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Hydrothermal Synthesis of Multiwall Carbon Nanotubes using Polystyrene: Purification and Characterization

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Abstract*: In the present research the multiwall carbon nanotubes (MWCNTs) were synthesized by hydrothermal treatment of polystyrene in the presence of catalyst. In the typical synthesis, the carbon precursor polystyrene was reacted with catalyst iron particle in the closed condition with pressure of hydrothermal condition. The obtained product was purified with strong, mixture of acid, which can helpful for the removal of metal catalyst from the product. Further, the purified MWCNTs were sintered at 400°C under nitrogen gas atmosphere to reduce the percentage of amorpous carbon. The MWCNTs were characterized by Field Emission Scanning Electron Microscopy (FESEM) for morphology study, EDX for elemental analysis, X-ray diffraction (XRD) study for crystalinity measurements, Raman spectroscopy to find out defects and graphitization of CNTs and Dynamic Light Scattering (DLS) process for particle size analysis.*
Keywords: Carbon nanotubes, hydrothermal, polystyrene, ferric chloride

I. INTRODUCTION

In the present and upcoming future, nanotechnology have enormous applications including bottom up technology in electronics, biological, pharmaceuticals, ecological industries, and in energy source field(1). The nanoparticles were synthesized in different methods such as chemical vapour deposition (2), electro chemical synthesis (3), pyrolysis (4), laser ablation method (5), hydrothermal synthesis (6), and flame synthesis (7) etc. CNTs having very interesting in the field of nanotechnology which operate the substance at nanometer level (8).

That is, their structural aptness, which had made them ideal objects to study and had attracted the attention of physicists, but in many instances, CNTs appeared to be difficult to handle, purify, sort, disperse, and mix(9). Yoshimura and co-workers synthesized CNTs from polyethylene, ethylene glycol and other sources with and without catalysts under hydrothermal conditions at 700–800 ^{o}C and 60–100 MPa (10). In the present research work also polymer was used as a carbon source for the CNTs synthesis, using hydrothermal method. Usually alkene derivatives were used to synthesis CNTs, such as hydrocarbon, polymers, benzene derivatives etc, because of the sp^2 behaviour of parent molecule help us to synthesis expected CNTs. In this regard choosing of polymers (alkene derivatives) as a carbon source is always best.

Transition metals such as Fe/Co/Ni etc were played an important role as a catalyst in the hydro solvothermal synthesis of nanoparticle, because of their variable oxidation states and high surface area under pressure.

Hydrothermal synthesis of nanoparticle is always a cost effective process under high and low temperature, which has a new turn and definition to advanced nanotechnology by Byrappa and Yoshimura. So hydrothermal is a novel synthesis method for the production of multiwall graphitic nanocarbons(11).

II. MATERIALS AND METHODS

In the present study, the CNTs were synthesized by hydrothermal method. All the chemicals used were of analytical reagent (A.R) grade purchased from Fischer chemicals. In a typical synthesis, 1.5 g of powdered polystyrene was added to 30ml dichloromethane, kept the solution mixture in magnetic stirrer for 30 minutes. 8 ml of above solution was transferred into 20 ml Teflon liners; add 6 ml of 20% sodium hydroxide solution and 0.25g of dried ferric chloride. The autoclaves placed in a hot air oven, heated at constant temperature 180^{o}Cfor the duration of 48 hr. Further the autoclaves were cooled down to room temperature. The products were collected and washed with 1 M Hydrochloric acid and alcohol, filter to get the black colour material. This resultant material was treated by sintering process at 400^{o}C for 2hr in the helium gas atmosphere.

A. Purification

In this study, a liquid-phase oxidation purification method was adopted to eliminate catalyst, support and amorphous impurities. This method was adopted to eliminate the possibility of damage to the structure of these materials. Since the CNTs usually synthesised by hydrothermal and CVD methods are accompanied by many other types of carbon particles, it was essential to use a strong oxidant to purify the raw CNTs. Therefore, the as-prepared CNTs were purified with a mixture of concentrated HNO_3 and H_2SO_4 in the ratio of 3:1by volume. The amount of acid used was determined from the stoichiometry of reaction between iron and these acids. The raw CNT samples were soaked in the mixture of these acids and vigorously stirred for 48 h at room temperature. They were subsequently washed repeatedly with distilled water until a pH of about 7 was achieved. They were dried in air at 120 ^oC for 12 h. The treated CNT samples were ground into fine particles and characterised using the XRD, FESEM, EDX, Raman and DLS.

