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Visible-Light-Induced Photocatalytic Degradation of Methyl Orange by Cobalt (II) Thiosemicarbazone Complexes

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Abstract: ***A series of p-substituted isonitrosoacetophenone-4-phenylthiosemicarbazone cobalt(II) complexes were synthesized and investigated for their photocatalytic activity toward the degradation of methyl orange (MO), a common azo dye pollutant in industrial wastewater. The synthesized complexes were characterized using elemental analysis, molar conductance, magnetic susceptibility, UV–Visible spectroscopy, FTIR spectroscopy, and TG–DTA. Photocatalytic degradation studies were carried out under visible-light irradiation using a tungsten filament lamp. The effects of irradiation time, catalyst dosage, and catalyst reusability were systematically evaluated. The cobalt(II) complexes exhibited efficient photocatalytic activity, resulting in significant degradation of methyl orange under the optimized reaction conditions. The enhanced catalytic performance is attributed to the favourable redox properties of cobalt(II) and the chelating nature of the thiosemicarbazone ligands, which facilitate the generation of reactive oxygen species during the photocatalytic process. The catalysts also demonstrated good stability and reusability over successive cycles. These findings indicate that the synthesized cobalt(II) complexes are efficient and environmentally benign photocatalysts with potential applications in the treatment of dye-contaminated wastewater.***

Keywords: Cobalt (II) complexes; Isonitrosoacetophenone phenylthiosemicarbazones; Photocatalytic degradation; Methyl orange; Wastewater treatment.

I. INTRODUCTION

Water contamination by synthetic organic dyes has become one of the major environmental challenges associated with rapid industrial development [1,2]. Effluents generated from textile, paper, leather, plastic, printing, pharmaceutical, and dye manufacturing industries contain significant amounts of colored organic compounds that are often resistant to natural degradation [1,2]. Even at very low concentrations, these dyes impart intense coloration to water bodies, reducing sunlight penetration, disrupting aquatic ecosystems, and adversely affecting photosynthetic activity [1–3]. Many synthetic dyes and their degradation products are toxic, mutagenic, and potentially carcinogenic, necessitating the development of efficient wastewater treatment technologies [1–3].

Among the numerous synthetic dyes, methyl orange (MO) is a widely used anionic azo dye and serves as a representative model pollutant for evaluating the efficiency of photocatalytic materials [4,14]. The presence of an azo (–N=N–) chromophore together with aromatic rings imparts remarkable chemical and photochemical stability to methyl orange, making it resistant to conventional treatment methods such as biological degradation, sedimentation, and filtration [2,4,14]. Consequently, the complete removal of methyl orange from industrial wastewater remains a significant environmental concern [7,12,14].

Several treatment techniques, including adsorption, coagulation–flocculation, membrane filtration, electrochemical oxidation, and biological treatment, have been employed for dye removal [1,2,5]. Although these methods are effective to some extent, they often suffer from limitations such as incomplete degradation, high operational costs, generation of secondary pollutants, or the requirement for additional disposal processes [1,5]. In contrast, photocatalytic degradation has emerged as an attractive advanced oxidation process because it transforms complex organic pollutants into environmentally benign products through oxidative mineralization rather than simply transferring contaminants from one phase to another [3,4,12].

The photocatalytic process is initiated when a photoactive catalyst absorbs light energy equal to or greater than its band-gap energy [3,4]. Excitation of the catalyst generates electrons in the conduction band and holes in the valence band [3,4]. These photogenerated charge carriers participate in surface redox reactions with dissolved oxygen and water molecules, producing reactive oxygen species such as hydroxyl radicals (•OH), superoxide radicals ($O_2^{\bullet -}$), and hydrogen peroxide [3,4,12].

These highly reactive intermediates attack the chromophoric structure of methyl orange, resulting in cleavage of the azo linkage, opening of aromatic rings, formation of low-molecular-weight intermediates, and ultimately complete mineralization into carbon dioxide, water, and inorganic ions [4,12,14].

In recent years, coordination compounds of transition metals have gained considerable importance as photocatalytic materials owing to their tunable electronic structures, variable oxidation states, and efficient charge-transfer characteristics [9–17]. Cobalt(II) complexes, in particular, have attracted increasing attention because cobalt can readily undergo reversible Co(II)/Co(III) redox transformations, facilitating electron-transfer reactions during photocatalysis [9,10,13,15].

Complexation of cobalt with Schiff base thiosemicarbazone ligands enhances the stability of the metal center, improves light absorption through ligand-to-metal and metal-to-ligand charge-transfer transitions, and suppresses rapid recombination of photogenerated electron–hole pairs [13,15,18,19]. These characteristics contribute significantly to improved photocatalytic efficiency under visible-light irradiation [10,13,16].

