



Synthesis of Lithium Copper Molybdate Nanoparticles and their Application for Photocatalytic Degradation of Malachite Green

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Authors' contributions

This work was carried out in collaboration between all authors. Author SCA designed the study and supervised the overall work and author MJ performed the experiment and wrote the first draft of the manuscript. Author RA helped in characterizing the prepared catalyst. Author PK helped in literature survey. All authors read and approved the final manuscript.

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ABSTRACT

In the present investigation, degradation of a non - biodegradable azo dye, malachite green has been carried out using lithium copper molybdate ($\text{Li}_2\text{CuMo}_2\text{O}_8$) as a photocatalyst. This catalyst was prepared by solid state reaction between Li_2CO_3 , CuO and MoO_3 . The effect of various parameters such as pH, concentration of malachite green, amount of lithium copper molybdate and light intensity has also been studied. Water quality parameters like chemical oxygen demand (COD), conductance, pH, TDS, salinity and dissolved oxygen have been determined before and after treatment. The rate of photodegradation of malachite green dye followed pseudo- first order kinetics. A tentative mechanism involving OH radicals as an oxidant for degradation of dye has been proposed.

Keywords: Malachite green; lithium copper molybdate; photodegradation; hydroxyl radical; oxidant.

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1. INTRODUCTION

Dyes are important source of environment contamination. Textile waste waters contain usually a considerable amount of unfixed dyes, many of which are azo dyes [1]. It was estimated that fifteen percent of the total world dye production is lost during dyeing process and it is released in textile effluents [2]. Moreover, environmental pollution by organic dyes also cause economical problem, which is increased by the fact that most of them are often toxic to microorganisms and have long degradation times in the environment [3]. Among these dyes, the azo dyes constitute the largest and the most important class of commercial dyes [4]. Azo dyes are toxic, carcinogenic mutagenic and known to be largely non-biodegradable under aerobic condition, and their stability is proportional to the structure complexity of their molecular structures [5]. Consequently, various methods like adsorption, osmosis, flocculation, biological and chemical methods such as chlorination, ozonation etc. are used traditionally to remove these dyes from the water bodies, but these methods suffer from some drawbacks [6]. Promising methods as an alternative to conventional methods for dye degradation is Advanced oxidation processes (AOPs) such as photo-Fenton, photocatalysis, sonolysis, etc which are being used to solve these environmental problems. This method involves the generation of hydroxyl radicals and use of these radicals as the primary oxidant for degrading organic pollutants. Modirshahla and Behnajady [7] reported the photooxidative degradation of malachite green (MG) by UV/H₂O₂ technology while Chen et al. [8] carried out the photodegradation of MG using TiO₂ nanoparticle under UV light irradiation. Bojinova et al. [9] studied the photocatalytic degradation of MG dye using TiO₂/WO₃ composite as photocatalyst. Asilturak et al. [10] studied the effect of Fe³⁺ ion doping on TiO₂ in photocatalytic degradation of MG dye under UV and visible light irradiation. Palladium modified tungsten trioxide used for photocatalytic degradation of MG dye in aqueous solution by Liu et al. [11].

Afshar et al. [12] worked on Pt/TiO₂/SiO₂ nanophotocatalyst, which is capable in photodegradation of MG under UV and visible light irradiation. α -Fe₂O₃ nanocrystals were synthesized by Pauer and Khajon [13] using sol-gel method and used these for photodegradation of MG under visible light irradiation. Tolia et al. [14] reported photocatalytic degradation of MG

using doped and undoped ZnS nanoparticles. Trabelsi et al. [15] studied sunlight activated photocatalysis of MG using TiO₂/cellulosic fiber. A hexagonal ZnO and anatase/rutile TiO₂ nanocomposite has been prepared by Zhang et al. [16] for photodecomposition of Rhodamine B and malachite green dye. Kaneva et al. [17] synthesized pure and SiO₂-ZnO nanocomposite for photodegradation of two organic dyes MG and methylene blue under UV and visible light irradiation. Panchal and Vyas [18] reported photocatalytic degradation of MG using undoped and iron doped zirconium dioxide.

