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Research Article

Sustainable MgO Nanoparticle Synthesis for Photocatalytic Applications in Dye Degradation

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Abstract

The dye degradation is a crucial area of concern for environmental research due to the extensive use of synthetic dyes in industries such as textiles and cosmetics. Dyes often exhibit high stability and resistance to conventional wastewater treatment. The harmful dyes block sunlight, disrupt photosynthesis, and deplete oxygen, thereby causing harmful effects on both human and aquatic organisms. Photocatalytic degradation driven by reactive oxygen species offers a sustainable solution by converting dyes into non-toxic by-products. In the present study, the synthesis of MgO nanoparticles using a Hog Plum leaf extract was carried out. The bio-synthesised nanoparticles were characterised using UV-Visible spectroscopy, Fourier Transform Infrared spectrophotometer, X-ray diffractometer, Scanning Electron Microscope and Energy Dispersive X-ray. The synthesised MgO nanoparticles were confirmed by UV-Visible spectroscopy. The nanoparticles exhibited good photocatalytic degradation, which was enhanced with the nanoparticles loading. The nanoparticles with 100mg loading showed 60% degradation. The degradation efficiency reduced with the increase in concentration of dye, and the optimum pH for maximum degradation efficiency was observed to be 12.

Keywords: MgO nanoparticles, Dye degradation, green synthesis, Hog plum,

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1. Introduction

Organic dyes are widely used in the textile industries, which are carcinogenic and non-biodegradable. The recent developments in the textile industries are associated with environmental contamination, mainly caused by effluents released into aquatic bodies. Nearly 12% of the discarded waste comprises textile dyes, which are toxic and, when released into water bodies, will increase the chemical oxygen demand, leading to a toxic environment for aquatic organisms and humans [1-4]. To overcome these environmental issues removal of toxic dyes is important. Methods like osmosis, adsorption, and photocatalytic degradation are employed in the removal of harmful dyes from aquatic bodies [5,6]. Heterogeneous photocatalysis is an advanced method used in the decolourisation of dye into nontoxic byproducts like water and carbon dioxide [7-9]. Methylene Blue is an organic cationic dye used in major textile industries. It causes harmful effects like nausea, vomiting, irritation, anaemia, etc, in humans [10].

Nanoparticles are frequently employed in the removal of dye.

Researchers are driven towards the approach of green synthesis for the synthesis of nanoparticles. Green synthesis is a broadly used method as an alternative to chemical synthesis of nanoparticles, as it is non-toxic, safe, clean, and eco-friendly. Leaves, fruits, bark, roots, stem, and some parts of plants are utilised in the synthesis of metal oxides [11-12]. Many biosynthetic metal oxides, like MgO, NiO, CaO, CoO, CeO_2, and ZnO, are employed in the degradation of dye. [11,13-14].

Magnesium oxide nanoparticles (MgO-NPs) are a type of adsorbent that is cheap, safe, and eco-friendly. They have high porosity, a larger crystalline size associated with a larger surface area, which makes them suitable for various applications. MgO nanoparticles have been synthesised using a variety of physical and chemical processes [15,16]. Numerous synthetic methods like the wet chemical method [17], sol-gel method [18], and precipitation method [19] have been reported for the synthesis of MgO nanoparticles. Likewise, many biogenic methods have also been reported. Magnesium Oxide nanoparticles were synthesised using Musa acuminata Cavendish (banana leaf) [1], Syzygium cumini fruit, Citrullus lanatus fruit, and aloe vera leaves were utilised as a fuel in the synthesis of pure MgO nanoparticles, which were further utilised in the degradation of direct green dye [3]. MgO nanoparticles was synthesized by fungal method by harnessing the metabolites which were secreted by Aspergillus niger strain F1 and was used in the degradation of real textile wastewater [20].

The present study reports the biosynthesis of pure MgO nanoparticles by Hog Plum leaf extract. The synthesised magnesium oxide nanoparticles were characterised by several analytical techniques like UV-Visible, XRD, FT-IR, SEM and EDX. Furthermore, the MgO nanoparticles were employed in the degradation of methylene blue dye.

