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Original Article

One-Pot Hydrothermal synthesis and Characterization of Heulandite/$NiFe_2O_4$ Nano-composite

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Abstract

We present the characterisation and one-pot hydrothermal production of a Heulandite-Nickel Ferrite (HEU/NiFe2O4) nanocomposite. Heulandite, a naturally occurring zeolite crystal, was pre-treated and then combined with nickel ferrite nanoparticles in a hydrothermal procedure that was set at 180˚C for eighteen hours in order to generate a nanocomposite. The hydrothermal treatment changed the structural and functional properties of Heulandite crystals, facilitating the incorporation of nickel ferrite nanoparticles onto their surface. The resulting HEU/NiFe2O4 nanocomposite was characterized using methods such as powder X-ray diffraction (P-XRD) and Fourier-transform infrared spectroscopy (FTIR). The presence of significant functional groups and metal-ferrite linkages was indicated by the FTIR spectra, which displayed characteristic bands connected to the nickel ferrite and heulandite aluminosilicate framework.

The successful synthesis and characterization of a heulandite-nickel oxide nanocomposite are demonstrated in this work. This study reports the successful synthesis and characterization of a Heulandite/$NiFe_2O_4$ nanocomposite using a one-pot hydrothermal method. Natural heulandite zeolite was combined with nickel and iron precursors and treated hydrothermally at 180 °C for 18 hours. The process resulted in the formation of a nanocomposite wherein nickel ferrite nanoparticles were effectively embedded onto the heulandite matrix. Structural and functional characterizations were carried out using Powder X-ray Diffraction (P-XRD) and Fourier Transform Infrared Spectroscopy (FTIR).

Keywords: *Heulandite, Nickel Ferrite ($NiFe_2O_4$), Nanocomposite, One-pot Hydrothermal Synthesis, Zeolite, FTIR Analysis, Powder X-Ray Diffraction (P-XRD), Magnetic Nanoparticles*

Introduction

Recently, there has been an increased interest in nanostructured magnetic materials owing to their prospective and current applications in information technology, such as high-density information storage [6] and magnetooptical devices [7]. Additionally, their use in magnetic resonance imaging (MRI) [4], magnetic fluids [5], biotechnology and biomedicine [2], [3] and catalysis [1] also reveals the multidisciplinary relevance of these materials.

Researchers have shown great interest in one type of magnetic nanomaterial, nickel ferrites, due to their peculiar magnetic and physical properties. They are used in important fields such as gas sensors [9] and catalysts [8], as well as in magnetic materials [12], microwave devices [11], and even in magnetic fluids [10]. Furthermore, nickel ferrites are considered an optimal matrix of inert anodes for aluminum electrolysis due to their pronounced corrosion resistance and electrochemical stability in molten cryolite–alumina [13]. The inverse spinel structure of nickel ferrite (NiFe2O4), a well-known soft magnetic material, shows that Ni2+ ions are only found in octahedral sites, while Fe3+ ions are halved to find tetrahedral and octahedral sites. The magnetic moment of anti-parallel spins between Fe3+ ions at tetrahedral sites and Ni2+ ions at octahedral sites is the source of ferrimagnetism [14]. Everyone knows that the grain size and microstructure of nickel ferrite nanoparticles affect their properties and that these processes undergo change due to the synthesis techniques used. Different synthesis techniques have created a significant nickel ferrite nanoparticles base.

