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Preparation of metal oxides for effective catalysts

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INTRODUCTION & AIM

Desired properties in catalysts include a nanosize and homogeneity of the particles that form the catalyst and/or its carrier. The creation of catalysts with the finest particles has been a hot topic of scientific research in recent decades. The particle sizes of catalytic oxides are set at the initial stage of forming; in wet-chemistry, this is a precursor to precipitation. It is possible to create optimal conditions by using homogeneous precipitation when the precipitant is formed in the solution itself due to a hydrolysis reaction. To solve this problem, urea was used in our work, and the hydrolysis products were ammonia and carbon dioxide (1).

As a result of precipitation, hydroxides, carbonates, or hydroxy carbonates of metals can be obtained.

METHOD

All precipitates were obtained from solutions of metal nitrates. The obtained hydroxides aluminum, indium, and iron, and the hydroxy carbonates nickel, cobalt, and zinc, were studied (2-7).

The following was found: metal hydroxides were obtained from aluminum, indium, and iron (III) nitrate solutions. Metal hydroxycarbonates were obtained from nickel, cobalt, and zinc nitrate solutions.

The oxides obtained from these materials by calcination at the temperature 450 °C during 2 hours. They form the structure of the catalyst. Their most important characteristics are crystallites sizes, specific surface and porosity. The all oxides and catalyst were characterized by XRD. To investigate the catalyst's structure XRD meter Bruker D&D discover (model D8) using the Cu-Kα radiation. The crystallites sizes were calculated by Willamson-Hall method. Specific surface areas of oxides and their porosities were obtained from the analysis of the nitrogen adsorption-desorption isotherms by using the BET and BJH procedure. The SEM images of the structures were obtained with Zeiss Evo25 with the electron-accelerating voltages in the gun in the range from 5 to 10 kV.

$$CO(NH_2)_2+H_2O=2NH_3+CO_2 \quad (1)$$

$$5Zn(NO_3)_2+5CO(NH_2)_2+13H_2O= \\ =Zn_5(OH)_6(CO_3)_2+10NH_4NO_3+3CO_2 \quad (2)$$

$$4Ni(NO_3)_2+4CO(NH_2)_2+10H_2O= \\ =2Ni_2(OH)_2CO_3+8NH_4NO_3+2CO_2 \quad (3)$$

$$2Co(NO_3)_2+4CO(NH_2)_2+7H_2O= \\ =2Co(OH)(CO_3)_{0,5}+4NH_4NO_3+3CO_2 \quad (4)$$

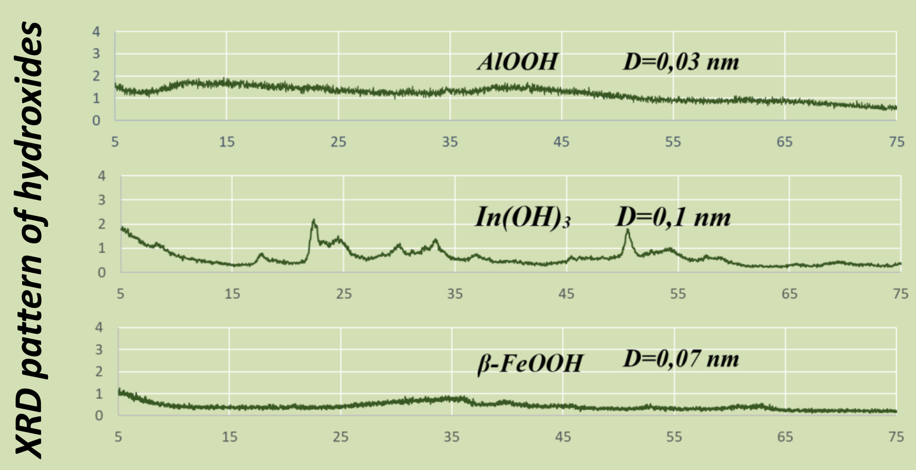
$$2Al(NO_3)_3+3CO(NH_2)_2+9H_2O= \\ =2Al(OH)_3+6NH_4NO_3+3CO_2 \quad (5)$$