2. Materials and Methods

2.1. Sample Collection

For the present research work, the reagents and chemicals used were of analytical grade. The magnesium nitrate hexahydrate $Mg(NO_3)_2.6H_2O$ and methylene blue dye were purchased from Sigma-Aldrich Chemical Pvt. Ltd. and used as received without any further purification. Hog plum leaves were collected from Mysore, Karnataka state, India.

2.2. Preparation of Hog Plum Leaf Extract

Fresh Hog plum leaves were collected, washed several times and dried in sunlight. The dried leaves were finely powdered mechanically using a motor and pestle. 5g of powdered leaves were weighed and mixed with 100ml distilled water, stirred for an hour at 80 ºC, cooled to room temperature and filtered. The collected extract was stored for further synthesis.

2.3. Synthesis of MgO Nanoparticles (MgO NPs)

MgO nanoparticles were successfully synthesised by the biogenic method. 1mg of $MgNO_3.6H_2O$ was weighed and dissolved in 90ml distilled water. To this, 10ml of Hog plum leaf extract was added and stirred for 30 minutes. Further, the mixture was heated on a hot plate for 90 minutes at 80 ºC. The obtained MgO nanoparticles were filtered, dried in an oven at 60 ºC and later calcinated for an hour at 500 ºC.

2.4. Characterization

The bio-synthesised MgO nanoparticles were characterised by several spectroscopic methods, including scanning electron microscopy, Fourier-transform infrared spectroscopy, X-ray diffraction, and a UV-visible spectrophotometer. The structural analysis was carried out using a Bruker D8 Advance X-ray diffractometer using Cu Kα radiation (λ = 1.5418Å). The surface and elemental analysis were carried out by SEM and EDAX. An FTIR spectrophotometer (Shimadzu 8400S) was used to analyse functional groups in the wavelength range of 400–4000 nm. The optical properties were determined by UV Shimadzu 1900 [21].

2.5. Photocatalytic Degradation

The biosynthesised MgO nanoparticles catalyst was used in the photochemical degradation of methylene blue dye. A known amount of MgO nanoparticles was taken in 20 ml of aqueous

solution of methylene blue, stirred continuously under UV light (40 W) until decolourisation of methylene blue took place. The supernatant was examined using a UV spectrophotometer at regular intervals of time. The degradation efficiency of methylene blue was calculated using Equation 1:

$$\%\, efficiency\ (\eta) = \frac{C_0 - C}{C_0} \times 100 \qquad \ldots (1)$$

Where 'C_0' and 'C' are the initial and final concentrations of methylene blue [22].

3. Results and Discussion

3.1. UV-Visible Analysis

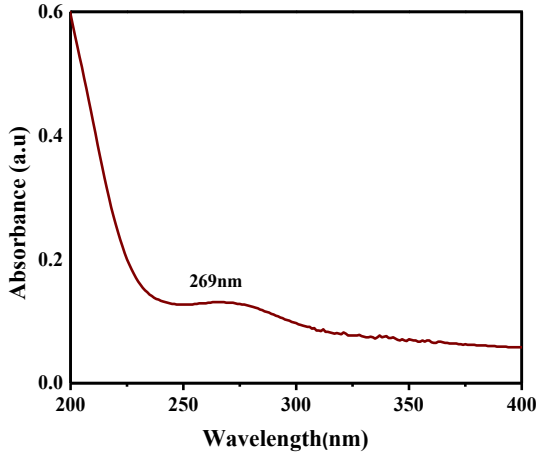


Fig. 1: UV-visible spectrum of synthesised MgO nanoparticle

The bio-synthesised MgO nanoparticles formation was confirmed by using a UV-Visible spectroscopy as shown in Fig. 1 The strong absorption peak was observed at 269 nm, attributed to the formation of MgO nanoparticles [23, 24].

