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A Chemical and Thermal Characterization Analysis of Kaolin

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Abstract: ***This study evaluates the chemical and thermal characterization of kaolin powder by means of Thermogravimetric Analysis (TGA) and Fourier Transform Infrared (FTIR) Spectroscopy. The TGA study was performed per ASTM E1131 by incrementally heating the sample from 22 °C to 1000 °C and results showed a total weight loss of 5.071 % with 4.354 % occurring between 400 °C and 800 °C as kaolinite dehydroxylated into metakaolin. A high residual mass of 94.930 % left on the basis residue at 1000 °C proved thermal stability and low volatility. Metakaolin being an amorphous aluminosilicate further improved thermal resistance and chemical inertness of the kaolin powder definitions of kaolin that had relevance reindustrial applications.In combination to the TGA study, the FTIR analysis was performed in accordance with ASTM E1252 methodology covering the infrared spectral range of 4000–500 cm^{-1} and thus confirmed the mineralogical composition. Firstly, the presence of kaolinite was confirmed by the very sharp Al–OH bending band at 925.87 cm^{-1} and additional minor overtone bands at 1984.25 cm^{-1} and 2149.94 cm^{-1} further confirmed structural composition. Furthermore, there was no significant absorption in the range of 1400–1600 cm^{-1} providing evidence for the absence of carbonate or organic impurities and further confirming the purity of the sample.TGA and FTIR evidence shows kaolin has high thermal stability, structural consistency, and mineralogical purity that can serve applications in refractories, insulation bricks, and advanced ceramics.***

Keywords: Kaolin, Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), Amorphous aluminosilicate, Metakaolin, Thermal Stability

Nomenclature			
TGA	Thermogravimetric Analysis	°C	Degree Celsius
FTIR	Fourier Transform Infrared Spectroscopy	cm^{-1}	Reciprocal Centimeter
ASTM	American Society for Testing and Materials	N_2	Nitrogen Gas
DTG	Derivative Thermogravimetry		

I. INTRODUCTION

Kaolin is a naturally occurring clay mineral of kaolinite ($Al_2Si_2O_5(OH)_4$) primarily used in ceramics, refractories, paper coating, thermal insulation,etc[1]. Kaolin has a variety of properties that make it suitable for many application, including chemical inertness, thermal stability, and structural stability[2-3]. Kaolin is overall performance and thermal performance at elevated temperature are functions of its thermal composition and behaviour [4].

The industrial importance of kaolin is largely due to its abundance as well as the unique physicochemical properties of kaolin that are determined by crystal structure, particle size distribution, structural stability and surface chemistry[5-6]. These properties ultimately control the thermal behaviour, phase transformations, and chemical inertness of kaolin at elevated temperatures, with the transformation from kaolinite to metakaolin, particularly critical when metakaolin may act as a precursor for high performance applications[7].

The amorphous aluminosilicate of metakaolin enhances pozzolanic activity in cementitious systems for durable, compressive strength and sustainable construction materials[8-9]. With kaolin's low thermal conductivity and high refractoriness, it has been applied to insulation bricks, refractory linings, ceramic bodies, or other high value engineering products, which rely on maintaining its performance characteristics at elevated temperatures[10-11].

Therefore, characterising kaolin's chemical and thermal behaviour utilising TGA and FTIR, will not only verify information on structural integrity and purity, but also solidify kaolin's full potential for industrial applications related to energy-efficiency or sustainable technology.

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In this study, we investigated the chemical and thermal characterization of a kaolin powder sample using both Thermogravimetric Analysis (TGA) and Fourier Transform Infrared (FTIR) Spectroscopy. TGA is an effective method, where the only measurement is the weight as temperature changes.

TGA enables the determination of thermal decomposition processes, stability, phases, and so on. Specifically, in the case of kaolinite, TGA can usually assess the total dehydroxylization of the kaolinite which is essential because that is the principal precursor of metakaolin which is an amorphous phase with higher thermal resistances. The thermal stability of TGA data also indicates the purity and volatile characteristics of a sample.

