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Study of Effect of Calcinations Temperature on Structural and Magnetic Properties of $MgMnO_{2-\lambda}$ Nanocrystallines

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ABSTRACT

The researchers are interested in studying nanoparticulates as a result of their specific Physical, Chemical and Biological traits. These nanoparticulates are additionally applicable to a wide array of industries, which includes solar cell coatings, transparent sunscreen, self-cleaning windows and scratch-resistant eyewear. Nanoparticulates have significant properties at nano scale because of high surface to volume ratio. The advanced properties of these nanoparticulates increase the interest of today's researchers and scientist. The metal oxide nanoparticulates of MgO have enhanced properties in the field of industries, defense and medical world. Metal oxide nanoparticulates like MgO and MnO_2 are currently the subject of substantial research because of their well-known antibacterial activity against several kinds of bacteria and their advanced uses in the field of storage for hydrogen gas. In the current work, pure MgO and Mn (10%) doped MgO nanoparticulates were prepared via a microwave irradiated chemical co precipitation process . The samples were further calcined at 200^o, 400^o, 600^o and 800^o Celsius for two hours constant heating. XRD, FTIR, FESEM, and VSM examinations have been conducted on the calcined materials. The XRD results revels that nanoparticulates were formed with FCC crystal structures and no phase transformation occurred with addition of Mn dopant concentration in MgO. However, the presence of Mn^{2+} ion in place of Mg^{2+} ion were recognized via broadening of peak resulted by micro strain lattice. The grain size of nanoparticulates were calculated by use of Debye Scherer estimation and crystalline size 29.47nm, 27.68nm, 36.80nm and 46.14 nm pattern shows that crystallite size increases with increase in calcinations temperature. The IR sharp peaks inspected at 409cm^{-1} (pure MgO/800^{O}C), 395cm^{-1}(pure MgO/600^{o}C), 402cm^{-1}(pure MgO/400^{o}C) and 409cm^{-1} (pure MgO/200^{o}C) respectively and for Mn doped MgO nanoparticulates sharp peaks inspected at 450 cm^{-1}(Mn10%/600^{O}C),400cm^{-1}(Mn20%/600^{o}C) respectively and were attributed by O-Mn-O molecule vibration were confirmed the XRD analyses that is Mn^{2+} ion exist at Mg^{2+} lattice site. The electron microscopic image of sample shows that cauliflower leaf like structure was seen with size nanometer in scale. The VSM characteristic of sample concluded that saturation magnetization gradually decreases with increase of calcinations temperature and all substances were ferromagnetic in behavior. The materials have recommended property in applications as electromagnet formation.

Keywords: Magnesium Oxide, XRD, FESEM and VSM.

INTRODUCTION

Nanoparticulates have been widely recognized to possess a significant impact on every aspect of our lives. They have revolutionized applications in the biomedical and pharmaceutical sectors as catalysts and devices for storing energy in the development of ever-smaller and better-performing devices for the electronic appliance industry, in the manufacturing of nanoscale transistors, flexible and transparent electronic gadgets, and in memory chips that increase computer power and storage capacity. Owing to their small size and a variety of physical, chemical and biological properties that are profoundly affected by the surface to volume ratio of the generated nanoparticulates, metal oxide nanoparticulates have been applied to all these diverse applications.

The material sciences, energy, medicine and electronics are a few domains where nanotechnology has the capability to revolutionize totally. The process of reverse engineering, a concept commonly observed in nature is used by nanotechnology for generating newer materials rather than lessening their mass. Through the application of this method goods can be manufactured at the nanoscale and practical application of atoms can then be progressed to much deeper scales. This technology facilitates the more intelligent manufacturing of materials that have improved strength, lowered weight, expanded reactivity, enhanced electrical conductivity and increased sieve-like properties. [1] The researchers have recently shown an intense interest in MgO nanoparticulates owing to their outstanding stability feature, molecular adsorption power, magnetic retention property and superior biocompatibility. These materials have multiple uses in biomedical science as antioxidant substances, antimicrobial agents, anti-diabetic agents and cancer fighting agents. MgO nanoparticulates are also of extreme importance in farming uses since they are harmless to animals as well as plants. Researchers aiming to take advantage of magnesium oxide nanoparticulates for further advantageous industrial and agricultural applications found them to be attractive targets since they have their anti-toxic properties. This work produces an array of magnesium oxide nanoparticulate samples through the use of the chemical co-precipitation methodology. Following that the sample is structurally assessed by techniques which involve FTIR, VSM, FESEM and XRD. [2]