The Tauc's plot of (αhv)2 v/s photon energy (E, eV) gives the energy gap of the MgO nanoparticles, where α is absorbance and h is Planck's constant, v is the velocity of light. From Tauc's plot as shown in Fig. 2, the band gap of biosynthesised MgO nanoparticles was found to be 4 eV.

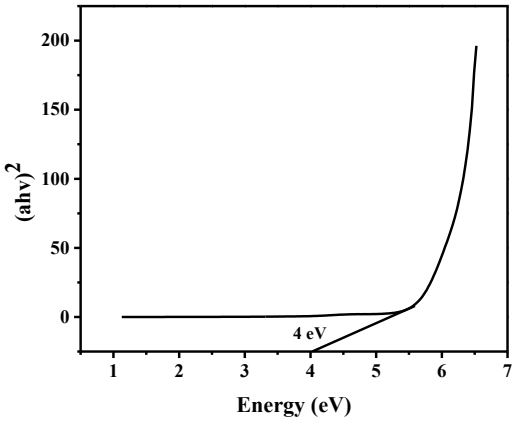


Fig. 2: Tauc's Plot of synthesised MgO nanoparticle.

3.2. X-Ray Diffraction Analysis

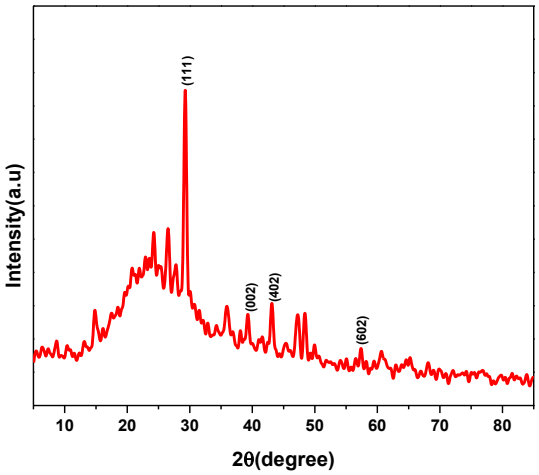


Fig. 3: XRD spectra of synthesised MgO nanoparticles.

The crystalline size of the synthesised MgO nanoparticles was investigated by XRD analysis [25]. Fig. 3 depicts the XRD spectrum of MgO nanoparticles. The peaks corresponding to 2θ values 29.1˚, 39.4˚, 43.4˚, 57 with crystal planes of (111), (002), (402), (602) respectively are due to MgO nanoparticles [26,27]. The synthesised MgO nanoparticles were found to have a crystalline nature and a cubic structure based on the XRD pattern [28]. The average crystalline size is calculated using the Debye-Scherrer equation as shown in Eq.2:

$$D = \left(\frac{k\lambda}{\beta\, cos\theta}\right) \qquad \ldots(2)$$

Here, D is the crystallite size (nm), k is the Scherrer constant whose value is 0.9, λ is the wavelength of the X-ray used (=0.1546 nm), β is the full-width at half-maximum (FWHM) of the diffraction peak (in radians), and θ is the Bragg angle (in degrees) [29, 30].

Additional parameters like micro-strain and dislocation constant are calculated using equations 3 and 4, respectively.

$$\varepsilon = \frac{\beta Cos\,\theta}{4} \quad \ldots(3)$$

$$\delta = \frac{1}{D^2} \quad \ldots(4)$$

The calculated values of biosynthesised MgO nanoparticles are given in Table 1.

