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# **Adsorption of Methylene blue by MgNiO<sub>2</sub> Nano Photocatalyst**

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**ABSTRACT:** The amount of wastewater generated is increasing day by day, hence the contamination and exhaust of freshwater resources have left us with no option but to treat the wastewater for reuse. Many methods used to treat the methylene blue dye involve biological, physical and chemical technologies but adsorption plays an important role in water treatment, due to its low cost, ease of application, and efficiency. In this paper, effective and rapid adsorptive removal of methylene blue dye has been studied experimentally using optimized MgNiO<sub>2</sub> adsorbent. Nano-photocatalyst MgNiO<sub>2</sub> powder was synthesized by solution combustion method and characterized with analytical techniques like powder X-ray diffraction (PXRD), Energy-dispersive X-ray spectroscopy (EDX) & Powders morphology was analyzed by Scanning electron microscopy (SEM). The effect of various adsorption parameters like adsorbate concentration (5-25) mg L<sup>-1</sup>, solution pH (3-11), adsorbent dosage (0.005-0.025) g, contact time (0-30) min on the extent of removal of methylene blue dye was studied in detail. The results show that the use of effective, low-cost and easily available MgNiO<sub>2</sub> photocatalyst for the decomposition of pollutants in water under natural sunlight.

**KEYWORDS:** Adsorption, Dye, Methylene blue, Sunlight, MgNiO<sub>2</sub>, Nanoparticle.

## **I. INTRODUCTION**

The wastewater generated in textile, leather, pulp, painting, paper, printing, photographs and cosmetic industries are associated with different coloring agents (dye) which enhances the COD, BOD and TOC of water. Besides they impart dark color to the water, which makes it toxic then it poses a great threat to the lives of the living organisms and the environment. The amount of wastewater generated is increasing day by day, hence the contamination and exhaust of freshwater resources have left us with no option but to treat the wastewater for reuse[1]. But the degradation of dyes is difficult due to complex aromatic structure which makes higher resistance towards ambient circumstances (bio, thermal, photodegradation). One of the important dye is the Methylene Blue dye [3,7-bis(Dimethylamino)-phenazathionium chloride Tetramethylthionine chloride], have chemical formula (C<sub>16</sub>H<sub>18</sub>CIN<sub>3</sub>S.3H<sub>2</sub>O), is a heterocyclic aromatic compound. Many methods used to treat the methylene blue dye involve biological, physical and chemical technologies but the adsorption plays an important role in the water treatment, due to its low cost (especially the natural adsorbent use), ease of application, and efficiency [2].

The present work reports the effects of different operational parameters on the degradation of dye and the adsorption of methylene blue (MB) by MgNiO<sub>2</sub> nanoparticles as photocatalyst under sunlight radiation have been studied.

## **II. EXPERIMENTAL METHODS**

### **A. Preparation and Characterization of Nano catalyst**

The MgNiO<sub>2</sub> nan catalyst was prepared by solution combustion method using magnesium nitrate, nickel nitrate and citric acid. Initially, a stoichiometric amount of magnesium nitrate, nickel nitrate and citric acid was dissolved in a minimum quantity of de-ionized water. The resulting mixture was kept under continuous stirring at a

temperature of 80°C for 2h. The obtained semi-transparent colloidal suspension solution was put into the pre-heated muffle furnace at 350°C. It swells and forms foam which ruptures with a flame. The product formed after combustion is a voluminous and foamy magnesium nickel oxide.

To determine the phase components and crystalline structure, the Powder X-ray diffraction (PXRD) measurement was carried out at room temperature. The measurement accelerating voltage and current 40 kV and 30 mA respectively and rotating anode Cu K<sub>β</sub> (1.3922 Å). The XRD patterns were obtained from 5° to 100° in 2(θ) range with step size 0.01°. The surface morphology of the adsorbent characterized with a scanning electron microscope (SEM), Energy-dispersive x-ray spectroscopy (EDS) was used to sense the nominal composition of the adsorbent.

