iJRASET
International Journal For Research in
Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 11 **Issue:** XII **Month of publication:** December 2023

DOI: https://doi.org/10.22214/ijraset.2023.57499

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
Volume 11 Issue XII Dec 2023- Available at www.ijraset.com

Bimetal Enriched Carbon Nanomaterials Synthesized from *Bagasse*- A Plant Waste Material as an Exceptional Adsorbent for Hydrogen Gas

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***Abstract:** In this study, the process of pyrolysis has been depicted for the synthesis of CNMs from the Bagasse-a plant waste materials at high temperature of 750°C in an inert atmosphere. The Taguchi optimization methodology is utilized to optimize the parameters for synthesis process. The adsorption of hydrogen was investigated using Sievert's apparatus. The SEM image demonstrates the bimetal enriched CNMs and HRTEM reveals the CNMs obtained were in cylindrical form as well as transparent sheet. The presence of various bimetal combination is confirmed by EDAX. The spectroscopic studies XRD and Raman reveals the graphitic nature of the CNMs. 4.96 weight percent hydrogen adsorption was demonstrated by CNMs synthesized from bagasse under the parameters predicted by the Taguchi optimization methodology.*

***Keywords:** Carbon nanomaterials, Bagasse, Taguchi optimization, Bimetal enriched, plant waste materials, Hydrogen adsorption, Sievert's apparatus.*

I. INTRODUCTION

The need to find new, efficient energy sources has grown in recent years due to an energy shortage brought on by the depletion of energy supplies and environmental catastrophe. Hydrogen is a perfect solution for energy converters due to its high efficiency and significant contribution to reducing air pollution. Hydrogen an energy carrier that may be used to provide valuable thermal energy used in a variety of practical scientific applications without disrupting the environment or natural cycles[1][2][3][4].Several distinct hydrogen storage systems, including liquefied hydrogen, compressed hydrogen, metal hydrides, and hydrogen physi-sorption on various substrates, including graphene materials and carbon nanomaterials (CNMs), have been proposed in recent years. [5][6][7][8][9].

Carbon materials is extremely encouraging among the numerous methods for storing hydrogen that have been researched thus far, since there is no chemical interaction between hydrogen and the surface of the materials utilized, providing completely reversible hydrogen and desorption[10][11][12][13]. Adsorption under encompassing circumstances on materials with uniform Van der Waals connections is believed to be able to meet the standards specified by the US Department of Energy (DOE) [14][15][16]. At room temperature, storing liquid hydrogen presents an explosion danger and is expensive. Metal hydride allows the ability to store hydrogen, but their substantial weight and inherently poor heat conductivity make the system uneconomical. A method of storing hydrogen using porous carbon materials has been developed to address these problems. [17][18][19][20][21][22]. Recently, both the academic and industrial sectors have paid close attention to carbon nanomaterials (CNMs), which have a large surface area, distinctive physical, mechanical, inherent high-aspect-ratio, hollow nano-geometry, among others [23][24][25][26][27]. Due to the physisorption and chemisorption processes involved, these materials have different hydrogen adsorption capabilities and inherent constraints [28][29][30][31]. Owing to the hydrogen spill-over effect, attaching or enriching CNMs with metal nanoparticles increases their capacity for storing hydrogen [32][33][34][35][36][37][38]. Nickel is a transition metal that is particularly promising for increasing hydrogen storage capacity since it is abundant and affordable compared to noble metals and is known metal for hydrogen catalysis [39], [40][41].

II. TAGUCHI METHODOLOGY

The goal of the current work was to standardize the pyrolysis of CNMs employing a horizontal furnace setup and plant waste materials (Bagasse) as precursors. This endeavour required a great deal of experimentation due to the consideration of several parameters and their variations. The Taguchi optimization approach was applied to tackle this issue. To obtain an understanding of the ideal conditions for obtaining the intended material, a large number of experiments have to be performed when four variable factors at three levels are to be employed.

In contrast, the Taguchi optimization technique requires an only a few experiments to be performed to obtain the same knowledge. The Taguchi optimization technique's specifics are covered separately in the following references-[42] [43] [44] [45] [46] [47].