Table 1: Calculated Data

Crystallite Size (nm)	Micro strain (ε)	Dislocation constant (δ)
12.68	0.0234	1.469

3.3. Fourier Transform Infrared Analysis

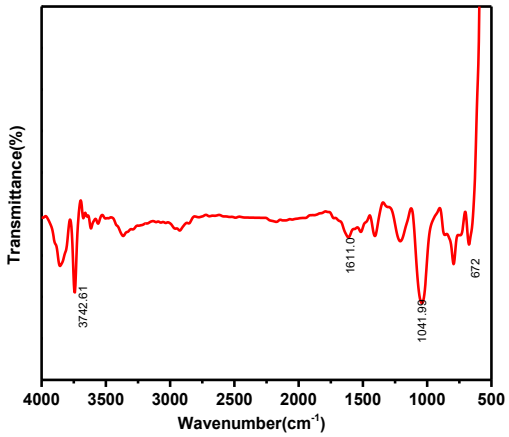


Fig. 4: FTIR spectrum of synthesised MgO nanoparticles.

The spectral behaviour of the bio-synthesized MgO nanoparticles were analysed using FT-IR spectroscopy as shown in Fig. 4 From the FTIR spectrum, the absorption peak obtained at 3748.61 cm^{-1}, 1611.0 cm^{-1}, and 1041.99 cm^{-1} corresponds to O-H, C=O and C=C stretching confirming the presence of biomolecules on the surface of the nanoparticles, which acts as capping agent to prevent the agglomeration of nanoparticles. The absorption peaks appeared at 672 nm due to Mg-O stretching vibrations. Thus, the FTIR spectrum of confirms the formation of the MgO nanoparticles successfully [22, 31-33].

3.4. SEM and EDX Spectra

SEM was employed in the analysis of the surface morphology of the synthesised Magnesium oxide nanoparticles. SEM images of MgO nanoparticles are depicted in Fig.5 [34]. From SEM image confirms that the MgO nanoparticles have an irregular shape. EDAX analysis was used to analyse the elemental composition of MgO nanoparticles, indicating the purity of MgO nanoparticles mediated by plants [35]. Furthermore, the EDX profile contains Mg and O. The characteristic peaks confirm the purity of magnesium oxide nanoparticles [36,37].

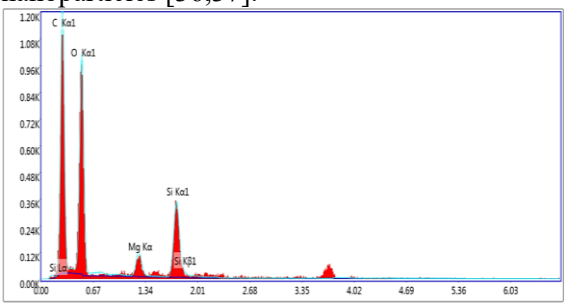


Fig. 5: SEM and EDX spectra of synthesised MgO nanoparticles.

4. Photocatalytic dye Degradation Studies

Methylene blue is an organic dye that exhibits blue colour in its oxidised form and remains colourless in its reduced form. Bio-synthesised MgO nanoparticles are employed in the oxidation of blue colored Methylene Blue. On oxidation, the blue colour of MB dyes fades away by splitting up into simpler molecules like carbon dioxide and water molecules. The synthesised MgO nanoparticle will provide an active site for the degradation of dye. The species responsible for the degradation are OH radical and protons. The UV irradiation time was about 90 minutes with a time interval of 15 minutes. The efficiency of degradation is calculated using equation 1 [38-40].

