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Preparation of Zinc Oxide Nanoparticles and its application in Dental Science

Glanish Jude Martis[1], Jenitta E.P[2], Divya S Nayak[3], Praveen S Mugali[4]
[1]*Alva's Centre for Research in Nanotechnology, Moodubidire, Karnataka, India.*
[2]*P.G Department of Biotechnology, Alva's College, Moodubidire, Karnataka, India.*
[3]*Department of Chemistry, Alva's College, Moodubidire, Karnataka, India.*
[4]*Department of P.G. Studies in Organic Chemistry, Alva's College, Moodubidire, Karnataka, India.*

Abstract: ***Zinc oxide can be called a multifunctional material which has high binding, antimicrobial, UV protection properties. These depend upon the size, shape and absorption peak. Zinc oxide nanoparticles are reported in attracting much attention due to their versatile and promising applications in biological sciences, such as antimicrobial, antifungal agent. Zinc oxide has the sealing ability for cavity filling, acting as cavity filler in the field of dentistry. Each of these factors were noteworthy in determining the properties of materials that lead to different dental applications.***

On the basis of above facts, we synthesized ZnO nanoparticles using $ZnSO_4$ pellets with aqueous NaOH solution which will result in $Zn(OH)_2$ and it was further decomposed to ZnO nanoparticles. The ZnO nanoparticles obtained were subjected for characterization using UV-Visible Spectroscopy, Fourier Transform Infrared Spectroscopy, Scanning Electron Microscopy and X-ray diffraction. The nanoparticles has a absorption peak of 379nm. FTIR showed bands from 2523cm^{-1} to 500cm^{-1}. The Scanning Electron Microscope revealed the nanoparticles to be in Spherical shape with size 96.3nm.The ZnO nanoparticles was subjected to antimicrobial activity using disc diffusion method with E.coli ATCC 25922, Klebsiella pneumonia, Streptococcus mutans MTCC 497, Lactobacillus acidophilus MTCC 10307 and Pseudomonas aeruginosa.These properties of nanoparticles will help in the application of dental science in preparing nanocavity filling material. As we synthesized molecules containing Zinc Oxide and subjected them for characterization, the strength and solubility tests are also determined for the obtained Zinc Oxide nanoparticles. The properties of nanoparticles will aid in the application for dentistry.

Keywords: Cavity Filler; SEM; Olive oil; Zinc Oxide Nanoparticles; Dental Science

I. INTRODUCTION

In recent years, application of nanoparticles within the size range of 1-100 nm has received significant attention due to their novel properties and has come up as an extensive research. Nanoparticles differ in characteristics from those of bulk materials. Due to numerous applications, studies on synthesis, characterization and properties of nanoparticles have received significant attention in the past several years. ZnO's resistance to radiation damages make it useful in various space applications. It has dragged much importance due to its unique applications in biology as an antimicrobial agent. [1]. The sealing capability of ZnO as a cavity filler is most promising in the field of dentistry. [2]. Studies have shown that Zinc Oxide can inhibit acid production *Streptococcus mutans* and *Lactobacillus*. [3]. Being low toxic nanomaterial, it has attracted great notice in various biomedical fields, including anticancer, antibacterial, antioxidant, antidiabetic and anti-inflammatory properties. [4, 5]. Zinc oxide is different in its own way bearing unique physical and chemical characteristics, such as high chemical stability and electrochemical coupling coefficient, very broad range of radiation absorption and quite good photo-stability, is a functional material in enormity. [6]. ZnO nanoparticles were synthesized by different methodologies. It is proved that the various applications of ZnO nanoparticles is dependent on the control of both physical and chemical properties like size, size dispersity, structure, state of the surface, crystalline structure. Due to these reasons, the synthesis of particles became prominent. Hong *et al.* used a controlled precipitation method. The precipitation of zinc oxide achieved using zinc acetate ($Zn(CH_3COO)_2 \cdot H_2O$) and ammonium carbonate ($(NH_4)_2CO_3$.[7]. Lanje *et al.* carried out an easy protocol for the precipitation of Zinc oxide. [8]. Another process of precipitation of zinc oxide in a controlled rate proposed by Wang *et al.* is attractive. Zinc oxide was obtained in a nanometric form by precipitation from aqueous solutions of NH_4HCO_3 and $ZnSO_4 \cdot 7H_2O$.[9]. Varsha Srivastava, Deepak Gusain, Y.C Sharma from Indian Institute of Technology, Varanasi prepared ZnO nanoparticles using 0.2M solution of Zinc Chloride dissolving it in distilled water and Ammonium Hydroxide solution in 2013. [10]. ZnO Nanoparticles also been noted in fighting against bacteria [11, 12], viruses [13] and fungi [14]. In this present work, ZnO nanoparticles have been synthesized by precipitation method using $ZnSO_4$ pellets with aqueous NaOH solution and is useful for dental applications with the use of cloves and olive oil.

