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Comparative Study of Synthesize of AG NP by Chemical and Green Method/Route

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Abstract: ***The particles with a diameter of 1-100 nm are called nanoparticles. They have unique properties due to their dimensions and high surface area. As a result of this characteristic, they are a suitable candidate for catalysis, imaging, medical applications, energy-based research, and environmental applications. In our work, silver nanoparticles (AgNP) were synthesized by two different routes, i.e., chemical and green routes. The chemical method synthesized AgNP from AgNO3 and NaBH4 as reducing agents. While for eco-friendly and non-hazardous green synthesis, fruit extract of Phyllanthus embolic- amla) was utilized. In this method, AgNP was synthesized with the help of silver nitrate precursors and an extract of Phyllanthus embolic, which acts as a reducing and stabilizing agent. Characterization results of both synthesized AgNP were compared using SEM, FTIR and XRD. Upon reaching the shape and size of AgNP with the chemical method, it was suggested that Phyllanthus emblica acts as a reducing agent for the synthesis of silver nanoparticles.***
Keywords: Silver Nanoparticles (Ag NP), Chemical synthesize, Green synthesize, Phyllanthus Emblica.

I. INTRODUCTION

With a view to special properties like high surface area, better reactivity, and enhanced catalytic property, nanoparticles have covered a vast area in their application. Nanomaterials exhibit altered physical, chemical and biological properties, which adds to their utilization in varied fields. Nanoparticle has found their application starting from waste treatment, diagnostic, antimicrobial and antibacterial agents, drug deliveries, electronics, bio-sensors, catalytic reaction, medicinal purpose, material science etc. It has been known that the ruby red hue of some historical glass paintings is the result of silver and gold nanoparticles. Metallic nanoparticles are so arranged in such a way that they artistically scatter light to give the material's immensely beautiful color-enhancing optical properties. In addition, carbon black was added during tyre manufacture to improve the life span of tyres (Matsuyama et al., 2019).

Some materials have a higher surface-to-volume ratio, which increases their efficiency as a catalyst. Surface area for any material is crucial; smaller particles react more quickly as more reaction sites are available. A material being bulk or in nano size depends upon its surface properties.

Physical, chemical and biological approaches can synthesize nanoparticles. However, the chemical procedure is preferred as it requires a short time to synthesize many nanoparticles. During chemical treatment, certain precursors are necessary for synthesizing in addition to a capping agent, which is used for stabilization. These chemicals, as well as limiting agents, may be expensive and toxic chemicals (Kadam et al., 2019). Therefore, there comes the need to go with certain eco-friendly routes (Ghoshal & Singh, 2022) (Zhang, 2016).

Nature is a storehouse of untreated extracted plants with unknown components, which gives way to a green chemistry approach to synthesizing nanoparticles. Literature showed that there had been certain routes that produce eco-friendly nanoparticles by a biological method where fungi, microorganisms and plants are used where it had been found that better result was shown by plant-based technique. Further, it is also an efficient technique because it removes the cost of isolating microorganisms (Ghoshal & Singh, 2022)

Different nanoparticles have been synthesized and utilized on the basis of their applications, such as Gold, aluminum, zinc, copper, iron, silver, etc. out of these; silver has frequently been used in electronic devices, water treatment, antimicrobial agents in wound dressings, bioengineering, anticancer/antifungal agents and many others because of its electrical conductivity, chemical stability, catalytic activity biological activity, physiochemical activity and antimicrobial activity (Dasaradhudu & Srinivasan, 2020).

In this work, we have synthesized silver nanoparticles by chemical and green routes. For the chemical route, $AgNO_3$ was used as a precursor, while for green synthesis, Indian gooseberry was used as a precursor. Necessary treatment was done for both cases and synthesized AgNP for both cases was characterized by FTIR, SEM and EDX to verify efficient synthesis by green route, which has been discussed further in the discussion.

II. EXPERIMENTAL PROCEDURE

Phyllanthus Emblica was purchased from the local market in Vadodara, Gujarat. Silver nitrate was used as a precursor purchased from Sulab Chemicals. Extract and solution was prepared with deionized (DI) water from DI plant at the university.

