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Facile and Efficient One-Pot Green Synthesis of Benzimidazoles Using Oxalic Acid as an Organocatalyst in Ethanol Solvent

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Abstract: ***An efficient, straightforward, and general synthetic method has been developed for the synthesis of benzimidazoles via the cyclo-condensation of aromatic aldehydes or acids with o-phenylenediamine in ethanol, using oxalic acid as an organocatalyst. This method offers several advantages over existing protocols, including shorter reaction times, improved selectivity, environmentally friendly conditions, simple product isolation, and excellent yields.***
Keywords: Benzimidazole, Organocatalyst, o-Phenylenediamine, Green synthesis.

I. INTRODUCTION

Organocatalysis has emerged as a powerful and versatile tool in modern synthetic chemistry, offering significant advantages over traditional catalytic methods. Its importance lies not only in its efficiency and selectivity but also in its alignment with the principles of green chemistry.[1] One of the most notable benefits of organocatalysis is its metal-free nature, which eliminates the need for toxic or expensive transition metals. This makes organocatalytic methods particularly attractive for applications in pharmaceutical synthesis, where metal contamination must be minimized. Organocatalysts are typically small, stable organic molecules that are often non-toxic, inexpensive, and easy to handle**,** further enhancing their practicality in both laboratory and industrial settings.[2] In the synthesis of benzimidazoles, oxalic acid acts as an organocatalyst by promoting the cyclo-condensation between *o*-phenylenediamine (OPD) and aromatic aldehydes or acids in ethanol. This process exemplifies organocatalysis by combining efficiency, simplicity, and sustainability. Benzimidazoles share structural similarity with key biomolecules such as adenine and guanine, two of the five nucleic acid bases, as well as uric acid and caffeine. Due to this fundamental resemblance, the benzimidazole nucleus has emerged as a biologically significant pharmacophore and is recognized as a privileged structure in medicinal chemistry. These compounds are also widely acknowledged as essential building blocks in various biopolymer frameworks. The first synthesis of benzimidazoles was reported by Hoebrecker in 1872, who obtained 2,5-dimethylbenzimidazole via the reduction and subsequent dehydration of 2-nitro-4-methylacetanilide.[3] This pioneering work laid the foundation for the extensive exploration of benzimidazole derivatives in medicinal and synthetic organic chemistry. Several methods have been developed for the synthesis of benzimidazoles under a wide range of conditions, employing a variety of catalysts. The most common synthetic method for the preparation of a wide range of benzimidazoles is Phillip's method, which involves the condensation of OPD with carboxylic acids or its derivatives, under heating conditions in the presence of concentrated hydrochloric acid. Alam et al. have reported the preparation of new benzimidazoles under reflux in xylene and polyphosphoric acid with equimolar amounts of o-phenylenediamine with *p*-aminobenzoic acid.[4,5] One-pot procedures were reported for the preparation of benzimidazoles using ammonium chloride as catalyst at 80-90˚C temperature,[6] and alumina, silica gel and zeolite HY under microwave and solvent free conditions.[7] Several methods were also reported for the preparation of benzimidazoles by reacting *o*-phenylenediamine with aromatic aldehydes by using sodium pyrosulfite $Na_2S_2O_5$ (sodium metabisulfite)[8] nitro benzene ($PhNO_2$),[9] lanthanum chloride ($LaCl_3$),[10] oxidation process with either air or several other oxidizing agents,[11] palladium or copper catalysts $(CH_3COO)_2Cu$,[12] and $[In(OTf)_3]$,[13]. There are some methods in the literature that describe the green synthesis of benzimidazole derivatives such as green synthesis of benzimidazoles using ZnO NPs as catalyst under solvent free conditions,[14] Cu-phen-MCM-41 and Cu-complex-bipy-MCM-41 as catalysts under ultrasonic irradiation,[15] nano-BiOCl under ultrasonication in water,[16] Silica trichloroacetic acid under sonication in aq. ethanol,[17] and photochemical protocol using 2,2-dimethoxy-2-phenylacetophenone as a photoinitiator.[18] Besides, benzimidazole derivatives are known to possess a diverse biological activities, including anticancer,[19] anti-inflammatory,[20] antioxidant,[21] anticonvulsant,[22] antitubercular,[23] antimicrobial,[24] and antiviral.[25]