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Monodispersed NiFe2O4 nanoparticles have been synthesized using Thermolysis of the Ni2+Fe23+–oleate complex [15] and the nonaqueous solvothermal method using organic and inorganic precursors [16]. NiFe2O4 nanoparticles have also been synthesized using flash microwave induced thermohydrolysis resulting in nanoparticles of high specific surface area (about 240 m2/g) and elementary size of 4-5 nm [17]. Various other techniques have also been reported. For example the reverse micelle technique [19] And the sonochemical decomposition process [18], have been used to create superparamagnetic NiFe2O4 nanoparticles with a diameter of less than 10 nm. NiFe2O4 nanospheres with a distribution region of 60–160 nm and room temperature saturation magnetization (Ms) values near 55 emu/g have been prepared using the reverse emulsion-assisted hydrothermal method [20]. By using the organic acid precursor method [21] and the template-assisted sol–gel method [22], NiFe2O4 nanoparticles with enhanced saturation magnetization were produced, with Ms values exceeding 60 emu/g. Most existing methods come with a lot of challenges. They often involve complicated procedures, require expensive chemicals and equipment, take a long time to complete, and usually give low yields. On top of that, they can produce unwanted byproducts, like nickel oxide (NiO). Because of these issues, there's still a strong need for a better, more practical approach—something that's simple, reliable, cost-effective, and environmentally friendly—especially when it comes to producing nano-ferrites on a large scale.

One promising alternative is the solid-state reaction method. This technique has several advantages: it doesn't need any solvents, it's less likely to cause contamination, it's affordable, and it can produce a high yield with good selectivity. So far, researchers have used this method to successfully synthesize a few types of oxide nanoparticles [23].

Ceylan et al. [24] used a solid state reaction process to create NiFe2O4 nanoparticles with core/shell structures. In order to create Ni33Fe67 alloy nanopowders, the necessary quantities of pure Ni and Fe metals are first simultaneously evaporated at 1500 °C with 5 Torr He pressure, and then inert gas condensation (IGC) is performed. The resulting Ni33Fe67 alloy nanopowders were then annealed at various annealing temperatures for 12 hours under ambient conditions to produce NiFe2O4 nanoparticles. NiFe2O4 nanoparticles were created using a solid-state reaction in our earlier studies [25], [26]. NiFe2O4 nanoparticles in a single phase were obtained by calcining the precursors made by adequately grinding FeSO4·Na2O, NiSO4·, 2O, and NaOH as reactants and NaCl as dispersant at room temperature.. To optimize the synthetic parameters, the impact of preparation method, dispersant content, calcination temperature, and heat preservation duration on particle size and shape has been thoroughly examined. The findings demonstrate that the solid-state reaction technique is regarded as a practical, affordable, and efficient way to prepare NiFe2O4 in a high yield. However, the manual grinding process has numerous drawbacks, including low efficiency and poor reproducibility, which make large-scale manufacturing challenging. Consequently, a different method for producing nickel ferrite nanoparticles in large quantities is still required..

In recent years, high-energy milling has attracted considerable attention for its ability to produce solids with enhanced or unique physical and chemical properties. This is largely due to the highly non-equilibrium nature of the milling process. For instance, high-purity $NiFe_2O_4$ coarse powders have been milled in a planetary ball mill at room temperature, yielding metastable nanocrystalline nickel ferrite with an average crystallite size of around 9 nm [12].

A combination of co-precipitation and mechanical alloying has also been used to synthesize ultrafine nickel ferrite particles, producing a relatively uniform structure with a mean particle size of approximately 10 nm [27]. Bid et al. [28] investigated nanocrystalline $NiFe_2O_4$ prepared by high-energy ball milling of a stoichiometric mixture of powdered NiO and α-Fe_2O_3. Their findings showed that extended milling led to a significant increase in ferrite formation, while particle size and lattice parameters gradually decreased.

Similarly, Hajalilou et al. [29] synthesized $NiFe_2O_4$ nanoparticles through planetary ball milling of stoichiometric NiO and Fe_2O_3 powders. During the mechanochemical reaction, Ni^{2+} and Fe^{3+} ions diffused and counter-diffused to form $NiFe_2O_4$ nanoparticles. These particles exhibited ferromagnetic behavior, with a saturation magnetization of approximately 8.5 emu/g and negligible coercivity.