To prepare fruit extract for green synthesis, the fruit was thoroughly washed and cut into 2-3 mm- sized cubes. The rough-cut pieces were washed with distilled water again, and they were then left to dry for between 24 to 48 hours. The dried pieces were weighed first, then 20 g of it, along with 70 % of methanol, were added to a beaker. This mixture was then brought to a boil and stirred magnetically at 250 to 300 rpm for about an hour. After being cooled to room temperature and filtered using Wittman filter paper, the extractant was kept at a temperature of 4-6 °C to be used as a reducing agent (Dhar et al., 2021). AgNPs were produced by mixing 20 ml of amla fruit extract and 180 ml of a solution containing 1 mM silver nitrate ($AgNO_3$). The mixture was heated to 65°-68°C and stirred continuously for about an hour. After that, a 24-hour incubation period was maintained. Followed by the incubation period, the color changed to a dark brown. A change in the color of the solution indicates the formation of AgNPs. The solution was centrifuged at 4000 rpm for an hour to separate the pure AgNPs from the mixture. The precipitates were then combined once more with. We kept the dried nanoparticles for additional characterizations.

The chemical synthesis was performed using a 250 ml conical flask containing 200 ml of a 0.02 M sodium borohydride ($NaBH_4$) solution and a 50 ml burette having a 0.01 mM silver nitrate ($AgNO_3$) solution in a beaker with ice cubes in an ice bath. The conical flask containing the $NaBH_4$. The solution is immersed in the ice bath for 25 to 30 minutes. Place the magnetic bead inside the conical flask in the center of the magnetic stirrer. A 700-900 rpm setting should be used for the magnetic stirrer. Stop stirring and remove the stir bar once the silver nitrate ($AgNO_3$) solution has been incorporated. After 2ml of $AgNO_3$ is added, the solution should turn dark brown. Maintain it for 24 hours in the incubator. The water should be drained from the beaker after the tiny particles have collected, and the particles should then be dried. The smaller silver nanoparticles are preserved for later characterization (Mughal & Hassan, 2022).

Because reactivity increases with temperature, the possibility of a reaction between nitrate ions and NaBH4 at higher temperatures leads to the need for extra ice. In addition, hot water will result in the formation of hydrogen and borax, which will reduce silver ions rather than borohydride ions. Cooling is required to slow the reaction to an appropriate rate and stop secondary reactions. A complete reaction is produced when sodium borohydride, also known as (NaBH4), is used, producing finer silver nanoparticles. To stabilize the silver (Ag) nanoparticles, excessive amounts of sodium borohydride ($NaBH_4$) are used.

III. CHARACTERIZATION TECHNIQUE

1) *X-Ray Diffraction:* An x-ray diffractometer (D8 DISCOVER – Bruker) employed at Central Instrumentation Facility, Indian Institute of Technology Gandhinagar, was used to check for the crystallographic structure of synthesized AgNP. CuKα radiation (k = 1.5406 A°) was used for x-ray generation. The range of scanning was kept from 20° to 80°.
2) *SEM:* For surface topography, synthesized AgNP were scanned in an electron field microscope (JEOL JSM-7900F) employed at Central Instrumentation Facility, Indian Institute of Technology Gandhinagar. The nanoparticles were placed in conducive carbon tape and that tap was fixed in a copper stub.
3) *FTIR:* Fourier transformed infrared technique was used to identify the functional present in a synthesized sample. FTIR instrument was available at Parul Institute of Pharmacy and Research, Parul University, was used for testing the synthesized samples.

IV. RESULT AND DISCUSSION

A. FTIR

FTIR spectra of silver nanoparticles for green and chemical methods as shown in Fig:4-1 and Fig:4-2 are in the 3500-4000 cm^{-1} corresponding to H-bonded alcohols stretched O-H and phenols. The peak at 1573-1685 cm^{-1} displays the C-H bond in the range of 1318-1573 $cm^{-1,}$ showing a stretch of N-H bond, while a stretch of AgNP was observed in the range of 500 – 1000 cm^{-1}. Consequently, created nanoparticles were surrounded by metabolites and protein functional groups, like those found in terpenoids (Hussain et al., 2019). From the FTIR result, we concluded that the carbonyl group from amino acids residue and protein have a strong affinity for metal, suggesting that protein may act as a cap for metal nanoparticle like silver to prevent agglomeration and stabilizes them. This also signifies that, from green synthesis, AgNP are formed in an aqueous medium and stabilized simultaneously. It was demonstrated by the carbonyl group that terpenoids or flavanols were absorbed on the surface of metal nanoparticles. In the absence of other powerful ligating agents in sufficient concentration, flavanones or terpenoids might interact with the carbonyl group to adsorb on the surface of the metal.