B. Characterization Techniques

The synthesized products were characterized by X-ray powder diffraction (XRD) performed on a Bruker D8 Advance powder X-ray diffractometer to check the purity and phase structure of products, Raman spectral studies on FT Raman (Perkin Elmer) instrument were made to understand the graphitic nature of the product. The surface morphology of the samples was examined with field emission scanning electron microscope (FESEM) using JEOL model JSM 6490 LV, the particle size distribution of synthesized MWCNTs were analyzed by dynamic light scattering (DLS) method and the elemental analysis was carried out by using energy dispersive X-ray spectrophotometer (EDX).

III. RESULTS AND DISCUSSION

Since being the special tool structural characterization of materials, the X-Ray diffraction technique has been employed to study the phase structure and nature (crystalline or amorphous) of the obtained product. The crystalinity of material was determined by powder X-ray diffraction (XRD) on a Rigaku miniplex II desktop diffractometer with Cu kα radiation (λ=1.5406 A^o) in the scan range of 6-80^o 2θ and the scanning rate was 0.02^o s^{-1}. The X-ray pattern of as synthesized material is shown in the Figure 1. In this figure the strong peak at 2θ angle of 25.96^o was found with maximum intensity with (0 0 2) plane and the second highest peak is at 2θ 42.87^o with (1 0 0) plane. The identification of homogeneous crystalline phases of carbon nanotubes were accomplished with JCPDS Number 00-058-1638.

