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Green synthesis of p-Co$_3$O$_4$/n-ZnO composite catalyst with Eichhornia Crassipes plant extract mediated for methylene blue degradation under visible light irradiation

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Abstract
The water pollution due to industrial effluents causes a great health problem. Hence, it is important to treat wastewater before discharging to the environment. In this work, water hyacinth (Eichhornia Crassipes) plant extract mediated ZnO, CO$_3$O$_4$, and p-Co$_3$O$_4$/n-ZnO composite catalysts were synthesized by green co-precipitation routes. The resulting samples were characterized by x-ray diffractometer (XRD), scanning electron microscope (SEM), Fortier transform infrared (FT-IR), and with other instruments. The catalytic activities of ZnO, CO$_3$O$_4$, and Co$_3$O$_4$/ZnO were tested for MB dye degradation under visible light irradiation. The catalytic degradation of MB with p-Co$_3$O$_4$/n-ZnO composite catalyst was 95.5%; while 72% and 79% of MB dye was degraded by ZnO and Co$_3$O$_4$ catalysts, respectively. The kinetic rate constants (k) in the degradation of MB dye with ZnO, Co$_3$O$_4$, and p-Co$_3$O$_4$/n-ZnO composite catalysts were also 0.014 min$^{-1}$, 0.018 min$^{-1}$, and 0.028 min$^{-1}$, respectively. The results showed that the presence of plant extract during the synthesis of the catalysts makes the catalyst more active and enhances the catalytic performances. Moreover, the formation of p-n junction in the p-Co$_3$O$_4$/n-ZnO catalyst also facilitates the photogenerated electron–hole separation and further enhances the catalytic efficiency. Hence, the formation of p-n junction is the key factor for enhancing the photodegradation of MB dye under visible light irradiation and the plant extract mediated catalyst synthesis also further improves its performance.

1. Introduction

Recently, the issues of getting clean water is one of the most challenging phenomenon due to increasing the growth rate of urbanization and industrialization in the globe and it may affect the living things [1]. Particularly, the releasing of hazardous organic pollutants such as dyes, pesticides, chemical warfare agents, phenol, and other organic pollutants from agricultural and industrial areas are the biggest challenges for human beings [2–6]. Due to this reason, it is advantageous to eliminate toxic and harmful pollutants from the polluted environmental areas [7–9]. Many researches have been performed to remove toxic organic and inorganic pollutants [10–12]. However, the reusability, costs, and efficiency in the treatments of wastewater are not still solved [13–15]. Therefore, the design and synthesis of cost effective and stable materials for the removal of toxic organic pollutants are needed.

Currently, photocatalysis is one of the methods used in the removal of toxic organic pollutants from wastewater [16, 17]. For this purpose, titanium dioxide (TiO$_2$) and zinc oxide (ZnO) semiconductor materials have been widely used due to their environmental friendly, photocatalytic activity, stability, and low cost [18–20]. Particularly, the exceptional properties of ZnO semiconductor permits the photocatalytic reactions on its surface effectively [20]. However, the photocatalytic activities of ZnO is limited because of its wider band gap energy (3.37 eV), high recombination rate of the photo-generated electron–hole pairs, narrow light absorption, and poor adsorption capacity [21]. Due to this reason, many researchers tried to modify the catalytic activities of
ZnO with different techniques [22, 23]. Among the techniques reported, the formation of heterogeneous p-n junctions with other semiconductor materials for the purpose of charge carriers separations and band gap engineering are well known [23, 24].

There are also many reports on the synthesis and application of n-type ZnO based semiconductor materials combined with other materials [25–28]. Diez-Pascual et al [29] synthesized the aminated poly(phenylene sulfide) and ZnO nanocomposites materials for medical applications. Silicate-bridged ZnO/BIVO₄ photocatalyst was also reported by Fu et al [30]. Shaf et al [31] also synthesized the NiO—ZnO heterostructures based photocatalyst material. The ZnO/CuO nano composite prepared with green method was also reported by Yulizar et al [32]. The Co₃O₄/ZnO nanocomposites for gas sensing applications was also reported by Bekermann et al [33]. However, the synthesis of green and plant extract mediated for different applications are still needed to improve the catalytic efficiencies.