FTIR spectroscopy are utilized to support or complement thermal analyses by characterizing the functional group and purity of the mineral. By using the typical absorption bands, indicates that kaolinite is present and potentially identifies impurities (e.g. carbonates and potential organics) that may adversely influence thermal and chemical behavior. Together FTIR and TGA can provide some understanding of the structure and chemistry of kaolin.

This kaolin sample has undergone thermal analysis using TGA from ambient temperature to 1000 °C (ASTM E1131), and FTIR from 4000 to 500 cm^{-1} (ASTM E1252), therefore fulfilling the aim of analyzing the mineralogy of the sample and provide thermal decomposition characteristics, as well as capabilities for more implementation as technical components for insulation bricks, ceramics (with a higher thermal stability), and purity.

Previous studies have carried out a systematic assessment of kaolin's thermal and chemical properties first using TGA and FTIR. Deju et al. (2021) reported that kaolinite showed a weight loss of ~11-12% between 400-700 °C as a result of its dehydroxylation. The sample after the dehydroxilation step indicated that metakaolin was always formed because of the high thermal stability. Azizi et al. (2024) found the same thermal transitions and weight loss recovery (~10%), respectively, but which had no bearing with regard to their general description on Moroccan kaolin. The authors reconfirmed the synergistic effects of dehydroxylation when using kaolin for thermal applications.[5-6].

In these studies and others (Saikia and Parthasarathy, 2010; Vaculíková et al., 2011), FTIR analysis showed sharp absorption bands at or around 900–930 cm^{-1}, corresponding to Al–OH bending of kaolinite's crystal structure. In addition, the absence of bands related to carbonate compounds in the range of 1400–1600 cm^{-1} due to purity of the minerals is significant when evaluating kaolin for its thermal stability. Ultimately, these methodologies remain standard for evaluating kaolin quality or properties for industrial applications[7-8].

II. METHODOLOGY

Kaolin powder was tested in its raw, unmodified state, as a viable possibility for insulation purposes at high temperature. Kaolin was received as approximately 50 grams of powder in a sealed plastic container. Sample was stored in a desiccator in order to minimize moisture absorption.

One hour prior to measuring the performance characteristics of the kaolin powder, the sample was preheated to 80 °C for one hour in order to remove surface moisture and to enhance thermal and spectroscopic measurements.

A. Thermogravimetric Analysis (TGA)

Thermogravimetric Analysis (TGA) was performed on a TA Instruments model (Platinum HT) to evaluate the thermal stability and decomposition behaviour of the kaolin powder. The analysis was carried out in a nitrogen (N_2) environment to avoid oxidation at a flow rate between 20 – 50 ml/min. A 10-gram sample was heated in a platinum crucible, at a consistent ramp of 20 °C/min, from 25 °C-650 °C.

B. Fourier Transform Infrared Spectroscopy (FTIR)

To clarify the chemical composition of the kaolin, Fourier Transform Infrared Spectroscopy (FTIR) analysis was carried out. The FTIR was conducted using the potassium bromide (KBr) pellet method where a kaolin slurry was prepared with KBr and sized into pellets. A transmission mode scan of the pellets was performed at a resolution of 4 cm^{-1} and a wavenumber range of 4000 cm^{-1} to 400 cm^{-1}.

Between 32 to 64 scans were performed and averaged to gain a clear, distinct spectrum which was free of noise. The data was collected using a DTGS detector or equivalent. Before the run was performed, the pellets were preheated at 80 °C for an hour to remove any moisture.

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III. RESULTS

A. Thermogravimetric Analysis (TGA) of Kaolin

The results from thermal gravimetric analysis (TGA) on the sample of kaolin powder (as previously indicated in Figure 1) determined a total weight loss of 5.071% of an initial 30.03 mg at a temperature range from 22 °C to 1000 °C with the highest relative mass loss observed between 400 °C and 800 °C. The mass loss during this temperature interval indicated dehydroxilation of kaolinite, (removal of hydroxyl groups from the crystal structure). The mass loss relates to kaolinite conversion to metakaolin (the amorphous state of aluminosilicate), which is commonly noted to enhance thermal stability and chemical inertness.

