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Plant extract assisted combustion synthesis and physical characterization studies of spinel Mn-Zn aluminate nanoparticles

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Abstract

Spinel Mn-Zn aluminate nano-catalyst was prepared successfully by a direct microwave heating method using corresponding metal nitrates as raw materials and *Aloe vera* extract used as the reducing agent. Powder XRD pattern analysis established the construction of single phase pure cubic spinel gahnite structure without any other impurities. The designed lattice parameter of the sample is 8.088 Å. The average crystallite sizes were predictable using Debye Scherrer's method, and it was found that 15.23 nm. FT-IR spectra confirmed the metal-oxygen stretching occurrences for the corresponding spinel structure of the samples. Surface morphology was studied by HR-SEM analysis and the observed images showed the features of well particle shaped crystals with nano-sized grains.

Keywords: Spinel Mn-Zn aluminate; *Aloe vera* plant extract; Microwave combustion.

1. Introduction

Recently, spinel type mixed metal oxide nano-crystals signify smart class of materials, which exhibits novel electro-chemical and magneto-optical characterization than those of their largeness materials [1-10]. Among the spinel oxides, spinel aluminates ($A^{2+}(Al^{3+})_2O_4$: A^{2+} = Zn^{2+}, Co^{2+}, Cu^{2+}) have become an significant materials, due to their impending applications in multidisciplinary areas [11-15]. Normal spinel represented by the formula $^{IV}(A^{2+})^{VI}(Al^{3+}Al^{3+})O_4$, whereas the inverse spinel with the formula $^{IV}(Al^{3+})^{VI}(A^{2+}Al^{3+})O_4$ [16-20]. Among various spinel type mixed metal oxide nano-crystals, zinc aluminate ($ZnAl_2O_4$), has attracted lot of interest in interdisciplinary areas, because of their opto-electronic and catalytic applications [21-25].

Various methods such as hydrothermal, polyol, co-precipitation, sol-gel, polymeric precursor methods [25-30], etc. have been used for the synthesis of spinel type mixed metal oxide nano-crystals. The above method has several disadvantageous such as time and energy overriding and also claims costly materials. Also, the preparation procedures were performed for long-time at high temperature. Consequently, in the present investigation, an effort is given to synthesize single phase cubic Mn-$ZnAl_2O_4$ spinel by *Aloe vera* plant extract supported microwave irradiation route. The importances of the microwave irradiation method produce a high degree of pure products [31]. Recently, *Aloe vera* plant extract has been used in the preparation $CoFe_2O_4$ spinel materials [32], *Aloe vera* extract supply higher yield nanosized functional materials with crystalline nature. The advantages of *Aloe vera* plant extract-assisted microwave irradiation route include the use of cheap and nontoxic precursors.

2. Experimental division

2.1. Materials and preparation methods

Nitrates of zinc, manganese, and aluminum as the raw materials, and *Aloe vera* extract as the reducing mediator were used by this method. Millipore water was used for this synthesis. *Aloe vera* -extract was prepared from a 5 g piece of systematically washed *Aloe vera* plant leaves were thinly cut and get the gel and liquefied in 10 ml of distilled water, stirred for 30 min upto get clear solution, which is known as *Aloe vera* plant extract. Nitrates of manganese, zinc, and aluminum were dissolved in the *Aloe vera* extract underneath stirred for 1 h and then located in a domestic microwave oven for 10 minutes, obtained solid powder and then washed with water and ethanol and followed drying at 70 ºC for 1h.

2.2. Characterization performances

Structural formation characterization of Mn-$ZnAl_2O_4$ spinel nano-crystals were carry out using a Rigaku Ultima XRD (λ = 1.5418 Å). The metal-oxide functional groups of the samples were analyzed by Perkin Elmer FT-IR spectra. Surface morphology was achieved with a Joel JSM 6360 HR-SEM analysis at desired magnification.

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3. Results and discussion

3.1. XRD structural analysis

Powder XRD pattern was used to investigate the phase structure and average crystallite size of Mn-$ZnAl_2O_4$ NPs. Fig. 1 shows the typical XRD result of spinel Mn-$ZnAl_2O_4$ NPs. The XRD peaks are corresponding to (220), (311), (222), (400), (331), and (620) diffraction planes. In relation with XRD pattern, all peaks diffraction could be absolutely confirmed as centered cubic structure of spinel Mn-$ZnAl_2O_4$ NPs (JCPDS card no. 05-0669).

