RASĀYAN *J. Chem.*
Vol. 19 | No. 4 | 2320-2325 | October-December | 2026
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

FACILE SYNTHESIS OF POLY (O-PHENYLENEDIAMINE)/$CoCrFe_2O_4$ NANOCOMPOSITES FOR PHOTOCATALYTIC INVESTIGATIONS

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ABSTRACT

We performed the preparation and properties of $CoCrFe_2O_4$ nanoparticles and nano composites $CoCrFe_2O_4$/poly (o-phenylenediamine), aiming to explore their potential in environmental applications. By the self-combustion method, $CoCrFe_2O_4$ nanoparticles were subjected to in-situ fabrication with polymer $C_6H_4(NH_2)_2$. The nanoparticles and nano composites were characterized by SEM, XRD, UV-Visible and FTIR spectroscopy. The nano composites demonstrated a synergistic effect where the conductive polymer matrix and nanoparticles enhanced each other's properties, making the material promising for applications. The photo-catalytic behavior of $CoCrFe_2O_4$ nanoparticles and $CoCrFe_2O_4$/PoPD nano composites were analyzed in the presence of visible light.

Keywords: PoPD.$CoCrFe_2O_4$, PXRD, SEM, Dye Degradation

RASĀYAN *J. Chem.*, Vol. 19, No.4, 2026

INTRODUCTION

The majority of rivers and running streams of water are polluted due to contamination from domestic wastewater and effluent from the dyeing industry.[1] Required attention to solve this problem is not effectively taken due to socioeconomic environment and political reasons. In spite of the established methods available to circumvent this problem, this pollution continues to happen due to a lack of cost-effective and less efficient materials used for the treatment of wastewater. The dyes in water impart reduction of COD and BOD levels of water essential for aquatic species to survive. Even after strict regulations have been implemented in all the countries, this problem of discharging effluent to water bodies persists.[2] So, there is a demand to invent a simple and cost-effective method to overcome this issue. Adsorption is one among the technique useful and effective if the available material is of low cost and possesses high adsorption capacity.[3] The insufficiency possessed by conventional processes in removing or degrading reactive dye, advanced oxidation process like photo catalysis becomes a technique under consideration.[4] Due to the non-degradable nature of reactive dyes, mixing with water sources disturbs aquatic life, induces carcinogenic effects and collapses the reproductive system.[5-6] The studies on metal oxide nanoparticles as a heterogeneous photo-catalyst for the deterioration of dyes. The mechanism of action in deterioration is concerned with the formation of reactive radicals in non-biodegradable pigments in polluted water. Preference for heterogeneous catalysts for photo-catalytic activity is due to stability, withstanding complex reaction environments and appreciable efficiency in the degradation process, rendering advantage to the metal oxides, metal ferrites and metal-sulphides to be used in the field of waste

http://doi.org/10.31788/RJC.2026.1949883
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water treatment. Many types of nanomaterials and nanocomposites like cobalt, chromium, nickel, copper and iron oxide continue to be investigated, and their boosted photo-catalytic efficiency towards different organic compounds because of their layered structure and self-trapped excitation was reported. The integration of nanomaterials into the polymer matrix is expected to modify photo-catalytic and photo-detecting properties. Moreover, the ease of incorporation of nanoparticles into polymeric materials for tailoring the properties of ferrites has brought interest among the researchers.[7-9]

EXPERIMENTAL

The nanoparticle $CoCrFe_2O_4$ was synthesized through self-combustion technique using Fe $(NO_3)_3.9H_2O$ (1mole), Cr $(NO_3)_3.9H_2O$ (0.6mole) and Co $(NO_3)_2.6H_2O$ (0.4mole) solubilized in 50 ml of water. The 50% urea (w/w) solution was mixed with constant stirring and then heated to about 160°C. The reaction mixture generated ferrite particles in a dry condition by self-propagating combustion. The dry particles were ground, centrifuged and calcinated at 800^0C for 6 h in a muffle furnace. Nano composite PoPD/$CoCrFe_2O_4$ was synthesized via oxidative method of polymerization. A 0.1 mole of o-phenylenediamine solution and a $CoCrFe_2O_4$ solution were mixed and stirred for 30 minutes at a temperature of 0–5°C. To this, 0.01 mole of ammonium persulphate solution was added with stirring. The resulting nano composite washed and dried. Similarly, one more composition has been prepared, and details are presented in Table-1.

