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Original Research Paper

Chemistry

Fe-MODIFIED COCONUT SHELL BIOCHAR AS AN EFFICIENT ELECTROCATALYST FOR CRYSTAL VIOLET REMOVAL

Jonty Rodrigues — Researcher, K J Somaiya College of Science and Commerce, Vidyavihar – 400077

Nishamol Kanat* — Associate Professor, K J Somaiya College of Science and Commerce, Vidyavihar – 400077 *Corresponding Author

Sheba Raju — Researcher, K J Somaiya College of Science and Commerce, Vidyavihar – 400077

ABSTRACT Crystal Violet is a persistent synthetic dye widely used in industrial applications and is difficult to remove from wastewater due to its stable aromatic structure and resistance to biodegradation. In the present study, Fe-loaded magnetite biochar derived from coconut shell biomass was synthesized and investigated for the electrochemical degradation of crystal violet in aqueous solution. The Fe-loaded biochar was prepared through $FeCl_3$ impregnation followed by pyrolysis at 700 °C and characterized using FTIR, XRD, and SEM–EDX analyses, which confirmed the formation of magnetite phases and the successful incorporation of iron species onto the biochar surface. Electrochemical degradation experiments demonstrated that the incorporation of Fe-loaded biochar significantly enhanced dye removal compared to electrochemical treatment alone. Near-complete decolourization of a 500 ppm crystal violet solution was achieved within 40 min under optimized conditions. Kinetic analysis showed that the degradation process followed pseudo-first-order behaviour with a high correlation coefficient, indicating efficient and rapid degradation within the electrochemical system. The results highlight the potential of Fe-loaded biochar as a low-cost and effective material for electrochemical wastewater treatment applications.

KEYWORDS : Biochar, Crystal Violet, Electrodegradation

INTRODUCTION

Emerging pollutants are contaminants that are not routinely monitored in the environment and whose ecological fate and long-term effects are not yet fully understood. Crystal Violet (CV) is one such synthetic dye widely used in industries including printing, textile, leather, pharmaceutical, varnish, and wax manufacturing [1]. Due to its aromatic ring structure, CV is highly stable and resistant to biodegradation, making it persistent in the environment [2]. In addition, its intense coloration reduces light penetration in water bodies, thereby affecting photosynthetic activity and aquatic life [3].

Various treatment technologies have been investigated for the removal of dyes from wastewater. However, many of these methods suffer from limitations such as high operational cost, sludge generation, and practical inefficiency for large-scale applications [4].

Biochar is a carbon-rich material produced through the pyrolysis of biomass under controlled temperature conditions, resulting in modified physicochemical properties depending on the pyrolysis temperature [5]. Biochar has been synthesized from various biomass sources and has been extensively explored for pollutant adsorption applications [6]. Surface modification remains an effective strategy to further enhance the properties of biochar [7]. Among these, iron loading has attracted considerable attention due to its ability to improve surface activity, electrochemical performance, and magnetic properties, thereby facilitating catalyst recovery and reuse [8].

Therefore, in the present study, Fe-loaded biochar derived from coconut shell biomass was synthesized and investigated for the electrochemical degradation of Crystal Violet in aqueous solution.

MATERIALS AND METHODS

Materials

Coconut shell biomass was collected from a local domestic market, washed thoroughly with distilled water to remove adhering impurities, sun-dried, and subsequently crushed and powdered using a domestic mixer grinder and hammer. Ferric chloride and Crystal Violet were procured from Loba Chemie, while potassium chloride was obtained from Molychem. All chemicals used in the study were of analytical grade and were used without further purification. Distilled water was used throughout the experiments.

Preparation of Fe-Loaded Biochar

Powdered coconut shell biochar was impregnated with an aqueous ferric chloride solution under continuous stirring to facilitate the loading of iron species onto the biochar surface. The impregnated material was subsequently dried and pyrolyzed at 700 °C for 4 h in a muffle furnace. The resulting Fe-loaded biochar was thoroughly washed with distilled water until neutral pH was attained to remove residual impurities and unbound iron species. The material was then dried and stored in airtight containers for subsequent electrochemical degradation experiments.