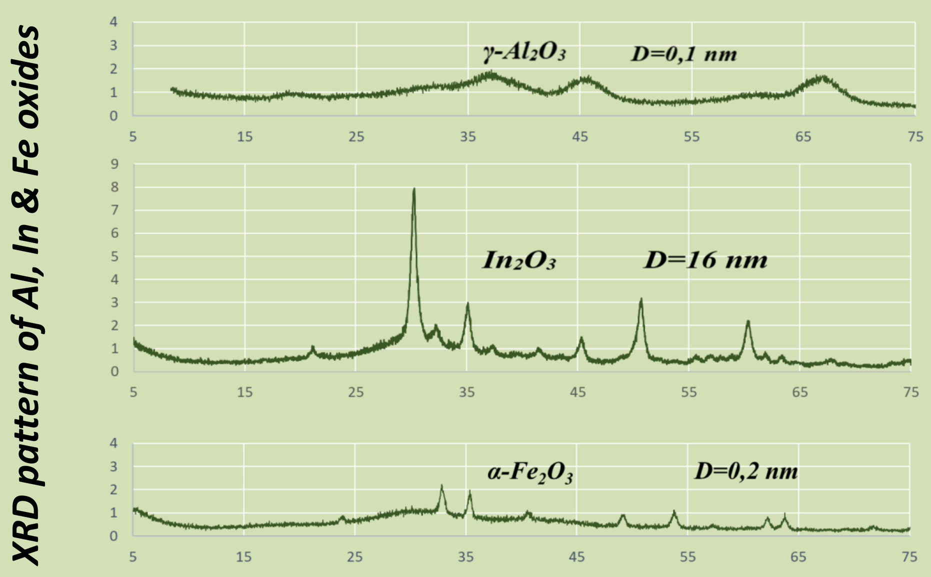
$$2In(NO_3)_3+3CO(NH_2)_2+9H_2O= \\ =2In(OH)_3+6NH_4NO_3+3CO_2 \quad (6)$$

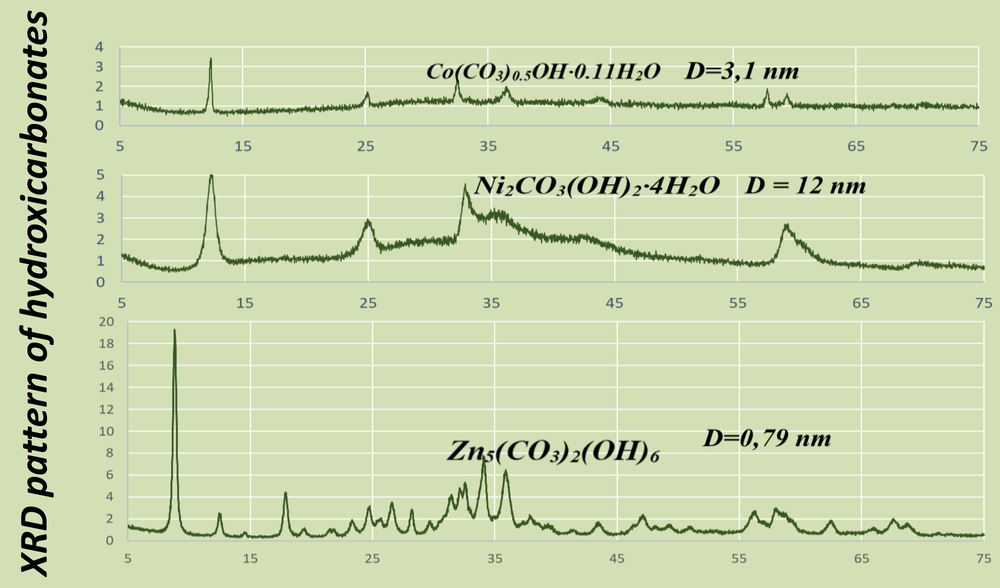
$$2Fe(NO_3)_3+3CO(NH_2)_2+7H_2O= \\ =2FeOOH+6NH_4NO_3+3CO_2 \quad (7)$$

