

SPINELS RENAISSANCE—PAST, PRESENT, AND FUTURE

**Impact of preparation method and chemical composition on physicochemical and photocatalytic properties of nano-dimensional magnetite-type materials†**

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**ABSTRACT**

Nickel ferrites with different Ni content,  $\text{Ni}_x\text{Fe}_{3-x}\text{O}_4$ ,  $0 \leq x \leq 1$ , are technologically important materials for microwave, electronic, and magnetic storage devices. They are members of solid-solution series of spinel-type materials ( $\text{Fe}_3\text{O}_4\text{--NiFe}_2\text{O}_4$ ) having specific magnetic properties and different degree of electron delocalization. They demonstrate good gas sensing properties and catalytic activity in various catalytic processes, such as complete oxidation of waste gases, oxidative dehydrogenation of hydrocarbons, decomposition of alcohols, etc. The preparation of such materials is still an actual problem due to several difficulties in their synthesis and the use of special techniques.

A series of nickel-containing ferrite materials  $\text{Ni}_x\text{Fe}_{3-x}\text{O}_4$  ( $x = 0.25, 0.5, 1$ ) were prepared by precipitation method using  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ,  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ , and  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  as precursors. The performed analyses show the dependence of the rate of formation of the spinel phase on the chemical composition. To obtain the exact conditions for a single-phase spinel material preparation several investigations have been performed: thermal analysis (thermogravimetry, differential thermogravimetry, and differential thermal analysis) and various studies of the intermediates by powder X-ray diffraction, Mössbauer spectroscopy (at room and liquid nitrogen temperature), BET method, and SEM. As a result, the appropriate conditions of obtaining monophase nanomaterials of doped magnetite are found. The synthesis involves a precipitation process combined with a low-temperature heat treatment of materials (at 300 °C and in argon atmosphere) or mechanochemical processing. Application of the second procedure leads to two interesting results: (1) synthesis of the target compounds under soft and clean conditions without heating, which is important for industrial technology and environmental protection and (2) the prepared samples have better characteristics (higher dispersion degree, better magnetic properties, and higher activity in photocatalytic purification of wastewater from the textile industry).

**Keywords:** Spinel, doped nano-sized magnetite, synthesis, mechanochemistry, relaxation phenomena, magnetic properties, photocatalytic activity