Experimental Details

All the supplies utilized during this investigation study were analytically graded. A sample was initially created using 400 CC of water that had been distilled and an adequate quantity of $Mg(NO_3)$ was added to dissolve in the water with a magnetic stirring device. An 8% NaOH (Sodium Hydroxide) solution was subsequently prepared and slowly added to the Magnesium Nitrate solution while being constantly agitated to obtain white precipitates. The sample that had been prepared was then allowed to mature for a complete day at the room temperature. After the sample matures, it is filtrated and the resultant filtrate is then washed twice with distilled water and Ethanol. Following the accumulation of this white precipitate on the filter paper as filtrate the sample was air dried for 15 minutes at 1500°C in an electric microwave. Researchers subdivided the sample into four portions and continuously calcined them at 200°C, 400°C, 600°C, and 800°C after heating it in a microwave for two hours continuously. The resultant product was a white powder. The final product was then constantly crushed until an extremely fine white powder consisting of pure or undoped MgO came out. Now, the exact same steps are performed again in order to generate a Mn doped sample of Magnesium Oxide nanoparticulates. Four separate 100ml of samples were prepared by dissolving a suitable amount of Magnesium Nitrate at concentrations of 10% Manganese. Next, NaOH was added drop wise, stirring constantly, until white precipitate occurred. After that the entire mixture was left to age for one full day. After that the samples had been separated, cleaned and microwave-dried. After being divided into four equal parts, each sample was heated steadily for two hours at 200°C, 400°C, 600°C and 800°C. The samples were constantly crushed until a fine powdered texture was attained after they were calcined.

RESULTS AND DISCUSSION

Structural Characterization

X-Ray Diffraction Analysis

The phase difference and atomic positions of pure and Mn-doped nanoparticulates were verified by XRD. Pure MgO nanoparticulates calcined at 600°C for two hours are represented in XRD graphs [Figure 1(a)]. The most intense peak for pure MgO is observed at angle 2θ =42.99. This angle indicates the largest periodicity and larger electron density fluctuation if compared to other angles, while the lower intensity peaks show the random order of the crystalline structure.

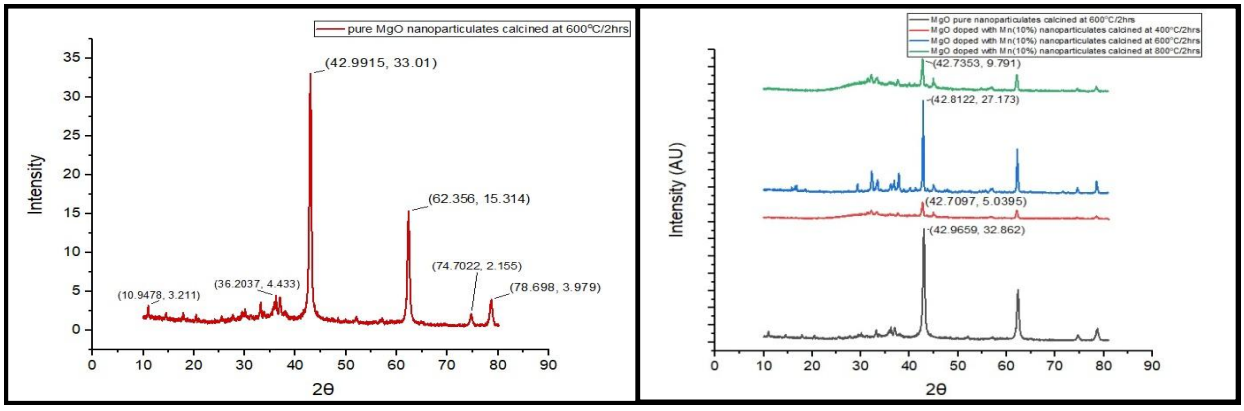

Figure-1: a) The XRD Spectrum of pure MgO nanoparticulates calcined at 600^{O}C/2hrs , b) MgO doped with Mn (10%) nanoparticulates calcined at different temperatures (400^{o}, 600^{o}, & 800^{o} C)

Figure 1(b) shows the XRD pattern of 10% Mn doped MgO nanoparticulates that were calcined at various temperatures (400^o C, 600^o C, and 800^o C) for the same amount of time (2 hours). The results reveal that the intensity of the highest peak was lowest at 400^o C and the intensity of the doped nanoparticulates was roughly comparable when the doped nanoparticulates were calcined at 600^o C. Furthermore, the intensity was discovered to decrease as the calcination temperature grows up to 800^o C.

