

VISIBLE-LIGHT-RESPONSIVE CLAY/POLYPYRROLE-GRAFTED SrCo_2O_4 NANOCOMPOSITE FOR PHOTOCATALYTIC DEGRADATION OF ORGANIC POLLUTANTS

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ABSTRACT

The synthesis and application of clay–polymer nanocomposites have driven extensive research as new classes of emerging nanomaterials due to their novel and improved properties compared with their pure constituents. In addition, polymer/clay nanocomposites can be used as supports for semiconductor oxides used as photocatalysts in water treatment. In this study, SrCo_2O_4 nanoparticles are added to the surface of polypyrrole-modified natural clay to successfully produce a photocatalytic system nanocomposite known as Clay/PPy/ SrCo_2O_4 (CPSC) to degrade Congo Red (CR) dye under visible light. The as-synthesized CPSC ternary nanocomposite and its individual constituents were fully characterized using structural (XRD), morphological (SEM), spectroscopic (FTIR), and thermal analysis (DSC and TGA) techniques. The photocatalytic activity of the as-synthesized photocatalyst is remarkably excellent. The photocatalytic degradation of CR by Clay/PPy/ SrCo_2O_4 achieved 99% in 100 min. The highest efficiency was observed at 1.0 g/L CPSC, 30 mg/L CR, and pH 4. Tests with the same pH and the same charge of the photocatalyst were carried out on solutions with lower concentrations of CR (10 and 15 mg/L), and we found that a duration of 30 min was sufficient to achieve complete decolorization (100%).

KEYWORDS: Photocatalysis ; nanocomposite ; clay ; polypyrrole ; SrCo_2O_4 ; Congo red.

1. Introduction

Providing clean, drinkable water to an exponentially growing global population has become a major concern for most countries due to scarce rainfall, excessive consumption, and high levels of pollution resulting from the industrial approach adopted on the one hand and the lack of clear environmental policies to preserve this vital resource on the other.¹ Water pollution comes from various sources, including human, industrial, and agricultural activities.² It should be emphasized that today, synthetic dyes used in several industries pollute a large quantity of water. Recent studies have confirmed that the annual consumption of synthetic dyes in the world represents more than 7×10^5 tons, of which approximately 84,000 tons are discharged into waterways.³⁻⁵ Dye compounds exhibit diverse properties and chemical structures; the effectiveness of their removal depends primarily on the technique implemented. Several conventional technologies have been used for the treatment of dye-containing wastewater, including, but not limited to, filtration, coagulation, membrane separation, biodegradation, and adsorption.⁶ However, these techniques are limited due to their high costs, their prolonged treatment times, and they often fail to achieve complete mineralization of pollutants.⁷ Heterogeneous photocatalysis is a promising low-cost green technology that differs from other techniques in its ability to transform hazardous organic pollutants into harmless byproducts (CO_2 and H_2O) without generating toxic residues.^{8,9} Photocatalysis is used in various applications, including wastewater treatment, photoinactivation, and production of hydrogen.^{10,11} Optimizing the conditions of a photocatalytic reaction requires the use of semiconductors with high chemical stability, adjustable band gap energy, and extremely high specific surface area.¹² Titanium dioxide (TiO_2), zinc oxide (ZnO), tin dioxide (SnO_2), and cerium oxide (CeO_2) are the most studied photocatalytic materials.^{13,14} However, their uses are limited, on the one hand, by their activation, which only takes place under UV irradiation due to their band gap values (> 3.0 eV), and on the other hand because of the rapid recombination of charge carriers (electrons/holes, e^-/h^+).¹⁵ Elemental dopants, surface defects, heterophase junction formation, composite formation, and the addition of reducing agents such as sodium borohydride (NaBH_4) and peroxydisulfate (PMS) remain effective strategies to improve the separation of photogenerated charges and increase photocatalytic activity accordingly.¹⁶⁻²⁰ Spinel oxides have been identified as promising functional materials for water treatment by photocatalysis under visible light. With the general formula AB_2O_4 , where A and B are transition metals, they exhibit several oxidation states, which promote redox reactions with efficient charge carrier transport. Furthermore, these catalysts are widely used as magnetic materials, offering the advantage of being easily separated from the reaction medium.^{21,22} Recently, special attention has been given to cobaltite oxide nanomaterials MCo_2O_4 (with $\text{M}=\text{Zn}, \text{Ni}, \text{Mn}, \text{Sr}, \dots$) as an ecological and inexpensive material for environmental applications due to their exceptional catalytic properties.²³⁻²⁵ However, these materials generally have some practical limits such as low adsorption capacity and high aggregation of nanoparticles, which considerably limits their use in photocatalysis in the treatment of organic wastewater. It is known that aggregation of photocatalyst nanoparticles can lead to a reduction in its specific surface area and a decrease in the number of accessible reaction sites necessary for the formation of reactive species.²⁶ Various strategies have been explored to limit this aggregation, such as the use of

