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FTIR Characterization and Structural-Functional Analysis of Kaolin Powder for Thermal Insulation Applications

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Abstract: ***Kaolin is an alumino-silicate mineral that occurs naturally, and has attracted attention as a suitable thermal insulation material because of its inherent structural stability and chemical functionality. Fourier Transform Infrared Spectroscopy (FTIR) characterization was performed to investigate the structural and functional properties of kaolin powder. The structural analysis was conducted using a Nicolet iS50 FTIR instrument and a Far-infrared spectrum (150–350 cm^{-1}), which indicated that distinct absorption bands attributed to Si–O and Al–O lattice vibrations were observed; the bands' representation confirms that the structural integrity and ordering of the crystalline framework was maintained. These attributes are correlated to the inherent high temperature stability of kaolin and the ability to maintain stability under thermal induced stresses. The typical functional analysis was compared in the Low and Mid Far Infrared region (1000–7000 cm^{-1}); strong bands corresponding to –OH stretching, Al–OH bending, and Si–O stretching vibrations were observed. These functional groups are representative of the presence of surface hydroxyls, interlayer hydrogen bonding, and silanol groups, thus proving the functional benefits that will additionally enhance kaolin's low thermal conductivity, refractory properties, as well as compatibility with composite systems. By incorporating structural resilience with functional reactivity, kaolin has the potential to be applied in many applications including insulating bricks, refractory linings, kilns, furnaces and energy-efficient construction materials. All in all, the dual FTIR analysis demonstrates kaolin to be a durable, sustainable and a viable thermal insulation material for modern industrial and greener technology purposes.***

I. INTRODUCTION

The increasing reliance on naturally occurring materials that are sustainable, energy-efficient and cost-effective has led to the study of naturally occurring minerals that can be applied as thermal insulators in industrial and building sectors. Among such minerals is kaolin, a hydrated alumino-silicate mineral, which has been of interest due to its abundance, low cost, structural stability and the provision of flexibility in usage[1], [2]. Though kaolin has long been utilized in ceramics, paper, paints and polymer composites, it is beginning to be studied more intensively for its potential as a thermal insulation and fireproof material in high-temperature uses[3], [4].

Kaolin's crystalline structure is distinct in that it has mechanical strength and chemical stability under heat stress. The sheeted silicate structure with alternating sheets of silica and alumina has rigid hydrogen bonding between the sheets with stability on heating. Furthermore, hydroxyl and silanol groups give reactive functional groups that enhance compatibility with other composites, rendering kaolin capable of acting as a multi-purpose material for sophisticated applications[5], [6], [7].

One specific method of characterization used to realize the structural and functional characteristics of kaolin includes Fourier Transform Infrared spectroscopy (FTIR). FTIR gives details of the vibrational characteristics of the bonds existing in the mineral, such as Si–O stretching, Al–O lattice vibration, and –OH stretching or bending, that gives details of both the crystalline structure and chemical activity of the minerals. By comparing absorption bands over the far infrared, mid-infrared, and low frequency, one can correlate the k measures of ordering structures and functional reactivity[8], [9], [10].

The significance of this lies in the fact that it is possible to utilize kaolin for industrial purposes, i.e., insulating bricks, refractory linings, furnaces, and energy-saving building materials[11], [12], [13]. Therefore, structural and functional FTIR, combined, not only verifies that kaolin is intrinsically stable and reactive in nature, but also indicates that kaolin can potentially be utilized as a stable and sustainable solution for thermal insulation of modern times and greener technology[14], [15].