Kaneva et al. [19] examined the photocatalytic efficiency of ZnO films for photodegradation of MG dye while Saikia et al. [20] studied the photocatalytic performance of ZnO nanomaterial for self sensitized degradation of MG dye under solar light irradiation. Josephine et al. [21] carried out the photocatalytic degradation of organic dye MG using ZnO doped Dy₂O₃ photocatalyst under UV light irradiation. H-Y. He [22] synthesized Ni_{1-x}Co_xFe₂O₄ (x = 0-1.0) ferrite nanoparticles using hydrothermal process for photodegradation of MG. Tadjarodi et al. [23] reported the photocatalytic activity of CdO for degradation of MG while Charanpahari et al. [24] carried out photocatalytic degradation of MG in the presence of Bi doped TiO₂-ZrO₂ ferromagnetic nanocomposite. A variety of semiconductor such as TiO₂, SiO₂-TiO₂, WO₃, ZnO, ZnS, have been used for photocatalytic degradation of MG. In the present work, lithium copper molybdate has been used for the degradation of malachite green.

2. MATERIALS AND METHODS

Malachite green 4{[4-(dimethylamino)phenyl] (phenyl) methylidene}-N,N-dimethylcyclohexa-2,5-dien-1-iminiumchloride, lithium carbonate, cupric oxide, molybdenum oxide, polyvinyl acetate, acetone K₂Cr₂O₇, HgSO₄, Ag₂SO₄, Na₂S₂O₃. 5H₂O were employed in the present study. Malachite green was used as a model system to investigate the photocatalytic activity of synthesized Li₂CuMo₂O₈. Its molecular formula is C₂₃H₂₅ClN₂ and molar mass 927.02 g/mol. 1.0 x 10⁻³ solution of MG (0.098 g MG in 100 mL water) was prepared with doubly distilled water and stored as a stock solution. The photocatalytic degradation of MG was observed by taking dye solution of 1.3 x 10⁻⁵ M and 0.10 g of Li₂CuMo₂O₈ at pH 10. Irradiation was carried out keeping the whole assembly exposed to a 200 W tungsten lamp (light intensity = 50.0 mWcm⁻²). The intensity of light was measured

with the help of a solarimeter (SM CEL 201). The pH of the solution was measured by the digital pH meter (Systronics Model 335). The desired pH of solution was adjusted by the addition of previously standardized 0.1 N sulphuric acid and 0.1 N sodium hydroxide solutions. The absorbance (A) was measured spectrophotometrically (Systronics Model 106). Some water parameters like pH, conductivity, salinity, TDS (Total dissolved solids), and DO (Dissolved oxygen), were determined with the help of water analyzer (Systronic Water Analyzer Model 371). Structure of malachite green is given in Fig. 1.

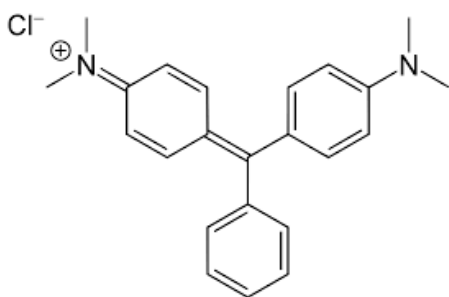


Fig. 1. Structure of malachite green

2.1 Synthesis of Semiconductor

A quaternary oxide was prepared by method of solid-state reaction. Stoichiometric amounts of Li_2CO_3 , CuO and MoO_3 were mixed and ground in an agate motor using acetone for

homogeneity. 5% solution of polyvinyl acetate in acetone was added as a binder.

2.2 Calcination of Semiconductor

The sample was kept in electric muffle furnace at 300°C for 3 hrs to evaporate the binder. Then, the sample was calcined in furnace at 650°C for 30 hrs. Then, the mixture was grind with a pestle in a mortar.