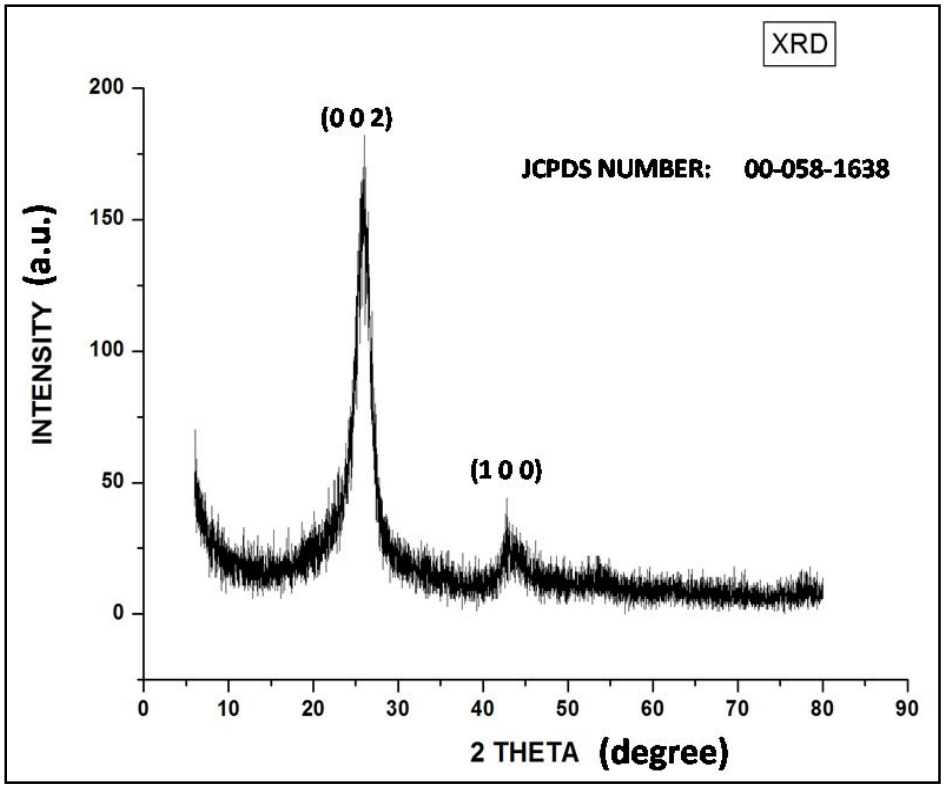

Figure1:X-ray diffraction pattern of carbon nanotubes

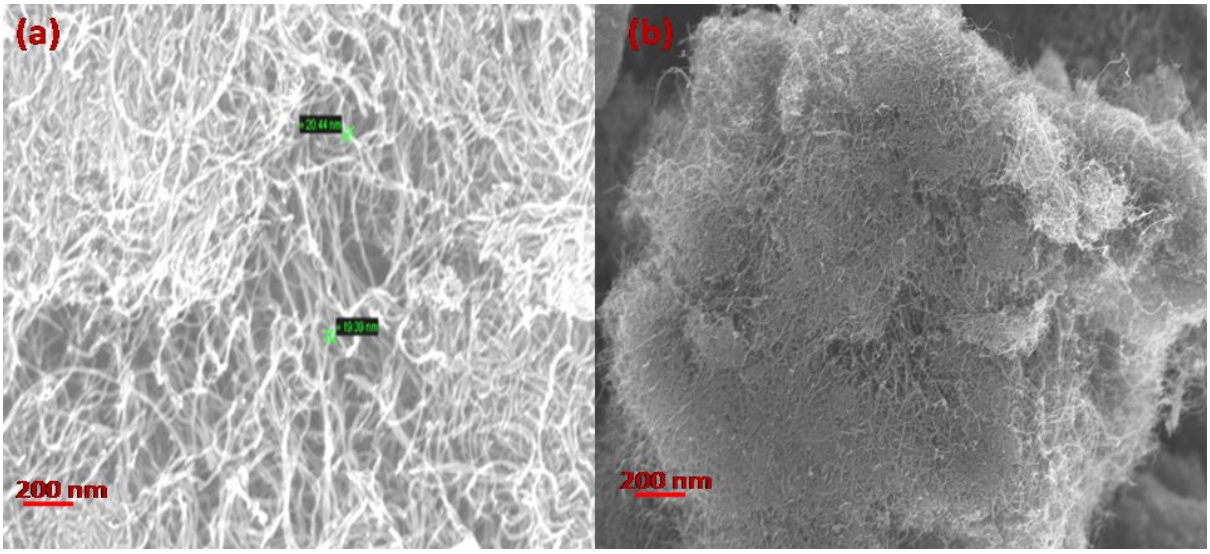

Figure 2.FESEM images of (a) purified and (b) as synthesized MWCNTs

The morphologies and nanostructure of the as-synthesized materials were characterized with Field Emission Scanning Electron Microscopy (FESEM). Figure 2(a and b) shows the FESEM images of the as synthesized MWCNTs with different magnifications indicating that the MWCNTs have outer diameters approximately 20 nm. The MWCNTs are about several millimetres long and their typical inner and outer diameters are 19 nm and 22 nm, respectively, with wall thickness of 5nm. The TEM images reveal that the CNT samples are relatively free of amorphous carbon. It is clearly seen that the MWCNTs were arranged in a disorder manner (entangled) that is the alignment was less and that the impurities are present (amorphous carbon and metal particles). The observation of samples by electron microscopy after hydrothermal treatment revealed aggregates of bundled nano size carbon tubes.

