iJRASET
International Journal For Research in Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 9 **Issue:** III **Month of publication:** March 2021

DOI: https://doi.org/10.22214/ijraset.2021.33523

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International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.429
Volume 9 Issue III Mar 2021- Available at www.ijraset.com

Microwave Assisted Green Synthesis, Characterization and Antibacterial Activity of Novel Schiff Base Ligand and its Metal Complexes Derived from 2-hydrazino benzothiazole and Isophthalaldehyde

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Abstract: ***The present work microwave assisted green synthesis of novel Schiff base ligand derived from 2-hydrazino benzothiazole and Isophthalaldehyde and its metal complexes of Mn(II), Fe(III), Ni(II), Cu(II), Co(II), Zn(II), Cd(II) and Ag(I). The novel Schiff base ligand was identified by melting point and thin layer chromatography. Characterization 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 thermogravimetric analysis. The antibacterial activities of the Schiff base ligand and its metal complexes were tested against Escherichia coli, Staphylococcus aurous and Salmonella Typhi.***
Keywords: Green synthesis, 2-hydrazino benzothiazole, Isophthalaldehyde, Schiff base ligand, antibacterial activity.

I. INTRODUCTION

Microwave assisted green synthesis is one of the modern synthetic methodology for Schiff base ligand and its metal complexes. This methodology was time-saving, solvent-free, simple reaction condition and larger yield [1-8]. Schiff base ligand containing an azomethine group was formed by reaction of carbonyl compound with primary amine [9-11]. Schiff base ligand plays an important role in inorganic chemistry as they form stable compound with transition metal complexes [12]. Schiff base ligand and their metal complexes have many applications in biological and analytical fields such as anticancer, plant growth inhibitors, insecticidal, antidepressant, antibacterial, anti-inflammatory, anti-tuberculosis, antimicrobial [13-21].

II. EXPERIMENTAL SECTION

A. Material and Method

All the starting chemicals are of analytical grade. 2-hydrazino benzothiazole and Isophthalaldehyde were purchased from Sigma Aldrich and metal salts from Loba chem and MERCK. The novel Schiff base ligand was synthesized by using scientific microwave oven. Syntheses of metal complexes were performed by reacting Schiff base ligand with metal salts in scientific microwave oven.

B. Techniques

Syntheses were performed in scientific microwave oven. Melting points were measured on digital melting point apparatus. The electronic spectra were recorded in the wavelength range 200 to 800 nm in DMSO using UV spectrophotometer. IR spectra were recorded on Simadzu Dr 8031.

^{1}HNMR spectra were recorded in DMSO D6 on Brakers 400Mhz 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^{0}c/min using Simadzu TGA 50H thermal analyzer. TLC analyses were performed on precoated aluminium plates.

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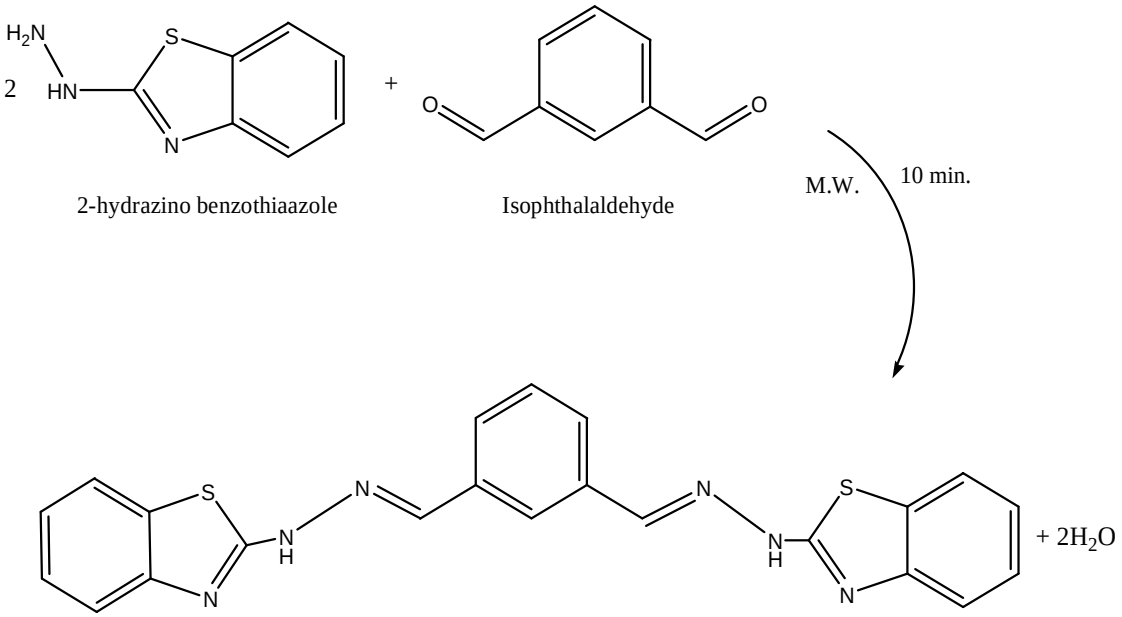
C. Preparation of Novel Ligand

