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Green Route of Synthesis, Characterization and Antibacterial Activity of Novel Ligand and its Metal Complexes Derived from 2-Amino Benzimidazole and Acenaphthenequinone

Pawar S R[1], Moharir S P[2], Undegaonkar M G[3], Sinkar S N[4], Mirgane S R[5]
[1]*Dept. of chemistry, Shri Siddheshwar Mahavidyalaya, Majalgaon, Dist- Beed*
[2]*Dept. of chemistry, Siddharth Arts Commerce and Science College Jafrabad, Dist. - Jalna*
[3]*Dept. of chemistry, Arts, Science and Commerce College Badnapur, Dist. - Jalna*
[4]*Dept. of chemistry, M.S.S. College Ambad , Dist. -Jalna*
[5]*Dept. of chemistry, JES College Jalna.*

Abstract: ***The microwave assisted green synthesis of novel ligand derived from 2-Amino benzimidazole and Acenaphthenequinone and its metal complexes of Mn(II), Fe(III), Ni(II), Cu(II), Co(II), Zn(II), Cd(II) and Ag(I). The novel ligand was identified by melting point and thin layer chromatography. Characterization of novel ligand was by elemental analysis, Infrared spectra, ^{1}HNMR spectra and mass spectroscopy. The metal complexes were identified by melting point, thin layer chromatography and their distinguishing colour metal complexes were characterized by Infrared spectroscopy, UV-visible spectroscopy and thermo gravimetric analysis. The antibacterial activities of the novel ligand and its metal complexes were tested against Escherichia coli, Staphylococcus aurous and Salmonella Typhi.***

Keywords: Green synthesis, 2-Amino benzimidazole, Acenaphthenequinone, Antibacterial activities.

I. INTRODUCTION

The azomethine group present in Schiff base ligand, which forms highly stable complex with transition metal ions. In the present work we focus on green and efficient synthesis of novel Schiff base ligand and their metal complexes. The main feature of microwave synthesis approach are shorter reaction time, larger the yield and simple conditions for reaction. The few reports are on the synthesis of metal complexes by microwave assisted method [1-3].

The use of microwave assisted irradiation in synthesis of drugs and organic compounds have proved that it is effective, safe and eco-friendly with shorter reaction time [4-5]. Compound containing azomethine/imine (C=N) group are known as ligand [6]. The product of ketone and aldehyde with primary amine are generally known as Schiff base ligand [7]. They are biological very active compounds, having biological activities like antibacterial [8-11], antimicrobial [12], anticancer [13], plant growth inhibitors [14] and so on.

II. EXPERIMENTAL SECTION

All the chemicals used in this work were of analytical grade. 2-Amino benzimidazole and Acenaphthenequinone form Sigma Aldrich and metal nitrates and chlorides from loba chem and MERCK. The novel ligand synthesized in scientific microwave oven. Metal complexes were synthesized by reacting novel ligand with metal salts in scientific microwave oven.

A. Techniques

Synthesis of novel ligand and its metal complexes were performing in microwave extraction system in scientific microwave oven. Melting points were measured on digital melting point apparatus. The electronic absorption spectra were recorded in the wavelength range 200 to 400 nm using UV spectrophotometer. IR spectra were analyses on Shimadzu Dr 8031. The ^{1}HNMR spectra was analyse in DMSO D6 on Brakers 400 MHz instrument. The mass spectrum was recorded by LCMS spectrophotometer. The TGA were carried out in dynamic nitrogen atmosphere (30ml/min) with heating rate of 10^0C/min using Shimadzu TGA 50H thermal analyser. TLC analysis performs on pre coated aluminium plates.

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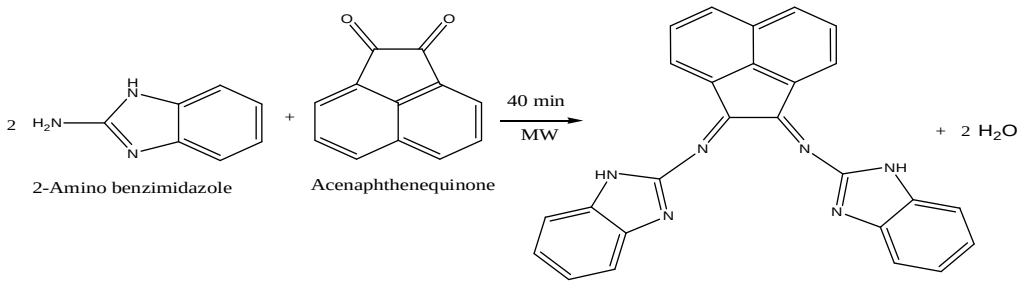
B. Preparation of Novel Ligand

The novel ligand was prepared by the reaction between 2-Amino benzimidazole (1.34 gm.) and Acenaphthenequinone (0.92 gm.) under solvent free condition in scientific microwave oven about 40 min. The irradiated product after cooling at room temperature washed with dry ether. The yield obtained was 2.12 gm. And melting point was 180^0C. The purity of the product confirm by TLC.