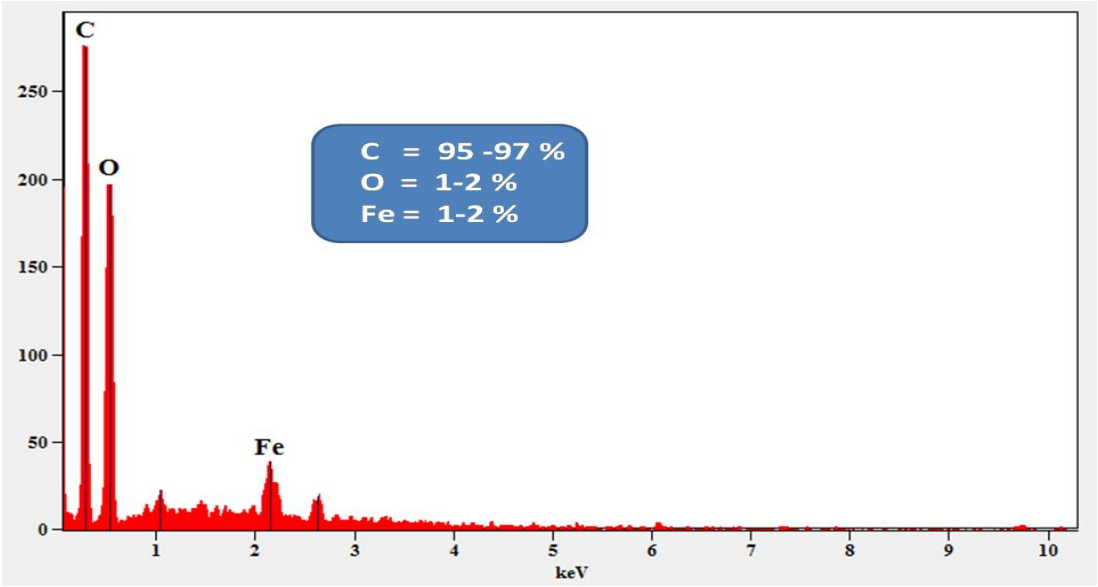

Figure 3: EDX spectra of purified MWCNTs

The elemental analyses of MWCNTs were characterized by using EDX spectrophotometer. The figure 3 shown that the presence of elements like Carbon, Oxygen and Iron. It was proved that the element Fe was not removed completely during the purification step, because the metal particles with nanosize were incorporated to the centre of carbon nanotube and therefore it was very difficult to complete removal of metal atom. Further, the purified MWCNTs were contain 95-97 % of carbon atom, which was present as tube like structure, 1-2 % of oxygen atom, which was bind carbon atom during synthesis and sintering process and finally it contains iron atoms, which was incorporated to the centre of tube during synthesis. From these results, it was concluded that the growth of MWCNTs was happened by the presence of surface of metal catalyst.

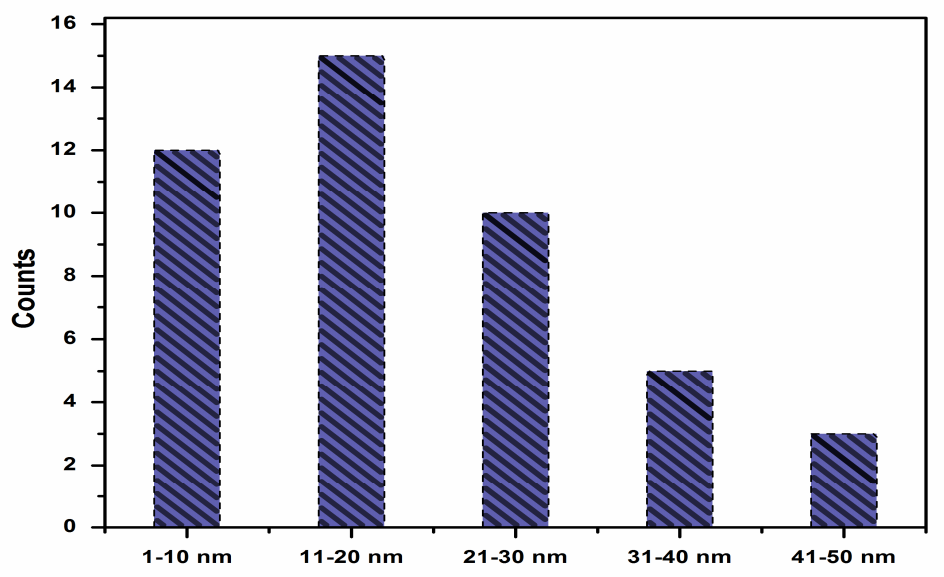

Figure 4. Particle Size Distribution of Carbon nanotubes.

