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Green synthesis: Synthesis and Characterization of Titanium Dioxide Nano Particles using Aloe Vera Extract

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Abstract: ***Green synthesis of nano particles aims at reducing generated waste and implementing sustainable processes. Green processes using mild reaction conditions and nontoxic precursors have been emphasized in the development of nanotechnology for promoting environmental sustainability. In the present work, nano particles of titanium dioxide were synthesized using an aqueous solution of Aloe Vera extract. Synthesized nano particles are characterized by X-ray diffraction (XRD), Scanning Electron Microscope (SEM), Energy dispersive spectroscopy (EDS), Ultra Violet Visible Spectrum Analysis (UV) and Fourier Transform Infrared Spectroscopy (FTIR).***
Keywords: Green Synthesis; Titanium tetra isopropoxide; TiO_2 Nanoparticles; Aloe Vera;

I. INTRODUCTION

A technology that measures and manipulate or incorporate materials with a critical dimension between 1-100nm. The unique physical and chemical properties are due to its high surface to volume ratio comparing with micro or bulk sized. Nanotechnology is the engineering of functional systems at the molecular scale. Nanomaterial's are of great interest because at this scale unique optical, magnetic, electrical and other properties emerge. The fabrication of nano particles with strict control size, shape, and crystalline structure has inspired the application of nanotechnology to numerous fields including catalysis, medicine and electronics.

Titanium dioxide (TiO_2) has become part of our everyday lives. It is found in various consumer goods and products of daily use such as cosmetics, paints, dyes and varnishes, textiles, paper and plastics, food and drugs, and even paving stones. TiO_2 is a promising material due to its photo–stability, chemical structure, physical, optical, electrical properties, low cost and excellent degradation of organic pollutants. Green synthesis is one of the newly introduced or recently developed non conventional methods to produce nanoparticles. In this method a number of biological systems which include plant extracts, green algae, micro organisms, such as fungi and bacteria are used to synthesis the nanoparticles. Nanoparticles produced by plants are more stable.

This method reduces the use of hazardous substances in the design, manufacture and application of chemical product. The main methods of nano particle production by chemical and physical method are costly and potentially harmful to the environment. It is desirable for biomedical applications such as drug carriers, cosmetics, and filling the material. Green synthesis of nanoparticles is an innovative branch of nanoscience

The objective of the present work is to prepare TiO_2 nanoparticles by green synthesis method using Aloe Vera extract.

II. EXPERIMENTAL METHOD

In the present work, the experimental procedure comprises of two stages. First, the preparations of plant extract and secondly the preparation of titanium dioxide nanoparticles using the prepared plant extract.

A. Preparation Of Plant Extract

The leaves of aloe Vera were separated from plant, which were thoroughly washed and cut into small pieces. For extract preparation 25g of the leaves in 100ml distilled water is taken and boiled for 2hrs at 90ºC. The extract was filtered using what man filter paper. The filtrate was stored for the synthesis of nano particles.

B. Preparation Of Tio_2 Nano Particles

To 0.1M of titanium tetra iso propoxide the aloe Vera plant extract was added drop wise under constant stirring to achieve a solution of pH 7. The mixture is subjected to stirring for 4hrs continuously. In this process, nanoparticles were formed and are separated using filter paper. The particles were washed with double distilled water repeatedly to remove the by-products if any. The nano particles were dried at 100ºC overnight and calcinated at 450ºC for 4hrs

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The characterizations of prepeared titanium dioxide were done by using X-ray diffraction (XRD), Scanning Electron Microscope (SEM), Energy Dispersive X- Ray Analysis (EDAX) , Ultra Violet Visible Spectrum Analysis (UV) and Fourier Transform Infrared Spectroscopy (FTIR). Flow chart depicting the green synthesis of Nano crystalline TiO_2 is shown in figure 2.2.1