Reaction



2-Amino benzimidazole Acenaphthenequinone

(*E*)-*N*-((*E*)-2-(1*H*-benzo[*d*]imidazol-2-ylimino)acenaphthylen-1(2*H*)-ylidene)-1*H*-benzo[*d*]imidazol-2-amine

C. Preparation of metal complexes

The metal complexes were synthesized under solvent free condition by irradiating metal nitrate or metal chloride with the required amount of the ligand. The reaction mixture was irradiated in microwave oven. The products were washed with dry ether, filter and dried at room temperature. The metal salts used were $MnCl_2$, $Fe(NO_3)_3.9H_2O$, $Co(NO_3)_2.6H_2O$, $Ni(NO_3)_2.6H_2O$, $Cu(NO_3)_2.3H_2O$, $Zn(NO_3)_2.6H_2O$, $Cd(NO_3)_2.4H_2O$ and $AgNO_3$.

III. RESULT AND DISCUSSION

All metal complexes and novel ligand are colored, solid and stable at room temperature. They possess sharp melting point. The complexes are insoluble in common organic solvents but soluble in DMF and DMSO.

A. Physical Properties

Physical properties of the novel ligand and metal complexes summarized in Table I

Table I

Sr. No	Molecular formula	Color	Melting point (^{0}c)	Time	Yield %
1	$C_{26}H_{16}N_6$	Yellow	180	40 min	93.80
2	$[C_{26}H_{16}N_6(H_2O)_2]Mn$	Light Yellow	118	360 sec.	95
3	$[C_{26}H_{16}N_6(H_2O)_2]Fe$	Brown	110	30 sec.	75
4	$[C_{26}H_{16}N_6(H_2O)_2]Co$	Purple	132	300 sec.	87
5	$[C_{26}H_{16}N_6(H_2O)_2]Ni$	Greenish	168	150 sec.	96
6	$[C_{26}H_{16}N_6(H_2O)_2]Cu$	Green	154	90 sec.	95
7	$[C_{26}H_{16}N_6(H_2O)_2]Zn$	Brownish Yellow	124	120 sec.	91
8	$[C_{26}H_{16}N_6(H_2O)_2]Cd$	Brownish Yellow	146	180 sec.	87
9	$[C_{26}H_{16}N_6(H_2O)_2]Ag$	Dark Yellow	176	360 sec.	95

B. Elemental Analysis

Elemental analysis (CHN) data of the novel ligand is summarized in Table II

Table II

Comp.	Empirical formula	Molecular Weight	C Found (cal.)	H Found(cal.)	N Found (cal.)
Novel Ligand	$C_{26}H_{16}N_6$	412	75.95 (75.73)	3.96 (3.88)	20.09 (20.39)

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C. Mass Spectral Studies

The mass spectrum study of novel ligand showed a peak at m/z. 413(M+1) that corresponds to the molecular weight of the ligand 412.

D. ^{1}HNMR Spectral Studies

Observed ^{1}HNMR peaks (ppm) of novel Schiff base ligand summarized in Table III.

Table-III

Compound	H-from Aromatic ring in ppm	H-from-NH of Imidazole in ppm
$C_{26}H_{16}N_6$	6.81 - 8.46	6.07

The ^{1}HNMR spectrum of novel ligand shows different peaks. The characteristic peak observed at 6.07 ppm is due to H-from NH-of Imidazole. The peaks observed at 6.81 – 8.46 ppm are due to H-from aromatic rings.

E. Infrared Spectra Analysis

Observed IR frequencies of novel ligand and its metal complexes summarized in Table IV.

Table-IV

Sr. No	Ligand/complex	C=N (cm^{-1})	N-H (cm^{-1})	C=C (cm^{-1})	M-N (cm^{-1})
1	$C_{26}H_{16}N_6$	1630	3350	1450	---
2	$[C_{26}H_{16}N_6(H_2O)_2]Fe$	1647.21	3400	1454.33	547.78
3	$[C_{26}H_{16}N_6(H_2O)_2]Cu$	1643.35	3360	1492.90	677.01

The IR spectrum of novel ligand show characteristics band at 1630 cm^{-1} which indicates (C=N) stretching vibration of azomethine group [15-20]. The vibrational band at 3350 cm^{-1} assigned N-H stretching in the ligand. Band observed at 1450 cm^{-1} corresponds to C=C stretching.