Particle size of the MWCNTs was determined by dynamic light scattering (DLS) technique. The particle size with the average diameter of 20 nm which matches with diameter of FESEM image for the purified MWCNTs as shown in the FESEM image. Dynamic light spectroscopy (DLS) is one measurement that provide information of nanomaterials size distribution. Although DLS measurement is suitable to determine the diameter of particles with tubes and sphere shape, the data could be used to evaluate variation of length distribution for MWCNTs. The measurement of particle size distribution of purified MWCNTs was analyzed in the solvent medium. For those dispersing in solvent, it could be noticed that the size distribution of MWCNTs with the diameter 11-20 nm (Figure 4). However it was concluded that the purified MWCNTs has the average diameter of 15 nm with highest particle size distribution.

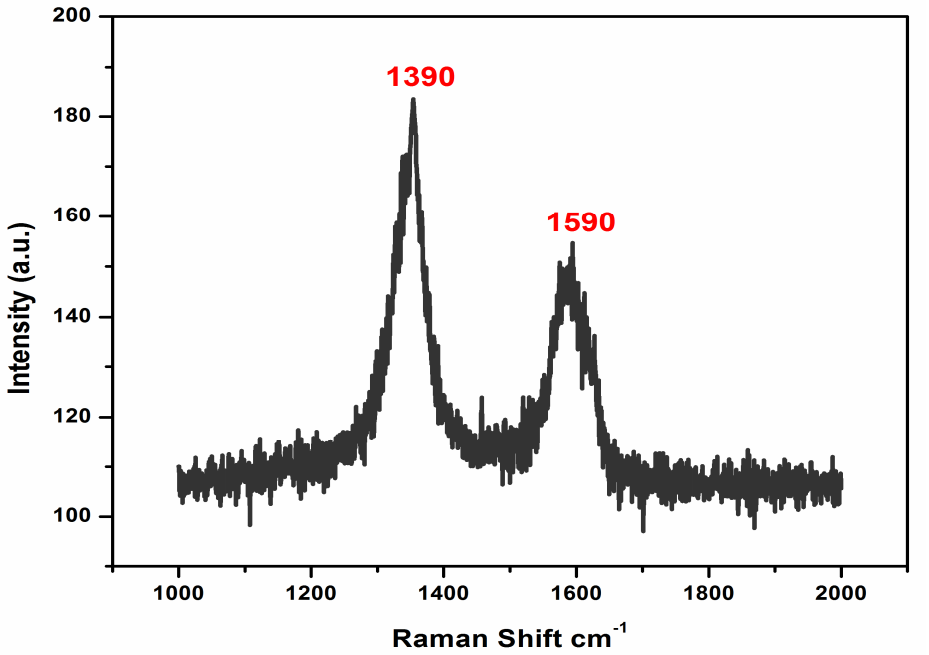

Figure 5. Raman spectra of Carbon nanotubes

Raman spectroscopy is an important tool for studying CNTs, which provides information about the crystal structure and the presence of the disorder in the sample. Raman spectra also provide the information about the removal of the amorphous and

structural disorders from CNTs after purification. Raman spectra of CNTs are based on phonon dispersion relationship of two-dimensional graphite, and the method of folding of the Brillouin zone. The Figure 5 of Raman spectra explain the presence of G-bands (at about 1590cm^{-1}) and D-bands (at about 1390 cm^{-1}) with different D/G ratio. The G-band revealed the vibration of carbon atoms in planar (sp^2) hexagonal lattice, indicates the presence of the graphitic mode (12), associated with nanotubes presence, while the D-band shows the presence of defects (amorphous, disorder carbon with fullerene etc). For the material synthesized from polystyrene, the D/G ratio was 0.8678.

IV. CONCLUSION

The above synthetic method can be considered to be a simple procedure and have an ability to produce CNTs on by hydrothermal method. The purification step was important and challenging to get good yield. The characterizations were carried out in different advanced instruments, which were basic tools for the carbon nanomaterials. The FESEM images of carbon nanotubes were taken at different magnifications to get good images. The Raman spectroscopy shows the better D/G ratio which was very important for CNTs.