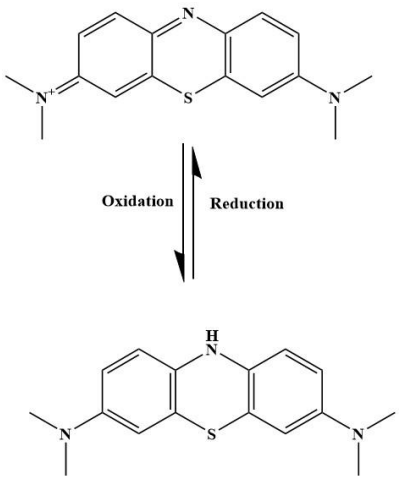


Fig 6: Oxidized and reduced form of methylene blue dye

4.1. Effect of Concentration of Photocatalyst

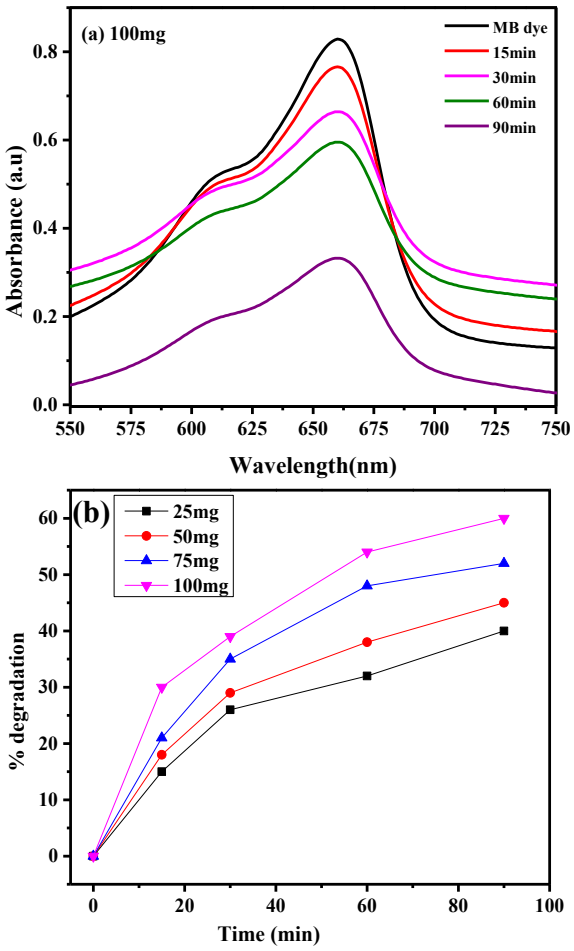


Fig. 7: Photocatalytic dye degradation of MB dye a) 100mg b) variable amounts of photocatalyst

The photocatalytic degradation of Methylene blue was carried out varying the amount of photocatalysts. As the amount of photocatalyst load increases, the degradation efficiency increases as the adsorption sites on nanoparticles increase [22]. The amount of photocatalyst was varied from 25–100mg. The bio-synthesised MgO nanoparticles have a degradation efficiency of 60% with 100 mg loading. The UV spectrum of photocatalytic dye degradation of methylene Blue with maximum loading of biosynthesised MgO nanoparticles, the variation in the amount of photocatalyst is represented in Fig. 7.

4.2. Effect of Concentration of dye

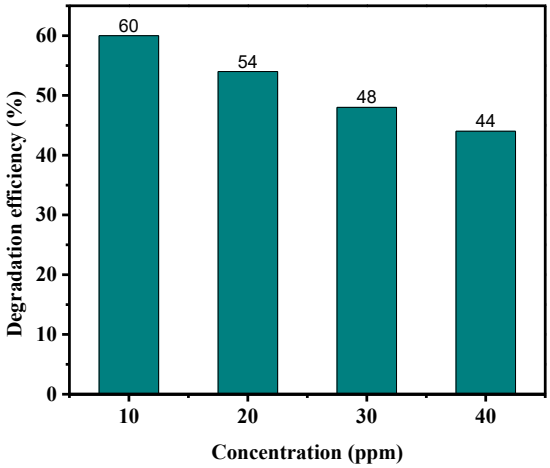


Fig. 8: Degradation efficiency of variable concentration of dye

The degradation efficiency also depends on the concentration of dye. The effect of concentration of dye was measured from 10 – 40 ppm as shown in Fig. 8 The highest degradation efficiency was observed with the lower concentration of 10ppm. As the concentration of dye increases to 40 ppm, the degradation efficiency drops by 16%. This may be because of the blocking of active sites on the photocatalyst by dye molecules, delaying the rate of breakdown [41-43].