III. EXPERIMENTAL TECHNIQUES

A. Synthesis of Carbon nanomaterials (CNMs) from Bagasse- a Plant Waste Material

The bagasse (*Saccharum officinarum*) fibers that were extracted from the plant waste materials were carbonized at 750°C in an inert atmosphere using a Lindberg horizontal quartz tube furnace. The carbon materials obtained were activated using various concentrations of alkali solutions as mentioned in Table 1. The activated carbons were loaded with Ni-Li, Ni-Mg and Ni-Al nanoparticles combinations. The resulting CNMs were then used to investigate the properties of hydrogen adsorption using Sievert's apparatus.

B. Determination of Hydrogen Adsorption

For the investigation 10 grams (approx.) of CNMs were utilized. Using Sievert's apparatus and the static volumetric technique, the hydrogen adsorption isotherms were measured at room temperature. Mukherjee et al. [40], [48] ,[49] have used Sievert's apparatus to study hydrogen adsorption on CNMs at a pressure about 60 bar.

TABLE 1: HYDROGEN ADSORPTION BY BIMETAL ENRICHED CNMs
(TAGUCHI ORTHOGONAL TABLE)

Experiments	Alkali-treatment	Bimetal loaded	Duration of annealing (Hr.)	Temperature (°C)	ADS wt.%
L1	NaOH	Ni-Li	2	600	3.0879
L2	NaOH	Ni-Mg	2.5	700	2.2633
L3	NaOH	Ni-Al	3	800	3.1768
L4	Na_2CO_3	Ni-Li	2.5	800	3.2561
L5	Na_2CO_3	Ni-Mg	3	600	3.1473
L6	Na_2CO_3	Ni-Al	2	700	3.2516
L7	**K_2CO_3**	**Ni-Li**	**3**	**700**	**4.9295**
L8	K_2CO_3	Ni-Mg	2	800	4.8368
L9	K_2CO_3	Ni-Al	2.5	600	3.6397

