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Nano-Particles Zinc Oxide for Water Cleaning

Nor Adilah Mohd Ali[1] , Nur Adrinna Nor Arifin[1], Jumaah Noaman Salman[2*]
[1]Universiti Sains Malasia, School of Physics, 11800, penang , malaysia
[2]Ministry of Industry & Minerals State Company for Inspection and Engineering Rehabilitation (SIER), Baghdad Iraq
*Corresponding author: drn_job@yahoo.com

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ABSTRACT

Organic dye is a major industrial pollutant and needs innovative methods to clear it. Zinc oxide (ZnO) is an attractive material for photo catalytic degradation of organic dyes. In this paper, the photo catalytic activity of ZnO nanoparticles (ZnO NPs) synthesized using chemical bath deposition (CBD) and laser-assisted chemical bath deposition (LACBD) are studied. Field Emission Scanning Electron Microscopy (FESEM) showed that ZnO NPs synthesized using LACBD obtained a more packed structure, resembling Nano sheets. This translates to a larger specific exposed area. Energy Dispersive X-ray (EDX) study shows ZnO NPs synthesized using the LACBD method exhibited a Zn:O ratio closer to 1:1, suggesting an adequate concentration of oxygen vacancies, which are crucial for photo catalytic degradation. For the photo catalyst degradation measurement, ZnO NPs are utilized as catalysts in water degradation applications, where 0.01 g of ZnO was added to 50 mL of 20 ppm methylene orange solution. The concentration of methylene orange solution was studied using UV-Vis measurements during photo catalyst degradation. ZnO NPs synthesized using LACBD exhibited the highest photo catalytic degradation performance under UV light, with the highest degradation rate constant, k, of 0.02521 min^{-1} and 71% photo degradation efficiency within 30 minutes.

Corresponding Author:

Nor Adilah Mohd Ali

Universiti Sains Malasia, School of Physics, 11800, penang , malaysia
Email: drn_job@yahoo.com

1. INTRODUCTION

One of the main pollutants in wastewater management is organic dye. Organic dye is a product that has been used in various industries, among them are; textile, food, printing, and cosmetics [1]. As these industries are widespread throughout the world, organic dye as waste poses a serious threat to the environment, which necessitates an effective method of removing it during wastewater treatment. Commercial dyes have skyrocketed, reaching tens of thousands, as listed in the U.S. "Color Index." Annually, a staggering 60,000 tons of dye-contaminated waste is dumped into the environment worldwide, with azo dyes comprising a significant 80% of this pollution [2]. Moreover, China holds the leading position in both fuel production and trade volume globally, and azo dyes constitute a substantial 70-80% of its dye production [2].

These printing and dyeing wastewater effluents contain a range of organic compounds with biological toxicity and exhibit three concerning properties: carcinogenic, teratogenic, and mutagenic. The complex dye composition within these effluents is characterized by high concentration, intense color, and recalcitrant biodegradation [2]. Consequently, the treatment of dye-polluted wastewater has become a pressing environmental concern [3]. Furthermore, research into wastewater management gains more traction when considering the fact that humans worldwide are losing access to clean water [4].

There are many different techniques for handling wastewater, primarily concerning organic dyes. Among them are physical, chemical, biological, and hybrid methods. Figure 1 toFigure 4 provide detailed methodologies grouped into physical, biological, chemical, and hybrid ways of handling dye-polluted waste [3].

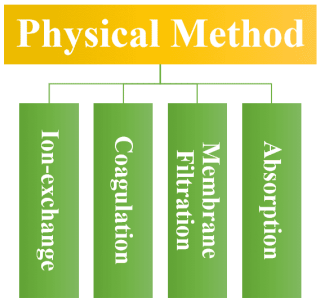

Figure 1: Physical methods for dye-polluted wastewater treatment [3].

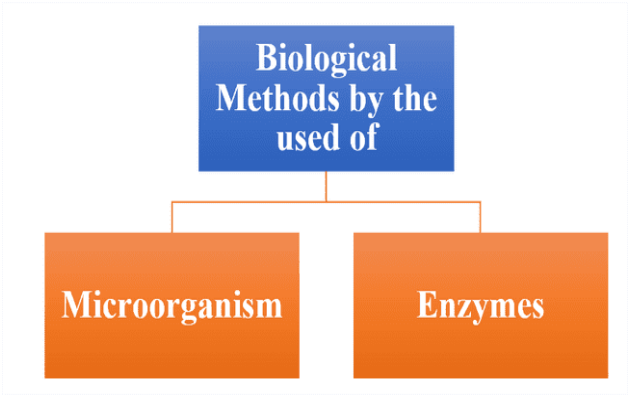

Figure 2: Biological methods for dye-polluted wastewater treatment [3].

