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Synthesis, Characterization and Studies of ZrO_2-PVA composites Using Super critical Fluid Method

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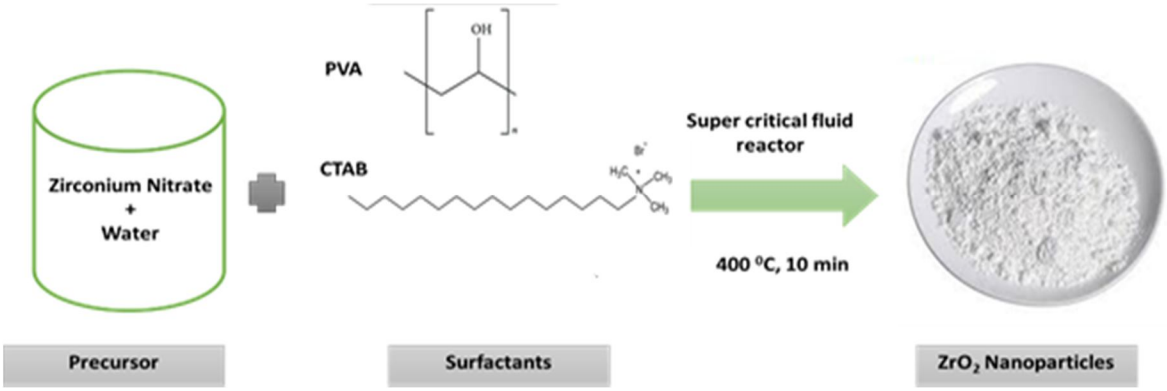
Abstract: ***In the new years, Supercritical liquid extraction (SCF) technique has yield great outcome in line of nanomaterials and its nanocomposites in the best and productive manner. In this philosophy, extraction medium is added to substances containing objective parts and extraction is performed in light of contrasts in solvency. As of late, it has stood out as a harmless to the ecosystem extraction technique that doesn't utilize hurtful natural solvents. Zirconium oxide can be consolidated in different polymer and metal composites to work on the thermo-mechanical properties of the base material. The construction and morphology of as pre-arranged ZrO2-PVA and ZrO2-CTAB Nanocomposite test was described by utilizing powder X-beam diffraction (XRD) device and by Scanning Electron Micrograph (SEM) instrument individually. Fourier Transform infrared (FTIR) otherworldly review was embraced to know the holding.***

Graphic abstract:



Keywords: Zirconium Oxide, PVA, Cetyl-tri-methyl ammonium bromide (CTAB), Supercritical fluid, Extraction (SCF).

I. INTRODUCTION

The Research in supercritical extraction innovation began around twenty years prior. In the interim, a few hundred supercritical liquid have been intended to work at very high pressures[1-3]. Normal applications, worked through supercritical liquids (SCFs), are the extraction which are performed on an enormous modern scale[4-6]. A few more modest modern units are likewise in activity for extraction of flavors for the food business and regular substances for use in beauty care products. Involving SCFs in various cycles might prompt the development of totally new items with specific qualities modest affecting the climate, like low energy utilization during the cycle, alongside wellbeing and security benefits. SCF can be utilized as solvents for precipitation and micronization. As response medium, as a portable stage for chromatography (supercritical liquid chromatography — SFC), thus on[7-10]. In any case, the most usually examined process where SCFs are utilized as dissolvable media is the supercritical liquid extraction (SFE) process[7-10]. Poly vinyl liquor (PVA) is utilized as most proficient fuel for microwave burning response. Metal oxides and its nanocomposites are ready by microwave illumination of metal hydroxides with PVA as fuel. At present the work is an endeavor to get ready nanosized ZrO2-PVA and ZrO2-CTAB nanocomposite materials[11-13]. These materials are ready by Super Critical Fluid involving PVA and CTAB as surfactant. At first metal hydroxide antecedents are ready and are microwave lighted with PVA as surfactant[14-17]. As framed ZrO2-PVA and ZrO2-CTAB Nanocomposite materials are utilized for its nanocomposite readiness. As pre-arranged metal oxides and its nanocomposite materials are very much described for its affirmation with different portrayal tools[18-20].

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II. EXPERIMENTAL

A. *Materials and Methods*

All chemicals used in the current experimentation were of AR (Analytical Reagent) grade. Double distilled solvents are used in the current work. Super Critical Fluid method was used for combustion conversion process. Polyvinyl alcohol is used as a fuel for the combustion. Zirconium Nitrate solution is used as Precursor, whereas PVA and CTAB are used as surfactants.

