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$Mn_{0.5}Zn_{0.5}Fe_2O_4$ Ferrite nanoparticles for temperaturesensitive ferrofluid

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ABSTRACT

This paper describes the synthesis and characterization of oleic acid coated $Mn_{0.5}Zn_{0.5}Fe_2O_4$ ferrite nanoparticles. The sample was characterized by XRD, TEM, VSM, and temperature dependent magnetization measurement. The XRD pattern confirms the synthesis of single crystalline phase of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ferrite nanoparticles. The magnetic measurements show superparamagnetic nature of the sample. The saturation magnetization and Curie temperature are 60emu/g and 124°C, respectively. These nanoparticles are used to synthesize temperature sensitive ferrofluid with kerosene as a carrier liquid. An experimental setup is developed to test their possible application in heat transfer enhancement based on thermo-magnetic convection. A considerable enhancement in heat transfer is observed with the application of magnetic field.

KEY WORDS: Mn0.5Zn0.5Fe2O4 ferrite nanoparticles, XRD, VSM, ferrofluid

INTRODUCTION

Dispersion of nanoparticles of different materials in a carrier fluid has been a subject of intensive investigations over decades due to their potential applications in heat transfer and electronic cooling. A possibility to induce and control the heat transfer process and fluid flow by means of an external magnetic field opened a window to a spectrum of promising applications including enhancement of heat transfer for cooling of high power electric transformers, and magnetically controlled heat transfer in energy conversion systems. Mn-Zn ferrites are among the most widely used electromagnetic materials for a broad category of applications such as high-density magnetic recording, transformer cores, multi-layer chip inductor, electromagnetic interference shielding, gas sensor and biological tagging. Recently, they are used to prepare temperature sensitive ferrofluids for their applications in heat transfer enhancement and energy conversion devices.(Azadmanjiri., 2007; Topfer *et al.,* 2011). The composite Mn-Zn ferrites are known to exist as mixed spinel structure wherein the concentration of zinc can modify the magnetic properties of the mixed ferrite. (Gopalan *et al.,* 2009) Thus one can aim at tuning the saturation magnetization value and Curie temperature of Mn-Zn ferrite nanoparticles by replacing the Mn ion with the nonmagnetic Zn ion. This makes them suitable for many technological applications including the synthesis of temperature sensitive ferrofluid. Herein, we report on the synthesis of superparamagnetic $Mn_{0.5}Zn_{0.5}Fe_2O_4$ferrite nanoparticles with special emphasis on controlled size and tunable magnetic properties. A ferrofluid is synthesized using these nanoparticles with kerosene as a carrier liquid and tested its suitability in heat transfer application based on thermo-magnetic convection. (Xuan *et al., 2007)*

EXPERIMENTAL METHODS

$Mn_{0.5}Zn_{0.5}Fe_2O_4$ferrite nanoparticles were synthesized by a facile chemical co-precipitation method. (Shahane *et al.*, 2010; Shahane *et al.*, 2013; Zipare *et al.*, 2015) AR grade $MnCl_2$, $ZnCl_2$ and $FeCl_3$ were used as starting materials. For synthesis, $MnCl_2$ (1M), $ZnCl_2$ (1M) and $FeCl_3$ (2M) were mixed in their stoichiometric ratio and homogenized at room temperature. Oleic acid was used as surfactant to prevent agglomeration of particles. The pH of the solution was adjusted by adding 1M NaOH solution. The mixture was then heated at 80^{o}C for about one hour. The particles were collected at this stage by magnetic decantation method and washed several times with de-ionized water to remove unwanted residual of salts. For the synthesis of ferrofluid, the wet slurry of oleic acid coated Mn-Zn ferrite nanoparticles was dispersed in kerosene using ultrasonic processor UP200S and centrifuged at 12,000 rpm for 15 min to separate out the larger size particles. The X-ray diffraction (XRD) patterns of the samples were recorded on Rigaku make powder X-ray diffractometer (Model- XRG 2KW) in the 2θ range from 20° to 80° using CuKα radiation (λ=1.54059 Å). The microstructure of the sample was analyzed by using transmission electron microscope (JEOL JEM-200CX). The magnetization measurements were carried out by vibrating sample magnetometer VSM Lake Shore Model 7307. The Curie temperature of the samples was determined from the temperature dependence of magnetization measured using a high temperature AC Susceptometer setup (MAGNETA ACSHT-1). An experimental setup is developed to test the application of synthesized ferrofluid in heat transfer enhancement. The variation of the temperature of the object without and with magnetic field was recorded.

RESULTS AND DISCUSSION

Fig 1 shows the XRD patterns of the sample. The ‘d’ values and intensities of observed diffraction peaks match with the single crystalline spinel form of the Mn-Zn ferrites (JCPDS Card No.074-2403).The absence of any additional lines, in any of the XRD patterns, confirms the purity of the $Mn_{0.5}Zn_{0.5}Fe_2O_4$ferrite phase in the sample. X-ray diffraction pattern shows broad peaks indicating ultrafine nature and small crystallite size of the sample. The lattice parameter was calculated and found to be8.4427Å.The crystallite size of each composition was then determined by the Scherrer relation and is of the order of 6.6nm.

