Preparation of La(OH)₃ colloids and tactoid structures

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With 5 figures

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Whilst impressive number of precipitation studies on metal hydroxides have been presented, relatively little was published on the phenomena of weak colloid interaction resulting in orientation of the colloid particles (1, 2). Our paper contributes to the knowledge about appearance of hydroxide tactoids and "schiller layers". Such aggregation types were already found in other colloidal systems (1, 3, 4) involving transient metal oxide/aqueous electrolyte equilibria.

Experimental

All the chemicals applied were of analytical grade purity. The concentrations of La(III) solutions were determined complexometrically (5). The solutions and bidistilled water were protected from atmospheric CO₂.

The results shown in figures 1, 2, 4 and 5 were obtained by "in situ" experimental method (6). This method was adopted as rapid and convenient for the investigation of precipitation phenomenology. The precipitation components were separated in two series of test tubes, each series containing the gradient in concentration of one precipitation component. The final systems were prepared by mixing the contents of two proper test tubes from both series.

For comparative study of La(OH)₃ colloids the repetitization of La(OH)₃ gel was performed. The gel was obtained using von Weimarn’s oversaturation principle. The precipitation was performed from concentrated LaCl₃ and ammonia or sodium hydroxide solutions in the ice-cooled bath. The gelatinous La(OH)₃ precipitate was converted to sol by washing out the salt and base with bidistilled water. The stable sol was obtained in pH 9.5—8.5 range as measured immediately after repetitization.

The intensity of the scattered light was measured at 535 nm which corresponds to the scattering maximum of the sol. As reference standard the polystyrene latex was used. The pH measurements were performed using potentiometric method and glass-calomel electrodes couple. The electron micrographs *) were taken using standard method.

Results and discussion

The results of the precipitation study are summarized in figure 1. The diagram in figure 1 indicates various phases in transition

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Fig. 2. The intensity of scattered light vs. dilution of lanthanum(III)hydroxide sol

The stability of the concentrated sol was examined by simple dilution with bidistilled water (fig. 2). The results indicate that the sol was in the same state of aggregation in the region approx. $3 \times 10^{-3}$ to $3 \times 10^{-4}$ mol/l La(OH)$_3$. Moreover, the slope of the line was constant over a period of two days. Similar results were obtained for the sols prepared by repectziation. It was concluded that the two classes of colloids should show the similar shape and aggregation characteristics. Actually, this was not the case. The sol obtained by repectziation gave needle-shaped tactoidally not orientated particles (fig. 3). For this difference we have no plausible explanation.

Figure 4 and figure 5 represent electron micrographs of the samples from the “sol region” of figure 1. From our preliminary results it follows that the formation of La(OH)$_3$ colloids is a relatively slow process, the fact which qualitatively accounts for the particles size uniformity. We adopt an explanation in terms of controlling influence of viscosity on the diffusion of polynuclear La(III) species

from homogeneous solution to the coarse La(OH)$_3$ sediment. The dashed line above the “sol” and “sol-sediment” regions indicates the limits of observation.

Fig. 3. Electron micrograph of lanthanum(III)hydroxide sol ($1.0 \times 10^{-2}$ mol/l La(III) and pH 8.5, colloid prepared by repectziation)

Fig. 4. Electron micrograph of lanthanum(III)hydroxide “schiller layers” formation ($1.05 \times 10^{-2}$ mol/l LaCl$_3$ and pH 8.1, “in situ” preparation)