Unlike previous studies which reported more significant losses in weight (~10–12%) for other kaolin clays (Deju et al., 2021; Azizi et al., 2024) the lower loss in weight from this sample indicates that it is a higher purity product or it simply may contain less volatile material. The remaining weight of 94.930% indicates the majority of this material is intact after thermal cycling which is pertinent for any materials that endure cumulative exposures to lengthy thermal exposures. thermal insulators[5-6].

The exothermic reaction observed during dehydroxylation confirms the stability and degree of non-volatility of the new product metakaolin indicates the suitability of kaolin powders for use in thermal insulation bricks. The considerable residue amounts and minimal weight loss indicate that they will experience dimensional stability upon the application of thermal energy, another beneficial characteristic among industrial ceramics and refractories.

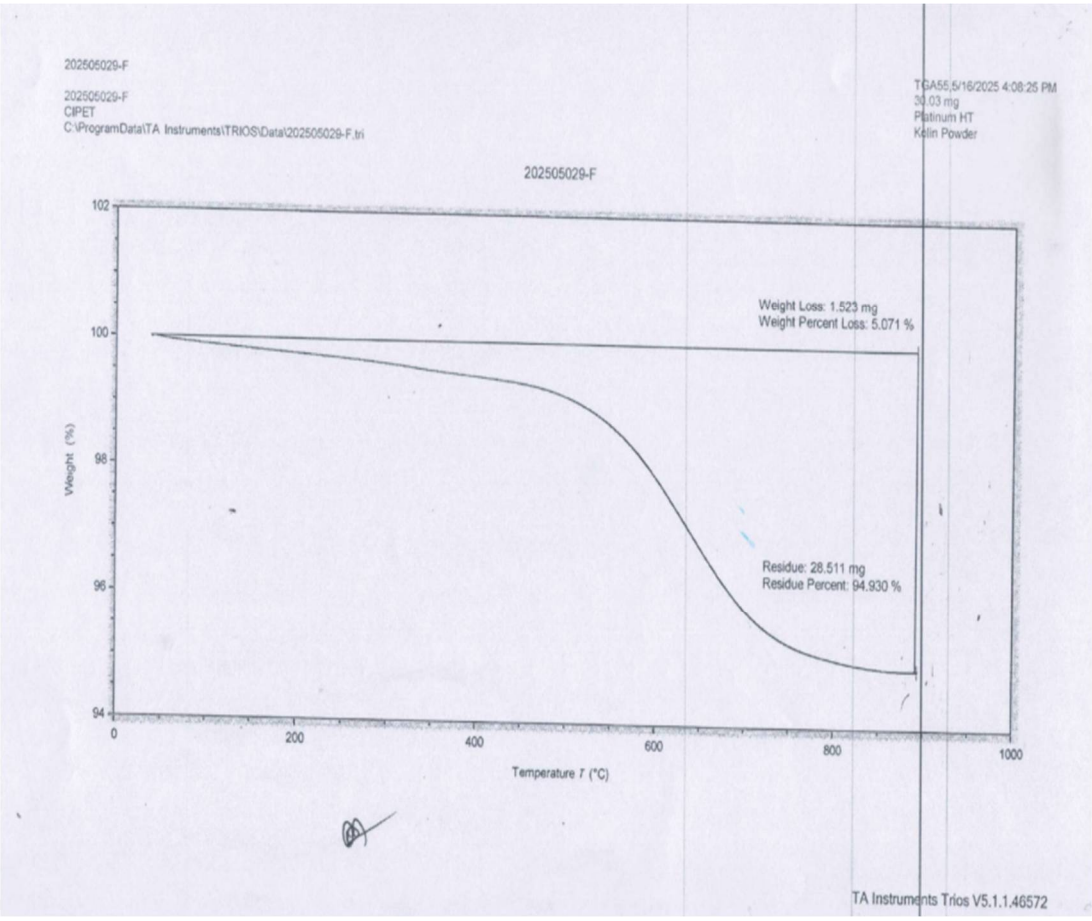


Figure 1. Thermogravimetric Analysis (TGA) graph of Kaolin.

B. FTIR Characterization Overview of Kaolin

The infrared spectrum of a kaolin powder demonstrates unique absorption bands that verify mineralogical identity and the purity of the kaolin sample. The strong, sharp band in the FTIR spectrum of the sample that occurs at 925.87 cm^{-1} relates to the Al−OH bending vibration, and this is consistent with the kaolinite layered aluminosilicate sheet (Vaculíková et al., 2011; Saikia and Parthasarathy, 2010)[7-8]. The sharp, intense absorption band demonstrates that the sample is crystalline kaolinite, which also confirms structural integrity (Figure 2).

