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Cuminum Cyminum (Jeeru) Seeds Extract as Eco-Friendly Corrosion Inhibitor for Aluminium in HCl Solution

R. T. Vashi[1], N. I. Prajapati[2]
[1]Associate Professor, Department of Chemistry, Navyug Science College, Rander road, Surat, India.
[2]Research Scholar, C. B. Patel Computer College & J. N. M. Patel Science College, Surat, Gujarat, India.

Abstract: The inhibition of the corrosion of aluminium in hydrochloric acid solution by Cuminum cyminum (Jeera) seeds extract has been studied using weight loss, Potentiodynamic polarization, Electrochemical Impedance Spectroscopic (EIS) and Scanning Electron Microscopic (SEM) techniques. Corrosion rate increases with the increase in acid concentration and temperature. As inhibitor concentration increases corrosion rate decreases while percentage of inhibition efficiency (I.E.) increases. Maximum I.E. of Cuminum cyminum seeds extract was found up to 88.39 % at 1.2 g/L inhibitor concentration in 1.0 M HCl solution. Polarization curve indicates that inhibitor act as mixed type. The results obtained from chemical and electrochemical techniques were in good agreement.
Keywords: Aluminium, HCl, Corrosion, Cuminum cyminum, Polarization, EIS, SEM.

I. INTRODUCTION

Corrosion is the deterioration of metal by chemical attack or by reaction with its environment. Aluminium is widely used in various industrial operations due to light weight, high thermal, electrical conductivity and relatively high mechanical strength. Aluminium and its alloy are widely used in construction, vessels, pipes, machinery and packing. Aluminium is used in electronics due to it is super purity [1]. Hydrochloric acid solution is one of the most currently used acids in the pickling and electrochemical etching of aluminium capacitor foil [2]. One of the methods used to reduce the rate of metal corrosion is the addition of inhibitors. Organic compounds are used as inhibitors, but because of its toxic nature and high cost now it is necessary to develop environmentally safe, readily available and cheap substances, as corrosion inhibitors. Many researchers [3-8] studied the corrosion inhibition of aluminium in HCl using various green inhibitors. Several green inhibitors were used to protect various metals in hydrochloric acid [9-11].

Cumin is the dried seed (Fig.1) of the herb *Cuminum cyminum*. *Cuminum cyminum* (Cumin, common name "Jeera") is a member of flowering plant in the family Apiaceae, native to a territory including the Middle East and stretching east to India. Today, the plant is mostly grown in the Indian subcontinent, Northern Africa and Mexico. The Cumin plant grows to 30-50 cm tall and harvested by hand. It is annual herbaceous plant, with a slender, glabrous, branched stem that is 20-30 cm tall and has a diameter of 3-5 cm [12]. Cuminaldehyde, cymene and terpenoids are the major volatile components of cumin oil which is used for a variety of flavours, perfumes and essential oil. Cumin can be used ground or as whole seeds. In traditional medicine practices of several countries, dried cumin seeds are believed to have medicinal purposes.

The seed is antispasmodic, carminative, galactagogue, stimulant and stomachic. A general tonic to the whole digestive system, it is used in the treatment of flatulence and bloating, reducing intestinal gas and relaxing the gut as a whole. In southern Indian states, a popular drink called jira water is made by boiling cumin seeds. It imparts an earthy, warming and aromatic character to food, making it a staple in certain stew and soups, as well as spiced gravies such as curry and chilli [13]. The main phytoconstituent of Jeera seeds is cuminaldehyde.

The extracts of Cuminum cyminum have been reported to effectively inhibit the acidic corrosion of metals [14-17]. The aim of the present study is to investigate the corrosion inhibition effect of *Cuminum cyminum* seeds as a cheap and environment friendly corrosion inhibitor for Al in 1.0 and 1.25 M of HCl by using weight loss, effect of temperature, polarization, EIS and SEM techniques.