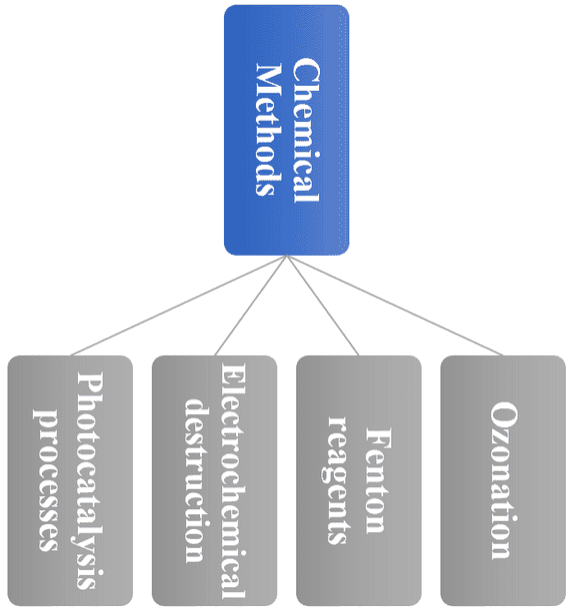

Figure 3: Chemical methods for dye-polluted wastewater treatment [3].

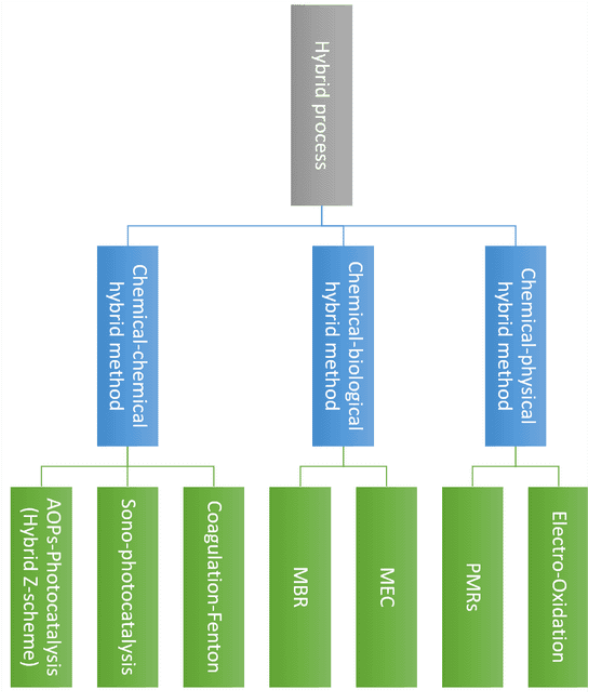

Figure 4: Hybrid methods for dye-polluted wastewater treatment [3].

An innovative way of dye removal involves dye degradation by photocatalysis [5]. In comparison to other technologies that are expensive and time-consuming, photocatalytic degradation by nanoparticles makes a strong argument for widespread implementation. Photocatalytic degradation occurs when pollutants (in the presence of catalysts) are destroyed in ultra-visible (UV-visible) light. This process is more beneficial as it does not produce byproducts such as polycyclic compounds [6, 7].

During a photocatalyst reaction, a catalyst medium that is exposed to light of energy larger than its bandgap will form an electron-hole pair. This pair then reacts with water molecules, forming highly reactive oxidizing or reducing radicals such as O^{2-} and OH^{-}. These radicals then react with organic or inorganic contaminants, such as dyes, to produce carbon dioxide (CO_2) and water (H_2O) respectively. Oxidation reactions are effective for mineralizing dissolved harmful organic compounds, the reduction reaction is useful for eliminating dissolved poisonous ions [8, 9]. Figure 5 provides a visual representation of a photocatalyst reaction [10].

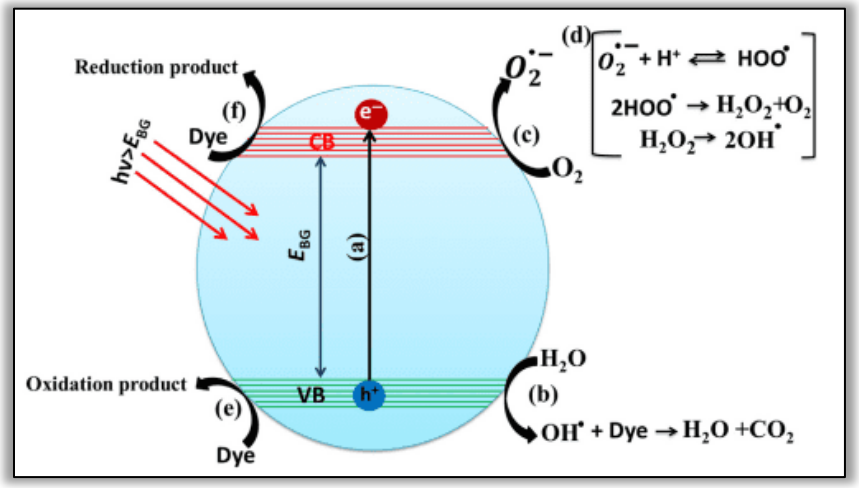

Figure 5: Visual representation of a photocatalyst reaction [10].

