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Synthesis, Spectral Study, Characterization and Antimicrobial Activity of Zinc (II) Complex of Chalcone of Pyridine-2-Carbaldehyde

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Abstract: ***Metal complex of Zn(II) has been synthesized with newly prepared biologically active ligand. This ligand was prepared by the Claisen-Schmidt condensation method of 2,6-dihydroxy acetophenone and pyridine-2-Carbaldehyde. The structure of the complex has been proposed by the analytical data, conductivity measurement, magnetic moment, IR spectrum, Electronic absorption spectrum, thermal studies and XRD analysis. Analytical data confirmed 1:2 stoichiometry and the electronic spectral data, IR, magnetic moment, TG-DTA suggests that Zn(II) complex has square planar geometry. Absence of coordinated water molecules in Zn(II) complex is confirmed by TG-DTA studies. The conductivity data show that the complex is non electrolyte. Antimicrobial activities of complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standards. In this paper we prepare chalcone of pyridine-2-Carbaldehyde by Claisen-Schmidt condensation method and synthesize Zn(II) metal complex and characterize them by Infrared, Electronic absorption spectra, magnetic susceptibility, CHO analysis, solution conductivity, XRD study, TG-DTA and antimicrobial activity.***
Keywords: Electronic absorption spectrum, Infrared spectrum, TG-DTA, Elemental analysis, Antimicrobial activities, Physico-chemical property, Magnetic susceptibility and Conductivity.

I. INTRODUCTION

Chalcones constitute an important group of natural products, chemically they consist of open chain flavonoids in which the two aromatic rings are joined by α, β unsaturated carbonyl system. The name chalcone is given by Kostanecki and Tambar [1]. Many complexes of chalcones are synthesized and studied in the literature [2-3]. It is believed that the (>CO–C=C<), moiety imparts biological characteristics to this class of compounds. Such *α,* β-unsaturated carbonyl compounds and their metal complexes possess interesting biochemical properties, such as antitumour, antioxidant, anti-fungal and antimicrobial activities [4]. The electronic absorption spectrum, infrared spectrum, magnetic moment, TG-DTA supports the square planar geometry of the metal complex of chalcone. All crystals of a substance possess the same elements of symmetry. The computer program, used for indexing data was powder-X. Furthermore, biological activities of complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standard [5].The X-ray powder diffractogram of the metal complexes was used for the structural characterization and determination of lattice dimensions.

II. MATERIALS AND METHODS

A. Synthesis of chalcone of Pyridine-2-Carbaldehyde

The reagents used for preparation of chalcone of pyridine-2-carbaldehyde are of A.R. grade. 2,6-dihydroxy acetophenone (0.01mol) and pyridine-2-carbaldehyde (0.01mol) are dissolved in ethyl alcohol (25 mL) and then potassium hydroxide 10 mL (40%) were added to it. The reaction mixture was heated for 3 hours till yellow-brown color was obtained. The progress of reaction was monitored by TLC, after completion of reaction the content was poured into ice cold water and then acidified by dil. HCl the solid obtained was filtered and the crude product was recrystallized from ethyl alcohol to give pyridine chalcone[6].

B. Synthesis of metal complex

The solution of 0.02 mole of chalcone of pyridine-2-carbaldehyde was taken in round bottom flask containing 30 ml of anhydrous methanolic solution and boiled for 10 minutes. A hot solution of 0.01 mole, of Zinc Nitrate in 20 ml of methanol was added drop wise to the solution of the chalcone of pyridine-2-carbaldehyde To this reaction mixture, 10% alcoholic ammonia was added up to slightly alkaline pH. The complex was precipitated at 9 pH range. The pH 8-10 range was definite for these complexes [7]. The

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contents were stirred on magnetic stirrer for one hour. The solid metal complex separated out and washed with methanol three to four times. Dried in vacuum desiccators over anhydrous granular calcium chloride. The melting point/decomposition a temperature of the complex was determined by Thiele's melting apparatus. The reactions of formation of Zn(II) complex is shown in figure (1).