Table-1: XRD data records of pure MgO nanoparticulates and Mn doped MgO nano-sized particulates calcined at different temperatures, with same concentration (10%) of dopant.

Sr. No.	Title of specimen	Spot of uttermost intense Alp (in degree)	Measurement of FWHM or (β) (in radian)	Grain size (in nm)
1	Pure MgO nanoparticulates 600^{O}C/2hrs	42.962	0.307	29.47nm
1.	MgO /Mn(10%) calcined at 400^{O}C/2hrs	42.709	0.287	27.68nm
2.	MgO /Mn(10%) calcined at 600^{O}C/2hrs	42.812	0.231	36.80nm
3.	MgO /Mn(10%) calcined at 800^{O}C/2hrs	42.735	0.203	46.14nm

Comparing the data to pure MgO nanoparticulates, researcher observed that the atomic radius measure of the Mn doped MgO nanoparticulates increases with increasing temperature but drops with decreasing temperature over calcinations. The Mn-doped MgO nanoparticulates were hydrated at 200°C, signifying that the dehydration energy was substantially lower than the temperature employed for calcinations. [3]

FTIR Spectroscopy Analysis

One of the most used techniques for characterizing both organic and inorganic nanoparticulates is FTIR. These investigations pinpoint the molecule's structure, chemical groups and impurity traces. Most of the time the informations obtained from these studies is extremely specific, allowing for precise differentiation between similar materials. [4] The transmission rate of unadulterated and doped specimens calcined at distinct temperatures (600°C and 800°C) and containing 10% concentration was assessed using an incident radiation wave number that ranged between 500 and 4000 cm^{-1}.

Figure 2(a,b) highlights the graphs obtained from the IR data which display different vibration peaks against the hydroxyl group and O-M-O vibration. [5]

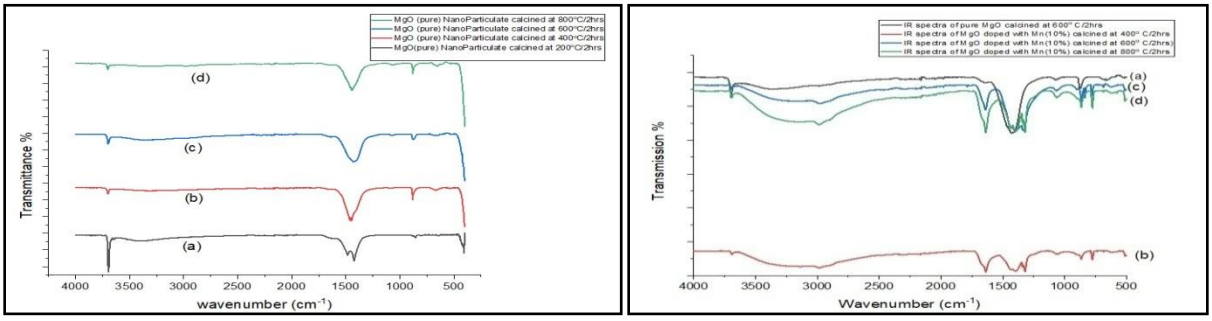

Figure-2:a)FTIR Spectra of pure Magnesium nitrate nanoparticulates calcined at different temperature(200°C, 400°C, 600°C & 800°C) , b) FTIR spectra of pure MgO and Mn doped (10%) MgO nanoparticulates calcined at different temperatures (400^{o}C, 600^{o}C & 800^{o}C)

The infrared radiation light employed for this study is represented by the horizontal axis as its wave number and the vertical line as its corresponding wave number-related transmission %. Solder, broad, minute and other types of peaks were among the multiple kinds observed. The region of a fingerprint below 1500 cm^{-1} is represented by an IR spectrum where the deepest peak uniquely indicates a molecule. The majority of the peaks in the analytical region were also referred to as the diagnostic region or the functional group region. Above 1500 cm^{-1} wave number are linked to specific functional groups.

The signal's shape is important for example, the broadest (U-shaped) and sharpest (V-shaped) signals have different intensities. Signals with the smallest depth are considered weak, while others with the deepest peak are considered dominant.

