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Precipitation of Thorium Hydroxide in the Presence of Gelatin and Sodium Nitrate

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The precipitation of thorium(IV) hydroxide and its interactions with gelatin were investigated in the system: gelatin – $Th(NO_3)_4$ – $NaNO_3$ – (HNO_3 or $NaOH$). The samples were prepared by mixing the aqueous solutions of the system components and kept at 293 K. The HNO_3 and $NaOH$ were pH-adjusting components, while $NaNO_3$ uncoiled gelatin molecules, thus enhancing their interacting power. The precipitation systems were investigated using nephelometry, microelectrophoresis, X-ray diffraction, IR spectroscopy and scanning electron microscopy. In dependence on pH, the characteristic sequence of states was observed: clear solution I, gel (very turbid, strong, elastic and thixotropic), clear solution II and flocks settled as sediment. This sequence of states was discussed with regard to three processes that occur with pH-increase: (a) hydrolysis of Th^{4+} ions followed by formation of hydroxy complexes and colloidal thorium(IV) hydroxide, (b) dissociation of the functional groups of gelatin and (c) interaction between the $Th(NO_3)_4$ component and gelatin. It was shown that the gel state consisted of colloidal particles of thorium(IV) hydroxide, which were linked by uncoiled gelatin molecules into a network of gel structure.

INTRODUCTION

Nuclear scientists used to pay great attention to the preparation of ThO_2 ceramics, because thorium can be used as nuclear fuel. Thorium undergoes nuclear reactions as follows:

$$^{232}_{90}Th + ^{1}_{0}n \longrightarrow ^{233}_{90}Th \xrightarrow{\beta^-} ^{233}_{91}Pa \xrightarrow{\beta^-} ^{233}_{92}U \qquad (1)$$
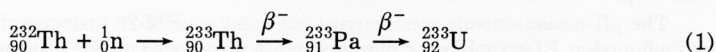
Highly dense ThO_2 and $(Th,U)O_2$ kernels are used in the fabrication of reactor fuel elements for the high temperature reactor (HTR).[1] Colloid-chemical procedures of the preparation of ThO_2 and $(Th,U)O_2$ microspheres are technologically more convenient than the powder metallurgical processes. An important step in the preparation of ThO_2 ceramics is the precipitation of $Th(OH)_4$ in accordance with the reaction:

$$Th(NO_3)_4 + 4NH_4OH \rightarrow Th(OH)_4 + 4NH_4NO_3 \qquad (2)$$
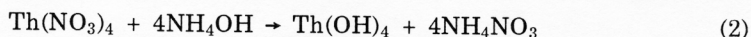
ThO_2 can be produced by calcination of $Th(OH)_4$:

$$Th(OH)_4 \rightarrow ThO_2 + 2H_2O \qquad (3)$$
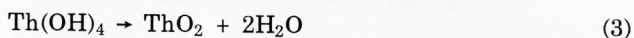
In order to obtain oxide ceramics of good properties, it is important to control the size of primary particles and their morphology. Sol-gel procedures are convenient for this purpose.[1,2]

In spite of the fact that thorium(IV) hydroxide is an important material in nuclear technology, its colloid chemistry has been insufficiently investigated compared to some other metal cations. Generally, Th^{4+} hydrolyzes at relatively low pH values and, in this process, the pH of the appearance of $Th(OH)_4$ phase from the aqueous solution is shifted to lower pH values in relation to Me(III)– and Me(II)– hydroxides. The distribution of different hydrolytical species of Th^{4+}, in dependence on pH, has been reported in the literature.[3]

In previous papers,[4-8] specific colloid interactions between Me(III)– or Me(II)– hydroxides with amphoteric polyelectrolyte gelatin were discussed. Under certain experimental conditions, Me^{3+} and Me^{4+} ions undergoing hydrolysis were found capable of forming gel through interactions with gelatin.

The aim of the present work was to obtain more information about the precipitation of thorium(IV) hydroxide and its interactions with gelatin, as well as to complete our results, obtained in our previous studies, on the influence of amphoteric polyelectrolyte gelatin on the precipitation processes and colloidal state of different metal hydroxides. The concentrations of $Th(NO_3)_4$ and pH-adjusting components were systematically varied.

EXPERIMENTAL

Analytical grade chemicals were used. A 0.80% gelatin (Merck, Art. 4078) in doubly distilled water was prepared before each experiment by swelling a weighed amount of gelatin in water for half an hour at 320 K, and then by cooling it to 293 K. The isoelectric point pH_{IEP} of this gelatin was determined at 4.86 ± 0.16.

The samples were prepared, within an interval of 15 seconds, by mixing aqueous solutions of the following components: I–gelatin, II–$Th(NO_3)_4$ and III–$NaNO_3$ + (HNO_3 or $NaOH$). I, II and III denote the respective test tubes. The sequence of mixing the contents of the test tubes was the following: III–I–III–I–II–III–II–III–II–III–I–II. Glass test tubes with flat bottoms and of the same wall thickness (outer $\Phi = 15$ mm and inner $\Phi = 13$ mm) were used.