II. LITERATURE REVIEW

Fourier Transform Infrared Spectroscopy (FTIR) is a well-established method that is utilized for characterizing kaolinite and other alumino-silicate clays as it is sensitive to lattice vibrations, hydroxyl groups and interlayer water. Vaculíková et al. differentiated between ordered and disordered kaolinite based on far-infrared peaks located near 200–210 cm^{-1} (Al–O vibrations) and ~340 cm^{-1} (Si–O vibrations) and suggested that these peaks indicate long-range crystallinity in kaolinite[16]. Similarly, Hussein et al. proposed that sharp bands at lower frequencies indicate well ordered alumino-silicate frameworks which can undergo high temperature treatment[17]. The OH stretching in the mid-infrared that has been associated with the inner-surface OH observed between 3620–3695 cm^{-1} has been attributed to the fingerprint for inner-surface OH in kaolin[18], [19], [20]. Deju et al. and Temuujin et al. observed that these OH bands disappeared from the spectra when kaolin was heated and attributed those bands to OH groups which were dehydroxylated during phase transformation to metakaolin[18], [19]. The H–O–H bending vibration near 1650 cm^{-1} noted by Temuujin et al. further verifies adsorbed[19].

III. METHODOLOGY

A. Sample Preparation

The selected sample material for characterisation was kaolin powder, which was used then in powdered form without any pre-treatment to avoid altering its original chemical state. The sample submission form indicated that the submission was classified as very low hygroscopic meaning that it would not readily absorb significant atmospheric moisture.

B. Instrumentation

The analysis used Thermo Scientific Nicolet iS50 FTIR spectrometer which is enabled for full-range spectral retrieval by advanced optics, high-resolution detector, KBr beam splitter and computer interface that allowed real-time monitoring, data acquisition and spectral analysis & processing (Fig 1). The FTIR assessments were performed at SAIF, IIT Madras with tightly controlled laboratory conditions to minimize interference from outside conditions.

Figure 1 :Thermo Scientific Nicolet iS50 FTIR spectrometer

Spectral characteristics of kaolin are governed by strong effect of structural hydroxyl groups and water, which were already intercalated in the kaolin clay, and decide kaolin's intercalation and water holding capacity. Saikia and Parthasarathy[20], report that Si–O stretching modes (1030–1110 cm-1 in the case of kaolin) predominate after the heat treatment, meaning that the structure is still alumino-silicate[21].

These findings show, as a whole, that FTIR is a reliable diagnostic method to evaluate kaolin's structural ordering, dynamic change, and functional properties, all of which are factors in use as refractory lining, insulating bricks and sustainability in thermal insulation technologies.

IV. RESULTS

A. FTIR Characterization and Structural-Functional Analysis of Kaolin Powder

Fourier Transform Infrared Spectroscopy (FTIR) was performed to investigate the structural and functional properties of the kaolin powder in two ranges - far-infrared (150–350 cm^{-1}) and low-to-mid infrared ranges (1000–7000 cm^{-1}). Both the spectra were recorded on a Nicolet iS50 FTIR at SAIF IIT Madras. The spectra analysis produces the double picture of kaolin lattice vibrations, hydroxyl functional groups, and silicate order, when all three are considered they are critical factors in deciding the thermal insulation and performance of a material.

B. Far-Infrared Spectrum (150–350 cm^{-1})

The kaolin powder's far-infrared spectrum (Fig. 1) showed well-defined and sharp bands between the range of 197–346 cm^{-1}. Especially, peaks at 197.6 cm^{-1}, 212.3 cm^{-1}, and 346.8 cm^{-1} were noted, which are due to the lattice vibrational modes of the alumino-silicate framework. In particular:

1) The 197-212 cm^{-1} range corresponds to Al–O lattice vibrations, due to octahedral aluminum sites in the kaolinite structure.

2) The ~346 cm^{-1} band is attributed to Si–O bending vibrations due to the tetrahedral silicate sheets.

