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Facile synthesis, structural and magnetic characterization studies of spinel copper aluminate nanoparticles

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Abstract

Spinel $CuAl_2O_4$ nanoparticles were prepared effectively by simplistic, economical microwave heating method using *Aloe vera* extract as reducing agent. The samples were successfully characterized by XRD pattern, EDX spectra, FT-IR analysis, HR-SEM analysis, and VSM instrumentation techniques. XRD, EDX and FT-IR results demonstrated that the products contain a pure single-phase spinel structure lacking of other secondary phase impurities. SEM results confirmed the spherical shaped nanoparticle morphology of the sample. Magnetic characterization property was confirmed by VSM analysis. VSM hysteresis loop established the superparamegnetism of the sample and the magnetization (M_s) value of $CuAl_2O_4$ is 0.025 emu/g.

Keywords: Spinel $CuAl_2O_4$; Nanocrystals; *Aloe vera* extract; Magnetic property.

1. Introduction

In recent times, spinel transition semiconductor oxide nanomaterials have been broadly studied, due to their sole opto-electromagnetic and catalytic/photocatalytic properties than those of their bulkiness materials [1-3]. Commonly, spinel aluminates ($A^{2+}(Al^{3+})_2O_4$: A^{2+} = Zn^{2+}, Co^{2+},

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Cu^{2+}) have develop into an significant materials, owing to their probable applications in different multidisciplinary areas [3-5]. Among various spinel aluminates manganese aluminate ($MnAl_2O_4$) has been investigated extensively [5]. Several techniques have been used to prepare the spinel type transition metal oxide semiconductor nanoparticles, for example solvothermal, co-precipitation, solvothermal, sol-gel and hydrothermal methods [6-10] etc. But, the above said methods meet several inconveniences for instance required long time procedures, high temperature and high-energy overriding, costly and complicated equipments and multifaceted procedures [11-16].

Amongst the above conservative methods, easy and cost proficient routes to prepare spinel metal oxide semiconductor nanoparticles by exploitation of inexpensive, cheap, low cost, non-toxic and environmentally benevolent precursors are unmoving key issues. Therefore, the enlargement of superficial and ecological gentle route is severely essential [11-16]. In this present work, spinel $CuAl_2O_4$ nanostructure was prepared by a simple microwave irradiation method using *Aloe vera* extract as the reducing agent. Currently, the plant extract-assisted microwave heating route has enthralled and extraordinary interest in fabricating useful nano materials [16]. Additionally, microwave irradiation method is a short time preparation route and no need the complex equipment, which making this route is very attractive. In recent times, the bio based synthetic route is much uncomplicated and provides pure and better yield materials with satisfactory possessions. *Aloe vera* extract act as a bio-reducing agent.

2. Experimental

2.1. Materials and methods

Aluminium nitrate, copper nitrate, and *Aloe vera* extract as the raw materials were used. Millipore water was used for this synthesis. *Aloe vera* extract was prepared from a 5 g piece of systematically washed leave was thinly cut then the gel obtained was liquefied in 10 ml of distilled water and stirred at 30 min to get clear solution, which is known as *Aloe vera* extract. Nitrates of manganese, and aluminum were dissolved in the *Aloe vera* extract under stirring for 1 h and then located in a domestic microwave oven for 15 min, solid powders are formed, and then washed with water and ethanol and kept at 70 ºC for 1h.

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2.2. Characterization

Structural formation of spinel $CuAl_2O_4$ nano-crystals were carry out using a Rigaku Ultima XRD (λ = 1.5418 Å). The corresponding metal-oxide group formation was analyzed by Perkin Elmer FT-IR spectra. Surface morphology was achieved with a Joel JSM 6360 HR-SEM analysis at desired magnification.

3. Results and discussion

3.1. Powder XRD analysis

Crystal nature, crystal formation, size and purity were established by analyzing the powder X- XRD pattern. Fig. 1 shows the XRD patterns of $CuAl_2O_4$ sample. The XRD diffraction peaks may possibly index single-phase spinel cubic structure of $CuAl_2O_4$.