Fig. 1 The Cumin seeds.

II. EXPERIMENTAL SECTION

A. *Preparation of Sample and Solution*

The aluminium specimens with a chemical composition of 99.54 % Al, 0.090 % Si, 0.320 % Fe, 0.0012 % Cu, 0.0034 % Mn, 0.0014 % Mg, 0.0042 % Cr, 0.0046 % Ni, 0.0020 % Zn, 0.0079 % Ti, 0.0005 % Pb, and 0.0026 % Sn are used in the present study. The metal sheet, test specimens of size 5.0 x 2.50 x 0.198 cm having an effective area of 0.2797 dm^2 are used. The specimens are cleaned by washing with distilled water, degreased by acetone, washed once more with doubled distilled water and finally dried and weighted by using electronic balance. Hydrochloric acid was used as corrosive solution having concentration of 1.0 and 1.25 M prepared by diluting analytical grade of HCl purchased from Merck using double distilled water.

B. *Preparation of Cuminum cyminum Seeds Extract*

The Cumin seeds are crushed into fine powder. The solution of *Cuminum cyminum* seeds are prepared by refluxing 10 gm of Cumin powder with 100 ml of distilled water for about 2 h and kept for 24 h, filtering and test solutions are prepared using stock solution with different concentration from 0.6, 0.8, 1.0 and 1.2 g/L by diluting in 1.0 M and 1.25 HCl [18].

C. *Weight loss Measurement*

For weight-loss measurement, the Al specimen completely suspended in 230 ml of 1.0 and 1.25 M HCl solution in absence and presence of 0.6, 0.8, 1.0 and 1.2 g/L concentration of *Cuminum cyminum* seeds extract using glass hooks at 301± 1 K for 24h. The coupons are retrieved after 24h washed with distilled water, dried well and reweighed. From the weight loss data, corrosion rates (CR) are calculated.

$$\text{C.R. } (mg/dm^2 d) = \text{Weight loss (gm.)} \times 1000 / \text{Area in } dm^2 \times \text{day} \quad (1)$$

Inhibition efficiency (I.E.) was calculated by using following equation:

$$\text{I. E. } (\%) = \{(W_u - W_i) / W_u\} \times 100 \quad (2)$$

where, W_u= Weight loss in absence of inhibitor, W_i = Weight loss in presence of inhibitor.

The degree of surface coverage (θ) of the aluminium specimen for different concentration of HCl solution have been evaluated by weight loss experiments using the following equation:

$$\Theta = (W_u - W_i) / W_u \quad (3)$$

D. *Potentiodynamic Polarization Measurement*

Both the potentiodynamic and EIS measurement were carried out using CHI608C-series, U.S. Model with CH- instrument. For polarization study, metal specimens are immersed with and without *Cuminum cyminum* seeds extract in 1.0 M HCl solution. In the electrochemical cell, aluminium specimens having an area of 1 cm^2 was used as a working electrode, Ag/AgCl electrode as a reference electrode and platinum electrode as an auxiliary electrode and allowed to establish a steady-state open circuit potential (OCP) for approximately 30 min. The polarization curves were plotted with current vs potential. An anodic and cathodic polarization curve gives corresponding anodic and cathodic Tafel lines. The intersect point of Tafel lines gives the corrosion potential (E_{corr}) and corrosion current (i_{corr}) [19].

E. Electrochemical Impedance Spectroscopy (EIS) Measurement

EIS measurements are made at corrosion potentials over a frequency range of 1 to 10^5 Hz by a sine wave with potential perturbation amplitude of 5 mV. A graph was drawn by plotting real impedance (Z') versus imaginary impedance (-Z''). From the Nyquest plots of Z' vs -Z'' the charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) are calculated. An experiment was carried out in absence and presence of inhibitor.