For a more effective photocatalyst rate of dye removal, the usage of a semiconductor photocatalyst was recommended, as the semiconductor photocatalyst can speed up the generation of electrons and holes. Therefore, much research is focused on utilizing photocatalysts that are composed of metal oxides, sulfides, or nitride-based semiconductors [11]. One of the semiconductor materials being studied is ZnO-based photocatalysts [12-14]. N-type ZnO possesses certain qualities that make it an efficient photocatalyst material; among them superior photo-reactivity, non-toxic, inexpensive for production, and relatively simple to utilize. These properties have made ZnO extensively researched in degrading various organic pollutants [15, 16].

ZnO nanoparticles exhibit superior quantum efficiency in photodegrading organic pollutants compared to TiO_2. This enhanced performance stems from their greater light absorption in wavelengths shorter than 400 nm, which constitutes approximately 5% of the solar radiation reaching Earth [17, 18]. A catalyst's photoactivity is directly linked to its ability to generate electron-hole pairs upon exposure to light. However, ZnO's primary drawback as a photocatalyst lies in its rapid recombination of photogenerated electron-hole pairs, which hinders the photodegradation process. Additionally, ZnO's optical absorption capacity, influenced by its wide band gap energy, affects its efficiency in converting solar energy. Consequently, significant research efforts have focused on narrowing the band gap energy and suppressing electron-hole recombination to improve ZnO's optical properties.

An important aspect of ZnO nanoparticles is that their morphology affects their photocatalytic activity. Therefore, the method employed in ZnO nanoparticle synthesis is important and warrants further study. In this work, two methodologies for ZnO nanoparticle synthesis are studied for their effectiveness in dye degradation via photocatalyst reaction, which will serve as the main focus of this study. The first method is via simple chemical bath deposition (CBD) and the second is via Laser-Assisted Chemical Bath Deposition (LACBD). Methyl orange (MO) was chosen as the indicator dye due to its widespread use in the textile industry [11]. As an azo dye, it exhibits resistance to complete biodegradation [19]. These characteristics make MO a suitable model dye for studying environmental remediation, including in this research. Furthermore, its concentration can be easily monitored using spectrophotometric measurements.

2. METHOD

2.1 Chemical Bath Deposition (CBD)

Figure 6 shows the step of synthesizing ZnO nanoparticles by the Chemical Bath Deposition (CBD). The materials used are 0.05 M of zinc acetate and zinc nitrate, 0.05 M of hexamine, and 50 ml of deionized water (DI). The needed amounts are calculated using the equation below.

$$W = (M)(M_w)(V) \quad (1)$$

Where W represents the weight (g), M and M_w are the molarity (M), and the molar weight of the material (zinc acetate = 219.49 g mol^{-1}, zinc nitrate =297.49 g mol^{-1}, hexamine = 140.19 g mol^{-1}) respectively, and V is the volume of the solution used (50 ml).

Firstly, 0.549 g of zinc acetate, 0.744g of zinc nitrate, and 0.35 g of hexamine are added to 50 ml of DI water and stirred on a magnetic stirrer for 1 hour. The mixture is placed into the water bath at 85 ºC for 2 hours. Next, the mixture is left at room temperature for around 30 minutes before being filtered to obtain the solid white precipitate. Then, the precipitate is baked in the oven at 100 ºC for 1 hour and is annealed at 400 ºC for 3 hours. Lastly, the obtained powder is grounded until its appearance is of fine powder