Lattice parameter was designed using the given formula in Eq. (1):

$$\sin^2\theta = \frac{\lambda^2}{4}\left[\frac{4}{3}\left(\frac{h^2+hk+k^2}{a^2}\right)+\frac{l^2}{c^2}\right] \quad \text{---- (1)}$$

where *'θ'* is the diffraction angle, *h*, *k*, and *l* are Miller's indices and *'λ'* is the incident wavelength (λ = 0.1540 nm). The lattice parameter of $CuAl_2O_4$ sample is 8.326 Å [3].

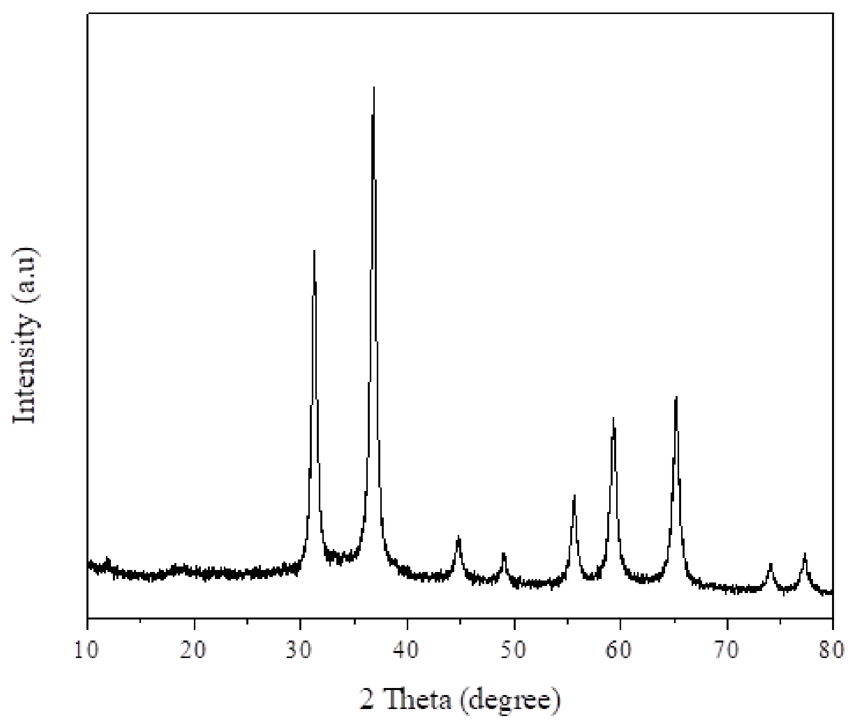

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Figure 1. Powder XRD pattern of spinel $CuAl_2O_4$ sample.

The crystallite size of was designed by Scherrer formula given in Eq. (2):

$$L = \frac{0.89\lambda}{\beta \cos\theta} \qquad \text{------ (2)}$$

where *'L'* is the crystallite size, *'θ'* is the Braggs angle diffraction, *'λ'* is the X-ray wavelength (1.5406 Å) and *'β'* is Full Width at Half Maximum (FWHM). The calculated crystallite size of spinel $CuAl_2O_4$ sample is 10.55 nm. Nevertheless, the microwave irradiation process, the microwave-oven has produced microwaves energy at a power of 850 W and converted into thermal energy, which resulting the functional nano-sized $CuAl_2O_4$ sample.

3.2. FT-IR spectroscopy

FT-IR spectra of spinel $CuAl_2O_4$ sample is given in Fig. 2. A wide-ranging vibration band at ~3420 cm^{-1} to 3250 cm^{-1} is connected with the OH vibration of water molecules, representing superior amount of exterior OH. In addition, two main wide M-O bands in the range of 400-950 cm^{-1} indicate the spinel materials [3], which is spinel $CuAl_2O_4$ sample.

