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RESEARCH PAPER

Co-precipitation Synthesis of $Cu_{0.25}Zn_{0.75}Cr_2O_4$ Nanoparticles as an Efficient Visible Light Photocatalyst for Degradation of Methylene Blue Dye

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ABSTRACT

Photocatalytic removal of organic pollutants requires an efficient photocatalyst of operating under visible light irradiation. In this work, synthesis of $Cu_{0.25}Zn_{0.75}Cr_2O_4$ nanoparticles (CZCO) was reported using the facile co-precipitation method. The CZCO nanoparticles exhibited a cubic phase with an average crystallite size of about 14.61 nm. Also, Field emission electron microscopy (FESEM) analysis confirmed a nanospherical morphology with particle sizes below 50 nm. The nanoparticles showed a strong visible light absorption with an optical band gap of 2.47 eV. The photocatalytic performance was examined by degradation of an aqueous solution of methylene blue (MB). More than 92% of the MB solution was degraded using 0.04 g of the nanoparticles under visible-light irradiation for 180 min. Studying of key parameters, including pH of MB solution and concentration of H_2O_2 oxidants, showed that alkaline dye solution (pH = 9) and 0.02 molar of oxidant enhanced the degradation efficiency to 95% and 99%, respectively. Furthermore, recyclability tests over six successive reaction cycles demonstrated the good reusability and structural stability of the synthesized CZCO nanoparticles.

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INTRODUCTION

In the 21st century, the development of practical approaches for removing of contaminants from water resources has become essential [1, 2]. The scarcity of clean and hygienic water has intensified over recent decades due to increasing industrial and agricultural activities [3, 4]. Consequently, environmentally sustainable wastewater treatment methods that generate fewer toxic by-products, exhibit higher efficiency, and operate at

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lower operational costs are receiving increasing attention [5, 6].

Among advanced treatment technologies, photocatalysis has introduced a revolutionary approach in water purification, owing to its potential capabilities for removing organic and inorganic pollutants form water media [7, 8]. Photocatalytic reactions exploit a renewable energy source—sunlight—to activate a photocatalyst material having a band gap associated with the energy of incident light [9, 10].

Although numerous materials have been investigated as photocatalysts, only a limited compounds demonstrate efficient activity under visible light irradiation [11]. Most photocatalysts suffer from low quantum efficiency due to the fast recombination of the photogenerated electrons and holes [12]. In order to enable photocatalysts to function effectively under the visible light, several strategies have been suggested, including incorporation of impurities (metallic or non-metallic ions) into crystalline structure of the photocatalysts [9, 13], combination of two or more photocatalyst materials [14, 15], and coupling photocatalysts with carbon nanomaterials [16, 17].

In this regard, many works have been published in literature relating to removal of contaminants using the visible-light photocatalysts. For example, Rahbar et al. introduced the photocatalytic system comprising Fe, and B co-doped TiO_2/CNT@WO_3 for treating the petrochemical wastewater under natural sunlight irradiation [18]. Derkaoui et al. reported $MnCo_2O_4$ spinel as the visible-light photocatalyst for removing rhodamine B [19]. Prasad et al. reported visible-light degradation of methylene blue using synthesized Cu doped triphasic TiO_2 [20]. In addition, Z-scheme Cu_2O@Ag/*g*-C_3N_4 heterojunctions was reported for the visible-light photocatalytic degradation of tetracycline [21].

In this work, we have synthesized $Cu_{0.25}Zn_{0.75}Cr_2O_4$ nanoparticles using facile and straightforward co-precipitation reaction. Co-precipitation method is recognized as simple and cost-effective synthetic route for preparation of fine metal oxide and mixed metal oxide nanoparticles [22, 23]. The $Cu_{0.25}Zn_{0.75}Cr_2O_4$ nanoparticles (CZCO), as a mixed metal oxide compound, consist of three transition metal oxides that synergistically encourage the photocatalytic activity by facilitating electronic transitions between energy levels of the components. For instance, Ahmed et al. suggested that the electron transfer from TiO_2 conduction band to conduction band of Cr_2O_3 prolongs the lifetime of the electron/hole pairs, and thereby increasing the photocatalytic activity [24].