supports to improve the dispersion of photocatalyst nanoparticles, thereby increasing the degradation rate of pollutants.²⁷ In addition to improving the dispersion and stability of photocatalysts in diverse wastewater environments, the use of clays and polymers facilitates the separation and transfer of photo-excited electrons and holes.²⁸ Indeed, the association of the inorganic functional groups of the clay with organic functional groups of the polymer on its surface results in a hybrid material with improved chemical and catalytic properties.²⁹ Clays in nanocomposites are modified with a wide variety of polymers, but conductive polymers, namely polyaniline (PANI), polythiophene (PTh), polypyrrole (PPy), and others, are the most widely used because polymers interact with organic pollutants due to the similarity of their conjugated molecular structures.^{30,31} Among these polymers, polypyrrole stands out as one of the best candidates for clay modification and the preparation of a matrix capable of securely holding the photocatalyst oxide nanoparticles on its surface, as polypyrrole possesses high electrical conductivity, good ion exchange capacity, low cost, and nontoxicity.^{32–34} To the authors' knowledge, the synthesis and application of Clay/Polymer/SrCo₂O₄ nanocomposites in photocatalysis have not been explored. Therefore, this work focuses on the synthesis of a novel photocatalytic nanocomposite, Clay/Polypyrrole/SrCo₂O₄, with high photocatalytic activity for wastewater treatment. The originality of this work lies in the creation of oxygen vacancies through a strong interaction between SrCo₂O₄ and polypyrrole (PPy). These vacancies represent a crucial aspect of photocatalytic degradation.³⁵ Oxygen vacancies serve as activation sites for O₂ molecules and enhance the oxidizability of SrCo₂O₄, which favors the generation of superoxides ($\cdot\text{O}_2^-$) on the surface of the photocatalyst.³⁶ In addition, the surface of clay has many active groups, such as hydroxyl groups ($-\text{OH}$), which play a crucial role in the oxidative photodegradation of organic pollutants.³⁷ Its performance was evaluated in the removal of Congo red (CR) dye in aqueous medium under visible light. The synthesized photocatalyst and its constituents were characterized by analytical techniques such as X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), UV-Vis reflectance spectroscopy, and differential scanning calorimetry and thermogravimetric analysis (DSC-TGA). We investigated various determining factors in the photo-catalytic process, including the initial concentration of CR, the initial pH of the solution, the dosage of the photocatalyst, and the temperature.

2. Materials and Methods

2.1. MATERIALS

Cobalt nitrate was purchased from Merck. Darmstadt. Strontium nitrate was purchased from Merck. Darmstadt. Citric acid was purchased from Biochem Chemopharma.

2.1.1. PROCEDURES FOR PREPARATION

Preparation of SrCo₂O₄

Initially, 1.571 g of Sr(NO₃)₂ in 40mL of ethanol and 4.713 g of Co(NO₃)₂ in 110mL of ethanol were mixed for 15 min at room temperature. A solution of 5.106 g citric acid in 100mL of distilled water

was added dropwise to the previous mixture and kept under thermal stirring at 70°C until a viscous liquid (gel) was formed; then the viscous gel gradually turned into a foam until a parched pink crust appeared. The resulting crust was then carefully ground to obtain a fine and well-dispersed powder, which was dried for 24 h at 70°C and then calcined in an electric furnace for 4 h at 600°C.

Preparation of nanocomposite Clay/PPY/SrCo₂O₄

Clay/PPY/SrCo₂O₄ composite was prepared using in situ oxidative chemical polymerization of pyrrole monomer containing clay and SrCo₂O₄ nanoparticles. Initially, 0.2 g of SrCo₂O₄ nanoparticles and 0.2 g of clay were dispersed in 10 mL of distilled water and kept under an ultrasonic bath for 3 h at 30°C. Then, 0.2 mL of pyrrole was added to the previous mixture, and the whole mixture was ultrasonicated for 2 h at the same temperature. The resulting homogeneous suspension was then transferred into a round-bottom flask placed in an ice bath and equipped with a magnetic stirrer, and then the polymerization was initiated by adding dropwise an iron chloride solution containing 1.48 g FeCl₃ dissolved in 10 mL of distilled water to the previous mixture. The reaction was then kept under constant stirring for 24 h at 5°C to complete the polymerization process. Once the time had elapsed, the resulting composite was separated from the solution by filtration, washed several times with distilled water followed by ethanol. Finally, the resulting product was dried in a desiccator for well over three days; then the recovered solid was ground to obtain a fine powder of Clay/PPY/SrCo₂O₄ composite (Fig. 1).

2.2. CHARACTERIZATION OF SYNTHESIZED NANOCOMPOSITE

Clay, polypyrrole, SrCo₂O₄, and nanocomposite were characterized using an XRD (X-ray diffractometer, Bruker D8 Twin-Twin) instrument for sample pattern recording from 10° to 80° at a scanning speed of 5°, 2θ per min. The infrared spectra were recorded using a Shimadzu 8400 Spectrometer in the wavenumber range from 400 cm⁻¹ to 4000 cm⁻¹. The surface morphology of the nanoparticles was studied by scanning electron microscopy (SEM) imaging using a Tescan Bruker model. Thermal analysis was performed using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA).

2.3. PHOTOCATALYTIC ACTIVITY OF NANOCOMPOSITE

The photocatalytic activity of the nanocomposite obtained by immobilizing spinel oxide photocatalyst (SrCo₂O₄) nanoparticles on the surface of local clay modified by *in situ* polymerization of polypyrrole was evaluated by the degradation of CR (C₃₂H₂₂N₆Na₂O₆S₂) under visible light irradiation. The photocatalytic reactor used was a cylindrical Pyrex container immersed in a cooling system. The photocatalyst was dispersed in 500 ml of an aqueous solution of the dye to be degraded. The mixture was stirred in the dark for 30 min. The suspension was then irradiated with a white LED light placed above the cylinder. Samples were taken every 30 min and centrifuged. The resulting samples were then analyzed by UV-visible spectroscopy.

