XRD, TEM AND VSM STUDIES OF GREEN SYNTHESISED MAGNESIUM OXIDE NANOPARTICLES AS PHOTOCATALYST FOR THE DEGRADATION OF RHODAMINE B-DYE AND METHYLENE BLUE DYE

A.Suba[1,4], P.Selvarajan[2,4*], J. Jebaraj Devadasan[3,4]

[1]Research scholar, Reg.No. 19132152132001, Department of Physics, Pope's College, Sawyerpuram, Thoothukudi-628251, Tamilnadu, India.
[2]Department of Physics, Aditanar College of arts and science, Tiruchendur, Thoothukudi-628216, Tamilnadu, India
[3] PG & Research Department of Physics, Pope's College, Sawyerpuram, Thoothukudi-628251,Tamilnadu, India.
[4]Manonmaniam Sundaranar University, Abishekapatti, Tirunelveli-627012, Tamilnadu, India.
***Corresponding author:** pselvarajanphy@yahoo.co.in

Abstract

The present work is focused on synthesis of MgO nanoparticles by using green method. MgO nanoparticles were prepared by using black grapes. In this green synthesis method, Magnesium nitrate is used as a precursor. The synthesised MgO nanopowder is analysed by Powder XRD, TEM with SAED, Vibrational Sample Magnetometer (VSM) and photocatalytic studies. Powder XRD studies shows that the average particle size was 11.18 nm. The TEM studies confirm the morphology, crystallinity, and particle size of the synthesised MgO nanoparticles. The magnetic properties of MgO nanoparticle is analysed by using vibrational sample magnetometer. The photocatalytic degradation of Rhodamine B-dye is evaluated using the as-prepared MgO nanoparticles under visible light.

Key words: Nanomaterial; MgO; Green synthesis; XRD; TEM; SAED

1. Introduction

The technology of twenty first century requires the miniaturization of the electronic devices into nanometer scale while their ultimate performance is significantly improved. This miniaturization of the electronic devices raises many concerns regarding the advanced materials for realizing precise functionality and selectivity. The different morphologies of nanostructured materials related to the new stem of advanced material science research are capturing a great deal of consideration because of their probable applications in the field such as electronic devices, optical devices, photo-catalysis application, ceramic. The technology of twenty first century requires the miniaturization of the electronic devices into nanometer scale while their ultimate performance is significantly improved. This miniaturization of the electronic devices raises many concerns regarding the advanced materials for realizing precise functionality and selectivity. The different morphologies of nanostructured materials related to the new stem of advanced nanocomposites, ferroelectric and ferromagnetic data storage devices and antimicrobial biological application [1-6]. Green synthesis of nanoparticles is less expensive, free of chemical contaminants and this method belongs to the category of bottom-up approach. In this method, the reducing agent used is an extract of a natural product and it can be used to stabilize the nanoparticles.

Grape (Vitis labrusca L.) juices are consumed worldwide and are considered an important source of phytochemicals, such as anthocyanins, biogenic amines, and polyphenolic compounds [7, 8]. From the literature survey, it is found that the researchers have used many biological agents except grape juice for synthesizing the MgO nanomaterial.

Dyes are extensively used in the textile industry. They are the copious source of coloured organics emanating as a waste from the textile dyeing process. Due to the high concentration of organics in the effluents and the higher stability of modern synthetic dyes, the conventional biological treatment methods are ineffective for the complete colour removal and degradation of organics and dyes [9]. Other conventional methods of colour removal from an aqueous medium include techniques like coagulation, filtration, adsorption by activated carbon and treatment with ozone [10]. Each method has its own advantages and disadvantages. Balakrishnan *et al* have prepared Microstructure, optical and photocatalytic properties of MgO nanoparticles. This paper explained the photocatalytic degradation of methylene blue dye is evaluated using the as-prepared MgO nanoparticles under UV light [11]. Patil *et al* have explained Vanadium-doped magnesium oxide nanoparticles formation in presence of ionic liquids and their use in photocatalytic degradation of methylene blue [12]. Ikram *et al* have prepared Graphene Oxide-Doped MgO Nanostructures for Highly Efficient Dye Degradation and Bactericidal Action [13]. The aim of the paper is to report the results obtained through green synthesis, Photodegradation action and magnetisation behaviour of MgO nanomaterial.