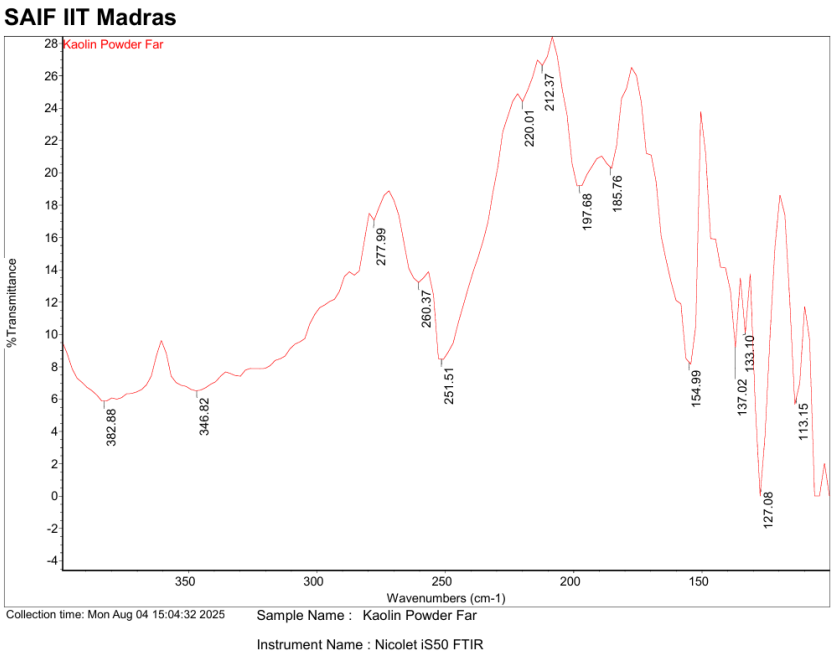

Figure 1. Far-Infrared FTIR Spectrum of Kaolin Powder (150–400 cm^{-1}) obtained on Nicolet iS50 FTIR at SAIF, IIT Madras

The presence of these peaks indicates the ordered crystalline structure of kaolin, since the powder maintains the original alumino-silicate framework with little evidence of structural disruption. This long-range order is generally accepted as a strong indication of thermal stability because disordered or amorphous phases usually weaken or broaden vibrational features.

Similar vibrational assignments have been reported in earlier FTIR studies. For example, Vaculíková et al. reported far-IR or ordered kaolinite bands at approximately 200–210 cm^{-1} (Al–O vibrations) and 340 cm^{-1} (Si–O vibrations), which are in full agreements with the foregoing observations[16]. Hussein et al. also verified that peaks in the far-IR with sharp contrast indicated the crystalline identity of kaolin[17].

This verifies that the kaolin sample examined possesses well-ordered crystalline domains which are responsible for the mechanical stability and tenacity necessary for long-term performance in high temperatures.

C. Low-to-Mid Infrared Spectrum (1000–7000 cm^{-1})

The low and mid-infrared region spectrum (Fig. 2) exhibited multiple strong absorption bands that can be correlated with functional groups present in kaolin. The multiple bands indicate the presence of hydroxyl groups, silanol groups, and interlayer water that define kaolin's physical and chemical properties.