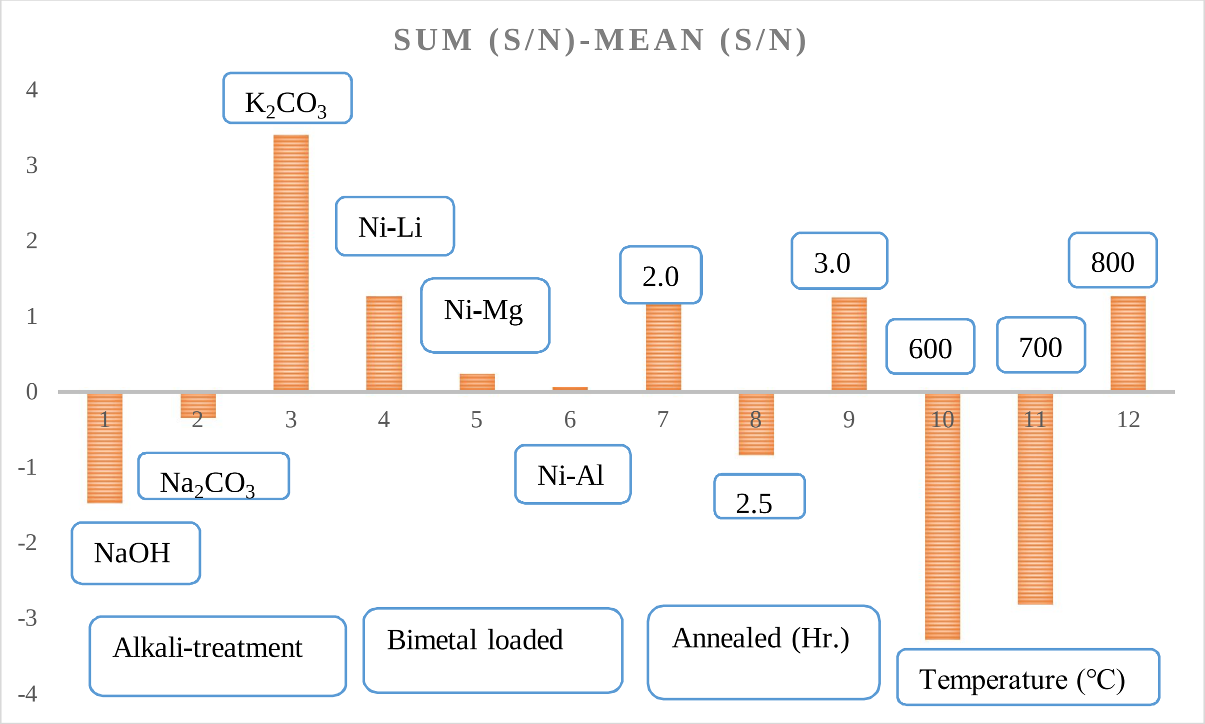

Fig.1 Histogram of sum (S/N) – mean (S/N) for synthesis of CNMs

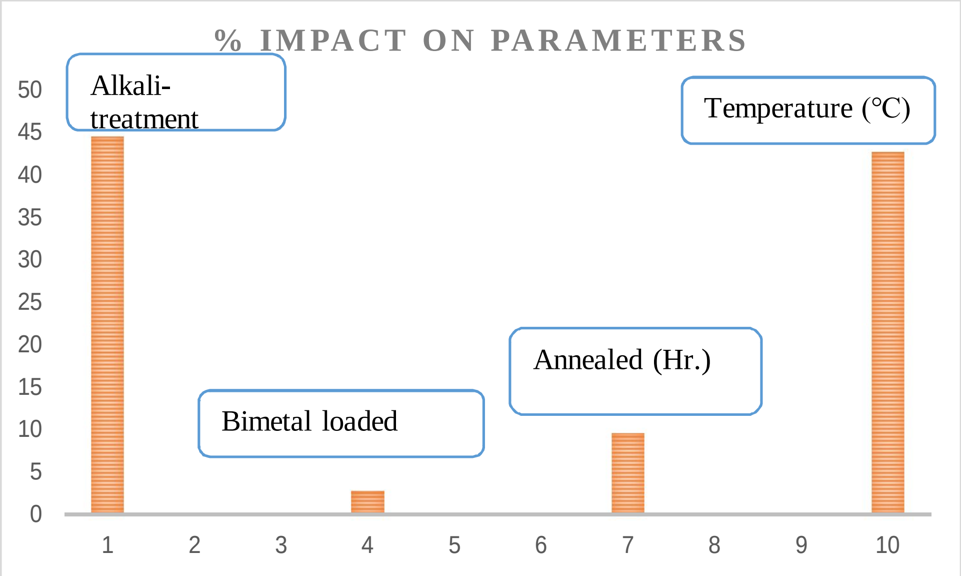

Fig.2 Histogram presenting % impact of different parameters on hydrogen adsorption by CNMs

The above histogram shows that the best results for hydrogen adsorption could be obtained when the bagasse is treated with K_2CO_3 loaded with bimetal Ni-Li combination and annealing for 3 hours at a temperature of 800 ℃. These conditions are comparable to those of L7, with the exception that temperature is required at 800 ℃ as opposed to 700 ℃ (Table 1). This is actually the benefit of Taguchi Optimization—it can forecast the optimal outcome even when the experiment may not have been conducted in that manner. To determine which factor or factors need to be monitored for better results, a histogram of the % impact of various parameters was also plotted (Fig.2). It is clear from the histogram (Fig.2) the percentage impact of the parameters that using alkali K_2CO_3 is the most crucial step in obtaining the carbon with the highest hydrogen adsorption capacity (44.64%). Additionally, the impact of bimetal is minimal with 3.90%.

The duration of annealing has a percentage effect of 2.89% whereas the temperature has the percentage effect of 42.79% on the sample. This suggests that a different temperature range needs to be investigated. Figure 1 suggests that the temperature to be at 800 °C, but L7 demonstrates that carbon synthesized at 700 °C has the maximum hydrogen adsorption capacity. To determine the optimal pyrolysis, it is evident that a thorough investigation of the effects of temperature between 750 and 850 °C is necessary.