### B. Photocatalytic Activity

The photocatalytic activities of MgNiO<sub>2</sub> were tested under sunlight irradiation. A known quantity of the MgNiO<sub>2</sub> was introduced into the 50 ml aqueous solution of methylene blue (5-25ppm). The mixture was placed on a magnetic stirrer under sunlight and stirred magnetically at 450 rpm to achieve uniform distribution with simultaneous exposure to sunlight. The known amount of decomposed dye solution was withdrawn at specific time intervals and centrifuged at 6000 rpm for 3 min. The absorbance was measured using UV-Spectrophotometer at 665 nm. The rate of photo-degradation of methylene blue (MB) was calculated by the following Eq. (1)

$$\text{Degradation}(\%) = \frac{(A_i - A_t) \times 100}{A_i} \quad (1)$$

Where A<sub>i</sub> is the absorbance of the initial MB solution; A<sub>t</sub> is the absorbance of the solution after illumination at the time 't' for MB solution.

## III. RESULTS AND DISCUSSION

### A. Powder X-ray Diffraction (PXRD)

Phase components and crystalline structure details of the adsorbent sample obtained by PXRD. MgNiO<sub>2</sub> crystal structure, PXRD pattern presented in fig.1. All the diffraction peaks at 2θ of 37.1°, 43.1°, 62.6°, 75.1°, 79.1° and 94.7° are perfectly matched with the JCPDS card Files No. 24-712 (Joint Committee on Powder Diffraction Standards cubic spinel structure) [3]. Further, the crystallite size was estimated by using the peak shape broadening method using Scherrer's and the average crystallite size was 6 nm, using the relation.

$$D = k\lambda / \beta \cos(\theta) \quad (2)$$

Where 'β' is the complete broadening of half maximum of a diffraction peak, 'θ' is the diffraction angle.

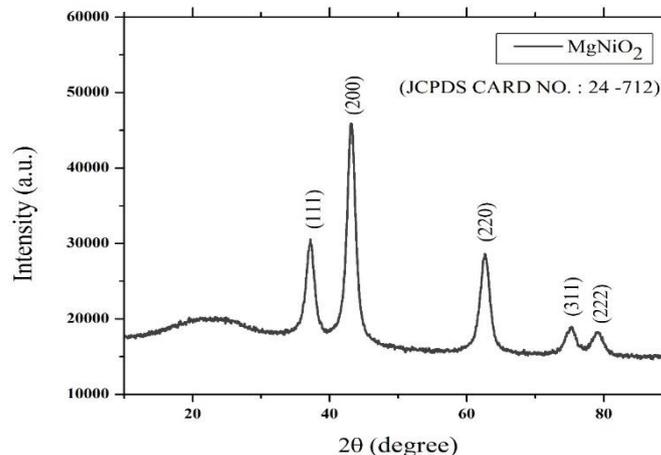


Fig. 1. P-XRD pattern for MgNiO<sub>2</sub> nanoparticle

**B. Scanning electron microscopy (SEM) and energy-dispersive x-ray spectra (EDS)**

The surface morphology of the adsorbent was determined using Scanning electron microscopy, Surface assimilation of the adsorbent is shown in micrographs fig.2(a). It is evident that the particles agglomerated, and the surface shows rough clumps and is found to have huge voids and pores providing more adsorption site and high adsorption MB uptake expected. The agglomeration is typical due to the solution combustion process. The voids and pores are due to the liberation of gases during combustion [4]. Energy-dispersive X-ray spectroscopy (EDS) was used to sense the nominal composition of the adsorbent is shown in fig. 2(b). It is a strong tool to predict the mapping mode to examine the fundamental distribution magnitude relation and to determine the compositions of the weather on the surface of  $MgNiO_2$  adsorbents. However, carbon was also detected as an impurity along with the expected elements. The presence of carbon in the adsorbent due to the fuel used in combustion [4].