The catalytic behaviour of cobalt complexes is strongly influenced by the coordination environment around the metal ion and the electronic nature of the coordinating ligands [13,18,19]. Phenyl thiosemicarbazone ligands provide multiple donor atoms, including nitrogen and sulfur, enabling the formation of stable chelate complexes with cobalt(II) [18,19]. The extended π-conjugation of these ligands facilitates charge delocalization and enhances light harvesting, while suitable substituents on the aromatic ring can further modify the electronic properties and catalytic activity of the resulting complexes [13,18,19].

In the present study, a series of cobalt(II) complexes of isonitrosoacetophenone phenyl thiosemicarbazone ligands were investigated for their photocatalytic degradation efficiency towards methyl orange dye under visible-light irradiation using a tungsten filament lamp. The degradation process was monitored by recording the decrease in absorbance of methyl orange at 480 nm using UV–Visible spectroscopy. The influence of irradiation time, catalyst loading, and catalyst recyclability on degradation efficiency was systematically evaluated. The results provide valuable insight into the potential application of cobalt(II) thiosemicarbazone complexes as efficient, recyclable, and environmentally sustainable photocatalysts for the treatment of dye-contaminated wastewater.

II. MATERIALS AND METHODS

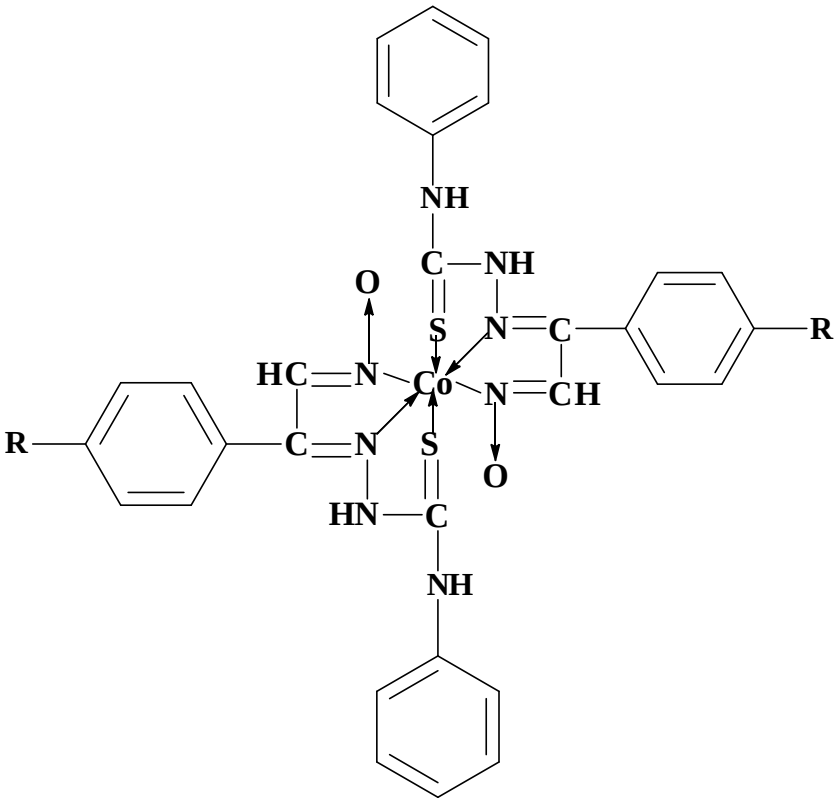
Analytical reagent grade chemicals were used throughout the study. Cobalt chloride hexahydrate ($CoCl_2 \cdot 6H_2O$) was obtained from SDFCL and used without further purification. Methyl Orange Dye was purchased from Sigma Aldrich. All solvents were purified and distilled by standard methods before use**[20]**.

The thiosemicarbazone ligands were prepared via the condensation of 4-phenylthiosemicarbazide with para-substituted isonitrosoacetophenones and characterized in our laboratory**[21]**.

A. Synthesis of Co(II) Complexes

The Co(II) complexes of ligands were synthesized **[22]** by using an aqueous solution of Co(II) chloride hexahydrate (1 mmol) and *p*-substituted isonitrosoacetophenones phenyl thiosemicarbazones(INAP-PTSC) (2 mmol). The preparation of cobalt complexes was carried in 2:1 molar ratio. The complexes derived from p-substituted isonitrosoacetophenones phenyl thiosemicarbazone (INAP-PTSC) were prepared by dissolving 2 moles of ligands in 20 ml ethanol and 1 mole of $CoCl_2.6H_2O$ metal salt in 10 ml ethanol; NH_3 was added dropwise with constant stirring. The pH was maintained between 7-8 and stirred for 30 minutes. The solid mass was separated by filtration and washed repeatedly with ethanol and dried.