C. Validation of Best Parameter Projected by Taguchi Optimization Methodology

It was necessary to conduct the experiments under the conditions predicted by the Taguchi optimization methodology because the first set of experiments (Table 1) did not follow the experimental conditions predicted by the Taguchi optimization methodology. The extension of above Taguchi (Table 1) was built in the manner indicated in following Table 2. The annealing process was run for 2.5 and 3.0 hours at a temperature between 750 and 850°C. The CNMs prepared from bagasse sample was given an alkali treatment using K_2CO_3 and loading of Ni-Li combination as it was the conditions predicted by the Taguchi Optimization methodology which revealed the highest observed hydrogen adsorption (Table 2). The maximum adsorption under these circumstances is 4.96 weight percent, surpassing the results of all L9 experiments (Table 1, L7: 4.92 weight percent).

Table 2: Outcomes of extension for Taguchi (Table 1)

Sr. No.	Duration of annealing (Hr.)	Temperature (°C)	ADS wt.%
1	2.5	750	4.1952
2	3	750	4.8570
3	2.5	800	3.8353
4	3	800	4.9634
5	2.5	850	3.7966
6	3	850	3.6646

IV. RESULTS AND DISCUSSIONS

A. Characterisation of CNMs

Using a Scanning Electron Microscopy (SEM) image, the microstructure and morphology of the carbon nanomaterial were examined[50] [51] [52][53][54]. The SEM image demonstrates the bimetal enriched CNMs made from plant waste materials. The CNMs are porous and have an average pores size between 35 and 60 nm [Fig.3 (a)]. Typically, amorphous carbon and carbon nanofibers with a disordered structure are combined to create the CNM [55] [Fig.3(b)].

The microscopic techniques to study the structure and the properties of the CNMs, the HRTEM was similarly used for the visualization of the samples[56] [57]. HRTEM reveals the CNMs obtained are in cylindrical form [Fig.3 (c)] as well as transparent sheet type [Fig.3 (d)]. The TEM image correspondingly demonstrates the bimetal enriched CNMs uniformly distributed in the diameter ranging from 15nm to 20nm [Fig.3 (c) and (d)]. The surface area was analyzed by BET (Brunauer-Emmett-Teller) [58] [59] and was found to be 517.875 m^2g^- [Fig.5 (a)] and 0.220 cc/g [Fig.5 (b)] as a pore volume of the CNMs.

B. Spectroscopic Studies

X-Ray Diffraction (XRD) techniques [60], [61] [61] [62] [63] are used to analyze the final CNMs that are obtained after annealing [Fig. 4 (a)]. Two theta values were obtained: one sharp peak at 26.21, corresponding to the (002) plane along with 43.74 and 44.28 corresponding to the (100) plane which suggest graphitic and partial crystalline nature of carbon nano materials.

Crystallographic orientation of the CNMs was also studied using Raman spectroscopy [64] [65] [66] [67] [68] [69] [70]. A *Raman spectrum* [Fig.4 (b)] displays one peak at 2594 cm^{-1} and another at 3180 cm^{-1} which states the second order D-band, also known as the 2D-band, is dependent on the packing in three-dimensional space, also is associated with the boundary point K in the graphite's Brillouin zone that divulges the graphitic nature of the materials having disordered carbon[71] . Furthermore, the peaks at 2594 cm^{-1} result from the CH_2 group's symmetric and asymmetric C–H stretching vibrations and the peak at 3180 cm^{-1} is produced due to one phonon lattice vibrational process.

Energy- dispersive X-ray analysis (EDAX) of samples [Fig.6 (a) (b) (c)] indicates the various bimetal combinations utilized for the enrichment of the CNMs.