B. *Synthesis of ZrO2-PVA Nano-composite*

To synthesize surface modified ZrO, 0.1 mol of zirconium nitrate and 1 wt% of PVA surfactants was added into 20 ml of Deionised water and stirred for 30 min, after complete dissolution the solution was transferred into super critical fluid (SCF) reactor and stored at 400 ^{0}C for 10 min. After reaction complete the reactor was quenched in cold water. The obtained product was taken out and washed in water and methanol consistently then dried at 60 ^{0}C overnight to get powder.

C. *Synthesis of ZrO2-CTAB Nano-composite*

The same steps were performed to synthesize surface modified ZrO, 0.1 mol of zirconium nitrate and 1 wt% of CTAB surfactants were added into 20 ml of Deionised water and stirred for 30 min, after complete dissolution the solution was transferred into super critical fluid (SCF) reactor and stored at 400 ^{0}C for 10 min. After reaction complete the reactor was quenched in cold water. The obtained product was taken out and washed in water and methanol consistently and dried at 60 ^{0}C overnight to get powder.

III. RESULTS AND DISCUSSION

A. *X-Ray diffraction*

Figs. 1a 1b and 1c show XRD pattern of ZrO_2 , ZrO_2-PVA and ZrO_2- CTAB Nanocomposite

Figs. 1a 1b and 1c show indexed XRD pattern using Supercritical fluid reactor of ZrO_2 , ZrO_2-PVA and ZrO_2- CTAB samples. The three patterns shows the presence of some Bragg's reflections confirms the formation of crystalline product. The d-spacing values of the ZrO_2 , ZrO_2-PVA and ZrO_2- CTAB samples match well with standard data of JCPDS file 36-1451 and 75-0594. Unit cell parameters were obtained by least-square refinement of the powder XRD data. This study reveals the samples are monophasic with cubic spinal structure having nanosized particles. Fig. 1b shows XRD pattern of as prepared ZrO_2-PVA nanocomposite. Fig. 1c shows XRD pattern of as prepared ZrO_2-PVA nanocomposite. The pattern shows the presence of sharp and highly intensed peaks due to crystalline nature. Bragg's reflections of ZrO_2 , ZrO_2-PVA and ZrO_2- CTAB are identified as ZrO_2 , ZrO_2-PVA and ZrO_2-CTAB in the composite pattern confirms the formation of different phased nanocomposite.