Experimental and Synthesis:

Materials

All chemicals: Anhydrous NiCl2, FeCl2.4H2O, NaOH, HCl, NH4OH, and distilled water were obtained from Fluka and used without further purification. Natural heulandite brought from Chattrapati Sambhaji Nagar (Previously Known as Aurangabad).

Synthesis of Heulandite/NiFe2O4 Nanocomposite

A one-pot hydrothermal method was employed to synthesize the Heulandite/$NiFe_2O_4$ nanocomposite. The detailed synthesis procedure is illustrated in Scheme 1. Initially, 0.314 g of $NiCl_2$ and 0.658 g of $FeCl_2·4H_2O$ were each dissolved in 25 mL of distilled water under continuous stirring. Subsequently, 0.70 g of zeolite (heulandite) was added to the mixed solution, followed by stirring for 30 minutes to ensure uniform dispersion. The pH of the solution was then adjusted to 11.8 by the dropwise addition of 4 mL of NH_4OH and 5 M NaOH.

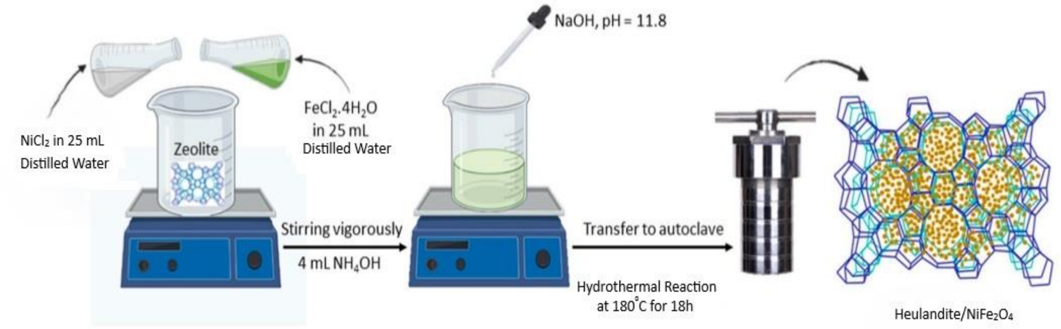
The resulting mixture was transferred into a 100 mL stainless-steel autoclave and subjected to hydrothermal treatment at 180 °C for 18 hours. For comparison, $NiFe_2O_4$ nanoparticles (NPs) were also synthesized under the same conditions, but in the absence of heulandite. The formation of the Heulandite/$NiFe_2O_4$ nanocomposite proceeds via a series of reactions, as described by equations (1)–(4).

$$Fe2+ + 2\ OH^- \rightarrow Fe(OH)2 \qquad (1)$$

$Fe(OH)_2 + OH^- \rightarrow FeOOH + H_2O$ (2)

$Ni^{2+} + 2\ OH^- \rightarrow Ni(OH)_2$ (3)

$Ni(OH)_2 + 2\ FeOOH \rightarrow NiFe_2O_4 + 2H_2O$ (4)



Scheme 1. Procedures for the synthesis of magnetic Heulandite/$NiFe_2O_4$ nanocomposite

Characterization of HEU/$NiFe_2O_4$ NCs:

X-ray diffraction (XRD) is a widely used technique for identifying crystalline phases and evaluating the structural properties of materials. In the present study, XRD analysis was conducted using a tabletop diffractometer (Miniflex 600, RIGAKU) to characterize the crystallinity and phase composition of the synthesized samples.