Material Characterization

Fourier Transform Infrared (FTIR) spectroscopy analysis was carried out in the range of 400–4000 cm^{-1} using the attenuated total reflectance (ATR) method to identify the surface functional groups present on the biochar. X-ray diffraction (XRD) analysis was performed over a 2θ range of 5–90° to investigate the crystalline phases and structural characteristics of the Fe-loaded biochar. Surface morphology and elemental composition of the prepared material were examined using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM–EDX).

Electrochemical Degradation Setup

Electrochemical degradation experiments were carried out in a 100 mL glass beaker with a working volume of 70 mL. Graphite and stainless-steel rod electrodes (0.3 cm diameter, 8 cm immersed length) were used as the cathode and anode, respectively, with an inter-electrode distance of 5 cm. Potassium chloride (0.1 M) served as the supporting electrolyte, while the applied voltage and initial Crystal Violet concentration were maintained at 7 V and 500 ppm, respectively. The reaction mixture was stirred continuously at 600 rpm without external aeration.

To investigate the role of Fe-loaded biochar, comparative

experiments were conducted under identical conditions: (i) electrochemical degradation without biochar, (ii) electrochemical degradation in the presence of Fe-loaded biochar (1 g/L), and (iii) biochar treatment without electric current to evaluate adsorption independently. These experiments enabled differentiation between adsorption and electrochemical degradation contributions during dye removal.

Analytical Methods

A linear calibration curve was prepared using standard solutions of Crystal Violet in the concentration range of 1–5 ppm. The concentration of crystal violet during the degradation experiments was determined using UV–Visible spectrophotometry by monitoring the absorbance of the dye solution at its maximum absorption wavelength.

The degradation efficiency of crystal violet was calculated using the following equation:

$$\frac{C_0 - C_t}{C_0} \times 100$$

where C_0 represents the initial concentration of crystal violet and C_t represents the concentration at time .

RESULTS AND DISCUSSION

The FTIR spectrum (Figure 1) of the Fe-loaded biochar revealed the presence of several functional groups associated with the carbonaceous structure and iron oxide species. Broad absorption bands observed in the region of 400–700 cm^{-1} may be attributed to Fe–O stretching vibrations, confirming the successful incorporation of iron species onto the biochar surface. In particular, the peaks around 564 cm^{-1} and 644 cm^{-1} are characteristic of magnetite-type iron oxide structures [9].

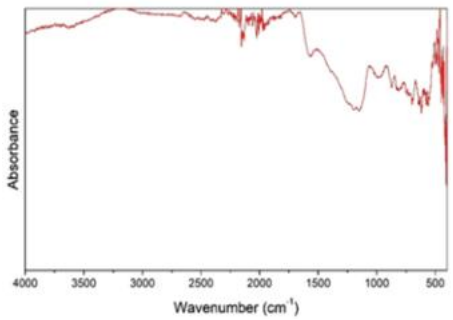

Figure 1: FTIR Spectra of Prepared Fe-Biochar

The absorption peak observed near 1158 cm^{-1} can be associated with C–O stretching vibrations originating from alcohol, ether, or phenolic functional groups present in the biochar matrix. The band around 1539 cm^{-1} corresponds to aromatic C=C stretching vibrations, indicating the presence of graphitic and aromatic carbon structures formed during pyrolysis [10].

The SEM micrographs (Figure 2) of the Fe-loaded biochar revealed a rough and heterogeneous surface morphology with irregular porous structures formed during pyrolysis. The layered and fragmented carbonaceous surface may enhance surface area and provide active sites for adsorption and electrochemical reactions, thereby facilitating improved interaction between the catalyst surface and Crystal Violet molecules.