The novel Schiff base ligand was prepared by the reaction between 2- hydrazino benzothiazole and Isophthalaldehyde under solvent free condition in scientific microwave oven for irradiating 10 minutes. The product after cooling at room temperature was washed with dry ether and recrystallized by absolute ethanol. The yield obtained was 1.39 gram and the melting point was 308^0C. The purity of the product was confirmed by TLC.



2-hydrazino benzothiaazole Isophthalaldehyde

2-[(z)-2-({3-[(12)-[2-(1,3-benzothiazol-2-yl)hydrazine-1-ylidene]methyl]phenyl}methylidene)hydrazine-1-yl]-1-3-benzothiazole

D. Preparation of Metal Complexes

The metal complexes were also synthesized under solvent free condition by mixing metal salts with the required amount of the Schiff base ligand. The reaction mixture was irradiated in microwave oven. The products were washed with ether, recrystallized by using absolute ethanol. 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 DISSCUSION

The synthesis of ligand and metal complexes was in two steps. In first 2-hydrazino Benzothiazole was irradiated with Isophthalaldehyde to get ligand. In second step the metal salts were irradiated with ligand to form metal complexes.

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

A. Elemental Composition Analysis

The elemental analysis (CHNS) data for this ligand is summarized in Table I

Table I

Compound	Empirical formula	Molecular Weight	C Found (Cal)	H Found (Cal)	N Found (Cal)	S Found (Cal)
Novel Ligand	$C_{22}H_{16}N_6S_2$	428	59.84 (61.68)	4.06 (3.74)	20.18 (19.63)	15.92 (14.95)

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B. Physical Properties

The detail physical properties of the novel ligand and its metal complexes summarized in Table II

Table II

Sr. No	Molecular Formula	Color	Melting Point (^{0}c)	Time	Yield %
1	$C_{22}H_{16}N_6S_2$	Dark brown	308	10 min.	79
2	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Mn$	Light grey	328	2 min. 30 sec.	95
3	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Fe$	Dark green	72	30sec.	68
4	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Co$	Dark brown	160	60sec.	95
5	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Ni$	Brown	99	40sec.	93
6	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Cu$	Dark green	126	10sec.	95
7	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Zn$	Grey brownish	214	40sec.	76
8	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Cd$	Dark grey	205	90sec.	98
9	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Ag$	Brown	138	3 min. 30sec.	90

C. Infrared Spectra Analysis

Table III

Sr. No	Ligand/Complex	Azomethine C=N (cm^{-1})	Aromatic C-H (cm^{-1})	N-H (cm^{-1})	Aromatic C=C (cm^{-1})	M-N (cm^{-1})	M-S (cm^{-1})
1	$C_{22}H_{16}N_6S_2$	1710	2950	3320	1575	---	---
2	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]$ Mn	1695.43	2940	3400	1554.63	468.70	414.70
3	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]$ Ni	1681.93	2900	3410	1580	455.20	424.34

The IR spectrum of novel Schiff base ligand L_4 show the characteristics band at 1710 cm^{-1} which assigned to (C=N) stretching vibration, which indicates the presence of azomethine group in the ligand [22-24]. The spectrum show vibrational band at 3320 cm^{-1} indicates (N-H) stretching in the ligand. The stretching at 1575 cm^{-1} corresponds to aromatic (C=C) bonding in the ligand. The band observed at 2950 cm^{-1} indicates aromatic (C-H) stretching.

