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Study on Photocatalytic Degradation of Methyl Orange Dye using Metal Complexes Obtained from 4-Chloroisonitrosoacetophenone cyclohexylthiosemicarbazone as a Heterogeneous Catalyst

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Abstract: ***The synthesized 4-chloroisonitrosoacetophenone cyclohexylthiosemicarbazone metal complexes have been employed to study the photocatalytic degradation of azo dye acting as a heterogeneous catalyst. The environmentally friendly catalysts were created with the intention of using them to degrade the organic dye. The photocatalytic activities of complexes were investigated using UV–Vis spectra, with methyl orange serving as a target dye impurity for degradation. Electronic absorbance spectra of methyl orange organic dye showed broad absorption at 480 nm. Using different irradiation periods (30, 60, 90 and 120 min), catalytic dosages (25, 50, 75 and 100 mg), the photocatalytic degradation of methyl orange was examined. Each complex exhibits strong catalytic activity for the degradation of methyl orange. Among the irradiation period, at 120 min catalysts show the highest percentage degradation. Similarly, in large amount of 100 mg of catalysts irradiation exposure of light, maximum degradation occurred. When the catalysts were tested on fresh dye samples (5 trials), the decolorization efficiency dropped effectively, indicating the efficient reusability of all the metal complexes. The possible mechanism for dye degradation has been suggested.***

Keywords: Photocatalytic activity, heterogeneous catalyst, azo dye, metal complexes, dye degradation

I. INTRODUCTION

The hazardous organic compounds, such as dyes from textile, drugs, leather-based colouring, cosmetics, paper formation and other industries, have been widely discarded. Amongst these, azo dyes are the main group of commercially produced dyes that are widely utilized in the textile sector due to their affordability and adaptability, but they also pose a serious environmental risk [[1]-[3]]. However, because these azo dyes are extremely susceptible to heat, light, and oxidizing agents, removing them from effluents effectively is a difficult task. Methyl orange [sodium 4-{[4-(dimethylamino) phenyl] diazenyl}benzene-1-sulfonate] is an orange anionic dye that comes from a variety of hazardous dyes. MO is an acidic azo dye that is frequently employed as a dyeing component in textiles and as an inductor in acid-base titrations. The most popular use of MO in both industry and research sectors is as an indicator [[4]]. The azo (-N=N-) bonds and the chromophores and auxochromes that go along with them determine the hue of azo dyes. The azo bonds, which can be reduced by electrons in the conduction band or oxidized by hydroxyl radicals or positive holes. Dyes become discoloured when -N=N- bonds are broken [[5]]. Numerous techniques, including as catalytic reduction, oxidation, photocatalytic degradation, and adsorption as well as reducing agents, and oxidizing agents have been suggested for the decolorization, detoxification, treatment of MO dye [[6]] biological treatments, Fenton systems [[7]], and coagulation/ flocculation [[8]]. Due to their high prices and requirement for safe processing, traditional dye removal techniques such precipitation, filtration, membrane separation, and biodegradation are frequently inadequate. Transition metal-based coordination compounds have demonstrated promise in increasing photocatalytic effectiveness, and photocatalytic methods are being developed to breakdown and mineralize poisonous organic dyes into less hazardous molecules [[9],[10]]. Thiosemicarbazones are powerful chelating ligands and have been thoroughly studied because of their noteworthy biological activities. The potential of TSC complexes to function as catalysts in a variety of reactions has been studied, and they are nearly limited to various organic reactions [[11]].

The study has demonstrated that thiosemicarbazones can be changed to enhance their catalytic efficiency, for example, by adjusting the metal coordination environment or changing the substituents on the terminal nitrogen [[12]]. While a number of heterocyclic metal(II) complexes of thiosemicarbazones and their mixed complexes with isothiocyanates or thiocyanates as co-ligands have been thoroughly studied. The crystallinity, shape, size, optical, and photocatalytic properties may be impacted by the addition of thiocyanates or isothiocyanates as co-ligands [[13]]. Because of their versatility, a variety of metal-thiosemicarbazone complexes can be investigated, resulting in a broad spectrum of catalytic actions and efficiency. Thus, thiosemicarbazone compounds are a new and efficient method for catalysing the hydrogen evolution reaction [[14]]. Through photocatalytic action, complexes and their oxides play a significant role in the degradation of dye molecules.