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Many existing methods for benzimidazole synthesis require strong acidic conditions, expensive reagents, prolonged reaction times, harsh experimental conditions, and tedious work-up procedures, often resulting in the generation of large amounts of toxic waste. Apart from this, these protocols are limited to either aldehydes or acids only and are unsuccessful to both aldehydes and acids. Therefore, the development of efficient, selective, and environmentally friendly (green) synthetic procedures remains a significant challenge. In particular, there is considerable interest in exploring simpler, cost-effective, and more sustainable approaches, including the use of metal-free organocatalysts.

Oxalic acid is a relatively stable, water soluble and readily available low cost organic substance. It is very easy to handle, less toxic and is not too much sensitive to air and moisture. Despite its favorable properties, there have been no reports on the use of oxalic acid as a Brønsted acidic organocatalyst for the synthesis of benzimidazoles. In this study, we report the first example of an oxalic acid-catalyzed, one-pot green synthesis of benzimidazoles via the cyclocondensation of aromatic aldehydes or acids with *o*-phenylenediamine in ethanol as the solvent.

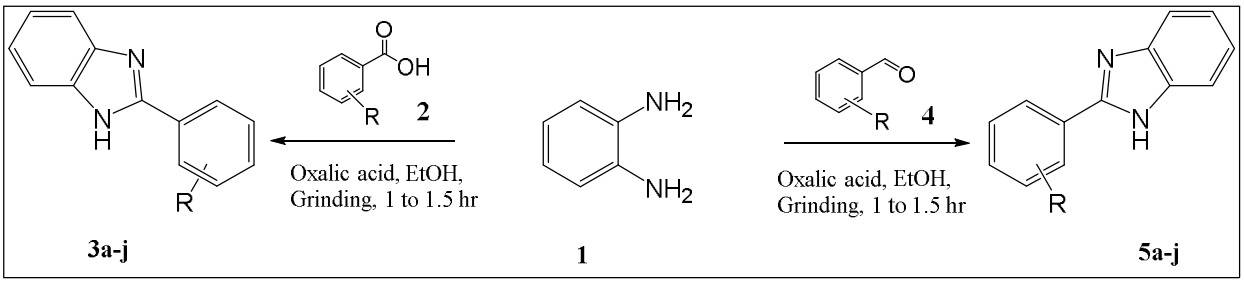
II. MATERIALS AND METHODS

The AR Grade chemicals, specifically Loba and Spectrochem made were purchased from local suppliers and were used without purification. Solvents were distilled off and used to conduct experiments. The structures of compounds were confirmed by IR, ^{1}H NMR, ^{13}C NMR and MS spectral analysis. The ^{1}H NMR and ^{13}C NMR spectra were obtained on a Varian Inova-400 spectrometer using DMSO-D6, and TMS as an internal standard. LC-MS analyses were performed on a HP-1100 LC-MS. Melting points were determined using a Büchi B-540 instrument. All melting points are uncorrected.

III. RESULTS AND DISCUSSION

Benzimidazole derivatives were successfully synthesized in 85-92% yield using aldehydes or carboxylic acids and *o*-phenylenediamine in the presence of 10 mol% oxalic acid under solvent-assisted grinding conditions. The reactions were performed in ethanol using a mortar and pestle, with reaction times ranging from 1 to 1.5 hours, as outlined in Scheme 1.