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II. MATERIALS&METHODS

A. Preparation of Zinc Oxide Nanoparticles

For synthesis of Zinc Oxide nanoparticles, 2g of Zinc Sulphate ($ZnSO_4$) pellets were taken and transferred to a beaker and 100ml of distilled water is added into it. The purpose of adding distilled water is to make an aqueous solution of $ZnSO_4$. It is dissolved and shaken well. After 10 minutes aqueous solution of 50ml NaOH is added dropwise slowly in a molar ratio 1:2 into the beaker containing Aqueous $ZnSO_4$ solution. It is then stirred vigorously in a magnetic stirrer for 12 hours, it forms a precipitate. The precipitate is now filtered and 2ml of distilled water is added into it. The residue obtained is placed in a Petri dish and kept in a hot air oven at 100ºC for 12 hours. After drying the precipitate is made to a fine powder. This fine powder is now Calcined at 300ºC, 500ºC, 700ºC and 900ºC for 2 hours. The powder obtained after calcination is Zinc Oxide nanoparticles.

$$ZnSO_4 + 2NaOH \longrightarrow Zn(OH)_2 + Na_2SO_4$$

$$Zn(OH)_2 \longrightarrow ZnO + H_2O$$

B. Characterization of ZnO Nanoparticles

1) *UV-Visible Spectroscopy:* The absorbance spectrum is obtained which shows the absorbance of a compound at different wavelengths and provide strong information as a tool of evidence. The amount of absorbance at any particular wavelength is due to the chemical structure of the molecule [15]. The spectrum of ZnO nanoparticles depends on method of fabrication, size, shape and temperature.
2) *Fourier Transform Infrared Spectroscopy (FTIR):* Fourier Transform Infrared Spectroscopy was used to recognize the functional groups bound specifically on the surface. The sample was used to examine at the spectra range of 400-4000 cm^{-1}.
3) *Scanning Electron Microscopy (SEM):* Scanning Electron Microscopy analysis of ZnO nanoparticles provides information about the surface, shape and size of the particles. Gold coating for the sample was done to prevent charging of the sample. It also increases the amount of secondary electrons that can be detected from the surface of the specimen in the SEM
4) *X-ray Powder Diffraction (XRD):* XRD is an analytical technique used for phase identification of a crystalline sample and is capable of providing information on cell dimensions [16].

C. Antimicrobial Activity

The ZnO nanoparticles was subjected to antimicrobial activity using disc diffusion method with *E.coli* ATCC 25922, *Klebsiella pneumonia*, *Streptococcus mutans* MTCC 497, *Lactobacillus acidophilus* MTCC 10307 and *Pseudomonas aeruginosa* where it showed sensitive zones for all pathogens except for *Pseudomonas aeruginosa.* 20µL of Olive oil is used as control and the discs of Amoxicillin, a broad-spectrum antibiotic, used here to test its effectiveness on specific pathogen.

D. Solubility Tests

The ZnO nanoparticles were treated with commonly used drinks like Tea (Red Label), Coffee (Nescafe) and Carbonated Lemonade (Limca) in order to determine the percentage of solubility and check for distortion caused if any after treatment.

III.RESULTS AND DISCUSSION

A. Characterization

1) *UV-Visible Spectroscopy:* UV-Vis spectroscopy was performed to know the formation of synthesized particles. The measurement was carried out after the dispersion of ZnO nanoparticles in distilled water. As shown in Figure 1, the absorption peak was observed at 379 nm which impute to the integral band-gap of Zn-O absorption and also this peak region is beneficial in antibacterial response.