Table-1: PoPD / $CoCrFe_2O_4$ Nano Composites Composition

Sample	Code	$CoCrFe_2O_4$ (g)	oPD (g)	(PoPD/ $CoCrFe_2O_4$) %
Poly ($C_6H_4(NH_2)_2$)	PoPD	-	2	-
PoPD / $CoCrFe_2O_4$	PNC I	0.22	2	90:10
PoPD / $CoCrFe_2O_4$	PNC II	0.50	2	80:20

RESULTS AND DISCUSSION

XRD Study

Figure-1 reveals the diffraction pattern of polymer (PoPD), $CoCrFe_2O_4$ nanoparticles, and PoPD/$CoCrFe_2O_4$ nano composites (PNC I and PNC II). The polymer (PoPD) shows diffraction peaks in the range of 2θ = 10°-55°, revealing its partial crystalline nature. The XRD peaks of $CoCrFe_2O_4$ nanoparticles correspond well with JCPDS cards 22-1086 and 01-1027. The XRD patterns of PNC I and PNC II confirm the successful incorporation of $CoCrFe_2O_4$ nanoparticles into the PoPD matrix.[10]

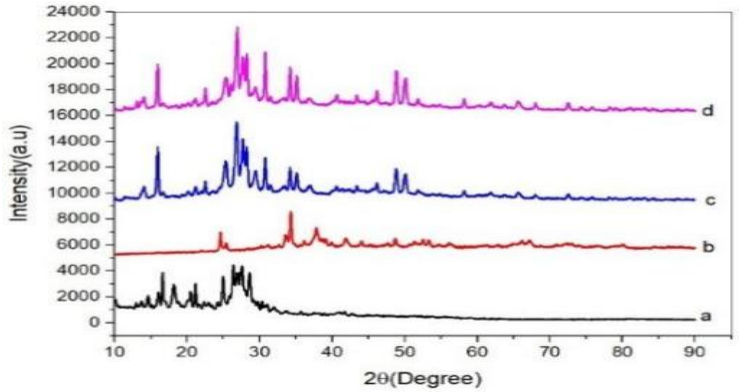


Fig.-1: XRD of (a) PoPD, (b) $CoCrFe_2O_4$ (c) PNC I, (d) PNC II

The crystallinity of the nanocomposites decreases with increasing $CoCrFe_2O_4$ content. The average crystalline sizes of $CoCrFe_2O_4$ nanoparticles and the nanocomposites (PNC I and PNC II) were calculated. The reduction in crystalline size of PNC I and PNC II with higher nanoparticle content is attributed to the confined segmental motion of the chains, which enhances dispersion of $CoCrFe_2O_4$ nanoparticles within the PoPD matrix. The average crystalline size of $CoCrFe_2O_4$ nanoparticles is 43.64nm decreased to 35.12nm (PNC I) and 29.68nm (PNC II) upon incorporation with PoPD. This reduction indicates that PoPD inhibits crystal growth and improves nanoparticle dispersion within the polymer matrix.

Vibrational Spectra

Figure-2 reveals the vibrational spectra of PoPD/$CoCrFe_2O_4$ and the PoPD polymer. The two peaks seen at 1675 and 1570 cm^{-1} are identified as the stretching of C=C in the benzenoid and quinoid structures. The peaks identified at 1365 and 1238 cm^{-1} are due to C-N stretching vibrations. The other peaks at 3315 and 3047 cm^{-1} are due to NH- and -NH_2 stretching vibrations.[11] From these analyses, we summarized that the polymer (PoPD) structure agrees with previous findings. The FT-IR peaks of PNC I and PNC II nano composites (PoPD/$CoCrFe_2O_4$) are marginally shifted upward with respect to the polymer (PoPD). The characteristic vibrational bands clearly suggested that the polymer (PoPD)/ $CoCrFe_2O_4$ nano composites have a good distribution of $CoCrFe_2O_4$ nanoparticles in the PoPD matrix.[12]

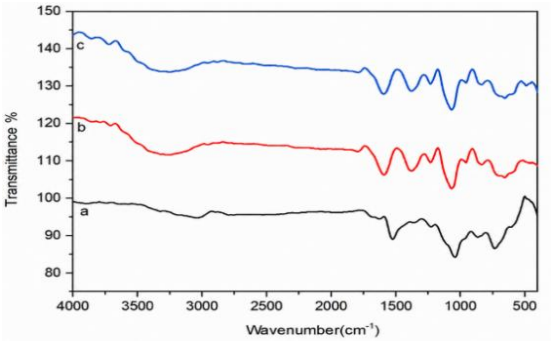


Fig.-2: Vibrational spectra of (a) PoPD, (b) PNC I, (c) PNC II

UV- Visible Spectra

In the absorption spectra of polymer (PoPD), PNC I and PNC II (PoPD/ $CoCrFe_2O_4$) nano composites are illustrated in Fig.-3. It shows three prominent bands appeared at 252, 391 and 460 nm for polymer (PoPD). The absorption peaks at 252 and 391 nm are described as polymer (PoPD) benzenoid rings due to π-π* transitions. The wide absorption band that appeared at 460 nm is assigned to intramolecular charge transfer between benzenoid and quinoid units. These absorption results clearly indicate that the polymer (PoPD) bands shifted to actual positions in the PNCI and PNCII (PoPD/$CoCrFe_2O_4$) nanocomposites, revealing that $CoCrFe_2O_4$ nanoparticles were successfully incorporated into the PoPD matrix. [13]