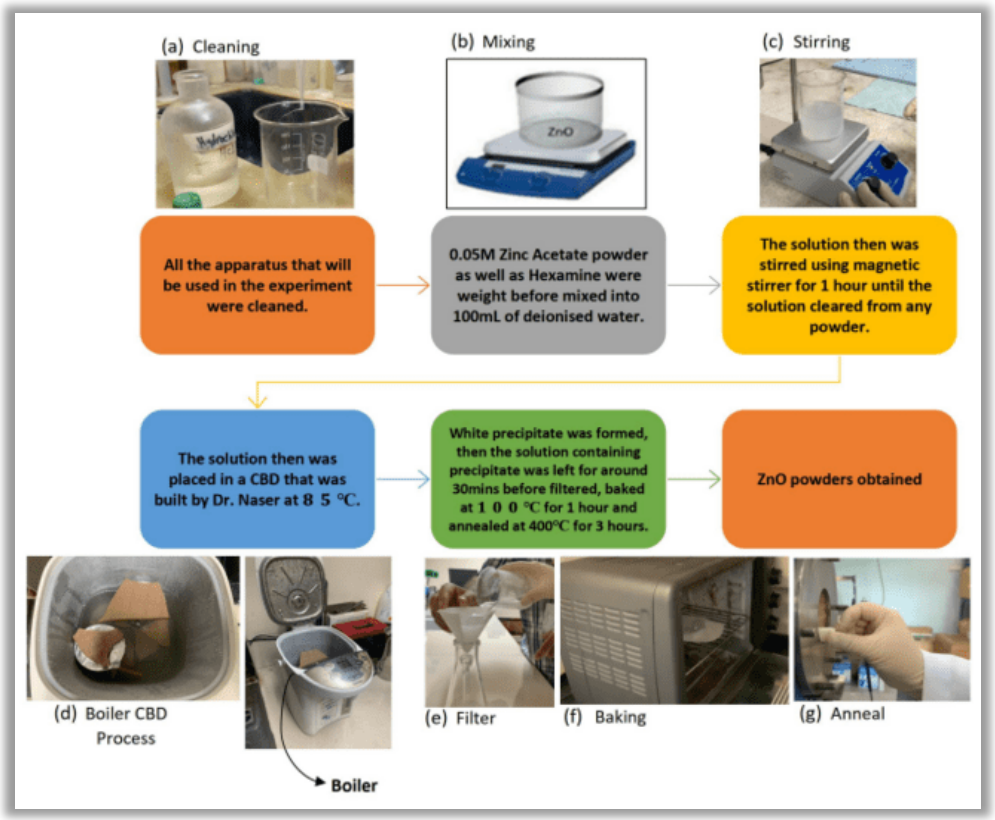

Figure 6: Flowchart on the methodology of the Chemical Bath Deposition synthesizing method

2.2 Laser-Assisted Chemical Bath Deposition (LACBD)

Figure 7 below shows the step of synthesizing ZnO nanoparticles by the Laser-Assisted Chemical Bath Deposition (LACBD). The materials used are 0.5M of Zinc Acetate/Zinc Nitrate, 0.5M of Hexamine, and 100 ml of Deionized water (DI). The amount needed are calculated using equation (1).

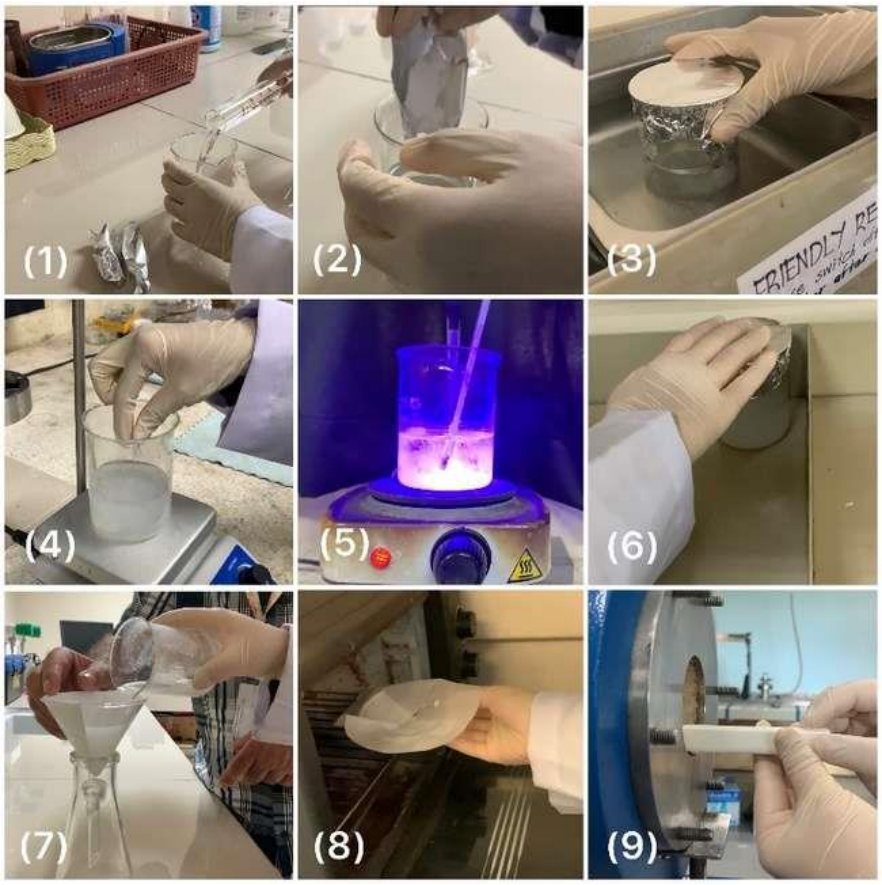

Figure 7: Steps for synthesizing of catalyst by LACBD method.