Herein, the visible-light photocatalytic capacity of the nanoparticles was assessed by diffuse reflectance spectroscopy. The visible-light photo-efficiency of the CZCO nanoparticles was studied for removal of methylene blue (MB). Some influencing factors, including pH of MB solution, loaded amount of CZCO nanoparticles, and concentration of oxidant agent (H_2O_2), on the photocatalytic efficiency of the CZCO nanoparticles were carefully investigated.

MATERIALS AND METHODS

Synthesis of $Cu_{0.25}Zn_{0.75}Cr_2O_4$ nanoparticles (CZCO)

The $Cu_{0.25}Zn_{0.75}Cr_2O_4$ nanoparticles were synthesized via co-precipitation reaction, as following: Initially, 1.0 g of polypropylene glycol (PPG), serving as a capping agent, was dissolved in 50 mL of deionized water. Subsequently, 0.25 mmol of $Cu(NO_3)_2.3H_2O$, 0.75 mmol of $Zn(NO_3)_2.6H_2O$, and 2 mmol of $Cr(NO_3)_3.9H_2O$ were introduced into the solution, followed by continuous stirring for 30 min. Next, pH of the solution was set to around 9 by adding NH_4OH solution (0.1 M), and stirring was continued for 30 min. The precipitated solid was collected by centrifugation at 5000 rpm for 15 min and subsequently rinsed twice with a 10% (V/V) ethanol/deionized mixture. The product was then subjected to oven drying at 100 °C for 5 h. finally, the product was calcined at 600 °C for 5 h to form the pure and crystalline brown powder.

Characterization

Crystallinity of the synthesized CZCO nanoparticles was investigated using X-ray diffraction pattern by Philips diffractometer Pro MPD at 2ϴ= 10°-80°. Surface properties of the CZCO nanoparticles were studied using field emission scanning electron microscope (FESEM) by TESCAN MIRA 3. Optical properties of the CZCO nanoparticles were studied using diffuse reflectance spectroscopy (DRS) (JASCO V-670).

Photocatalytic experiments

To investigate the visible light photocatalytic activity of the synthesized CZCO nanoparticles, the degradation of methylene blue (MB) in aqueous solution was selected as the model reaction. The visible light irradiance was supplied using a white color LED lamp (50 W).

Different amounts of the nanoparticles (0.02, 0.04, and 0.06 g) were used to conduct the photocatalytic reaction for degradation of constant concentration of MB solution (50 ppm). To obtain the higher photocatalytic efficiency, reaching an adsorption/desorption equilibrium between the MB molecules and CZCO nanoparticles is imperative. Hence, the photocatalytic reaction was firstly carried out in the darkness under continuous stirring for 30 min to achieve this goal. Then, the photocatalytic process was initiated under the visible light irradiation for 180 min. The photocatalytic degradation efficiency of MB was determined at 30-min intervals by recording the UV/Vis absorption spectra at its characteristic maximum absorption wavelength of 664 nm.

RESULTS AND DISCUSSION

Structure and morphology

The crystallinity of the CZCO nanoparticles was studied using the XRD pattern, shown in Fig. 1. The observed diffraction peaks at 2*Θ* values of 18.4°, 30.3°, 35.7°, 37.4°, 43.4°, 47.6°, 53.9°, 57.5°, 63.1°, 66.4°, 67.4V, 71.6°, 74.7°, 75.7° are in agreement with the reported reflections for the cubic structure of chromium copper zinc oxide (JCPDS file no 01-076-1566). Since the synthesized CZCO nanoparticles crystalized in the cubic structure ($a = b = c$; $\alpha = \beta = \gamma = 90°$), the lattice constant (a) was calculated to be 8.24 Å using following equation (Eq. 1):

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad (1)$$

, where d is the interplanar space, a is lattice constant, and h, k, l are miler indices.

Also, the average size of the crystallites was determined using the Scherrer equation [25], which was found to be around 14.61 nm.

The FESEM micrograph in Fig. 2a displays the spherical structure of the CZCO nanoparticles, with an average particle size below 50 nm. Additionally, the nanoparticles were elementally investigated using the EDX spectroscopy (Fig. 2b), which was revealed that the synthesized nanoparticles are consisted of Cu (5.39 wt%), Zn (20.25 wt%), Cr (45.21 wt%), and O (29.15 wt%).