Analysis of ligand-Mn complex: The band observed at 1695.43 cm^{-1} attributed to the stretching vibration of azomethine (C=N) group, whereas in spectrum of ligand same band is observed at 1710 cm^{-1} [25-26]. The bands observed at 2940 cm^{-1}, 3400 cm^{-1} and 1554.63 cm^{-1} were assign to aromatic (C-H) stretching, (N-H) stretching vibration and aromatic (C=C) stretching respectively, whereas same bands are observed at 2950 cm^{-1}, 3320 cm^{-1} and 1575 cm^{-1} in spectrum of ligand respectively. The most characteristics bands appeared at 468.70 cm^{-1} and 414.70 cm^{-1} was due to (M-N) and (M-S) stretching respectively, which confirms the formation of metal ligand bonding [27]. The weak bands observed at 1045.42 cm^{-1} and 885.33 cm^{-1} were due to OH wagging mode of vibration, indicating coordination of water molecule in metal complex [28-31]. 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 spectrum of ligand-Ni complex: The characteristics band appeared at 1681.93 cm^{-1} assign to azomethine (C=N) stretching in the complex molecule, whereas same azomethine stretching is observed at 1710 cm^{-1} in spectrum of ligand. The bands appeared at 2900 cm^{-1}, 3410 cm^{-1} and 1580 cm^{-1} assign to aromatic (C-H) stretching, (N-H) stretching and aromatic (C=C) stretching vibration in the complex, whereas same stretching's are observed at 2950 cm^{-1}, 3320 cm^{-1} and 1575 cm^{-1} in spectrum of ligand respectively. The characteristics band appeared at 455.20 cm^{-1} was indicate metal nitrogen bonding [32-33] and band appeared at 424.34 cm^{-1} was justified metal sulphur bonding. These bands confirm stable metal ligand bonding in the complex molecule. The weak bands observed at 929.69 cm^{-1} and 883.40 cm^{-1} were due to OH wagging mode of vibration, indicating coordination of water molecule in metal complex [28-31]. Above characteristics bands which are appeared in spectrum of complex are not appeared in spectrum of ligand that confirms the formation of metal complex with stable metal ligand bonding.

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D. ^{1}HNMR Spectral Studies

Table IV

Compound	H-from Azomethine In ppm	H-from Aromatic ring In ppm	H-from-NH of Hydrazine In ppm
$C_{22}H_{16}N_6S_2$	9.00	6.96 - 8.25	5.01

The ^{1}HNMR spectrum of novel ligand shows different peaks. The characteristic peak observed at 5.01 ppm is due to H-from NH-of hydrazine. The peaks observed at 6.96 – 8.25 ppm are due to H-from aromatic rings. The peak observed at 9.00 ppm is due to H-from azomethine.

E. Mass Spectral Studies

The mass spectrum study of novel Schiff base ligand show a peak at m/z 428.4, which was corresponds to molecular weight of the novel Schiff base ligand i.e. 428.

F. Electronic Spectra

Table V

Sr. No	Complex	UV-visible Major Bands. Absorption Maxima cm^{-1} (nm)	Assignment	Proposed Geometry
1	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Mn$	38197.17 (261.80)	$^6A_{1g} \rightarrow {}^4T_{1g}$	Octahedral
		43103.44 (232.00)	$^6A_{1g} \rightarrow {}^4E_g(G)$	
		44091.71 (226.80)	$^6A_{1g} \rightarrow {}^4T_{2g}(G)$	
2	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Ni$	39215.68 (255.00)	$^3A_{2g} \rightarrow {}^3T_{2g}(F)$	Octahedral
		46598.32 (214.60)	$^3A_{2g} \rightarrow {}^3T_{1g}(F)$	
		47036.68 (212.60)	$^3A_{2g} \rightarrow {}^3T_{1g}(P)$	

The electronic spectrum of the metal complexes shows absorption bands, these transition may be attributed to the charge transfer band which proves the coordination of the ligand to the metal ion [34].

Electronic spectral data of Mn: Electronic spectrum of Mn(II) complex shows absorption maxima at 38197.17 (261.80), 38197.17 (261.80) and 43103.44 (232.00) assign to $^6A_{1g} \rightarrow {}^4A_{1g}$, $^6A_{1g} \rightarrow {}^4E_g(G)$ and $^6A_{1g} \rightarrow {}^4T_{2g}(G)$ transitions respectively indicating that complex possess octahedral geometry [35-36] .