It is also well known that the green synthesis method of catalysts has got a great attention and the resulting materials have been used in various applications [34–40]. Particularly, the green synthesis of nanomaterials had various benefits in which the method is non-toxic, easy to scale up, cost effective, and environmentally friendly [41–46]. For example, to synthesis metal nanoparticles with the required morphology and size, plant extracts could play a major role thereby acting as a natural reducing, capping, and stabilizing agent [47]. Moreover, using plant extract mediated synthesis of catalyst can also create highly porous materials and high dispersion and specific surface area [48]. This phenomenon will help to enhance the catalyst activities of the materials with facilitating the charge transfer [49].

Researchers also used different plant extracts to enhance the photocatalytic activities of semiconductors. For examples, the green synthesis of p-NiO/n-ZnO nanocomposites was reported for organic pollutant removal from wastewater [49]. The Melissa Officinalis L. leaf extract for the synthesis of CuO/ZnO was used in the reduction of 4-nitrophenol and Rhodamine B [50]. The tunable ZnO nanostructures synthesized with biosynthesis method was also used for catalytic applications [51]. The Ag–Cu decorated ZnO synthesized with green method was also used for the removal of toxic organic and detection of nitrite ions [52]. Moreover, the Eichhornia Crassipes plant has been also used for different application such as in the removal of heavy metal, biofuel production, bio-composite, supercapacitors, water purification and other applications [53–55]. However, there is no a significant research work done on Eichhornia Crassipes plant extract used for the synthesis of n-type ZnO and p-type Co₃O₄ semiconductor nanocomposite for wastewater purification.

In this work, Eichhornia Crassipes plant extract was used as a template/capping agent for the synthesis of plant extract mediated p-Co₃O₄/n-ZnO composites catalysts. The resulting composite catalysts were characterized and used for the degradation of methylene blue (MB) under visible light irradiation. Moreover, a possible degradation mechanism with p-Co₃O₄/n-ZnO composites catalysts was also proposed. It is expected that the synthesis of photocatalyst materials by using Eichhornia Crassipes plant extract as a template/capping for n-type ZnO and p-type Co₃O₄ composite catalysts will have high catalytic activities.

2. Materials and methods

2.1. Chemicals and reagent

Zinc nitrate hexahydrate (Zn(NO₃)₂.6H₂O) (Sigma Aldrich, 99%), sodium hydroxide (NaOH), cobalt nitrate (Co(NO₃)₂.6H₂O (Sigma Aldrich, 98%), and ethanol were used. All chemicals and reagents were used without further purification. Distilled water was also used throughout the experiment.

2.2. Preparation of plant extracts

Eichhornia Crassipes was collected from Koka Lake, Ethiopia. The Eichhornia Crassipes plant was first washed with tap water to remove unwanted dusts from the plant followed by distilled water. Then, the plant was subjected to dry at room temperature and crushed. The resulting 15 g of water hyacinth powder was mixed with 430 ml of distilled water and stirred at 50 °C for 1 h. The solution was filtered using filter paper and the resulting plant extract was stored for further application.

2.3. Synthesis of plant extracted mediated p-Co₃O₄/n-ZnO composite catalyst

Eichhornia Crassipes mediated synthesis of p-Co₃O₄/n-ZnO was prepared through co-precipitation method according to the reported literature [49, 56]. In a particular procedure, 40 ml of Zn(NO₃)₂.6H₂O (0.1 M) and 40 ml of Co(NO₃)₂.6H₂O (0.1 M) solutions were dropped in to 40 ml of plant extract and stirred for 2 h. Subsequently, 2 M of NaOH was dropped to form hydroxide colloidal solution. The solution was centrifuged and washed with distilled water followed by ethanol. The precipitate was dried at 60 °C and calcined at 500 °C for 2 h. For comparison purposes, the plant extract mediated ZnO and Co₃O₄ catalysts were synthesized with the
same procedure mentioned above. Scheme 1 shows the synthesis of n-type ZnO and p-type Co$_3$O$_4$ composite catalyst by using water hyacinth plant extract.

2.4. Characterization of the samples
The phase and crystallinity of ZnO, Co$_3$O$_4$ and p-Co$_3$O$_4$/n-ZnO composites samples were determined by x-ray diffraction (XRD) (Shimadzu XRD-7000) with Cu Kα radiation. The morphologies of the samples were also examined by scanning electron microscopy (SEM) equipped with EDX (COXIEM-30). Fourier transform infrared (FTIR) analysis was done by using Spectrum 65 FT-IR(PerkinElmer) in the range 4000–400 cm$^{-1}$ using KBr pellets. Shimadzu–3600 Plus UV–vis spectrophotometer was used for evaluating the degradation of MB.