Firstly, 1.10 g of zinc acetate, 1.49 g of zinc nitrate, and 0.70 g of hexamine are added into 100 ml of DI water and sonicated for 10 minutes at room temperature. The mixture is then irradiated to a laser of wavelength 441 nm, alongside being heated up to a temperature of 65 ºC for 2 hours, resulting in a white precipitate being formed. The temperature of the mixture is measured by a thermometer. The final solution then is left to cool down for about 30 minutes before being filtered. This is followed by being baked at 100 ºC for 1 hour and annealed at 400 ºC for 3 hours. The obtained solid then is ground into a fine powder.

2.3 Preparation of Methylene Orange

For the preparation of methylene orange (MO) solution, 2 mg of methylene orange powder is added to 100 ml of DI water. The solution is then sonicated for 5 minutes. The resulting solution will have a concentration of 20 ppm. The solution is then divided into 2 batches, whereby 50 ml of the solution is tested using ZnO nanoparticles synthesised using the CBD method, and the other 50 ml is tested using ZnO nanoparticles synthesised using LACBD.

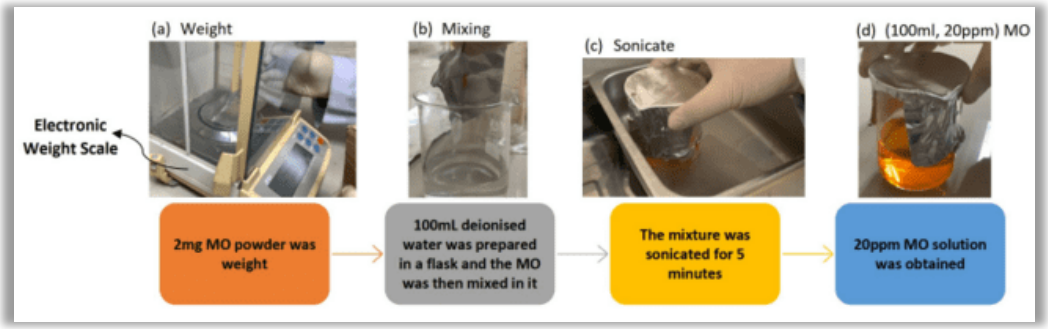

Figure 8: Flowchart on the methodology of Methylene Orange solution preparation.

2.4 Characterizations

The morphology of the synthesised ZnO NPs is characterized using a Field Emission Scanning Electron Microscope (FESEM- JSJ6460 LV Energy, accelerating voltage: 30 kV). Energy Dispersive X-ray (EDX) was performed on the samples in conjunction with FESEM to determine the elemental composition of the synthesised ZnO NPs. Structural analysis was performed using X-ray analysis using a diffractometer (PANalytical X'Pert Pro MRD PW3040). The X-ray radiation source is monochromatized Cu Kα (λ = 1.542 Å) at 40 kV and 35 mA. The range of the scan is 20-80°.

The photocatalytic activity of ZnO NPs synthesised using both CBD and LACBD techniques was assessed by measuring the degradation of MO at regular intervals under constant room temperature conditions. A catalyst content (100 mg ZnO) was used in a medium beaker containing 50 ml of MO solution with an initial concentration of 20 ppm. UV-light irradiation at a wavelength of 385 nm was initiated. The dye solution was sampled every 15 minutes. Photocatalytic analysis was conducted using a UV-Vis spectrophotometer in absorbance mode. The absorption peak of MO was measured at 464 nm. Photodegradation efficiency (%) was calculated based on the maximum photodegradation observed at each interval. The photodegradation rate constant, k, was determined for all samples. A control graph was generated by excluding UV-light irradiation to assess the effect of absorption alone.

3. RESULTS AND DISCUSSION (10 PT)

3.1 Structural Properties of ZnO synthesize by CBD and LACBD

3.1.1 Field Emission Scanning Electron Microscopy (FESEM)

Figure 9 shows the FESEM images of ZnO NPs synthesised using (a) CBD and (b) LACBD methods. As shown in Figure 9 (a), the ZnO NPs synthesised using the CBD method showed a more spherical morphology, which was in agreement based on previous research by El-Bindary et al. [20]. Some of the ZnO NPs can be seen to have a hexagonal shape. The average particle diameter was calculated to be 1.5431µm. Whereas for Figure 9 (b), the ZnO NPs synthesised using the LACBD method showed a more packed structure, losing its spherical morphology. Furthermore, this synthesis method created ZnO NPs with a morphology that showed a larger specific exposed area [21] and thus provided better photocatalytic degradation.