Electronic spectral data of Ni: Electronic spectrum of Ni(II) complex shows absorption maxima at 39215.68 (255.00), 46598.32 (214.60) and 47036.68 (212.60) assign to $^3A_{2g} \rightarrow {}^3T_{2g}(F)$, $^3A_{2g} \rightarrow {}^3T_{1g}(F)$ and $^3A_{2g} \rightarrow {}^3T_{1g}(P)$ transitions respectively indicating that complex possess octahedral geometry [37-38].

G. Thermo Gravimetric Analysis of Metal Complexes

Table VI

$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Mn$		$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Ni$	
Weight Loss %	Temperature ^{0}C	Weight Loss %	Temperature ^{0}C
0	30.24	0	31.39
10	201.47	10	184.99
20	256.34	20	202.60
30	299.29	30	212.84
40	321.80	40	220.92
50	441.78	50	241.75
-----	--------	60	321.65
-----	--------	70	374.41
52.74% (Total Wt. Loss)	450	73.28% (Total Wt. Loss)	490

The TGA of metal complexes were carried out in the temperature range from room temperature up to 500^0C. The heating is carried out in the dynamic nitrogen atmosphere. Heating rate was controlled at $10^0 Cmin^{-1}$.

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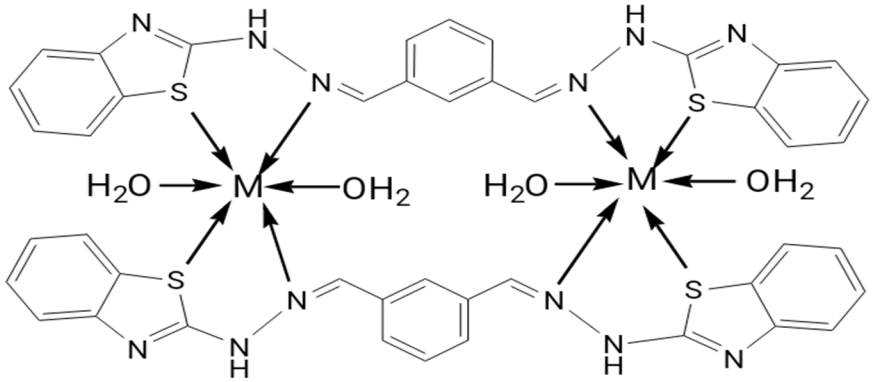
The ligand-Mn complex thermogram clearly shows, Total weight loss of 52.74%. In first step water of crystallization got removed in the range of 30.24^{0}c to 201.47^{0}C with 10% weight loss observed. After this weight loss of water of crystallization, loss of organic moiety took place with total weight loss of 52.74% up to 450^{0}C. A stable curve indicates formation of stable metal oxide.
The ligand-Ni complex thermogram clearly shows, Total weight loss of 73.28%. In first step water of crystallization got removed in the range of 31.41^{0}C to 184.99^{0}C with 10% weight loss observed. After this weight loss of water of crystallization, loss of organic moiety took place with total weight loss of 73.28% up to 490^{0}C. A stable curve indicates formation of stable metal oxide.



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

Table VII

Sr. No.	Compound	Minimum Inhabitation Concentration (ug/ml)		
		E. Coli	S. Aureus	S. Typhi
1	$C_{22}H_{16}N_6S_2$	500	125	250
2	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Mn$	250	125	250
3	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Fe$	500	125	250
4	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Co$	500	250	500
5	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Ni$	100	125	250
6	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Cu$	500	500	100
7	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Zn$	62.5	500	250
8	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Cd$	125	250	100
9	$[(C_{22}H_{16}N_6S_2)_2(H_2O)_2]Ag$	125	250	50

Antibacterial activity of synthesized novel Schiff base ligand L_4 and its metal complexes were screened. The cultures of Escherichia coli, Staphylococcus aureus and Salmonella typhi were grown overnight at 37^{0}C temperature, minimum inhibitory concentration (MIC) were evaluated against test bacteria with concentration ranging between 0.4ug/ml to 100ug/ml. The comparative antibacterial study of L_4 and its metal complexes show that the MIC value of Zn(II) shows excellent and Ni(II) shows good antibacterial activity on E.coli bacteria as compared to other complexes and parent Schiff base ligand. The MIC value of Mn(II), Fe(III), Ni(II) and L_4 shows good antibacterial activity against S. aureus bacteria as compared to other metal complexes. The MIC value of Ag(II) shows excellent and Cu(II), Cd(II) shows good antibacterial activity on S.Typhi bacteria as compared to other complexes and parent Schiff base ligand. It concludes that in some quantity novel Schiff base ligand L_4 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 Schiff base ligand was synthesized from 2-hydrazino benzothiazole and Isophthalaldehyde. It act as a tetra dentate ligand and forms stable binuclear 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 Schiff base ligand and its eight metal complexes show good antibacterial activity.