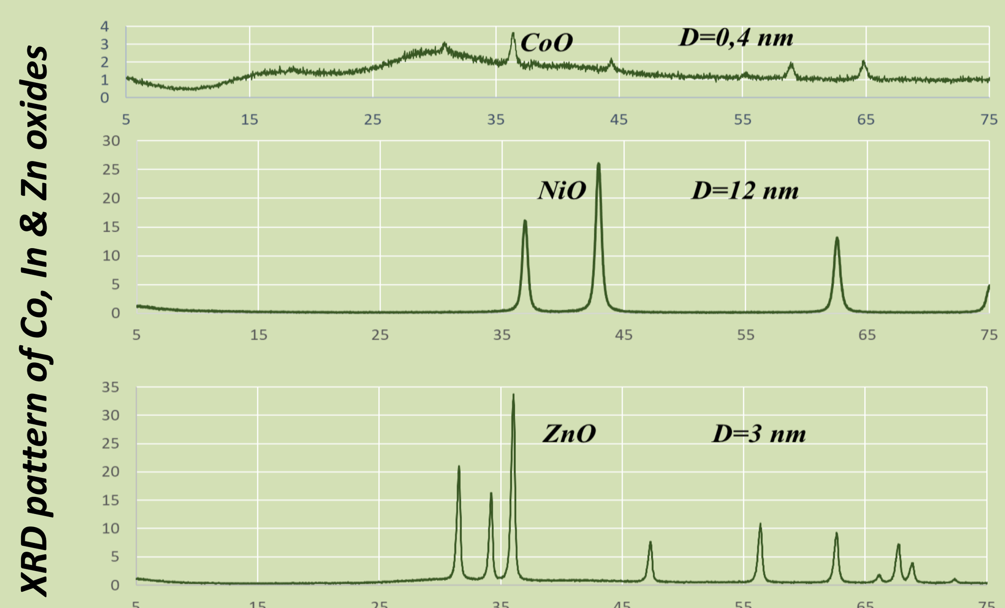
RESULTS & DISCUSSION

According to the XRD patterns, it was established that the crystallite sizes of the obtained hydroxides were, respectively, 0.1, 0.03, and 0.07 nm. The oxides obtained by the calcination of these hydroxides have similar sizes, from 0.1 to 16 nm. The crystallite sizes of these compounds are quite large and exceed 10 nm. However, their thermal decomposition allows us to obtain oxides with crystallite sizes less than 15 nm.The specific surface area and porosity of several of the obtained samples were also measured. It was found that the obtained oxides have a specific surface area that is significantly higher than similar samples obtained by other methods. Most of the porous volume and surface area is located in the mesopores.

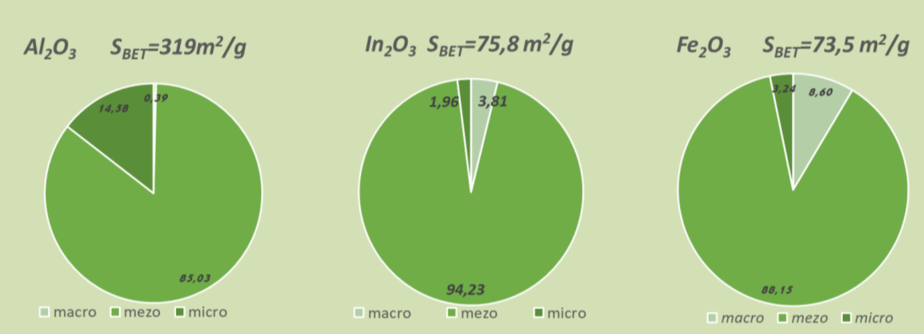




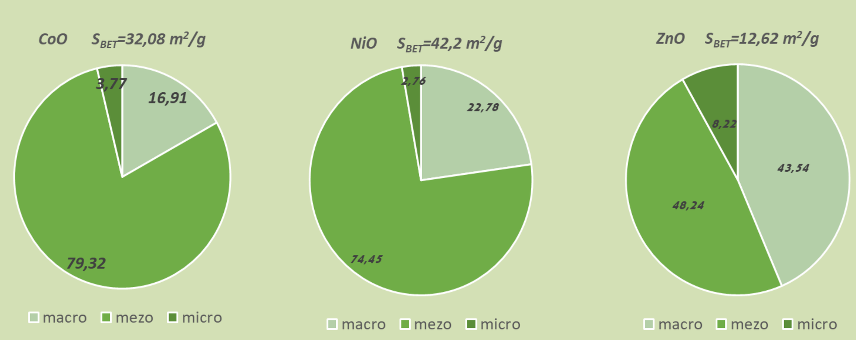




Generally, oxides formed by the calcination of hydroxycarbonates have a larger volume of macropores.
This is due to both the presence of carbon dioxide in their composition and the number of hydroxy groups. These components are removed during calcination, forming a porous structure of oxides.