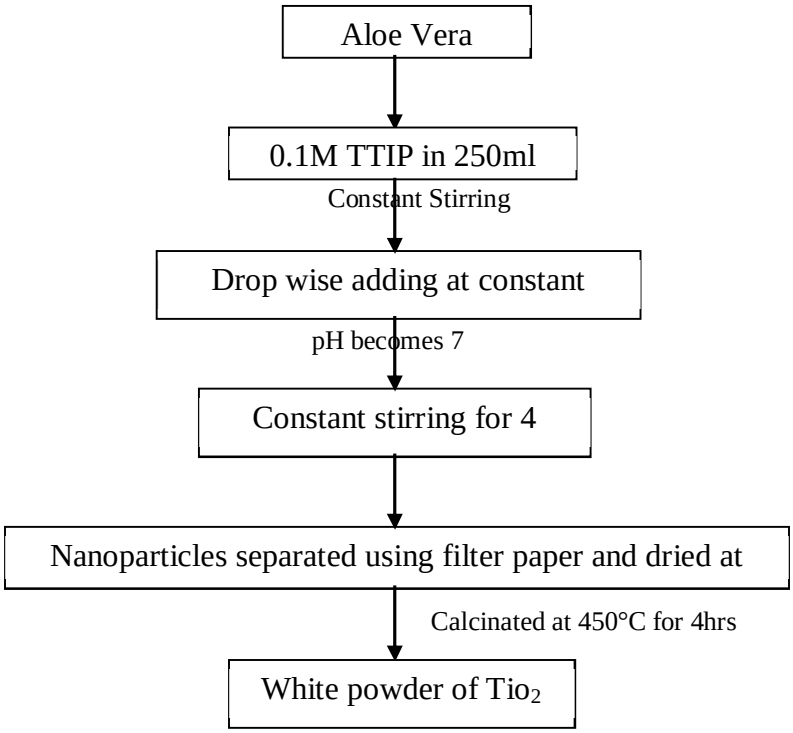

Fig 2.2.1 Flow chart for the green synthesis of TiO_2 Nanoparticles

III. RESULT AND DISCUSSION

A. X-Ray Diffraction Analysis

X−ray Powder Diffraction (XRD) is an efficient analytical technique used determination of grain size, composition of solid solution, lattice constants, and degree of crystalline in a mixture of amorphous and crystalline substances. It is a general technique for the study of crystal structures, atomic spacing, crystallite sizes, stress analysis, lattice parameters, and quantitative phase analysis and can provide details on unit cell dimensions.

This information is important for relating the production of a material to its structure and hence its properties. For this reason, in order to investigate the phase formation and micro structure studies, the XRD analysis was carried out on the prepared TiO_2 nanoparticles using plant extract.

The XRD pattern of Titanium dioxide nano particle prepared using green synthesis method is shown in figure (3.1). The average crystalline size was calculated by using the data from X-ray diffraction pattern with the Debye-Scherrer formula.

The Debye-Scherrer equation is,

$$D = k\lambda / \beta \cos\theta$$

Where,

D = crystallite size
λ = wave length of the filament used in XRD machine (1.5418 Aº)
β = Full Width Half Maximum (FWHM)
θ = angle of the corresponding peak
k = shape factor (0.94)

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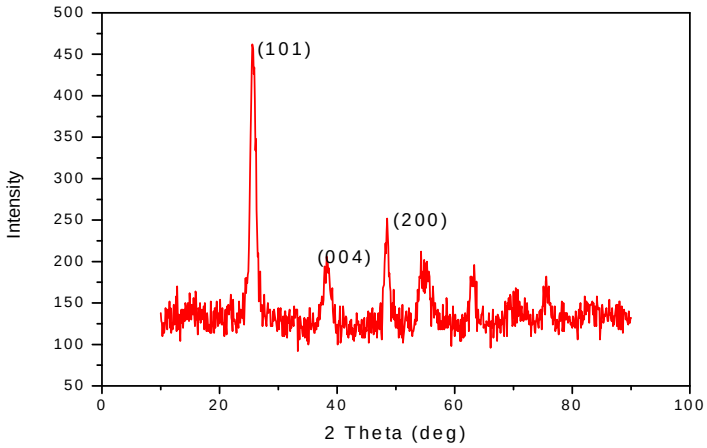

Fig 3.1.1 XRD pattern of TiO_2 nano particles.

The XRD spectrum showed peaks around 25^0, 38^0, 48^0, 54^0, 62^0 and 75^0. The most intense peaks observed around at $2\theta = 25.68°$, 48.43°, 38.25° indexed the planes (101), (004), (200) respectively. The diffraction data were in good agreement with JCPDS card no 21- 1272. The observed peaks indicate the nature of TiO_2 nanoparticles. The average crystalline size was found to be in the range of 5-8 nm**.** The TiO_2 sample possessed evident diffraction peaks characteristics of anatase, which indicates that the TiO_2 nanoparticles prepared are predominantly of the anatase crystal phase. The peak details of the TiO_2 nanoparticles are listed below in the table 3.1.1

Table 3.1.1 – The peak details of the TiO_2 nanoparticles.