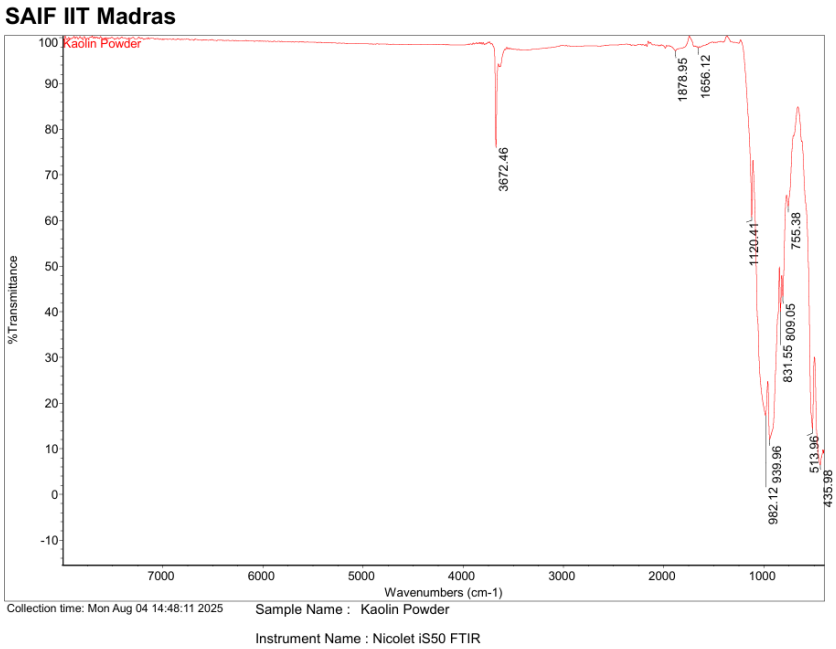

Figure 2. Low-to-Mid Infrared FTIR Spectrum of Kaolin Powder (750–4000 cm^{-1}) obtained on Nicolet iS50 FTIR at SAIF, IIT Madras

1) *Hydroxyl Stretching Region (3600–3700 cm^{-1})*
- Sharp peaks distinctive were noted between 3620–3695 cm^{-1}, indicating the stretching vibrations of inner-surface hydroxyl groups attached to octahedral aluminum.
- These hydroxyls are involved in interlayer hydrogen bonding, which is the cause of kaolin's thermal stability to 500–600 °C.
- The vibrational assignments conform with the results of Deju et al. who too demonstrated the same OH-stretching frequencies in unmodified kaolin samples[18].

2) *H–O–H Bending (1630–1656 cm^{-1})*
- A strong band occurring at about 1656 cm^{-1} is related to the bending vibration of interlayer water molecules.
- This indicates that there should be interlayer or adsorbed water molecules present on kaolin and this may influence the dehydration and dehydroxylation properties of kaolin under thermal treatment.
- Temuujin et al. also referred to these H–O–H bending modes indicating that the preservation of this characteristic means water is in the nature of ambient moisture adsorbed physically on the kaolin's surfaces[19].

3) *Si–O Stretching (1000–1120 cm^{-1})*
- The prominent absorption bands at 1030–1110 cm^{-1} are attributed to Si–O stretching vibrations of the tetrahedral silica layers.
- Most prominent bands, this region of the spectrum is regarded as a kaolin signature.
- Saikia and Parthasarathy have reported that these modes remained prominent even after the thermal treatment, suggesting that the silicate framework is rigid[20].

4) *Al–OH Bending and Si–O–Al Vibrations (750–950 cm^{-1})*
- Absorptions at 750–800 cm^{-1} are attributed to Al–OH bending vibrations whereas weaker bands approximating 900–950 cm^{-1} can be attributed to Si–O–Al linkages
- These functional modes help to emphasize the connectivity of the alumina and silica sheets that are responsible for structural stability with heating.

5) *Broad High-Wavenumber Range (4000–7000 cm^{-1})*
- Intensity features in the region above 4000 cm^{-1} are portrayed as overtones and combinations of bands.
- They are not so diagnostic but tend to provide an indication of the complex vibrational coupling phenomenon in the kaolin layered lattice.

Overall, the functional group analysis indicates that kaolins are hydroxyl-rich, structurally-unaltered, and capable of maintaining hydrogen-bonded stability. This can be significantly pertinent to use in insulation, composites, and refractory-based technologies.

D. Comparative Discussion with Literature

The FTIR data we obtained parallels similar studies on kaolin and related alumino-silicates:

1) Hydroxyl Vibrations: The intense bands at 3620–3695 cm^{-1} are well-documented as OH vibrations in the inner surface of kaolin[18], [19]. Moreover, Deju et al. demonstrated that with thermal treatment, these bands start to collapse which can be a sign of dehydroxylation[18].

2) Water Vibrations: The band at 1650 cm^{-1} is assigned to molecular water consistent with the findings of Hussein et al., and Temuujin et al. It is mentioned that kaolin reacts with atmospheric moisture and this could affect thermal and intercalation characteristics[17], [19].

3) Silicate Framework: The Si–O vibrations of stretching 1000–1120 cm^{-1} are universally considered to be the most diagnostic banding kaolin signature and appear in many studies such as Vaculíková et al[16]. It is considered that these bands are proof of the silica framework's rigidity and provide assurance of thermal stability.