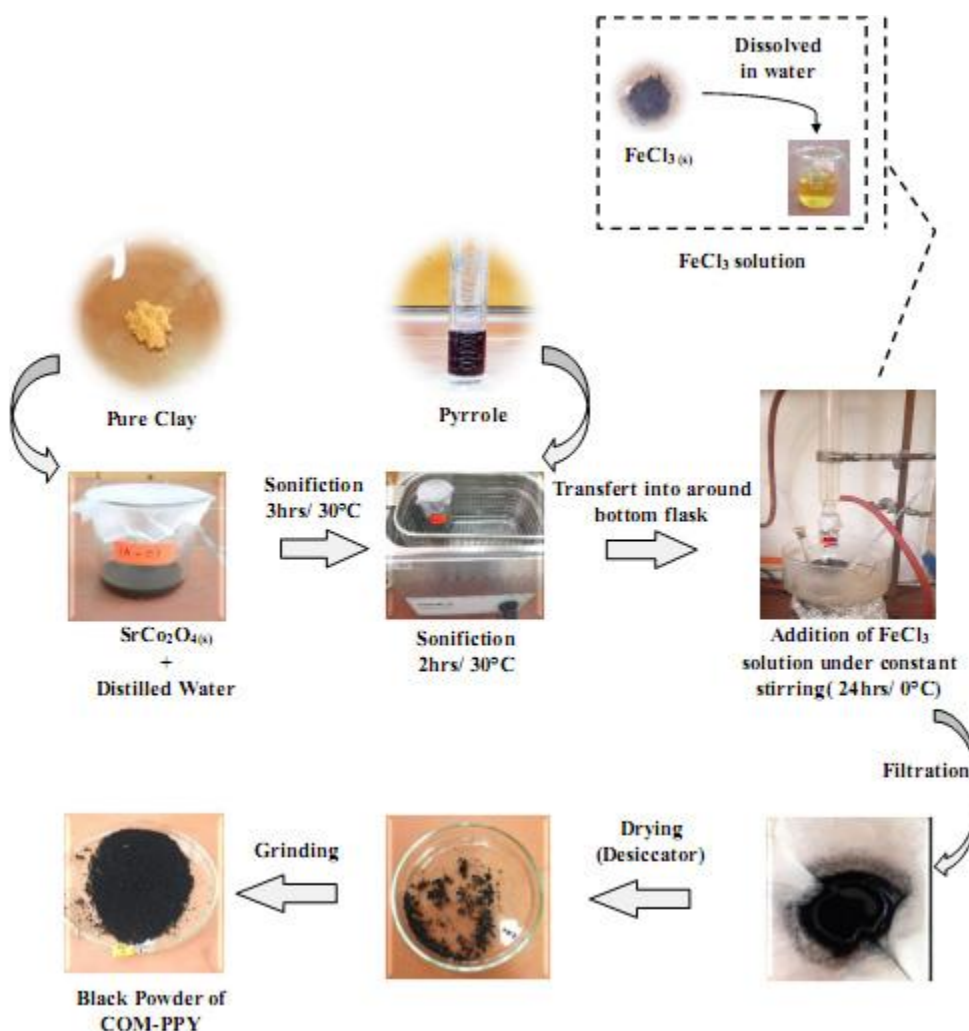


Fig. 1. Schematic illustration for synthesizing Clay/PPY/SrCo₂O₄ nanocomposite.

3. Results and Discussion

3.1. CHARACTERIZATION

The structural characteristics of the Clay/PPY/SrCo₂O₄ nanocomposite were confirmed by the XRD. The amorphous nature of the polypyrrole, the crystallinity of the clay, and the good crystallinity of the spinel oxide are highlighted by characteristic reflections illustrated in the diagram in Fig. 2(a). The polypyrrole diffraction peak does not appear in the XRD diagram because polypyrrole is amorphous in nature.³⁸ On the other hand, the principal reflections corresponding to the crystal planes (111), (220), (311), (222), (400), (422), (511), and (440) of the spinel phase of SrCo₂O₄ are well illustrated and located at 2° ¼ 18:85°; 31:19°; 36:81°, 38:39°; 44:87°; 55:45°; 59:31°, and 65:33°, respectively.³⁹ After the polypyrrole intercalation step, the majority of the diffraction peaks characteristic of clay were significantly reduced. Furthermore, the principal reflection of kaolinite

($2\theta \approx 12.4^\circ$) shifted to lower 2θ angles ($2\theta \approx 11.42^\circ$), which is characteristic of the insertion of polymer chains into the interlayer space of clay.

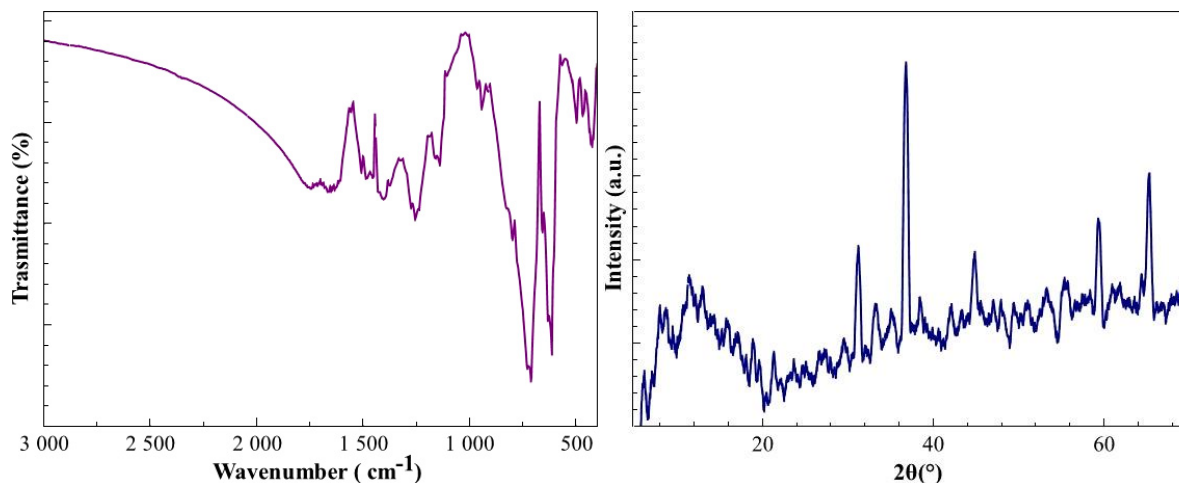


Fig. 2. (a) FTIR spectrum and (b) XRD pattern of Clay/PPY/SrCo₂O₄ nanocomposite.

