

#### Ceramics International

Volume 47, Issue 20, 15 October 2021, Pages 28623-28633

# Magnetically retrievable nanoscale nickel ferrites: An active photocatalyst for toxic dye removal applications

Swapnil A. Jadhav A. Mangesh V. Khedkar A. Sandeep B. Somvanshi A. M. Jadhav A. Show more 

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https://doi.org/10.1016/j.ceramint.2021.07.021 

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#### **Abstract**

Nickel ferrite (NiFe<sub>2</sub>O<sub>4</sub>) <u>nanoparticles</u> were synthesized through the sol-gel auto-combustion method using urea and glycine as mixed fuel. The prepared <u>nanoparticles</u> were investigated for their structural, optical, and magnetic characterizations. Rietveld refined X-ray diffraction (XRD) patterns revealed the development of single-phase cubic spinel. The <u>crystallite size</u> was calculated by using Modified Scherrer's method and the W-H plot was found in the order of 26.6nm and 25.4 respectively which are nearly the same. The infrared spectrum showed the typical characteristic absorption bands in the range of 400cm<sup>-1</sup> to 600cm<sup>-1</sup> belonging to cubic spinel structure. Scanning electron microscopy images showed the spherical nature of the nanoparticles along with agglomeration to some extent. As per the optical study, the prepared nanoparticles have an optical bandgap of 2.59eV. The <u>magnetic properties</u> were studied through the M-H hysteresis curve showing superparamagnetic nature, the value of saturation magnetization (M<sub>s</sub>), coercivity (H<sub>c</sub>) was observed 46.20 emu/gm, and 383.20e respectively. The photocatalytic activity of nickel ferrite was studied based on the degradation of methylene blue (MB) dye as a model compound, where the result showed that prepared nanoparticles possessed a good photocatalytic activity against dye degradation. Up to four times catalyst exhibits nearly the same reutilization.

#### Introduction

The increasing human population contributes to more air, water, and soil pollution day by day. Pollution directly affects human health, nature, animals, underwater creatures, and birds. Due to environmental pollution, serious issues are occurring nowadays [1]. Due to air pollution, asthma and other diseases of the lungs have increased rapidly. The soil pollution occurring due to heavy metals has a direct impact on agriculture; therefore, many types of stomach diseases are observed [2]. Water pollution is the most serious issue nowadays, which occurs due to the generation of huge amounts of wastewater from the textile and other chemical industries [[3], [4], [5]]. About 70% of diseases are caused due to polluted water; therefore, water purification is the best and prime option to restrict this problem. From the last few decades, semiconductor materials were employed for various wastewater treatments and water purification processes [6,7]. However, the main problem with semiconductors is that visible light cannot be absorbed by the semiconductor nanomaterials having a wide band difference. Through mixing active metal and/or non-metal into bulk semiconductor materials, the activity of these may be increased. For the decolorization of dye contaminants, semiconductor photocatalysts such as TiO<sub>2</sub>, ZnO, CdO, NiO, and Fe<sub>2</sub>O<sub>3</sub> have been used [[8], [9], [10]]. However, the use of these materials in wastewater treatment is restricted due to some crucial issues which need to be tackled as early as possible [11]. The first issue is that these materials are only effective in the UV range and ineffective in the visible spectrum range. Second, recovery of these materials after their use is complex and thus, they are non-reusable. Lastly, these materials are not scalable to large applications [[12], [13], [14]].

Magnetic nanoparticles with a spinel structure are known as spinel ferrites. Ferrites are a kind of spinel that has the general formula of MFe<sub>2</sub>O<sub>4</sub> (M=Fe, Zn, Ni, Co, etc.), based on their crystal structures and magnetic properties [15]. The M and Fe are the metallic cations that are found at two separate locations, namely tetrahedral (A-site) and octahedral (B-site), respectively, in the case of normal spinel and some M preferred B-site in the case of inverse spinel [16]. Nickel ferrite (NiFe<sub>2</sub>O<sub>4</sub>), a well-known inverse spinel, is extremely valuable due to its numerous applications. One of its most common spinel ferrites is nickel ferrite (NiFe<sub>2</sub>O<sub>4</sub>). Its characteristics such as high magnetocrystalline anisotropy, high saturation magnetization, and peculiar magnetic composition catch the attention of the researchers [17]. NiFe<sub>2</sub>O<sub>4</sub> has a wide range of magnetic properties based on its particle size and morphology, such as paramagnetic, superparamagnetic, or ferrimagnetic conduct [18]. The magnetic nature such as paramagnetic, superparamagnetic, or ferrimagnetic of NiFe<sub>2</sub>O<sub>4</sub> depends on its particle size and morphology.