To complement the structural analysis, Fourier Transform Infrared Spectroscopy (FTIR) was employed to investigate the functional groups present in the material. The FTIR spectra were recorded using a spectrophotometer (Shimadzu, Japan). FTIR operates by measuring the absorption of infrared radiation by molecular bonds, where each functional group exhibits characteristic absorption peaks at specific wavenumbers (cm^{-1}), producing a unique spectral fingerprint. This allows for precise identification of the chemical functionalities within the sample

Results and discussion:

P-XRD

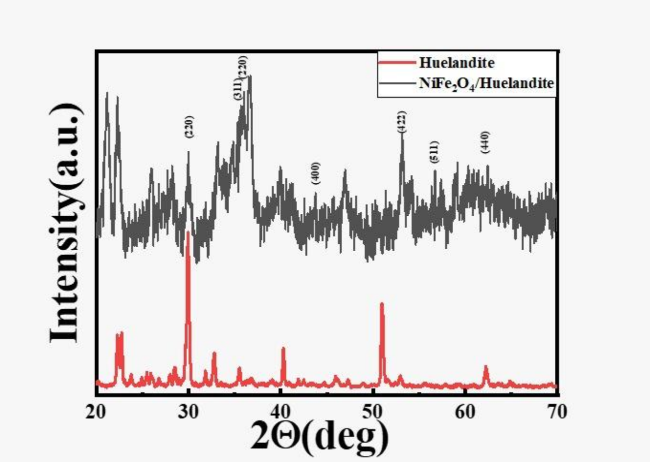


Fig.1. XRD patterns of Heulandite (Red), Heulandite/$NiFe_2O_4$ nanocomposite (Line)

Heulandite is a naturally occurring zeolite mineral characterized by a complex crystalline structure. In the XRD pattern, pure heulandite (represented by the red line) exhibits multiple sharp and well-defined peaks, indicative of a high degree of crystallinity. The narrow width of these peaks suggests a well-ordered crystal lattice, with distinct 2θ values reflecting the characteristic diffraction pattern of the heulandite phase.

In the case of the Heulandite/$NiFe_2O_4$ nanocomposite (represented by the black line), the XRD pattern becomes more complex due to the incorporation of nickel ferrite ($NiFe_2O_4$) nanoparticles into the heulandite matrix. Compared to pure heulandite, the composite exhibits additional peaks and modified intensity profiles, confirming the successful formation of the $NiFe_2O_4$ phase. These new peaks partially overlap with and obscure those of heulandite, indicating structural integration.

The characteristic diffraction peaks of the $NiFe_2O_4$ spinel structure are observed at 2θ values corresponding to the (220), (311), (222), (400), (422), (511), and (440) planes. The appearance of these peaks confirms the successful synthesis of the spinel-type $NiFe_2O_4$ within the zeolite matrix.

Additionally, the broadened peak profiles suggest that the $NiFe_2O_4$ particles are in the nanometer size range.

Changes in the intensity and broadening of the heulandite peaks further indicate an interaction between the two phases. Specifically, the reduced peak intensity and broadening in the composite sample suggest a decrease in crystallinity, which may result from two factors: (1) **Disturbance of the Heulandite framework** due to the incorporation of $NiFe_2O_4$, and (2) **Lower overall crystalline content** within the nanocomposite. These observations collectively support the formation of a well-integrated Heulandite/$NiFe_2O_4$ nanocomposite structure

FTIR

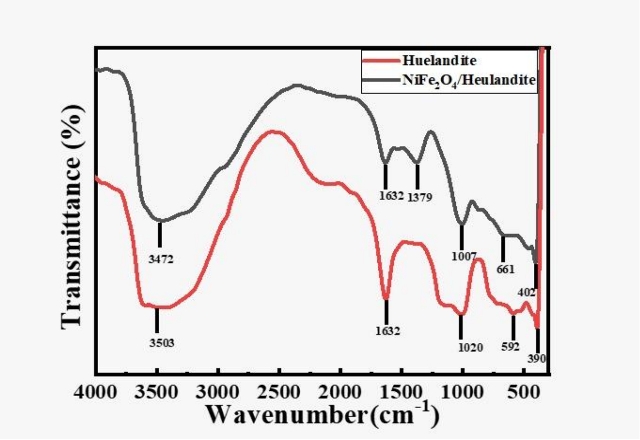


Fig.2. FTIR of Heulandite (Red), Heulandite/NiFe2O4 nanocomposite (black)