F. Scanning Electron Microscope (SEM) Study

The Al specimens immersed in 1.0 M HCl (blank) and containing 1.2 g/L of *Cuminum cyminum* seeds as green inhibitor for a period of one day at 298 K. After exposure, specimen was removed, rinsed with double distilled water, dried and observed in scanning electron microscope to examine the surface morphology. The SEM image of polished aluminium specimen was also taken. The surface morphology measurements of aluminium specimens were examined using JEOL-5610 LV (Made in Japan) computer control SEM.

III. RESULTS AND DISCUSSION

A. Weight loss Experiments

Effect of acid concentration- Results showed in Table 1 indicates that as the acid concentration increases corrosion rate increases. Corrosion rate was 6163.74 and 8283.87 mg/dm^2.d corresponding to 1.0 and 1.25 M HCl respectively for an immersion period of 24 h at 301± 1 K. At constant inhibitor concentration, the I.E. decreases with the increase in acid concentration (Table 1).

Effect of inhibitor concentration- At constant acid concentration, as the inhibitor concentration increases corrosion rate decreases while I.E. increases. e.g., in case of *Cuminum cyminum* seed extract in 1.0 M HCl the I.E. was found to be 65.19, 72.15, 79.11 and 88.39 % corresponding to 0.6, 0.8, 1.0 and 1.2 g/L inhibitor concentration respectively (Table 1).

Table 1 Effect of HCl concentrations on corrosion rate (C.R.) and I.E of Al having different concentration of Cuminum cyminum seed extract.

Inhibitor concentration (g/L)	Acid concentration			
	1.0 M		1.25 M	
	CR (mg/dm^{2}d)	I.E. (%)	CR (mg/dm^{2}d)	I.E. (%)
Blank	6163.74	-	8283.87	-
0.6	2145.15	65.19	3571.68	56.88
0.8	1716.12	72.15	2799.42	66.20
1.0	1287.09	79.11	2188.05	73.58
1.2	715.05	88.39	1784.05	78.46

B. Potentiodynamic Polarization Study

Potentiodynamic polarization study was carried out for aluminium in 1.0 M HCl in absence and presence of 1.2 g/L of *Cuminum cyminum* seeds extract. Electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), anodic Tafel slope (βa), cathodic Tafel slope (βc) and percentage I.E. are given in Table 2. Inhibition efficiency (I.E.) from polarization study was calculated using following equation: [20]

$$\text{I. E. } (\%) = \{(icorr_{(uninh)} - icorr_{(inh)})/icorr_{(uninh)}\} \times 100 \qquad (4)$$

where, $i_{corr\ (uninh)}$ is corrosion current density for uninhibited acid and $i_{corr\ (inh)}$ corrosion current density for inhibited acid.

From Table 2, it was observed that the addition of *Cuminum cyminum* seeds extract in acid solution indicates the significant decrease in corrosion current density (i_{corr}) and decrease in corrosion rate with respect to blank. There is significant change in the anodic and cathodic slopes after the addition of the inhibitor. In general, an inhibitor is anodic or a cathodic if the variation E_{corr} against the blank is higher or above 85 mV [21,22]. In this study, the displacement of E_{corr} was 20 mV (Table 2) which suggest that the *Cuminum Cyminum* function as a mixed type of inhibitor. Polarization curves (Tafel plots) were shown in Fig. 2.

Table 2 Potentiodynamic polarization data and I.E. of 1.2 g/L of *Cuminum cyminum* seeds extract as an inhibitor for Al in 1.0 M HCl.