4.3. Effect of pH

pH plays a vital role in the degradation of dye. The charge on MgO nanoparticles photocatalyst will vary with the change in pH. Methylene blue dye is a cationic dye that tends to absorb negative species. The photocatalyst is effective in the presence of OH radical, thus indicating that the efficiency of degradation is higher at basic pH. The surface of the

photocatalyst is free to attract the positive species, which are only cationic dyes in the basic pH range. The optimum pH for the degradation of methylene blue dye is 12, as shown in Fig. 9 [7,23,44].

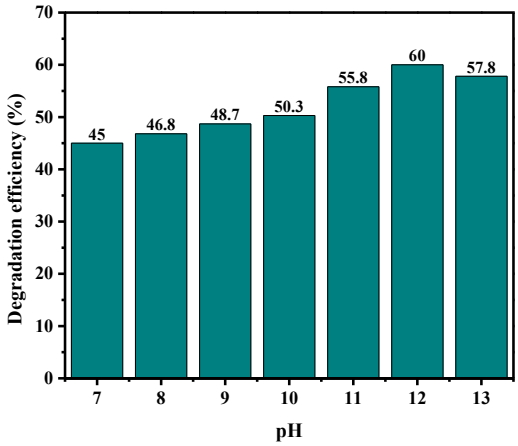


Fig. 9: Degradation efficiency of Methylene blue with variable pH

4.5. Reusability and Scavenger Studies

The bio-synthesised MgO nanoparticle retains its stability up to 4 consecutive cycles. The degradation efficiency was reduced during the fifth cycle, as shown in Fig. 10 (a). From Fig. 10(b), it is observed that the degradation efficiency is reduced in the presence of radical scavengers. The radical scavengers like ammonium oxalate and t-butanol were involved, which showed varying amounts of reduced degradation efficiency. The major reduction was observed in the presence of t-butanol, which is a hydroxyl radical scavenger. Thus, it can be concluded that the major degradation is facilitated by hydroxyl radical [16,45,46].

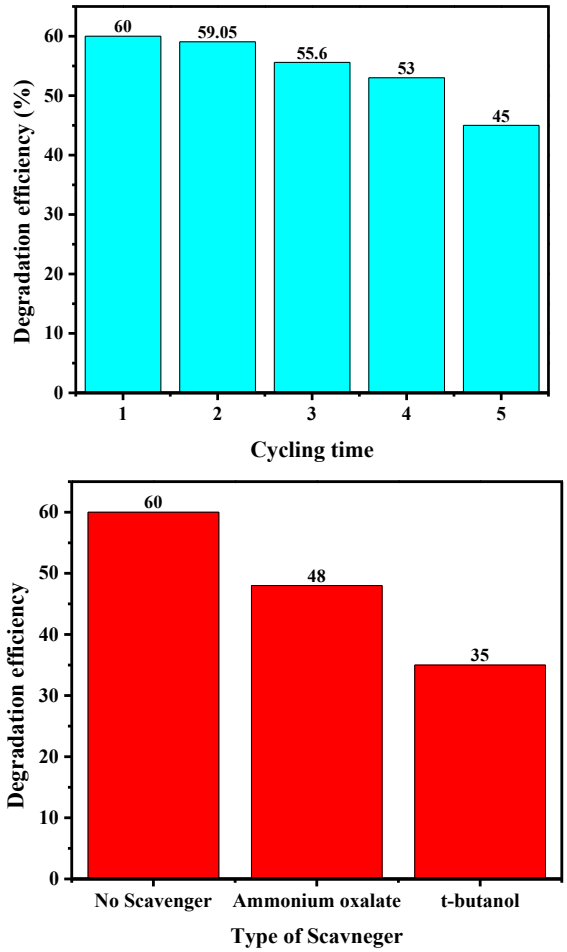


Fig. 10 (a): Reusability of MgO nanoparticles and **(b)** Degradation efficiency of methylene blue using different scavengers