IR spectral study of Fe metal complex: The band appeared at 1647.21 cm^{-1} corresponds to azomethine (C=N) stretching, whereas same azomethine band is observed at 1630 cm^{-1} in spectrum of ligand. Which indicate coordination of ligand with metal ion [21]. The band observed at 3400.00 cm^{-1} assign to (N-H) stretching, whereas in spectrum of ligand it is observed at 3350 cm^{-1}. The vibration observed at 1454.33 cm^{-1} due to aromatic (C=C) stretching. The characteristics band appeared at 547.78 cm^{-1} assign to (M-N) vibration, which confirms coordination of azomethine and metal ion [22-23]. The weak bands observed at 893.04 cm^{-1} and 1041.56 cm^{-1} were due to OH wagging mode of vibration, indicating coordination of water molecule in metal complex [24-27]. Above bands which are appeared in spectrum of complex are not appeared in spectrum of ligand that confirm the formation of metal complex with stable metal ligand bonding.

IR spectral study of Cu metal complex: A stretching observed at 1643.35 cm^{-1}, which corresponds to azomethine (C=N) stretching vibrations, whereas same stretching is observed at 1630 cm^{-1} in spectrum of ligand. The band observed at 3360.00 cm^{-1} assign to (N-H) stretching, whereas in spectrum of ligand it is observed at 3350 cm^{-1}. The vibration observed at 1492.90 cm^{-1} due to aromatic (C=C) stretching.

The coordination of metal to nitrogen was justified by stretching observed at 677.01 cm^{-1} [28]. The weak bands observed at 893.04 cm^{-1} and 1043.49 cm^{-1} were due to OH wagging mode of vibration, indicating coordination of water molecule in metal complex [24-27]. Above bands which are appeared in spectrum of complex are not appeared in spectrum of ligand that confirm the formation of metal complex with stable metal ligand bonding.

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F. Electronic Spectra

UV-Vis spectral data and probable geometry for the metal complexes summarized in Table V

Table-V

Sr. No.	Complex	UV-visible major bands. Absorption Maxima cm^{-1}(nm)	Assignment	Proposed geometry
1	$[C_{26}H_{16}N_6(H_2O)_2]Fe$	43706.29 (228.80)	$^6A_{1g}(S) \rightarrow {}^4T_{1g}(G)$	Octahedral
		49019.60 (204.00)	$^6A_{1g}(S) \rightarrow {}^4T_{2g}(G)$	
		49455.98 (202.20)	$^6A_{1g}(S) \rightarrow {}^4E_g, {}^4A_{1g}$	
2	$[C_{26}H_{16}N_6(H_2O)_2]Cu$	44682.75 (223.80)	$^2B_{1g} \rightarrow {}^2A_{1g}$	Octahedral
		45289.85 (220.80)	$^2B_{1g} \rightarrow {}^2B_{2g}$	
		47619.04 (210.00)	$^2B_{1g} \rightarrow {}^2E_g$	

UV-Vis spectrum of both metal complexes Fe(III), Cu(II) recorded in the wavelength region 200nm to 400nm in DMSO solution.

UV-Vis spectral data of Fe: Electronic spectrum of Fe(III) complex shows absorption maxima at 43706.29 (228.80), 49019.60 (204.00) and 49455.98 (202.20) assign to $^6A_{1g}(S) \rightarrow {}^4T_{1g}(G)$, $^6A_{1g}(S) \rightarrow {}^4T_{2g}(G)$ and $^6A_{1g}(S) \rightarrow {}^4E_g, {}^4A_{1g}$ transitions respectively indicating that complex possess octahedral geometry[29-30].

UV-Vis spectral data of Cu: Electronic spectrum of Cu(II) complex shows absorption maxima at 44682.75 (223.80), 45289.85 (220.80) and 47619.04 (210.00) assign to $^2B_{1g} \rightarrow {}^2A_{1g}$, $^2B_{1g} \rightarrow {}^2B_{2g}$ and $^2B_{1g} \rightarrow {}^2E_g$ transitions indicating that complex possess octahedral geometry[31-32].