B. Scanning Electron Microscope

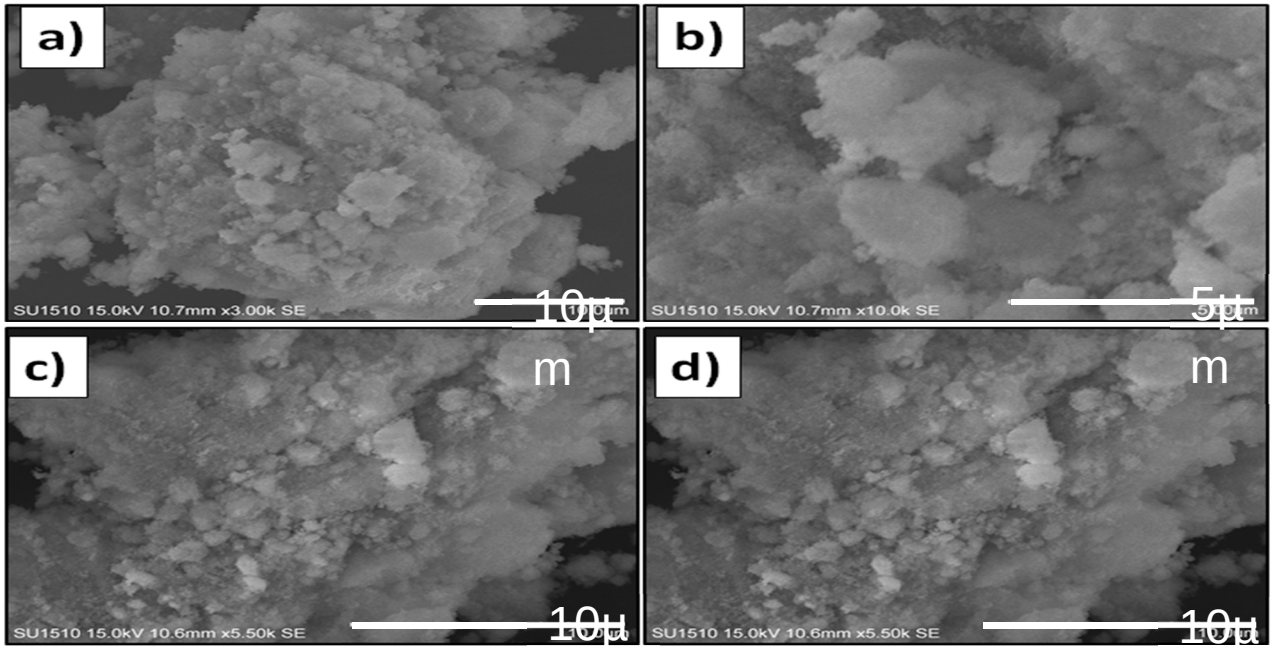
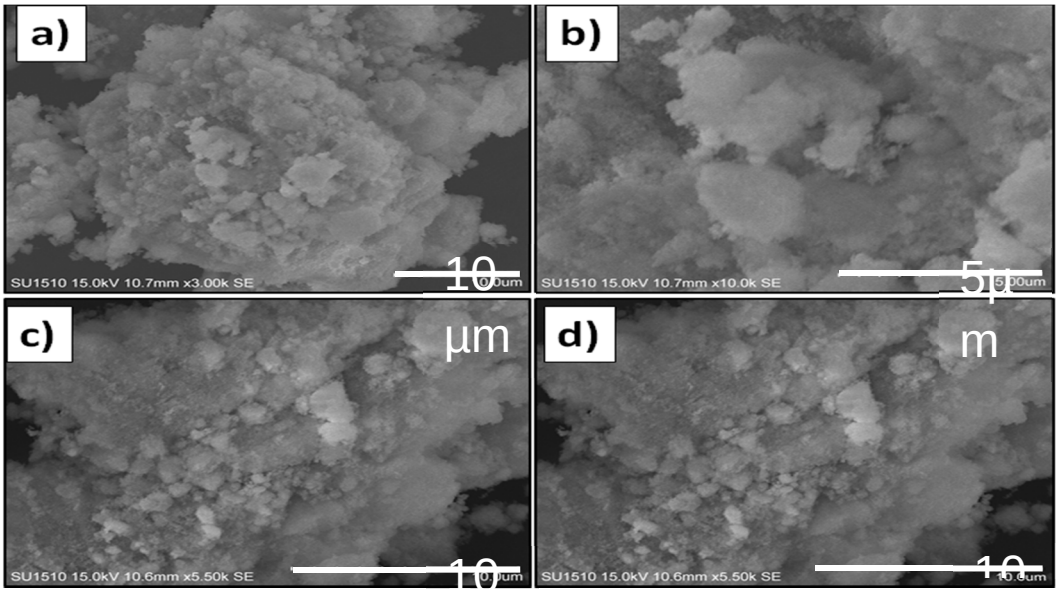


Fig. 2 SEM Images of 2 a & b: $ZrO_2$and 2 c & d: ZrO_2-PVA

The surface morphology of the above prepared ZrO_2-PVA and ZrO_2- CTAB nanocomposite is studied by scanning electron micrograph. Figure 2(a-b) shows the SEM image of as prepared ZrO_2-PVA and ZrO_2- CTAB nanocomposite materials at various resolutions. The image shows irregular shaped particles of fine agglomeration with dense and compact structure. Dispersion of the oxide nanoparticles into PANI matrix masked with somewhat crystalline nature. However in higher resolution it is clearly observed that a sheet of polymer containing fine particles of oxide increased the compact nature confirms the formation of ZrO_2-PVA Nanocomposite.



XRD Patterns of 3 a & b: ZrO_2 and 3 c & d: CTAB-ZrO_2

The surface morphology of the above prepared ZrO_2-PVA and ZrO_2- CTAB nanocomposite is studied by scanning electron micrograph. Figure 3(a-b) shows the SEM image of above prepared ZrO_2-PVA and ZrO_2- CTAB nanocomposite materials at various resolutions. The image shows irregular shaped particles of fine agglomeration with dense and compact structure. Whereas Figure 3(c-d) shows the SEM image of as prepared ZrO_2- CTAB nanocomposite materials shows its oxalate precursor at low and high magnification, respectively. Self-assembled nanosphere like structure of ZrO_2- CTAB, forming globular network with micro dimensions, are observed. On higher magnification, some nanosized spherical particles are also observed

C. FT-IR Spectroscopy

Figure 4 shows FT-IR spectrum of ZrO_2, ZrO_2-PVA and ZrO_2- CTAB nanocomposite.