The broad absorption band observed at 3472 cm^{-1} in the FTIR spectrum corresponds to the O–H stretching vibrations of adsorbed water molecules and structural hydroxyl groups within the Heulandite framework. The bending vibrations of H–O–H from these water molecules contribute to the band at 1632 cm^{-1}. The peaks at 1007 cm^{-1} and 661 cm^{-1} are associated with Si–O stretching vibrations in the Heulandite silicate framework, which is composed of interconnected SiO_4 tetrahedra, characteristic of aluminosilicate zeolites. Additionally, the peak at 402 cm^{-1} is likely due to Si–O–Si bending vibrations within the zeolite lattice. Other peaks—such as those at 3503, 1379, 1020, and 592 cm^{-1}—are also attributed to vibrations within the Heulandite structure, possibly involving Al–O bonds or related structural units.

In comparison to pure Heulandite, the FTIR spectrum of the Heulandite/$NiFe_2O_4$ nanocomposite exhibits noticeable broadening and shifts in peak positions, which indicate significant interactions between Heulandite and nickel ferrite. The O–H stretching peak at 3472 cm^{-1} remains present but with altered intensity and shape, suggesting that the introduction of nickel ferrite affects the hydroxyl groups and/or the water adsorption behavior of Heulandite.

The Si–O stretching bands at 1007 cm^{-1} and 661 cm^{-1} also display changes, further supporting the occurrence of structural interactions between the two phases. In the nanocomposite spectrum, new peaks or changes in existing peak intensities may arise from the vibrational modes of nickel ferrite itself. Notably, a peak observed between 590 and 600 cm^{-1} is commonly linked to Fe–O stretching vibrations in the tetrahedral sites of the $NiFe_2O_4$ spinel structure, confirming the presence of $NiFe_2O_4$ within the composite.

These spectral changes—including peak broadening, shifts, and intensity variations—indicate that nickel ferrite and Heulandite interact, potentially through mechanisms such as physical adsorption, chemical bonding, or ion exchange. Moreover, the observed modifications may reflect changes in crystallinity or particle size distribution in the nanocomposite. The identification of specific functional groups thus provides valuable insight into the surface chemistry and structural composition of both the pure Heulandite and the resulting composite.

Conclusion

The successful formation of the Heulandite/$NiFe_2O_4$ (HEU/$NiFe_2O_4$) nanocomposite is supported by both XRD and FTIR analyses. XRD confirms the incorporation of $NiFe_2O_4$ nanoparticles and suggests nanoscale structural interactions with the Heulandite matrix. Complementary FTIR analysis reveals significant shifts and broadening of characteristic vibrational bands, indicating strong interactions between the two components at the molecular level. These findings collectively confirm the formation of a true nanocomposite and provide critical insights into its chemical structure and potential applications in areas such as catalysis, environmental remediation, and magnetic materials.