Two colloid-chemical effects, elasticity and thixotropy, were observed during investigation of the samples. Selected samples of the gels »$Th(OH)_4$–gelatin«, which showed elastic behaviour, could return to the starting position after the mechanical force was stopped. The effect of thixotropy was observed in a traditional manner.

The pH-measurements were carried out using a pHM-26 instrument and electrodes produced by Radiometer. Electrophoretic measurements were based on the ultramicroscopic method using

a double cell of a circular cross section.[9] The relative nephelometric values, τ, of the samples were determined for proper times, with a Zeiss SPECOL-10, in scattered light at an angle of 135°, using the wave length at 535 nm. XRD analysis of the precipitates was performed with a Phillips diffractometer. IR spectra were recorded using a Perkin-Elmer 580 B spectrometer. Selected samples were also investigated by scanning electron microscopy (SEM).

RESULTS AND DISCUSSION

The precipitation phenomena observed for the system $Th(NO_3)_4$–HNO_3 or NaOH are summarized in Figures 1 to 5. These figures show the characteristic changes during the precipitation of thorium(IV) hydroxide in dependence on pH. Figures 1 to 3 indicate the presence of two separated nephelometric maxima, while in Figures 4 and 5 these two nephelometric maxima become one. The pH values of the appearance of the first nephelometric maximum, shown in Figure 1 to 3, are in accord with the microelectrophoretic data. Microelectrophoretic determination of particle charge for the precipitation systems, shown in Figures 1 to 3, gave pH_{IEP} at 5.9 to 6. With increasing $Th(NO_3)_4$ concentration, the pH of the clear solution/sediment boundary was shifted to lower pH values. The observed pH values of the clear solution/sediment boundary in the precipitation systems investigated were in accord with literature data.[10,11] In Figures 1 – 5, the heights of the sediments formed in the test tubes after 1500 min of aging are also given.

The precipitation of thorium(IV) hydroxide in the presence of 6×10^{-1} M $NaNO_3$ was investigated for the following concentrations: 5×10^{-4}, 7.5×10^{-4}, 1×10^{-3}, 3×10^{-3}, 5×10^{-3}, 8×10^{-3} and 1×10^{-2} M $Th(NO_3)_4$. The characteristic precipitation phenomena observed are summarized in Figures 6 to 8. In these systems a »neutral electrolyte«, $NaNO_3$, serves as the coagulating agent. On the other hand, in the presence of gelatin, $NaNO_3$ is needed to uncoil the gelatin polymers. By increasing the
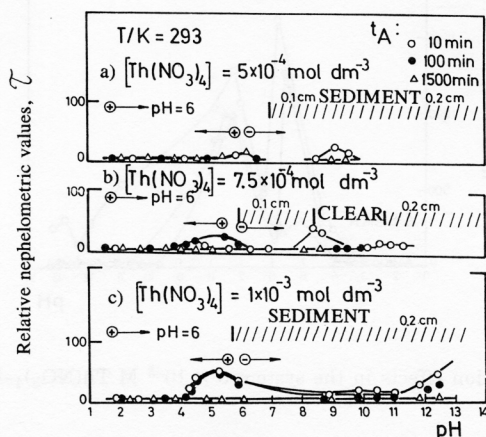

Figure 1. Precipitation effects in the system $Th(NO_3)_4$–HNO_3 or NaOH. a) 5×10^{-4} M $Th(NO_3)_4$–HNO_3 or NaOH, b) 7.5×10^{-4} M $Th(NO_3)_4$–HNO_3 or NaOH, c) 1×10^{-3} M $Th(NO_3)_4$–HNO_3 or NaOH; pH region of the sediment at 1500 minutes of aging.
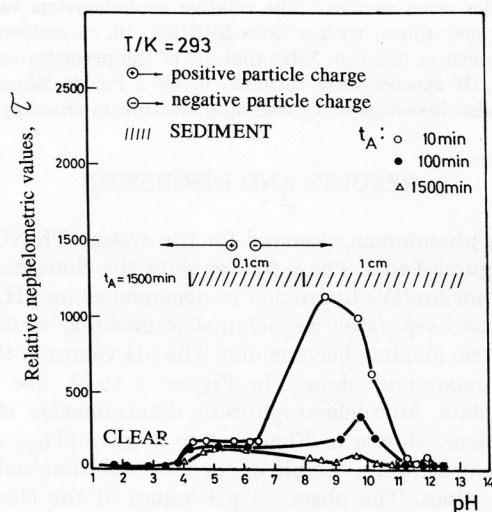
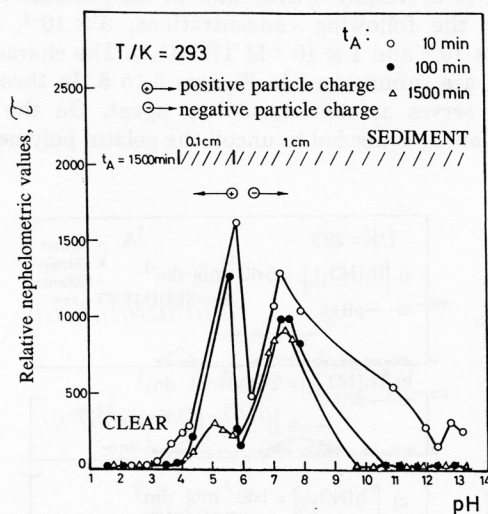Figure 2. Precipitation effects in the system 3×10^{-3} M $Th(NO_3)_4$–HNO_3 or NaOH.