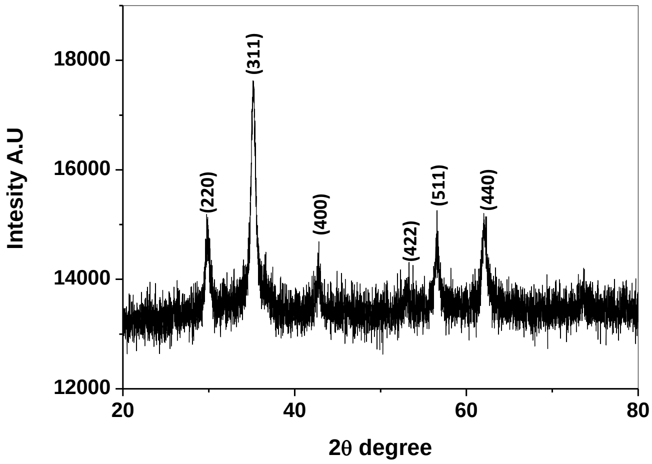


Fig 1: XRD patterns of $Mn_{0.5}Zn_{0.5}Fe_2O_4$nanoparticles

The TEM image of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ferrite nanoparticles, corresponding histograms showing the particle size distribution, the selected area electron diffraction (SAED) patterns and the high resolution TEM (HRTEM) image are shown in Fig 2. The sample contains nanoparticles of nearly spherical in shape with narrow size distribution. The average particle size is of the order of 10 nm. The observed particle sizes are in good agreement with the XRD values. The SAED patterns show diffused rings with small spots arising from Bragg reflection from individual crystallites indicating nanocrystalline nature of sample. The patterns provide the d-spacing consistent with those obtained from XRD studies. The lattice fringes of the core, as observed from the HRTEM image, also confirm the single crystalline nature. These observations imply that high quality uniform phase pure $Mn_{0.5}Zn_{0.5}Fe_2O_4$ferrite nanoparticles are obtained with our facile wet chemical method. This feature is quite important and points out towards the fact that the highly crystalline nature of the samples is favorable for achieving high saturation magnetization. (Shinde *et al., 2013)*

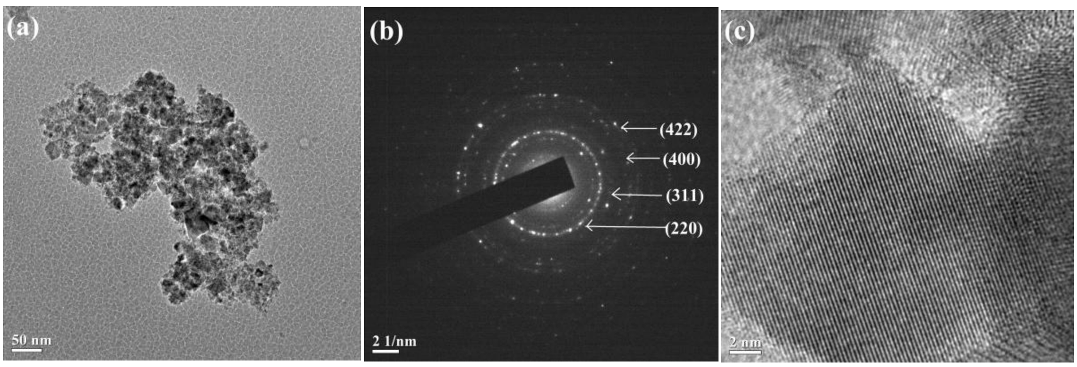
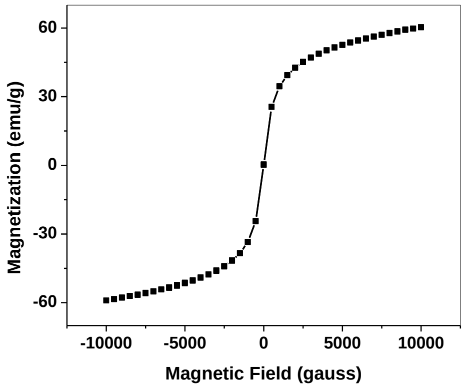


Fig 2: TEM, SAED and HRTEM images of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ ferrite nanoparticles

The magnetic measurements were carried out on these samples at room temperature using vibrating sample magnetometer. Fig 3 shows the variation of magnetization with applied field. The magnetization curve demonstrates superparamagnetic behavior of the sample with negligible remanence and coercivity. The saturation magnetization is 60emu/g. The superparamagnetism of these nanoparticles can be attributed to their small crystallite size, which makes it easier for them to be thermally activated to overcome the magnetic anisotropy. (Iyer *et al.*, 2009; Chand *et al.*, 2011). Zhang *et al.*, (2009) have reported the superparamagnetic particle size limit for Mn-Zn ferrite nanoparticles particle is about 25nm. The size of the synthesized $Mn_{0.5}Zn_{0.5}Fe_2O_4$ferrite particles is about 6.6nm which is well below the critical size limit showing the superparamagnetic behavior of the sample. The Curie temperature of the sample was determined from the temperature dependence of magnetization. Fig 4 displays the temperature dependence of magnetization for $Mn_{0.5}Zn_{0.5}Fe_2O_4$ sample. The Curie temperature was estimated by extrapolating the linear section of the curve andis found to be 124^{o}C.