2.5. Photocatalytic measurements
The photocatalytic activity measurements were performed according to the method report with modification [57]. In a particular procedure, 130 ml of MB dye (10 mg l$^{-1}$) solution was used as a pollutant. Then, 20 mg of the catalyst was dispersed in to the pollutants under stirring for 30 min in dark for adsorption–desorption equilibrium confirmation between the catalyst and pollutant. After 30 min stirring under dark, light was on and halogen lamp (250 W) was used as visible light sources. For kinetic studies, 5 ml aliquot with interval of time was taken and centrifuged. Then, the aliquot was measured with UV–vis spectrophotometer.

The stability of the p-Co$_3$O$_4$/n-ZnO photocatalyst was also checked and 77 mg of the catalyst was dispersed in to 500 ml MB (10 mg/l) solution. The mixture solution was stirred under dark for 30 min and exposed to light for 60 min. Then, 5 ml of aliquot was taken for UV–vis measurement. The remaining solution was separated from the catalyst and the supernatant solution was removed by decantation. The catalyst remained in the bottom of the beaker left was reused for the next photocatalytic reaction run with similar procedure as mentioned above.

3. Results and discussion

The crystalline structures of ZnO, Co$_3$O$_4$, and p-Co$_3$O$_4$/n-ZnO samples were characterized by using XRD and showed in figure 1. As it is showed in figure 1(a), ZnO hexagonal (wurtzite) was observed which had a good agreement with reported results (JCPDS 36-1451) [58]. Moreover, the XRD peaks in figure 1(b) located at 19.0°, 31.3°, 36.8°, 38.7°, 44.9°, 55.80°, 59.3°, 65.2°, and 77.3° also indicates the cubic phase of Co$_3$O$_4$ (JCPDS 42-1467) [59]. As it is showed in figure 1(c), both ZnO and Co$_3$O$_4$ phases were observed. The result indicates that the p-Co$_3$O$_4$/n-ZnO composite catalyst was successfully synthesized. The crystalline sizes of the ZnO and p-Co$_3$O$_4$/n-ZnO were also calculated according to the Scherrer formula [60]. The average crystalline sizes of the
ZnO and p-Co$_3$O$_4$/n-ZnO catalysts were found to be 24.62 and 16.68 nm, respectively. The result indicates that the p-Co$_3$O$_4$/n-ZnO had smaller crystalline size after p-Co$_3$O$_4$ semiconductor was incorporated.

Figure 2 shows the FT-IR spectra of the synthesized ZnO and p-Co$_3$O$_4$/n-ZnO. The absorption peaks located at 3415, 2925, and 2837 cm$^{-1}$ attribute to the stretching and bending vibrational absorptions of O–H resulted from the adsorbed water molecule [61]. Absorption bands at 1738, 1634 and 1375 cm$^{-1}$ are also attributed to the stretching vibration of NO$_3$– ions which may be originated from the reaction intermediates or residue of cobalt and zinc nitrate precursors [61, 62]. Moreover, the peaks at 665 cm$^{-1}$ and 576 cm$^{-1}$ showed the stretching vibration of Co–O, and corresponding to the tetrahedral and octahedral coordination of Co$^{2+}$ and Co$^{3+}$, respectively [63]. Similarly, the absorption peak at 425 cm$^{-1}$ represents the stretching vibrational mode of Zn–O. Thus, the presence of Co–O and Zn–O confirms that the p-Co$_3$O$_4$/n-ZnO composite was synthesized successfully.

**Figure 1.** XRD patterns of (a) ZnO, (b) Co$_3$O$_4$, and (c) p-Co$_3$O$_4$/n-ZnO catalysts.

**Figure 2.** FT-IR spectra of the synthesized ZnO and p-Co$_3$O$_4$/n-ZnO catalysts.
The surface morphologies of the samples were examined by SEM. As it is observed in figures 3(a)–(b), the morphologies of ZnO, Co3O4, composite catalysts were the clusters of close packed organization and amorphous dense masses. However, the p-Co3O4/n-ZnO shows- hampering and relatively smaller and both metal oxide pieces randomly agglomerated (figure 3(c)). Moreover, the elemental composition of synthesized catalyst was examined and the noticeable peaks for Zn, Co, and O elements were observed as it shown in figure 3(d).