V. ACKNOWLEDGEMENTS

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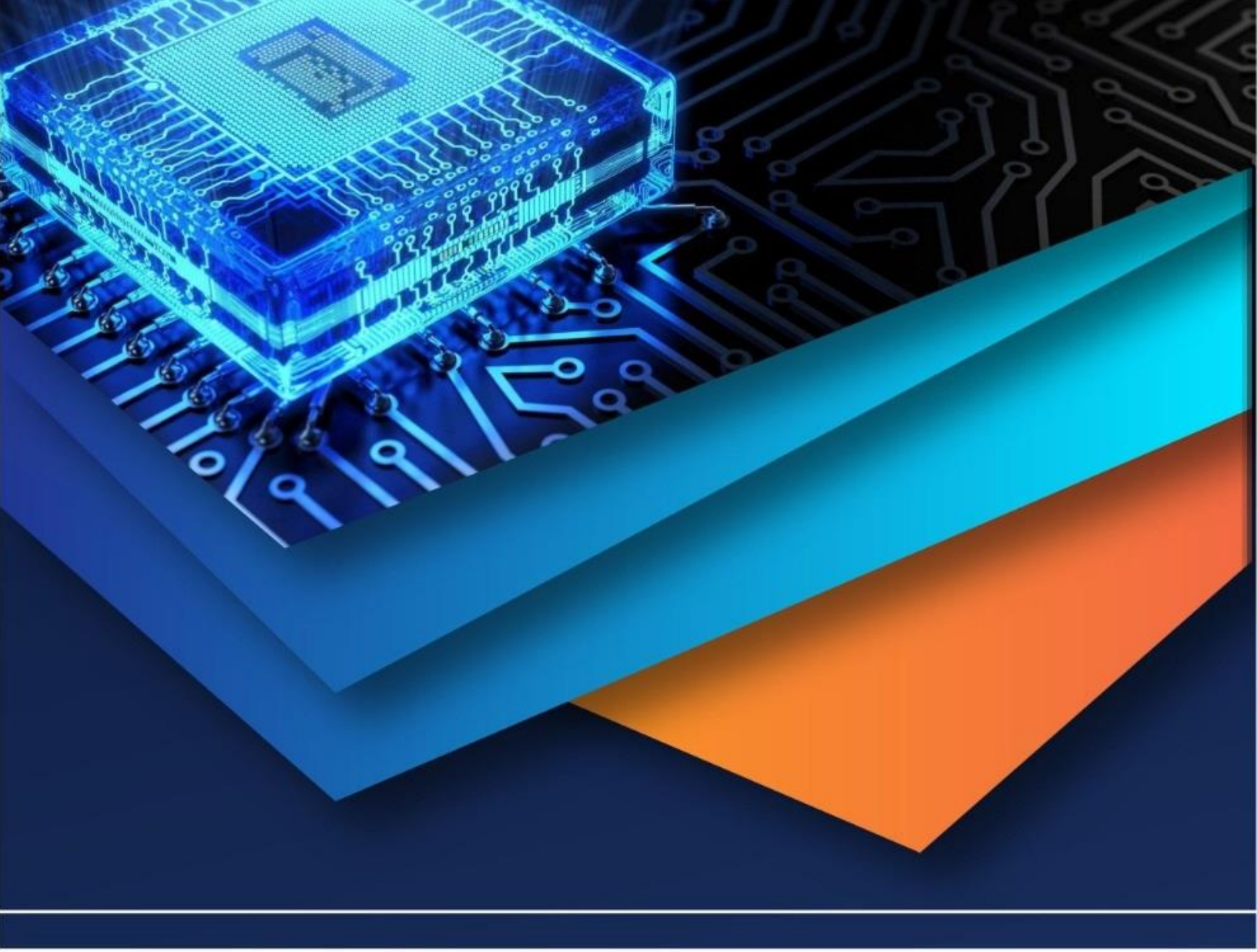

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