System	E_{corr} (V)	I_{corr} ($\mu A/cm^2$)	Tafel slope (V / decade)			I.E. (%) Calculated from	
			Anodic (+βa)	Cathodic (-βc)	β (V)	Polarization method	Weight loss method
Blank	- 0.896	7433	0.63	1.11	1.73	-	-
Cuminum Cyminum	- 0.916	621.1	4.212	3.111	0.989	91.64	88.39

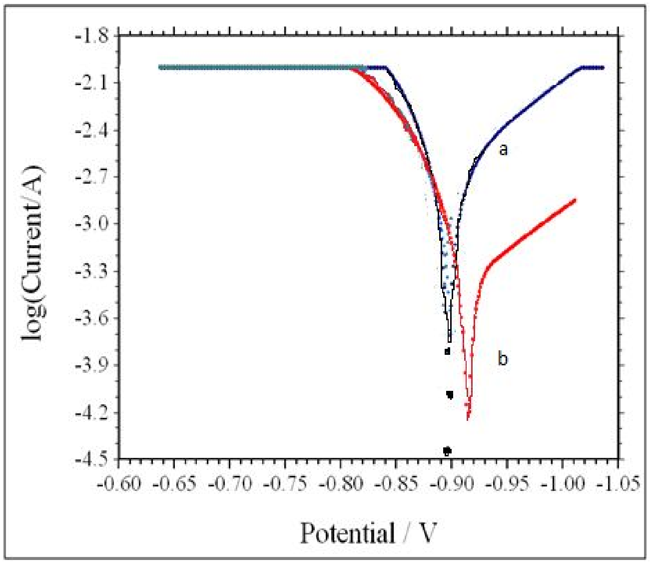

Fig. 2 Polarization curves for corrosion of aluminium in (a) in 1.0 M HCl (Blank) (b) in 1.0 M HCl in presence of 1.2 g/L Cuminum cyminum seeds extract.

C. Electrochemical Impedance Spectroscopy (EIS) Measurement

Nyquist plots for the corrosion of aluminium in 1.0 M HCl solution in absence and presence of *Cuminum Cyminum* seeds extract was examined by EIS method at room temperature was shown in Fig. 3 and EIS parameters in Table 3. It is observed from Fig. 3 that the impedance diagram is almost semicircle. The difference has been attributed to frequency dispersion. The semicircle nature of the plots indicates that the corrosion of aluminium is mainly controlled by charge transfer process.

Table 3 EIS parameters for corrosion of Al in 1.0 M HCl containing1.2 g/L *Cuminum cyminum* seeds extract.

System	R_{ct} (Ω cm^2)	C_{dl} ($\mu F/cm^2$)	I.E (%) Calculated from	
			EIS method	Weight loss method
Blank	40	199.04	-	-
Cuminum cyminum	310	3.42	98.24	88.39

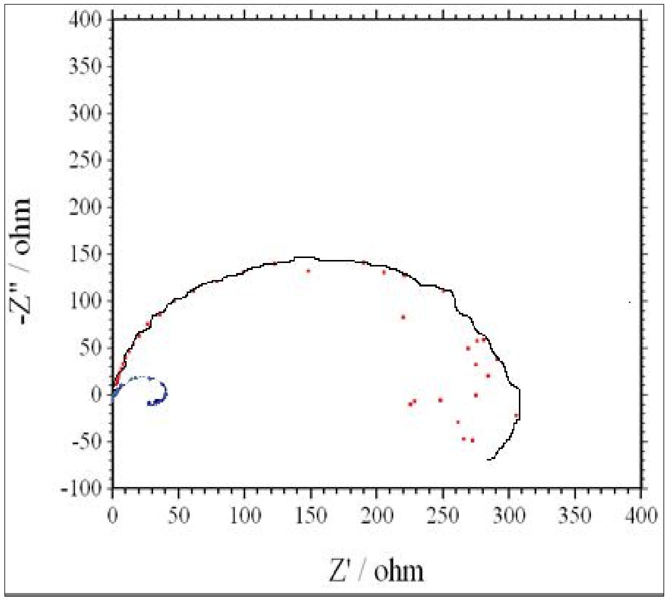

Fig. 3 Nyquist plot for Al in 1.0 M HCl in absence and presence of 1.2 g/L *Cuminum Cyminum* seeds extract.