The 4-chloroisonitrosoacetophenone cyclohexylthiosemicarbazone ligand and their Co(II), Ni(II), Cu(II) and Zn(II) metal complexes were synthesized and characterised by various analytical techniques like elemental analysis, magnetic susceptibility, molar conductance, TG-DTA, UV–Visible spectroscopy, FTIR, ^{13}C-NMR and ^{1}H-NMR spectra [[15]]. The prepared metal complexes were also characterised by using other tools like cyclic voltammetry, powder XRD and structure optimization by using Chem 3D software.

The studied literature revealed that the 4-chloroisonitrosoacetophenone cyclohexyl thiosemicarbazone ligand and their metal complexes have not been investigated as photocatalytic agents. The main goal of this current work is to study the photocatalytic degradation by using metal complexes as a heterogeneous catalyst to degrade the methyl orange dye. Due to their non-chemical reactivity, non-toxicity, and ease of recovery using filtration techniques, heterogeneous [[16],[17]] photocatalysts—particularly those utilizing metal ligand complexes, have been extensively employed for efficient photocatalytic dye degradation [[18],[19]].

II. MATERIALS AND METHODS

All the chemicals and reagents utilized in this investigation was of reagent grade and was employed without any additional purification. The protocol standard was followed in the use, purification, and distillation of solvents. Sigma-Aldrich Methyl Orange dye Content 85% of ACS reagent grade were used.

A. Synthesis of (ML_2) complexes

The metal complex (ML_2) was produced using a molar ratio of 1:2 (Metal:Ligand). The appropriate metal(II) chloride salt was added gradually to the ethanolic solution of 4-chloroisonitrosoacetophenone cyclohexyl thiosemicarbazone to form complexes. The reaction mixture was dropwise supplemented with NH_3 to keep the alkaline pH between 7 and 8. The above mentioned reaction was kept at room temperature for 30 minutes while being constantly agitated, and then it was let to stand overnight. Whatmann filter paper no. 1 was used to filter the solid product. The solid compounds were dried and weighed as a metal complex (Fig. 1)

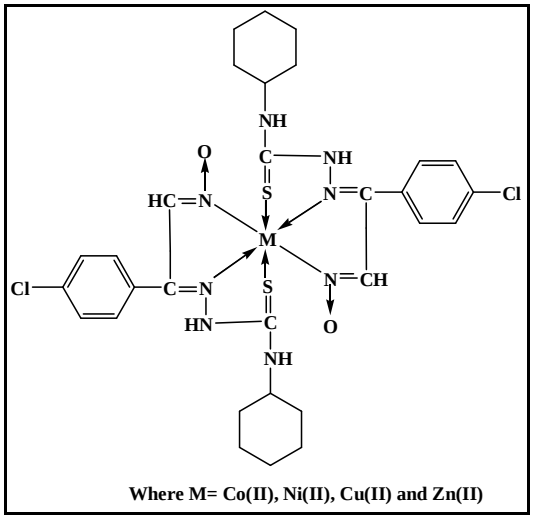

Fig. 1. Structure of 4-chloroisonitrosoacetophenone cyclohexylthiosemicarbazone metal complexes (ML_2)

B. Preparation of Methyl orange dye solution

To make a 100 ppm dye solution, weigh out 100 mg of methyl orange dye and dissolve it in 1000 mL of distilled water. A 10 ppm solution was made from stock by diluting 50 mL of a 100 ppm solution with 500 mL of distilled water. The dye was photocatalytically degraded using the 10 ppm dye sample solution.

C. Photocatalytic activity

The methyl orange dye degradation in the existence of a tungsten filament lamp was used to test the photocatalytic dye degradation efficiency of the complexes. Two conical flasks A and B of 25 cm^3 were taken. Flask A: Methyl orange solution (control) and Flask B: Methyl orange + ML_2 complex (test). The initial absorbance (A_0) of both the solutions obtained at λ_{max} = 480 nm. The photocatalytic degradation was examined using varied catalyst amounts (25, 50, 75, and 100 mg), dye concentrations (10 ppm), and time intervals (30, 60, 90 and 120 min). The absorbance of dye sample before and after exposed to light and with and without a catalyst was determined. Upon irradiation, the residue of the catalyst has been separated by centrifugation, and a UV-visible spectrophotometer set to 480 nm was used to measure the absorbance of the filtrate solution of the methyl orange dye sample. The following formula was used to get the photocatalytic degradation(%) [[20]]:

$$\% \text{ Degradation} = \frac{A_o - A_t}{A_o} \text{ X } 100$$

Where A_o and A_t denotes the absorbance before and after photocatalytic reaction for methyl orange dye