Polar groups (-OH, -NH, which can form hydrogen bonds), CO (which is polar but gives weak connection within functional group due to electromagnetic interactions) and non-polar functional groups (which typically depict strong peaks) are used to represent broad signals. Any functional group's wave number will drop with rising mass number but as bond strength rises with wave number, so does the wave number when conjugation increases.[6,7]

a) The peak position for pure MgO nanoparticulates were observed at approximate 3699cm^{-1}, 3706cm^{-1}, 3706cm^{-1} and 3713cm^{-1} maybe accredited due to presence of O-H-O vibrations in atmospheric air of sample. Whereas, sharp peaks inspected at 409 cm^{-1} (pure MgO/800^{o}C), 395cm^{-1}(pure MgO/600^{o}C), 402cm^{-1}(pure MgO/400^{o}C) and 409cm^{-1}(pure MgO/200^{o}C) respectively and were attributed by O-Mg-O (metal oxide) vibration. Whereas, 1415cm^{-1} (pure MgO/800^{o}C), 1458cm^{-1}((pure MgO/600^{o}C), 1429cm^{-1}(pure MgO/400^{o}C) and 1444cm^{-1} (pure MgO/200^{o}C) were attributed by CO_2, NO_2 molecule vibration.
b) Peaks position of various kinds such as solder, minute, broad band ,sharp were observed in spectroscopy results. The observed peak position for Mn doped MgO nanoparticulates was at approximate 3711cm^{-1}(Mn 0% calcined at 600^{o}C/2 hours) ,3697 cm^{-1}(Mn 10% calcined at 400^{o}C/2 hours), 3707 cm^{-1}(Mn 10% calcined at 600^{o}C/2 hours) and 3694 cm^{-1}(Mn 10% calcined at 800^{o}C/2 hours) were accredited by presence of O-H-O vibrations (water) in atmosphere of sample. Whereas, sharp peaks observed at 763 cm^{-1}, 779cm^{-1}, 843cm^{-1} and 869cm^{-1} respectively were attributed by O-Mn-O molecule vibration. Peaks observed at 1407 cm^{-1}, 1643 cm^{-1}, 1597 cm^{-1}, 1642 cm^{-1} respectively were attributed by O-Mg-O molecule vibration and therefore, confirmed the presence of MnO in MgO nanoparticulates.

Field Emission Scanning Electron Microscopy (FESEM)

The most advanced technique called FESEM is utilized to image the microstructure of nanoparticulates. Due to the tendency of gas molecules to interfere with the electron beam, the secondary and backscattered electrons that are generated were used for imaging, this examination is often carried out in a vacuum. This characterization method yields higher resolution, clearer, less electro statically distorted images. [8, 9]

The electron microscope images of the pure MgO nanoparticulates that were calcined at 600°C for two hours (Figure 3(a)) and the Mn (10%) doped $MgNO_3$ nanoparticulates that were calcined at 600°C for two hours were almost identical to the typical electron microscope images of sample scanning. Figure 3(b) shows a micrograph of 10% Mn-doped MgO nanoparticulates that were calcined at 600^{o}C for two hours.

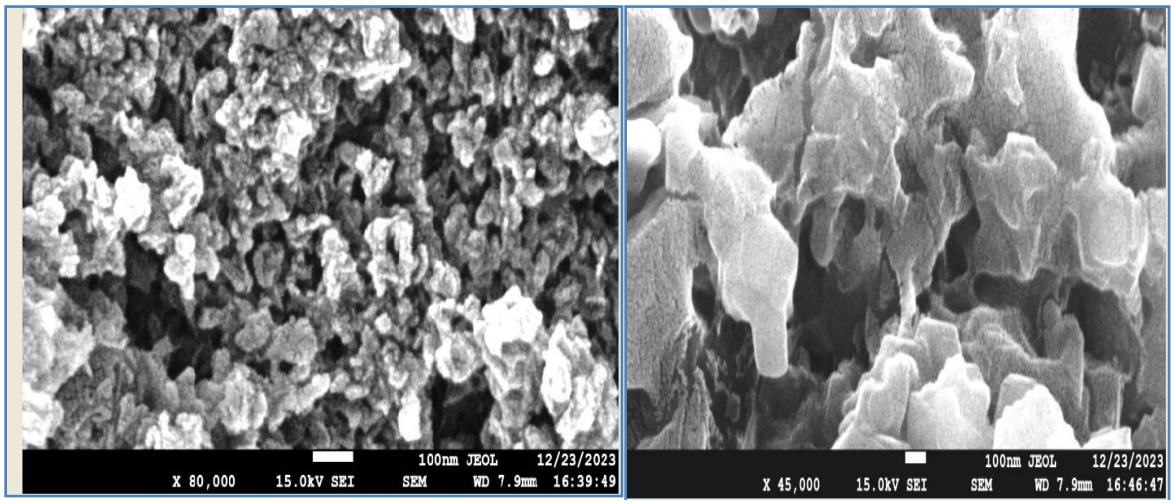

Figure-3(a) FESEM images of pure MgO calcined at 600^{o}C/2hrs (b) Mn (10%) doped MgO nanoparticulates calcined at 600^{o}C/2hrs