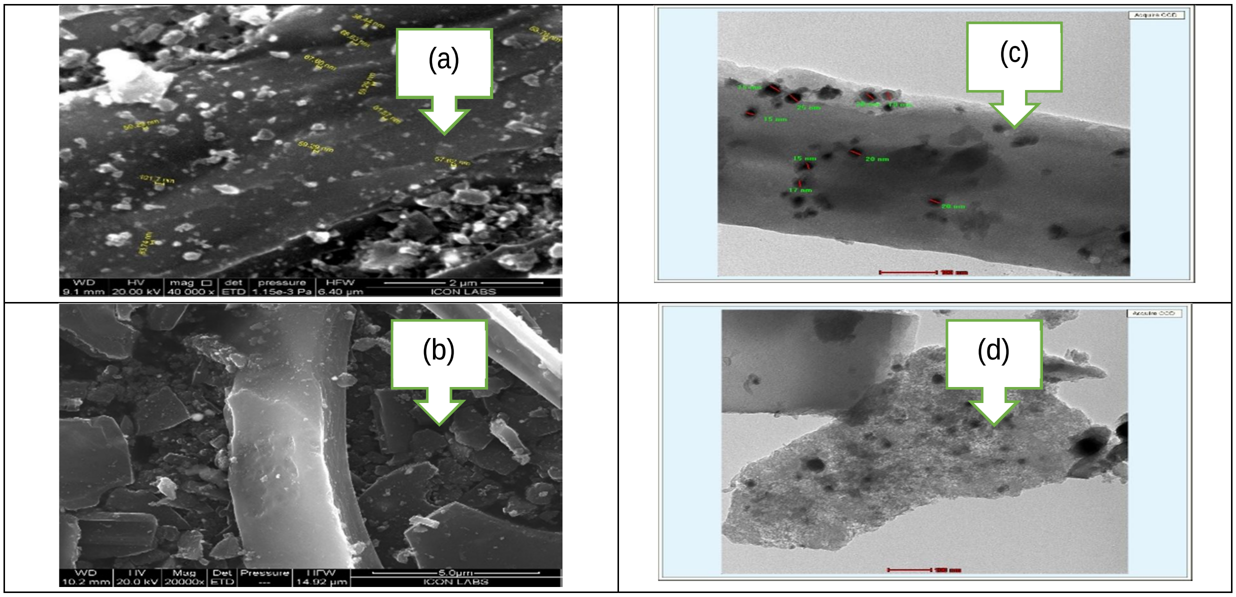

Fig.3 CNMs obtained by pyrolysis of bagasse-a plant waste materials [(a) and (b) Scanning Electron Microscopy (SEM) of bimetal enriched samples]; (c) and (d) High-Resolution Transmission Electron Microscopy (HRTEM) of cylindrical and transparent sheet of CNMs.