Infrared spectroscopy analysis was used for the qualitative identification of the surface functional groups of the prepared nanocomposite. The FTIR curve obtained on nanocomposite is shown in Fig. 2(b). The Si–O–Si stretching is represented by the peak observed at 467cm^{-1} .⁴⁰ The peak at 1030cm^{-1} characterizes the O–Si–O stretching and deformation vibrations, while the peak at 914cm^{-1} is associated with the Al–O–H deformation vibration.⁴¹ The peak at 799cm^{-1} is attributed to C–H wagging.^{42,43} The peak at 1545.04cm^{-1} is due to $\text{C}\equiv\text{C}$ stretching vibrations.⁴⁴ The two sharp and intense peaks at 562cm^{-1} and 663cm^{-1} are attributed to stretching vibrations of the metal–oxygen bond in the octahedral and tetrahedral (Sr–O) and (Co–O) sites, respectively.⁴⁵

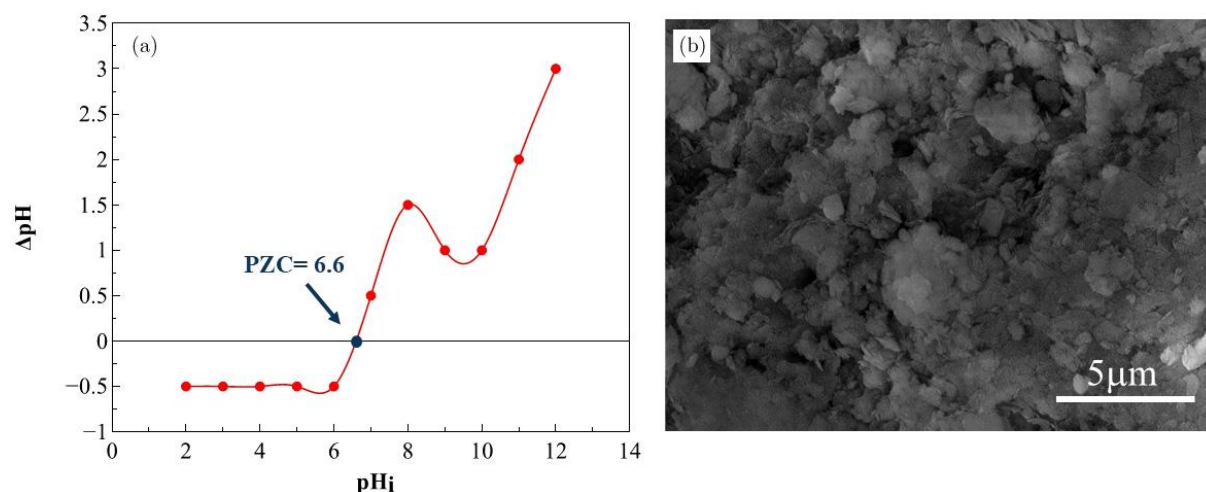


Fig. 3. (a) Point of zero charge (PZC) and (b) SEM image of Clay/PPY/SrCo₂O₄ nanocomposite.

As shown in Fig. 3(a), the δpH value is zero for an initial pH of 6.6. Therefore, the pH Point of zero charge (pHPZC) of the prepared Clay/PPy/SrCo₂O₄ nanocomposite is 6.6. In a solution with a pH below 6.6, the photocatalyst is positively charged and becomes negatively charged at a pH above pHPZC.⁴⁶

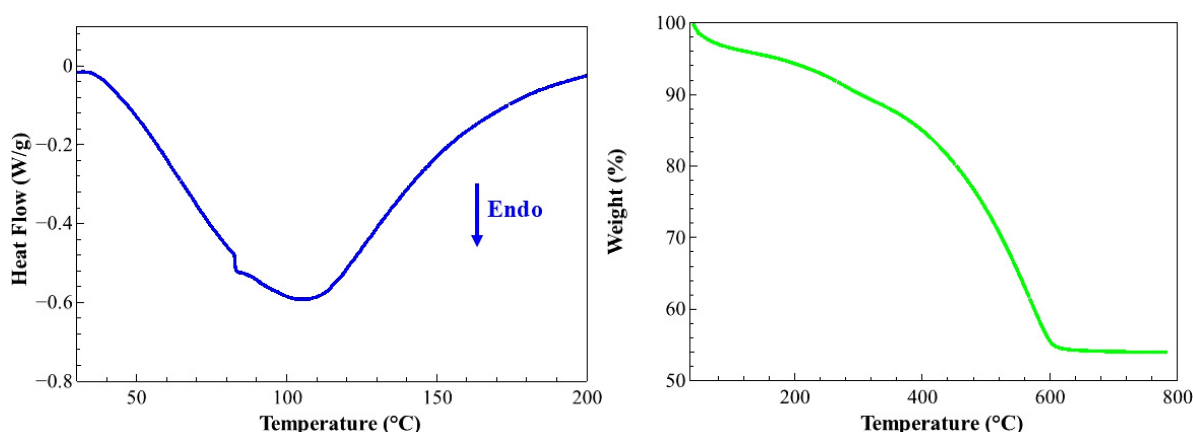


Fig. 4. (a) DSC thermograms of normalized heat flow (W g^{-1}) and (b) TGA thermograms against temperature ($^{\circ}\text{C}$) of Clay/PPy/ SrCo_2O_4 nanocomposite.

