Photocatalytic efficiency of brilliant green dye on ZnO loaded on cotton stalk activated carbon

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Abstract
In this study, ZnO loaded on cotton stalk activated carbon (ZnO/CSAC) has been successfully synthesized by the chemical precipitation method. XRD patterns showed that ZnO/CSAC have two phases are hexagonal wurtizite and amorphous structures. The average particle size is reduced (12.4 nm) upon loading CSAC than the ZnO NPs. The band gap value is decreased (2.50 eV) upon loading on CSAC compared to ZnO NPs. The functional groups identify using by FT-IR spectrum. FE-SEM images a pure ZnO revealed a 15–40 nm spherical structure and 10–35 nm large agglomerations spherical structure for ZnO/CSAC and then, EDAX spectrum affirmed the formation of ZnO/CSAC. BET surface area and pore volume of the 22.18 m² g⁻¹ and 0.210 cm³ g⁻¹ for the ZnO NPs and make it increased surface area and pore volume due to the adding CSAC to 263.18 m² g⁻¹ and 0.306 cm³ g⁻¹. The photocatalytic degradation efficiency of brilliant green (BG) dye and results shows that ZnO/CSAC sample improves photocatalytic activity than the ZnO NPs. Amidst all; ZnO/CSAC showed the maximum degradation the efficiency (90.26%) than the ZnO. These results show that the ZnO/CSAC sample is due to the synergistic effect between ZnO (photocatalysts) and CSAC (adsorption), its also possible mechanism and pseudo-first-order model are used to analysis the kinetics.

1. Introduction
Numerous applications of organic dyes and pigments have increased in a considerable way in recent years. Many types of organic dyes and pigments are used in the process such as paper, carpet, printing, plastic, pulp, ceramic, glass, cosmetic, fiber, pharmaceutical, leather, textile, and other industrial have been always a major problem. The effluents containing dyes are largely colored and causing water pollution [1]. The brilliant green (BG) is a cationic dye, it is described as an irritant, causing skin, eye burns, vomiting, diarrhea, and abdominal pain [2, 3]. Nowadays, dyes removal in the enormous value. The semiconductor materials were studied like TiO₂ [4], CdS [5], SnO₂ [6], ZnS [7] and ZnO [8] as photocatalysts are used to remove organic dyes of pollutants. Among these, Zinc oxide is an II-VI group of n-type semiconductors due to its wide bad gap (3.37 eV) and its large exciton binding energy (60 meV) at room temperature and high photosensitivity, non-toxic nature and low cost [9]. The many methods have been used for the synthesis of ZnO NPs are studied like hydrothermal [10], chemical vapour deposition [11], sol-gel [12], chemical precipitation method [13]. Among these, we have chosen, chemical precipitation is using by one of the simplest methods. In several researchers are reported by, it has found that activated carbon (AC) prepared from agricultural wastes such as coconut shells [14], bamboo [15], acorn shell [16] and etc. AC one of the most widely used for adsorbent; it possesses a large surface area and pore volume. AC has been suggested as an effective loaded for semiconductor materials in the removal of the organic pollutants [17]. Keeping in view of the above, many researchers studied by nanomaterial such as SnO₂/TiO₂ [18], ZnO/CdS [19] and CuO/MnO₂ [20] have been recently improved. Nguyen Xuan et al reported...
on the metal ions doping nanomaterials can also act as electron–hole recombination sites, resulting in low photocatalytic efficiency [21]. ZnO loaded on non-metal ions (AC) is recently developed in several researchers reported because of its good properties such as, high surface area, large pore volume, decrease particle size, decrease band gap and increasing active site which can improve its photocatalytic activity [22]. It is clear that the ZnO NPs loaded on AC shows a synergistic effect of the organic dye pollutants [23]. Herein, we reported a study of CSAC loading on the structure, optical, functional, morphology, and surface area of the ZnO nanoparticles. The photocatalytic efficiency of the consequent pure ZnO NPs and ZnO loaded on CSAC was studied in detail.