4) Lattice Order: The reported here far-infrared bands are comparable to that of Vaculíková et al. who distinguished between ordered vs disordered kaolinites[16]. The ordering of clear peaks at ~200 and ~340 cm^{-1} in our sample verifies long-range lattice order, further verifying refractoriness.

Overall, the FTIR results are consistent with a broad range of published literature and verify the usability of kaolin in thermal analysis and thermal treatment processes.

E. Implications for Thermal Insulation Applications

Kaolin's combination of structural stability and functional reactivity has unique advantages:

1) Refractory and Structural Strength

- Short-range order exhibited by examination of Al–O and Si–O molecular vibrations produced a robust structure which is suited for refractory linings, kilns, and furnaces.
- This characteristic ensures kaolin-based ceramics and bricks preserve structural strength under thermal cycling without all or partial collapse.

2) Low Thermal Conductivity

- Hydroxyl functionalities and hydrogen bonding capabilities in kaolin contribute to phonon scattering, resulting in low thermal conductivity.
- This is beneficial for utilizing kaolin in energy-efficient insulating materials.

3) Composite Compatibility

- Hydroxyl and silanol groups provide reactive sites for bonding with polymers, cements, or resins.
- This aspect of kaolin's structure makes it valuable in nanocomposites coatings and structural reinforcements[22], [23].

4) Green and Sustainable Uses

- Because kaolin is a naturally occurring material that is naturally abundant and low-cost, a suitable material and a candidate for a sustainable resource to replace or contribute to synthetic (energy-inefficient) insulators.
- Kaolin has overlapping properties, which makes it a brazen candidate for green technology in construction by contributing to energy efficiency and environmentally damaging methods.

F. Extended Discussion with References

Multiple articles support the reliability of FTIR to link properties and function of kaolin:

1) Thermal Transformation: Temuujin et al. and Tironi et al. monitored kaolin's conversion to metakaolin, such as disappearance of hydroxyl bands on increasing temperature[24], [25].

2) Modification Studies: Hussein et al. and Aroke et al. each indicated how the movement or replacement of components in kaolin altered FTIR profiles in manners that indicated how to alter kaolin properties for other applications[17], [26].

3) Clays Differentiation: Vaculíková et al. and Saikia both demonstrated FTIR was able to distinguish kaolinites of various deposits, signifying the reliability of this technique to detect natural variations[21], [27].

Thus, the present FTIR findings indicate kaolin possesses inherent structurally dynamic and functionally rich characteristics, and the existing body of literature supports kaolin as being effective as a promising candidate material for modern thermal insulation prospective applications[17], [21], [24], [25], [26], [27], [28].

V. CONCLUSION

The FTIR characterization of the kaolin powder in the far-infrared and mid-to-low infrared range signifies an evident correlation between functional flexibility and structural stability of the mineral. In the far-infrared range (197–346 cm^{-1}), the highly defined and sharp nature of lattice vibrations signifies a very ordered alumino-silicate structure. Order in the structure is required for crystallinity, and hence the value of kaolin in refractoriness, mechanical strength, and resistance to high temperature with low phase transformations.

In the low-to-mid infrared region (1000–3700 cm^{-1}), we can investigate the chemical functional groups of kaolin by characteristic hydroxyl stretching bands, silicate framework vibrations, and water bending modes. Octahedral aluminum is bonded to hydroxyl groups and plays a dual role, they stabilize the layered lattice through interlayer hydrogen bonding and are possible sites for reactive surface modification or composites with kaolin. The Si–O stretching vibrations verify the structural hardness of a framework of silica, allowing for low thermal conductivity and high durability, and the bending band of H–O–H demonstrates kaolin's inherent ability to retain moisture and how this interaction affects dehydration and thermal transformation processes.

Together with the current literature, these consistently verify the widely reported FTIR signatures of kaolinite and hence endorse kaolin for industrial application. The three qualities of kaolin which are crucial - refractoriness, low thermal conductivity, and chemical compatibility with other materials - can now be merged with the abundance, low price, and environmental friendliness of kaolin to complement kaolin in green building technologies by offering advantage to sustainability and performance in energy-efficient building.