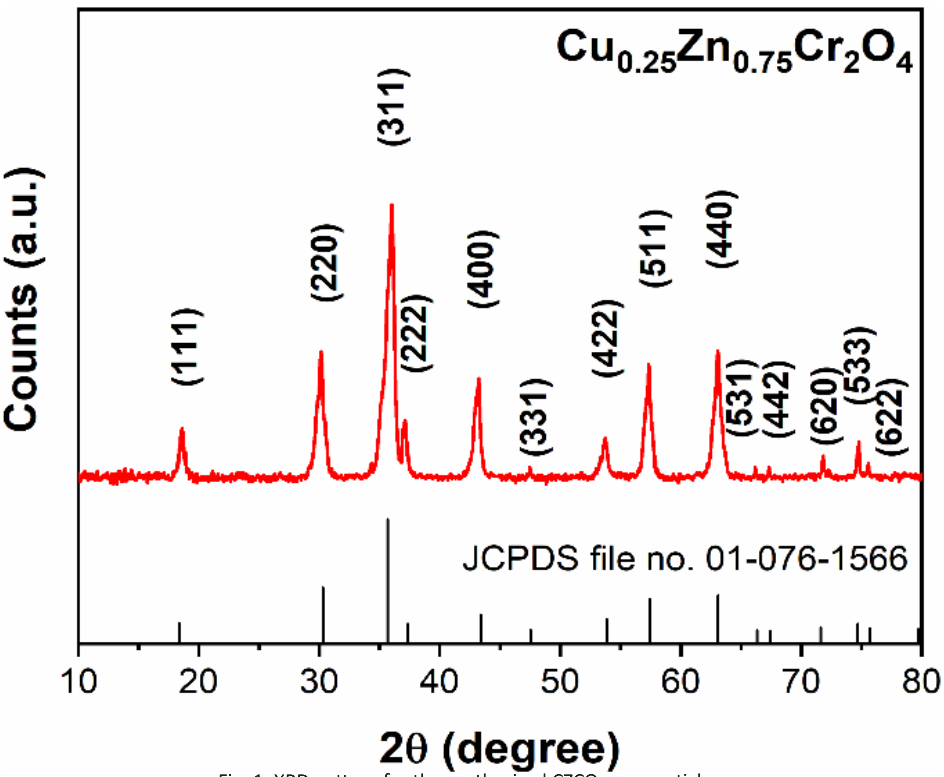

Fig. 1. XRD pattern for the synthesized CZCO nanoparticles.

Optical properties

The UV/Vis spectrum of the CZCO nanoparticles was depicted in Fig. 3, confirming that the CZCO nanoparticles possess the noticeable absorption in the visible region. Also, the calculated optical band gap using the Tauc method [26] was depicted in the inset to the Fig. 3. The optical band gap value (2.47 eV) clearly confirms the capacity of the synthesized CZCO nanoparticles to act as the efficient visible light photocatalyst. This results reveals that the interactions of different energy levels of different metal oxides can significantly enhance the visible-light sensitivity and increase the photocatalytic efficiency [27-29].

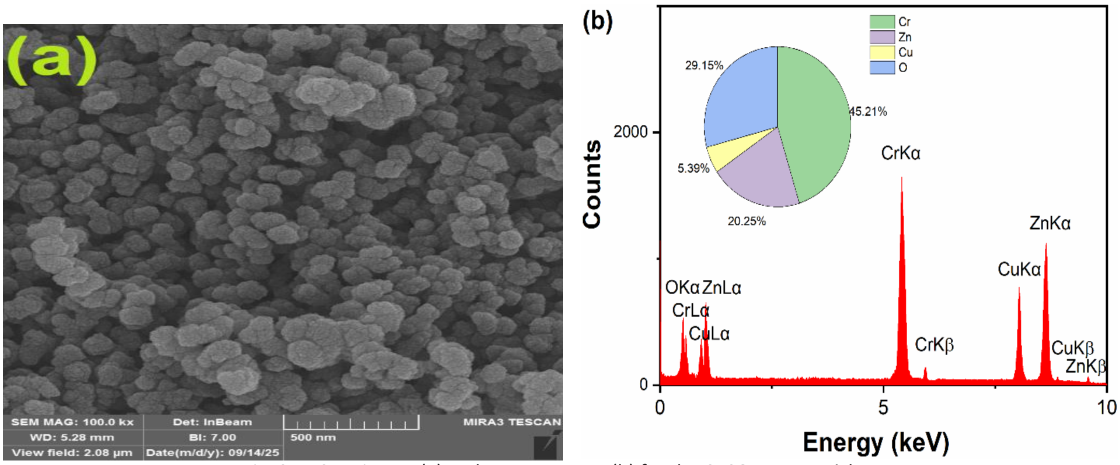

Fig. 2. FESEM image (a) and EDX spectrum (b) for the CZCO nanoparticles.