Figure 3. Precipitation effects in the system 5×10^{-3} M $Th(NO_3)_4$–HNO_3 or NaOH.

$Th(NO_3)_4$ concentrations, the pH of the appearance of the sediment is shifted to lower pH values (Table I).

Addition of gelatin to the system $Th(NO_3)_4$–$NaNO_3$ + (HNO_3 or NaOH) caused significant changes in the phenomenology of precipitation processes. In the 5×10^{-4} M
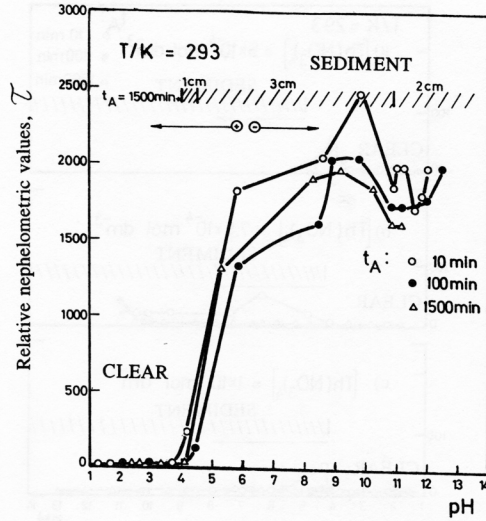
Figure 4. Precipitation effects in the system 8×10^{-3} M Th(NO$_3$)$_4$–HNO$_3$ or NaOH.
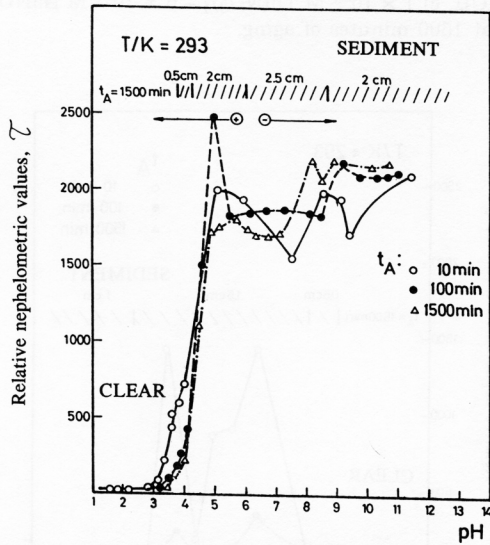
Figure 5. Precipitation effects in the system 1×10^{-2} M Th(NO$_3$)$_4$–HNO$_3$ or NaOH.

