

# ANALYSIS OF PRODUCTS FORMED IN HYDROTHERMAL PROCESSING OF YTTRIUM NITRATE AND YTTRIUM CHLORIDE

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The investigation of the effect of initial salt counter-ion on the result of hydrothermal processing of yttrium salts is presented. Powder X-ray diffraction analysis and scanning electron microscopy have been used to characterize the properties of the products. It has been established that 3–6  $\mu\text{m}$  crystals of  $\text{Y}_2(\text{OH})_{5.14}(\text{NO}_3)_{0.86} \cdot \text{H}_2\text{O}$  structure are formed from yttrium nitrate and 200–500 nm crystals of  $\text{Y}_2(\text{OH})_{4.86}\text{Cl}_{1.14} \cdot 1.07\text{H}_2\text{O}$  structure are formed from yttrium chloride.

*Keywords: hydrolysis of yttrium salts, hydrothermal synthesis, layered materials, X-ray diffraction analysis, scanning electron microscopy.*

## Introduction

The widespread use of yttrium compounds in the preparation of ceramics and catalysts for organic synthesis promotes the study of these compounds, as well as the search for new methods of synthesis. Yttrium hydroxide is often used as the basis for obtaining crystalline yttrium oxide, with the specified structure [1]. Yttrium hydroxide is practically impossible to form in its pure form from yttrium salts by hydrolysis due to the tendency for forming a layered structure [2]. Yttrium hydroxide nitrate or yttrium hydroxide chloride and other compounds are formed depending on which precursor has been used. In [3] yttrium hydroxide nitrate and yttrium oxyhydroxide nitrate have been synthesized by ultrasonic hydrothermal and hydrothermal methods. The resulting precipitate may include other ions such as carbonate ions in its structure [4].

Structures with various morphologies can be obtained by varying the conditions of hydrothermal synthesis [1]. In particular, nanotubes of various sizes [5], spheres, rods of various sizes, connected with each other at multifarious angles, as well as micro-ribbons which can fold to form tubes [6]. In [7] nanotubes with undetermined composition were obtained using yttrium chloride as a precursor at pH 9.5 and a temperature of 200 °C.

In [1] it has been found by X-ray diffraction analysis (XRD) that, depending on the hydrothermal synthesis conditions, several main types of compounds can be formed:  $\text{Y}_2(\text{OH})_{5.14}(\text{NO}_3)_{0.86} \cdot \text{H}_2\text{O}$ , hexagonal  $\text{Y}(\text{OH})_3$  and monoclinic  $\text{Y}_4\text{O}(\text{OH})_9(\text{NO}_3)$ , in addition, a small amount of monoclinic  $\text{Y}(\text{OH})_3$  in the form of flakes, together with  $\text{Y}_4\text{O}(\text{OH})_9(\text{NO}_3)$  or hexagonal  $\text{YO}(\text{OH})$  can be produced at relatively high temperatures and high pH values.

Review of the literature shows that the systematic analysis of regularities for hydrothermal treatment of yttrium salts has not been found. Earlier we investigated the hydrolysis of yttrium nitrate by the precipitation method [8]. In the present study the hydrothermal treatment of yttrium nitrate and chloride has been investigated for the same concentrations of salts and the hydrolytic agent, X-ray analysis and the study of the morphology by scanning electron microscopy have been carried out.

## Experimental

For the synthesis of samples yttrium nitrate and yttrium chloride were used at 0.1 mol/L concentration, the hydrolytic agent was the aqueous solution of sodium hydroxide with 0.1 mol/L concentration. The synthesis was carried out at various pH values. The pH values of the sample synthesis (5, 7, 8) were located below pH of the yttrium hydroxide point of zero charge (9.2–9.3). The synthesis was carried out for 24 hours in a thermostat at 180 °C in an autoclave with self-regulating pressure, with the capacity of 40 mL, 50 % full. The samples were washed five times with distilled water (until counter-ions disappeared in the washings), then they were dried at 50 °C under vacuum to constant mass.

## Краткие сообщения

X-ray diffraction patterns were recorded on the diffractometer Rigaku Ultima IV. The SEM-image was obtained with the use of the scanning electron microscope Jeol JSM-7001F.

### Discussion

Fig. 1 shows typical diffraction patterns of the samples synthesized from solutions of chlorides and nitrates. The analysis of diffraction patterns shows that the "chloride" sample is substantially a crystalline phase with  $Y_2(OH)_{4.86}Cl_{1.14} \cdot 1.07H_2O$  composition, while "nitrate" sample is  $Y_2(OH)_{5.14}(NO_3)_{0.86} \cdot H_2O$ . The most intense reflections in the range 10 degrees  $2\theta$  speak about large interplanar distance, typical for layered materials [2]. It is possible to suppose that the "chloride" and "nitrate" samples have the planar structure.