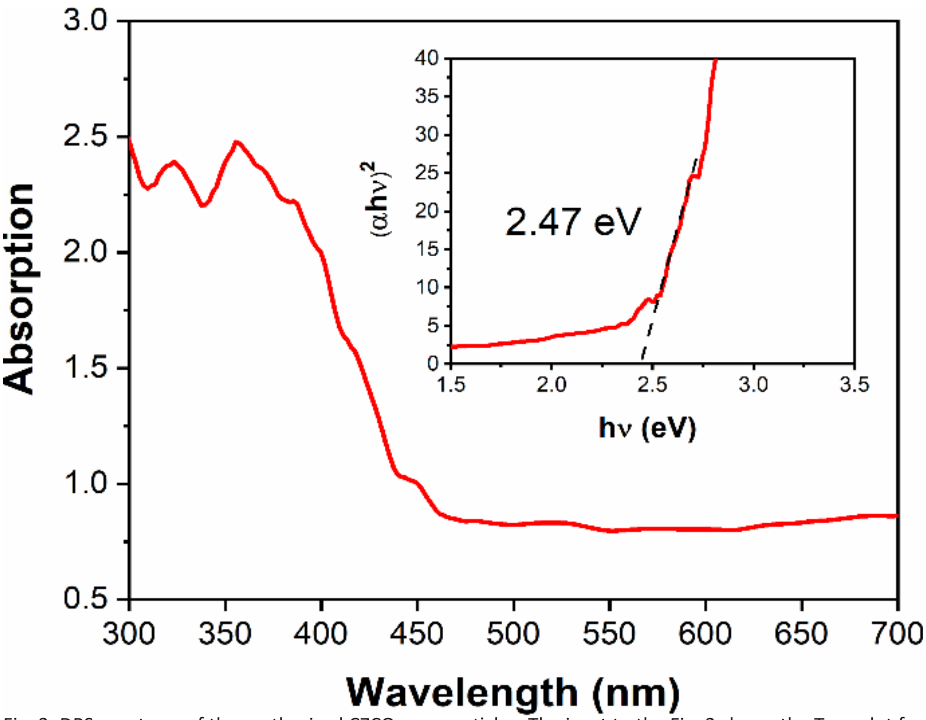

Fig. 3. DRS spectrum of the synthesized CZCO nanoparticles. The inset to the Fig. 3 shows the Tauc plot for determining the optical band gap of CZCO nanoparticles.

Photocatalytic experiments

The photocatalytic removal of the MB solution using various dosages of the CZCO nanoparticles (0.02, 0.04, and 0.06 g) was presented in Fig. 4a. As can be seen, the highest degradation level (92.7%) of the MB solution was achieved with 0.04 g of the photocatalyst. However, the degradation efficiency decreased to 83.4% using 0.06 g of the synthesized photocatalyst. This can be explained by the excessive cloudiness of the suspension, which limits light transmission through the solution [30-32]. In addition, UV/Vis spectra of the degraded MB solution under different irradiation time in the presence of the 0.04 g of the CZCO nanoparticles was shown in Fig. 4b. The absorption of the MB solution markedly decreased after 180 min visible-light illumination upon 0.04 g of the photocatalyst.