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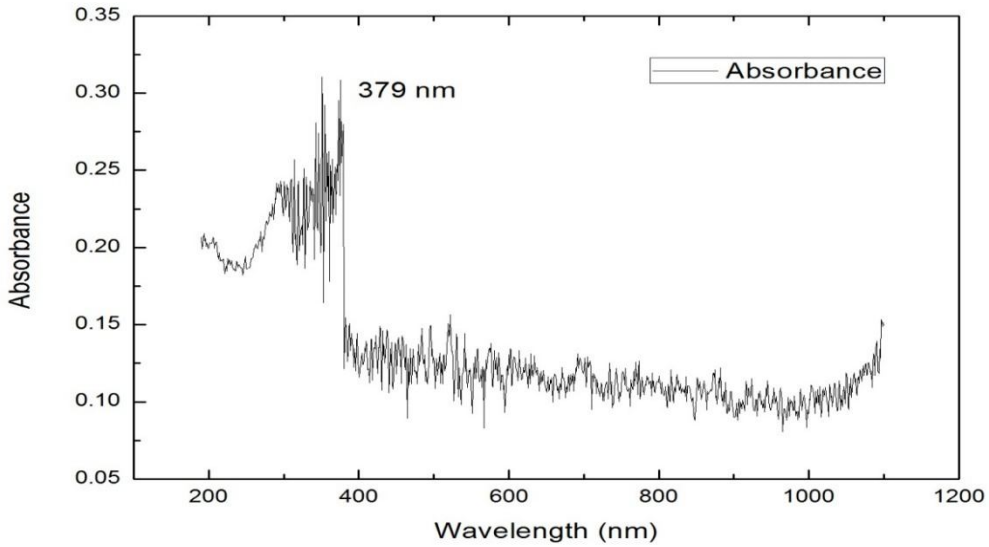

Fig. 1 UV-Visible Spectroscopy

2) *Fourier Transform Infrared Spectroscopy (FTIR):* As shown in Figure 2, the absorption frequency nearly 450 cm^{-1} is corresponding to Zn-O stretching mode of vibration.