The cobalt content of the synthesized complexes was estimated by complexometric titration using standard analytical procedures**[23]**. The molar conductance of the complexes was measured in 10^{-3} M DMF solutions using a EQUIP-TRONICS EQ-660A digital conductivity meter fitted with a magnetic stirrer and having a cell constant of 1. Room-temperature magnetic susceptibility measurements were carried out using a Gouy balance (Batra Trading Company, Model GMX-TR2), with $Hg[Co(SCN)_4]$ employed as the standard calibrant. The effective magnetic moments were calculated after applying diamagnetic corrections for the ligand moieties using Pascal's constants**[24]**. The electronic spectra of the cobalt(II) complexes were recorded in DMF on a Shimadzu UV-1800A UV–Visible spectrophotometer. Thermal studies, including thermogravimetric (TG) and differential thermal analysis (DTA), were performed using a Rigaku Thermo Plus-8120 TG-DTA instrument. Fourier-transform infrared (FT-IR) spectra of the ligands and their corresponding metal complexes were obtained in the 4000–400 cm^{-1} region using an INFRA-3000 spectrometer and interpreted with reference to reported literature data**[25]**. The proposed structures of the cobalt(II) complexes are depicted in given scheme.1



Where R= -H, -CH_3, -OCH_3, -OH, -Cl, -NO_2

Co(II) Complex of Isonitrosoacetophenonephenylthiosemicarbazone

Scheme 1: Co(II) complexes of Isonitrosoacetophenonephenylthiosemicarbazone

B. Preparation of Methyl orange dye solution

The stock solution of methyl orange (MO) was prepared at a concentration of 100 ppm by dissolving 100 mg of the dye in 1000 mL of deionized water. A 10 ppm working solution was then prepared by transferring 50 mL of the stock solution into a 500 mL volumetric flask and making up the volume with deionized water. This diluted solution was employed for the photocatalytic degradation studies.

C. Photocatalytic Activity

The photocatalytic activity of the cobalt(II) complexes was assessed through the degradation of methyl orange (MO) dye under illumination from a tungsten filament lamp. The degradation experiments were performed using a 10 ppm MO solution at different irradiation times (30, 60, 90, and 120 min) and with varying catalyst dosages (25, 50, 75, and 100 mg). The absorbance of the dye solution was recorded both before and after exposure to light, in the presence as well as the absence of the catalyst. Following irradiation, the catalyst was recovered by centrifugation, and the absorbance of the resulting supernatant was measured using a UV–Visible spectrophotometer at 480 nm. The percentage degradation efficiency was determined from the change in absorbance using the following equation:

$$\% \text{ Degradation} = \frac{A_0 - A_t}{A_0} \times 100$$

Where A_0 and A_t denotes absorbance before and after photocatalytic degradation of Methylorange

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III. RESULTS AND DISCUSSIONS

A. Photocatalytic Activity

For determination of λ_{max} Methyl orange dye was used of 100 ppm concentration (100 mg/L) and was found at 480 nm shown in Fig.1.

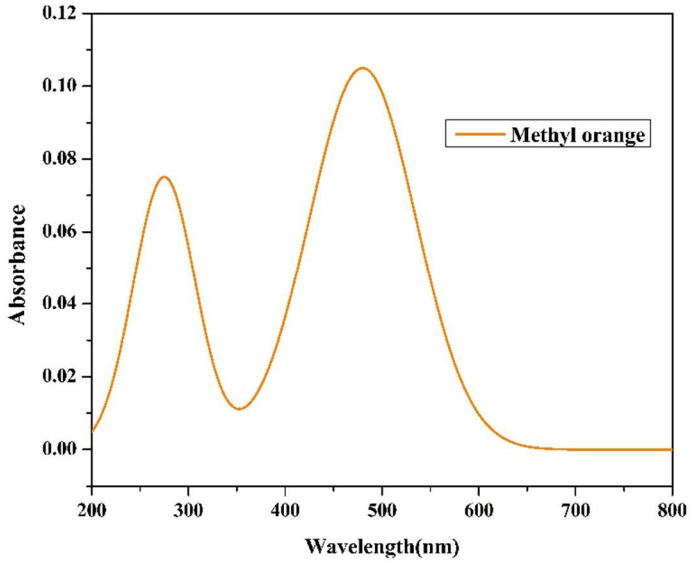

Fig. 1. Wavelength of maximum absorbance of Methyl orange dye λ_{max} 480 nm

B. Determination of initial absorbance (Ao)

For determination of initial absorbance (A_o) 10 ppm dye solution was used and absorbance obtained at A_o = 0.105 is show in Fig 1.