The nanoparticle morphology of the prepared sample was observed using SEM analysis and the result is presented in Fig. 3(b). The image reveals that the nanocomposite sample consists of aggregates of particles of varying shapes and sizes resulting from the exfoliation of clay during polymer formation. In addition, several small, spherical particles of different colors are scattered across the surface, corresponding to the oxide nanoparticles.

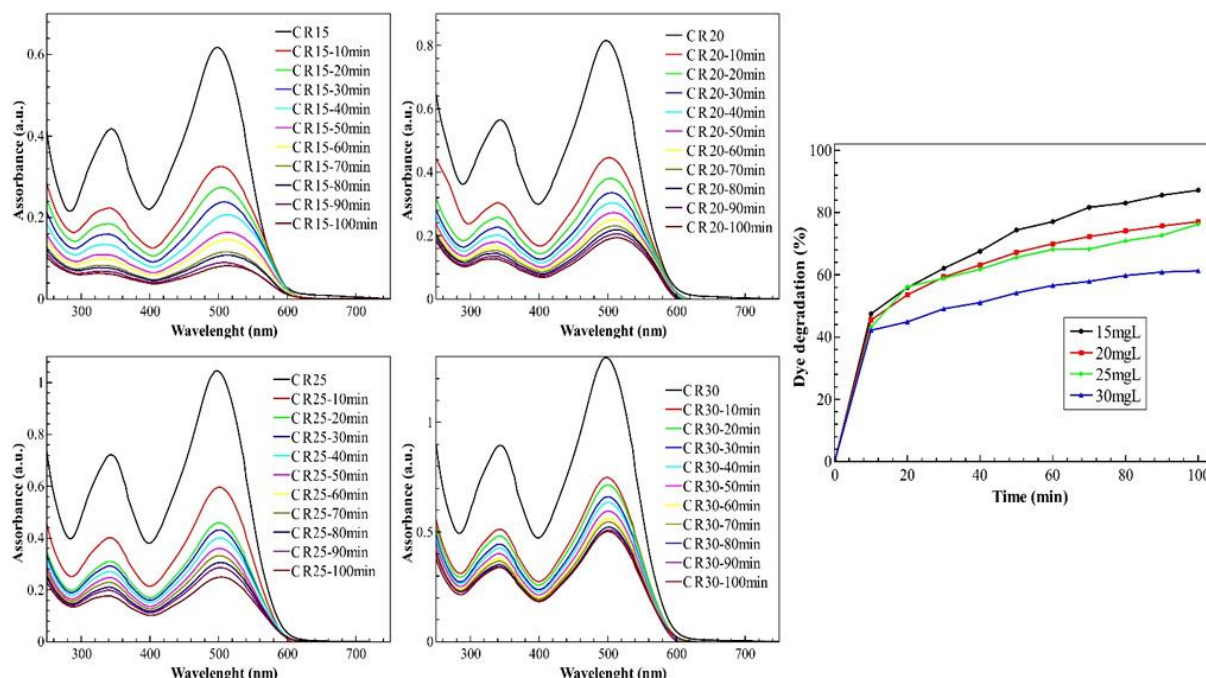


Fig. 5. The UV-vis spectra of CR dye from 0 min to 100 min under visible light irradiation.

DSC analysis measures heat flows into and out of a material during temperature variations associated with heat-induced macromolecular transitions.⁴⁷ Figure 4(a) shows the DSC heating curve of the prepared Clay/PPy/ SrCo_2O_4 nanocomposite in the temperature range from 0°C to 300°C . The broad and well-resolved endothermic drop around 105°C corresponds to the glass transition temperature of polypyrrole. The shape of the drop indicates that water loss and the glass transition of the polymer occur simultaneously.^{48,49}

The TGA thermogram of the nanocomposite (Fig. 4(b)) shows a slow decomposition curve extending from 60°C to 600°C, leaving a residue of approximately 54 wt.%. Three stages of mass loss are recorded. The first step starts at 45°C and continues up to 165°C, showing a mass loss of approximately 5%, corresponding to the loss of adsorbed and bound solvent. The second stage between 165°C and 350°C shows a mass loss of 9% associated with the temperature at the first degradation of the polymer. The third stage begins at 355°C and continues up to the temperature of 600°C, leading to a 40% mass loss caused by the second degradation of the polymer.

3.2. PHOTOCATALYTIC ACTIVITY

3.2.1. EFFECT OF INITIAL CONCENTRATION OF CR

The UV–visible absorption spectra of the CR dye in the presence of the nanocomposite under visible light, as a function of time and for different initial dye concentrations (15–30 mg/L), are shown in Fig. 5. The pH of the solution was not adjusted, and the temperature was set at 19°C (room temperature). The corresponding degradation percentages are also shown in the figure. This figure shows that the CR dye degraded by approximately 87% at an initial concentration of 15 mg/L and that this degradation rate decreased with increasing dye concentration.

3.2.2. EFFECT OF PHOTOCATALYST LOAD

The photocatalyst dosage is a critical parameter that significantly influences the efficiency of photocatalytic dye degradation. It plays a crucial role in controlling and implementing optimal conditions for a given concentration of the dye to be removed. Figure 6 illustrates the degradation efficiency of CR dye by varying the amount of photocatalyst (1.0, 1.5, and 2.0 g/L). The pH of the solution was not adjusted, and the temperature was set at 19°C (room temperature). The results showed that the degradation rate increases with increasing amounts of photocatalyst in suspension. This increase in efficiency is explained by the multiplication of photogenerated charges and, consequently, a greater availability of active sites on the photocatalyst surface.