G. Thermo Gravimetric Analysis of Metal Complexes

Thermo gravimetric analytical data of metal complexes were summarized in Table VI.

Table-VI

$[C_{26}H_{16}N_6(H_2O)_2]Fe$		$[C_{26}H_{16}N_6(H_2O)_2]Cu$	
Weight loss %	Temperature ^0C	Weight loss %	Temperature ^0C
0	29.76	0	31.79
10	179.04	10	172.61
20	287.10	20	214.19
30	431.47	30	295.40
----	----	40	372.32
-----	----	50	423.73
-----	-----	60	432.89
-----	----	70	449.31
------	----	80	485.49
33.231% total wt. loss	500	82.323% total wt. loss	500

The TGA curve of Fe(III) was carried out in the temperature range from 28.55^0C to 500^0C. The heating was carried out in the nitrogen atmosphere, with heating rate $10^0C\ min^{-1}$.

In the range of 29.76^0C to 179.04^0C water of crystallization lost with 10% weight loss is observed. Then loss up to organic moiety total weight loss of 33.231% at 500^0C. Stable curve indicates formation of metal oxide.

The TGA curve of Cu(II) was carried out in the temperature range from 30.58^0C to 500^0C. The heating was carried out in the nitrogen atmosphere, with heating rate $10^0C\ min^{-1}$. The thermogram of Cu(II) shows total weight loss of 82.323%. The water of crystallization was loses in the range of 31.79^0C to 172.61^0C. The loss of organic moiety with total weight loss at 500^0C was 82.323%. A stable curve shows the formation of metal oxide of copper.

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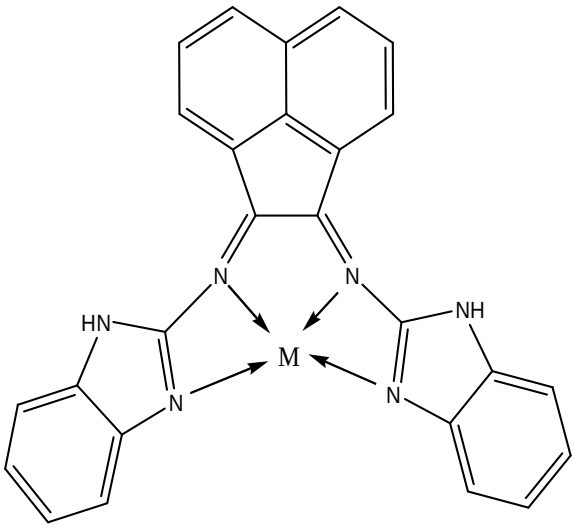

Proposed structure of metal complex (M)=Mn(II),Fe(III),Co(II),Ni(II),Cu(II),Zn(II),Cd(II),Ag(I).

H. Bioactivity study

Antibacterial activity of novel Schiff base ligand and its metal complexes were summarized in Table VII.

Table-VII

Sr. No.	Compound	Minimum inhabitation concentration (ug/ml)		
		E. Coli	S. Aureus	S. Typhi
1	$C_{26}H_{16}N_6$	250	125	250
2	$[C_{26}H_{16}N_6(H_2O)_2]Mn$	62.5	125	200
3	$[C_{26}H_{16}N_6(H_2O)_2]Fe$	100	250	100
4	$[C_{26}H_{16}N_6(H_2O)_2]Co$	100	100	250
5	$[C_{26}H_{16}N_6(H_2O)_2]Ni$	500	250	500
6	$[C_{26}H_{16}N_6(H_2O)_2]Cu$	100	125	250
7	$[C_{26}H_{16}N_6(H_2O)_2]Zn$	500	500	100
8	$[C_{26}H_{16}N_6(H_2O)_2]Cd$	62.5	500	250
9	$[C_{26}H_{16}N_6(H_2O)_2]Ag$	125	250	100

Antibacterial activity of synthesized novel ligand and its metal complexes were performing against Escherichia Coli, Staphylococcus Aureus and Salmonella Typhi. Which were grown overnight at 37^0C temperature. The minimum inhibitory concentration (MIC) was evaluated against test bacteria. Concentration ranging is in between 0.4 ug/ml to10 ug/ml.

The comparative antibacterial study of novel ligand and its metal complexes show that the MIC value of Mn(II) and Cd(II) shows good antibacterial activity on E.Coli bacteria as compared to rest of metal complexes and parent ligand. The MIC value of Co(II) complex shows good antibacterial activity on S.aureus bacteria as compared to rest of metal complexes and parent ligand. The MIC value of Fe(III), Zn(II) and Ag(I) shows good antibacterial activity on S.Typhi bacteria as compared to rest of metal complexes and parent ligand. It get result as that in some quantity novel Schiff base ligand and its metal complexes are greatly useful against E. Coli, S. Aureus and S. Typhi.