Scheme 1: Synthesis of benzimidazole derivatives from acids (2) or aldehydes (4) and OPD (1) catalyzed by oxalic acid



To establish the optimum reaction conditions for this transformation, three different solvents, ethanol, water, and a mixture of ethanol and water were evaluated along with varying molar ratios of oxalic acid. In a preliminary investigation, the condensation of *o*-phenylenediamine with benzaldehyde was carried out under both neat (grinding) and solvent-assisted conditions in the presence of oxalic acid as an organocatalyst for 2 hours. The influence of catalyst loading and solvent choice was systematically studied, as summarized in **Table 1**.

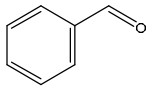
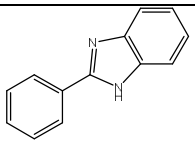
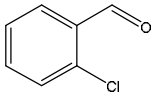
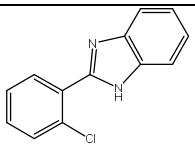
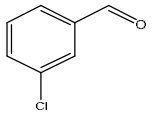
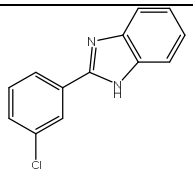
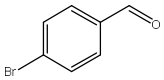
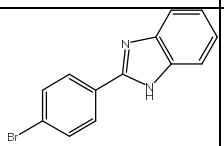
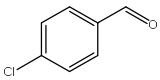
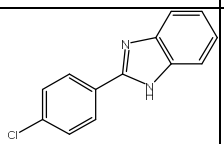
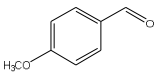
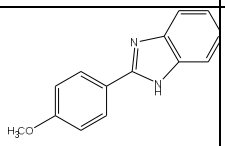
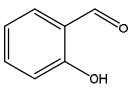
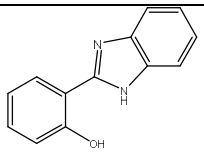
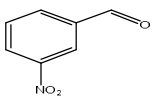
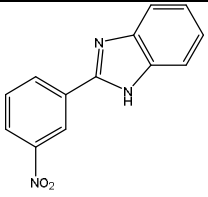
Table 1. Catalyst optimization in the synthesis of 2-phenyl-1H-benzo[d]imidazole

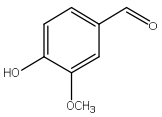
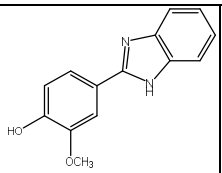
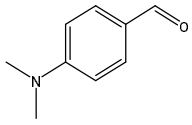
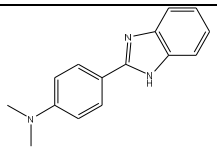
Entry	Catalyst	Solvent	% Yield	Solvent	% Yield	Solvent	%Yield
1	0 mol %	Ethanol	0	Water	0	Etanol+Water (1:1)	0
2	1mol %	Ethanol	10	Water	5	Etanol+Water (1:1)	5
3	5 mol %	Ethanol	50	Water	20	Etanol+Water (1:1)	30
4	10 mol %	Ethanol	90	Water	50	Etanol+Water (1:1)	70
5	15mol%	Ethanol	90	Water	50	Etanol+Water (1:1)	70

Among the tested conditions, ethanol proved to be the most effective solvent, yielding benzimidazole with an excellent 90% conversion when 10 mol% of oxalic acid was employed. Although the reaction also proceeded in water and ethanol-water mixtures, the yields were significantly lower, at 50% and 70%, respectively.

Taking into account the efficiency of oxalic acid in the model reaction, a series of transformations were performed on substituted benzaldehydes. The percentage yields, reaction times, and physical constants for these reactions are summarized in **Table 3**.