A broad peak from 3500-3000 cm^{-1} shows the presence of moisture . A straight peak at 1850 and 1550 cm^{-1} is due to presence of CH stretching and some overtunes. The peaks below 1000cm^{-1} correspond to metal oxygen modes. Peaks at 750cm^{-1} may be attributed due to Zr-O peaks confirm the formation of ZrO_2, ZrO_2-PVA and ZrO_2- CTAB nanomaterials.

D. UV-Vis Study

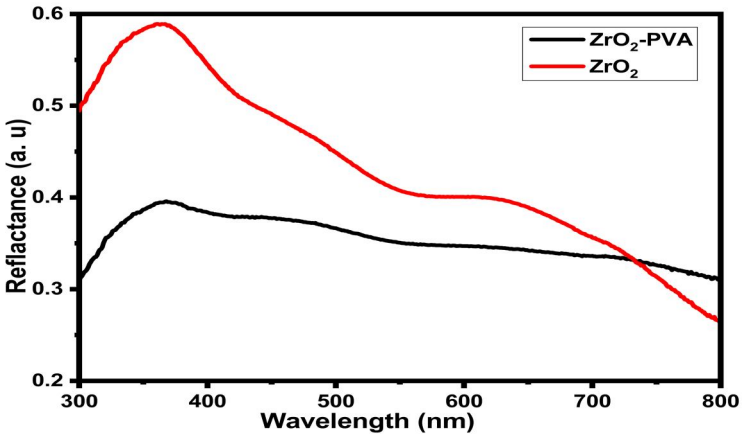


Figure 5 shows the UV absorbance spectra of ZrO_2, ZrO_2-PVA composite. It is observed that the maximum absorbance occurs at 700–800 nm. The UV spectra for monodispersed particles should be narrow, while the absorption spectrum of wide size distribution is broad. The single-peak absorbance spectra indicate the presence of spherical size of the particles, but in the above spectra it is observed the double-peak absorption spectra that lie between 300–400 nm indicate that the shape of the particles may be Spherical in ZrO_2-PVA composite.

E. TG-DTA (Thermo gravimetric – Differential Thermal Analysis)

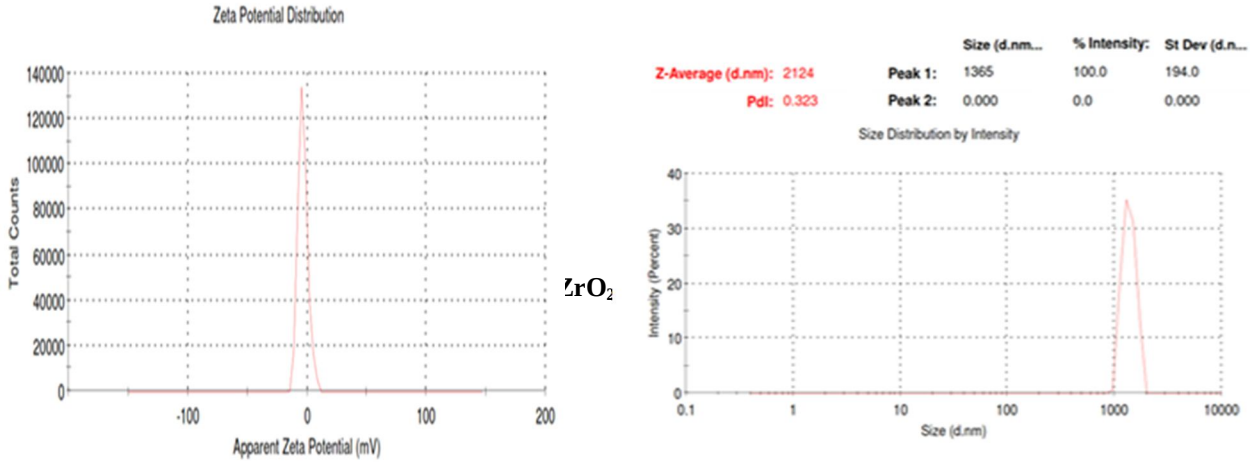
Figure 6 thermal analysis (TGA) of sample ZrO_2-PVA