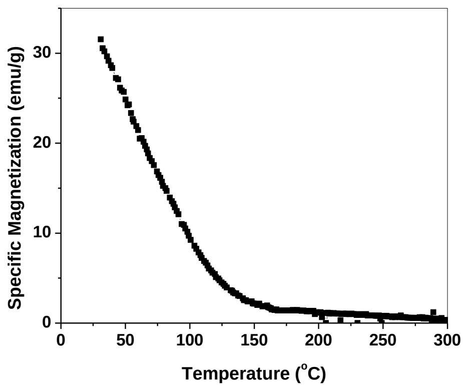


Fig 3: Magnetization curve for Mn-Zn nanoparticles **Fig 4: Curie temperature determination**

Fig 5 shows the prototype experimental setup developed to evaluate the performance of thermomagnetic circulation in the flow loop. The Teflon tubes are used as the primary fluid channel along with a copper heat source tube, and a copper heat rejection section to form a closed flow loop. The heat source section is made of a copper tubing, whose OD is electrically insulated using Teflon tape, and a small electric heater is placed in contact with it. The heat source section is then wrapped with glass wool for thermal isolation. The heat rejection (cooling) section is an annular tube design. It includes a central copper tube which is a part of FF flow loop, surrounded by an outer concentric copper tube which allows cooling water to flow. The cooling water is provided by a constant temperature water circulator system. A solenoid is used as the magnetic field source which is placed close to the heater. This experimental setup allows for the measurement of temperature within the FF flow between the heated section and heat rejection section, as well as the temperature of the heat source surface.

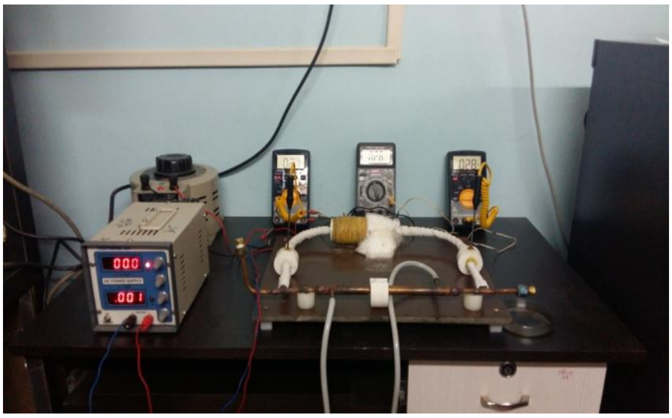


Fig 5: Experimental setup developed to study the thermomagnetic convection

Fig 6 shows the variation of temperature of the system without and with magnetic field. It is observed that a decrease in temperature is observed with the application of magnetic field. The decrease in temperature can be attributed to motion of the ferrofluid due to both temperature and magnetic field gradients, which alter the induced magnetic body force and generate the net flow driving force in the flow direction and parallel to the tube axis; the ferrofluid is attracted toward regions with larger field strength, while near the heat source the fluid temperature approaches the Curie temperature of the FF. In this region the fluid loses its attraction to the magnetic field, and is displaced by colder fluid. This shows the potential of the ferrofluid in heat transfer enhancement based on thermo-magnetic convection.

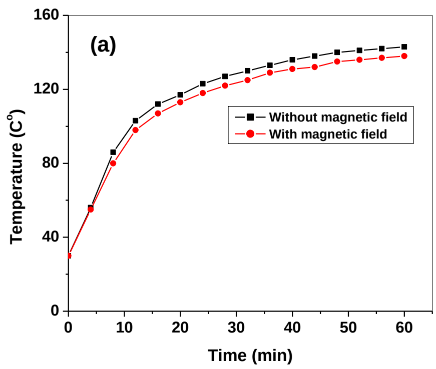


Fig 6: Variation of temperature of the systemwithoutandwith magnetic field

CONCLUSION

A low temperature facile chemical co-precipitation method is used for synthesis of nanocrystalline $Mn_{0.5}Zn_{0.5}Fe_2O_4$ferrite particles. The XRD pattern confirms the synthesis of single crystalline phase of $Mn_{0.5}Zn_{0.5}Fe_2O_4$ ferrite nanoparticles. The magnetic measurements show superparamagnetic nature of the sample. The saturation magnetization and Curie temperature are 60emu/g and 124^{o}C, respectively. These nanoparticles are used to synthesize temperature sensitive ferrofluid with kerosene as a carrier liquid. A considerable enhancement in heat transfer is observed with the application of magnetic field. This demonstrates the potential of synthesized ferrofluid for heat transfer applications.

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