2. Experiment

2.1. Material
Zinc acetate dihydrate (Zn (CH₃COO)₂ 2H₂O), ammonium carbonate ((NH₄)₂CO₃), and Potassium hydroxide (KOH) were analytical reagent grade purchased from Merck and SD fine chemicals. Furthermore, double distilled water is used all over the experiments.

2.2. Synthesis of cotton stalk activated carbon (CSAC)
Cotton Stalk (CS) agricultural wastes were collected from the local area in Harur Taluk, Dharmapuri District, Tamilnadu, India. The CS was washed several times with tap water and then with double distilled water to remove impurities and furthermore dried in an oven at 110 °C for 24 h. The dried CS was then ground and sieved to about 2 mm is less than size. The CS was then impregnated in a solution of potassium hydroxide with a mass ratio of 1:2 (KOH : CS). After 5 h of a magnetic stirrer, the sample is filtered from the solution and then dried at 110 °C for 24 h. After, the product was then dried and activated by heating (rate of 20 °C min⁻¹) at 600 °C during 2 h flow in a muffle furnace under a N₂ atmosphere. After cooling down to ambient temperature, the CSAC produced is washed five times with hot double distilled water and cold double distilled water to remove the base until washing was near of neutral (pH ≈ 7). The product was then dried at 110 °C for 24 h and then finally, to obtain the cotton stalk activated carbon (CSAC).

2.3. Synthesis of pure ZnO NPs and ZnO loaded on cotton stalk activated carbon (ZnO/CSAC)
Pure ZnO NPs and ZnO loaded on CSAC are synthesis by a chemical precipitation method. 0.5 M of Zn (CH₃COO)₂ 2H₂O was dissolved in 50 mL of double distilled water in which 1.0 M of NH₄)₂CO₃ was dissolved in 50 mL solution is added drop wise and the mixture is stirred for 1 h, to obtain pure ZnO. A similar method, ZnO/CSAC was synthesis by adding 0.5 g of CSAC and then, mixture was stirred at 60 °C for 4 h. The product materials were filtered and washed with many times with double distilled water and ethanol, dried at 110 °C for 6 h. Finally, the obtained samples were annealed in a muffle furnace at 400 °C for 2 h, to obtain the ZnO/CSAC. A similar method of synthesis without the addition of CSAC is used to synthesis pure ZnO NPs.

2.4. Characterization
The XRD patterns of pure ZnO and ZnO/CSAC are recorded by X’PERT-PRO diffractometer with a Cu Kα radiation (λ = 1.5406 Å). The UV–Vis absorption spectra were obtained using a Varian/Carry 5000 in the range of 300–800 nm. The FTIR spectra were analyzed by SHIMADZU-8400, wave number range of 4000–400 cm⁻¹. The morphologies of samples are analyzed by FE-SEM (CARL ZEISS) with Energy-dispersive x-ray spectroscopy (Bruker). The N₂ adsorption–desorption is measured at 77 K (Micrometrics ASAP 2010).

2.5. Photocatalytic experiment
The photocatalytic performance of pure ZnO NPs and ZnO/CSAC are tested by photodegradation of BG dye. The experiments were carried out between 11.00 am to 02.00 pm under sunlight and the solar intensity (1250 × 100 Lu ± 100). They are ZnO/CSAC (0.20 g L⁻¹) was added on to a quartz photoreactor containing 100 mL of a 30 mg L⁻¹ concentration of BG dye. After this, the reaction mixture was stirred keeping irradiated under sunlight. The preordained regular intervals, an adequate quantity of solutions were taken out and centrifuge to remove undissolved particles (Remi centrifuge, 85414 with 3000 RPM). The brilliant green (BG) dye solution and supernatant are analyzed by a UV–Vis (Shimadzu UV-1800) at λ max of 624 nm. The percentage of degradation of dye solution was calculated is,

\[
\%D = \frac{C_o - C_i}{C_o} \times 100
\]

where \(C_o\) is the initial concentration of BG dye solutions (mg L⁻¹), \(C_i\) is the concentration of dye after irradiation after a selected time interval (mg L⁻¹) [24].
3. Results and discussion

