Synthesis of Vanadium Ferrite Nanoparticles By Microwave Assisted Technique

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Abstract
Among the magnetic materials ferrites are of great interest due to its distinctive properties. Ferrites exhibit a spontaneous magnetic moment below a Curie temperature and act as insulators at low temperature. Since they have been used in number of technological applications like microwave devices, magnetic and magneto optic recording, data storage etc. The incredible magnetic, electric and dielectric properties of ferrites make them more attractive and applicable in the current field of science and technology. In the present study vanadium ferrite nanoparticles were synthesized by microwave assisted method. The structural properties of synthesized nanoparticles are carried out from X-ray diffraction technique. The presence of peaks confirms the crystalline nature of the vanadium ferrite nanoparticles. The crystal structure, crystalline size (D), Dislocation density (δ) is determined from XRD data. Also using Williamson-Hall plot micro strain value is calculated. The different molecular vibrations are confirmed through Fourier Transformed Infrared Spectroscopy (FTIR) vibration spectra. From the Ultra Violet Spectroscopic data using the Tauc relation, the energy gap (Eg) at the edge of absorbance band is calculated as 4.28eV for direct and 4.84 eV for indirect transition. The morphological studies are done through Scanning Electron Microscope (SEM). From the SEM micrographs we confirm the presence of spherical nanoparticles and they are arranged uniformly. From VSM analysis, we calculated the saturation magnetization as 20.00 emu, coercivity as 125.96 g, and retentivity is 130.45 emu.

1. Introduction
The history of ferrites and their applications are known for several centuries ago because ferrites are considered as better magnetic materials than pure metals. It consists of metal oxide (MO) and ferric oxide (Fe2O3) as their main constituents. Depending upon the crystal structure, ferrites are classified as Spinel Ferrite, garnet, Ortho - ferrite and hexagonal ferrites. Based on its application it is classified as soft and hard ferrites. The Soft ferrite is a class of magnetic material which can easily change their magnetization behavior. The soft ferrite materials are used in the fields of high frequency inductors, transformer cores, micro wave emitting antennas, transformers etc [1-3]. The Hard ferrites are mostly used in permanent magnets mainly in loud speakers, micro motors. It can be magnetized by large external field. In the past few years the preparation of metallic ions such as (Fe, Zn, Mn, Ni, Co) substituted ferrite nano particles especially in nanoscience and nano technology is due to its physical and chemical behaviors [4-7]. Some metal substituted ferrites have high potential applications, low cost, excellent corrosion resistance, easy manufacture, superior magnetization, small size or large specific surface area. The Super paramagnetic
ferrite nanoparticles have outstanding application in the field of heat transfer, optical filter etc [8-10]. Among varies ferrite materials vanadium ferrite is one of the material shows super paramagnetic properties. These materials can be used in very high frequencies without any limitations as normal requirement of magnetic materials. In particular vanadium ferrite are expected to play an important role in catalysis, sensor and electro chemical capacitors [11-13]. This magnetic materials can be synthesized by various techniques like sol-gel, Microwave assisted method, hydrothermal, solvothermal, co-precipitation method etc [14-15]. The particle Size and surface area of any materials can be controlled by synthesis techniques and reaction parameters. The microwave assisted method is one of the modern methods for synthesis of magnetic nanoparticles. The major advantages of this method is Uniform heating, high purity, fast reaction, high yield, low cost, less hazardous. In the present investigation vanadium ferrite nano particles are synthesized by microwave assisted method. The properties of synthesized vanadium ferrite nanoparticles were characterized by different characterization techniques like XRD, FTIR, UV-Vis, SEM and VSM.

2. Experimental Procedure

All the chemical reagents are used in the synthesis were of analytical grade without further purification. Vanadium pentoxide [V2O5] and ferric nitrate [Fe(NO3)3 9H2O] are taken 1:2 molar ratio. Defined ratio of vanadium pentoxide and ferric nitrate are dissolved pinch by pinch in 100ml of distilled water by using magnetic stirrer with continuous stirrer for half an hour. At the same time sodium hydroxide [NaOH] pellets is dissolved in 20 ml of distilled water and allowed continuous stirring for half an hour. After half an hour sodium hydroxide solution is added drop by drop into initially prepared solution and we get the heterogeneous solution. This solution is continuously stirrer for half an hour. Then the prepared heterogeneous solution is kept in microwave oven at 110ºC for 3 hours. Finally the powder form as yellow colored vanadium ferrite is obtained. The final product was collected and again rinsing with distilled water and ethanol. The prepared vanadium ferrite sample is dried at 100ºC. Then the sample is calcinated at 400ºC for one hour to remove unwanted impurities present in the sample. Further the prepared vanadium ferrite properties are studied from given different characterization techniques.