2. Experimental

2.1. Materials Used

Magnesium nitrate, purchased from Merck India, is used as a precursor. Grapes were bought from a fruit stall in Tamil Nadu. Grape juice was collected after the grapes had been washed and air dried. The solvent was double distilled water.

2.2. Synthesis of MgO nanoparticles

MgO nanoparticles were synthesised using the green co-precipitation route, which is a simple and environmentally friendly method. MgO nanoparticles were prepared using black grapes. The grape juice was mixed with a 0.5 M aqueous magnesium nitrate solution. This mixture was continuous stirring for 3 hours at 30º C. Precipitate of brown colour was formed and it was filtered out. Magnesium hydroxide is found in this dried powder. Calcination was performed with a muffle furnace at 400ºC for 18 hours. Following calcination, magnesium hydroxide was transformed into magnesium oxide.

3. Results and Discussion

3.1 Powder XRD analysis

X-ray diffraction (XRD) is useful for finding the crystal structure of the material. The XRD data is analyzed for the reflection angle to calculate the inter-atomic spacing or d spacing. The prepared sample of MgO nanoparticles has been subjected to powder X-ray diffraction (PXRD) analysis to confirm the lattice constants of the sample and to confirm the reflection planes. The sample was scanned over the required range for 2θ values (10°– 80°). X-ray diffraction data gives the angle of scattering (2θ) and the corresponding intensities of diffracted beam for each reflection [14]. The recorded PXRD pattern of the sample is depicted in figure 1. The d-spacings corresponding to different peak positions were calculated using the Bragg's law $2d \sin\theta = n \lambda$ where d is the interplanar spacing, θ is the Bragg's angle, n is the order of diffraction and λ is the wavelength of X-rays. The Bragg's diffraction peaks were indexed for the cubic crystal system. The broad peaks of the XRD pattern indicate that the prepared sample is nano sample. The diffraction peaks of sample are indexed as (111), (200),(220),(311) and (222), which is matched with JCPDS No. (No.04-005-4664).The sample exhibits the reflection corresponding to the cubic MgO phase. The obtained value of lattice parameter for MgO nanoparticle is a = 4.220 Å.

On the basis of Powder XRD analysis, MgO sample has the cubic structure. The particle size has been calculated by Debye-Scherrer's formula Particle size $D = K\lambda/\beta\cos\theta$, where K is a constant and it is equal to 0.9, λ is wavelength of X-rays (1.5406 Å) and β is full width at half maximum. The crystelline size of Magnesium Oxide Nanoparticles is 11nm.

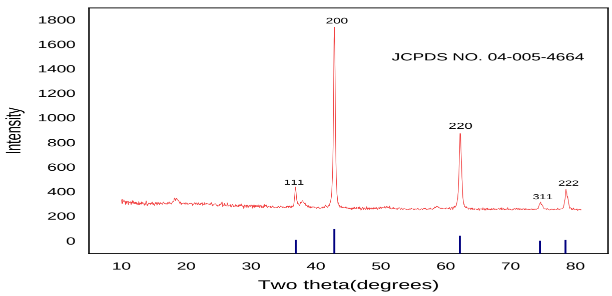

Fig.1: Powder XRD pattern of MgO nanoparticles

Table1. Powder XRD data for MgO Nanoparticles

Two-theta (degrees)	d-spacing (A°)	Relative Intensity (%)	hkl
36.86	1.89	30.55	111
42.82	1.4	18.35	200
62.17	2.3	100	220
74.51	1.79	1.37	311
78.44	0.77	43.06	222

3.2 TEM studies

The morphology and particle size of MgO nanomaterial were investigated using a transmission electron microscope (TEM) at Cochin University. Figure 2 shows a TEM image of the sample. The image shows that the MgO sample has nanostructures with average particle sizes ranging from 5 to 25 nm. In the photograph of the TEM image shows the spherical shape of the nanoparticles. The selected area electron diffraction (SAED) pattern on a single MgO nanoparticle is shown in the figure 3 and it displays a set of diffraction spots. The spots on the pattern indicate that MgO nanoparticles form the monocrystalline nature of the sample [15]. The selected area electron diffraction pattern is coinciding with d-spacing of powder XRD pattern. These two results are displaying the same result. Thus Magnesium oxide nanoparticle is simple cubic structure.

