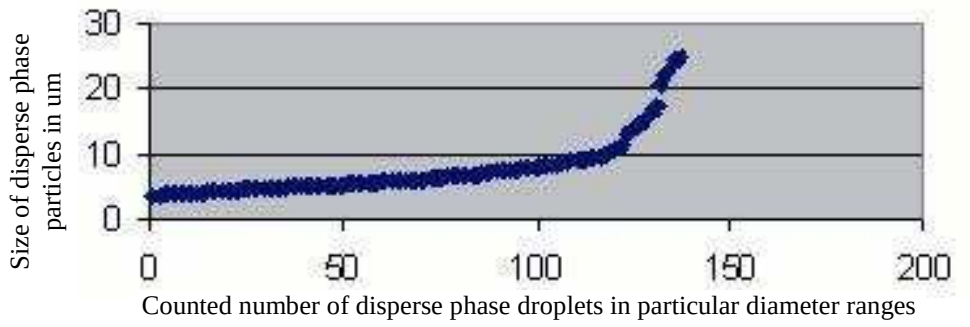

Fig.6 . Number of disperse phase particles in emulsion containing 30% water 20 minutes after homogenization, determined on the basis of observed particles

The application of distance wires and springing push-buttons on cover glasses made it possible to observe emulsion of constant layer thickness, thin enough to obtain an image in transmitted light. Attempts to observe the sample on a plate with the cooling chamber with "distance wire" diameter of 0.05 mm did not yield proper results as the amount of light passing through the examined sample is not sufficient. Consequently, microscopic observation is not possible, nor are measurements of water droplet sizes in the emulsion. The research results have shown that recurrent results can be obtained by placing droplets of fuel-water emulsion on polycarbonate plates with a cooling chamber. Plates of the same design

made of glass caused coagulation, thus the size of water droplets increased significantly when in contact with the base or cover glass. The phenomenon was observed as early as three minutes after placing the emulsion on the plate. It was sufficient to cool the chamber for microscopic observation with a stream of water at a constant temperature of 25^0C provided for by an ultrathermostat.

3. Examination of the water in fuel emulsion with the use of a MALVERN Mastersizer 2000 device

A more accurate distribution of the disperse phase droplet diameters in the produced emulsion was obtained by examining fuel-water emulsion based on residual fuel The research aimed at developing methods of measuring the distribution of water droplet size in an emulsion produced on the basis of residual fuel, considered as a supplementary measurement to microscopic observations. Droplet diameter measurements were performed in a MALVERN Mastersizer 2000 device operating on the principle of laser light diffraction measurements. As residual fuel is practically non-transparent (light can pass through a very thin fuel layer), and examinations using this device require the specimen obscuration ranging from 10 to 20%, measurements can be performed only when the fuel or emulsion is properly diluted in a special thinner.

Experience gained from previous research shows that the best thinner for this purpose is n-heptane. Residual fuels, apart from very different physical and chemical properties, also have varied optical properties. Therefore, for measurements to be carried out, it was necessary to dissolve residual fuel of the examined specimen to 0.4 ml in 1000 ml of n-heptane so that the required obscuration could be obtained. Any under fuel under examination has to have such a concentration that the initial obscuration reached by subsequent approximations does not exceed 11 -12%. The determination of water droplet size distribution in an emulsion is considered as a relative measurement in reference to residual fuel containing a large amount of asphaltene particles and solid and liquid contaminants in a size range from nanometers to about 100 μm. This leads to a conclusion that almost all water droplet diameters will fall into the same size range as ashaltene and contaminant particles. The Mastersize device is not capable of identifying which particles (or droplets) it has to deal with; whether these are spheres or irregular particles, whether they are transparent or perhaps absorb light completely; or what is the refraction coefficient of particles and that of the liquid. The device works according to a model of particle distribution assumed by the operator, in which the above parameters have to be strictly defined. So now the basic difficulty of the planned measurement can be seen: in n-heptane there will be both spherical particles (water) and irregular ones (the remaining particles), transparent and non-transparent and those with very different refraction coefficients. It was assumed that first diameter distribution measurement will be executed for a specimen of residual fuel, then another measurement of emulsion specimen containing the same amount of fuel as the reference sample. The difference in measurements should give the examined distribution of water droplet diameter in the emulsion. As water is the medium of our interest, not asphaltenes, both measurements should be performed for the water droplet model. The examinations brought unexpected results, that is diameter distributions divided into three groups of diameter sizes for both fuel and emulsion. This was most probably due to the fact that the computer treated asphaltene particles as water particles. An attempt to determine the difference in results in order to define the distribution of water particles gave no results, because the differences in the diagram were partly positive and partly negative. It was assumed that this explains the behaviour of water droplets in the emulsion which attract polar particles of resins and asphaltenes. Therefore, from the optical point of view a water droplet in the residual fuel-based emulsion should be

treated as an asphaltene particle. The computer program of the measuring device makes it possible to calculate the results according to a new model without repeating the examination. Presented below are examples of the results obtained from files counted by Malvern2000 device.