The reduction of metal ions and formation of metal nanoparticles may have been caused by the presence of sugar in the solution. It's possible that terpenoids contribute for the reduction of the metal ion by converting molecules from aldehydic group to carboxylic acids. Once various Phyllanthus extracts have been identified, separated and individually assessed for reduction of metal ions, then the issue can be resolved (Dhar et al., 2021).

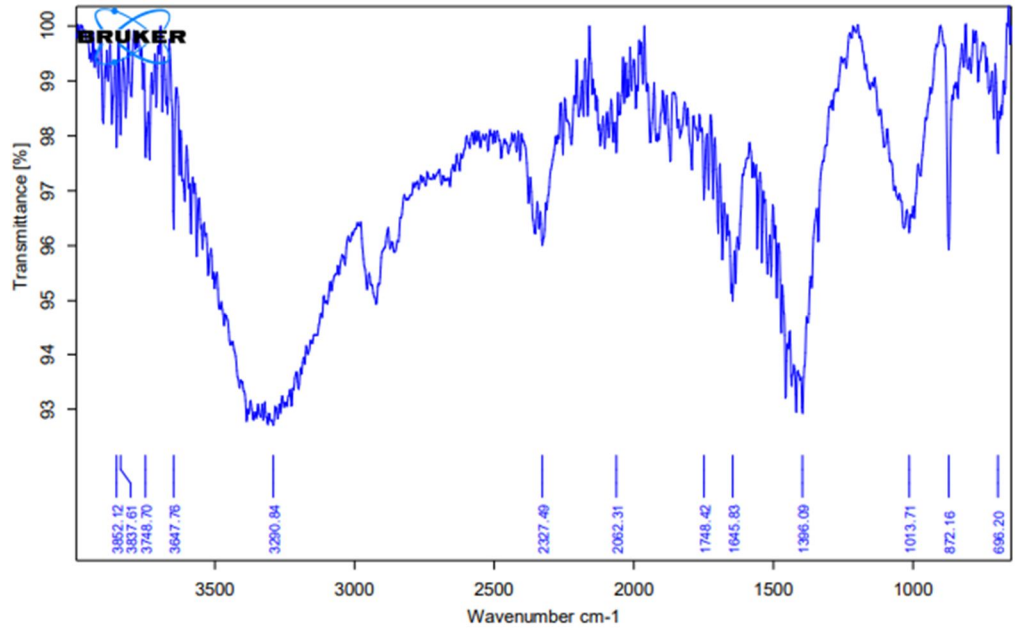

Fig. 1 FTIR spectra for AG NP from green route

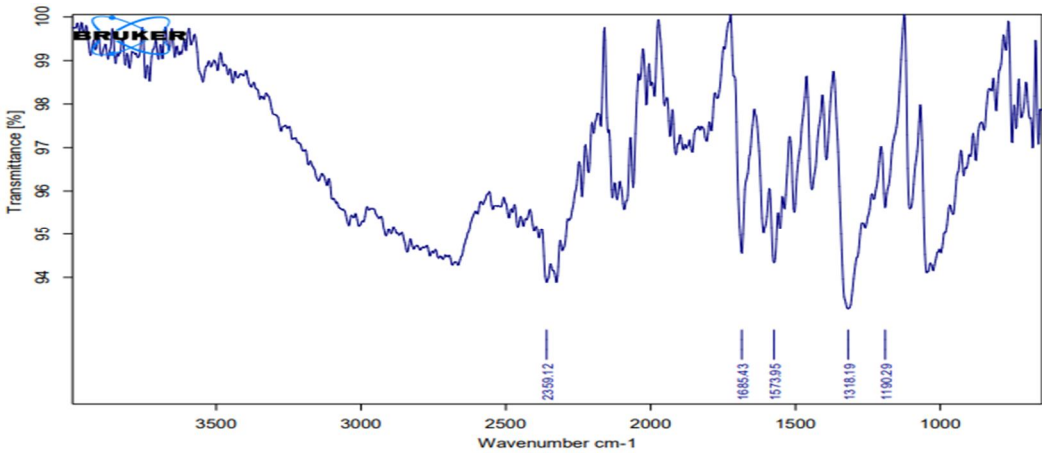

Fig. 2 FTIR spectra for Ag NP from Chemical route

B. 4.2 XRD

XRD spectra in Fig:4-3 confirms the crystalline property of the synthesized sample. Diffraction peak around 38°, 44°, 64°, 77° was visible in the XRD spectrum, which is indexed by the cubic face-centered silver. Possible causes for these sharp Bragg peaks include capping agents stabilizing the AgNP. Strong X-ray scattering centers are thought to be in the crystalline phase and may be caused by capping agents, according to intense Bragg reflections. The centrifugation and redispersing of the pellet in distilled water after nanoparticle formation as a part of the purification process ruled out independent crystallization of the capping agents (Youssef et al., 2014). As a result, XRD results indicated that the organic phase (Green synthesis) and chemical phase (chemical synthesis) crystallize on the surface of the silver nanoparticles or vice versa. In most cases, particle size effects are to blame for the broadening of peaks in solids XRD patterns. Broader peaks reflect the effects of the experimental conditions on the nucleation and growth of the crystal nuclei and indicate smaller particle sizes.