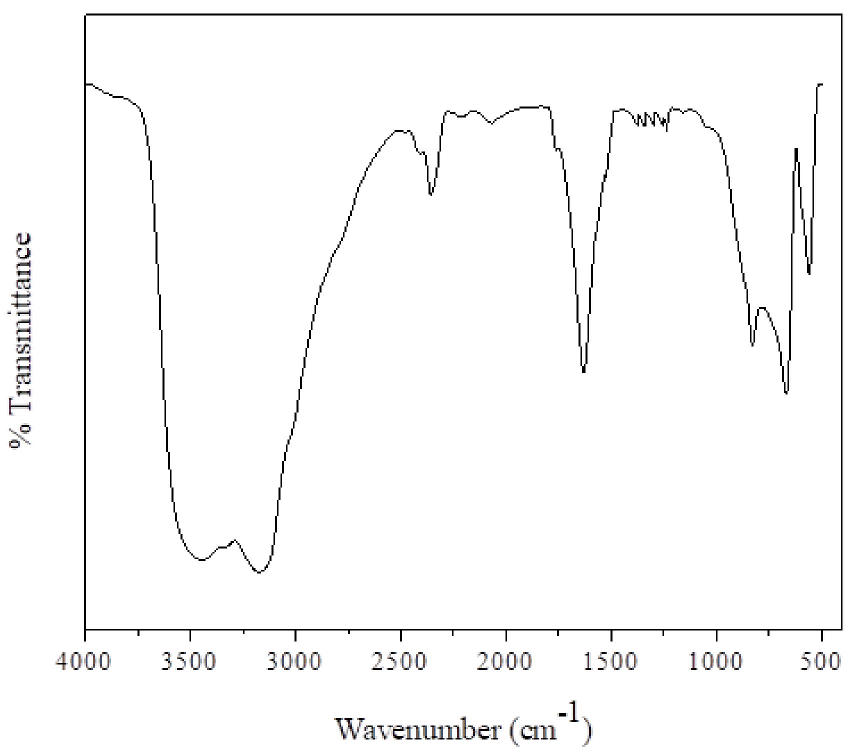

Figure 2. FT-IR spectra of spinel $CuAl_2O_4$ sample.

3.3. HR-SEM analysis

The surface morphology was analyzed by HR-SEM analysis and is exposed in Fig. 3. Fig. 3 shows the HR-SEM image of spinel $CuAl_2O_4$ sample exhibit homogeneous sphere-like nanoparticles. The smaller agglomerations of the products are mainly due to the influence of microwaves for the homogeneous distribution of the samples, which makes agglomeration and also magnetic relations between the resources.

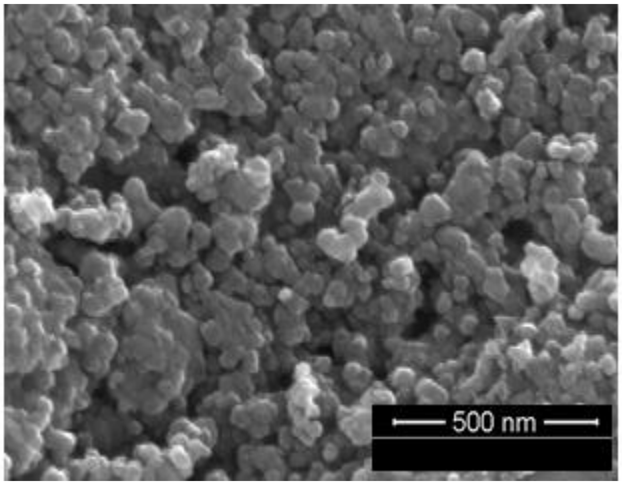

Figure 3. HR-SEM image of spinel $CuAl_2O_4$ sample.

3.4. EDX analysis

The elemental and sample purity was confirmed by EDX technique. Fig. 4 shows the EDX spectra of $CuAl_2O_4$ sample, which contains the peaks of Al, Cu and O and the absence other secondary peak observation, confirmed the purity products.

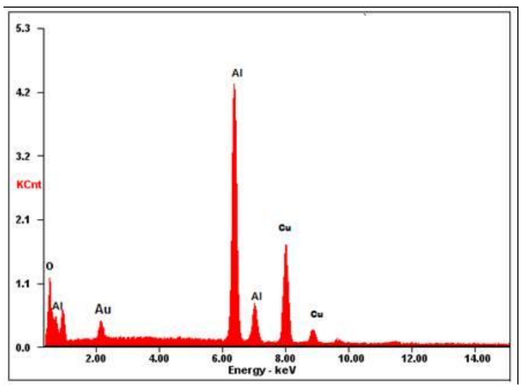

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Figure 4. EDX spectra of spinel $CuAl_2O_4$ sample.