to 1×10^{-3} M Th(NO$_3$)$_4$ concentration range, with pH increasing, the precipitation regions denoted as clear I, gel, clear II, and sediment were observed. The clear II precipitation region separates the precipitation regions denoted as gel and sediment.
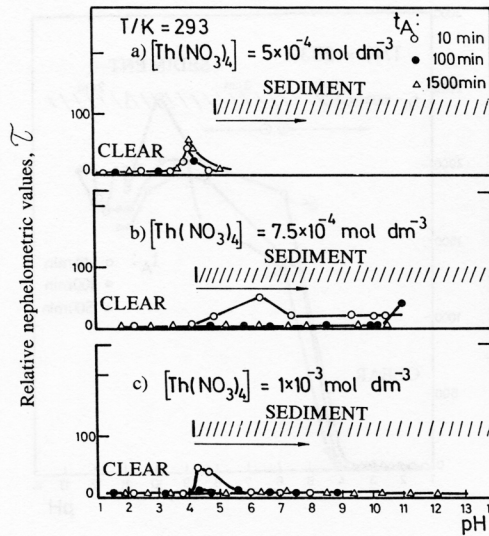
Figure 6. Precipitation effects in the system Th(NO$_3$)$_4$–NaNO$_3$–HNO$_3$ or NaOH. a) 5 × 10^{-4} M Th(NO$_3$)$_4$ – 6 × 10^{-1} M NaNO$_3$–HNO$_3$ or NaOH, b) 7.5 × 10^{-4} M Th(NO$_3$)$_4$ – 6 × 10^{-1} M NaNO$_3$–HNO$_3$ or NaOH, c) 1 × 10^{-3} M Th(NO$_3$)$_4$ – 6 × 10^{-1} M NaNO$_3$–HNO$_3$ or NaOH; pH region of the sediment at 1500 minutes of aging.
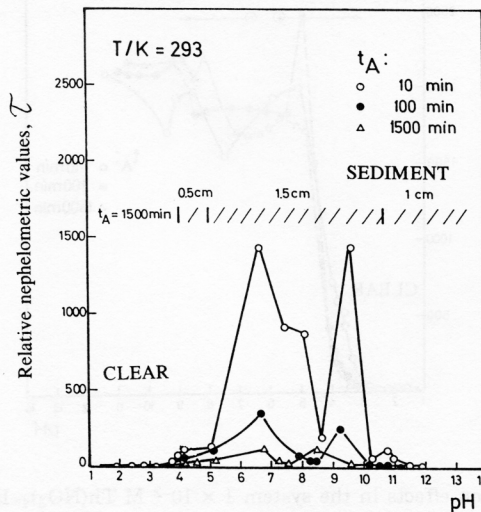
Figure 7. Precipitation effects in the system 3 × 10^{-3} M Th(NO$_3$)$_4$ – 6 × 10^{-1} M NaNO$_3$–HNO$_3$ or NaOH.
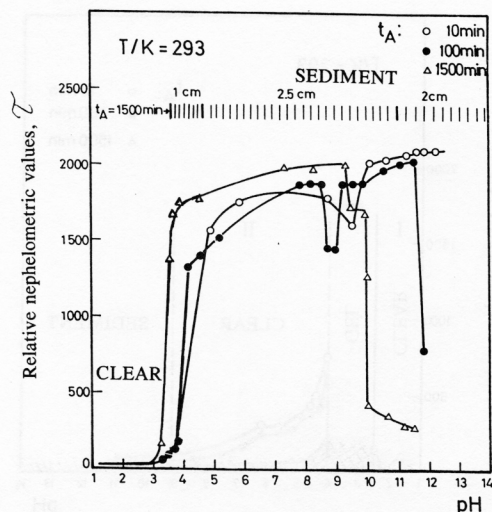
Figure 8. Precipitation effects in the system 8×10^{-3} M Th(NO$_3$)$_4$ – 6×10^{-1} M NaNO$_3$–HNO$_3$ or NaOH.

TABLE I

pH values of the appearance of the sediment during the precipitation of thorium(IV) hydroxide, for different Th(NO$_3$)$_4$ concentrations, t_{aging} = 1500 min.

Th(NO$_3$)$_4$ concentration / (M)	NaNO$_3$ concentration / (M)	pH
5×10^{-4}	6×10^{-1}	4.8
7.5×10^{-4}	6×10^{-1}	4.3
1×10^{-3}	6×10^{-1}	4.2
3×10^{-3}	6×10^{-1}	4.02
5×10^{-3}	6×10^{-1}	3.75
8×10^{-3}	6×10^{-1}	3.65
1×10^{-2}	6×10^{-1}	3.55