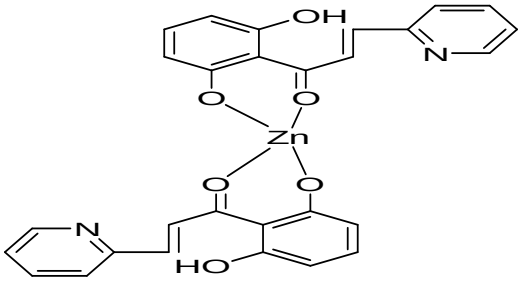

Fig.(1): Metal complexes of Zinc (II) with chalcone of pyridine-2-carbaldehyde

III. RESULTS AND DISCUSSION

A. Infra Red Spectrum

1) *Infrared spectrum of Zn(II) complex:* The infrared spectra of ligands and Zn(II) complex of chalcone of pyridine-2-carbaldehyde was recorded on a Perkin-Elmer Spectrum RX-IFTIR Spectrophotometer over the range 4000-400 cm^{-1} using KBr pellet at CIL, Chandigarh, Punjab.

Seema Habib assigned [8] the ligand which shows a weak broad band around 3047-3029 cm^{-1}. In Zn complexes do not show any absorbance for –OH of the coordinated water molecule. In the IR spectra of all ligands, an intense band appearing around 1656 cm^{-1} is attributed to C=O group. The medium intensity band appearing around 1530 cm^{-1} in the ligand and the complexes are assigned to C=O aromatic. The M-O band for Zn(II) was observed at 500 cm^{-1}.

Chiara Sulpizio assigned that, [9] Zn^{+2} do not show any significant shift compared to the free ligand. While the Zn^{+2} complexes show a shift of C=0 stretching mode from 1630 to 1610 cm^{-1} indicating coordination of carbonyl oxygen to a metal ion.

In the chalcone of pyridine-2-carbaldehyde a sharp strong band observed at 1597 cm^{-1} is attributed to (C=C) stretching mode in the IR spectra of ligand. This band observed at 1437 cm^{-1} in Zn(II) complexes. The slight shift in the band of (C=C) stretching frequency is due to change in electron distribution across (C=C) bond in the metal complexes[10].

The presence of phenolic –OH is confirmed by peaks at 3055 cm^{-1} in the ligand, In the spectra of Zn(II)) complexes, there is the complete disappearance of the peak at 3055 cm^{-1} in chalcone which suggests absence of phenolic group –OH indicates its coordination. (C-O-C) is shifted to a lower wave number compared with a free ligand. In the IR spectra of ligand, the strong bands appeared in the region 1623 & 1666 cm^{-1} are assigned to υ C=O) of stretching frequency [11]. It is shifted towards lower frequency than corresponding ligands and appeared at 1599 cm^{-1} in metal complexes. Such a lowering in stretching vibration of υ (C=O) in chalcone indicates the participation of chalcone carbonyl in complexation. In the IR spectra of Zn(II) complexes, In Zn(II) complexes new band is observed at 507 cm^{-1} due to the (M-O) bond.

Table no. (1): Infrared absorption frequencies (cm^{-1}) of chalcone of pyridine-2-carbaldehyde Zn(II) complex.

Ligand/ Metal complexes	υ(OH) cm^{-1}	υ (H_2O) cm^{-1}	υ ((-CO-CH=CH-))cm^{-1}	υ ((C-O-C) cm^{-1}	υ (C=C) cm^{-1}	Aromatic ring (C=C)cm^{-1}	υ(M-O)cm^{-1}
chalcone of pyridine-2-carbaldehyde	3055		1666	1099	1597	1435	--
[Zn (chalcone of pyridine-2carbaldehyde)$_2$]	3068	-	1599	1052	1437	1369	507

