Optical study of the concentration and particle size distribution of octadecylamine in water

N M Skornyakova, A V Anikeev and V A Parshin

V.A. Fabrikant Physics Department, National Research University "Moscow Power Engineering Institute", Krasnokazarmennaya 14, Moscow, 111250 Russia

E-mail: nmskorn@mail.com

Abstract. The objective of this study was to determine the size of the investigated particles in water. The particles studied were octadecylamine inclusions which are invisible to the naked eye. To increase the accuracy of the results obtained, the study was carried out using two optical methods: direct video surveillance in a digital stereo microscope and laser diffraction method.

1. Introduction

Octadecylamine is a chemical compound used for the surface preservation of metals [1]. It is applied in power and other areas. Derivatives and salts of octadecylamine are toxic to bacteria and microorganisms. To implement the process of sorption of octadecylamine molecules on the functional surfaces of structural materials, it is necessary to carry out preparatory operations for the preparation of a highly concentrated aqueous emulsion and for its transportation to the circuit. It is assumed that the method of the emulsion preparation affects the particle size and, accordingly, the properties of the emulsion.

The objective of this study was to determine the size and concentration of the studied particles in water. The particles studied were octadecylamine inclusions. The main difficulty was that visually these inclusions were not observed, since the particles of octadecylamine are transparent [2]. To improve the accuracy of the results obtained, the study was carried out using two optical methods: direct video surveillance in a digital stereo microscope and laser diffraction method.

Stereo microscopes belong to the category of optical microscopes. They work on the principle of light reflected from the object being studied. Stereo microscopes are equipped with two eyepieces, the image in which is displayed at slightly different angles. This is what makes it possible to obtain a volumetric enlarged image and makes it possible to evaluate the dimensions of the investigated object.

Stereo microscopes are used in areas where it is necessary to obtain a three-dimensional image, and the perception of contrast and depth is important for the study of the object. In addition, the optical system of the stereo microscope gives a "direct" image of the sample, which allows the use of similar equipment in industry for manipulating microscopic objects. As a rule, stereo microscopes have an upper illumination. However, the presence of a lower backlight is not excluded. Also, manufacturers often make it possible to understaff a microscope with additional sources of illumination: side and ring illumination. For illumination, LED, halogen, fluorescent lamps and even incandescent lamps can be used. Many stereo microscopes show a relatively small magnification of the image: from 10 to 100 times. Although there are instances that can increase the image 200 times.

Laser light (monochromatic, coherent light) interacts with the particles, which have to be characterized in terms of particle size. In dependence of the particles' size, the light waves are scattered
by the particles in a characteristic manner: the larger the particles are, the greater is the scattering in forward direction. With particles smaller about 100 nm, the scattering intensity is nearly identical in all directions. Laser diffraction analysis is based on the Fraunhofer diffraction theory, stating that the intensity of light scattered by a particle is directly proportional to the particle size [3]. The angle of the laser beam and particle size have an inversely proportional relationship, where the laser beam angle increases as particle size decreases and vice versa [4].

2. Experimental setup

2.1. Research with stereoscopic microscope
The Altami SM0745 digital stereo microscope was used in this work for direct observation of octadecylamine inclusions in water. In the study, in some cases, an additional ring illuminator was additionally used. Since in this case it was assumed not a single measurement of the particle diameter, but a set of statistical information to obtain the distribution of inclusions by size and determination of concentration, then the obtained images were simultaneously recorded by each method and transferred to a computer.

Together with a stereo microscope, a PL-B621-MF video camera was used, designed to work with microscopes. The PL-B621-MF uses the standard FireWire interface for plug-and-play connection with a host computer. The camera is equipped with a 1.3 MPx CMOS sensor and can provide 30 frames per second at full resolution, or 100 frames per second at VGA resolution. PL-B623-MF is TWAIN, WDM, and IIDC 1.31 (DCAM) compatible.

2.2. Research using a laser diffraction method
Currently, a large number of optical methods for diagnosing flows have been developed: shadow, interference, polarization, holographic. The essence of the laser diffraction method is to determine the size of a particle located in the path of the beam propagation by the period of the emerging diffraction pattern. Laser measurement methods are created to solve problems when the environmental parameters are determined from the measured parameters of the laser radiation at the outlet of the medium under study and the measured (known) parameters of the laser radiation at the input. The laser diffraction method or the small angle method is used. In it, the calculation of the diameter of the present particle is performed according to the formula:

\[ d = \frac{1.22\lambda}{R_{\min}} \]

where \( d \) is the particle diameter; \( \lambda \) is the light wavelength; \( R \) is the distance from the investigated plane or lens to the registration plane; \( x_{\min} \) is the position of the first diffraction minimum.

Experimental setup was developed to solve this problem. It is shown in figure 1. The light from the laser (1) passes through the optical system (2), the elements of which are selected so that the object under study (3) is in the waist of the laser beam. Then the scattered radiation enters the screen (4), the image of it is recorded on the camera (5). The test object is a microliter volume cuvette filled with the test liquid. If the particle under investigation is caught in the path of the laser beam inside the volume under investigation, a diffraction pattern appears on the screen.