The diameter of capacitive loop in the presence of inhibitor is bigger than that in the absence of inhibitor. The high frequency capacitive loop is related to the charge transfer resistance (R_{ct}). To calculate the double layer capacitance (C_{dl}), the frequency at which the imaginary component of the impedance is maximum was found as presented in the following equation: [23]

$$C_{dl} = 1/2\pi F_{max} R_{ct} \quad (5)$$

where, 'f' is the frequency at the maximum height of the semicircle on the imaginary axis and R_{ct} is the charge transfer resistance [24]. Inhibition efficiency (I.E.) from EIS method was calculated using following equation:

$$\text{I. E. } (\%) = \{Cdl_{(uninh)} - Cdl_{(inh)}/Cdl_{(uninh)}\} \times 100 \quad (6)$$

where, $C_{dl(uninhi)}$ is double layer capacitance for uninhibited acid and $C_{dl(inhi)}$ is double layer capacitance for inhibited acid.
The addition of inhibitor increases R_{ct} value while decreases in C_{dl} values which is due to the adsorption of inhibitor on the metal surface. The results suggest that the inhibitor acts by the formation of a physical protective layer on the surface that retards the charge transfer process and therefore inhibit the corrosion reaction, leading to increase in R_{ct} values. Moreover, the adsorbed inhibitor species decrease the electrical capacity of electrical double layer values at the electrode/solution interface and therefore decrease the value of C_{dl} [25].

D. Scanning Electron Microscopy (SEM) Study

The SEM images of the aluminium surfaces exposed to 1.0 M HCl in absence and presence of 1.2 g/L of inhibitor for 24 h were presented in Fig.4 & Fig.5

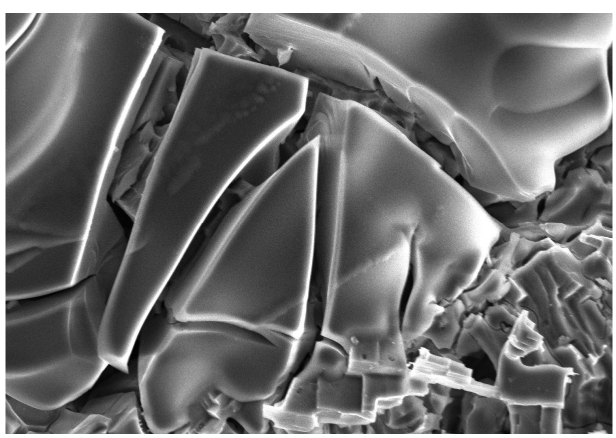

Fig. 4 SEM micrographs of Al surface after immersion in 1.0 M HCl in absence of inhibitor (5000 X).

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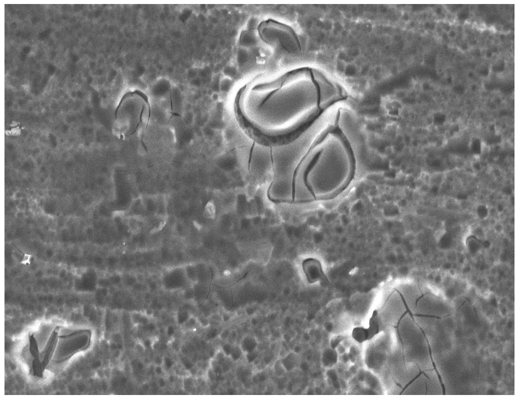

Fig. 5 SEM micrographs of Al surface after 24 h immersion in 1.0 M HCl in presence of 1.2 g/L Cuminum cyminum seed extract at 301 K.

Fig. 4 shows the morphology of uninhibited Al surface and the micrograph reveals that the roughness of the metal surface which indicates the dissolution of aluminium. The corrosion products appear very uneven and lepidoteral-like morphology. Fig. 5 showed the SEM image of the Al surface exposed in inhibited acid (1.0 M HCl + 1.2 g/L inhibitor). The metal surface is smooth and almost free from corrosion due to the formation of insoluble complex on the surface of metal. In the presence of inhibitor, the surface is covered by a thin layer of inhibitor which effectively controls the dissolution of aluminium.