3.2.3. EFFECT OF PH

The pH of the CR with photocatalyst suspension is one of the most important parameters used to optimize conditions favorable for the photocatalytic degradation of the dye. The effect of pH on the photocatalytic CR degradation using Clay/PPy/SrCo₂O₄ under visible light irradiation was investigated. The 500ml of 30mg/L initial dye concentration, 1.0 g/L photocatalyst, and reaction time of 100min was used. The pH of the CR-photocatalyst suspension was adjusted by adding negligible volumes of 0.1 M solutions of HCl and NaOH. Based on Fig. 7, the optimum degradation efficiency under visible light irradiation was achieved at a solution pH of 4 with the percentage of degradation of 99%. The pH of 4 corresponds to the limit of the dye's transition zone.⁵⁰ At this pH, the dye is negatively charged, given that the isoelectric point of CR is approximately 3.⁵¹ Indeed, the surface of the photocatalyst is positively charged in an acidic environment. CR, as an anionic dye, interacts favorably with such a surface, resulting in efficient degradation of the dye.⁵² The degradation

efficiency becomes slightly lower at basic pH. However, at neutral pH, the degradation of CR is not perceptible.

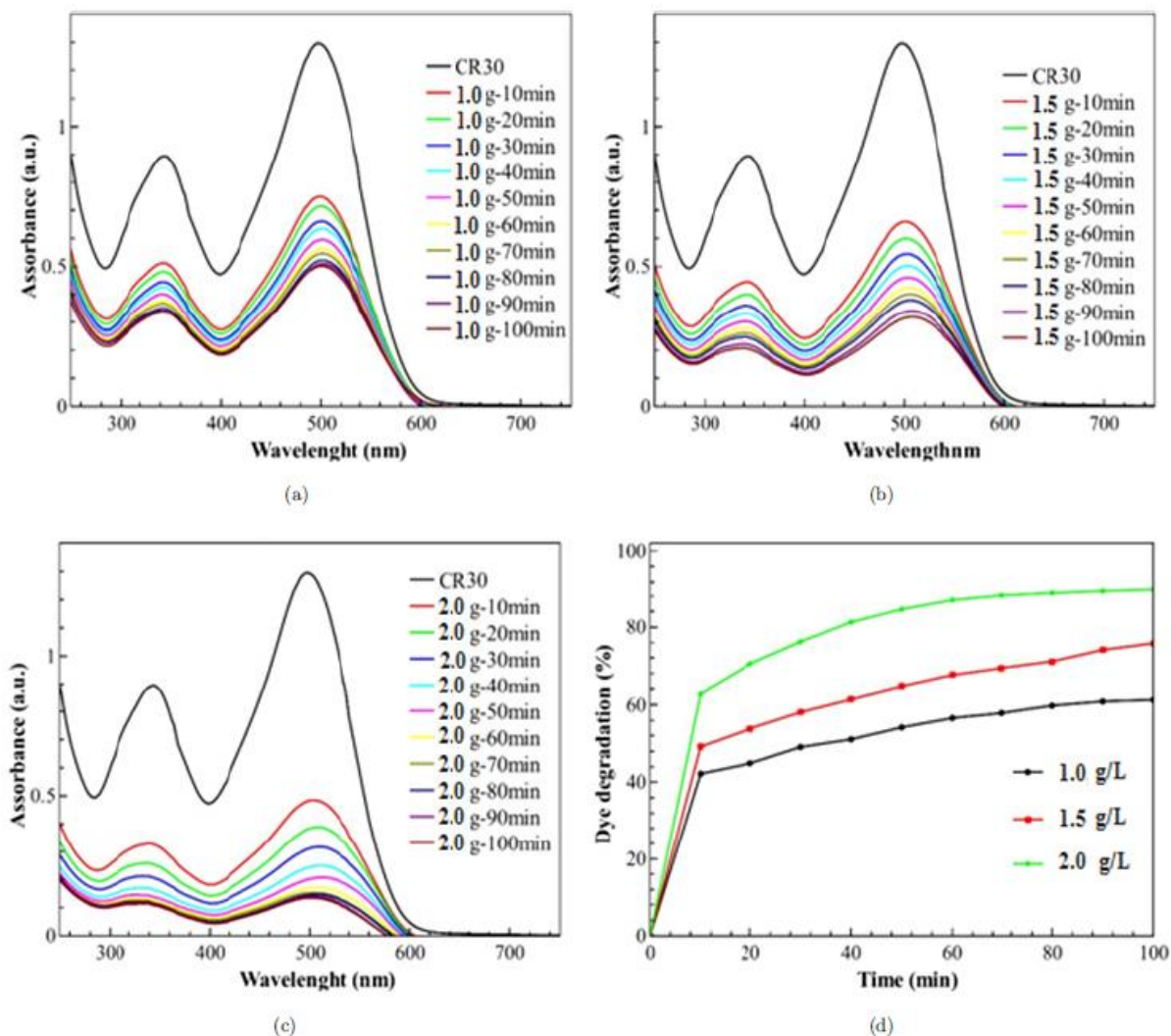


Fig. 6. Effects of photocatalyst dose.

3.2.4. EFFECT OF TEMPERATURE

In general, the photocatalytic act is independent of temperature. Although the collision frequency increases with temperature, this contributes only very slightly to increasing the reaction rate.^{53,54} The study of the effect of temperature on the photocatalytic degradation of CR was carried out at four different temperatures (19°C, 24°C, 29°C, and 35°C). As illustrated in Fig. 8, degradation is effective across all tests, with a slight decrease in the degradation rate recorded with increasing temperature.