Effect of pH of MB solution

To study the dependence of the photocatalytic activity on the pH of the MB solution, the degradation reactions were conducted under different pH conditions, including 3, 5, 7, 9, and 11. As shown in Fig. 5, the MB solution is degraded to more than 95.2% at the pH of 9. However, the photocatalytic efficiency decreased to 71.4%

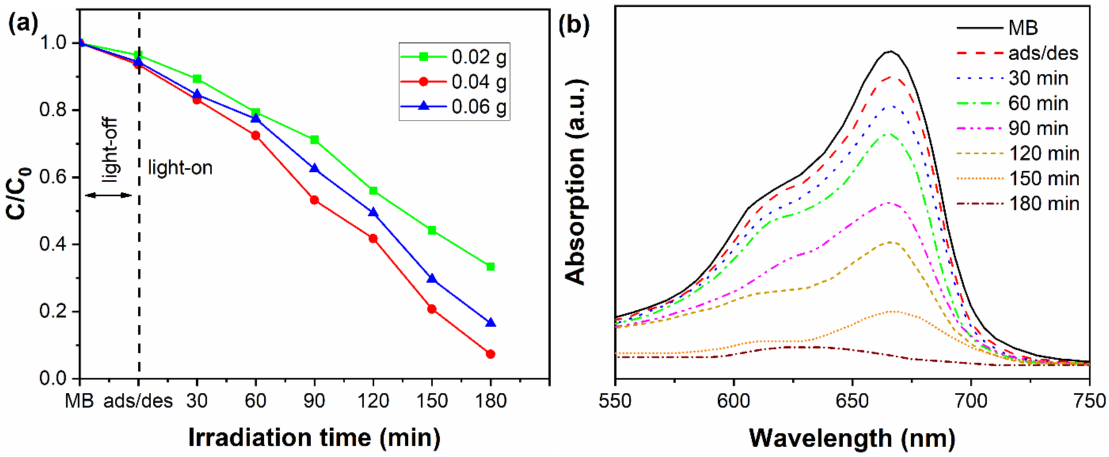

Fig. 4. Photocatalytic degradation of MB solution using different amounts of CZCO nanoparticles (a), UV/Vis spectra of MB solution under different irradiation time (b).

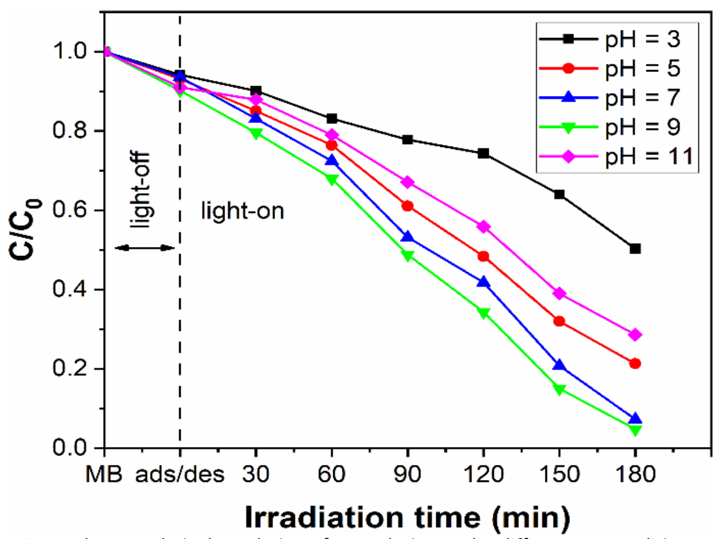

Fig. 5. Photocatalytic degradation of MB solution under different pH conditions.

at the pH of 11. Moreover, the photocatalytic degradation level under acidic conditions is not as high as the alkaline conditions. So that, the MB solution was degraded only to 49.7% at the pH of 3. To reason these observations, it can be concluded that the MB solution, as a cationic dye [33, 34], has the great tendency to attract onto the negatively charged CZCO surface at alkaline condition (pH = 9). As the photocatalytic process is a surface-dependent reaction, increased adsorption of dye molecules facilitates greater photocatalytic efficiency. Because of the photocatalytic reaction primarily occurs at the photocatalyst-solution interface, higher adsorption rates directly promote better performance [35].