IV. CONCLUSION

The microwave method assures the principle of green chemistry. The novel ligand was synthesized from 2-Amino benzimidazole and Acenaphthenequinone. It forms stable complexes with transition metal ions such as Mn(II), Fe(III), Ni(II), Cu(II), Co(II), Zn(II), Cd(II) and Ag(I). The novel ligand and its eight metal complexes show good antibacterial activity.

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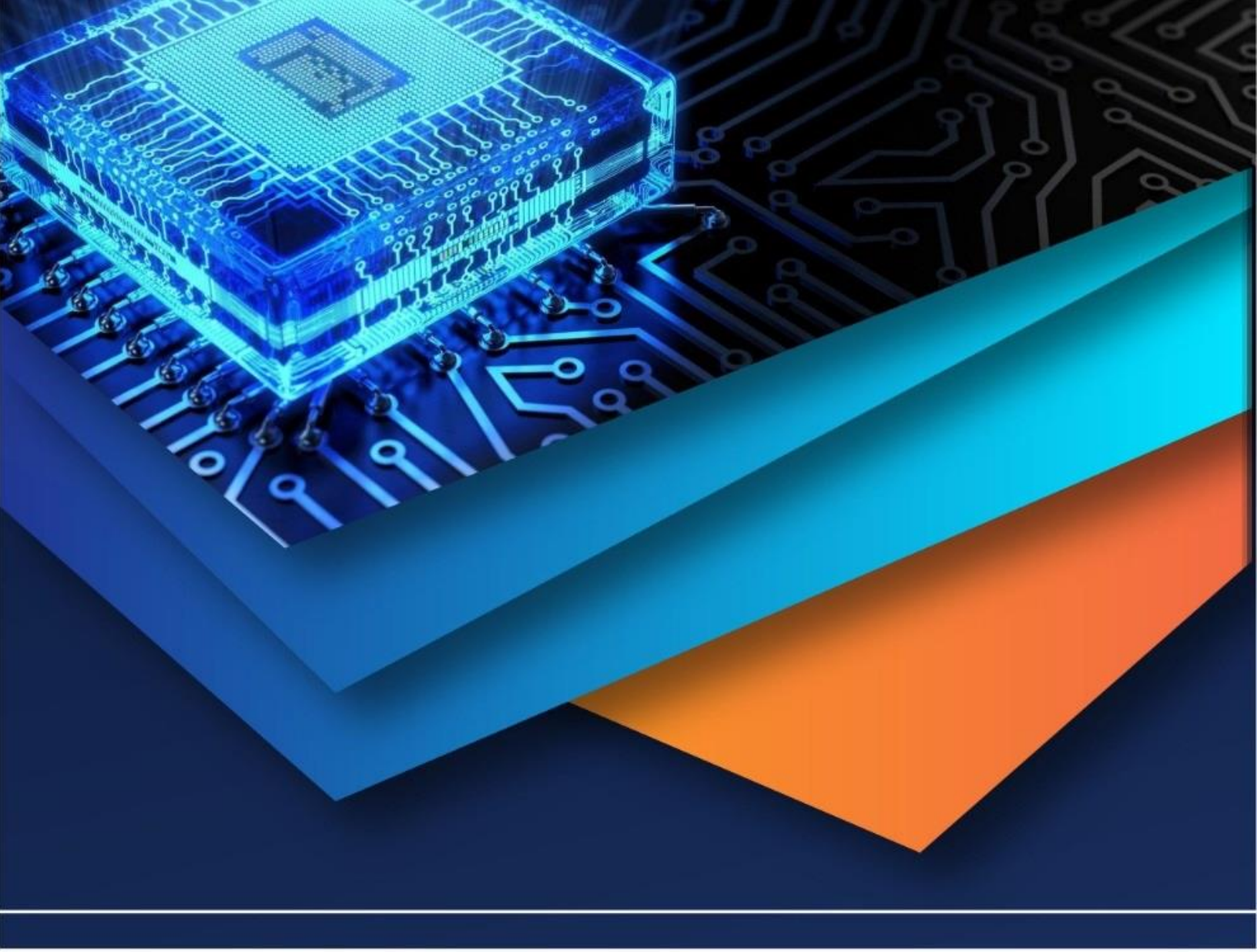

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