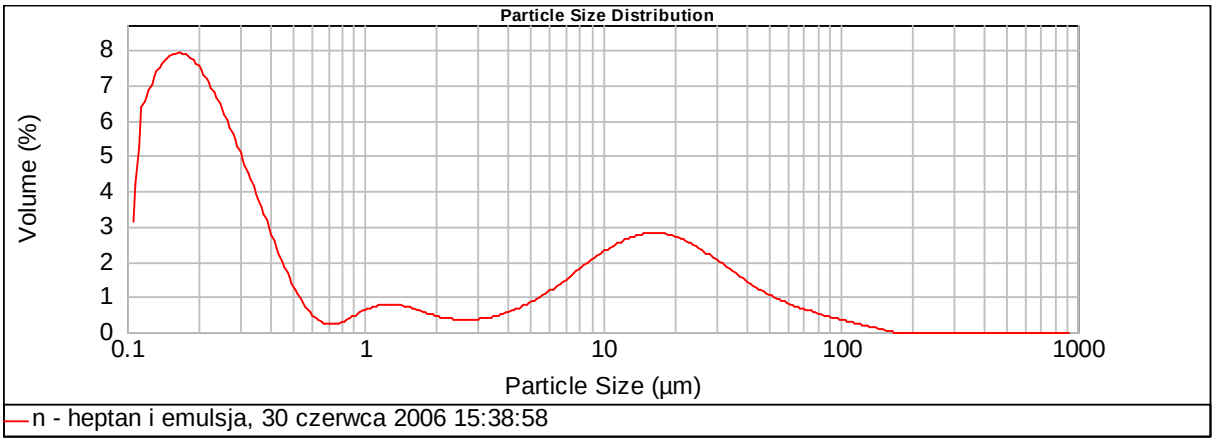

Fig. 7. Emulsion observed as water droplets

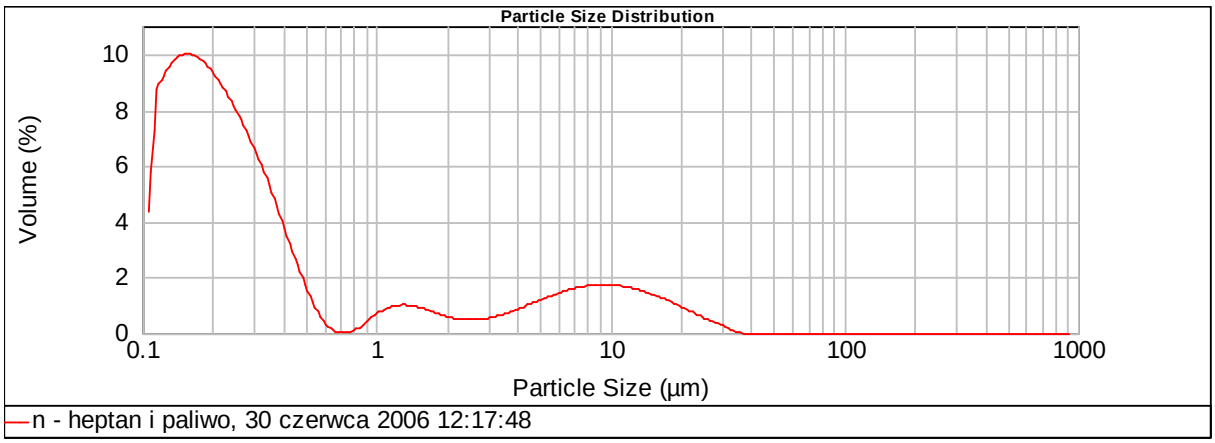

Fig. 8. The background fuel

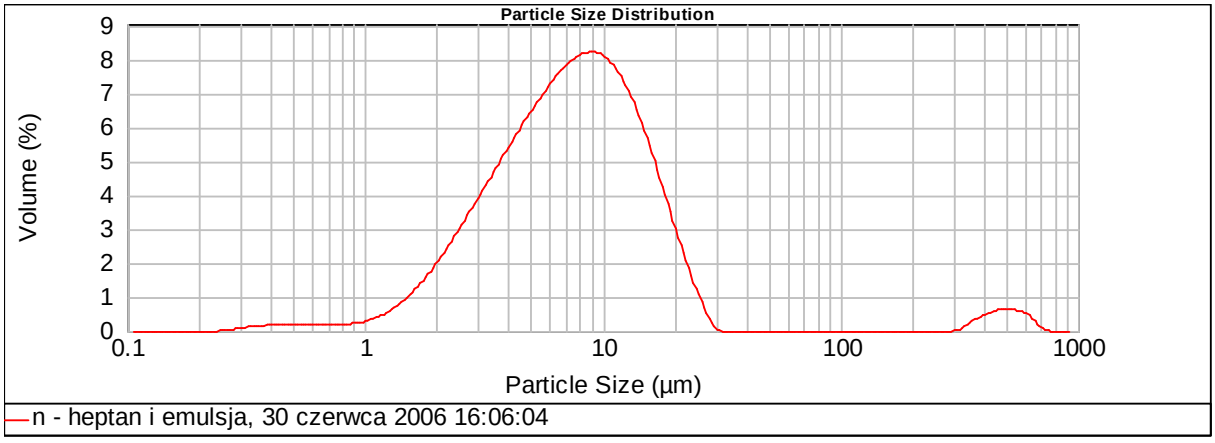

Fig.9. Emulsion observed as asphaltenes after taking away the background

The distribution for residual fuel is a model one, whilst that for the emulsion is somewhat ‘too small’. The reason for this was the presence of various particles in the solution that were not additive in the measuring device program. The use of base fuel solution as the background turned out to solve the problem. The above methodology of measuring the

emulsion of water in residual fuel should be verified by microscopic examination. However, it should be done for the value of refraction coefficient for water droplets covered with resins and asphaltenes and for their value of light absorption coefficient, not for the procedures developed. The values of the coefficients used during the examinations described are only approximate, but very probable.

4. Conclusion

The executed tests resulted in a method allowing to obtain reliable information on the refinement of the disperse phase (water droplets) in fuel-water emulsions and the distribution of water droplet size in the emulsion, which allows to observe and record changes in emulsions to be used in the research. In this way the comparative analysis for various types of emulsion will be burdened with smaller errors.

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