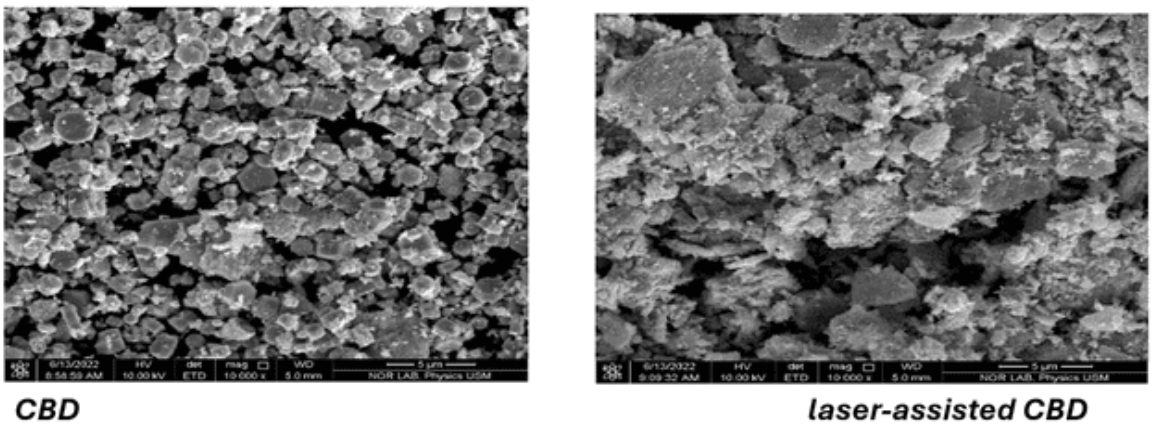

Figure 9: FESEM images of ZnO NPs synthesised using (a) CBD and (b) LACBD methods.

3.1.2 Energy Dispersive X-ray (EDX)

Energy-dispersive X-ray analysis (EDX) is founded on the principle that each element's unique atomic structure generates a characteristic set of peaks in its X-ray spectrum [22]. The EDX instrument was utilized to characterize, analyze, and determine the surface morphology, structure, and elemental composition of the samples [23]. Figure 10 shows the EDX spectra of the ZnO NPs synthesised using (a) CBD and (b) LACBD methods and their corresponding atomic and weight composition. The ZnO NPs obtained using the CBD method showed an elemental composition of zinc (Zn) at 58.29 % and oxygen (O) at 41.71 %, with a Zn:O ratio of 1.40. The ZnO NPs obtained using the LACBD method showed an elemental composition of Zn at 55.22 % and oxygen (O) at 44.78 %, with a Zn:O ratio of 1.23. For ZnO NPs synthesised using the LACBD method, their Zn:O ratio showed a closer value to 1:1 compared to NPs synthesised using the CBD method [23]. This indicates that the sample contains a sufficient amount of oxygen vacancies, which is crucial to improve photocatalytic efficiency [24].

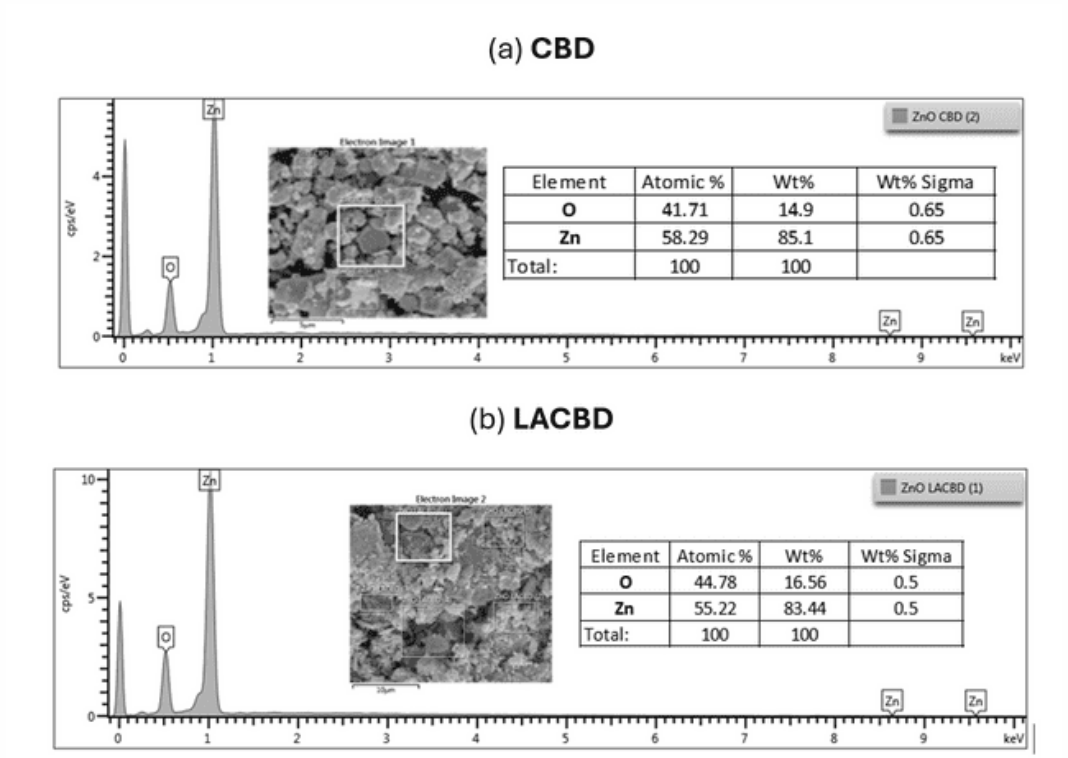

Figure 10: EDX analysis on (a) ZnO synthesized by CBD, and (b) ZnO synthesized by laser-assisted CBD.