Effect of H_2O_2

The photoactivity of the CZCO nanoparticles

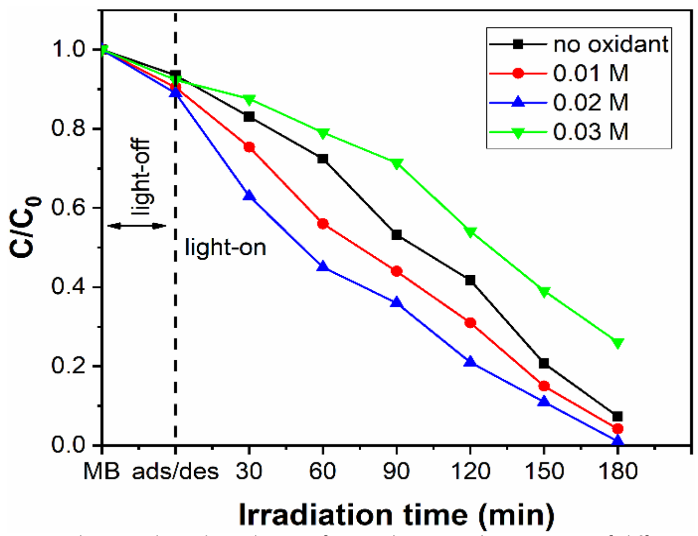

Fig. 6. Photocatalytic degradation of MB solution in the presence of different concentrations of H_2O_2.

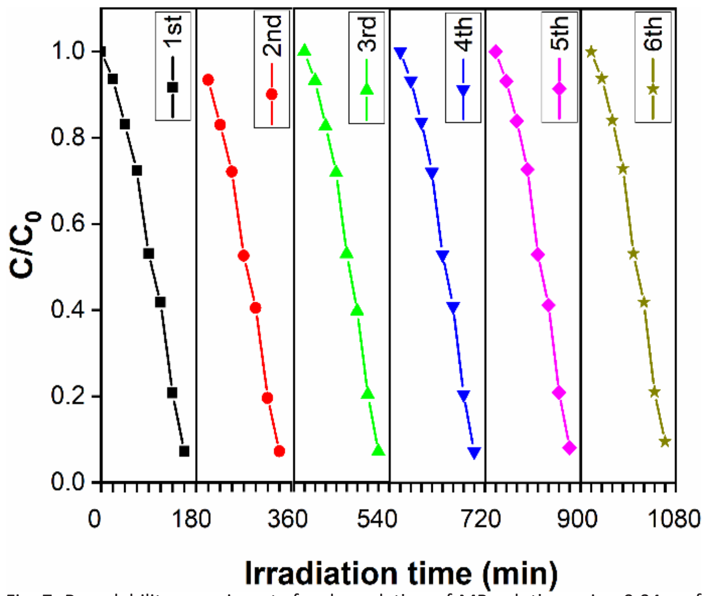

Fig. 7. Recyclability experiments for degradation of MB solution using 0.04 g of CZCO photocatalyst.

was evaluated in the presence of different concentrations of H_2O_2 as an oxidant agent (0.01, 0.02, 0.03 M), as shown in Fig. 6. As can be seen, the MB solution is completely degraded using 1 mL of H_2O_2 (0.02 M). However, increasing the H_2O_2 concentration to 0.03 M led to the significant decline (73.9%) in the removal efficiency. These results confirm that the addition of proper amount of the H_2O_2 enhances the production of reactive radicals during the photocatalytic reaction [36]. Nevertheless, at concentrations exceeding the optimal level, the photocatalytic performance decreases due to the scavenging of the radicals by excess H_2O_2 [37].

Recyclability experiments

Fig. 7 shows the recyclability of the synthesized CZCO nanoparticles for the degradation of MB solution. The reactions were repeated over 6 successive cycles under constant experimental conditions. After each cycle, the photocatalyst particles were recovered by centrifugation at 7000 rpm for 15 min. The collected nanoparticles were subsequently rinsed with the 10% (V/V) ethanol/deionized water solution, followed by drying at 90 °C for 5 h in an oven.

As seen in Fig. 7, the synthesized CZCO photocatalyst exhibits the great recyclability over 6 reaction cycles, confirming the insignificant decline in the photocatalytic activity. Notably, the photocatalytic degradation level of the MB solution remained almost constant after 4th reaction, and the decrement in the efficiency was only about 2.28 % after 6th reaction. This noticeable recyclability can be explained by the structural stability of the CZCO nanoparticles, which was confirmed by providing the XRD patterns for the pristine and reused CZCO nanoparticles. As seen in Fig. 8, no evident changes are observed in the XRD patterns for the CZCO nanoparticles used in the 6 consecutive reaction cycles compared to the pristine nanoparticles.