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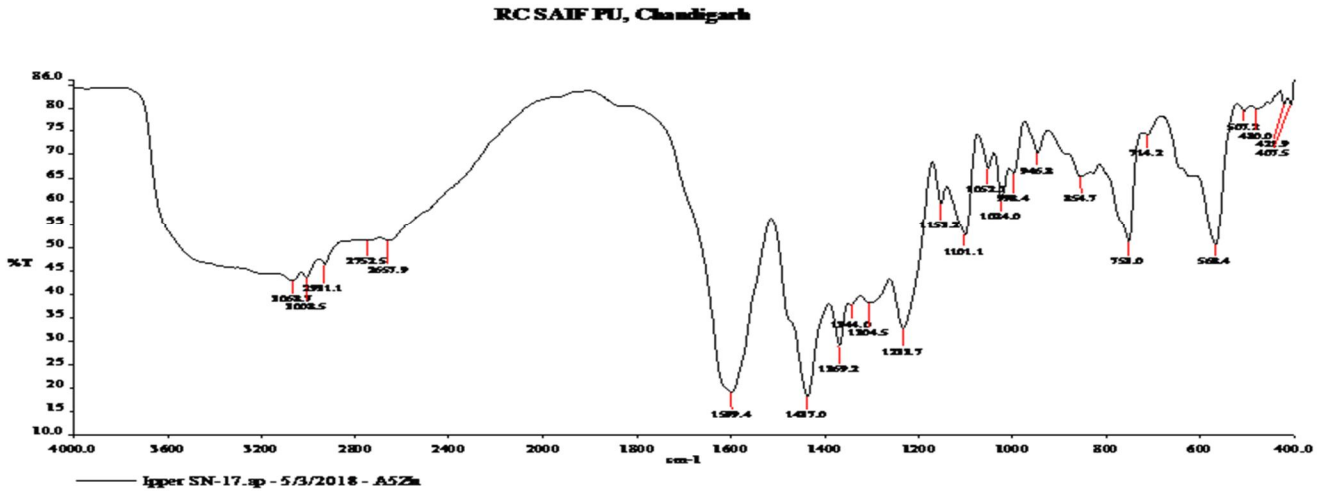

Fig. (2): Infrared spectra of Zn metal complex.

B. Physical Parameters

Table no.(2): Physical parameters of Zinc (II) complex.

Metal complex	Ligand	p^H range ppt	Color	M.P. ^0C
Zinc(II) complex	chalcone of pyridine-2-carbaldehyde	7.0-7.5	Reddish brown	280

C. CHO Analysis

The carbon, hydrogen, oxygen, cobalt and copper metals percentage in Zn(II) complex of chalcone measured at SAIF Cochin, Kerala. The calculated and measured values of CHO analysis are matching and are given in the table no.(3).

Table no. (3): CHO analysis

Metal complexes	Chemical formula	Mol. Wt.	Elemental analysis : % found (calculated)						
			C	H	N	O	S	X(Br)	M
Zn (II) complex	$[C_{28}H_{20}O_6N_2Zn]$	545	61.61 (57.80)	3.69 (4.16)	5.13 (4.81)	17.58 (21.99)	-	-	11.90 (11.23)

D. Magnetic Susceptibility, Solution Conductivity And Electronic Absorption Spectral Data

1) *Magnetic Susceptibility:* The μ_{eff} (B.M.) values at room temperature for Zn(II) complex is dimagnetic these values agree with Square planar geometry of the metal complex [12-13].
2) *Solution Conductivity:* The solution conductivities of 10^{-3} M solution of metal complex in DMSO were measured on EQUIPTRONICS digital conductivity meter EQ - 660 with 20 μΩ to 200 μΩ at 298K temperature. In the present investigation Zn(II) complex is reddish brown in color, stable to air and moisture. Decomposes at high temperature rather than showing sharp melting points. They are insoluble in water and soluble in DMSO, DMF. The low conductivity values in DMSO solution (10^{-3} M) are given in table no.(4) indicates non-electrolytic nature.
3) *Electronic Absorption Spectral Study:* Electronic absorption spectrum was measured on SL159, single beam UV-VIS spectrophotometer.

The electronic spectrum of Zn(II) complex studied in the present investigation exhibit absorption band at 27247(367 nm) cm^{-1} which are assigned to charge transfer band.

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Fig. (3): Electronic absorption spectra of Zn (II) complex of chalcone of pyridine-2-carbaldehyde

Table no. (4): Solution conductivity, magnetic and electronic absorption spectral data of Zn(II) complex.