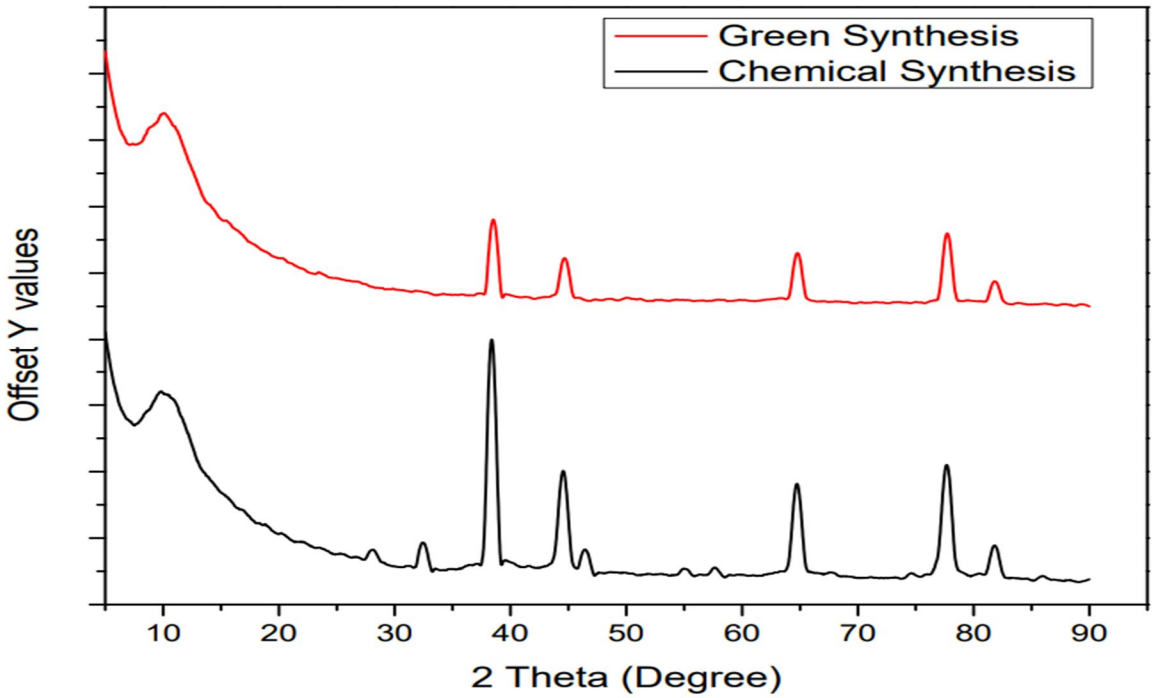

Fig. 3 XRD spectra for combined Ag NP

C. 4.3 SEM and EDX

The images obtained using a scanning Electron Microscope for various magnification factors are shown below Fig 4.4. From the images obtained from the scanning electron microscope, the particle size is in the range of 6-8 nm; this variation in the size is because of the agglomeration of silver nanoparticles. Synthesized microstructures are spherical in shape. In