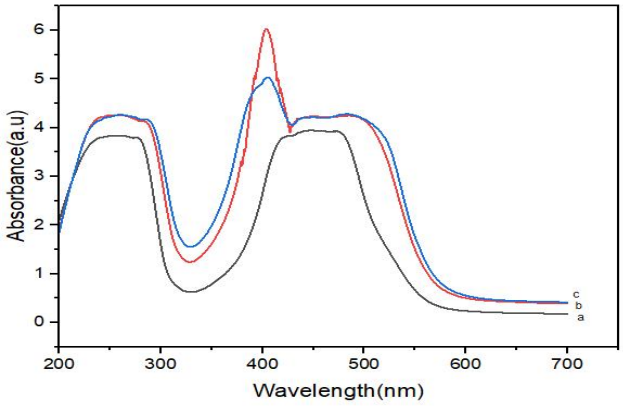


Fig.-3: UV – Visible spectra of (a) PoPD, (b) PNC I, (c) PNC II

SEM

The SEM micrographs of nanoparticles $CoCrFe_2O_4$ and nano composites PNC I and PNC II (PoPD/ $CoCrFe_2O_4$) are illustrated in Fig.-4. It is globular morphology of nanoparticles $CoCrFe_2O_4$. The SEM images of nano composites PNC I and PNC II (PoPD/ $CoCrFe_2O_4$) show a distinct morphology compared with nanoparticles $CoCrFe_2O_4$. These SEM results of PoPD/ $CoCrFe_2O_4$ nano composites, demonstrates that the morphology is modified upon incorporation of $CoCrFe_2O_4$ nanoparticles.[14] Further, the proportion of $CoCrFe_2O_4$ nanoparticle is also leads to morphological changes of PoPD/ $CoCrFe_2O_4$ nano composites. EDAX spectra of the nanoparticles and nano composites are illustrated in Table-2. The results show that

Co, Cr and Fe metals are present in the expected composition. Further, nanoparticles $CoCrFe_2O_4$ are well dispersed in the nano composite PoPD/ $CoCrFe_2O_4$.