C. Effect of irradiation time on the photocatalytic degradation of methyl orange

The photocatalytic degradation of methyl orange (MO) was investigated using a series of synthesized cobalt(II) complexes (CL1–CL6) under visible-light irradiation.

The experiments were carried out by maintaining a constant methyl orange concentration of 10 ppm and a catalyst loading of 50 mg. The degradation process was monitored by measuring the absorbance of the dye solution at its characteristic absorption maximum (λ_{max} = 480 nm) after irradiation intervals of 30, 60, 90 and 120 min.

The cobalt(II) complexes exhibited excellent photocatalytic activity towards the degradation of methyl orange. A gradual decrease in the absorbance of the dye solution was observed with increasing irradiation time for all the synthesized complexes, indicating continuous degradation of the dye molecules. Correspondingly, the percentage degradation increased steadily throughout the irradiation period.

Among the cobalt(II) complexes, CL1 exhibited the lowest photocatalytic activity with 19.04% degradation after 30 min, which increased to 92.38% after 120 min. In contrast, CL6 showed the highest activity, achieving 28.57% degradation after 30 min and 97.14% degradation after 120 min. The remaining complexes displayed intermediate degradation efficiencies.

The outstanding photocatalytic performance of the cobalt(II) complexes can be attributed to the efficient photoinduced charge separation at the cobalt centre, which promotes the formation of highly reactive hydroxyl (•OH) and superoxide ($O_2^{\bullet -}$) radicals. These reactive oxygen species effectively attack the azo bond and aromatic framework of methyl orange, resulting in progressive degradation of the dye. Furthermore, the substituent present on the ligand framework influences the electronic environment around the cobalt ion, thereby affecting charge transfer efficiency and photocatalytic performance.

The nitro-substituted complex (CL6) exhibited the highest activity, whereas the unsubstituted complex (CL1) showed comparatively lower degradation efficiency.

Table 1: Effect of time on degradation

Sr. No	Complex	Time	Absorbance	% Degradation
1	CL1	30	0.085	19.04
		60	0.05	52.38
		90	0.022	79.04
		120	0.008	92.38
2	CL2	30	0.083	20.95
		60	0.048	54.28
		90	0.02	80.95
		120	0.007	93.33
3	CL3	30	0.081	22.85
		60	0.046	56.19
		90	0.019	81.9
		120	0.006	94.28
4	CL4	30	0.079	24.76
		60	0.044	58.09
		90	0.017	83.8
		120	0.005	95.23
5	CL5	30	0.077	26.66
		60	0.042	60
		90	0.016	84.76
		120	0.004	96.19
6	CL6	30	0.075	28.57
		60	0.04	61.9
		90	0.014	86.66
		120	0.003	97.14