image Fig4-4 (b) shows SEM images of AG NP synthesized from green route. Here size of Ag NP are varying from 5–60 nm. This variation is significant in comparison to chemical method. Size variation can be controlled efficient by controlling rate of reduction with capping agent.

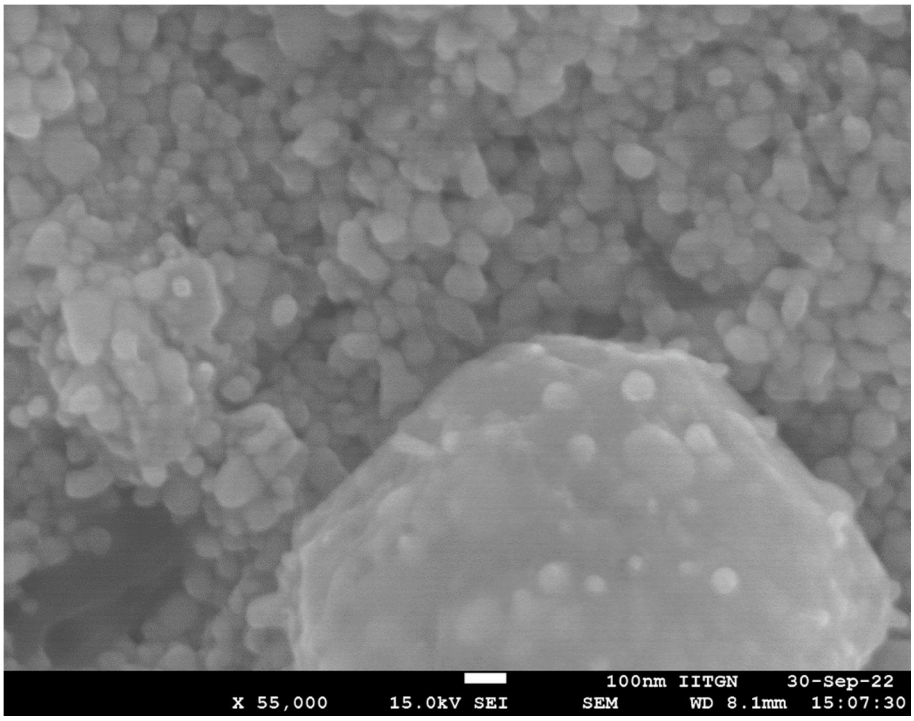

Fig. 4 SEM image of AG NP synthesized by Green route

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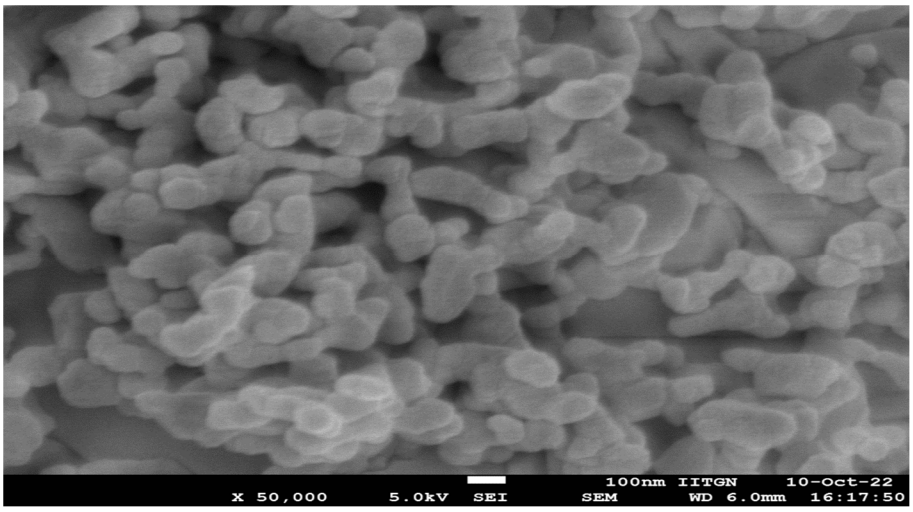