The photocatalytic performances of the synthesized samples were also tested with the degradation of MB dye under visible light irradiation. Figures 4(a)–(c) indicate the photocatalytic performances of ZnO, Co3O4, and p-Co3O4/n-ZnO catalyst towards the degradation of MB. In the degradation processes, the absorbance peaks located at 664 nm were decreased when the degradation reactions time increased in the presence of catalysts. The highest catalytic degradation of MB was achieved by p-Co3O4/n-ZnO and 95.5% of MB dye was degraded within 60 min. However, only the 72% and 79% of the degradation of the MB dye was achieved by ZnO and Co3O4 catalysts, respectively. The results indicated that the presence of plant extract during the synthesis of the catalysts followed by calcinations makes the catalyst more porous and enhances the catalytic performances. It is also indicated that the adsorption of MB dye under dark was maximum and makes the pollutant concentration lower in the solution. Moreover, the degradation of MB dye with p-Co3O4/n-ZnO also further enhanced. The highest degradation efficiency of the composite catalyst might be due to the porous surface of the samples and the formation p-n junction in the catalyst system in which the electron and hole recombination rate is decreased.

As shown in figure 5a, the degradation kinetic of the MB dye was examined by the fitting data according to the equation: \[ \ln \left( \frac{C_t}{C_0} \right) = -kt, \] where \( k \) is the apparent first-order rate constant; \( C_0 \) and \( C_t \) are the initial concentration and the concentration at a time \( t \). The linear correlation between irradiation time and \( \ln \left( \frac{C_t}{C_0} \right) \) indicates that the photocatalytic degradation of the MB dye shows pseudo-first-order kinetics [64]. The rate constants \( k \) in the degradation of MB dye with ZnO, Co3O4, and p-Co3O4/n-ZnO catalysts were 0.014 min\(^{-1}\), 0.018 min\(^{-1}\), and 0.028 min\(^{-1}\), respectively (figure 5b).

The reaction mechanism for the degradation of MB dye was also proposed (figure 6). When the p-type Co3O4 is coupled with n-ZnO semiconductor, a p–n heterojunction will be formed in the interface. The holes will diffuse to n-type semiconductor while the electrons will diffuse to p-type semiconductor region because of the availability of concentration gradient carriers in the interfaces [64, 65]. In the photocatalysis reaction process, the photogenerated electrons will transfer to n-type ZnO conduction band and holes will transfer to p-type Co3O4 valence band. The p–n heterojunction will be formed between Co3O4 and ZnO semiconductors.
that can facilitate the photogenerated electrons and holes separation. The electrons will interact with surface adsorbed oxygen and the holes will interact with water molecules to form active oxygen species. The resulting active oxygen species will react with organic pollutants and changed to CO₂ and H₂O. Hence, the p-Co₃O₄/n-ZnO photocatalyst could enhance the photocatalytic degradation activities of organic pollutants. Moreover, the stability of the p-Co₃O₄/n-ZnO photocatalyst was also checked as shown in the figure 7(a). After the 5th run, 89.8% of the MB dye was degraded. The XRD analysis was also performed after reused and there was no significant change from the XRD peaks as shown in figure 7(b).
4. Conclusion

The green and cost-effective method used to synthesize the *Eichhornia Crassipes* plant extract mediated p-Co$_3$O$_4$/n-ZnO composite catalyst is important. The degradation efficiency of the p-Co$_3$O$_4$/n-ZnO was the highest and 95.5% of MB dye was degraded within 60 min. However, the ZnO and Co$_3$O$_4$ catalysts degrade only 72% and 79% of the MB dye, respectively. The results indicated that using unwanted *Eichhornia Crassipes* plant extract for the synthesis of catalyst is greatly appreciated. Moreover, the formation of p-n heterojunction between, p-type Co$_3$O$_4$ and n-type ZnO semiconductors enhances the photocatalytic activities due to the photogenerated electrons and holes separation. Hence, the *Eichhornia Crassipes* plant extract mediated p-Co$_3$O$_4$/n-ZnO composite catalyst may be important in the wastewater treatment technologies.

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