L1= INAP-PTSC L2 = p-Me INAP-PTSC L3 = p-OMe INAP-PTSC,
L4 = p-OH INAP-PTSC L5 = p-Cl INAP-PTSC , L6 = p-NO_2 INAP

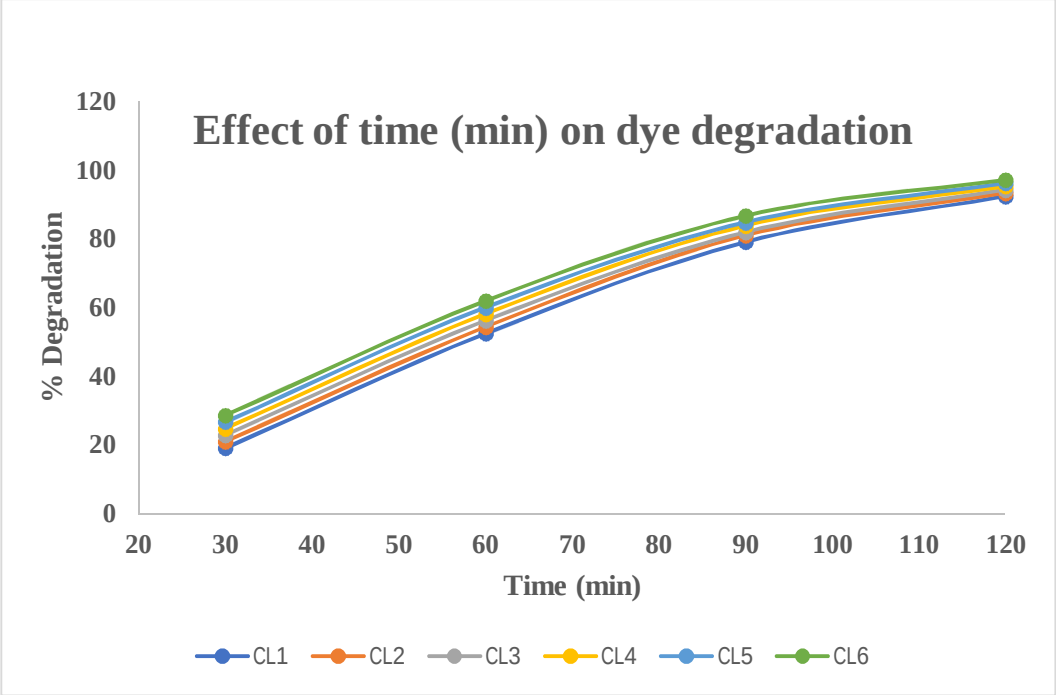

Fig.2. Effect of time (min) on dye degradation for Cobalt complexes

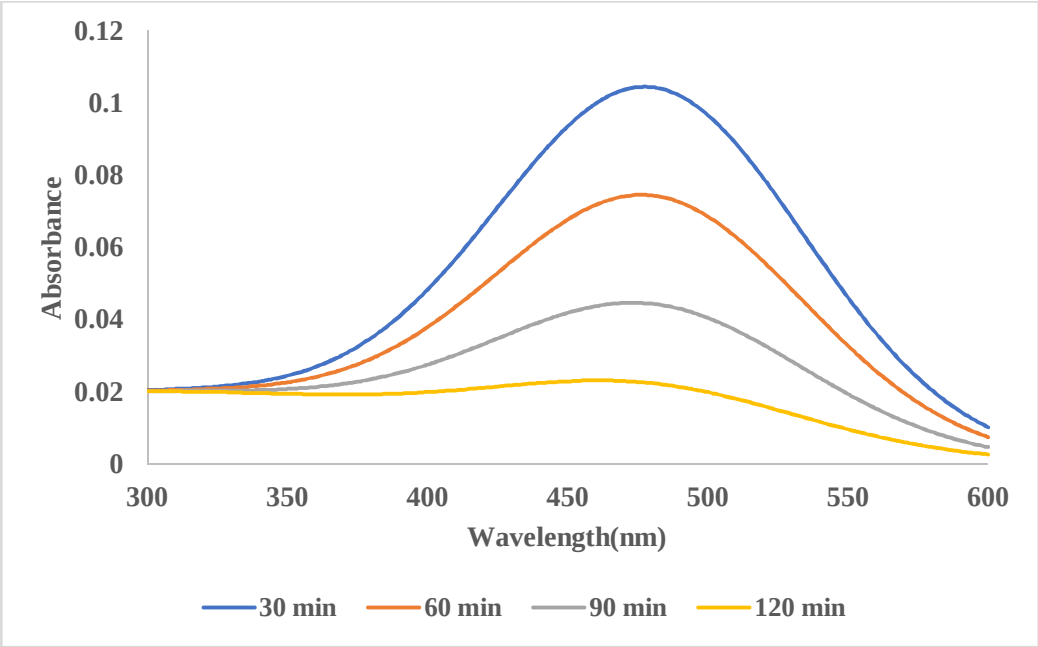

Fig.3. UV–Vis absorption spectra of methyl orange solution recorded at different irradiation times(30–120 min) in the presence of the representative CL1 complex photocatalyst.

D. Effect of catalyst dosage on the photocatalytic degradation of methyl orange

The influence of catalyst dosage on the photocatalytic degradation of methyl orange was investigated using the synthesized cobalt(II) complexes (CL1–CL6). The experiments were performed by maintaining a constant methyl orange concentration of 10 ppm and a fixed irradiation time, while varying the catalyst dosage from 25 to 100 mg. The degradation efficiency was evaluated by monitoring the decrease in absorbance at the characteristic absorption wavelength of methyl orange (λ_{max} = 480 nm).

The photocatalytic degradation efficiency of the cobalt(II) complexes increased progressively with increasing catalyst dosage. For all complexes, the absorbance of methyl orange decreased continuously as the catalyst loading was increased from 25 to 100 mg, indicating enhanced photocatalytic activity due to the availability of a greater number of active surface sites.

Among the cobalt(II) complexes, CL1 exhibited degradation efficiencies of 27.61%, 79.04%, 84.76% and 89.52% at catalyst dosages of 25, 50, 75 and 100 mg, respectively. Similarly, CL2 achieved degradation efficiencies ranging from 29.52% to 90.47%, while CL3 showed values between 31.42% and 91.42%. The hydroxyl-substituted complex CL4 exhibited a degradation efficiency of 92.38% at a catalyst dosage of 100 mg. The highest photocatalytic activity was observed for CL6, which exhibited percentage degradation efficiencies of 37.14, 86.66, 90.47 and 95.23 at catalyst dosages of 25, 50, 75 and 100 mg, respectively.