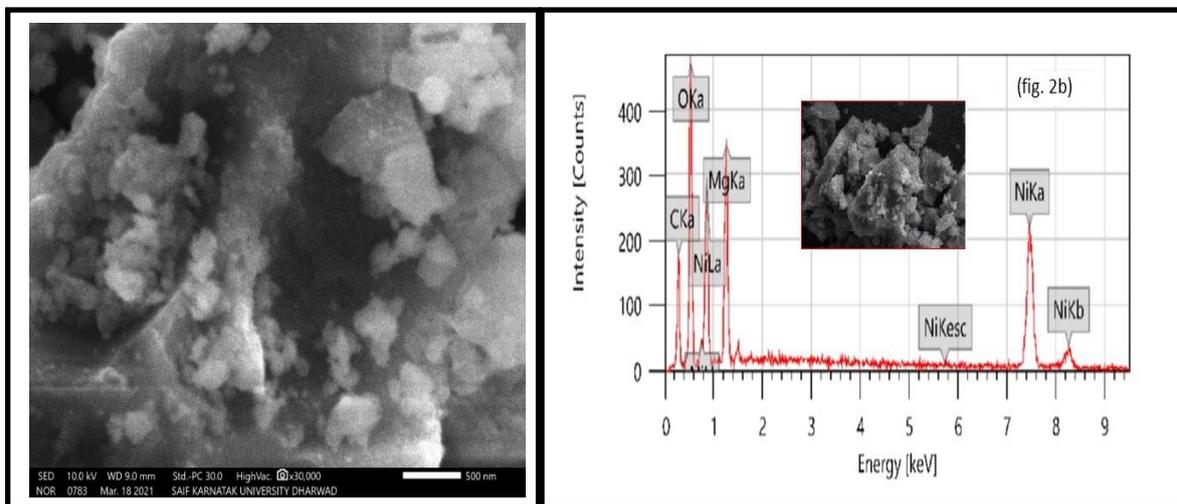


Fig.2.(a):SEM micrograph of  $MgNiO_2$  nanoparticle. (b):EDS spectra of  $MgNiO_2$  nanoparticle

**C. Photocatalytic degradation of MB**

To understand the photocatalytic degradation of MB dye using  $MgNiO_2$  as a catalyst, we studied the effect of several factors on the degradation process including photocatalyst loading, contact time, adsorbate concentration, and solution pH [5].

**C.1 Effect of photocatalyst loading**

The optimization of the  $MgNiO_2$  loading was studied by varying the  $MgNiO_2$  loading from 0.005g to 0.025g per 50mL of MB dye solution. The outcome is shown in Fig. 3. Which declares that percent removal of MB increases on increasing  $MgNiO_2$  loading? It is because an increase in  $MgNiO_2$  loading eventually increased the number of sites available on  $MgNiO_2$  which enhanced the adsorption capacity of  $MgNiO_2$ . The maximum percent removal of MB was observed at 0.015g of  $MgNiO_2$ . On further increase in the  $MgNiO_2$  loading, the turbidity of the solution has been observed.

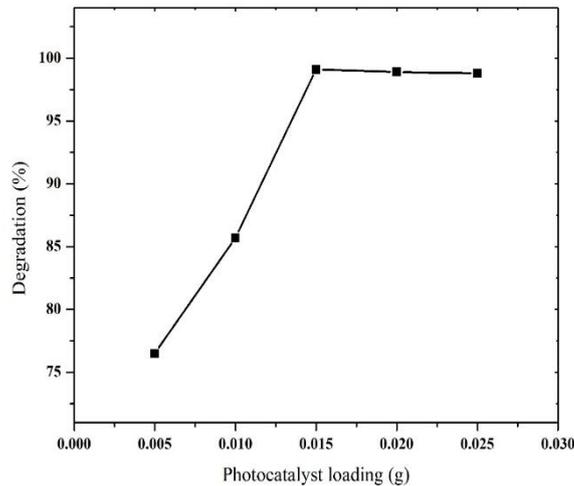


Fig. 3. Effect of photocatalyst loading on percent removal of MB.

### C.2 Effect of contact time

The optimization of contact time was obtained by varying it from 0 to 30 min at optimized  $\text{MgNiO}_2$  loading 0.015g for the same volume and concentration (50mL, 5ppm) of dye as shown in Fig. 4. It was observed that the percent removal of MB by  $\text{MgNiO}_2$  increased with an increase in contact time. It is because more time allows maximum contact period for the adsorbent to interact with the dye. The maximum removal was observed at 25 min beyond which it became constant. Thus  $\text{MgNiO}_2$  and dye should be in contact for 25 min to attain maximum adsorption.