E. Mechanism of Corrosion

Being a strong oxidizing agent, HCl is capable of attacking aluminium. Aluminium is corroded to Al^{+3} in HCl solution and no oxide film is formed to protect the surface from the attack of the corrosive medium. The chronology of the mechanisms can be determined as follows. In the anodic half-cell reaction, the dissolution of aluminium takes place according to the following equation: [26,27]

$$Al_{ads} \rightarrow Al^{3+} + 3e^{-} \qquad (7)$$

The oxygen reduction occurs at cathodic half-cell yielding the following equation:

$$1/2\ O_2 + 2H_2O + 3e^{-} \rightarrow 3\ OH^{-} + 1/2\ H_2 \qquad (8)$$

The overall equation is represented by the following equation:

$$Al^{3+} + 3OH^{-} \rightarrow Al\ (OH)_3{\cdot}3H_2O \qquad (9)$$

In chloride containing solution like HCl, the aluminium anion undergoes hydrolysis:

$$Al^{3+} + H_2O \longleftrightarrow H^{+} + Al\ (OH)^{2+} \qquad (10)$$

Aluminium hydroxide reacts with chloride:

$$Al\ (OH)^{2+} + Cl^{-} \rightarrow Al\ (OH)\ Cl^{+} \qquad (11)$$

It further reacts with water consequently producing acidic conditions:

$$Al\ (OH)\ Cl^{+} + H_2O \longleftrightarrow Al\ (OH)_2Cl + H^{+} \qquad (12)$$

F. Mechanism Inhibition by Cuminum cyminum Seeds Extract

The main constituents of cumin are Cuminaldehyde, *p*-cymene and terpenoid [28]. The main phytoconstituent of Jeera seeds is Cuminaldehyde (Fig. 6). It is likely to impart good corrosion inhibition activity due to presence of aromatic ring, aldehyde and isopropyl groups as substituents. Cuminaldehyde (4-isopropylbenzaldehyde) is a natural natural organic compound with the molecular formula $C_{10}H_{12}O$.

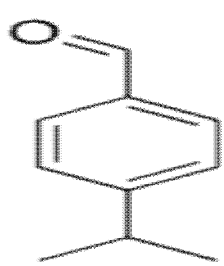

Fig. 6 Structure of Cuminaldehyde

When cuminaldehyde adsorbs on the Al surface, electrostatic interaction takes place by partial transfer of electrons from the polar O-atom and the delocalized π electrons around the benzene ring to the metal surface [29]. Our results are in good agreement with the results obtained by Ladha et al. [28] in their work on cumin extract as corrosion inhibitor for aluminium in HCl medium and study of Patel et al. [17] on *Cuminum cyminum* (Jeeru) seeds extract a green corrosion inhibitor for brass in HNO_3 media.

IV. CONCLUSION

As acid concentration increases corrosion rate increases while I.E. decreases. At constant acid concentration, as inhibitor concentration increases corrosion rate decreases while I.E. increases. The seed extract of *Cuminum cyminum* showed maximum I.E. of 88.39 % at 1.2 g/L inhibitor concentration in 1.0 M HCl solution. A polarization curve indicates that *Cuminum cyminum* seed extract act as a mixed type of inhibitor with predominance of cathodic inhibitor on aluminium surface. EIS study shows that the charge transfer resistance increases and double layer capacitance decreases with increases in concentration of inhibitor. The SEM observation proves that the inhibition of corrosion is due to formation of an adsorbed passive film on the aluminium surface. From this experimental study, it is clear that *Cuminum cyminum* can be used effectively as eco-friendly green inhibitor for corrosion of aluminium in HCl solution.