Table 3: Synthesis of benzimidazoles from aldehyde and *o*-phenylenediamine

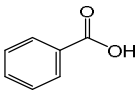
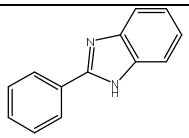
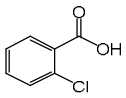
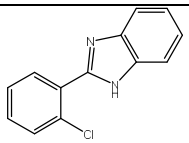
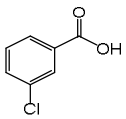
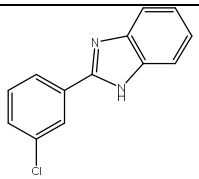
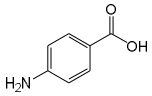
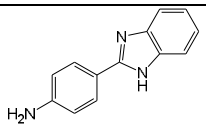
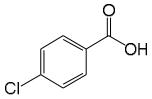
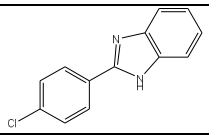
Entry	Aldehyde	Benzimidazole	Reaction Time (h)	(%) Yield[a]	Mp(˚C)[b]
5a			1.5	90	289-291
5b			1.5	89	231-233
5c			1.5	92	234-236
5d			1.5	92	297-299
5e			1.5	88	292-295
5f			1.5	92	218-221
5g			1.5	90	155-156
5h			1.0	85	203-205

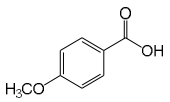
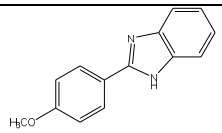
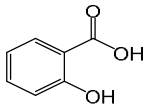
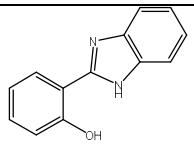
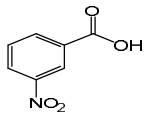
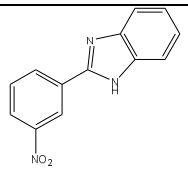
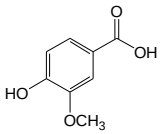
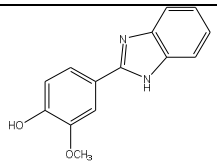
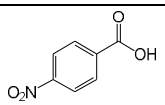
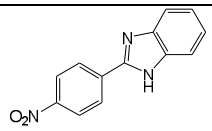
5i			1.5	89	242-244
5j			1.5	90	238-240

Reaction conditions: *o*-phenylenediamine (1mmol), aldehyde (1mmol), oxalic acid (10 mol %) were grind for 1 to 1.5h in Ethanol, [a]isolated yields and [b]melting points.

Following the success of aforementioned chemical transformations, the efficiency of oxalic acid was further investigated in the condensation reaction between benzoic acid and *o*-phenylenediamine in ethanol. Remarkably, the model reaction proceeded smoothly, affording an excellent yield (92%) of 2-phenylbenzimidazole. Purification of the compound was done by washing with cold water and recrystallization from ethyl alcohol. The purified compound was dried and its molecular structure was confirmed by using FT-IR, ^{1}H NMR, ^{13}C NMR and mass spectroscopic techniques and melting points. The same methodology was applied for other chemical transformations on substituted benzoic acids and their results are summarized in Table 2.

Table 2: Synthesis of benzimidazoles from acids and *o*-phenylenediamine

Entry	Acid	Benzimidazole	Reaction Time (h)	(%) Yield[a]	Mp(˚C)[b]
3a			1.5	92	289-291
3b			1.5	89	231-233
3c			1.5	92	234-236
3d			1.5	90	235-237
3e			1.5	88	292-295

3f			1.5	92	218-221
3g			1.5	90	155-156
3h			1.0	88	203-205
3i			1.5	90	242-244
3j			1.5	90	312-314

Reaction conditions: *o*-phenylenediamine (1mmol), acid (1mmol), oxalic acid (10 mol %) were grind for
1 to 1.5h in Ethanol, [a]isolated yields and [b]melting points
In both cases, the organocatalyst functions with full efficiency and without the formation of side products. Thus, the present methodologies for the synthesis of benzimidazole derivatives are highly significant compared to existing protocols, as no previously reported method employs a single catalyst for the conversion of both aldehydes and acids into the corresponding benzimidazole derivatives within a short reaction time.