Figure 9 shows the phenomenology of the precipitation system 0.40% gelatin – 5×10^{-4} M Th(NO$_3$)$_4$ – 6×10^{-1} M NaNO$_3$ at different pH values. For the concentration 5×10^{-4} M Th(NO$_3$)$_4$, after 1500 min. of aging, the formation of gel was observed at pH 2.7 and that of the sediment at pH 9.2. In the 2.7–3.0 pH interval, only a small amount of gel was formed. However, in this pH interval, a very turbid colloid also existed. At pH 3.0–3.9, the solution above the gel was clear. The sediment appeared at pH 9.2 in the form of big flocks, which changed with time to gelatineous thorium(IV) hydroxide. For the concentration 7.5×10^{-4} M Th(NO$_3$)$_4$, after 1500 min. of aging, gel appeared at pH 2.5 and a gelatineous sediment of thorium(IV) hydroxide at pH 8.8. In the 2.3–2.5 pH interval, a turbid colloid was formed and this region was followed by gel formation at pH 2.5 to 4.8, while some slightly turbid colloid existed above the »gel-flocks« at pH 4.8 to 5.2. The region called clear I was observed at pH ≤ 2.3, and region clear II at pH 5.2 to 8.4. Figure 10 shows the phenomenology of the precipitation sys-
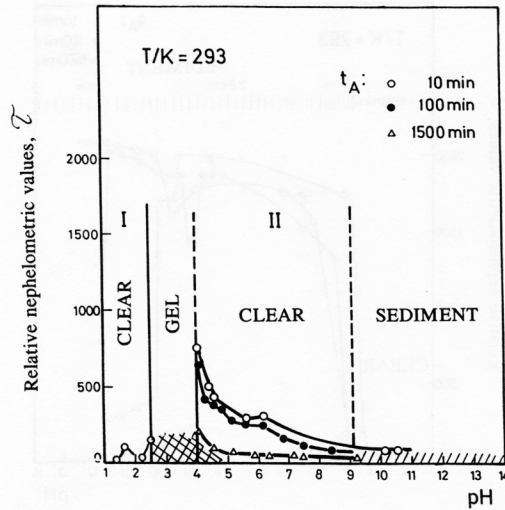
Figure 9. Precipitation effects in the system 0.40% gelatin – 5×10^{-4} M Th(NO$_3$)$_4$–6×10^{-1} M NaNO$_3$ – HNO$_3$ or NaOH.
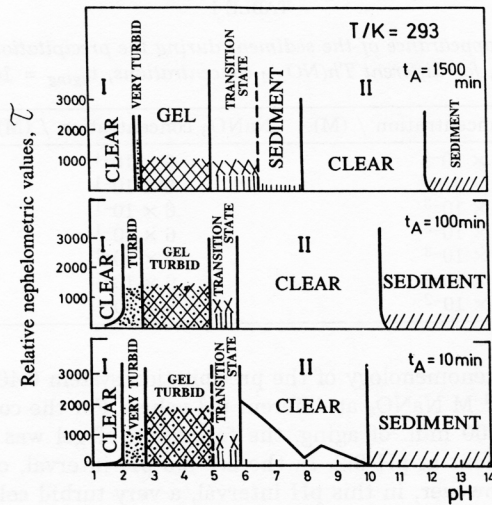
Figure 10. Precipitation effects in the system 0.40% gelatin – 1×10^{-3} M Th(NO$_3$)$_4$ – 6×10^{-1} M NaNO$_3$ – HNO$_3$ or NaOH.

tem 0.40% gelatin – 1×10^{-3} M Th(NO$_3$)$_4$ – 6×10^{-1} M NaNO$_3$ – (HNO$_3$ or NaOH). After 1500 min. of aging, the following colloid phenomena were observed: clear solu-

tion I at pH $\leq$ 2.7, very turbid colloid at pH 2.7–2.8, formation of gel at 2.8–4.9, »gel-flocks« (transition state) up to pH 6.5, a small amount of hydroxide sediment up to pH 8, clear solution II up to pH 12, and again, hydroxide sediment at pH > 12.

At higher concentrations, up to 1×10^{-2} M Th(NO$_3$)$_4$, region clear II disappeared. The chief difference between these precipitation systems and the ones previously described was: region clear II disappeared, the flock settled as sediment and some turbid colloid was observed instead. In the present work, precipitation systems for concentrations greater than 1×10^{-2} M Th(NO$_3$)$_4$ were not investigated.

Microelectrophoretic investigation of precipitation systems in the absence of NaNO$_3$ and containing a constant amount of gelatin, showed shifts of pH$_{IEP}$ with increasing Th(NO$_3$)$_4$ concentrations. Table II indicates that the pH$_{IEP}$ values changed from 4.86 to 5.9 when Th(NO$_3$)$_4$ concentration was increased from 5×10^{-4} to 1×10^{-2} M.

In order to explain the complex interactions between thorium(IV) hydroxide and gelatin, we shall pay attention to the chosen systems 0.40% gelatin – 1×10^{-3} M Th(NO$_3$)$_4$ – 6×10^{-1} M NaNO$_3$ – (HNO$_3$ or NaOH), to the precipitation system without gelatin, as well as to the properties of gelatin. On the basis of the experimental results obtained in the present work, we established characteristic precipitation regions, which are schematically shown in Figure 11a,b.

The system 0.40% gelatin – 1×10^{-3} M Th(NO$_3$)$_4$ – 6×10^{-1} M NaNO$_3$ – (HNO$_3$ or NaOH) shows a characteristic sequence of states with pH-increasing as shown in Figure 11a: clear solution (A), turbid colloid (B), gel (very turbid, very strong, elastic and thixotropic) (C), »gel-flocks« (D), clear solution (E) and sediment (settled flocks) (F). Turbid colloid (B) and »gel-flocks« (D) represent transitional states. The action of gelatin becomes evident when each of the colloidal states shown in Figure 11a is compared, at the same pH, with the colloidal states shown in Figure 11b. Instead of the states, clear solutions (G) and (H), shown in Figure 11b, when no gelatin was present, the formation of turbid colloid (B) and gel (C) was observed after incorporation of gelatin in the precipitation system, as shown in Figure 11a. Instead of flocks, settled as sediments (J) and (K) when no gelatin was present (Figure 11b), formation of gel (C), »gel-flocks« (D) and clear solution (E) was observed after the incorporation of gelatin into the corresponding precipitation system (Figure 11a). At high pH, the influence of the gelatin component failed and the flocks settled as sediments (L) and (F), as shown in Figures 11b and 11a, respectively.