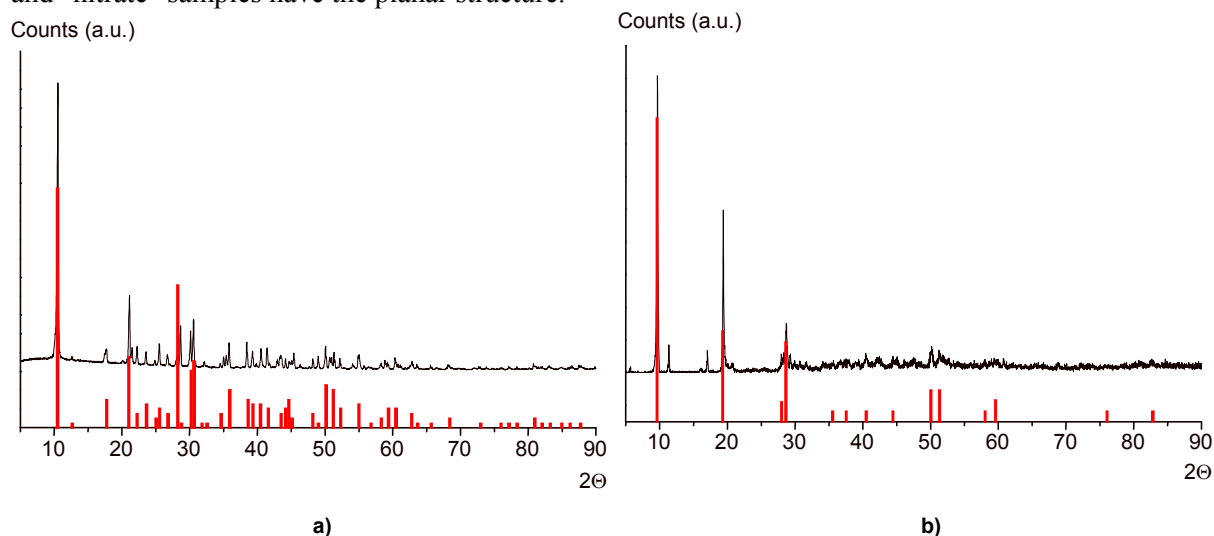


Fig. 1. XRD analysis of samples obtained from (a) yttrium chloride and (b) yttrium nitrate

Electron microscopy also shows significant differences in the morphology of the samples obtained from different precursors. Fig. 2 shows the SEM-images of "chloride" and "nitrate" precipitation. It can be seen that the crystals of the "chloride" sample are much smaller than of the "nitrate" one; and, moreover, they contain an admixture of objects whose morphology is different. The size of yttrium hydroxide nitrate crystal is 3–6 micrometers, and yttrium hydroxide chloride crystals are 200–500 nm in size.

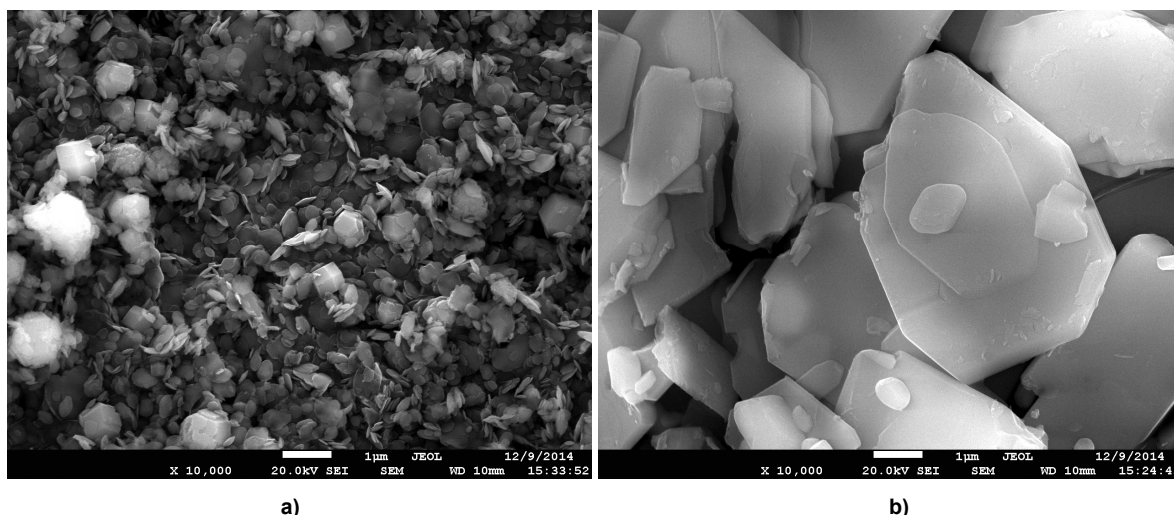


Fig. 2. SEM-images of samples obtained from (a) yttrium chloride and (b) yttrium nitrate

### Conclusion

It has been found that the nature of the counter-ion of the original yttrium salt has a significant impact on the structure, morphology and general composition of the hydrothermal hydrolysis products. The formed objects are probably layered materials, which include counter-ions in the matrix structure, they

are not removed by washing with water. Hydrothermal synthesis is a promising method for obtaining nanoscale yttrium oxides.

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## АНАЛИЗ ПРОДУКТОВ ГИДРОТЕРМАЛЬНОЙ ОБРАБОТКИ НИТРАТА И ХЛОРИДА ИТТРИЯ

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В работе представлено исследование влияния природы противоиона исходной соли на результат гидротермальной обработки солей иттрия. Для изучения свойств полученных продуктов использовались сканирующая электронная микроскопия и порошковый рентгеноструктурный анализ. Установлено, что из нитрата иттрия образуются кристаллы размером 3–6 мкм с составом  $Y_2(OH)_{5,14}(NO_3)_{0,86} \cdot H_2O$ , а из хлорида иттрия – кристаллы размером 200–500 нм с составом  $Y_2(OH)_{4,86}Cl_{1,14} \cdot 1,07H_2O$ .

## Краткие сообщения

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*Ключевые слова: гидролиз солей иттрия, гидротермальный синтез, слоистые материалы, рентгеноструктурный анализ, сканирующая электронная микроскопия.*

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