The crystal size was calculated by Scherrer's formula,

$$D = \frac{0.89\lambda}{\beta \cos\theta}$$

where 'D' is the crystallite size, 'β' is the full width at half maximum (FWHM),'λ' is the X-ray wavelength, and 'θ' is the Bragg diffraction angle. The designed crystal size of the sample is 15.23 nm.

The lattice parameter was intended based on the following Eq.

$$a = d_{hkl}(h^2 + k^2 + l^2)$$

where, 'a' is the lattice constant, 'd_{hkl}' the inter planar spacing matching to the Miller indices, *'h', 'k', and 'l'* the miller indices [33]. The obtained lattice parameter value is 8.088 Å.

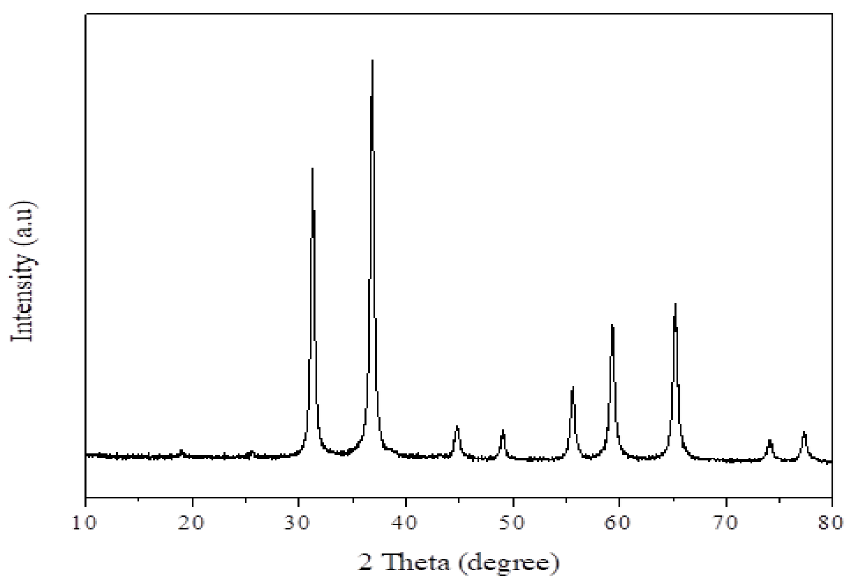

Figure 1. Powder XRD pattern of spinel Mn-$ZnAl_2O_4$ NPs.

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3.2 FT-IR spectral analysis

FT-IR spectra of Mn-$ZnAl_2O_4$ NPs are given in Fig. 2. Water molecule has a well-built and wide-ranging fascination band centered in the range of 3255-3445 cm^{-1}. It can be seen that the peaks at around 3435 cm^{-1} and at around 1622 cm^{-1}, assigned to the -OH stretching and O-H bending vibrations of adsorbed H_2O, respectively. The absorption peak at 2344 cm^{-1} may be the stretching vibration of CO_2 from environment. Mn-$ZnAl_2O_4$ NPs, the M-O stretching vibrations are accounted in the range 550-900 cm^{-1}, connected with the sensations of Al-O and M-O-Al peaks [13-15].