III. RESULTS AND DISCUSSION

A. Photocatalytic activity

A 100 ppm concentration of methyl orange dye was employed to determine the λ_{max}, which was observed at 480 nm [[21]], as shown in Fig. 2. Under light irradiation, the degradation of the organic dye methyl orange was assessed both with and without the catalyst. The catalyst was used to degrade the dye over time in a dark environment. Catalysts did not influence the rate of dye degradation. The light exposure at various catalyst amount and durations led to deterioration. To optimize the degradation experiment, 25-100 mg of catalyst was utilized. Photocatalytic degradation is influenced by catalyst quantity, with more active sites on the surface leading to faster deterioration [[22]].

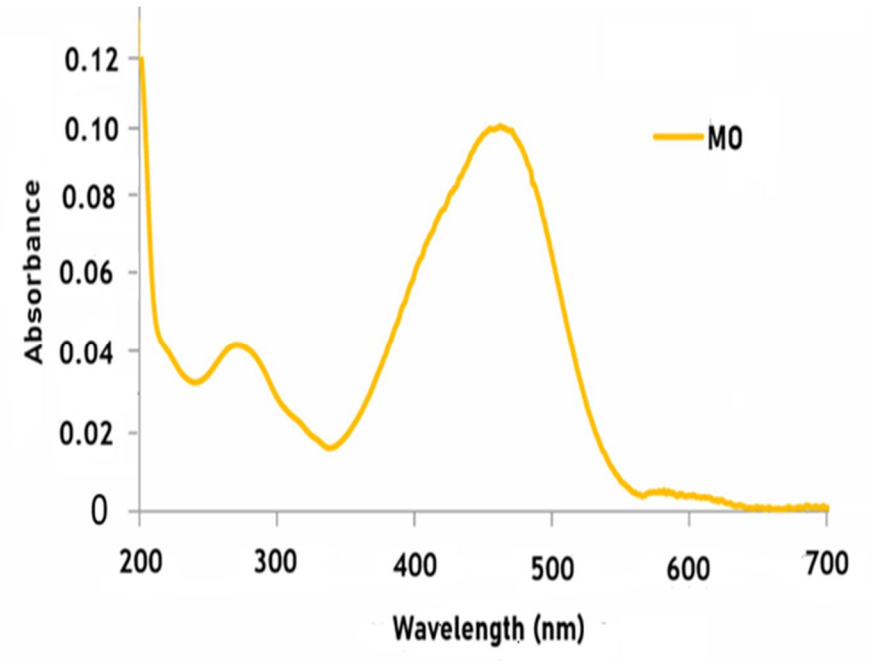

Fig. 2. Wavelength (λ_{max}) of maximum absorbance of methyl orange dye at 480 nm

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B. Determination of Initial absorbance (A_o) of dye

Using a 10 ppm dye solution, the initial absorbance (A_o) was determined. The absorbance at A_o = 0.103 is displayed in Fig. 3.

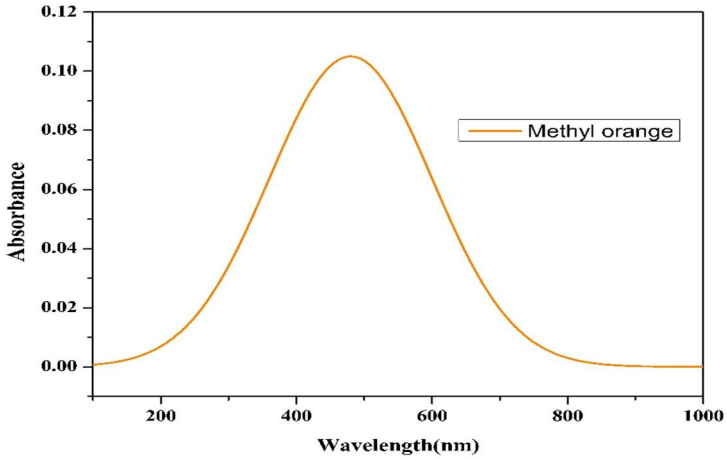

Fig. 3. Initial absorbance (A_o) of methyl orange dye without light irradiation

C. UV-visible spectra of representative CoL_2 complex

The plot of UV-Vis spectra of representative CoL_2 catalyst at variations of time period shown in Fig. 4.