Figure 6 shows the thermal analysis (TGA) of as-obtained sample ZrO_2-PVA, which gives the information about the percentage weight loss of Zirconium oxide upon increasing the temperature, which in turn indicates thermal stability, The thermogram indicates two-step mass loss with prevalent weight loss of ZrO_2-PVA sample. Initially,it starts decomposition gradually with a rise in temperature up to 350 °C. The major decomposition of nanocomposite occurs at 350–440 °C, indicating thethermal stability of composite, and is also ascribed to strong interaction between zirconium and PVA in the composite. The inserted ferrite increases the thermal stability in comparison with pure PVA. The existence of strong binding force in the composite is due to the interaction between ZrO_2-PVA nanomaterials and PVA backbone. The primary mass loss from 10 to 100 °C is a moderate adversity that demonstrates the loss of moisture and organic element from nanoparticle.

F. DLS (Dynamic Light Scattering)

Dynamic light scattering was carried out to determine surface charges and dispersion stability of the synthesized ZrO_2 and PVA-ZrO_2. Zeta potential was determined by knowing the mobility of colloidal particles suspended in a water by applying electric field. The hydrodynamic size and poly dispersion intensity was determined to confirm the surface functionalized moieties and dispersion stability of the synthesized ZrO_2 and PVA-ZrO_2. The zeta potential of ZrO_2 and PVA-ZrO_2 is shown in figure 7 a & b.

The ZrO_2 surface modified with PVA shows -3 mV zeta potential whereas without modified ZrO_2 has +3 mV. The PVA-ZrO_2 shows negative charge because of negative charges on the surface. Which confirms the modification of ZrO_2 with PVA[13]. The hydrodynamic size and poly dispersion intensity of both ZrO_2 and CTAB-ZrO_2 is shown in figure 7 c & d. The average hydrodynamic size of pure ZrO_2 was found to be 178.7 nm with poly dispersion intensity of 0.475. There were two peaks one at 200 nm and another at 5281 nm which indicates the aggregation of pure ZrO_2 nanoparticles in other words poly dispersion. In CTAB-ZrO_2 the hydrodynamic size was found to be 1365 nm with 0.323 poly dispersion intensity[14]. The higher hydrodynamic size probably due to denser surface attached PVA molecules and there was no other peak. Which, indicates there was no aggregation and hence no poly dispersion occurred in CTAB modified ZrO_2. More uniform size and much stable particles are suitable for plastic scintillator preparation. Therefore, CTAB modified ZrO_2 were found to be more suitable in plastic scintillator preparation[15].



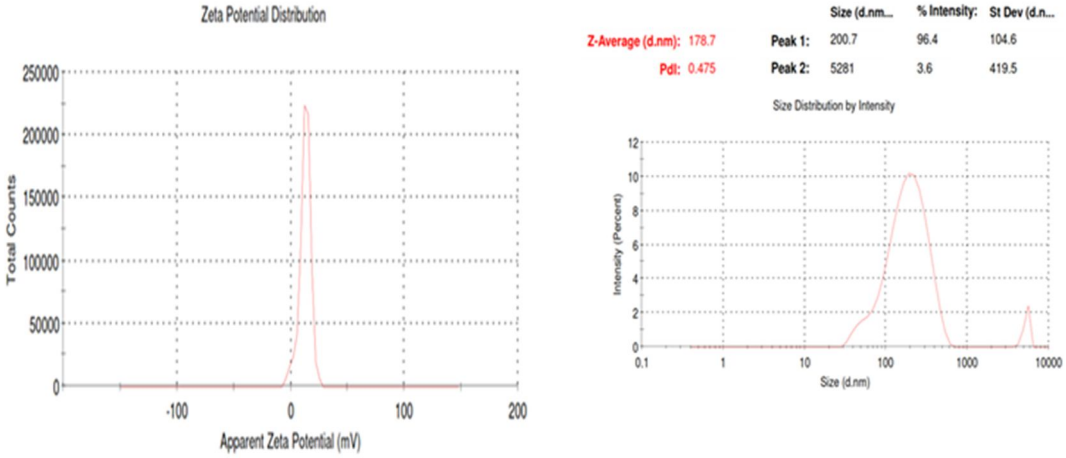


Fig 7 c. Zeta Potential of PVA-ZrO_2 and 6 d. Size distribution of PVA-ZrO_2

IV. CONCLUSION

Supercritical fluids are cheap, inert and non toxic. Thus, they are readily disposed off after an extraction is completed by allowing them to evaporate into the atmosphere. In this study, we envisage a simple and efficient synthetic procedure in the synthesis of different metal oxide nanoparticles choosing a single precursor. The use of PVA as a fuel in the synthesis is new and the present synthetic procedure may be applied for the synthesis of other metal oxides and even for the synthesis of mixed metal oxide nanoparticles.