2.3 Photocatalytic Degradation

The photocatalytic degradation of malachite green dye using nanosized $\text{Li}_2\text{CuMo}_2\text{O}_8$ photocatalyst under visible light was investigated. The concentration of dye in the form of absorbance during photocatalytic degradation was measured at 620 nm (λ_{max}). A 200 W tungsten lamp was used as the visible light source. The progress of the photocatalytic reaction was observed by taking absorbance at regular time intervals. The change in absorbance versus irradiation time was measured. A typical run is presented in Fig. 2. It was observed that the absorbance (A) of malachite green solution decreased in presence of the photocatalyst and light. The plot of $\log A$ versus time was linear and hence, this reaction pseudo- first order kinetics. The rate constant (k) for this reaction was determined from the expression $k = 2.303 \times$ slope and it was $8.52 \times 10^4 \text{ sec}^{-1}$ under optimum conditions. The plot of a typical run is presented in Fig. 2.

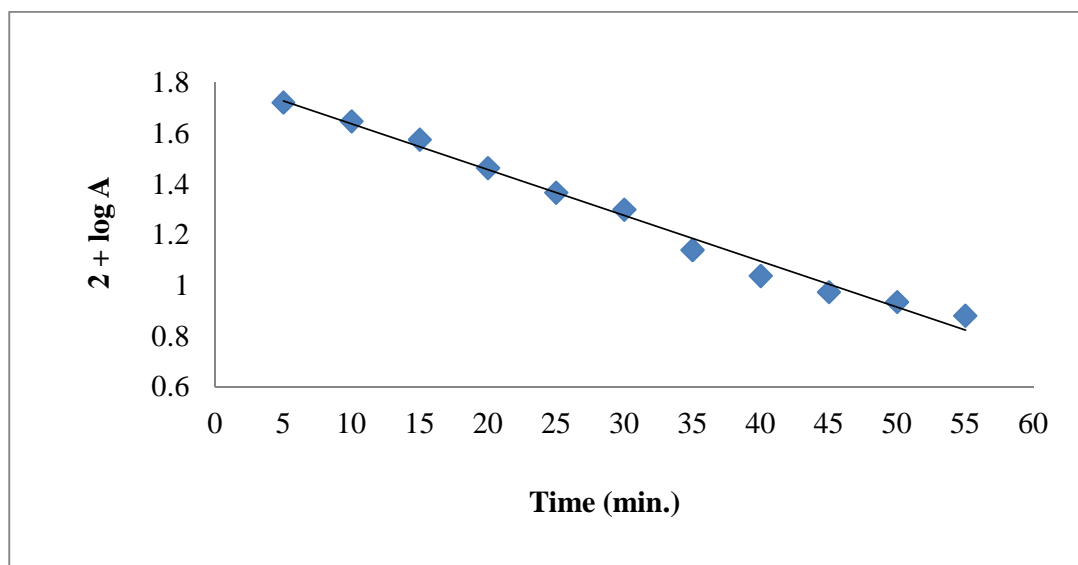


Fig. 2. A typical run

3. RESULTS AND DISCUSSION

3.1 Effect of pH

The effect of pH on photocatalytic degradation was investigated in the range 5.0- 10.0. The results are reported in Table 1.

Table 1. Effect of pH

pH	Rate constant (k) × 10 ⁴ (sec ⁻¹)
5.0	0.06
5.5	0.06
6.0	0.07
6.5	0.23
7.0	1.63
7.5	1.69
8.0	2.50
8.5	4.13
9.0	5.37
9.5	6.33
10.0	8.52
10.5	9.98

[Malachite green] = 1.3 × 10⁵ M; Light intensity = 50.0 mW cm⁻²; Lithium copper molybdate = 0.10 g

It was observed that the rate of degradation of malachite green increases on increasing pH regularly. All other variations have been made at pH 10 as the reaction becomes very fast on increasing pH of the reaction medium above 10. It may be due to the increasing attraction between cationic dye and negatively charged surface of the semiconductor, which increases as more and more OH⁻ ions are adsorbed on increasing pH of the reaction medium [25].

3.2 Effect of Concentration of Malachite Green

The concentration of dye was varied from 1.0 × 10⁵ to 1.9 × 10⁵ M. The result are reported in Table 2.