The increase in degradation efficiency with catalyst dosage is attributed to the increased number of photo catalytically active sites and enhanced generation of photogenerated electron–hole pairs. As the catalyst loading increases, more hydroxyl (•OH) and superoxide ($O_2^{\bullet -}$) radicals are produced, leading to more efficient oxidation of methyl orange molecules. Within the investigated dosage range, no decline in activity was observed, indicating that 100 mg provided the highest photocatalytic efficiency under the selected experimental conditions.

Table 2. Effect of amount of catalyst (cobalt complexes) on degradation

Sr.No	Complex	Amount	Absorbance	% Degradation
1	CL1	25	0.076	27.61
		50	0.022	79.04
		75	0.016	84.76
		100	0.011	89.52
		25	0.074	29.52

2	CL2	50	0.021	80
		75	0.015	85.71
		100	0.01	90.47
3	CL3	25	0.072	31.42
		50	0.019	81.9
		75	0.014	86.66
		100	0.009	91.42
4	CL4	25	0.07	33.33
		50	0.018	82.85
		75	0.013	87.61
		100	0.008	92.38
5	CL5	25	0.068	35.23
		50	0.016	84.76
		75	0.012	88.57
		100	0.007	93.33
6	CL6	25	0.066	37.14
		50	0.014	86.66
		75	0.01	90.47
		100	0.005	95.23

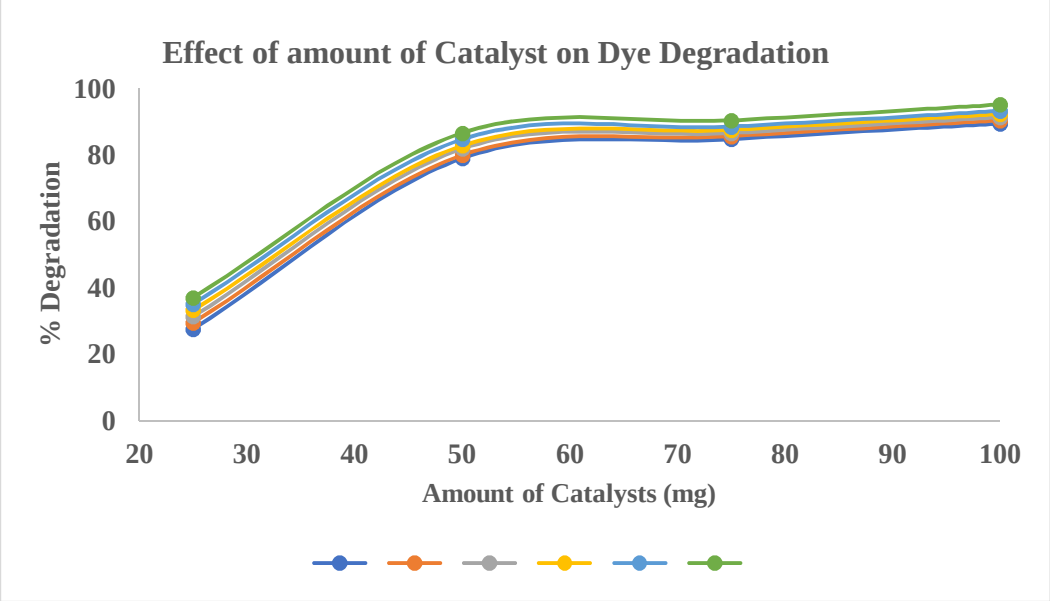

Fig.4. Effect of amount of catalyst on dye degradation for Cobalt complexes

E. Reusability Study of the Cobalt Metal Complexes

The reusability of the synthesized Co(II) complexes was evaluated by performing five consecutive photocatalytic degradation cycles under identical experimental conditions. After each cycle, the catalyst was recovered by filtration, washed thoroughly with distilled water followed by ethanol to remove any adsorbed dye molecules, dried, and reused in the subsequent experiment. The absorbance of the degraded dye solution was measured after each catalytic cycle, and the corresponding percentage degradation was calculated.

The reusability results demonstrate that all the synthesized complexes retained a significant proportion of their photocatalytic activity after repeated use. A slight increase in absorbance and a corresponding decrease in degradation efficiency were observed with increasing catalytic cycles, indicating gradual deactivation of the catalyst. This reduction in activity may be attributed to minor catalyst loss during recovery, partial blockage of active surface sites by reaction intermediates or degradation products, and slight structural or surface modifications occurring during repeated irradiation.