TABLE II

pH$_{IEP}$ values measured for different concentrations of Th(NO$_3$)$_4$ in the system 0.40% gelatin—Th(NO$_3$)$_4$—HNO$_3$ or NaOH, t_{aging} = 1500 min.

Th(NO$_3$)$_4$ concentration / (M)	pH$_{IEP}$	pH$_{IEP}$ of gelatin	pH$_{IEP}$ of Th(OH)$_4$
		4.86 ± 0.16	5.9 to 6.0
5×10^{-4}	4.90		
7.5×10^{-4}	5.00		
1×10^{-3}	5.08		
3×10^{-3}	5.45		
5×10^{-3}	5.72		
8×10^{-3}	5.88		
1×10^{-2}	5.90		

In order to explain the observed action of gelatin on the state of thorium(IV) hydroxide, it was necessary to establish the characteristics of all components and to clarify the interactions between these components. Either HNO_3 or the NaOH component was used to adjust the pH of the system. The »neutral electrolyte«, $NaNO_3$, has the role to uncoil[12,13] gelatin molecules, thus enhancing their interacting power,[14] and has also the role of a coagulating agent. Vainerman *et al.*[15] investigated the effect of self-association of gelatins, with different isoionic points, on their electrostatic complexation. Electrostatic complexation and self-association processes competed and were dependent on pH, temperature, concentration, the sequence of mixing, the presence of urea and some ions. The swelling properties of gelatin in aqueous media, as a function of pH and ionic strength, were also investigated.[16]

The pH values govern three processes: (a) hydrolysis of Th^{4+}, (b) dissociation of the functional groups of gelatin, and (c) interaction between the $Th(NO_3)_4$ component and gelatin. Regarding the $Th(NO_3)_4$ component, an increase in pH brings about, *via* the hydrolysis of Th^{4+} ions, formation of soluble hydroxy complexes and formation of colloidal $Th(OH)_4$ particles at the corresponding pH values (vertical arrow and asterisk in Figure 11c). At pHs lower than (M) in Figure 11b, the colloidal particles are positively charged, at higher pHs they are negatively charged. Simultaneously, owing to the same pH-increase, the functional groups of gelatin dissociate[8,17,18] and, therefore, the concentration of negatively charged $-COO^-$ groups increases while the concentration of positively charged $-NH_3^+$ groups decreases (Figure 11c). The consequence of the mentioned two processes, which both depended on pH, was the interaction of the Th^{4+} ion, its hydroxy complexes or colloidal hydroxide, with gelatin. The state of the colloidal product of these interactions, as well as the final equilibrium, depended, therefore, on pH, but also on the ratio of total gelatin concentration and $Th(NO_3)_4$. The sequence of the colloidal states, shown in Figure 11a, can be explained as follows.

At low pH, gelatin carries $-COOH$ and $-NH_3^+$ groups, Th^{4+} is not hydrolyzed, no interaction occurs and a clear solution is found (state (A) in Figure 11a).

During the following step of pH-increase, the $-COOH$ groups of gelatin dissociate and the concentration of negatively charged $-COO^-$ groups is enlarged (interval (N) in Figure 11c). Simultaneously, the Th^{4+} ion hydrolyzes, forming soluble complexes and colloidal hydroxide. The negatively charged $-COO^-$ groups also adhere to the positively charged colloidal thorium(IV) hydroxide particles, since pH is lower than that in (M) in Figure 11b. These colloidal particles are linked by chains of uncoiled gelatin molecules into a network of gel structure (state (C) in Figure 11a).

During the subsequent pH-increase, the concentration of the $-COO^-$ groups in the gelatin increased, but the OH^- ion concentration remained relatively low. Therefore, the process of formation of the soluble gelatin complex with the Th^{4+} ion and/or its soluble hydroxy complexes predominates in the competition with the formation of the colloidal thorium(IV) hydroxide. In this process, the gelatin component hindered the formation of thorium(IV) hydroxide and, as a consequence, a clear solution was formed (state (E) in Figure 11a).

