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Synthesis and Investigation of Nanocrystalline Powder of Neodymium Oxide

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The starting chemicals for the synthesis of neodymium oxide were neodymium metal and commercially available reagent grade nitric acid. Highly dispersed powders of neodymium sesquioxide Nd₂O₃ were synthesized by template method. According to this method hydrated cellulose fibers (medical cotton) were impregnated with a neodymium nitrate water solution of the concentration 5 times as less as it corresponds to the maximal stoichiometric value obtained when neodymium metal is dissolved in 63% nitric acid. These wet fibers were air-dried at 80 °C for 3 hours and subsequently air-calcined for 9 hours at 550 °C. The choice of the calcinations temperature value was due to a requirement that this value should be as low as possible, since at low synthesis temperatures the formation of nanostructured particles is highly favorable and because of that the complete thermal decomposition of cellulose fibers was shown in our previous works within the project implementation to proceed at temperatures well above 500 °C. Alternatively, the calcination temperature was raised up to 1000 °C.

The composition and structure of the prepared oxides were studied by advanced methods.

The obtained powders had light-blue appearance, while XRF spectrum of the neodymia prepared by the thermal decomposition of neodymium nitrate (Fig. 73) at 550 °C shows no presence of metals other than neodymium. The discussion of other results should take into account that like other rare earth sesquioxides neodymia readily absorbs water vapours and carbon dioxide from the surrounding air (Adachi and Imanaka 1998). With this respect thermo gravimetric analysis, BET, IR and XRD spectroscopy and elemental analysis for "light" elements, i.e. carbon, hydrogen and oxygen (Table) are in a good agreement with each other. The thermal analysis (thermo gravimetry) evidences that on heating all compounds both presented in the bulk and on the surface (hydroxides and carbonates) will be decomposed.

Table: Results of the elemental combustion (for C and H) and pyrolysis (for O) analyses of neodymia obtained by the calcination under air at 550 °C of hydrated cellulose fibers impregnated with aqueous solutions of neodymium nitrate taken at low concentration

sample	C, mass.%	H, mass.%	O, mass.%	N, mass.%
550 °C, low concentration	1.10±0.10	0.53±0.03	5.51±0.24	0.34±0.10

The microstructure of neodymia synthesized at 550 °C as derived by high resolution electron microscopy and electron micro diffraction is very fine with the crystallite size of about 20-30 nm. An increase in the annealing temperature up to 1000 °C leads to an increase in the degree of crystallinity of neodymia, but the crystallite sizes do not markedly change, as evidenced by electron microscopy data. XRD analysis of neodymia samples obtained by the thermal decomposition at 550 °C and 1000 °C of neodymium nitrate taken at low concentration evidences (Fig.) the presence of one crystal phase of Nd₂O₂, with the structural type of A-form of rare-earth sesquioxide, its unit cell is characterized: $a = 0.383\pm0, 002$ nm; $c = 0.600\pm0, 003$ nm

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