Zn(II) Complex	Molar Conductance $Ohm^{-1}cm^2mol^{-1}$	μ_{eff} (B.M.)	Absorption Maxima (LMCT) cm^{-1} Charge transfer.
Chalcone of pyridine-2-carbaldehyde	8.04	Diamagnetic	27247(367)

E. Thermal Analysis Zn(II) Complex Chalcone of Pyridine-2-Carbaldehyde

The simultaneous thermogravimetric, differential thermal analysis of Zn(II) complex chalcone of pyridine-2-carbaldehyde was performed in an inert nitrogen atmosphere on Perkin Elmer STA 6000 at SAIF, Cochin, Kerala. The heating rate was 10˚/min and flow rate of nitrogen 50 ml/min. The reference substance used was α Al_2O_3 in platinum crucible and sample weighed in the range of 4-12 mg. The thermogram of Zn(II) complex of chalcone of pyridine-2-carbaldehyde is presented in figure (4). This curve reveals that there is absence of lattice as well as coordinated water in the complex.

The TG-DTA curve of Zn(II) complex chalcone of pyridine-2-carbaldehyde reveals that the complex is thermally stable decomposes above 250˚C and there is no weight loss up to 250˚C indicating the absence of lattice water as well as coordinated water. The first step shows rapid decomposition within a temperature range 250-330˚C with a weight loss of 22.67% (calc. wt. loss 23.79%) which may be due to loss of non-coordinated part two pyridine ring fragments of the ligand. This is confirmed by an endothermic peak at 293.00˚C in DTA. The second step decomposition with a weight loss of 36.05% in the range 340-480˚C, corresponds to the decomposition of coordinated part of the complex. This is confirmed by an endothermic peak at 414.05˚C in DTA. The third step decomposition with a weight loss of 28.98% in the range 490-600˚C, corresponds to the decomposition of remaining coordinated part of the complex. This is confirmed by an endothermic peak at 557.36˚C in DTA. The compound finally decomposes above 600 ˚C with a weight loss 14.92% and form ZnO as final product.

The thermal behavior of zinc metal complexes in the present study indicates high thermal stability. Decomposition of all the complexes is started at a relatively higher temperature (~250˚C), finally giving a metal oxide residue. Thermograms of all the Zn complexes indicate the absence of lattice water as well as coordinated water molecules and it exhibit higher thermal stability.

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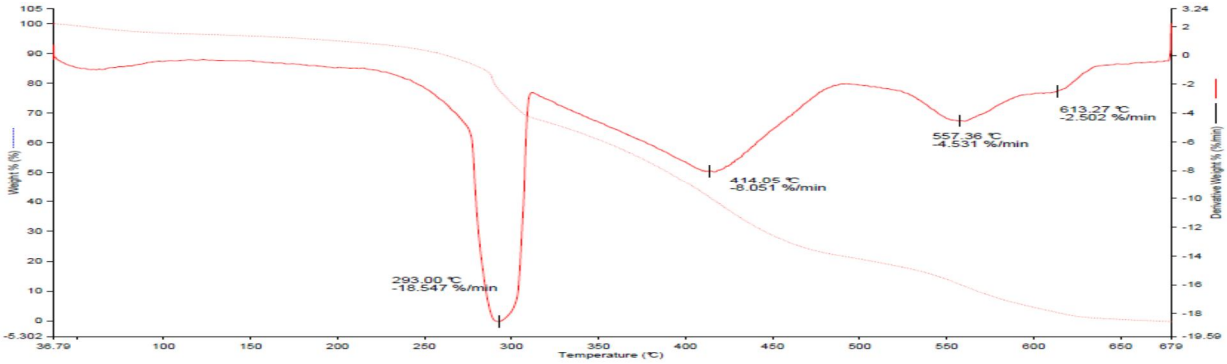

Fig. (4): TG-DTA curve of Zn(II) complex of chalcone of pyridine-2-carbaldehyde

F. X-Ray Diffraction Spectral Studies Of Metal Complex Of Zn(Ii) Complex Of Chalcone Of Pyridine-2-Carbaldehyde

The XRD spectral study has been done at SAIF, Cochin, Kerala. The X-ray diffraction patterns of Zn (II) complex is shown in (Fig .5). The standard deviation observed is within the permissible range. [14] The observed and calculated densities of Zn(II) complex of chalcone of pyridine-2-carbaldehyde are 1.909 gcm^{-3} and 1.816 gcm^{-3} respectively. Chalcone of pyridine-2-carbaldehyde was of Zn(II) complex is found to be tetragonal lattice type with space group P2/m and lattice parameters are a (Å) = 4.9162 b (Å) = 4.9162 c (Å) = 5.4089 α = 90˚ β = 90˚ γ= 90˚ unit cell volume (V) =328.46 $Å^3$