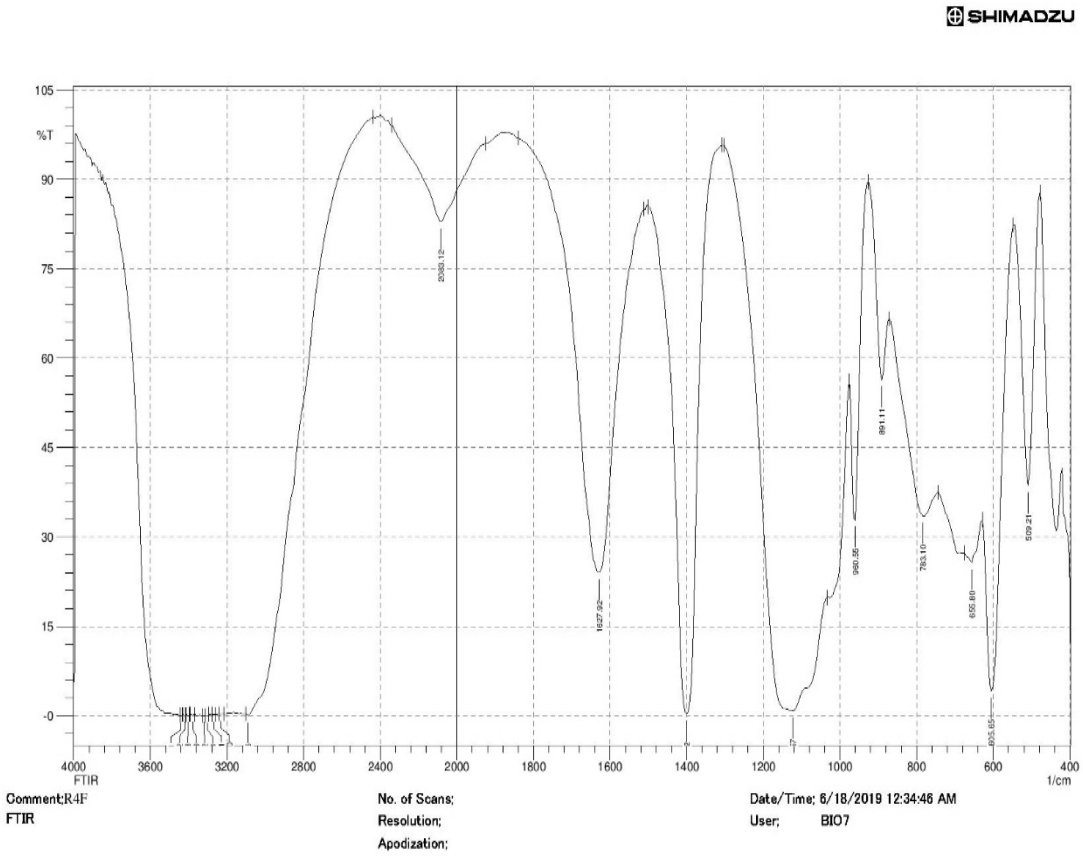

Fig. 1 Fourier Transform Infrared Spectroscopy

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3) *Scanning Electron Microscopy (SEM):* Figure 3.shows the size of the synthesized nanoparticles to be 96.3 nm.

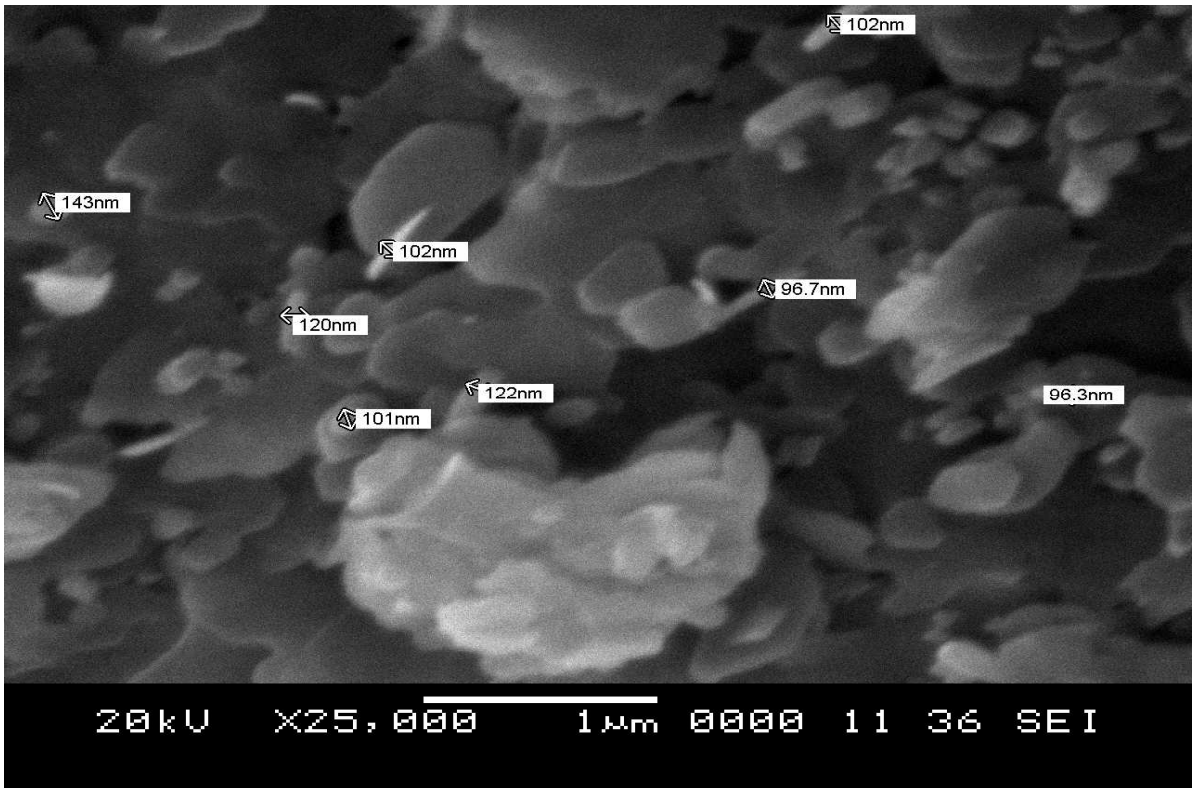

Fig. 3 SEM Image of ZnO nanoparticles

4) *X-ray Powder Dioffraction (XRD):* XRD of ZnO nanoparticles, the results of nanoparticle size measurement of samples of XRD and SEM indicate that the size of the ZnO nanoparticles was about 96.3 nm.