3.1.3 X-ray diffraction (XRD)

Figure 11 shows the XRD patterns of ZnO NPs synthesised using CBD (top) and LACBD (bottom). This finding aligns with previous research conducted by El-Bindary et al. and S.H.Zyoud et al. [22, 23]. As illustrated in Figure 11, the XRD diffraction peaks of the synthesized samples exhibit a wurtzite structure with hexagonal crystal lattice parameters, matching the reference (JCPDS card no. 01-080-0075). Pure ZnO typically displays three prominent peaks at 2θ = 31.728°, 34.400°, and 36.212°, corresponding to the (100), (002), and (101) planes respectively. Additional peaks observed include (102), (110), (103), (200), (112), and (201). The absence of impurity peaks in the diffraction patterns suggests highly crystalline samples with excellent crystal quality, indicating high purity of the final products and the absence of undesired reactions during the synthesis process [23, 25]. Pure ZnO nanostructures demonstrate a strong (002) preferred orientation, indicating preferential growth along the c-axis, as observed in the ZnO sample synthesized by LACBD. This result suggests a single-crystalline structure with vertical growth on the substrate.

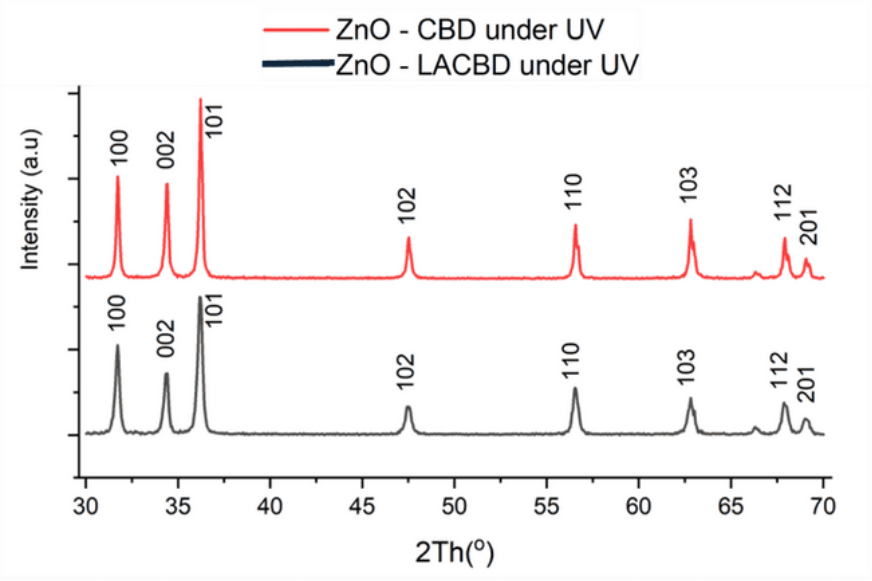

Figure 11: XRD patterns of ZnO NPs synthesised using CBD (top) and LACBD (bottom).

3.2 Photocatalytic activity

3.2.1 UV-Visible (UV-VIS) Spectrophotometry

Figure 12 shows the UV-Vis absorption curves of MO, which undergoes photocatalysis degradation under UV light using ZnO NPs synthesised using (a) CBD and (b) LACBD. The experimental results demonstrate that the morphology of the synthesized ZnO nanomaterials significantly influences their photocatalytic activity. Referring back to Figure 10, ZnO NPs synthesized by LACBD exhibit the largest specific surface area compared to the other two. Consequently, ZnO NPs synthesized by LACBD significantly accelerated MO degradation, as evident in both graphs. MO degradation by ZnO NPs synthesized by LACBD reached 71% completion, while 16% was observed for ZnO synthesised by CBD, both within 30 minutes.