1) Unit cell data and crystal lattice parameters for Co(II)

Unit cell data and crystal lattice parameters

a (Å) = 4.9162 b (Å) = 4.9162 c (Å) = 5.4089 α = 90˚ β = 90˚ γ= 90˚ Volume (V) =328.46 $Å^3$

Density (obs.) = 1.2196 gcm^{-3} Density (cal.) = 1.1265 gcm^{-3} Z = 4 Crystal system= Tetragonal

Space group = P2/m Standard deviation (%) = 0.027

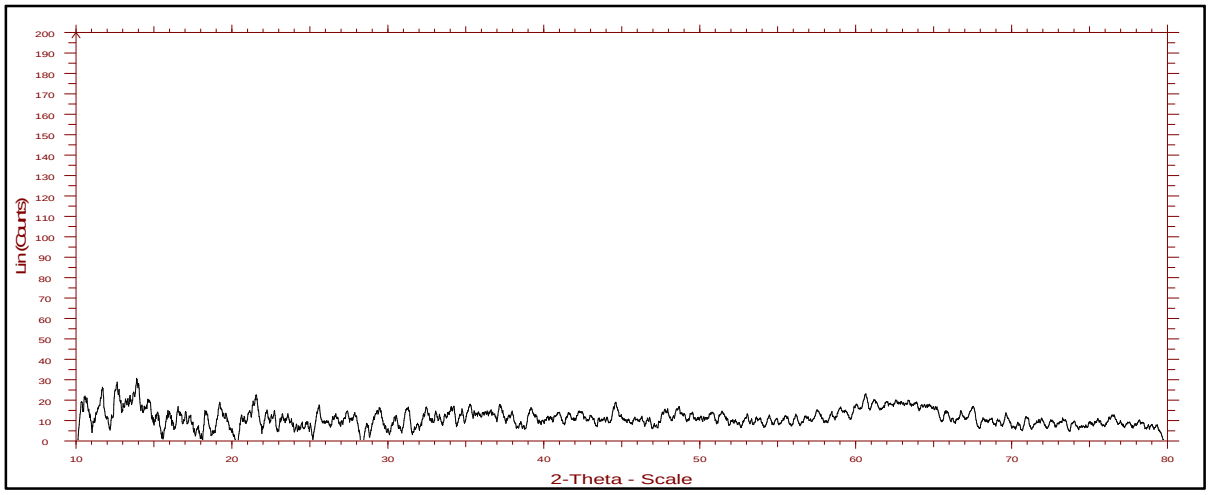

Fig. (5): X-ray diffractogram of Zn(II) complex of chalcone of pyridine-2-carbaldehyde

G. Antimicrobial Activity

Antimicrobial activity was assayed by cup plate agar diffusion method [15] by measuring inhibition zones in mm. In vitro antimicrobial activity of all synthesized compounds and standard have been evaluated against strains of The fungal toxicity of Zn(II) complex was studied in vitro against Aspergillus niger ATCC 16404, Saccharomyces cerevisiae ATCC 9763, Candida albicans ATCC10231 fungal pathogens at fixed 1% concentration. The antibacterial activity of Zn(II) complex was studied, for evaluating antibacterial activity Gram positive and Gram negative bacterial pathogens were used. Staphylococcus aureus ATCC 6538, Bacillus megaterium ATCC 2326, Bacillus subtilis ATCC 6633 were Gram positive pathogens used in this study. Escherichia coli ATCC8739, Salmonella typhi ATCC9207, Shigella boydii ATCC 12034, Enterobacter aerogenes ATCC13048, Pseudomonas aerogenosa ATCC9027, Salmonella abony NCTC6017 were the Gram-negative pathogens used in this stud. From the results of antimicrobial activity of ligands and complexe it is clear that the Zn(II) complex shows enhanced activity than ligand. The increase in antimicrobial activity is due to faster diffusion of metal complex as a whole through the cell membrane or due to the combined activity of the metal and ligand.