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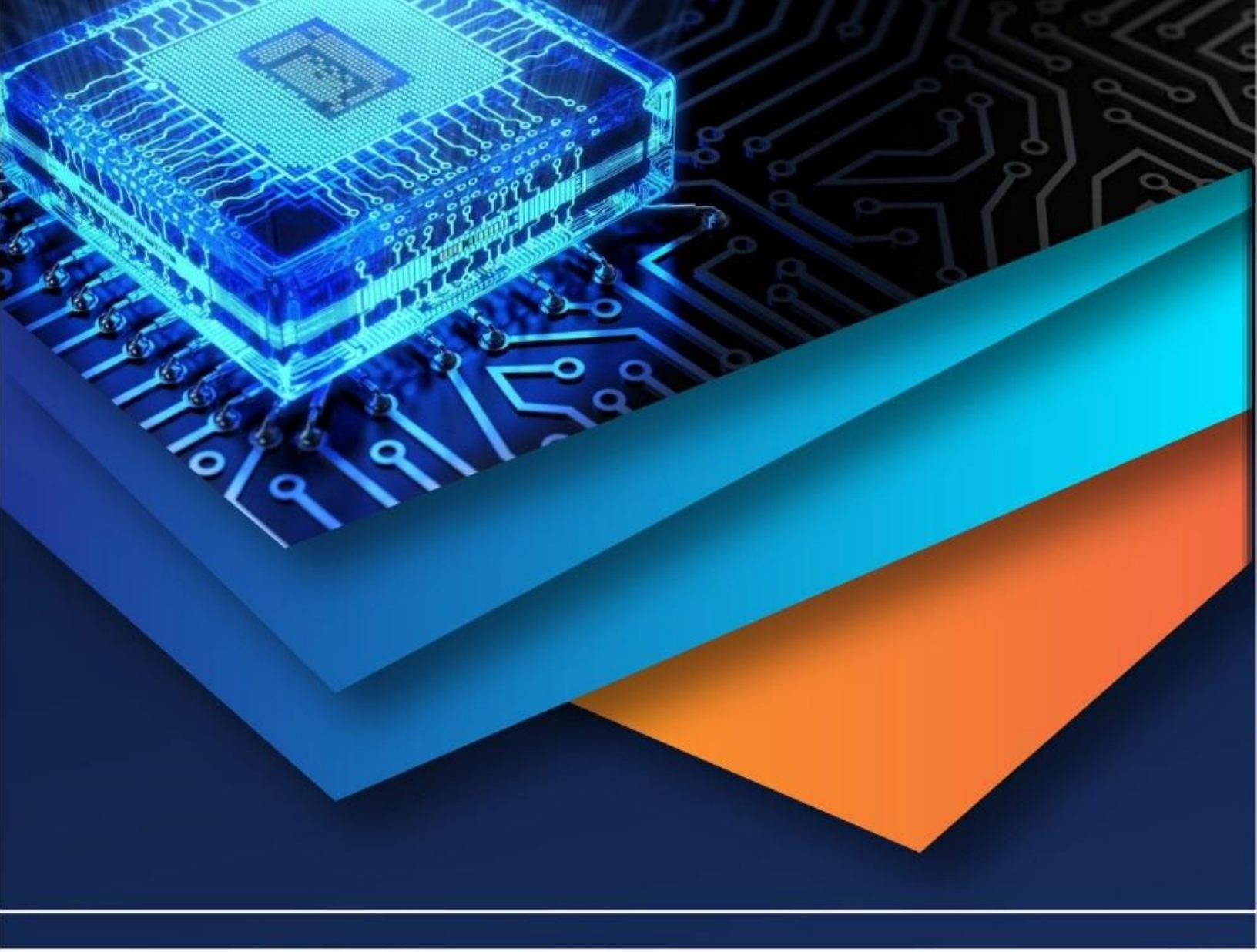

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