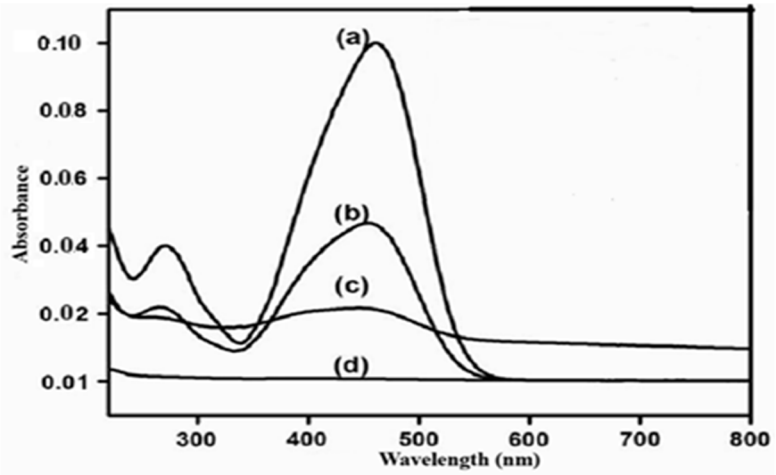

Fig. 4. UV–Vis spectrum of representative CoL_2 catalyst for photocatalytic reduction of MO after (a)30 min, (b)60 min, (c)90 min and (d)120 min

The photocatalytic activity of complexes used as heterogeneous catalyst studied on two parameters: Effect of time on degradation and Effect of catalyst amount on degradation

D. Effect of time on degradation of azo dye

A fix 10 ppm concentration of dye solution (control) and the rection mixture of dye solution and 50 mg of ML_2 complexes were used to measure absorbance at a wavelength of 480 nm for the time intervals of 30, 60, 90 and 120 mins. As the degradation time of a dye increases, absorbance of all the ML_2 complexes decreases [[23]]. TABLE 1 displays the MO photodegradation rate at different time intervals using the slope of the curves in Fig. 5. In this studied parameter, %degradation of ML_2 complexes increases with increasing degradation time. At time 30 min, %degradation found at 6.79, 7.76, 9.70 and 11.65 for CoL_2, NiL_2, CuL_2 and ZnL_2 catalysts respectively; %degradation at the time of 60 min obtained 36.89, 38.83, 39.80 and 41.74. For 90 min deterioration% shown 65.04, 66.99, 67.96 and 68.93; the dye degradation at 120 min obtained 74.75, 77.66, 78.64 and 80.58 of CoL_2, NiL_2, CuL_2 and ZnL_2 catalysts respectively.

TABLE 1
EFFECT OF TIME ON DYE DEGRADATION

Sr. No.	Complex	Time (min)	Absorbance (A_t)	% Degradation
1.	CoL_2	30	0.096	6.79
		60	0.065	36.89
		90	0.036	65.04
		120	0.026	74.75
2.	NiL_2	30	0.095	7.76
		60	0.063	38.83
		90	0.034	66.99
		120	0.023	77.66
3.	CuL_2	30	0.093	9.70
		60	0.062	39.80
		90	0.033	67.96
		120	0.022	78.64
4.	ZnL_2	30	0.091	11.65
		60	0.060	41.74
		90	0.032	68.93
		120	0.020	80.58