FESEM images revealed that the cauliflowers like leaf structures were obtained with in nanoscale parameters.

VSM (Vibrating Sample Magnetometers)

The magnetic characteristics of ferromagnetic, anti-ferromagnetic, paramagnetic and diamagnetic materials can all be found using VSM. With this method of characterization the strength of the magnetic field generated by the sample and its susceptibility to an external magnetic field are measured. [10]

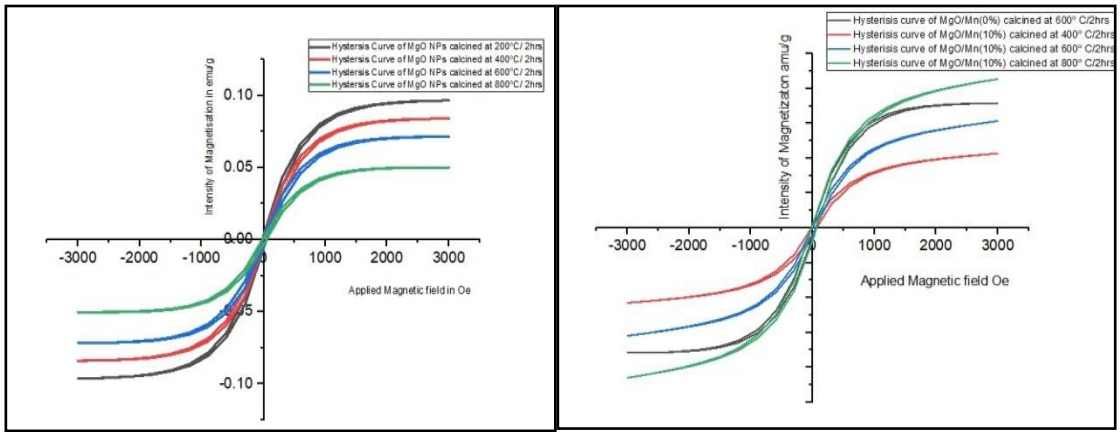

Figure-4(a) VSM pattern of pure MgO nanoparticulates calcined at various temperatures(400^0 C , 600^0 C & 800^0 C for 2 hrs) , (b)VSM pattern of Mn (10%) doped MgO nanoparticulates calcined at various temperatures (400^0 C , 600^0 C, 800^0 C for 2 hrs)

A close look at the graph above reveals that all of the compounds were ferromagnetic in behavior and that the saturation magnetism steadily decreased as the calcination temperature increased. The materials are advised for use as electromagnet materials.[11]

CONCLUSIONS

The synthesis of Mn-doped MgO nanoparticulates with a 10% concentration was accomplished by the use of microwave-treated co-precipitation synthesis procedures. The nanocrystalline grains that formed were 29.47, 27.68, 36.80 and 46.14 nm in size. The pattern indicates that the size of the crystallites grows as the temperature of calcination rises. Sharp peaks in the infrared spectrum were observed at 409 cm^{-1} (pure MgO/800^{o}C), 395 cm^{-1} (pure MgO/600^{o}C), 402 cm^{-1} (pure MgO/400^{o}C) and 409 cm^{-1} (pure MgO/200^{o}C) respectively. Similarly for Mn doped MgO nanoparticulates, sharp peaks were observed at 450 cm^{-1} (Mn 10 %/600^{o}C), and 400 cm^{-1} (Mn 20%/600^{o}C) respectively. These were attributed to O-Mn-O molecule vibration, which was confirmed by XRD weitage indicating the presence of Mn^{2+} ion at the Mg^{2+} lattice site. The sample's electron microscopy image reveals the presence of a nanometer-scale structure resembling a cauliflower leaf. The sample's VSM characteristic revealed that all of the components exhibited ferromagnetic behavior and that saturation magnetization steadily decreased as the calcination temperature increased. The materials are advised for use as electromagnet materials.

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