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IV. CONCLUSION

The Zn (II) complex was colored, insoluble in most of the organic solvent but soluble in organic solvent. The stoichiometry ratio of the metal complexes obtained has been found to be 1:2.Solution conductivity of this metal complex reveals nonelectrolytic nature. The infrared spectral data indicate that the ligand act as mononegative bidentate species towards Zn(II) complex. The electronic spectral data, IR spectrum, magnetic moment, TG-DTA suggests that Zn(II) has Square planar geometry. The CHO analysis gives C, H, and O percentage in the metal complex. The XRD parameters shows that the structure of Zn (II) is tetragonal and has space group = P2/m.

V. ACKNOWLEDGEMENT

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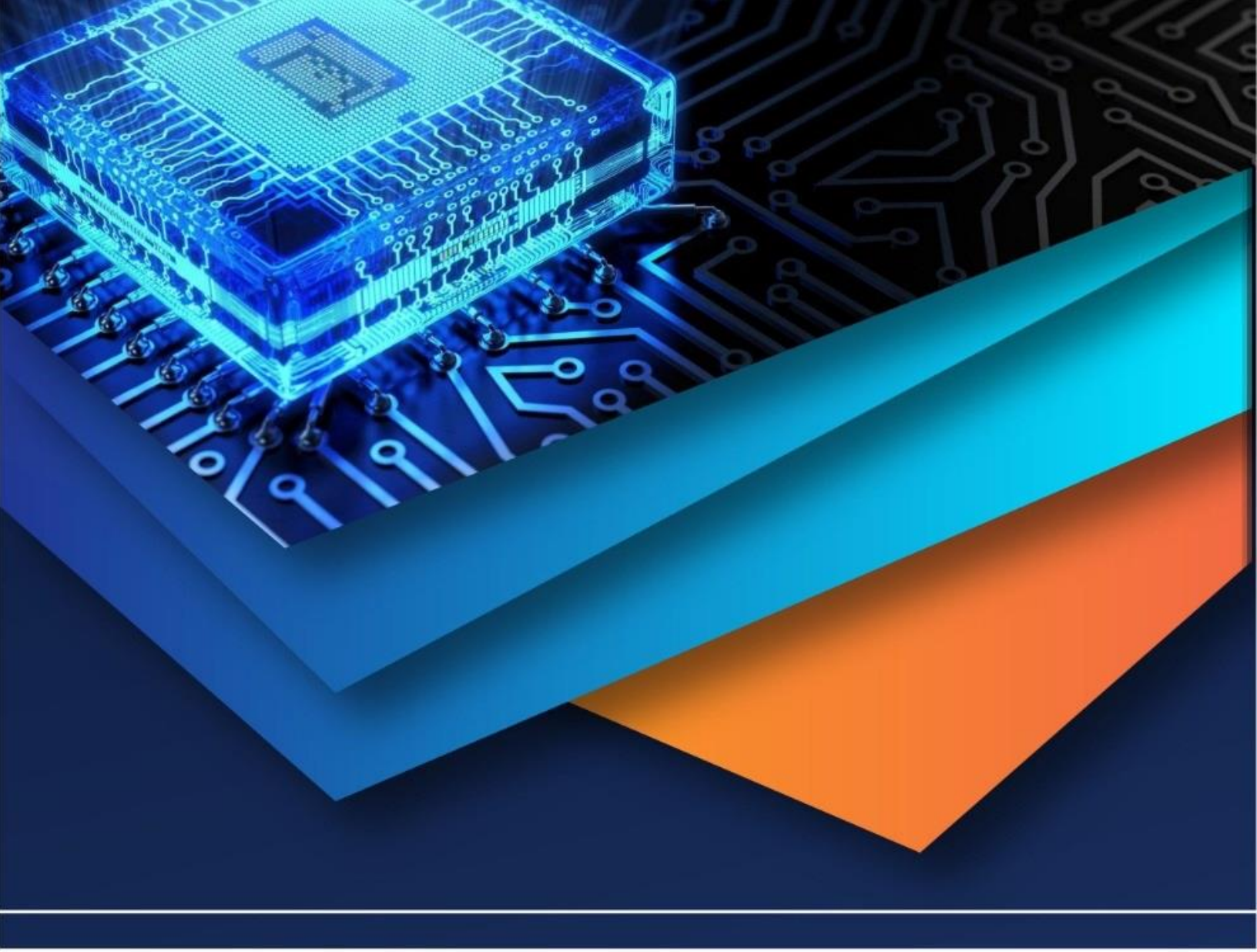

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