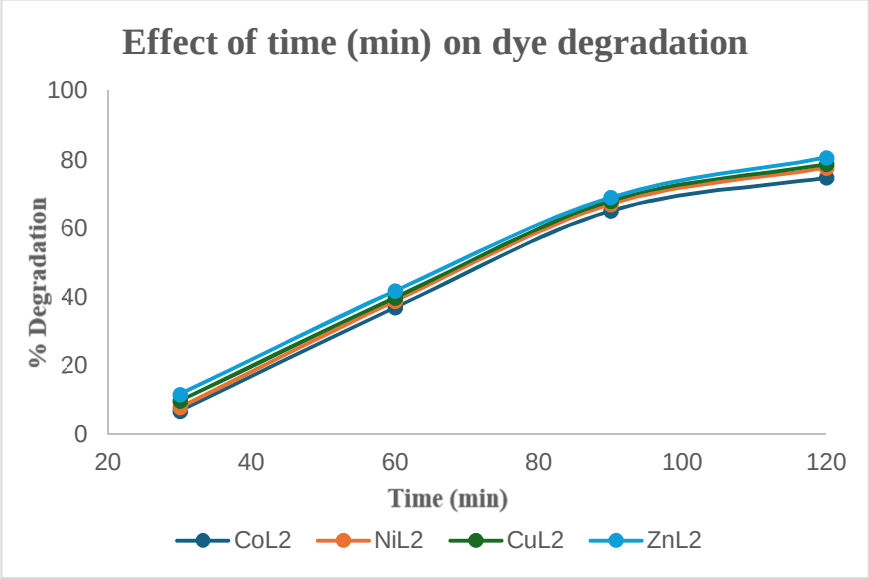

Fig. 5. Effect of time(min) on dye degradation

E. Effect of catalyst amount on degradation of azo dye

The sample was exposed to light for 120 minutes while the amount of catalyst was varied and the dye concentration was fixed at 10 ppm. The absorbance of the dye solution was determined following irradiation. The proportion of dye degradation was determined using the absorbance readings. The amount of catalyst utilized enhanced the rate of degradation [[24]]. TABLE 2 show the effect on MO photodegradation rate by amount of catalyst with the slope of the curves in Fig. 6. In this investigation, it was discovered that the proportion of dye sample degradation raised as catalyst quantity increased. By using 25 mg of the catalyst, %degradation obtained 12.62, 13.59, 14.56 and 16.50 for the catalysts CoL_2, NiL_2, CuL_2 and ZnL_2 respectively. After using 50 mg of catalyst, degradation percentage noted for the catalysts are 63.10, 64.07, 66.01 and 66.99. Used of 75 and 100 mg of catalyst, the observed percentage are 66.99 and 74.75 for CoL_2, 67.96 and 76.69 for NiL_2, 68.93 and 77.66 for CuL_2, 69.90 and 79.61 for ZnL_2 respectively.

TABLE 2
EFFECT OF AMOUNT OF CATALYST ON DYE DEGRADATION

Sr. No.	Complex	Amount of catalyst (mg)	Absorbance (A_t)	% Degradation
1.	CoL_2	25	0.090	12.62
		50	0.036	63.10
		75	0.034	66.99
		100	0.026	74.75
2.	NiL_2	25	0.089	13.59
		50	0.037	64.07
		75	0.033	67.96
		100	0.024	76.69
3.	CuL_2	25	0.088	14.56
		50	0.035	66.01
		75	0.032	68.93
		100	0.023	77.66
4.	ZnL_2	25	0.086	16.50
		50	0.034	66.99
		75	0.031	69.90
		100	0.021	79.61