Among the cobalt complexes, CL6 exhibited the highest catalytic stability, maintaining a degradation efficiency of 79.36% during the first cycle and 74.41% after the fifth cycle, corresponding to a decrease of only about 4.95 %. Similarly, CL5, CL4, CL3, and CL2 also retained high degradation efficiencies after five successive cycles, indicating excellent recyclability. Although CL1 showed a comparatively lower degradation efficiency, it also remained catalytically active throughout the study, demonstrating acceptable operational stability.

The small decrease in degradation efficiency observed after repeated reuse confirms that the synthesized metal complexes possess good structural integrity and excellent recyclability. These findings suggest that the complexes are promising reusable photocatalysts for the degradation of methyl orange dye and may be suitable for practical wastewater treatment applications due to their sustained catalytic performance over multiple reaction cycles.

Table 3. Reusability of Catalysts for Cobalt complexes

Comp	No.of Catalytic cycle	Absorbance (At)	% Degradation	Comp	No.of Catalytic cycle	Absorbance (At)	% Degradation
CL1	1	0.035	65.34	CL4	1	0.025	75.18
	2	0.037	65.36		2	0.026	74.81
	3	0.04	60.39		3	0.029	71.14
	4	0.04	60.39		4	0.03	70.73
	5	0.04	60.39		5	0.031	70.31
CL2	1	0.029	70.36	CL5	1	0.022	77.42
	2	0.03	70.05		2	0.023	77.03
	3	0.033	66.11		3	0.026	73.36
	4	0.034	65.75		4	0.027	72.92
	5	0.035	65.32		5	0.028	72.48
CL3	1	0.027	72.84	CL6	1	0.02	79.36
	2	0.028	72.48		2	0.021	78.95
	3	0.031	68.72		3	0.024	75.28
	4	0.032	68.28		4	0.025	74.84
	5	0.033	67.85		5	0.026	74.41

F. Mechanism for Dye Degradation

Upon exposure to visible light irradiation, cobalt complex absorbs photons and undergoes photoexcitation, resulting in the generation of electron–hole pairs. The efficient separation of these charge carriers enhances the photocatalytic activity of the complex and suppresses electron–hole recombination. The photogenerated electrons react with dissolved oxygen molecules to produce superoxide radical anions ($O_2^{\bullet -}$), while the photogenerated holes oxidize water molecules or surface hydroxyl groups to generate highly reactive hydroxyl radicals ($^{\bullet}OH$).

The superoxide radicals may further react with H^+ ions to form hydroperoxyl radicals ($HO_2^{\bullet}$), which contribute to the formation of additional reactive oxygen species. These radicals, particularly $^{\bullet}OH$ and $^{\bullet}O_2^-$, play a crucial role in the oxidative degradation of Methyl Orange molecules. The reactive species attack the azo (–N=N–) bond and aromatic rings of the dye, resulting in cleavage of the chromophoric structure and progressive decomposition into smaller organic intermediates.

Further oxidation of these intermediates leads to their mineralization into environmentally benign products such as carbon dioxide, water, and inorganic ions. The synergistic action of hydroxyl and superoxide radicals significantly accelerates the degradation and decolorization of Methyl Orange. Therefore, the cobalt complex acts as an efficient photocatalyst by facilitating charge separation and promoting the generation of reactive oxygen species responsible for dye degradation.

IV. CONCLUSION

The photocatalytic degradation of Methyl Orange (MO) was examined using a series of cobalt(II) complexes derived from p-substituted isonitrosoacetophenone phenyl thiosemicarbazone ligands. The synthesized complexes showed notable photocatalytic performance, with degradation of more than 85% of MO achieved after 120 min of irradiation.

Among the studied complexes, CL6, incorporating the p-nitro-substituted ligand, showed the maximum degradation efficiency. The enhanced photocatalytic behaviour of CL6 can be associated with the strong electron-withdrawing effect of the nitro group, which may facilitate charge separation and decrease the probability of electron–hole recombination. Consequently, the formation of reactive oxygen species may be enhanced, contributing to the effective breakdown of the MO molecules. CL5, containing the p-chloro substituent, also exhibited relatively high photocatalytic activity compared with the unsubstituted and electron-donating derivatives.

The complexes containing electron-releasing groups, namely methyl, methoxy, and hydroxyl substituents, exhibited comparatively reduced photocatalytic efficiencies. These groups increase electron density in the ligand system and may not promote charge separation as effectively as electron-withdrawing substituents. Among the series, the unsubstituted complex CL1 showed the lowest degradation efficiency.