Different synthesis methodologies have been introduced to produce materials with suitable physicochemical properties such as sol-gel auto-combustion method, hydrothermal, and coprecipitation. The sol-gel auto-combustion method is a two-step procedure that includes the analysis of a precursor and its auto-ignition [19]. This process has the advantages such as homogeneous molecules, high purity and uniformity of the sample, and application of simple laboratory equipment. Individual and complex metal oxides were synthesized using a variety of

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fuels such as urea, glycine, citric acid, EDTA, PVA, sucrose, dextrose, and others [20]. The surface region, particle size distribution, and agglomeration of solution combusted powders are all affected by flame temperature and the volume of emitted gaseous compounds, which are in turn influenced by the type of fuel and the fuel-to-oxidant ratio [21]. As opposed to a substance formed with a single fuel, the use of a mixture of fuels allows for a decrease in particle size due to its better regulation of flame temperatures as well as the form and quantity of gaseous goods released.

Sen et al. reported that the nickel ferrite nanoparticles were synthesized by the sol-gel method using urea as fuel and studied its structural, morphological, elemental, and optical properties [22]. Shanmugam et al. synthesized cobalt ferrite nanoparticles applying urea and citric acid as the fuels through the sol-gel method [23]. They concluded that the different fuels affected the structural, elemental, and morphological properties of cobalt ferrite.

This research aims to show how the fuel mixing ratio of urea and glycine affects the structure, morphology, magnetic, and catalytic properties of  $NiFe_2O_4$  nanoparticles made by the sol-gel autocombustion method. The prepared  $NiFe_2O_4$  nanoparticles exhibit highly remarkable structural, magnetic, optical, and photocatalytic activity. The obtained results demonstrate excellent magnetic, optical, and photocatalytic actives.

# Section snippets

### Preparation of ferrite nanoparticles

The sol-gel auto-combustion process was used to make NiFe<sub>2</sub>O<sub>4</sub> nanoparticles. The raw materials used were nickel nitrate (Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O), ferric nitrate (Fe(NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O), urea (CH<sub>4</sub>N<sub>2</sub>O), glycine (C<sub>2</sub>H<sub>5</sub>NO<sub>2</sub>), and ammonia (NH<sub>3</sub>), all of which was 99.9% pure. The overall oxidizing and reducing valences of the materials were used to test the Stoichiometric compositions of metal nitrates and fuels, and the oxidizer to fuel ratio was set to 1: (60:40). To make a blended solution, metal nitrates were first...

# Thermal analysis

The thermal analysis was performed to understand the decomposition of metal hydroxide, removal of water, and ferritization temperature of the prepared NiFe<sub>2</sub>O<sub>4</sub> nanoparticles under a nitrogen atmosphere as shown in Fig. 3. And in the temperature range from 27°C to 1200°C with a heating rate of 20°C/min, the TG-DTA curve of the NiFe<sub>2</sub>O<sub>4</sub> nanoparticles is shown in Fig. 3 From TG-DTA analysis, it is clear that those three different events of weight loss were exhibited in the TGA curve and one...

#### Conclusion

In conclusion, urea and glycine as mixed fuel have been used to synthesize NiFe<sub>2</sub>O<sub>4</sub> nanoparticles. The processes of NiFe<sub>2</sub>O<sub>4</sub> development, as well as the characterization of the resulting compounds, were carried out with the help of X-ray diffraction, FTIR, Raman spectra, UV-vis diffuse reflectance spectroscopy, and FE-SEM. The values of crystalline size calculated from Modified Scherer's and W-H plot are nearly the same and exhibit nanocrystalline nature. The Debye temperature calculated from...

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper....

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