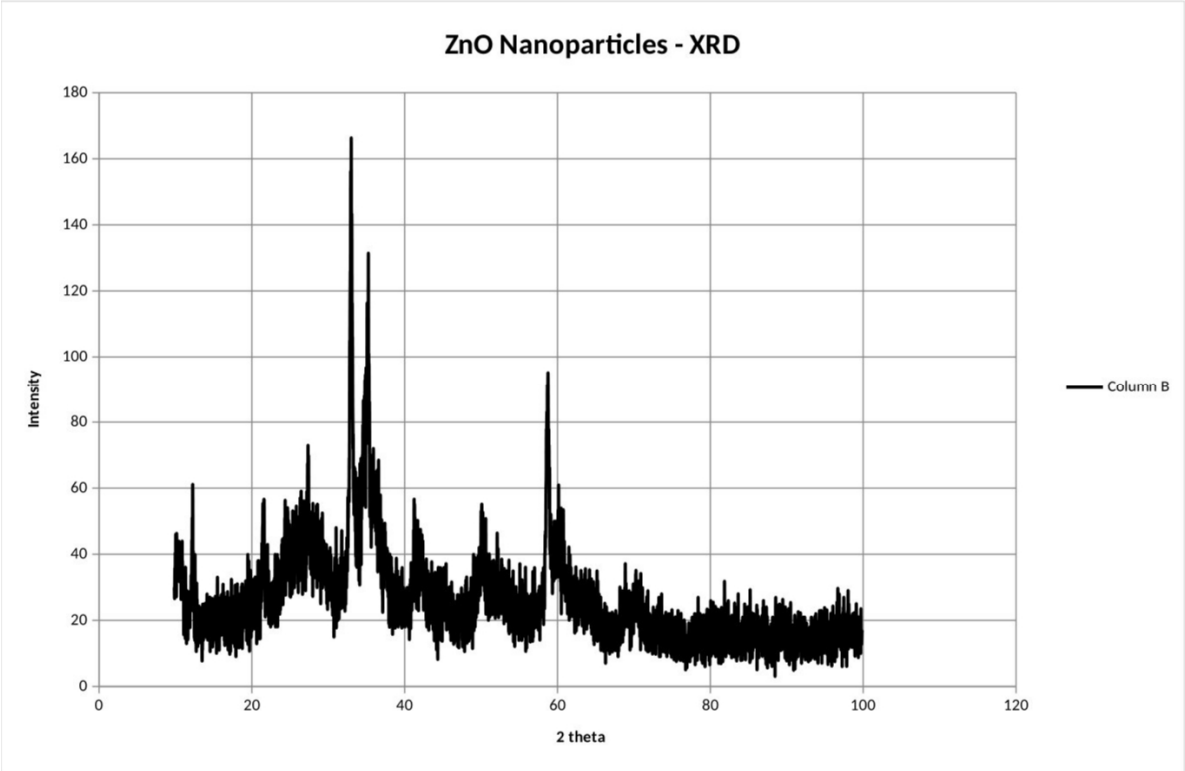

Fig. 4 XRD Pattern of ZnO nanoparticles

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B. Antimicrobial Activity

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TABLE I
RESULTS OF ANTIMICROBIAL ACTIVITY

Sl. No.	Species	Inhibition Zone (mm)
1	*E. coli* ATCC 25922	16
2	*Klebsiella pneumonia*	15
3	*Streptococcus mutans* MTCC 497	16
4	*Lactobacillus acidophilus*	15
5	*Pseudomonas aeruginosa*	Nil

C. Solubility Tests

Percentage of solubility was nearly same for all the three hot and cold beverages. No distortion took place in the nanoparticles after subjecting daily consuming drinks containing various metabolites etc.