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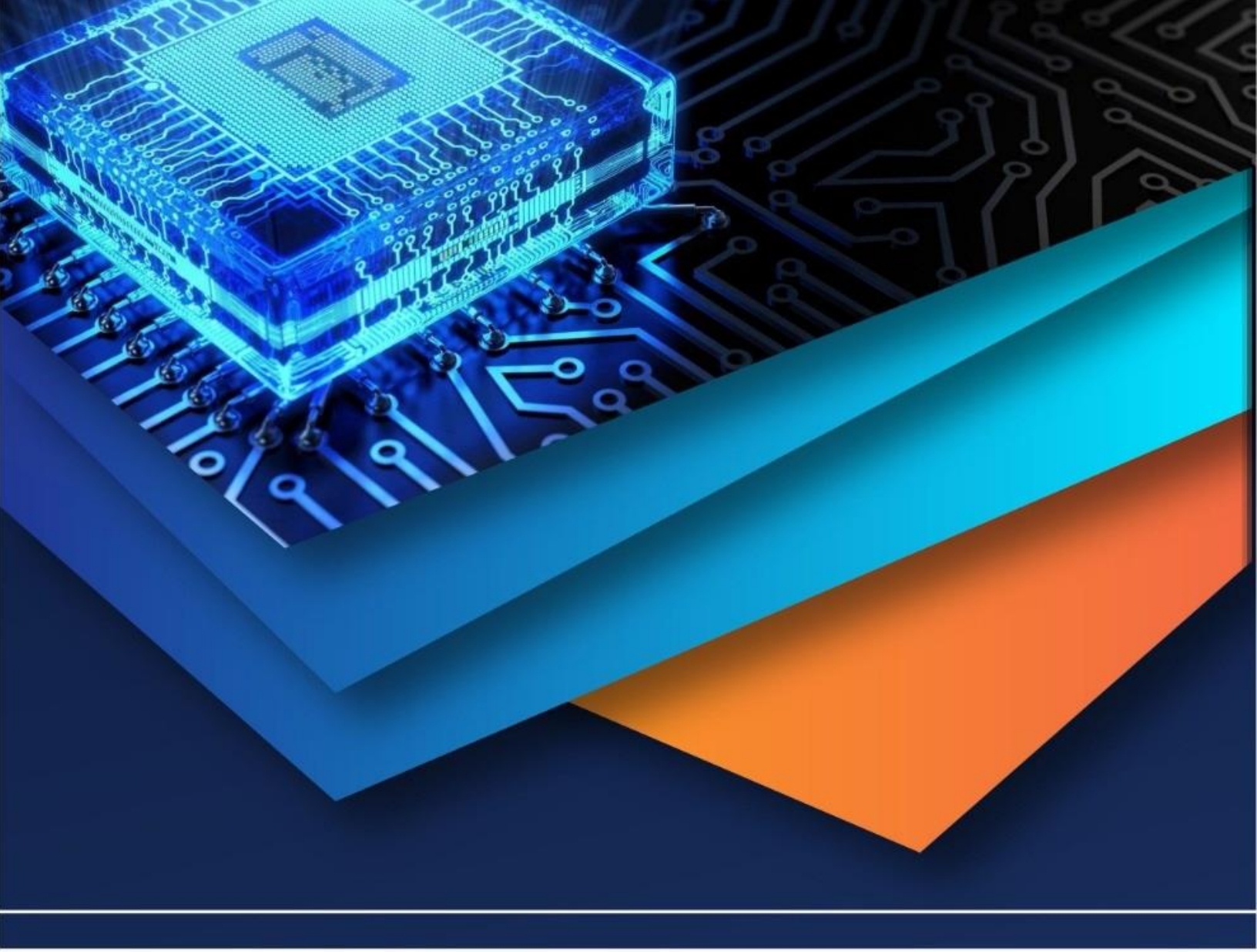

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