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Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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32. Recent advances on nickel nano-ferrite: A review on processing techniques, properties and diverse applications
Garima Rana, Pooja Dhiman , Amit Kumar, Dai-Viet N. Vo, Gaurav Sharma , Shweta Sharma, Mu. Naushad
33. Preparation and Characterization of Nickel ferrite Nanoparticles via Co-precipitation Method
34. Synthesis of surfactant-assisted nickel ferrite nanoparticles (NFNPs@surfactant) to amplify their application as an advanced electrode material for high-performance supercapacitors Muhammad Waheed Mushtaq*[a], Muhammad Shahbaz[b], Rabia Naeem[b], Shahid Bashir[c], Shahzad Sharif*[b], Kainat Ali[a] and Naveed Aslam Dogar[a]
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37. Clinoptilolite-Heulandite Nomenclature (David L. Bish and Jeremy M. Boak)
38. Synthesis of a heulandite-type zeolite by hydrothermal conversion of zeolite Pl (Sherwin Khodabandeh and Mark E. Davis) Sherwin)
39. Clinoptilolite-heulandite: applications and basic research (Thomas Armbruster)
40. Luminescence from Zinc Oxide Nanostructures and Polymers and their Hybrid Devices Magnus Willander*, Omer Nur, Jamil Rana Sadaf, Muhammad Israr Qadir, Saima Zaman, Ahmed Zainelabdin, Nargis Bano and Ijaz Hussain
41. The selective heavy metal ions adsorption of zinc oxide nanoparticles from dental wastewater Mengqin Gua, Liying Haoa, Yigan Wanga, Xiyu Lia, Yifan Chenc, Wei Lia, Li Jianga)
42. ZnO as a Functional Material, a Review (Michał A. Borysiewicz)
43. A review of ZnO nanoparticles as solar photocatalysts: Synthesis, mechanisms and applications (Chin Boon Onga, Law Yong Ngb, Abdul Wahab Mohammad
44. Effect of biogenic nanoparticles on growth physiological, biochemical traits and antioxidant on olive tree in vitro (Luca Regna, Daniele Del Boone et.al)
45. Properties and application of zeolite (CHRISTOPHER J. RHODES
46. Hydrothermal synthesis of nickel ferrite powders, their properties and sintering (Miroslaw M Bucko, Krzysztof Haberko)
47. Preparation and magnetic properties of nano size nickel ferrite particles using hydrothermal method (Kamellia Nejati & Rezvanh Zabihi)
48. Synthesis and characterization of nickel ferrite magnetic nanoparticle (P. Sivakumar [a], R. Ramesh [b], A. Ramanand [c], S. Ponnu samy [b], C. Muthamizhchelvan)
49. Current Development in Synthesis and Characterization of Nickel Ferrite Nanoparticle (Nitish Gupta, Pranat Jain, Ravindra Rana , Sanjay Shri vastava)
50. Synthesis *and magnetic characterization of nickel ferrite nanoparticles prepared by co-precipitation route* (K. Maaz [a] [b], S. Karim [a], A. Mumtaz [b], S. K. Hasanain [b], J. Liu· J. L. Duan)
51. Influence of preparation method structural and magnetic properties of nickel ferrite nanoparticles (Binu P. Jacob, Ashok Kumar, R. P. Pant, Sukhvir Singh & E. M. Mohammed)
52. Structural, magnetic, dielectric and optical properties of nickel ferrite nanoparticles synthesized by co-precipitation method(Seema Joshi, Manoj Kumar, Sandeep Chhoker , Geetika Srivastava , Mukesh Jewariya , V. N. Singh)
53. Solvothermal synthesis and magnetic properties of size-controlled nickel ferrite nanoparticles (Jingying Wang· Fenglian Ren, Ran Yi, Aiguo Yan, Gu anzhou Qiu, Xiaohe Liu)
54. Synthesis of nickel ferrite nanoparticles by sol-gel method (Dong-Hwang Chen, Xin-Rong He)
55. Synthesis and characterization of nickel ferrite nanoparticles by sol-gel auto combustion method (R Kesavamoorthi ,A N Vigneshwaran ,Vijayalakshmi Sanyal, C. Ramachandra Raja)
56. Synthesis of ferrite and nickel ferrite nanoparticles using radio-frequency thermal plasma torch (S. Son, M. Taheri, E. Carpenter, V. G. Harris, and M. E. McHenry)
57. nickel ferrite Nanopartical: Synthesis, Modification by Surfactant and Dye Removal Ability (Niyaz Mohammad Mahmoodi)
58. Facile synthesis of magnetic and porous zeolite/SnFe2O4 nanocomposite for cationic and anionic dyes deterioration (Eman F. Aboelfetoh*, Heba G. El-Attar, Ehab A. Okba.