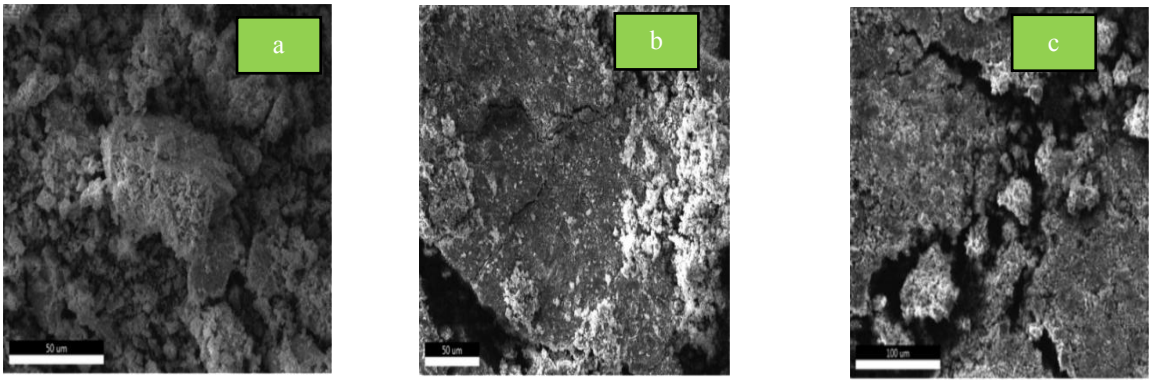


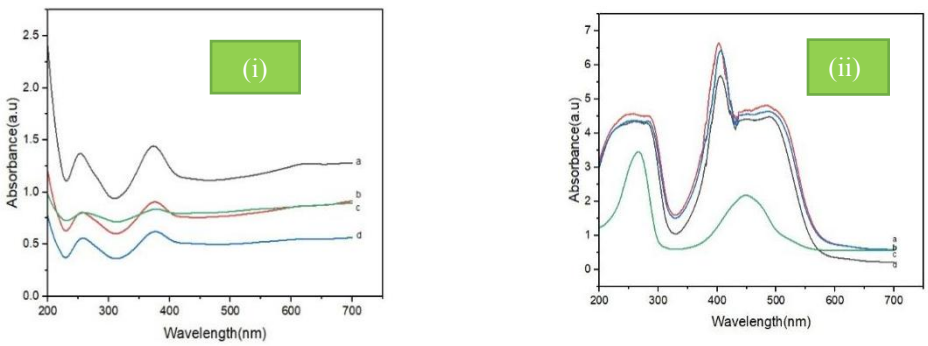
Fig.-4: SEM Images of (a) $CoCrFe_2O_4$, (b) PNC I, (c) PNC II

Table-2: SEM- EDAX Data for $CoCrFe_2O_4$ Nanoparticles

Element	$CoCrFe_2O_4$ nanoparticles		PNC I		PNC II	
	Wt %	Atomic %	Wt %	Atomic %	Wt %	Atomic %
CK	3.65	10.54	35.77	48.07	33.57	47.29
OK	18.69	40.50	16.09	20.01	14.39	17.38
NK	-	-	21.36	21.09	23.48	24.83
SK	-	-	15.18	7.48	7.76	4.10
CrK	24.68	16.46	3.58	1.09	6.57	2.14
FeK	41.30	25.63	5.94	1.68	11.20	3.39
CoK	11.67	6.86	2.18	0.58	3.03	0.87

Photo-catalytic Degradation

To evaluate the photo-catalytic performance of nanoparticles and nano composites, degradation studies were carried out using malachite green (MG) dye under controlled conditions to assess the catalytic efficiency under visible light. The dye concentration in the solution was monitored at regular time gap using UV–Visible spectroscopy, where a decrease in absorbance at 380 and 440 nm indicated the removal of MG dye.[15] The absorption peaks of $CoCrFe_2O_4$ nanoparticles and nano composites (PNC1 and PNC II) and the change in dye concentration are illustrated in Fig.-5. It shows the % degradation of malachite green over time under continuous visible light illumination in the presence of nanoparticles and nano composites. As seen, the degradation rate increased steadily with time, reaching a maximum degradation within 24 h of exposure. It shows the % degradation of malachite green over time under continuous visible light illumination in the presence of nanoparticles and nano composites.[16] As seen, the degradation rate increased steadily with time, reaching a maximum degradation within 24 h of exposure.



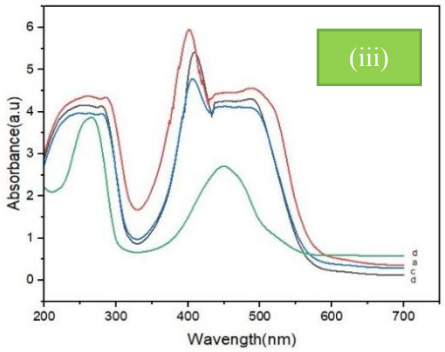


Fig.-5: Photo-catalytic Degradation of (i)$CoCrFe_2O_4$ Nanoparticle (ii) PNC I (iii) PNC II (a)Dye (b)1hr (c) 2hr (d) 24hr

CONCLUSION

The $CoCrFe_2O_4$ nanoparticles were synthesized by the self-combustion method, and PoPD/$CoCrFe_2O_4$ nano composites were created using in-situ chemical oxidative polymerization of o-phenylenediamine compound, followed by the dispersion of varying concentrations of $CoCrFe_2O_4$ nanoparticles. XRD, FT-IR and UV–Vis spectral analyses confirmed the interaction between PoPD and $CoCrFe_2O_4$ nanoparticles in the PNC I and PNC II composites. SEM images revealed PoPD/$CoCrFe_2O_4$ nanocomposites, which are significantly influenced by the incorporation of $CoCrFe_2O_4$ nanoparticles. Photocatalytic studies demonstrated that both the synthesized $CoCrFe_2O_4$ nanoparticles and the PoPD/$CoCrFe_2O_4$ nano composites exhibit high degradation efficiency, which depends on the specific surface area.

ACKNOWLEDGEMENTS

The author sincerely acknowledges the support from Dr N.G.P. Arts and Science College for this research.

CONFLICT OF INTERESTS

The authors state that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present study is associated with the *SDG-6: Clean Water and Sanitation*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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Cite this article as: M. Mohanraj *et al.*, *Rasayan J. Chem.*, 19(4), 2320-2325(2026), https://doi.org/10.31788/RJC.2026.1949883

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[RJC-9883/2026]