Fig. 5 SEM image of Ag NP synthesized by Chemical Route

EDX is one of the techniques used to identify elemental composition in synthesized samples. The reduced silver nanoparticles were subjected to EDX analysis with an optical absorption characteristic peak at 3 keV. Based on the results obtained as shown in Fig: 4-5 and Fig:4-6, it can be confirmed that silver has been synthesized by chemical and green routes. However, the minor peak of Si and Cl have been observed with green synthesis. That's can be because of incomplete reduction of Phyllanthus Emblica to Ag (Dhar et al., 2021).

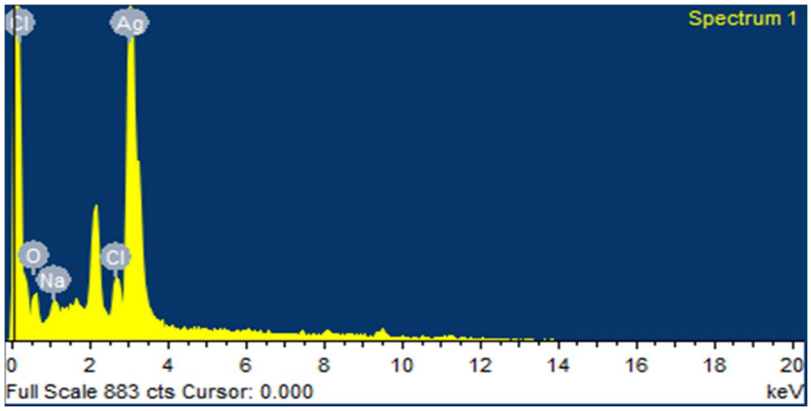

Fig. 6 EDX image of Ag NP synthesised by Green route

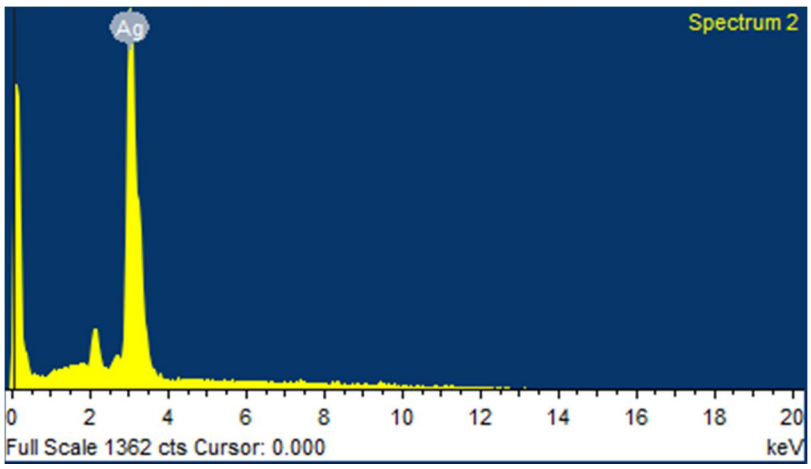

Fig. 7 EDX image of Ag NP synthesized by Chemical route

V. CONCLUSION

For the green synthesis we have use the plant extract of Phyllanthus emblica plant because it is simple, easy to use and offers a benign nanoparticle. While for the chemical synthesis, we have used the chemicals like $NaBH_4$ and $AgNO_3$.The size range for the green method ranged from 100 nm-100 µm, while for the chemical method, 100 nm to 100 µm. The size of the nanoparticles was bigger in chemical synthesis compared to the green method. The structure of the nanoparticles was seen clearer in chemical synthesis than in green synthesis using SEM analysis. From the FTIR analysis, we can conclude that the green synthesis peaks were broad compared to the chemical synthesis. From XRD analysis, it can be concluded that the peaks were sharp in the chemical method rather than in the green method. From the EDX analysis composition of other elements present in green synthesis are (Si, Cl) apart from Ag and in chemical synthesis, only Ag was present was noticed.