Sample	2θ(degree)	Phase and orientation (hkl)	FWHM (deg)	Crystallite size(nm)	d_{hkl}
TiO_2 Nanoparticles	25.68	(101)	1.1214	7.265	3.46
	48.43	(004)	0.9700	8.979	1.87
	38.25	(200)	1.4500	5.798	2.35

B. Scanning Electron Microscope (FESEM)

The grain size, shape and surface properties like morphology of the prepared titanium dioxide nanoparticles were investigated by SEM analysis. The SEM image was observed within the magnification of 0.5µm. TiO_2 nanoparticles showed irregular agglomerated particle structure. The size was found to be 50 nm. The magnified image of titanium dioxide nanoparticles is shown in figure (3.2.1).

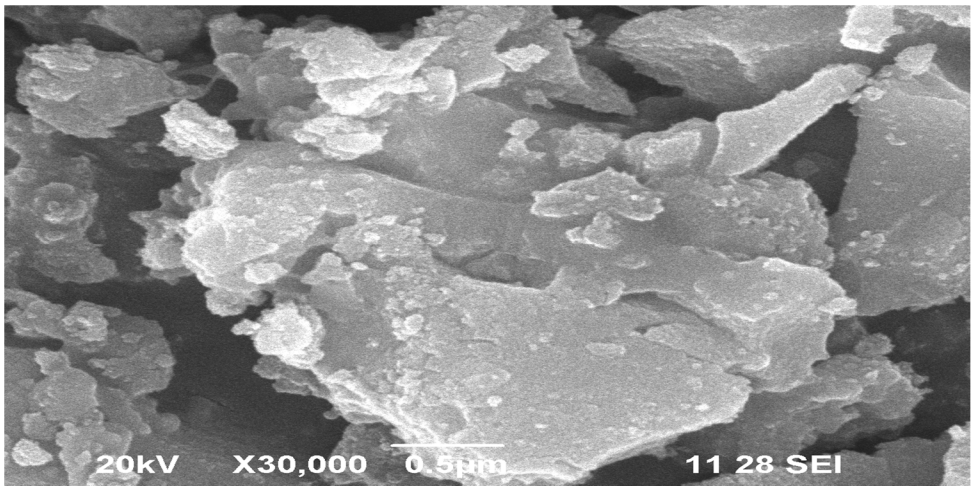

Fig 3.2.1 SEM image of TiO_2 nanoparticles

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C. Energy Dispersive X-Ray Analysis

The EDAX spectrums of Titanium dioxide nanoparticles prepared using Aloe Vera extract is shown in figure (3.3.1). The EDAX spectrum revealed the presence of titanium and oxygen in the prepared samples. The EDAX analysis confirmed the formation of titanium dioxide nanoparticles. It also indicates the absence of any impurities in the prepared sample.

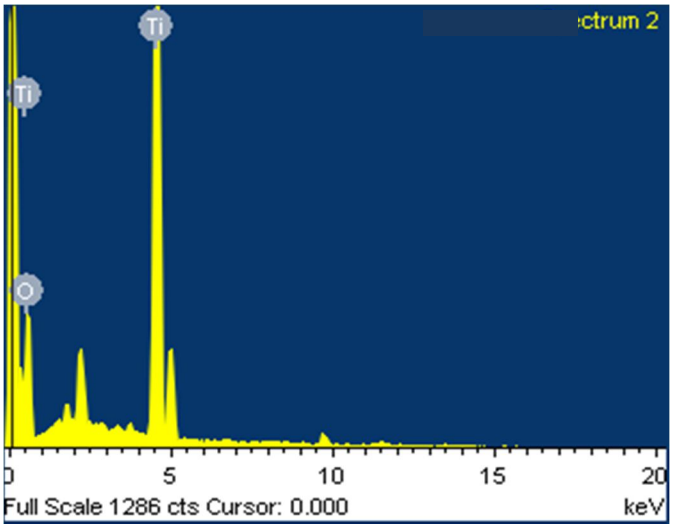

Fig 3.3.1 EDAX spectrum of TiO_2 nanoparticles.

D. UV - Visible Spectroscopy

The energy band gap value was determined based on the mathematical derivative of the optical absorption coefficient. The fundamental absorption method corresponds to band to band transitions by using energy relation. The absorption spectrum of TiO_2 nanoparticles exhibits a strong absorption at 410 nm. The energy band gap was determined to be 3.02 eV by using the energy relation.

$$\mathbf{E_g = 1240/\lambda_g}$$

The UV-VIS spectrum of TiO_2 nanoparticles is shown in fig. (3.4.1).