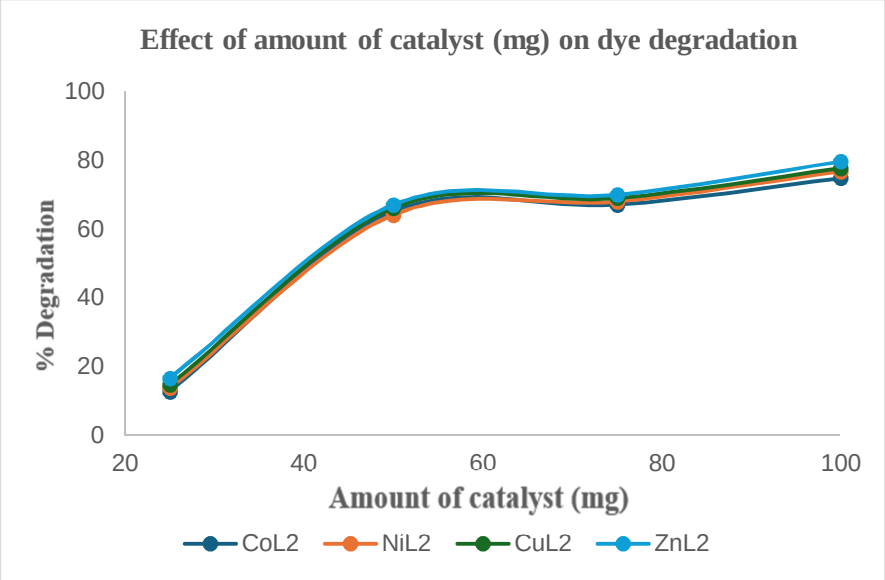

Fig. 6. Effect of amount of catalyst (mg) on dye degradation

F. Decolorization of methyl orange dye

Before being exposed to light, the dye sample in the typical metal complex had a higher colour intensity. However, after being exposed to light and by increasing the time exposure, the dye molecule's degradation is evident in Fig. 7, where its colour intensity decreases [[25]]. This shows that the dye has degraded based on data showing a decreasing solution absorbance value.

Fig. 7. Degradation of colour intensity of the MO dye after exposure time of (a)30 min, (b)60 min, (c)90 min and (d)120 min

G. Reusability of catalyst

Photocatalysts play a crucial role in dye degradation due to their low cost, ease of availability, and unconventional properties. No significant decrease in dye degradation was seen after five continuous cycles. The catalysts used in the dye degradation process were recovered. The loss of photocatalyst during sample handling and centrifugation is linked to the drop in the percentage degradation efficiency [[26]]. Catalytic cycle tests were carried out under certain parameters, including a reaction period of 120 minutes, a dye concentration of 10 ppm, and 50 mg of catalyst, in order to assess the catalyst's sustainability. According to the study, the efficiency of CoL_2, NiL_2, CuL_2 and ZnL_2 complexes progressively dropped with subsequent catalytic cycles, suggesting a decrease in catalytic activity with time. When CoL_2 was used for the first time, it could destroy 66.01% of the dye, with an average decrease in efficiency to 61.16% and NiL_2 efficiency fall from 68.93% to 63.10% after five uses. 72.81% to 65.04% reusability rate have been found in the case of CuL_2 and 76.24% to 69.30% degradation efficacy of the catalyst ZnL_2 was obtained after taken all the cycles (TABLE 3).

TABLE 3
REUSABILITY OF PHOTOCATALYST

Sr. No.	Complex	No. of catalytic cycles	Absorbance (A_t)	% Degradation
1.	CoL_2	1	0.035	66.01
		2	0.037	64.07
		3	0.040	61.16
		4	0.040	61.16
		5	0.040	61.16
2.	NiL_2	1	0.032	68.93
		2	0.035	66.01
		3	0.038	63.10
		4	0.038	63.10
		5	0.038	63.10
3.	CuL_2	1	0.028	72.81
		2	0.033	67.96
		3	0.036	65.04
		4	0.036	65.04
		5	0.036	65.04
4.	ZnL_2	1	0.024	76.24
		2	0.028	72.27
		3	0.031	69.30
		4	0.031	69.30
		5	0.031	69.30

IV. MECHANISM FOR DYE DEGRADATION

A complex increases photocatalytic activity when it is exposed to light because it absorbs photons and promotes charge separation at the interface [[27]]. The interaction between the methyl orange dye molecule and light results in the production of excited dye molecules. Positive dye radicals and negative O_2^- radicals are created when the excited dye molecules react with oxygen [[28]]. Additionally, the dye molecules are destroyed by superoxide radicals (•OOH), which are created when negative radicals combine with H^+ ions released from H_2O. Photogenerated holes promote the adsorption of O_2^- from H_2O by reducing electron recombination and the surface of OH groups. O_2 is reduced to O_2^- species by the photoformed electrons, which can subsequently combine with H_2O to create more oxygenated radicals, mainly hydroxyl (•OH) radicals [[29],[30]]. Both hydroxide and superoxide radicals significantly speed up the degradation of dye molecules, which is why methyl orange dye decolorizes effectively.

V. CONCLUSION

In conclusion, the photocatalytic decolorization of industrial textile dye, Methyl orange, under tungsten filament lamp light was investigated employing CoL_2, NiL_2, CuL_2 and ZnL_2 as photocatalysts. The solution of methyl orange dye absorbs at the wavelength of 480 nm and initial absorbance (A_o) recorded. The 10 ppm MO dye solution (blank) and MO dye solution with catalysts (test) were used to employed the photocatalytic activity. The observed results show the intensity of the dye colour goes on degraded with increasing time interval of the light exposure and as well as increasing the amount of the catalysts. The absorbance of the samples was noted after every 30 min and after every addition of 25 mg of the catalysts. The absorbance goes on decreasing and degradation rate enhances with increasing time duration. The absorbance also falls down and deterioration increases with increasing the amount of catalyst. These findings clearly demonstrated that the synthesized metal complexes act as a very effective catalyst for the study of degradation of azo dye.

