Introduction

In connection to our investigation of Ludox colloidal silica (1) it was desirable to determine the particle size by a method entirely different from light scattering in order to compare and estimate the reliability of the results obtained by means of this method. Electron microscopy seemed as the most convenient method, more so since it does not give only data of particle size, but also of polydispersity and of aggregation of particles. The comparison between light scattering and electron microscopic data was most convenient method, more so since it is a proof of the reliability of the correctness of the theories involved.

Experimental

The samples of Ludox(3) were the same as those used for light scattering work [see Table 2 in ref. (1) for technical specifications and relevant data]. The samples were investigated by electron microscopy both uncentrifugated and centrifugated. The centrifugations were performed as previously (1).

For electron microscopic investigations a Trüb, Täuber & Co. instrument (Model KM 4) was used. The distortion of the image was compensated by separate adjustment and careful measurement of the currents in the double electromagnetic projective lens (5). The instrument was calibrated by means of single samples of striated diatom cell walls, the selected parts of which were at first photographed in the electron microscope and afterwards measured by means of an ordinary microscope at a magnification of 1000 times. The obtained results were then compared with measurements on monodisperse standard polystyrene latex samples (4). Both data were in a good agreement. The working conditions of the electron microscope were kept constant by inspection of all measuring devices. All photographs were taken at a direct magnification (i.e. on the screen) of 10,500 times. The measurements of particle sizes were done directly from photographic plates using a calibrated low power microscope at a magnification of 10 times.

The Ludox samples used for electron microscopy were diluted with distilled water immediately before use (1 drop of Ludox to 20 ml. of water). Small droplets of these solutions were transferred by means of fine capillary tubes to bronze grids coated with a thin “Mowital” membrane stripped from glass slides. The irradiation of the object was short as the different parts of the preparations were brought into the electron beam immediately before the exposure of the plates. However, no perceivable changes of the samples could be observed even at a longer exposition to the electrons.

For the statistical analysis counts of about 200 particles were made, except in one case when more than 500 particles were counted.

References


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Electron Microscopy of Ludox Colloidal Silica

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With 5 figures in 6 details and 2 tables

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Results and Discussion

The initial aim of our electron microscopic study of Ludox was an attempt to detect the differences in the degree of aggregation of uncentrifugated and centrifugated samples of Ludox. These differences were revealed by light scattering measurements and fully confirmed by present studies. We have chosen as representative examples of electron micrographs those on Figs. 1 and 2 for Ludox sample II, which was the oldest one and in which the phenomena of aggregation were mostly pronounced. Similar differences in aggregation of uncentrifugated and centrifugated preparations were found in all Ludox samples investigated. Although some aggregating of particles during the process of drying is not completely improbable, the differences for the two preparations are striking. Thus, the electron microscopic findings paralleled closely the results of light scattering dissymmetry measurements [see Table 8 in ref. (1)].

Unfortunately, it was not possible to correlate the degree of aggregation with the age of the samples. All samples (except the