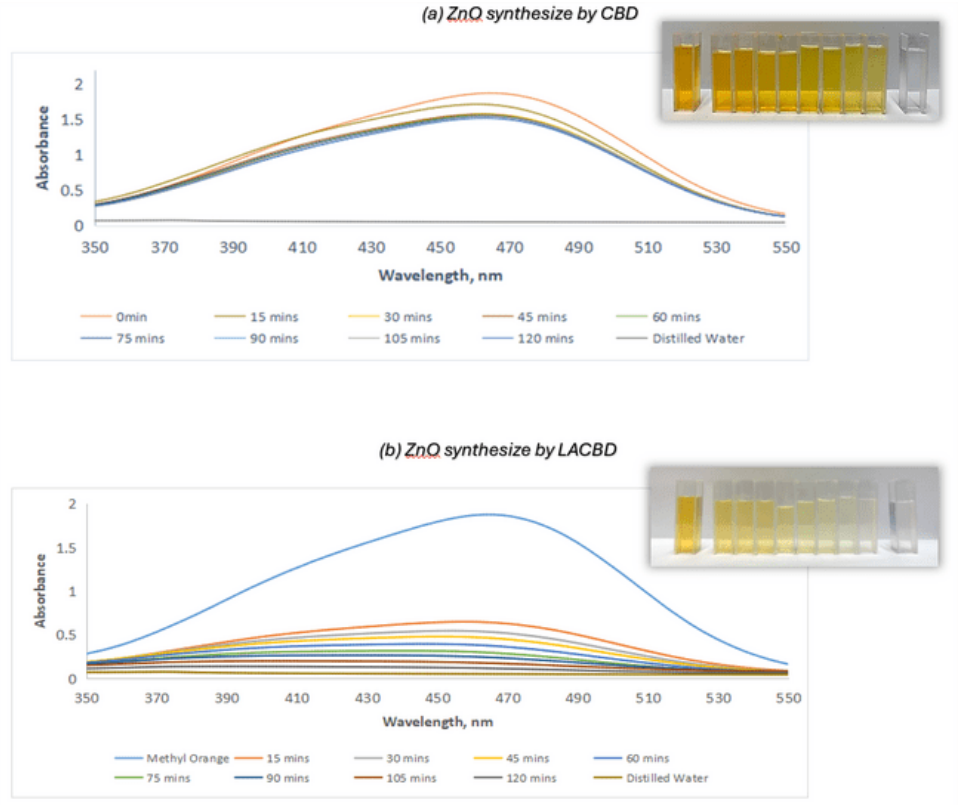

Figure 12: UV-Vis absorption curves of MO, which undergoes photocatalysis degradation under UV light using ZnO NPs synthesised using (a) CBD and (b) LACBD. The inset of each graph shows the photographs taken of the MO solution after each subsequent 15 minutes.

4. CONCLUSION

In conclusion, ZnO NPs have been successfully synthesized using both CBD and LACBD methods. Observations revealed that ZnO NPs synthesized by LACBD possess a larger specific exposed area, ideal for light absorption applications, with a fine flat-shaped morphology resembling nanosheets. EDX analysis showed ZnO NPs synthesised using LACBD obtained a Zn:O ratio closer to 1:1, indicating sufficient oxygen vacancies important for photocatalyst degradation. Lastly, ZnO synthesized by LACBD exhibited the highest degradation efficiency of 71% within 30 minutes, corroborated by UV-Vis measurements vs the CBD method at 16 %.

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الخلاصة

الصبغة العضوية هي ملوث صناعي رئيسي وتحتاج إلى طرق مبتكرة لإزالتها. أكسيد الزنك (ZnO) هو مادة جذابة للتحلل الضوئي للأصباغ العضوية. في هذا البحث، تمت دراسة النشاط التحفيزي الضوئي لجسيمات نانوية من أكسيد الزنك (ZnO NPs) المصنعة باستخدام الترسيب الكيميائي للحمام (CBD) والترسيب الكيميائي بمساعدة الليزر .(LACBD) أظهر المجهر الإلكتروني الماسح للانبعاث الميداني (FESEM) أن جسيمات نانوية من أكسيد الزنك المصنعة باستخدام LACBD حصلت على بنية أكثر حزماً تشبه صفائح النانو. وهذا يترجم إلى مساحة مكشوفة محددة أكبر. تُظهر دراسة الأشعة السينية المشتتة للطاقة (EDX) أن جسيمات نانوية من أكسيد الزنك المصنعة باستخدام طريقة LACBD أظهرت نسبة Zn:O أقرب إلى 1:1، مما يشير إلى تركيز كافٍ من شواغر الأكسجين، والتي تعد ضرورية للتحلل الضوئي. لقياس تحلل المحفز الضوئي، يتم استخدام جسيمات أكسيد الزنك النانوية كمحفزات في تطبيقات تحلل الماء، حيث تمت إضافة 0.01 جرام من أكسيد الزنك إلى 50 مل من محلول الميثيلين البرتقالي بتركيز 20 جزء في المليون. تمت دراسة تركيز محلول الميثيلين البرتقالي باستخدام قياسات الأشعة فوق البنفسجية المرئية أثناء تحلل المحفز الضوئي. أظهرت جسيمات أكسيد الزنك النانوية المصنعة باستخدام LACBD أعلى أداء تحلل ضوئي تحت ضوء الأشعة فوق البنفسجية، مع أعلى ثابت لمعدل التحلل، k، 0.02521دقيقة-1 وكفاءة تحلل ضوئي 71٪ في غضون 30 دقيقة.