3.2.5. COMPARISON AND MECHANISM OF PHOTOCATALYTIC EFFICIENCY

To highlight the photocatalytic efficiency of the developed nanocomposite, a comparative study with other composites of the same nature was conducted, drawing on the literature and presented in Table 1. Furthermore, a probable mechanism is proposed to illustrate the photocatalytic activity of the Clay/PPy/SrCo₂O₄ photocatalyst, as shown in the diagram in Fig. 9.

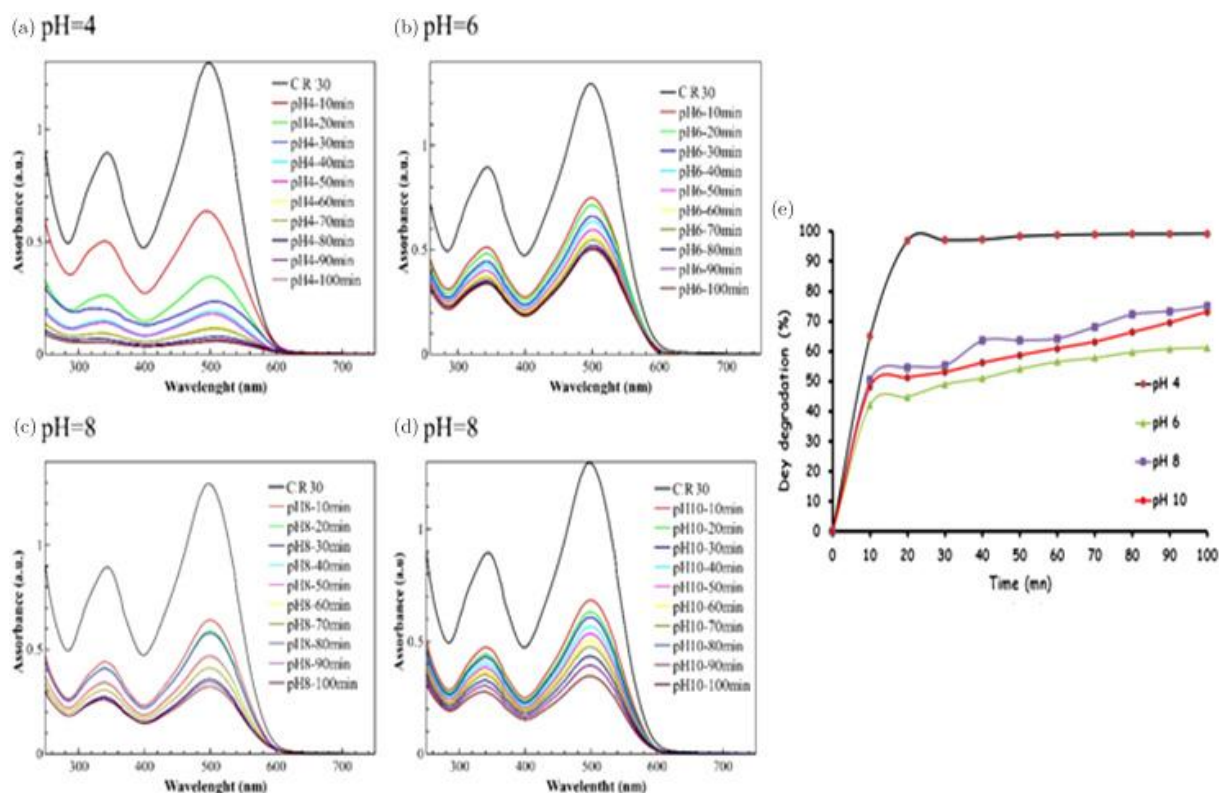


Fig. 7. Effect of pH on CR degradation.

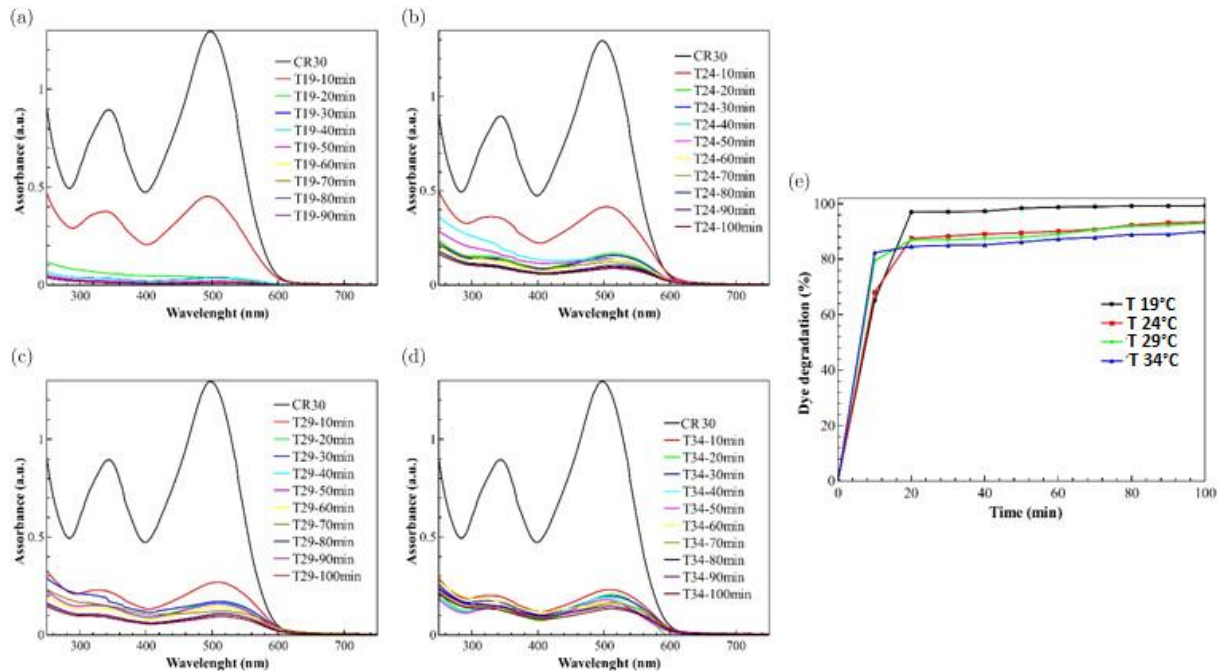


Fig. 8. Plots of dye degradation at different temperatures.