3.1. XRD analysis

The XRD patterns of the CSAC, ZnO NPs and ZnO loaded on CSAC (figures 1(a)–(c)). In can be observed (figure 1(a)) that the two broad diffraction peaks appeared at 2theta is 26 degree and 43 degree, which corresponding to the (002) and (100) planes of graphite [25, 26]. The ZnO NPs (figure 1(b)) shows that only all the diffraction peaks assigned to the good crystal hexagonal wurtzite structure [JCPDS 36-1451]. The ZnO NPs upon loading on the CSAC pattern of the two phases is formed with hexagonal wurtzite and amorphous structures and then, all the diffraction peaks show that a reduced upon loading CSAC (figures 1(b)–(c)). The average crystallite sizes of the ZnO NPs and ZnO/CSAC were estimated by Debye Scherrer’s formula [27]. The average crystallite sizes of 33.20 nm and 12.4 nm for ZnO NPs and ZnO/CSAC, respectively. It is clear that the decrease in the crystallite size with respect to loading on CSAC and the improved surface area of the ZnO/CSAC [28].

3.2. UV–Visible absorption spectrum of pure ZnO and ZnO loaded on CSAC

The UV–Vis absorption spectra of pure ZnO NPs and ZnO/CSAC are shown in figure 2. We can see that the absorption edges of pure ZnO NPs are about 380 nm. The absorption edge of ZnO loaded on CSAC exhibited a red shift compared with ZnO NPs [29]. Figure 2(b) shows that the band gap ($E_g$) of the ZnO NPs and ZnO/CSAC calculated by Tauc’s plot ($\alpha h\nu$)$^2$ versus $h\nu$ [30]. The band gap value of ZnO NPs and ZnO/CSAC is 2.63 and 2.50 eV, respectively. The decrease in the band gap value of ZnO/CSAC sample, this leads to the change of energy level is loading on CSAC. These reduced band gap value and favorable surface properties for hydroxyl radical formation [31].

3.3. FTIR analysis

The FTIR spectra of the CSAC, ZnO NPs and loaded on CSAC (figures 3(a)–(c)). In CSAC, sample O–H stretching vibration broad bands at 3392–3112 cm$^{-1}$ [32]. The band is center at 1579 cm$^{-1}$ was assigned to the aromatic C=C groups [33, 34]. It is aromatic ring groups confirm that form of CSAC. The band around 1188 cm$^{-1}$ is assigned to C=O stretching vibration of carboxylic groups [35]. In ZnO NPs and ZnO/CSAC (figures 3(a), (b)), the absorption two bands at 3438 cm$^{-1}$ and 1594 cm$^{-1}$ which attributes to O–H stretching and bending vibrations. The band at

Figure 1. XRD patterns of (a) CSAC, (b) ZnO and (c) ZnO/CSAC.
446 cm$^{-1}$ was assigned to the stretching vibration mode of Zn–O bond [36]. The FTIR spectra results show that the presences of various functional groups prove the formation of ZnO/CSAC.

3.4. Field emission scanning electron microscopy with energy dispersive x-ray analyses
The products of ZnO NPs and ZnO/CSAC have been analyzed by FE-SEM and corresponding to the EDAX analysis (figures 4(a)–(c)). It is an image clearly observed that the ZnO NPs show spherical particles with a size in the range of $\sim 15$–$40$ nm (figure 4(a)), while the ZnO loaded on CSAC it sees that the show irregular agglomerated spherical particle with size in the range of $\sim 10$–$35$ nm (figure 4(b)). The elemental analyses of the ZnO loaded on CSAC (EDAX analysis) as shown in figure 4(c). From the figure, we can see that the peaks of Zn, C, and O in the weight percentages are 52.31, 27.04 and 20.65. As well as the quantitative analysis confirm with form of ZnO loaded on CSAC.

