

Plant Extract Assisted Green Synthesis and Structural Studies of Spinel NiAl₂O₄ Nano-Catalysts

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Abstract: Spinel NiAl₂O₄ nanoparticles were synthesized via Aloe vera plant extract microwave heating method. Aloe vera plant extract-assisted combustion method makes straightforward synthesis, which is alternative process of spinel preparation. The synthesized spinel NiAl₂O₄ nanoparticles was successfully characterized by powder XRD pattern, FT-IR, EDX analysis, HR-SEM analysis, and VSM techniques. XRD, FT-IR and EDS results established the development of spinel and single cubic phase NiAl₂O₄ nanocrystals. The formation of spherical shaped nanoparticles was affirmed by HR-SEM technique. VSM measurements revealed that NiAl₂O₄ sample have superparamagnetic behavior.

Keywords: Spinel NiAl₂O₄; Nanoparticles; Aloe vera extract; Magnetic properties;

I. INTRODUCTION

Recently, nanostructured spinel type materials are attractive in materials science and nanotechnology, for the reason that of their novel physical, chemical and catalytic activities than that of their same bulkiness materials [1-5]. Among the various spinel materials, Nickel aluminate (NiAl₂O₄), has gained much attention in interdisciplinary areas due to their high mechanical strength, and chemical stability [6-8]. Various synthesis routes have been used to prepare the spinel type nanomaterials [8-10]. But the methods have some disadvantageous such as costly equipments and materials and difficult synthetic procedures. Recently, a facile microwave combustion method (MCM) has been used. In this route, the nanomaterials are synthesized at lower temperatures and also enough low cost with good controlled size of the products [10-12].

The present work focused on the synthesis of spinel NiAl₂O₄ nanoparticles by using green method on extract from Aloe vera microwave combustion method. Aloe vera is a permanent juicy belonging to the Liliaceal family [13-17]. To our knowledge, no literature is available on the synthesis of NiAl₂O₄ nanostructures by Aloe vera plant extract microwave heating method. Nevertheless, Aloe vera plant extracts using as the reducing agent by microwave heating method. Moreover, spinel NiAl₂O₄ nanomaterials are non-toxic, inexpensive, comparatively higher surface area

and the properties formulate them proper for use as inexpensively feasible nano-catalysts [18-28]

II. EXPERIMENTAL METHOD AND TECHNIQUES

A. Materials

Nitrates of Nickel and aluminum, plant extract as the raw materials were used by this method. Millipore water was used for this synthesis. Aloe vera-extract was prepared from a 5 g piece of methodically washed leaves were thinly cut and the gel obtained was liquefied in 10 ml of distilled H₂O and stirred for 30 min, which is known as Aloe vera plant extract. [20-26] Nitrates of Nickel, and aluminum were dissolved in the plant extract under stirring for 1 h and then located in a domestic microwave oven for 10 mins. After completion of the reaction, the attained powders were labeled as NiAl₂O₄

B. Characterization

The structural characterization of spinel NiAl₂O₄ nano-crystals were carry out using a Rigaku Ultima XRD ($\lambda = 1.5418 \text{ \AA}$). The functional groups were analyzed by Elmer FT-IR. The morphology was achieve with a Joel JSM 6360 HR-SEM analysis. Magnetic properties were carried out 3900 model VSM.

III. RESULTS AND DISCUSSION

A. XRD analysis

The structure, crystallite size, phase formation of the powder were established by the XRD pattern. Fig. 1 shows the XRD of spinel NiAl₂O₄ nanoparticle. The peaks of 31.13, 36.84, 38.64, 44.53, 49.14, 55.73, 59.43, 65.53, 74.25 and 77.52° can be absolutely indexed as fcc spinel NiAl₂O₄ (JCPDS card no. 38-0814).

The crystallite size measured using Scherrer's Eq. (1),

$$D = \frac{0.89\lambda}{\beta \cos \theta} \quad \text{---- (1)}$$

The calculated crystallite size is 18.35 nm.

B. FT-IR analysis

A wide-ranging band appeared in the expanse 3220-3440 cm⁻¹ showed the vibrations of H₂O (Fig. 2). A band at around 1630 cm⁻¹ was assigned by H-O-H vibration. The band at 2357 cm⁻¹ is due to the CO₂ vibration. The M-O stretching bands in the range 550-850 cm⁻¹, connected to the Al-O and Ni-O-Al [14, 15].

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C. SEM analyze

Fig. 3 shows HR-SEM image of NiAl₂O₄ sample. HR-SEM image clearly shows the agglomerated particle-like nano-crystals. From the results, it is assumed the microwave heating process, the microwaves are involved for nucleation and formed the final products with narrow size range of particles within few minutes.

D. VSM study

The magnetization of NiAl₂O₄ nanoparticle was analyzed through external field between ± 10 kOe by VSM at room temperature. Magnetization (M) versus applied field (H) curve is shown in Fig. 4. From the M-H curve, we can infer that a soft magnetic nature of NiAl₂O₄ material and also indicate superparamagnetism at ± 15 kOe. The obtained results show that the value of Ms is 1.45×10^{-4} emu/g. However, it is noted that lower Ms, Hc and Mr values confirmed the NiAl₂O₄ nanoparticles have soft nature of superparamagnetism [16-23].

IV. CONCLUSIONS

Spinel NiAl₂O₄ nano-catalysts were prepared by a easy microwave heating route using Aloe vera plant extract. The results of the prepared sample have a spinel. The manifestation of wide-ranging band between 550 and 850 cm⁻¹ exposed the arrangement of Al-O and Ni-O-Al bond appearance. SEM image showed the construction of well residential particle with nano grains. VSM studies revealed that NiAl₂O₄ showed superparamagnetism

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