At high pH (state (F) in Figure 11a), the colloidal particles of thorium(IV) hydroxide are negatively charged (because pH is higher than that in (M) in Figure 11b) and gelatin molecules carry only a very low number of oppositely, *i.e.* positively charged groups (Figure 11c). Gelatin molecules cannot adhere to colloidal particles, and no effect of the gelatin component is observed. The colloidal state (F), shown in Figure 11a,
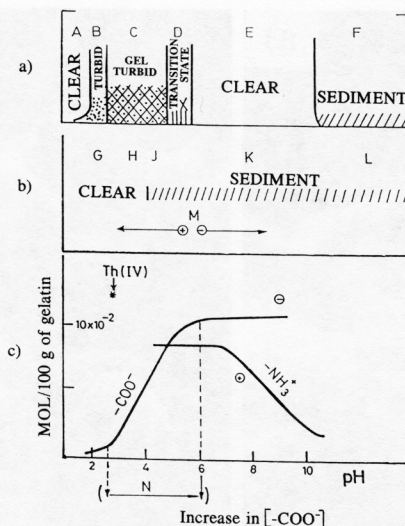
Figure 11. a) Precipitation effects in the system 0.40% gelatin – 1×10^{-3} M Th(NO$_3$)$_4$ – 6×10^{-1} M NaNO$_3$ – HNO$_3$ or NaOH, t_A = 100 min. In dependence on pH, following states appear: clear solution (A), turbid colloid (B), gel (very turbid, strong elastic and thixotropic) (C), »gel-flocks« (transition state) (D), clear solution (E) and sediment (settled flocks)(F). b) Precipitation effects in the system 1×10^{-3} M Th(NO$_3$)$_4$ – 6×10^{-1} M NaNO$_3$ – HNO$_3$ or NaOH, t_A = 100 min. In dependence on pH, the following states appear: clear solution (G) and (H), and sediments (settled flocks) (J), (K) and (L). Label (M) is pH$_{IEP}$, determined for Th(OH)$_4$. c) Concentration/(Mol/100 g of gelatin) *versus* pH. Label (N) represents the pH-interval of the increasing concentration of –COO$^-$ groups of gelatin.

is, therefore, like state (L) of the system without gelatin component, as shown in Figure 11b.

The suggestion that the gel consisted of colloidal thorium(IV) hydroxide particles, linked by the gelatin molecules into the network of gel structure, can be supported by other results. The presence of colloidal particles in the gel was indicated by its high turbidity. The gel was elastic and thixotropic, which is also a sign of the network structure. Radioanalytical analysis of the gel showed that thorium was the constitutive element of the gel structure and that the content of thorium in the gel increased with an increase in pH. X-ray diffraction of the gel state showed its amorphous nature. The IR spectrum of 13 times washed gel, which was prepared at lower pH values, still contained the characteristic peak of the nitrate group at 1385 cm^{-1} On the basis of these results, it can be suggested that some part of hydroxyls in thorium(IV) hydroxide is substituted by NO$_3^-$ groups. On the other hand, the IR spectrum of the settled flocks at high pH values did not give any evidence of the substitution of some part of hydroxyls in thorium(IV) hydroxide with NO$_3^-$ groups. Šmidt *et al.*[19] investigated also the properties of precipitates formed in the precipitation system Th(NO$_3$)$_4$–HNO$_3$ or NaOH. They suggested that in the pH 3.8–4.1 range, there is formation of basic Th(IV) nitrates, such as Th(OH)$_{1.5}$O$_{0.75}$NO$_3$ and Th(OH)$_2$O$_{0.75}$(NO$_3$)$_{0.5}$.
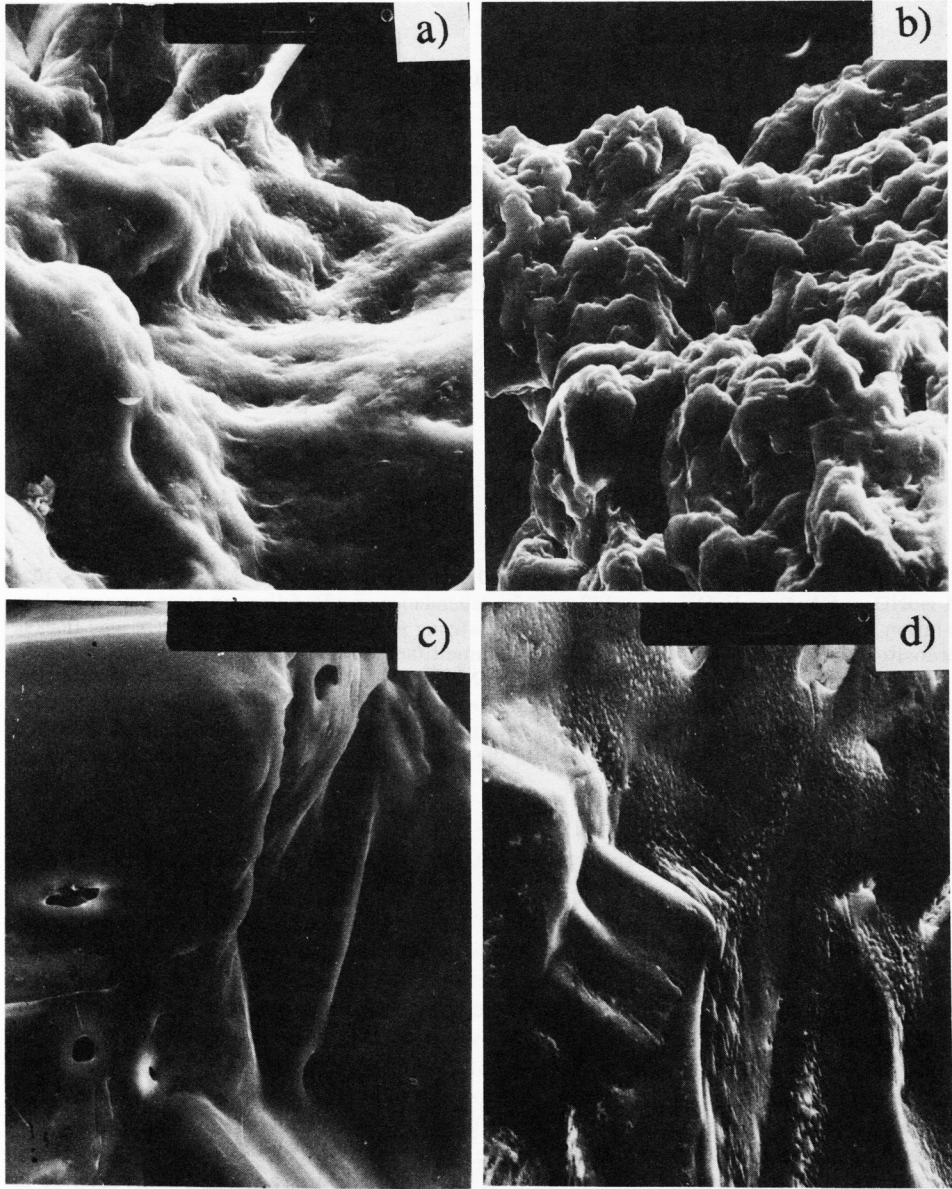
Figure 12. Scanning electron micrographs of the gel state for the following precipitation systems: a) 0.40% gelatin – 1×10^{-2} M $Th(NO_3)_4$ – 6×10^{-1} M $NaNO_3$ at pH 3.40, t_{aging} = 1 day, magnification 880 × ; b) 0.40% gelatin – 3×10^{-3} M $Th(NO_3)_4$ – 6×10^{-1} M $NaNO_3$ at pH 3.68, t_{aging} = 2 months, magnification 880 × ; c) 0.40% gelatin – 1×10^{-3} M $Th(NO_3)_4$ – 6×10^{-1} M $NaNO_3$ at pH 3.33, t_{aging} = 8 months, magnification 528 × ; d) 0.40% gelatin – 5×10^{-3} M $Th(NO_3)_4$ – 6×10^{-1} M $NaNO_3$ at pH 4.55, t_{aging} = 1 year, magnification 880 × .