3.5. Nitrogen adsorption-desorption isotherms
The specific surface area and pore volume distribution of ZnO NPs and ZnO/CSAC (figures 5(a), (b)). Both the sample exhibits a type IV $H_3$ hysteresis loop with mesopores structure [37]. The specific surface area and pore volumes are $22.18$ m$^2$ g$^{-1}$ and $0.210$ cm$^3$ g$^{-1}$ for ZnO NPs, since, $263.18$ m$^2$ g$^{-1}$ and $0.306$ cm$^3$ g$^{-1}$, for ZnO/CSAC (insets of figures 5(a), (b)), respectively. Its results indicate that the high specific surface area and pore volume of ZnO/CSAC sample exhibit higher photocatalytic activity compared to the ZnO NPs.
3.6. Effect of catalyst dose
The effect of catalyst dose versus percentage degradation of BG was studied in 100 mL dye solution, initial dye concentration \((30 \text{ mg L}^{-1})\) at RT \((30^\circ \text{C})\) for 120 min. The increases in the catalyst doses is 0.10 to 0.30 g L\(^{-1}\) (figure 6), it is seen that the highest percentage of removal is 72.60% and 90.26% for the dye solution observed at catalyst dose \((0.20 \text{ g L}^{-1})\) are ZnO NPs and ZnO/CSAC and after that the percentage of degradation is observed to be decreased at a high catalyst dose \((0.30 \text{ g L}^{-1})\). The results show that the decreased in the removal efficiency of BG at initial dye concentration of above 30 mg L\(^{-1}\) are due to the light reflectance by CSAC [38].

3.7. Photodegradation of BG dye
The absorption spectra of irradiated ZnO NPs and ZnO/CSAC samples are recorded at different time intervals \((0 \text{ to } 120 \text{ min})\) and removal of BG dye are shown in figures 7(a), (b). It can be observed that the most absorption peak at 624 nm of the BG dye solution containing the ZnO/CSAC sample, after the absorption peak decreased gradually with increasing irradiation time and finally, almost disappeared after 120 min. The results confirm that the photocatalysts degradation of BG dye solution on the ZnO loaded on CSAC is an improvement compared to ZnO NPs.

Figure 8 show the effects of contact time and percentage degradation of BG dye solution at initial dye concentration \((30 \text{ mg L}^{-1})\). It may see from that, the percentage degradation increased from 21.73 to 72.60% and 24.69 to 90.26% with an increase in the contact time from 0 to 120 min for ZnO NPs and ZnO/CSAC. Initially, a fast increase in the dye adsorption may be due to the availability of a large number of free surface active sites on ZnO/CSAC sample for dye adsorption. After some instant of time, the adsorption of dye was slow down and finally attained equilibrium. It may be due to the saturation of adsorbed surface and repulsive force build between dye molecules on the adsorbent surface.

3.8. Kinetic studies
The pseudo-first-order reactions kinetic of the BG dye solution on ZnO NPs and ZnO/CSAC samples [39]. The plots of \(\ln \left(\frac{C_0}{C_t}\right)\) versus irradiation time \((t)\), are shown in figure 9. The rate constants \((k)\) and linear regression
The coefficient ($R^2$) for all samples are given in table 1. It can be seen from the table that the rate constants values $k$ is 0.010 and 0.019 min$^{-1}$ for ZnO and ZnO/CSAC, respectively. The highest rate constants ($k$) value of ZnO/CSAC indicating its best degradation ability for the organic pollutant.

3.9. The Mechanism of Photocatalytic degradation of the ZnO/CSAC

The mechanisms of ZnO/CSAC degrade a synergistic effect such as CSAC (adsorption) and ZnO NPs (photocatalysts) to removal of the BG dye solution is proposed schematic diagram are shown in figure 10. Now, ZnO/CSAC removal a synergistic effect, further, ZnO photocatalytic activity (72.60%) and loading CSAC adsorption (90.26%) due to increase the removal percentage. In this possible reaction in the photocatalytic degradation mechanism of BG by ZnO/CSAC are proposed as follows [40].