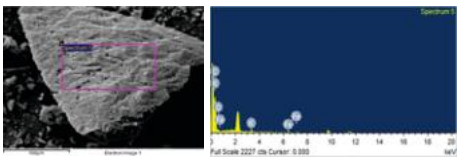

Figure 2: SEM Image and EDX-Graph

EDX analysis confirmed carbon and oxygen as the dominant elements in the biochar matrix, along with iron species introduced during FeCl impregnation. The material primarily consisted of carbon (71.97 wt%) and oxygen (25.10 wt%), while the presence of iron (1.57 wt%) confirmed successful iron loading onto the biochar surface.

The XRD diffractograms (Figure 3) confirmed the successful formation of Fe^{2+}/Fe^{3+} magnetite phases corresponding to JCPDS card No. 01-075-1609. Broad diffraction features observed in the spectra indicated the amorphous nature of the biochar carbon matrix. The average crystallite size of the synthesized iron oxide particles, estimated using the Scherrer equation, was found to be approximately 10 nm, suggesting nanoscale dispersion of iron species over the biochar surface.

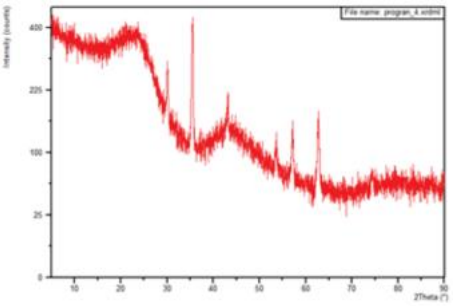

Figure 1: Powder-XRD Patter of Fe-Biochar

Figure 3: Powder-XRD Patter of Fe-Biochar

Electrodegradation Studies

Comparative Studies

The electrochemical degradation studies demonstrated that the incorporation of Fe-loaded biochar significantly enhanced the removal of Crystal Violet within the electrochemical system. Comparative experiments showed that the combined electrochemical–biochar system exhibited substantially higher decolourization performance than the control setups.

After 40 min of treatment, the electrochemical system containing Fe-loaded biochar achieved near-complete decolourization of the dye solution (residual concentration < 5 ppm), whereas the electrochemical setup without biochar retained a residual dye concentration of approximately 140 ppm. In contrast, the setup containing biochar without the application of electric current showed considerably lower removal efficiency, with the dye concentration remaining at approximately 410 ppm after the same treatment duration.

These findings indicate a synergistic interaction between the electrochemical process and the Fe-loaded biochar. The enhanced degradation may be attributed to the combined effects of adsorption, catalytic oxidation, and improved electron transfer facilitated by iron species dispersed on the conductive biochar surface.

Reaction Kinetics

Degradation kinetics of Crystal Violet were investigated using the Fe-loaded biochar electrochemical system. At predetermined time intervals, aliquots of the reaction mixture were collected, filtered, and immediately quenched using sodium thiosulphate to terminate the degradation reaction. The resulting concentration data were fitted using pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models.

The degradation process was well described by the pseudo-first-order model over the majority of the reaction period (0–30 min, $R^2 = 0.988$), with an apparent rate constant of 0.059 min^{-1}. The good linearity suggests a relatively constant concentration of electrogenerated reactive chlorine species during this stage. A slight acceleration in degradation was observed during the final stage of the reaction, where the remaining dye concentration rapidly decreased between 30

and 40 min. This behaviour may be attributed to an increased local oxidant-to-dye ratio near the Fe-loaded biochar surface as the bulk dye concentration decreased. The kinetic behaviour further supports the role of chloride-derived oxidizing species and the synergistic contribution of the Fe-loaded biochar in enhancing localized catalytic degradation.

CONCLUSION

Fe-loaded magnetite biochar was successfully synthesized and utilized for the electrochemical degradation of Crystal Violet. The results demonstrated that although degradation of the dye occurred in the absence of biochar, the incorporation of Fe-loaded biochar significantly enhanced the degradation efficiency of the system. The biochar acted as a catalytic and conductive support, facilitating improved adsorption, electron transfer, and localized oxidative degradation of the dye molecules. The enhanced performance observed in the presence of the Fe-loaded biochar highlights its potential as a low-cost and effective material for wastewater treatment applications involving persistent dye pollutants.

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