VI. ACKNOWLEDGEMENT

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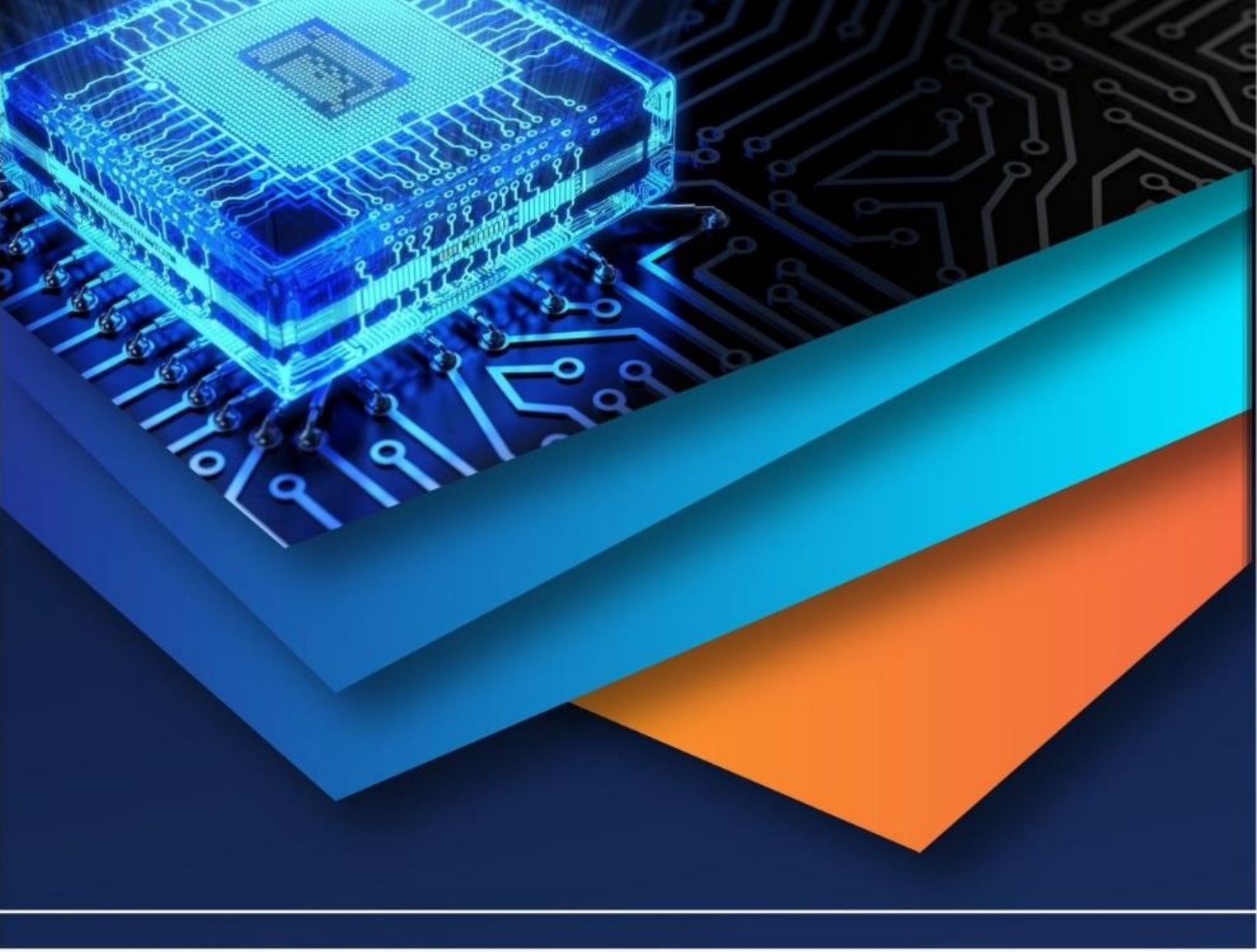

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