V. ACKNOWLEDGEMENT

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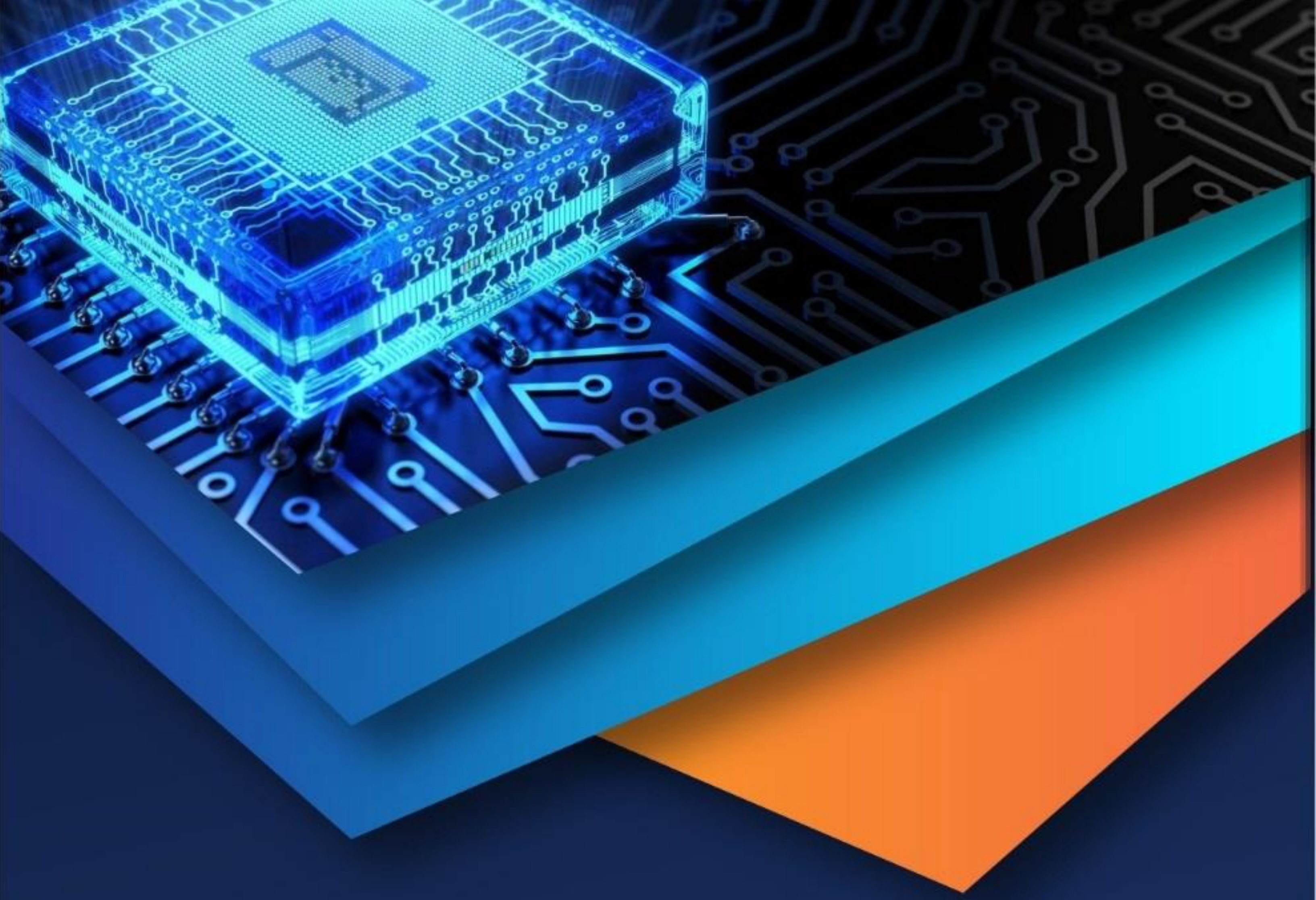

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