4.6. Mechanism of dye Degradation and Kinetics

The MgO photocatalyst present in the aqueous solution of methylene blue dye will adhere the dye molecules which induce the electronic transition from HOMO to LUMO. When exposed to UV light, the electronic transition gives rise to electron holes and an equal number of free electrons. The excited electron of biosynthesised MgO nanoparticles is transferred from the valence band to the conduction band of MgO nanoparticles. The electron holes generated will react with water to release hydrogen protons and Hydroxyl radicals, the important species responsible for the decolourisation of methylene blue dye [7, 47]. From Fig.11, it is evident that as the exposure time increases, the intensity of the absorbance peak decreases, indicating enhanced degradation efficiency. Kinetic studies have been conducted to study the

photocatalytic activity of bio-synthesised MgO nanoparticles. Fig.11 shows the first-order kinetics evaluated in the time range of 15 – 90 min with 100mg of MgO nanoparticles and 10ppm of Methylene Blue dye. The rate constant was found to be 0.0103 min^{-1} and has the corresponding correlation coefficient (R^2) of 0.840.

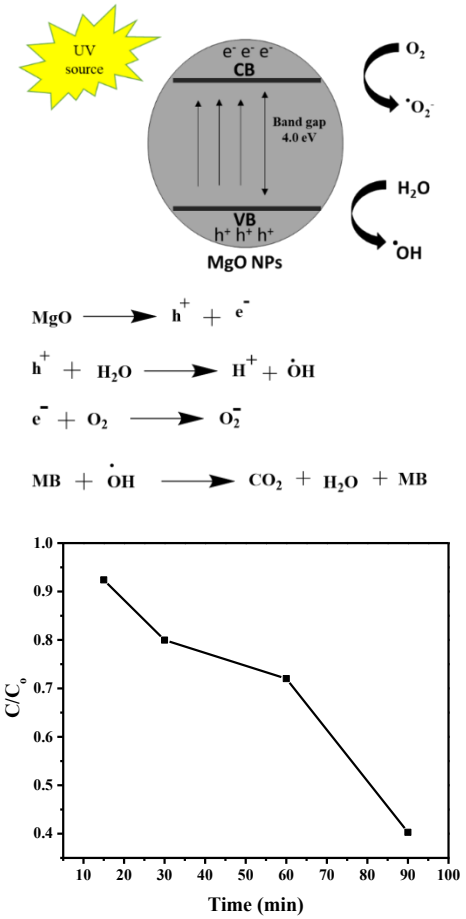
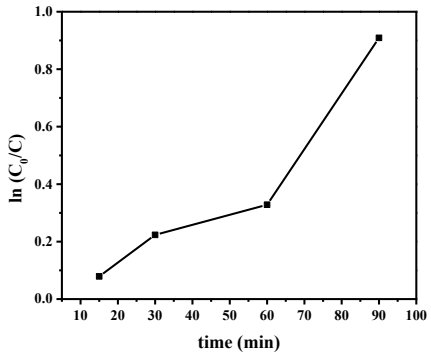


Fig. 11 (a): C/C_0 vs irradiation time curves and **(b)** Natural logarithm C_0/C vs irradiation time



5. Conclusion

The MgO nanoparticles were synthesized by employing the green synthesis method by using Hog-Plum leaf extract. The formation of MgO nanoparticles was confirmed by UV-Visible spectroscopy. The functional group analysis was carried out by FT-IR. The crystal size, dislocation constant and micro strain were calculated using XRD Analysis. The shape and elemental composition were confirmed by employing SEM and EDAX. The bio-synthesised MgO nanoparticles were employed as a photocatalyst in the degradation of methylene blue dye. From the photocatalytic degradation data, it was observed that the degradation efficiency increased with the nanoparticles concentration, and decreased with dye concentration. At an optimum pH of 12, the MgO nanoparticles showed the highest degradation efficiency towards methylene blue dye. The scavenger study reveals that the degradation is mainly due to the presence of hydroxyl radicals. The reusable stability of MgO nanoparticles was seen up to 4 consecutive cycles. It is also evident that the degradation of methylene blue dye using the bio-synthesised MgO follows first-order kinetics.

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