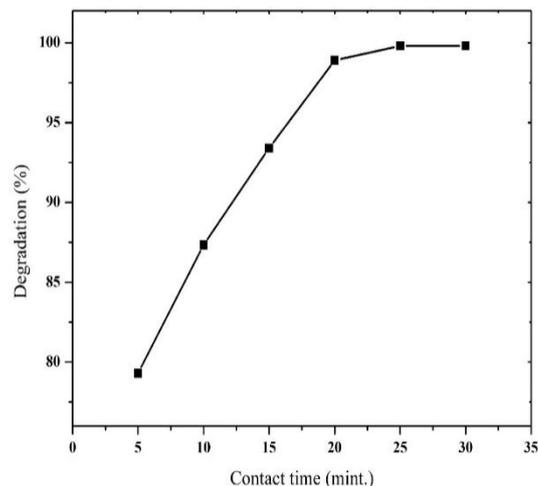


Fig. 4. Effect of contact time on percent removal of MB.

### C.3 : Effect of adsorbate concentration

The optimization of MB dye concentration was carried out to obtain the maximum adsorption capacity of  $\text{MgNiO}_2$ . the concentration of MB dye was optimized by studying the percent removal of MB for varying initial concentrations (5-25  $\text{mg L}^{-1}$ ) at optimized adsorbent dose and time. The results are shown in Fig. 5. This reveals that

the percent removal of MB dye decreases with an increase of dye concentration. This decrease is due to the saturation of activesites of  $\text{MgNiO}_2$  after adsorption of a certain concentration of MB.

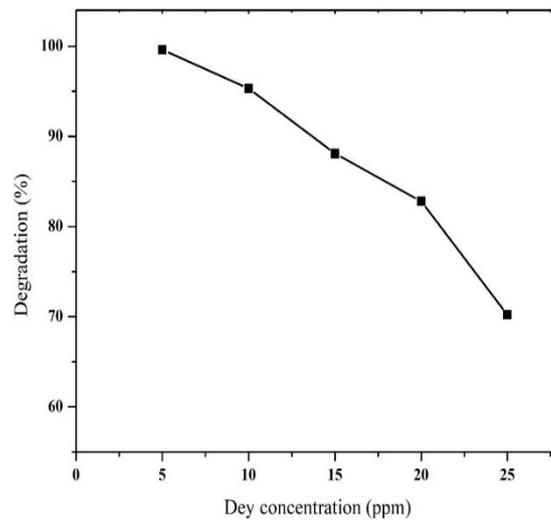


Fig. 5. Effect of dye concentration on percent removal of MB

#### C.4 : Effect of solution Ph

The optimization of pH was studied by varying it from 3 to 11 in 50mL MB dye solution ( $5 \text{ mg L}^{-1}$ ). To each solution, 0.015g of  $\text{MgNiO}_2$  was added. From Fig. 6. It has been observed that the removal efficiency of MB by  $\text{MgNiO}_2$  is almost 99.8% for the entire pH range (3-11). Based on adsorption results, it can be concluded that the proposed nanomaterial efficiently works in a wide pH range for the removal of MB dye.

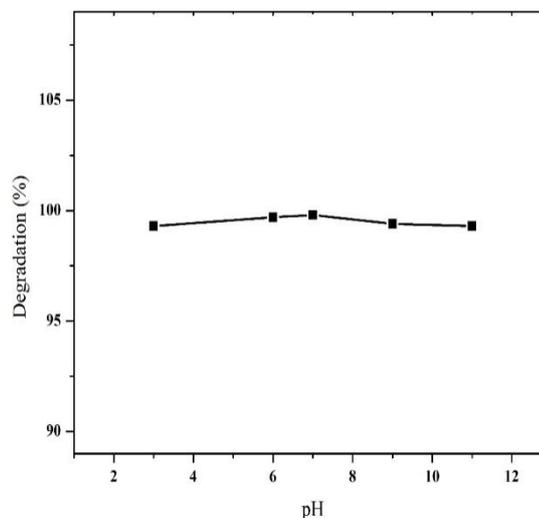


Fig. 6. Effect of solution pH on percent removal of MB.



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## IV. CONCLUSIONS

In this summary, MgNiO<sub>2</sub> nanoparticles have been successfully prepared by the solution combustion method. The structure and morphology of the nanoparticle were studied using X-ray diffraction and scanning electron microscopy analysis. The obtained particles were in the average crystallite size of 6 nm. The experiment of decolorizing MB confirmed that the obtained photocatalyst can perform the significant photocatalytic activity in solar light irradiation. Therefore, the MgNiO<sub>2</sub> nanoparticle is a good catalyst in the treatment of industrial wastewater to eliminate organic pollutants, which can cause severe harm to the environment.

## V. ACKNOWLEDGMENTS

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