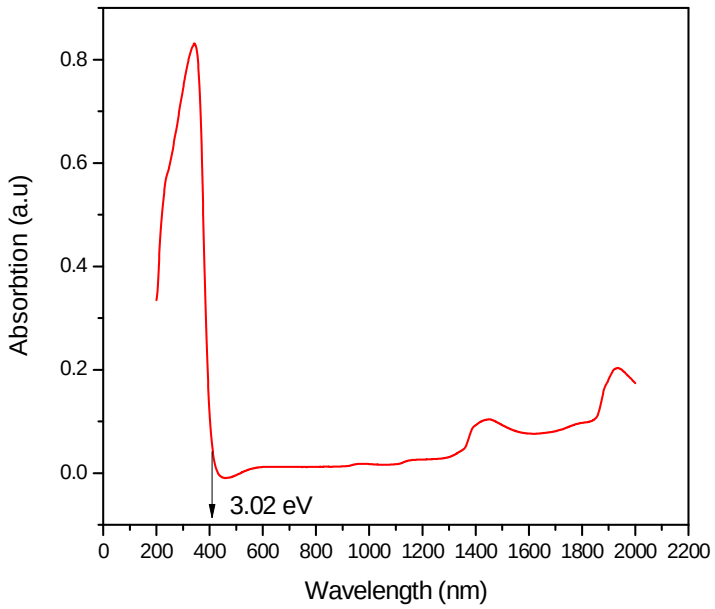

Fig 3.4.1 Absorbance spectrum of TiO_2 nanoparticles.

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E. Fourier Transform Infrared Spectroscopy

The FTIR spectrum of TiO2 nanoparticles calcinated at 450°C for 4 hours is shown in figure. The diffraction peaks corresponding to the broad band centered at 447 cm-1 which is the characteristic of Ti-O Skeletal vibration.

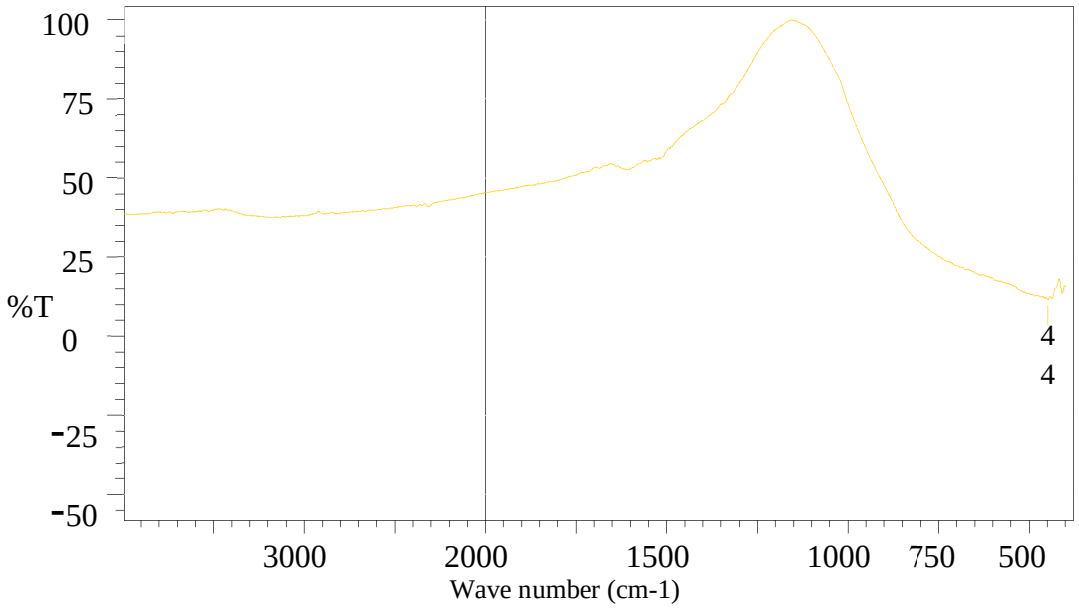

Fig. 3.5.1 FTIR spectrum of TiO2 nanoparticles

IV. CONCLUSION

The Titanium dioxide nanoparticles were synthesized by Green synthesis method using Aloe Vera extract. The prepared sample was analyzed using various characterization techniques such as XRD, SEM, EDAX, UV/VISIBLE spectrum and FTIR. The XRD pattern shows a strong diffraction peaks at 2θ = 25. 68º, 48.43º and 38.25º and the peak values were in good agreement with the JCPDS card No.21-1272 representing a tetragonal structure. The particle size ranges from 5nm to 8 nm. The optical absorption UV spectrum is analysed for the TiO_2 nanoparticles. The band gap energy is calculated as 3.43eV and the increase in band gap is due to the defects level present in the sample as the sample contains some by-products of Aloe Vera. The SEM image shows the irregular particle structure and the size obtained as 50nm.The EDAX spectrum shows the presence of Ti and O in the prepared sample. The FTIR analysis shows the skeletal mode of vibration for the peak with wavelength of 447nm.