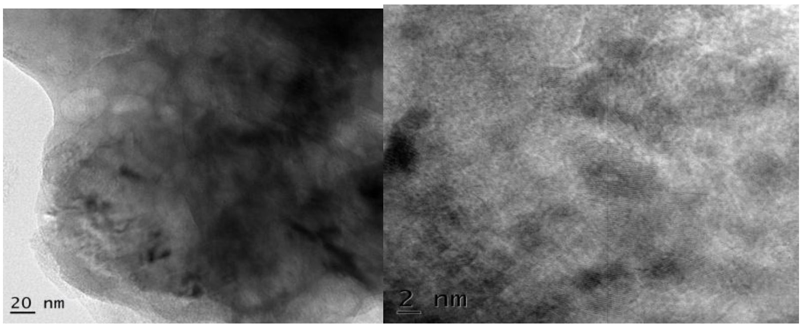

Fig.2: TEM image of MgO nanomaterial

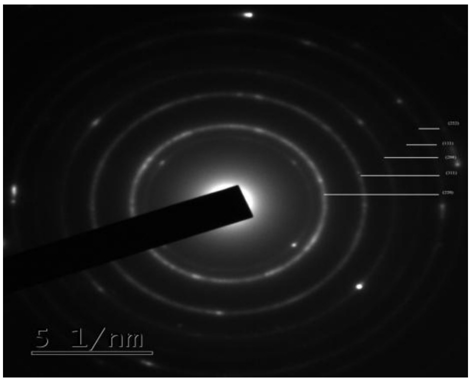

Fig.3: SAED pattern of MgO nanoparticle

3.3 Vibrating Sample Magnetometer (VSM)

A vibrating sample magnetometer (VSM) system is used to measure the magnetic properties of materials. The vibrating component causes a change in the magnetic field of the sample, which generates an electrical field in a coil based on Faraday's Law of Induction. If the sample is placed within a uniform magnetic field H, a magnetization M will be induced in the sample. In a VSM, the sample is placed within suitably placed sensing coils, also held at the desired angle. This study is taken out from SAIF at the Indian Institute of Technology, Chennai. Model: Lakeshore 7410S/ MH-1-5T. The magnetic behaviour of magnesium oxide nanoparticles depends on the particle's surface, cation distribution, spin order etc. In order to study the magnetic behaviour of the synthesized MgO nanomaterial the dependence of magnetization on the applied magnetic field has been measured at room temperature using VSM in the field range of -15000 to +15000 G. The magnetization curve of MgO nanoparticles is shown in figure 4. The curve exhibits the diamagnetic nature of MgO nanoparticles.

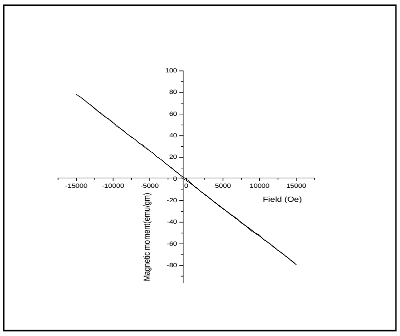

Fig. 4: Magnetization curve of magnesium Oxide nanoparticle

From the figure, the graph is plotted between magnetic field and magnetization. This graph is identified the material is diamagnetism. Because, the behaviour of the sample whose atoms spins are oriented parallel but inversely to the external field. Its magnetization is weak and in the opposite direction to the field and therefore its susceptibility, χ is negative and of very low values [16].

3.4 Photocatalytic Activity

The photocatalytic behaviour of the prepared MgO was tested through the degradation of Rhodamine B Dye and Methylene Blue Dye.

The photocatalytic activity of the rhodamine B dye was evaluated by the degradation efficiency of rhodamine B under visible light (Philips, 40 W). Prior to the photocatalytic reaction, the mixed solution was stirred magnetically in a dark room for 30 min to make the catalyst uniformly dispersed. It also ensured that the dye molecules reached an adsorption/desorption equilibrium on the catalyst surface. The mixture was subjected to a photocatalytic reaction under visible light. The concentration of the RhB dye was measured by UV-Vis spectrophotometer with a test wavelength of 554 nm. Rhodamine B was prepared at 10 µM concentration. Photocatalyst concentration (2.5 mg, 5 mg, 10 mg and 25 mg), to the blank photocatalyst was not added, it was incubated for 2 h and photocatalytic activity (%) determined.