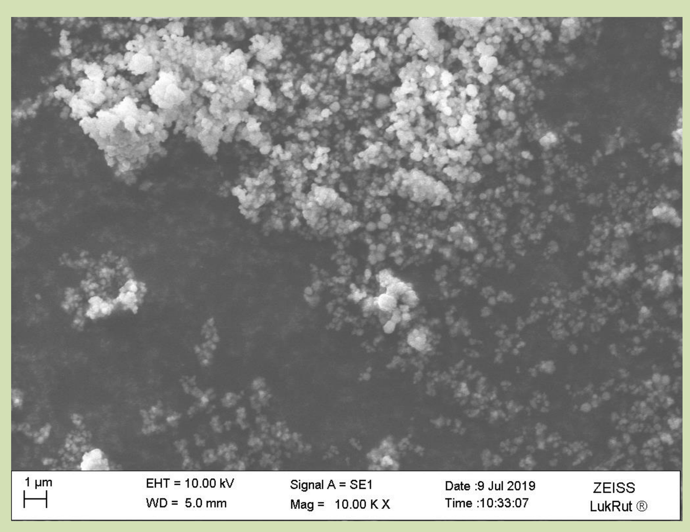


The pore distribution of Al, In & Fe oxides

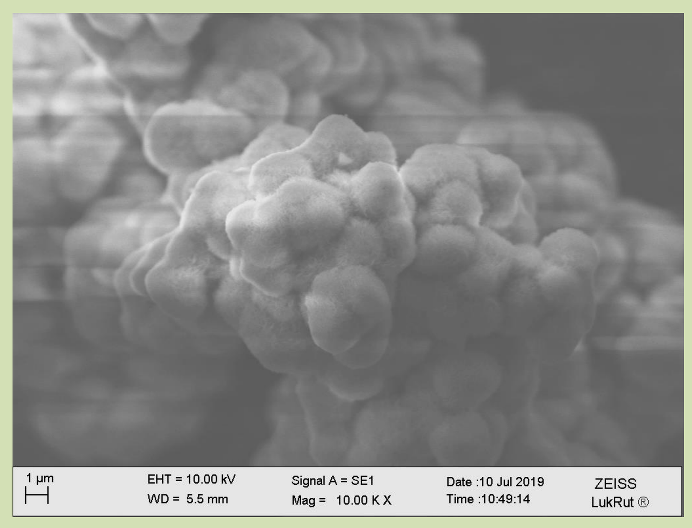


The pore distribution of Co, Ni & Zn oxides

a



b



SEM images of FeOOH (a) and Nickel hydroxycarbonate (b)

The SEM precipitate investigations show the particles formed by carbamide precipitation from iron (III) nitrate solution. The particles are rods combined in peanut-like superstructures1.
The SEM image of the Nickel Hydroxycarbonate shows the uniform spherical particles that are combined in agglomerates.

CONCLUSION

Thus, nanosized oxides were obtained that can be included in the composition of catalysts. All the obtained oxides have nanosized crystallites and are capable of catalyzing selected reactions. Another important characteristic is the specific surface area and porosity. It is believed that mesoporous nanosized catalysts are the optimal combination for the manifestation of catalytic activity. The above properties of nanosized oxides were formed due to the fact that their precursors - hydroxides and hydroxycarbonates – consist of monodispersional particles. The particles are formed as a result of homogeneous precipitation, the peculiarity of which is the formation of precipitators as a result of the hydrolysis reaction and the simultaneous creation of a large number of crystallization centers of precursor precipitates throughout the solution volume.

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