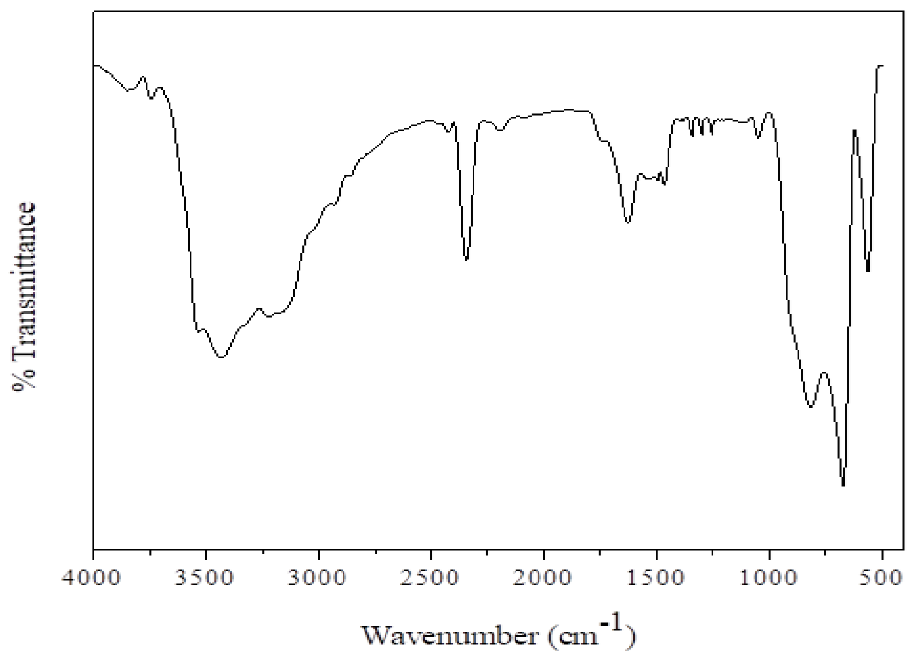

Figure 2. FT-IR spectra of spinel Mn-$ZnAl_2O_4$ NPs.

3.3 SEM morphology study

HR-SEM technique was used to find the morphologies of the spinel Mn-$ZnAl_2O_4$ NPs. Fig. 3 shows HR-SEM image of Mn-$ZnAl_2O_4$ NPs. The surface morphology of Mn-$ZnAl_2O_4$ NPs consists of well-developed particles with different shape and size of crystals with less uniform; varying size distribution with relatively well crystallized grain size lesser than 50 nm.

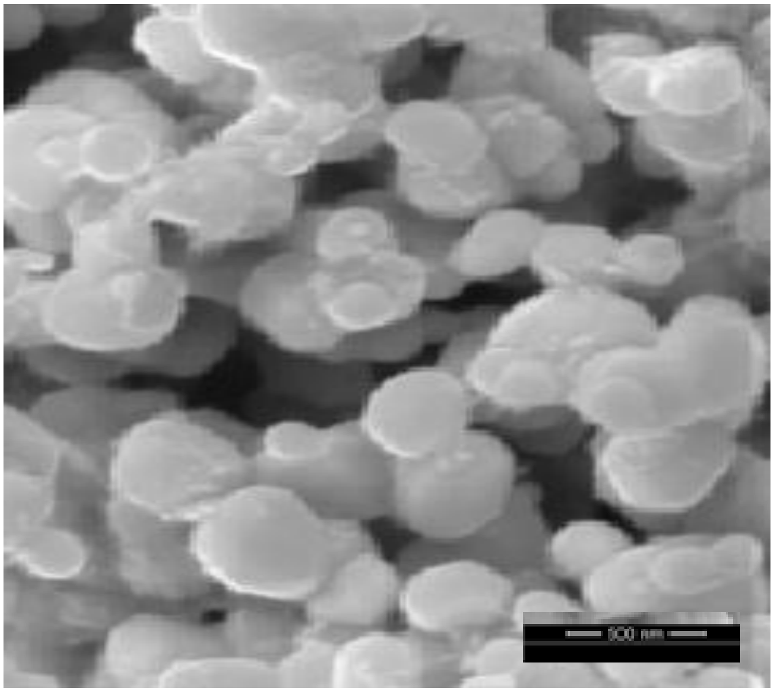

Figure 3. HR-SEM image of spinel Mn-$ZnAl_2O_4$ NPs.

3.4 EDX spectral study

EDX spectral study of the prepared spinel Mn-$ZnAl_2O_4$ NPs is given in Fig. 4. Fig. 4 shows the EDX spectra of $ZnAl_2O_4$. EDX results showed that the peaks of Zn, Al and O elements in Mn-$ZnAl_2O_4$ NPs and the absence of other secondary peak, which inveterate the sample is pure [13-15].

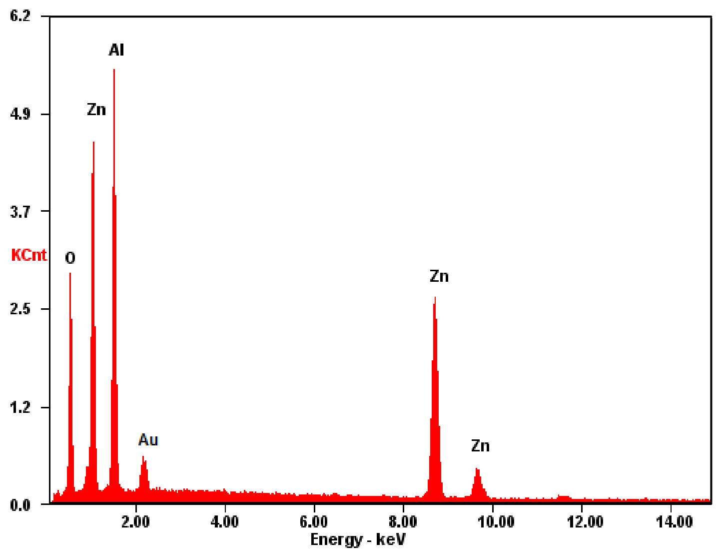

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Figure 4. EDX spectra of spinel Mn-$ZnAl_2O_4$ NPs.