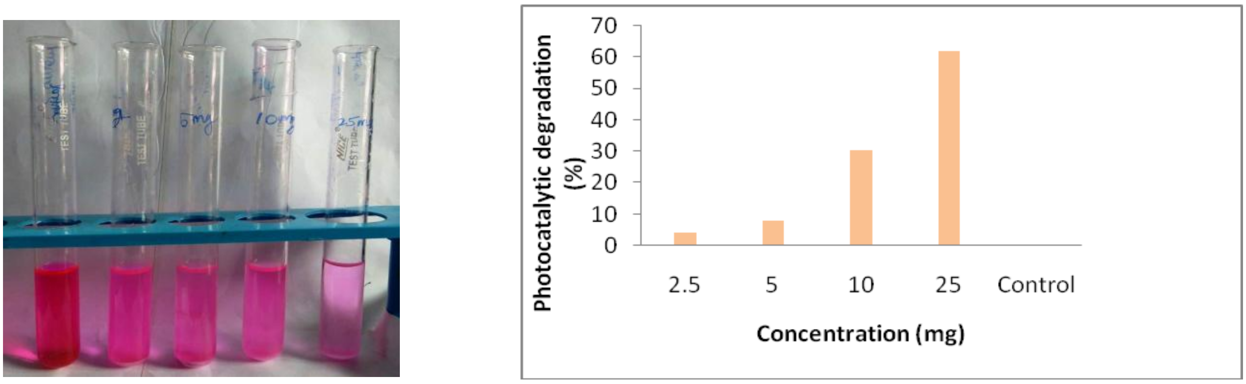

Fig. 5: Photo degradation effect of Rhodamine B Dye

Table 2: Degradation efficiency chart of MgO Nanoparticle

Concentration(mg)	Dye degradation (%)
2.5	4.2
5	7.8
10	30.2
25	61.7
Control	0

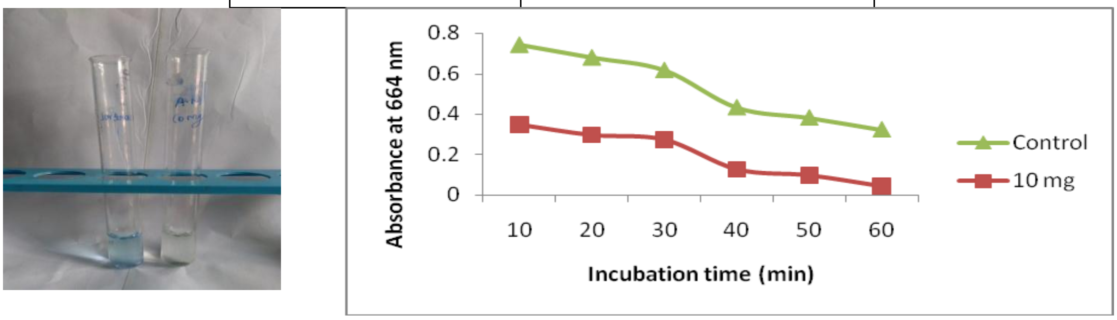

Fig.6. Photo degradation effect of methylene blue dye

From the observation, green synthesised Magnesium Oxide Nanoparticles had high performance towards the degradation of Rhodamine B dye. Figure 6 demonstrates that Methylene Blue Dye degrades quickly even at very low concentrations (10mg). A UV-Vis spectrophotometer was used to measure the Methylene Blue Dye concentration using a test wavelength of 664 nm. It was created with a 10 mg dose of methylene blue dye. When compared to Rhodamine B Dye photo degradation, this approach is distinct. RhB dye photodegradation from these two concentrations proceeds much more slowly than methylene blue dye photodegradation. Mao-Yuan, C. and Heh-Nan, L. (2015) Enhanced photocatalysis of ZnO.

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4. Conclusion

Magnesium Oxide nanoparticles are widely being used in various domains such as automotive, aircraft, agriculture, environmental and biomedical, etc., and its use is spreading to almost every field due to its unique properties. Nanoparticles are showing promising application since near past and are to capture almost every field in near future because of their unbeatable properties. From Powder XRD study, an average particle size of the sample was found to be 11 nm. Transmission of electron microscope is used to identify the shape and size of the nanoparticles. The selected area electron diffractions show the uniform pattern of diffraction spot. The image is showing the nature of crystal structure of nanoparticle is simple cubic structure. This result is also coinciding with the Powder XRD analysis. Magnetization Properties are analysed with the help of VSM. The photocatalytic behaviour of the prepared MgO was tested through the degradation of Rhodamine B Dye.

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