A. . Experimental Section

1) General procedure for the synthesis of benzimidazoles

A mixture of *o*-phenylenediamine (1 mmol), aldehyde or acid (1 mmol) and oxalic acid (10 mol %) in ethanol (5 ml) was placed in a clean and dry mortar and grinding was continued till completion of reaction. The progress of the reaction was monitored by TLC (Hexane (8): EtOAc (2)), after completion of the reaction, the reaction mixture was poured into ice-cooled water (100g), it was quenched with aq. saturated solution of sodium bicarbonate and stirred for 10min. The solid product obtained after stirring was filtered off, washed by cold water 4-5 times, dried and purified by column chromatography on silica gel using n-hexane x ethyl acetate mixture (8:5). The aqueous layer was acidified by dilute solution of acetic acid and distilled off to leave oxalic acid.

2) Spectral data for selected compounds

2-Phenylbenzimidazole (3a):

White solid; IR v_{max} (KBr, cm^{-1}): 3445(-NH str.), 3075(C=C-H str.), 1623(C=N str.); ^{1}H NMR (400Hz, DMSO-d6): ^{1}H NMR: δ13.04 (br s, 1H, -NH), 8.15 (d, 2H), 7.62-7.47 (m, 3H), 7.23-7.08 (m, 2H), 7.87-7.81 (m, 2H); ^{13}C NMR (400 MHz, DMSO-d6): δ113.9, 117.5, 122.5, 122.6, 127.2, 127.4, 128.7, 129.1, 129.2, 130.7, 137.5, 137.6, 152.2; (LC-MS) m/z: 195.08 $[M+H]^{+}$.

2-(2-Chlorophenyl) benzimidazole (3b):

Red solid; IR v_{max} (KBr, cm^{-1}): 3484(-NH str.), 3085(C=C-H str.), 1521(C=N str.)^{1}H NMR (400Hz, DMSO-d6): δ12.80 (br s, 1H), 7.91-0.89 (m, 1H), 7.67-7.62 (m, 3H), 7.57-7.52 (m, 2H), 7.25-7.23 (m, 2H); ^{13}C NMR: δ 113.4, 116.7, 121.7, 121.9, 127.8, 127.9, 128.2, 128.8, 129.8, 131.9, 137.1, 138.0, 152.1; (LC-MS) m/z: 229.04 $[M + H]^{+}$.

2-(3-Chlorophenyl) benzimidazole (3c):

Colourless solid; IR v_{max} (KBr, cm^{-1}): 3484(-NH str.), 3081(C=C-H str.), 1520(C=N str.) ^{1}H NMR (DMSO-d6): δ13.05 (br s, 1H), 8.42 (s, 1H), 8.27 (d, 1H), 7.81-7.72 (m, 4H), 7.49-7.47 (m, 2H); ^{13}C NMR: δ 113.5, 116.8, 121.9, 122.0, 123.3, 123.3, 126.3, 129.1, 130.4, 136.0, 137.1, 137.5, 151.9; (LC-MS) m/z: 229.04 $[M + H]^+$.

2-(4-Chlorophenyl) benzimidazole (3d):

Colourless solid; IR v_{max} (KBr, cm^{-1}): 3486(-NH str.), 3101(C=C-H str.), 1518(C=N str.) ^{1}H NMR (DMSO-d6): δ12.9 (br s, 1H), 8.13 (d, 2H), 7.64-7.50 (m, 4H), 7.21 (d, 2H); ^{13}C NMR: δ 113.2, 115.7, 118.0, 121.5, 122.0, 127.0, 127.6, 128.3, 128.5, 134.9, 136.9, 137.0, 151.5; (LC-MS) m/z: 229.04 $[M + H]^+$.