TABLE II
RESULTS OF SOLUBILITY TESTS

Test Samples	ZnO Nanoparticles (in grams)	Percentage of solubility
Tea (Red Label)	0.06	1.2
Coffee (Nescafe)	0.059	1.18
Cold Drink (Limca)	0.065	1.3

IV. CONCLUSIONS

The work was itself a satisfactory after subjecting the synthesized nanoparticles of ZnO into various physical and analytical techniques like UV-Visible Spectroscopy, FTIR, SEM and Powder XRD. The results obtained from these curriculums are noteworthy in preparing the temporary cavity filler. Antimicrobial activity suggest the inhibitory zones when the culture was swabbed with different microbial strains. Solubility Tests reveal the tiny percentage of solubility under both hot and cold conditions using beverages and carbonated drink.

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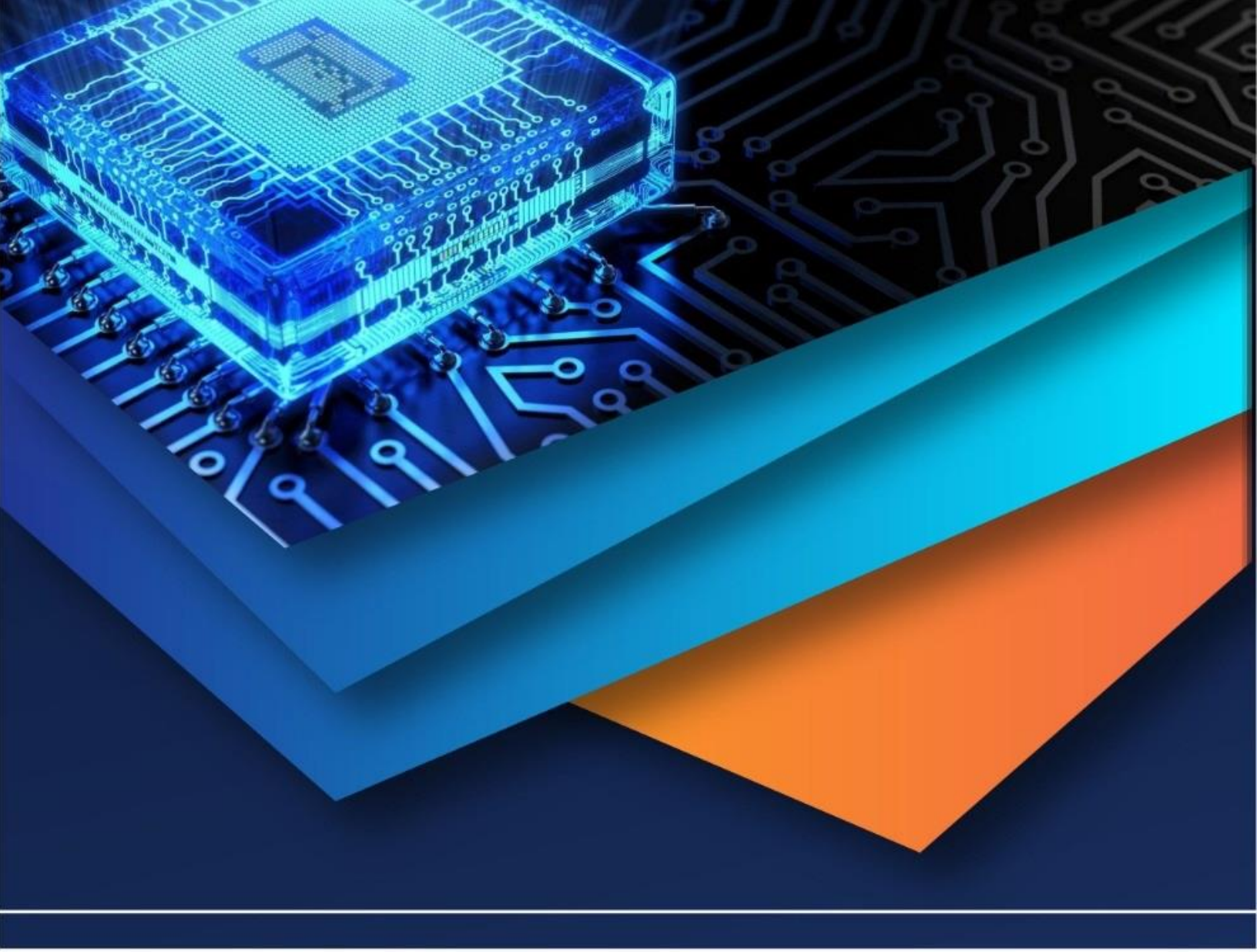

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