$$\text{ZnO/CSAC} + \hbar \nu \rightarrow \text{ZnO} (e^-_{\text{CB}} + h^+_{\text{VB}}) \quad (2)$$

$$\text{ZnO/CSAC}(e^-_{\text{CB}}) + O_2 \rightarrow \text{ZnO/CSAC} + O_2^- \quad (3)$$

$$\text{ZnO/CSAC}(h^+_{\text{VB}}) + H_2O \rightarrow \text{ZnO/CSAC} + H^+ + OH^- \quad (4)$$

ZnO NPs are irradiated by sunlight with an energy band gap, an valence band ($h^+$) can react with the adsorbed H$_2$O or OH$^-$ groups on ZnO NPs, a creation of OH$^-$ and conduction band ($e^-$) can react with the adsorbed O$_2$ to form ·O$_2^-$. Both OH$^-$ and ·O$_2^-$ radical can react with BG dye, however, OH$^-$ plays the main role in the photocatalytic degradation of BG using ZnO NPs and loaded on CSAC. In these results that the best degradation efficiency of ZnO/CSAC sample, because of the following factors, CSAC in the formation of reduced particle size, decrease band gap, high surface area and high pore volume on the photocatalysts [41].

3.10. Reusability of ZnO/CSAC

The reusability of ZnO/CSAC sample is used for the percentage of degradation of BG dye solution in the four cycles are shown in figure 11. The results showed that the BG dye removal of 88.80% was sustained after four cycles of reuse of ZnO/CSAC. ZnO/CSAC exhibits remarkable photostability as the BG degradation percentages are 1$^{st}$ cycle (90.26%), 2$^{nd}$ cycle (89.50%), 3$^{rd}$ cycle (88.8%) and 4$^{th}$ cycle (88.8%). The results indicate that the ZnO/CSAC are stable and can be recycled with a slight loss of their photocatalytic activity.
Figure 5. (a) N\textsubscript{2} adsorption and desorption isotherms and the corresponding pore size distribution curve (inset) of ZnO nanoparticles, (b) ZnO/CSAC.

Figure 6. Effect of catalyst dosage on photocatalytic oxidation of BG dye in presence of ZnO NPs and ZnO/CSAC.
Figure 7. (a) Time dependent UV–Vis absorption spectra of the photocatalytic degradation of BG in the presence of ZnO nanoparticles and (b) ZnO/CSAC.

Figure 8. The effect of BG dye degradation by ZnO NPs and ZnO/CSAC.
Figure 9. Kinetic study of photodegradation of BG in the presence of ZnO NPs and ZnO/CSAC.

Table 1. The rate constants and linear regression coefficients for the degradation of BG in the presence of ZnO NPs and ZnO/CSAC.

| Samples     | Rate constant (min⁻¹) | R²   |
|-------------|-----------------------|------|
| ZnO         | 0.010                 | 0.965|
| ZnO/CSAC    | 0.019                 | 0.980|

Figure 10. Schematic diagram of the adsorption and photocatalytic degradation of BG on the ZnO/CSAC under sunlight irradiation.
4. Conclusion

The synthesis of ZnO/CSAC has been successfully by a chemical precipitation method under annealed at 400 °C. The XRD and EDAX results confirmed the formation of ZnO/CSAC and the XRD analysis of ZnO NPs and ZnO/CSAC showed the crystalline sizes of 33.20 nm and 12.4 nm. The decrease in the crystalline size can be related to the observed increase in the surface area and pore volume. Optical properties of samples are studied by UV-Vis spectra and the effective loading of CSAC decrease the band gap of ZnO. EDAX reveals the presence of Zn, O and C in the ZnO/CSAC. BET surface area of ZnO/CSAC is higher than ZnO NPs due to the increase surface area and pore volume of ZnO/CSAC with enhanced photocatalytic activity. The high rate constants k is 0.019 min⁻¹ of the BG degradation using ZnO/CSAC using pseudo-first-order reaction kinetics. The mechanism of dye degradation is proposed for the higher photocatalytic activity of ZnO/CSAC. The high efficiency of this photocatalysis is due to the synergistic effect between ZnO NPs (photocatalysts) and CSAC (adsorption).

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