2-(4-Methoxyphenyl) benzimidazole (3f): Colourless solid; IR v_{max} (KBr, cm^{-1}): 3484(-NH str.), 3105(C=C-H str.), 1522(C=N str.) ^{1}H NMR (DMSO-d6): δ12.90 (br s, 1H), 8.21 (d, 2H), 7.70-7.68 (m, 2H), 7.38-7.36 (m, 2H), 7.21 (d, 2H), 3.88 (s, 3H); ^{13}C NMR: δ 54.9, 113.1, 114.8, 115.7, 116.0, 117.7, 121.8, 121.9, 128.9, 129.0, 137.2, 137.4, 152.1, 160.3; (LC-MS) m/z: 225.07 $[M + H]^+$.

2-(3-nitrophenyl) benzimidazole (3h): Off-white solid; IR v_{max} (KBr, cm^{-1}): 3480(-NH str.), 3103(C=C-H str.), 1520(C=N str.);^{1}H NMR (DMSO-d6): δ13.2 (br s, 1H), 9.02 (s, 1H), 8.60 (d, 1H), 8.33 (d, 1H), 7.81 (t, 1H), 7.71-7.50 (m, 2H), 7.20 (t, 2H); ^{13}C NMR: δ 113.7, 117.5, 121.0, 122.3, 122.6, 124.0, 124.1, 124.2, 131.3, 137.4, 137.5, 148.2, 152.5. (LC-MS) m/z: 240.06 $[M + H]^+$.

4-(1*H*-benzo[d]imidazol-2-yl)-2-methoxyphenol (3i): White solid; IR v_{max} (KBr, cm^{-1}): 3495(-NH str.), 3482(-OH str.), 1530(C=N str.); ^{1}H NMR (400 MHz, DMSO-d6): δ 12.97 (1H, br, s, -NH), 3.68 (3H, s), 7.45-7.03 (2H, dd,), 7.81-7.04 (4H, m), 7.34 (1H, dd), 9.79 (1H, s, -OH); ^{13}C NMR (400 MHz): δ 56.0, 113.7, 114.5, 115.1, 117.3, 122.2, 122.8, 124.5, 128.1, 137.1, 137.4, 137.2, 147.9, 150.4, 152.5. (LC-MS) m/z: 141.09 $[M+H]^+$.

2-(4-Nitrophenyl)-1H-benzo[d]imidazole (3j): (Yellow solid; IR v_{max} (KBr, cm^{-1}): 3480 (N-H str.), 3042(C=C-H str.), 1604(C=N str.); ^{1}H NMR (400 MHz, DMSO-d6): *δ* 7.10 to 7.15 (m, 2H), 7.30 (d, 1H), 7.35 (d, 1H), 7.40 (t, 1H), 7.45 (t, 1H), 8.0 (dd, 2H), and 13.0 (brs, 1H); ^{13}C NMR (400 MHz): δ 113.8, 116.3, 117.8, 119.5, 122.4, 122.7, 122.7, 128.2, 133.5, 137.3, 137.9, 147.1, 152.1; (LC-MS) *m/z*: 240.06 $[M + H]^+$.

IV. CONCLUSIONS

Oxalic acid has been employed as a novel and efficient organocatalyst for the synthesis of benzimidazoles from *o*-phenylenediamine and a variety of aldehydes or carboxylic acids. The reactions were performed under grinding conditions using a mortar and pestle, with 10 mol% oxalic acid in ethanol as a green solvent, yielding products within 1 to 1.5 hours. This method proceeds under mild conditions, offering short reaction times, high efficiency, and minimal environmental impact. Notably, the catalyst can be recovered and reused for multiple cycles, enhancing the sustainability and practicality of the protocol. Overall, this method offers significant advantages over existing procedures in terms of simplicity, efficiency, and environmental friendliness.