It has been observed that the rate of photocatalytic degradation increases with increasing the concentration of the dye up to 1.3 × 10⁵ M. This may be due to the fact that as the concentration of the malachite green was increased, more dye molecules were available for excitation and consecutive reaction. Hence, an increased in the rate was observed. The rate of photocatalytic degradation was found to decrease with further increase in the concentration of dye. This may be attributed to the fact that the dye started acting as a filter for the incident light and it does not permit the

desired light intensity to reach the photocatalyst surface in a limited time domain; thus, decreasing the rate of photocatalytic degradation of malachite green [26].

Table 2. Effect of dye concentration

[Malachite green] × 10 ⁵ M	Rate constant (k) × 10 ⁴ (sec ⁻¹)
1.0	5.13
1.1	6.83
1.2	7.13
1.3	8.52
1.4	7.83
1.5	7.29
1.6	7.13
1.7	6.83
1.8	6.27
1.9	6.23

pH = 10.0; Light intensity = 50.0 mW cm⁻²;
Lithium copper molybdate = 0.10 g

3.3 Effect of the Amount of Photocatalyst

The effect of the amount of photocatalyst is also likely to affect the process of dye degradation and therefore, different amounts of photocatalyst were used. The result are reported in Table 3.

Table 3. Effect of lithium copper molybdate

Lithium copper molybdate (g)	Rate constant (k) × 10 ⁴ (sec ⁻¹)
0.02	6.42
0.04	6.65
0.06	7.18
0.08	7.72
0.10	8.52
0.12	8.51
0.14	8.51
0.16	8.49
0.18	8.49

pH = 10.0; Light intensity = 50.0 mW cm⁻²;
[Malachite green] = 1.3 × 10⁵ M

The rate of reaction was found to increase on increasing the amount of semiconductor, lithium copper molybdate. The rate of degradation reached to its optimum value at 0.10 g of the photocatalyst. Beyond 0.10 g, the rate of reaction becomes almost constant. This may be explained on the basis that as the amount of semiconductor was increased, the exposed surface area of the semiconductor also increases. However, after a particular value (0.10 g), an increase in the amount of semiconductor will only increase the thickness of

layer of the semiconductor and not its exposed surface area [27].

3.4 Effect of Light Intensity

The effect of the variation of the light intensity on the rate was also investigated and the result are reported in Table 4.

Table 4. Effect of light intensity

Light intensity (mW cm ⁻²)	Rate constant (k) × 10 ⁴ (sec ⁻¹)
20.0	5.06
30.0	6.52
40.0	7.21
50.0	8.52
60.0	7.84
70.0	6.83

*pH = 10; [Malachite Green] = 1.3 × 10⁻⁵ M;
Lithium copper molybdate = 0.10 g*

The data indicate that photocatalytic degradation of malachite green was enhanced with the increase in intensity of light, because an increase in the light intensity will increase the number of photons striking per unit area per unit time of photocatalyst surface. There was a slight decrease in the rate of reaction as the intensity of light was increased beyond 50.0 mW cm⁻². Therefore, light intensity of medium order was used throughout the experiments [28].

3.5 Determination of Water Quality Parameters

Quality of water before and after photocatalytic degradation has been tested by measuring some parameters and results are summarized in Table 5.

Table 5. Water quality parameters

Parameter	Before photocatalytic degradation	After photocatalytic degradation
pH	10	8.8
Conductivity (µS)	139	170
TDS (ppm)	159	370
Salinity (ppt)	0.39	0.42
DO (ppm)	12.9	14.4
COD (mg/L)	40	20

3.5.1 pH

pH of sample indicates the extent of its pollution by acidic and alkaline wastes. All chemical and biological reaction directly depends upon the pH

of medium. pH of malachite green is in basic range before the degradation but after the photocatalytic degradation of the dye, pH reaches to towards neutral range (Slightly basic). Dyes are not in permissible limit in water which is not suitable for irrigation and drinking purpose according to WHO standard, whereas pH of photocatalytically treated water by Li₂CuMo₂O₈ is suitable for animal and aquatic biota. This result indicates that pH value of sample was in alkaline range but within the ISI permissible limits [29].