3. Results and Discussion

The structural properties of synthesized ferrite were studied from powder X-ray diffraction studies. From XRD, the crystalline nature, lattice parameters hence the structures, particle size, strain and dislocation densities of the synthesized Vanadium ferrite are studied. The different molecular vibrations are present in the prepared vanadium ferrite were studied from FTIR spectroscopy. The optical properties of the synthesized vanadium ferrite studied from UV-Visible spectroscopy. From UV-Visible data, absorbance, transmittance, cut-off wavelength and energy band gap (Eg) are also determined. The surface morphological study of the prepared material is studied from scanning electron microscope (SEM). The Magnetic behaviors of the prepared vanadium ferrite were studied from Vibrating sample magnetometer (VSM).

3.1 X-Ray Diffraction Analysis

The crystalline phase of the synthesized sample were identified by using powder X-Ray diffractometer with the wavelength \( \lambda = 1.5406\AA \). A Scanning was performed from 20 degree to 80 degree with a step size of 0.04 degree. The diffraction peaks for prepared ferrite nanopowder is shown in figure 1. The crystalline nature, dislocation density, crystalline size and the lattice parameters are studied from XRD data [16-17]. Crystalline nature of the material confirms by the presence XRD peaks. The lattice parameter values are found to be \( a = 11.54 \), \( b = 3.56 \) & \( c = 4.36 \) with intercepts angles \( \alpha = \gamma = 900\& \beta = 102.50 \) from this we can conclude that the synthesized nanoferrites has monoclinic structure and well indexed with JCPDS card no 00-016-0714.
Fig. 1 XRD Analysis of vanadium ferrite nanoparticles

The average particle size calculated by using below Debye Scherer’s relation [18].

\[ D = \frac{K\lambda}{\beta\cos\theta} \]  \hspace{1cm} (1)

Dislocation density (\(\delta\)) = \frac{1}{D^2} \hspace{1cm} (2)

Where D is the particle size, \(\lambda\) is the wavelength of the X-ray radiation (\(\lambda=0.15406\) nm) for CuK\(\alpha\) and K is usually taken as 0.89 for spherical nanoparticles. The \(\beta\) is the line width at half-maximum height. From the maximum intensity peak (2\(\theta=20.352\)) the Full Width at Half Maximum (FWHM) used for calculating the particle size. The average particle size is found to be 76nm. Dislocation is linear defect in a crystal. The dislocation density is defined as the length of dislocation line per unit volume of the material. The dislocation density \(\delta\) has been calculated as 1.16520 x 1014 m-2.

3.1.1 Williamson-Hall plot

Fig. 2 Williamson -Hall plot for vanadium ferrite

The lattice strain (\(E\)) of the prepared ferrite can be determined using XRD data using Williamson-Hall relation [19].
A graph is plotted between the $\beta \cos \theta$ and $4\sin \theta$. The figure 2 shows the strain value of the vanadium ferrite. From the above graph the calculated lattice Strain ($\varepsilon$) values are found to be 0.9938.

3.2 Fourier Transformation Infrared Spectroscopy

FTIR spectroscopy is a spectroscopic technique which helps us to identify the different functional groups and molecular vibrations. The FTIR spectrum is recorded in the wave number range from 400 – 4000cm$^{-1}$. The FTIR spectra for the synthesized material as shown in below figure 3.

![FTIR Spectra for vanadium ferrite nanoparticles](image)

**Fig. 3 FTIR Spectra for vanadium ferrite nanoparticles**

The C-H Stretching of alkyl group present in 470 cm$^{-1}$ and the presence of peak at 509.21 cm$^{-1}$ show C-Br Stretching vibrations (alkyl). In 675.09 cm$^{-1}$ peak shows Alkyl Stretching vibrations (alkyl). The functional group like aromatic and alkenes are confirmed by presence of peak 837.11 and 960.55 cm$^{-1}$. The C-O stretching vibrations are observed in the wave number of 1107.14cm$^{-1}$.The peaks observed from the region 1633.71 cm$^{-1}$ gives the N-O stretching vibrations [20-21].

3.3 UV-Visible Spectroscopy

![UV-Visible Spectra of Vanadium ferrite](image)

**Fig. 4 UV-Visible Spectra of Vanadium ferrite**
In the present report the optical properties of the vanadium ferrite was studied from the absorption spectrum measured using UV-VIS spectrophotometer in the wavelength range 200 to 1200nm can be done. The absorption spectrum of the synthesized material is shown in the figure 4. The cut off wavelength is found to be 211.31nm. The synthesized material shows a good optical transparency in the entire visible region and near UV region. This shows that the synthesized nanomaterials can be used for manufacturing UV sensors, UV detectors and Opto-electronic devices [22].