3.5 VSM measurements

The magnetic behavior of spinel Mn-$ZnAl_2O_4$ NPs were investigated by using the external magnetic field between ±15 kOe using room temperature vibrating sample magnetometer (VSM). Magnetizations (M) versus magnetic field (H) behavior plots are shown in Fig. 5. These M-H curves are typical for a soft magnetic material and indicate paramagnetism in the field ranges of ±15 kOe [31-33].

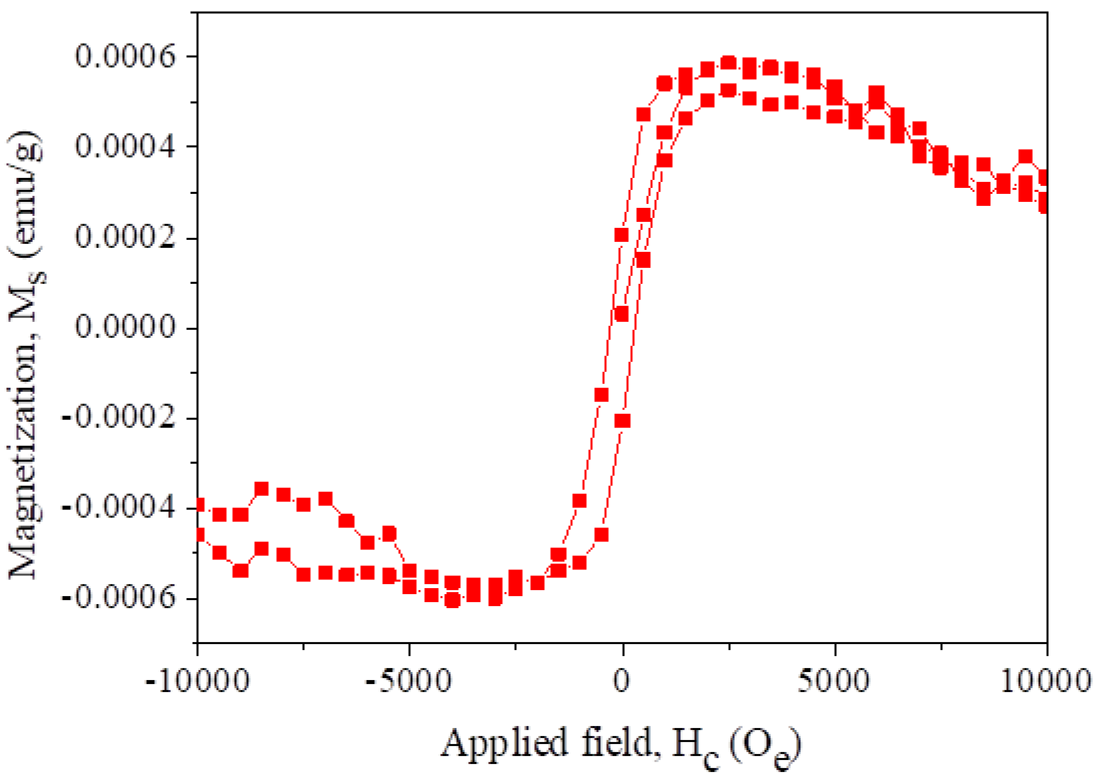

Figure 5. VSM analysis of spinel Mn-$ZnAl_2O_4$ NPs.

4. Conclusions

Spinel Mn-$ZnAl_2O_4$ nano-crystals were prepared by microwave irradiation route using *Aloe vera* extract as reducing agent. Powder XRD pattern suggested the formation of pure gahnite Mn-$ZnAl_2O_4$ spinel. Also, the XRD and EDX results specify that the synthesized $ZnAl_2O_4$ nano-crystals have spinel lattice. The manifestation of two peaks between 550 and 900 cm^{-1} in FT-IR spectra exposed the formation of spinel Mn-$ZnAl_2O_4$ structure. HR-SEM image depict the well urbanized particle-like crystal morphology with nano-sized grains on the surfaces.

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