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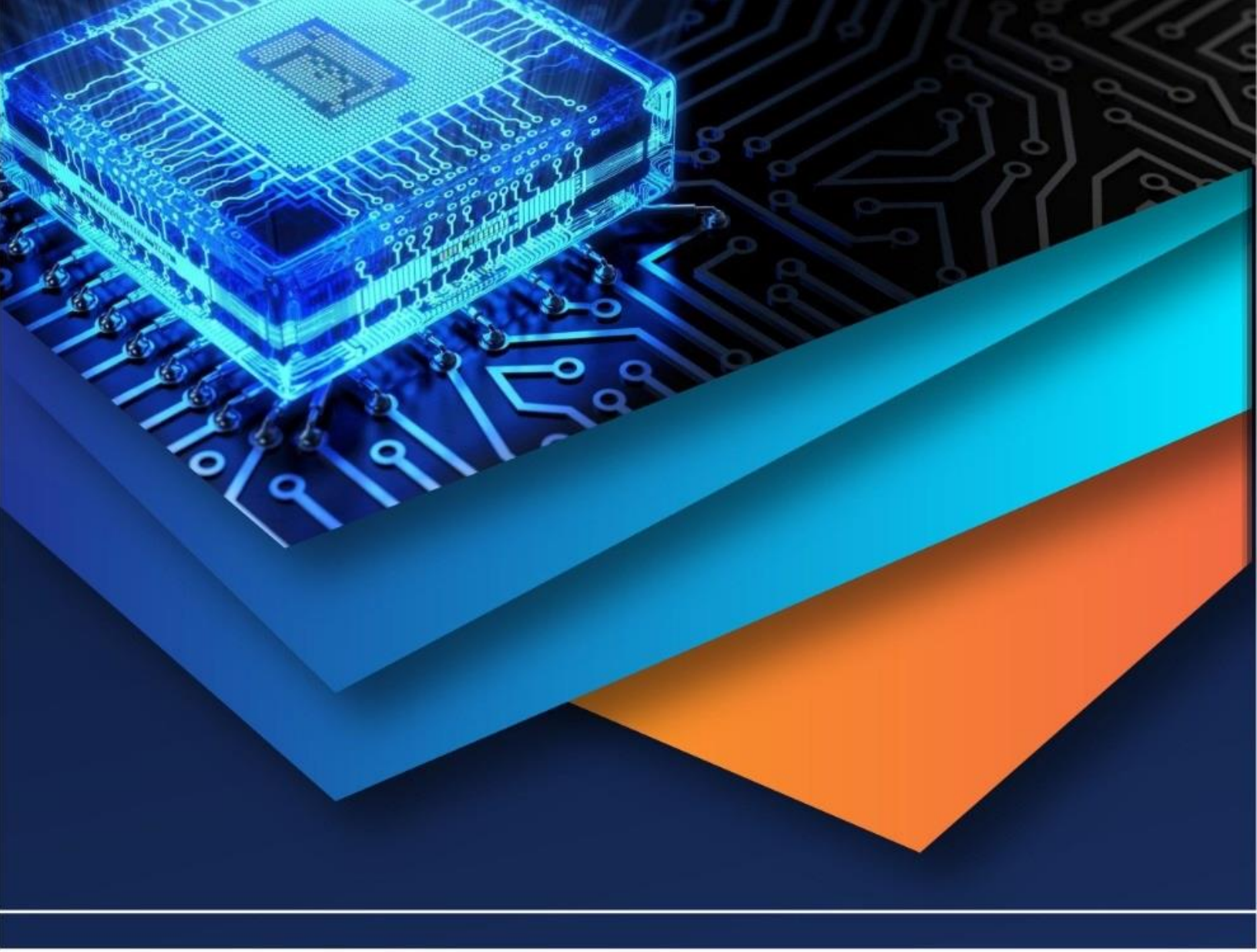

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