3.5. VSM measurements

The magnetic assets of the spinel $CuAl_2O_4$ sample was analysed by VSM at field ranging upto ± 10 kOe is exposed in Fig. 5. VSM hysteresis (M-H) loop confirmed superparamagnetism. The saturation magnetization (M_s) value was obtained to be 0.023 emu/g. From the VSM results, it was inference the magnetic property of the products depending on their size, and shape of the nanopowders.

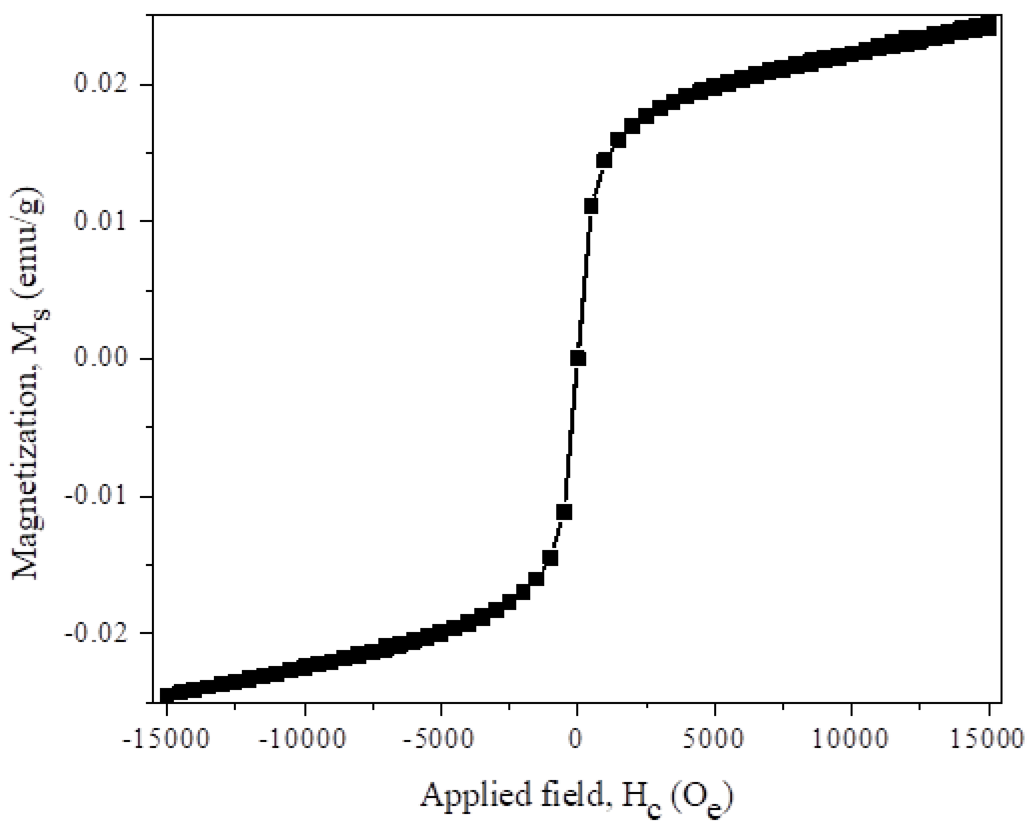

Figure 5. Magnetic (M-H) hysteresis loop of spinel $CuAl_2O_4$ sample.

4. Conclusions

Spinel $CuAl_2O_4$ sample was synthesized successfully by a facile microwave heating route using *Aloe vera* extract. XRD, EDX and FT-IR results specified that the prepared spinel $CuAl_2O_4$ sample have spinel structure with well crystalline product and also free from other

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phase impurities. HR-SEM result revealed that spinel $CuAl_2O_4$ sample contain nanoparticle-like morphology. The specific M_s values were obtained to be 0.023 emu/g for spinel $CuAl_2O_4$ sample.

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