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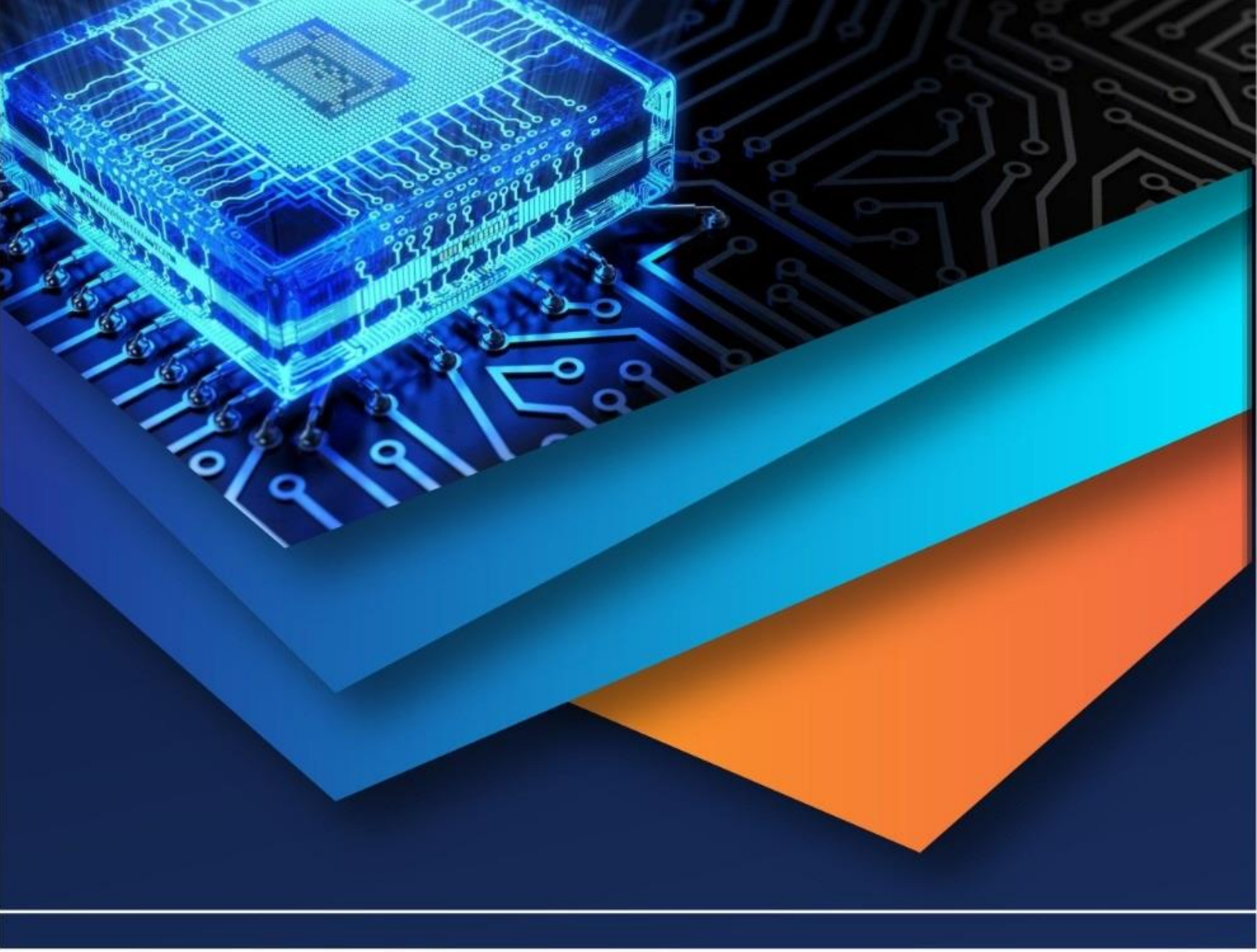

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