Table 1. Comparison of the photocatalytic performance of Clay/PPy/SrCo₂O₄ composite under visible light (this work) with some composite photocatalysts reported in the literature.

Composite	Pollutant	Light	Time	Efficiency (%)	References
Fe ₂ (MoO ₄) ₃ @PPy	Crystal violet	Visible-light	120 min	97	55
PPy-WO ₃	Methylene blue	UV light	160 min	98.31	56
PPy/TiO ₂	Rhodamine B	Visible-light	120 min	85	57
CdS/PPy	Methylene blue	Direct SunLight	300 min	77	58
MoSe ₂ -PPy	Rhodamine B	SunLight	15 min	84.4	59
MoSe ₂ -PPy	Congo red	SunLight	15 min	98.5	59
PPy/TiO ₂ -Fe	Methylene blue	Visible-light	120 min	91.92	60
SnO ₂ /C/PPy	Rhodamine B	Visible-light	20 min	90	61
TiO ₂ /PPy/GO	Victoria blue	Visible-light	180 min	95	62
Bi ₂ WO ₆ @g-C ₃ N ₅ /PPy	Rhodamine B	Visible light	60 min	96	63
Bi ₂ WO ₆ @g-C ₃ N ₅ /PPy	Methylene blue	Visible light	60 min	98	63
Clay/PPy/SrCo ₂ O ₄	Congo red	Visible light	100 min	99	This work

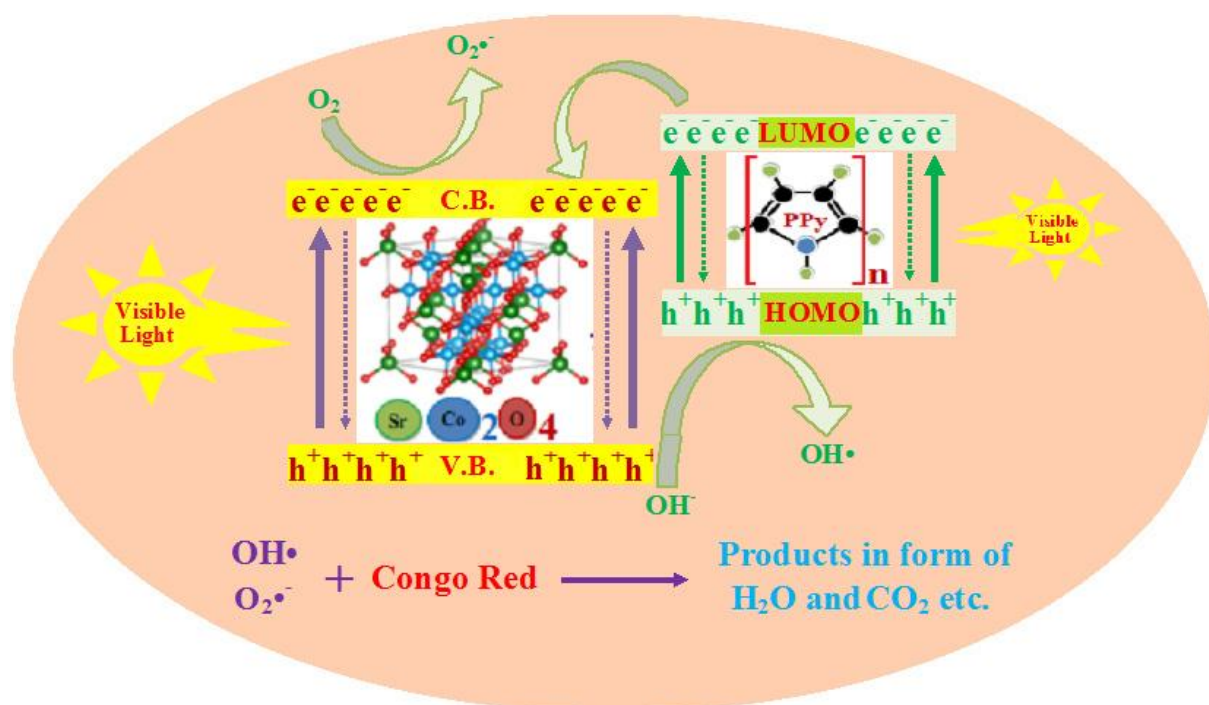


Fig. 9. Proposed CR dye photodegradation mechanism using Clay/PPy/SrCo₂O₄.

4. Conclusion

In conclusion, a Clay/PPy/SrCo₂O₄ nanocomposite was prepared. XRD, FTIR, SEM, DSC, and TGA techniques were used to characterize the composite produced. Photodegradation activity was examined by removing CR dye in aqueous solution. The photocatalytic degradation activity of the nanocomposite as a photocatalyst on the degradation of the CR dye was examined by varying the parameters: initial dye concentration, photocatalyst dose, initial pH, and temperature. The results

demonstrated that the Clay/PPy/SrCo₂O₄ nanocomposite exhibits high catalytic performance. With optimal pH and temperature conditions, the composite displays strong photodegradation of the dye even with relatively high pollutant concentrations.

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