The results clearly indicate that the nature of the substituent attached to the ligand has a considerable influence on the photocatalytic performance of the cobalt(II) complexes. Electron-withdrawing groups, particularly the nitro group, appear to favour enhanced photocatalytic degradation of MO. Hence, the synthesized cobalt(II) complexes may be considered potential photocatalysts for the removal of organic dyes from contaminated water and for environmental remediation applications.

$$\text{Co(II) Complex} + h\nu \longrightarrow e^- + h^+$$

$$e^- + O_2 \longrightarrow O_2^{\bullet-}$$

$$h^+ + H_2O \longrightarrow {}^{\bullet}OH + H^+$$

$${}^{\bullet}OH, O_2^{\bullet-} + \text{MO} \longrightarrow \text{Degraded Products}$$

$$\text{Products} \longrightarrow CO_2 + H_2O + SO_4^{2-} + NO_3^-$$

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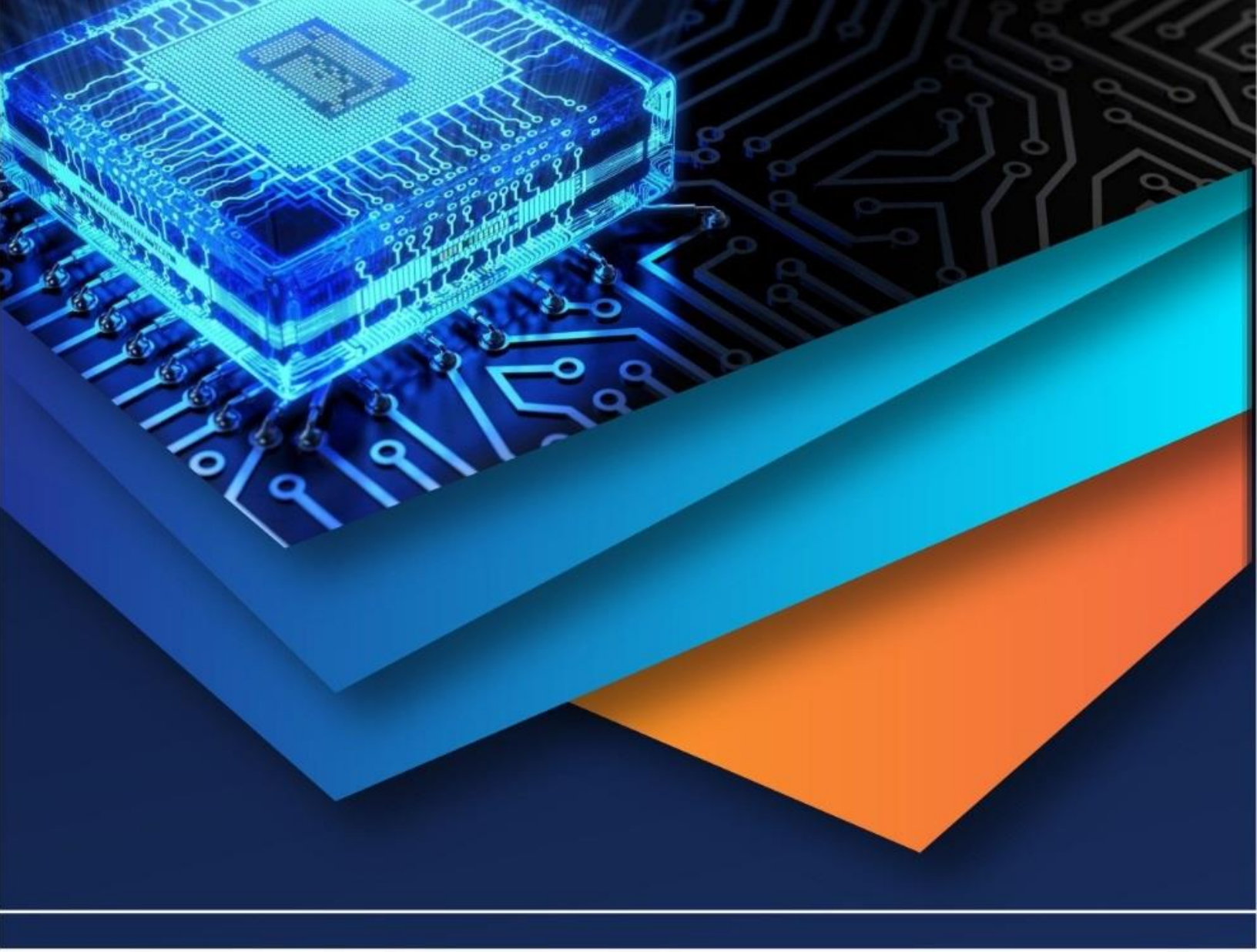

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