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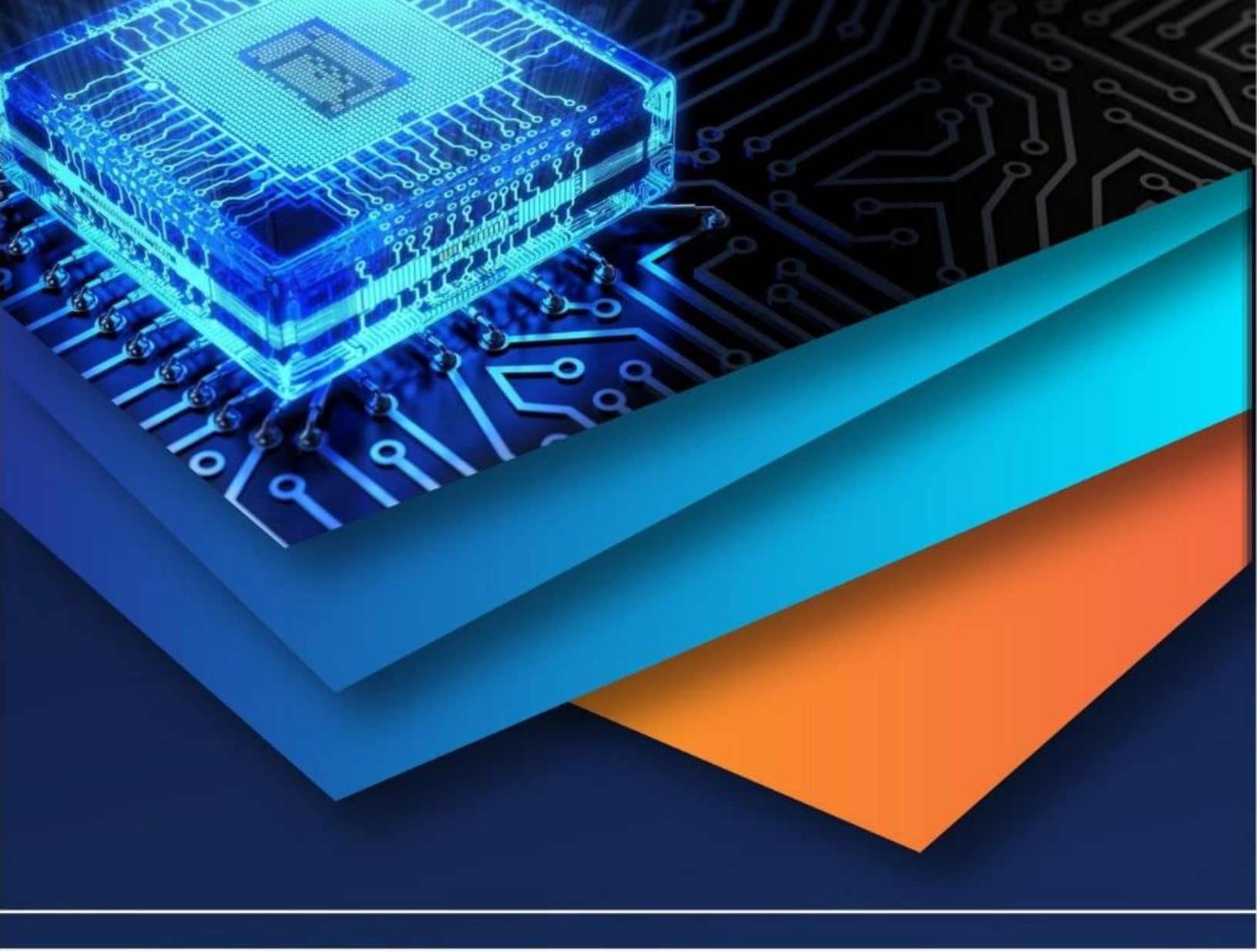

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