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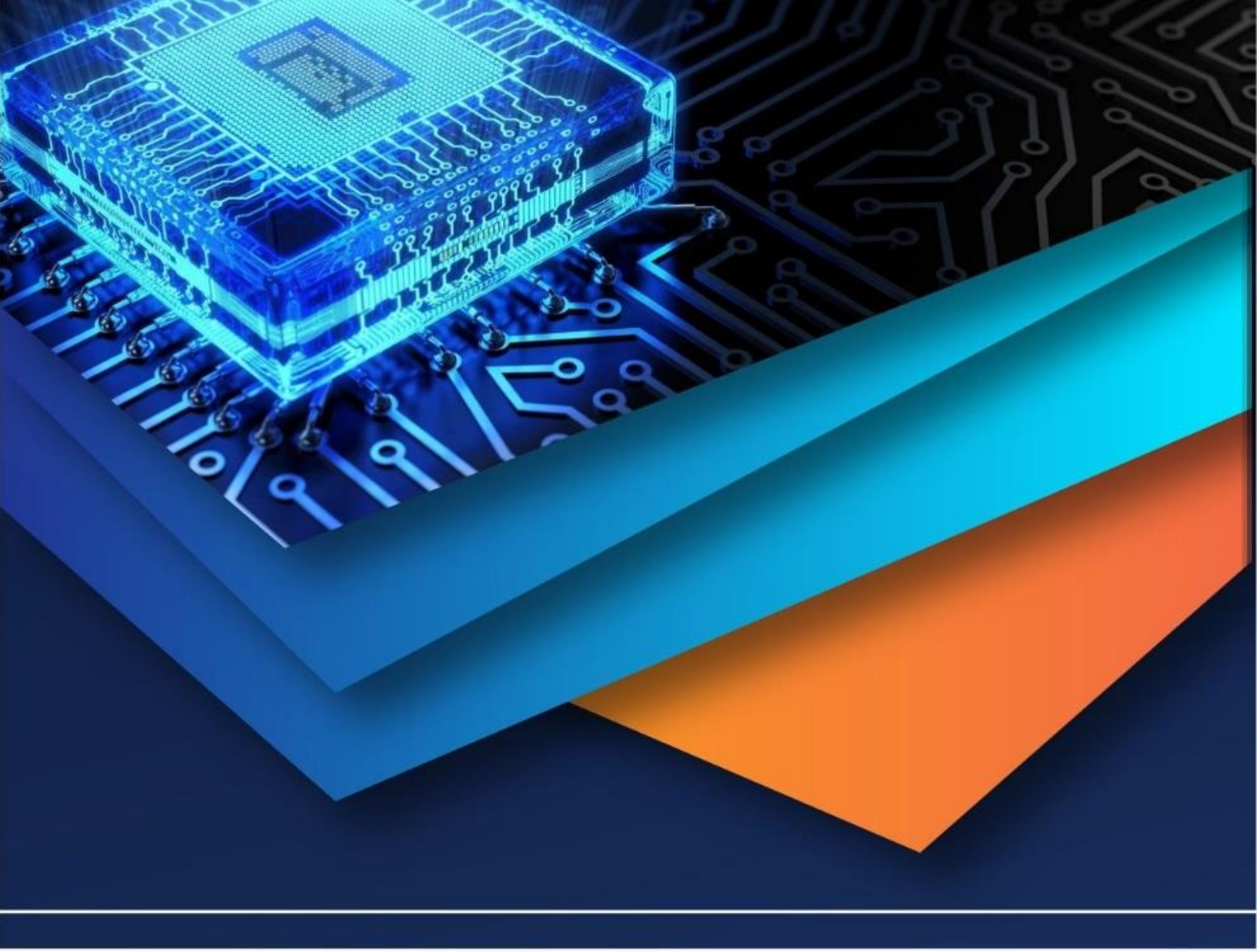

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