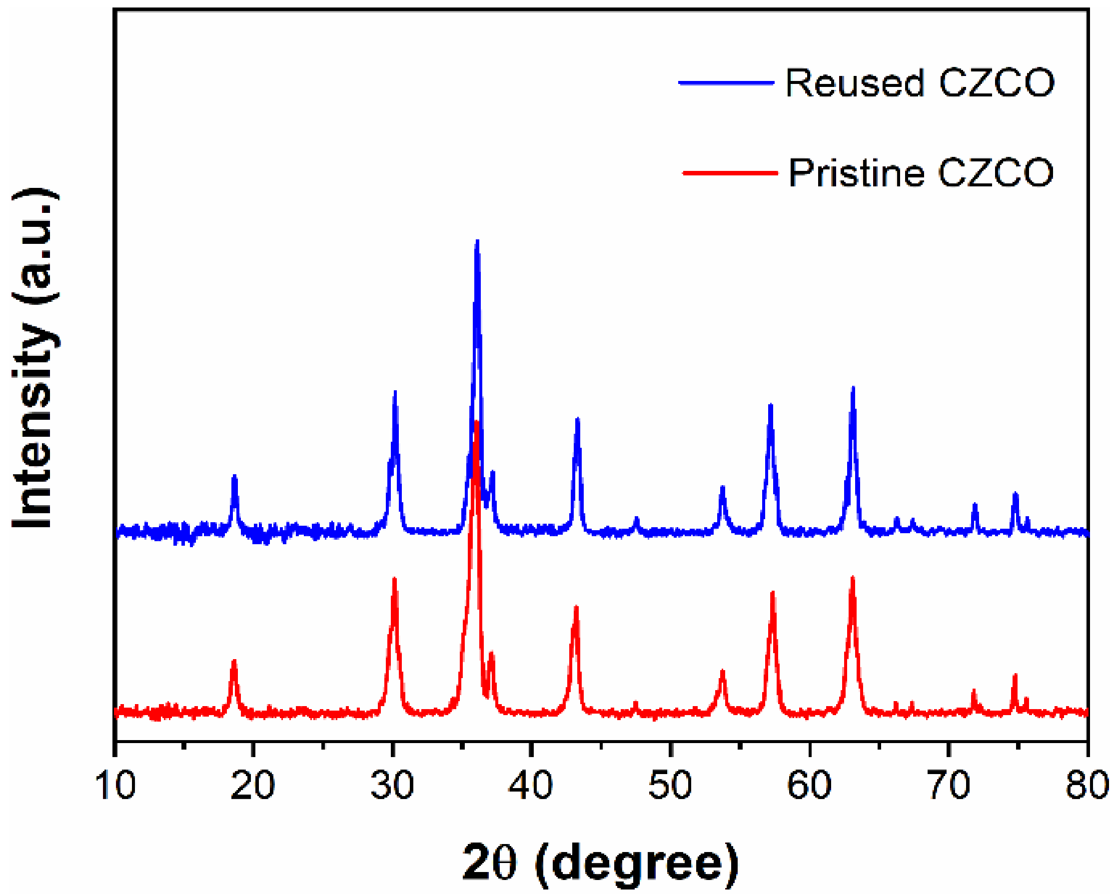

Fig. 8. XRD patterns for CZCO nanoparticles for before and after reusability experiments.

CONCLUSION

The $Cu_{0.25}Zn_{0.75}Cr_2O_4$ nanoparticles (CZCO) were successfully synthesized using simple co-precipitation reaction. The FESEM image confirmed the formation of nano-sized spherical particles. Through the optical examination using DRS spectroscopy, it was found that the band gap of the CZCO nanoparticles is in the range of the visible light. Therefore, the visible-light photoactivity was suggested for the CZCO photocatalyst. The photocatalyst activity of the CZCO nanoparticles was studied using degradation of the MB solution under visible-light irradiation from the LED lamp (50 W) for 180 min. It was observed that over 92% of the MB solution was degraded using 0.04 g of the synthesized photocatalyst. Furthermore, the enhancement of the degradation level was achieved under alkaline condition (pH = 9) and addition of 0.02 M of H_2O_2 oxidant. Additionally, the CZCO nanoparticles possessed the high structural stability which reflected in the recyclability experiments, showing the insignificant decrement in the photocatalytic activity after 6 successive reaction cycles.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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