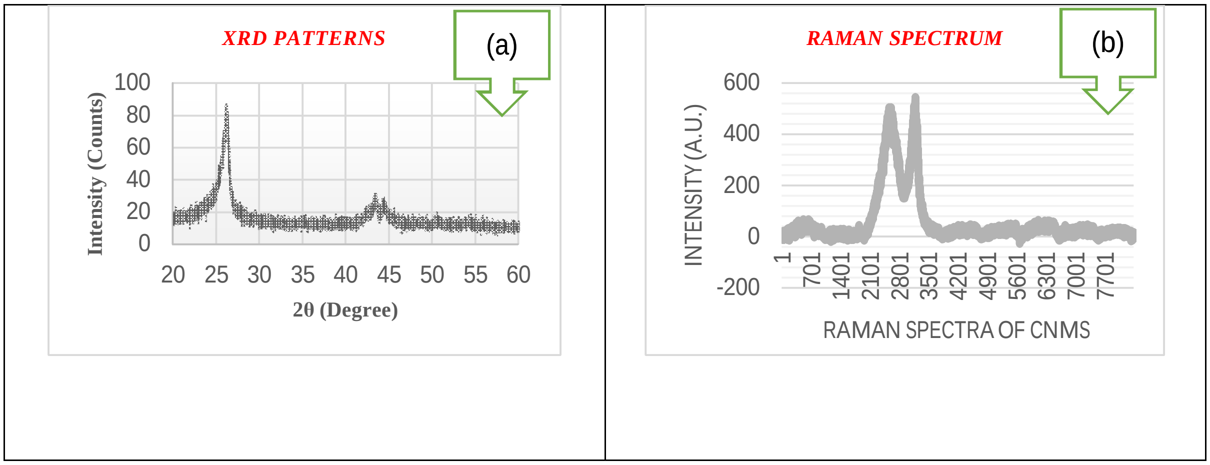

Fig.4 Spectroscopic studies

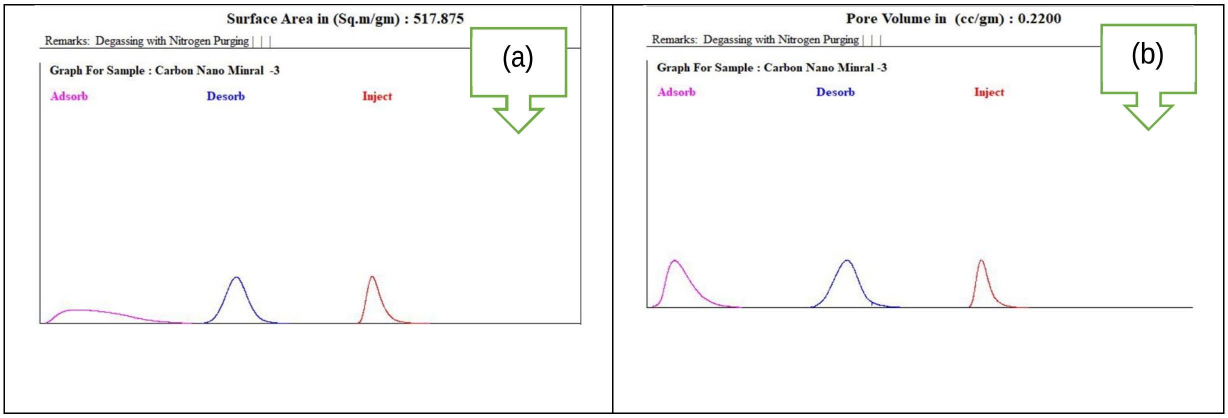

Fig.5 BET of CNMs

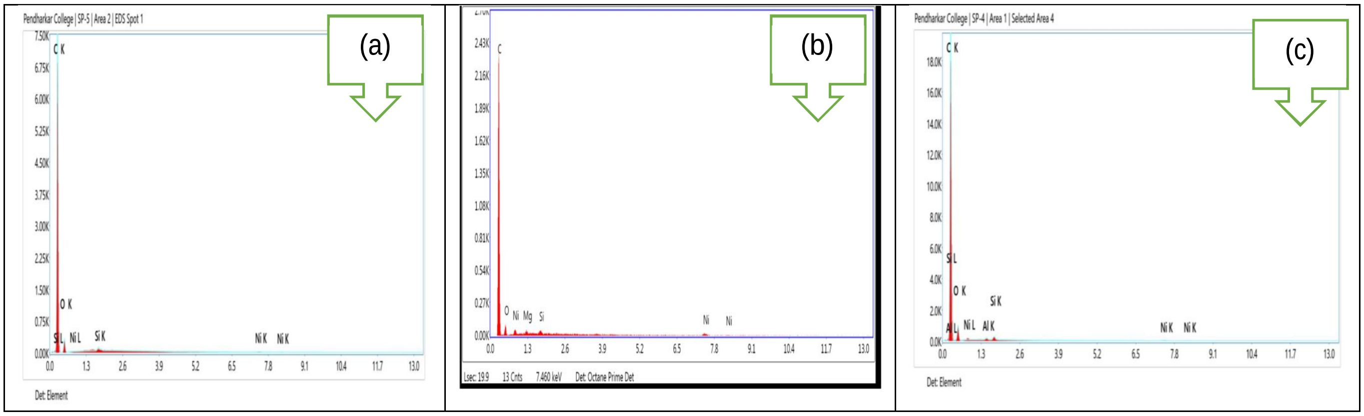

Fig.6 EDAX of CNMs

V. CONCLUSION

The maximum hydrogen adsorption capacity of 4.96 weight percent was achieved when pyrolyzed bagasse sample was given alkali treatment using K_2CO_3 followed by bimetal loading of Ni-Li combination annealed for 3 hours at a temperature of 800 ℃ as predicted by Taguchi optimization methodology. The CNMs thus obtained by above mentioned process is found to be cylindrical as well as transparent with graphitic properties.