Figure 12 shows some impressively plastic forms of the gel state. These scanning electron micrographs were taken for different times of aging of the gel state, up to a one-year period. The absence of visible Th(OH)$_4$ particles, up to 8 months of aging, in these micrographs, confirmed the previous conclusions that Th(OH)$_4$ particles are present in the form of very fine particles embedded in the network of gelatin molecules. Also, these micrographs confirmed the high stability of the gel state for long periods of its aging.

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SAŽETAK

Taloženje torijevog hidroksida u prisutnosti želatine i natrijevog nitrata

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Taloženje torij(IV)-hidroksida i interakcije taloga sa želatinom istraživani su u sustavu: želatina–$Th(NO_3)_4$–$NaNO_3$–(HNO_3 ili NaOH). Uzorci su pripravljeni miješanjem vodenih otopina komponenti zadanog sustava, a zatim su ostavljeni da stare pri 293 K. Komponente HNO_3 i NaOH služile su za ugađanje pH, a $NaNO_3$ za odmatanje molekule želatine, čime se povećava njihova interakcijska moć. Taložni sustavi istraživani su primjenom nefelometrije, mikroelektroforeze, difrakcije X–zraka, IR spektroskopije i skanirajuće elektronske mikroskopije. Karakterističan niz pojava opažen je u ovisnosti o pH: bistra otopina I, gel (vrlo mutan, jak, elastičan i tiksotropan), bistra otopina II i sedimentirane flokule. Navedeni niz pojava razmatran je s obzirom na tri moguća procesa koji se odigravaju s povećanjem pH, a to su: (a) hidroliza iona Th^{4+} te nastajanje Th(IV)-hidroksi kompleksa i koloidnog Th(IV)-hidroksida, (b) disocijacija funkcijskih skupina želatine, i (c) interakcija između $Th(NO_3)_4$ i želatine. U radu je pokazano da koloidno-kemijsko stanje gel predstavljaju koloidne čestice Th(IV)-hidroksida koje su vezane preko odmotanih molekula želatine u čvrstu mrežu strukture gela.