3.5.2 Conductivity

Conductivity as a summation parameters is a measure of the level of concentration of ion in a solution. It is directly proportional to its dissolved mineral matter content. Consequently it is an index of the salt load in waste water or the impurity of water. However, conductivity is only a quantitative measurement. Only ionizable material will contribute to conductivity. Conductivity was found to increase in treated water. Slight decrease in pH and increase in conductivity also confirms the mineralization of dye into CO₂ and inorganic ions such as CO₃²⁻, NO₃⁻, etc.

3.5.3 Total Dissolved Solid (TDS) and salinity

Dissolved solids refer to any minerals, salts, metal, cations, or anions dissolved in water. This includes anything present in water other than pure water molecules and suspended solids. In general, the TDS results in an undesirable taste, which could be salty, bitter or metallic. It could also lead to gastrointestinal irritation. TDS value of malachite green was increased from 159 ppm to 370 ppm due to mineralization of dye particles.

3.5.4 Dissolved Oxygen (DO)

Dissolved oxygen gives an idea about physical and biological activity in water. The minimum standard limit is 5 ppm. Oxygen can serve as electron sink to trap the excited conduction band electron from reactive oxygen species. DO has been proposed to be responsible for cleavage of aromatic rings in dye molecule. DO value of malachite green solution increased from 12.9 to 14.4 ppm [30].

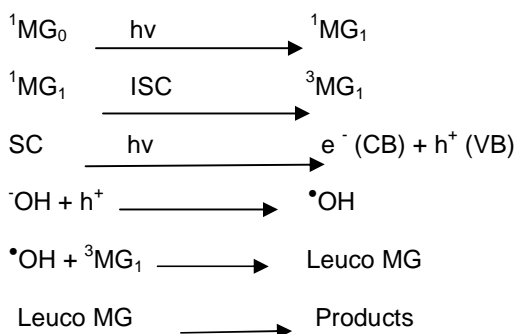
3.5.4 Chemical Oxygen Demand (COD)

The chemical oxygen demands are widely used as an effective technique to measure the organic strength of wastewater. This allows us the

measurement of waste in terms of the total quantity of oxygen required for the oxidation of organic matter to CO₂ and water. The COD of the dye solution before and after the treatment was estimated. The reduction in COD values of the treated dye solution indicates the mineralization of dye molecules along the colour removal [31].

3.6 Mechanism

On the basis of experimental observations, a tentative mechanism for photocatalytic degradation of malachite green may be proposed as:



Malachite green absorbs radiations of desired wavelength and it is excited giving its first excited singlet state. Further, it undergoes intersystem crossing (ISC) to give its more stable triplet state. Along with this, the semiconducting lithium copper molybdate (SC) also utilizes this energy to excite its electron from valence band to the conduction band. An electron can be absorbed from hydroxyl ion by hole (h⁺) present in the valence band of semiconductor generating $\cdot\text{OH}$ radical. This hydroxyl radical will oxidize malachite green to its leuco form, which may ultimately degrade to products. It was confirmed that the $\cdot\text{OH}$ radical participates as an active oxidizing species in the degradation of malachite green as the rate of degradation was appreciably reduced in presence of hydroxyl radical scavenger (2-propanol). The presence of NO₃⁻ and Cl⁻ ions were confirmed by their usual chemical tests. Carbon dioxide and water were major products of degradation.

4. CONCLUSION

Photocatalytic degradation of malachite green dye was studied as model system using Li₂CuMo₂O₈, quaternary oxide, as photocatalyst. From kinetic studies, it was found that the photodegradation is dependent on parameters

such as pH, dye concentration, amount of semiconductor and light intensity of the reaction system. The optimum amount of photocatalyst was 0.10 g for the degradation of 1.3 × 10⁻⁵ M malachite green solution with the pH value of 10.0 at 50.0 mW cm⁻² light intensity. Photocatalytic treatment increased the biodegradability of dye containing polluted water. It helps to reduce COD and pH increased and the DO, conductivity, salinity and TDS of dye solution. It can be concluded that the Li₂CuMo₂O₈ assisted photocatalytic degradation of dye may be versatile, economic, environmental friendly and efficient method of treatment.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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