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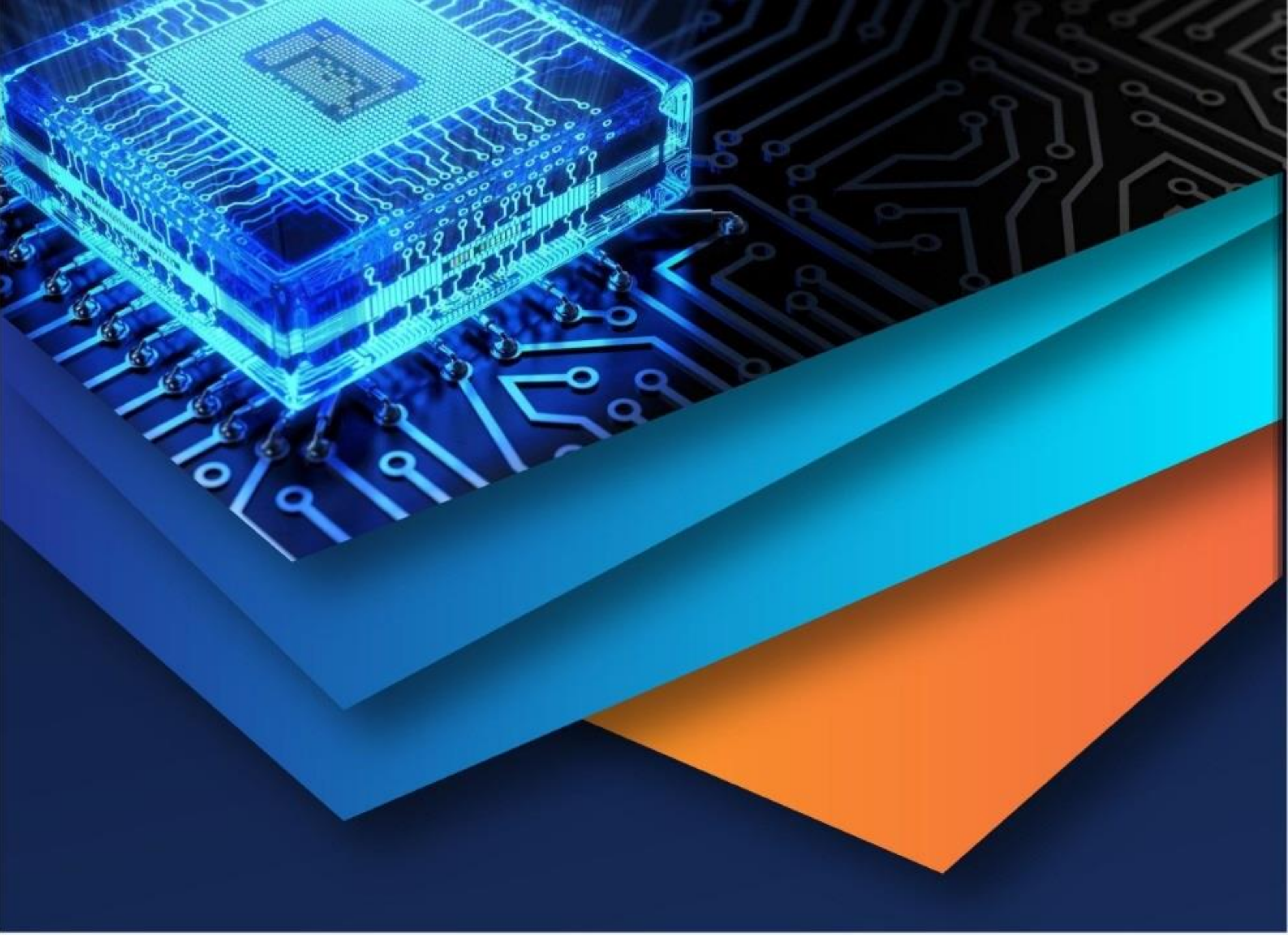

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