3.3. 1 Tauc plot using uv-visible spectroscopic data

The optical band gap of the material can be studied from the Tauc relation [23-24],

\[(\alpha h\nu)^n = A(h\nu - E_g) \]  (3)

Here, h is plank constant, \(\alpha\) is the absorption coefficient, \(E_g\) is optical band gap energy and B is proportionality constant. The value of the exponent denotes the nature of the electronic transition either Direct or Indirect. For direct allowed transition n have integer values and half-integer values of indirect allowed transitions. The graphs are plotted between \((\alpha h\nu)^n\) along y-axis and \(E_g\) along x-axis. The Tauc plot is plotted for both direct band gap and indirect band gap mode. The figure 4a and 4b explains the direct and indirect band energy gap of prepared vanadium ferrite. From the graphs it’s observed that the direct band energy gap is 4.28eV and indirect band energy gap is 4.85eV

![Fig. (4a) direct band gap value of Vanadium ferrite](image)

![Fig. (4b) indirect band gap value of Vanadium ferrite](image)
3.4 Surface Morphological Study

Figure 5 shows the morphological study of vanadium ferrite nanoparticles. The scanning of image carried out under at different nano and micrometrical range 500nm, 1μm and 2μm respectively. The micrograph shows the nanoparticles at 500 nm. The formation of vanadium ferrite particles are dense and are assembled on the surface in 1μm and 2μm magnification [26-28].

![Fig. 5 SEM image for vanadium ferrite](image)

The morphological studies allow us to examine the diameter, shape and length of the nanostructure materials. From the above figure gives the formation vanadium ferrite nano particles have uniform grain size and shapes. The pure vanadium ferrite has closed grains with well defined boundaries. SEM reveals that the particles are spherical in shape. The closed grains are takes a major role in the thermal stability of the prepared material. From figure 5 the average grain size values are found to be 71.14nm, 73.74nm, 74.54nm, 74.74nm.

3.5 Vibrating Sample Magnetometer

The magnetic properties of the prepared material were studied using Vibrating Sample Magnetometer at room temperature. The magnetization curve for the vanadium ferrite at room temperature is as shown in figure 6. The sample presents ferromagnetic behavior with coercivity (Hc) 358.48 G and retentivity (Mr) of 301.41 E-6 emu. The saturation magnetization (Ms) of the sample is found to be 8.748 E-3 emu/g. It is obvious from the magnetization curve that ambient temperature is enough to overcome the coupling force between neighboring atoms. The ferromagnetic behavior is clearly observed in the hysteresis. The low coercive value indicates that the particle can be easily magnetized with low flux loss. In this type of material undergoes the soft magnetic material [29-30]. This significant property allows this type of material can be used in fabrication of magnetic storage media. The non saturation of M-H loop and the
absence of hysteresis remanence and coercivity at room temperature strongly suggest the presence of super paramagnetic behavior.

![VSM for vanadium ferrite nanoparticles](image)

**Fig. 6 VSM for vanadium ferrite nanoparticles**

### 4. Conclusion

Vanadium ferrite nanoparticles were synthesized successfully by microwave irradiation assisted method. The crystalline nature of the nanoparticles is evident from the presence of peaks in the X-Ray diffraction pattern. The synthesized material is grouped under the monoclinic system, matched with JCPDS file with the card no. 00-019-0641. The average crystallite size is found to be 76nm calculated using Debye-Scherer formula. The strain value is calculated as 0.99378 from Williamson Hall plot. The dislocation density is found to be 1.16520X10^14 m^-2. The different functional groups and molecular vibrations are conformed by Fourier transform Infra-red Spectroscopy. The cut off wavelength of sample is determined as 211.31nm by UV-VISIBLE spectroscopic studies. From Tauc plot it is found to be 4.28 eV for direct and 4.85 eV for indirect transition. From the graphs, the most linear response is observed for direct transition. This indicates that synthesized materials can be used for manufacturing Opto-electronic devices. The morphological survey of vanadium ferrite is done by using SEM at 500nm, 1μm and 2μm respectively. From the micrographs we can confirm the presence of nanoparticles and they are uniformly arranged. From VSM analysis, we calculated the saturation magnetization as 8.7480E-3 emu, coercivity as 358.48 g, and retentivity is 301.41 E-6 emu.

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