As the recording device, a Nikon J1 NIKKOR camera was used. An optical system composed of two collecting lenses was used to obtain the required radius of the waist of the laser beam at the measurement point. The volume of the cuvette used in the experiment was 2 ml. The geometric dimensions of the cell (length, width, height) are equal respectively: 24.1 mm, 7.4 mm and 40.2 mm. Since it was necessary to scan the cross section of the test volume with a laser beam, the cuvette was mounted on a two-coordinate table with micrometric screws vertically and horizontally.
3. The results of visualization

As a result of visualization of inclusions using a stereo microscope, it was possible to determine that two types of particles are present in the provided emulsion samples. The first type is close in shape to spherical or elliptical particles; hereinafter, it will be called “typical” inclusions. The inclusions of the second type are shapeless, can be threadlike structures, both elongated and looped, in what follows we will call “atypical” inclusions. Examples of observed images are presented in figure 2.

Figure 2. The examples of visualization in stereo microscope.

A study of the provided samples by diffraction method was performed at room temperature. Liquid was scanned from the edge of the cell to its bottom for each series of measurements. The movement of the cell in the cross section of the beam was carried out by rotating micrometer screws. The image on the screen was recorded each time a particle fell into the measuring region. Examples of observed diffraction patterns are presented in figure 3.
Figure 3. The examples of observed diffraction patterns.

4. The results of measurements
A study was made of the particle size in a suspension of octadecylamine depending on the method of creating the emulsion. The first emulsion was obtained by mechanical stirring of a “powder” of octadecylamine in hot (80°C) water. The second was prepared by dosing the octadecylamine melt through an ejector into a stream of water at 80°C; sampling was carried out immediately after the ejector.

Figure 4 presents the statistical results of the study of the first emulsion. In this emulsion, only “typical” inclusions were observed. On the histogram along the horizontal axis: the diameter of the encountered particles in µm, the left boundary is 3.68 µm, then in increments of 0.7 µm; along the vertical axis: the percentage of particles are given in range of diameters in the liquid (100% – all particles in the test volume). The largest number of particles between diameters of 3.7 µm and 4.4 µm is associated with the resolving power of the microscope, that is, all particles of a smaller diameter also fall into this range.

Figure 4. Histogram of particle size distribution in emulsion 1.
By analysing images from a stereo microscope, it is also possible to determine the concentration of inclusions in the emulsion. The total volume of particles per 1 mm$^3$ of liquid was $4.6 \times 10^{-5}$ mm$^3$. The density of octadecylamine is 0.86 g/cm$^3$ or 0.86 mg/mm$^3$ [5]. Accordingly, the concentration of 39.56 mg/L.

In the emulsion prepared by the second method, both “typical” inclusions and “atypical” were observed. If for “typical” particles a histogram of the diameter distribution was constructed (assuming that the particles are spherical), then for the “atypical” ones, histograms of the horizontal, vertical, and interspersed areas were constructed.

In figure 5 a bar graph of particle diameter distribution for “typical” particles are presented. On the histogram along the horizontal axis: the diameter of the encountered particles in µm, the left boundary is 1.401 µm, then in increments of 0.42 µm; along the vertical axis: the percentage of particles in a given range of diameters in the liquid (100% – all particles in the test volume).

![Figure 5. Histogram of “typical” particle size distribution in emulsion 2.](image)

Figure 6 shows the histograms of the distribution of the diameters of the “atypical” inclusions separately horizontally and vertically. Since the particles are far from always oriented horizontally or vertically, the histograms in figure 6 do not give complete information. It will be statistically more correct to compare the inclusions in the liquid over the area. The histogram of the area distribution for “atypical” particles is shown in figure 7. On the histogram along the horizontal axis: the area occupied by particles in square µm, the left border is 27.04 µm$^2$, then with a step of 14.07 µm$^2$; along the vertical axis: the percentage of particles in a given range of areas in the liquid (100% – all particles in the test volume).

The total volume of “typical” particles per 1 mm$^3$ of liquid is $9.6 \times 10^{-6}$ mm$^3$. Accordingly, the concentration is 8.26 mg/l (for "typical" particles). The total volume of “atypical” particles per 1 mm$^3$ of liquid is $8.2 \times 10^{-5}$ mm$^3$. Accordingly, the concentration is 70.52 mg/l (for “atypical” particles).

For clarity, the difference between the characteristics of the first and second emulsions, a comparative histogram is shown in figure 8. Blue color (empty rectangles) – liquid 1, red color (filled rectangles) – liquid 2.
Horizontally

**Figure 6.** Histogram of “atypical” particle size distribution in emulsion 2.

Vertically

**Figure 7.** Histogram of the distribution by area of “atypical” particle in emulsion 2.

**Figure 8.** Comparative histogram.
5. Conclusion
The possibility of complex diagnostics by optical methods of the parameters of optically transparent inclusions in water is shown. During the study it was determined that the parameters of the resulting octadecylamine emulsions depend on the method of their preparation. With mechanical stirring of the “powder” of octadecylamine in hot (80°C) water, the particle diameters are obtained in the range from 3.7 to 18.2 µm, while all are of a “typical” type. When preparing the emulsion by dosing the octadecylamine melt through an ejector into a water stream at 80°C, the scatter in the diameters of the “typical” particles is smaller and ranges from 1.4 to 11.8 µm, but the emulsion is an order of magnitude larger (than "typical") the number of "atypical" particles.

6. References
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