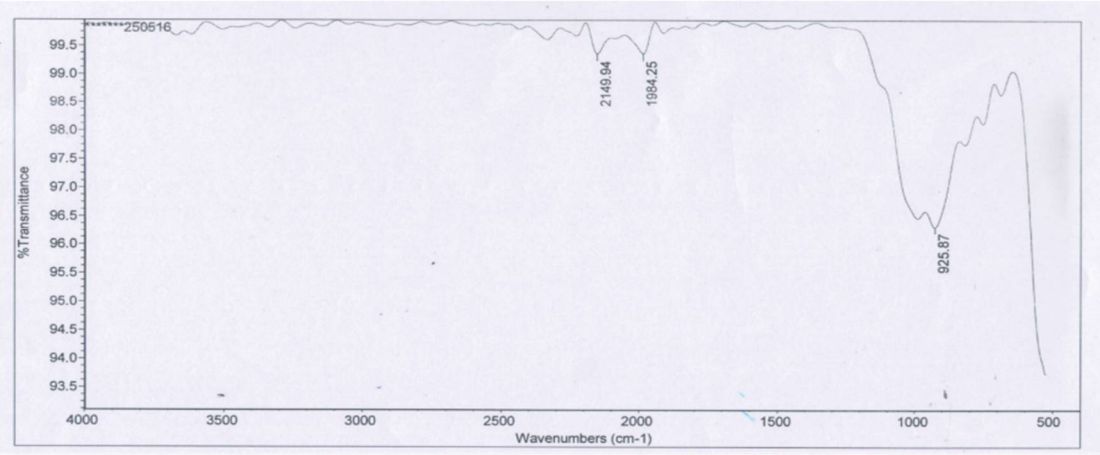


Figure 2. FTIR spectrum of Kaolin showing key functional groups.

Further absorption features appear at wavenumbers 1984.25 cm^{-1} and 2149.94 cm^{-1}, which are overtone and combination modes of vibration which summarize the molecular structure of kaolinite, purely vibrating (Vaculíková et al., 2011; Saikia and Parthasarathy, 2010). The documented bands identified in the 1400-1600 cm^{-1} range are only some that would be considered and possessing minor significant and identifiable presence. These portions of the spectrum represent approximative reference to identify carbonate species or next imply organic contamination within kaolin, therefore the absence of band absorption indicates a possible indication of mineralogical purity of kaolin. Mineralogically pure kaolin powder is desired for KOOL kaolin is superior thermal stability and chemical inertness, thus limiting reactivity is essential to fuller structural strength.

The FTIR spectral characteristics documented in these kaolin samples are very closely matched with literature documented data, during analysis closely matched band wavenumbers and absence of other impurities have been assigned to kaolinite suitable for high performance thermally insulating applications or refractor applications (Vaculíková et al., 2011). The identification of presence and confirmation of Al–OH groups in kaolinite through FTIR data to the stability of a chemical composition stable against thermal degradation at elevated temperatures.

C. Integrated Interpretation and Application Potential

There was a kaolin powder profile from the TGA and FTIR that exhibited good stability and chemical resistence. Low mass loss and relatively high residue indicates the material probably contains no substantial volatiles or chemically reactive constituents - a key consideration for high-performance ceramic products and energy-efficient insulation.

The metakaolin phase described above (dehydroxylation), along with bonding capacity for the size of the materials having lower reactivity than kaolin will provide it improved thermal stability. The FTIR findings also confirm this since the layered structure was intact and had no indication of impurities that are likely to be a reason for a decline in performance.

Features mentioned above have major implications on applications such as thermal insulation bricks for industrial furnace or high temperature refractories linings, high performance ceramics for abrasive and chemically aggressive thermal cycling. The simplicity of high purity and structure of this product also facilitate the possibility of materials modification or composites to be created to enhance mechanical or thermal performance.

IV. CONCLUSION

The current research provides significant chemical and thermal analysis of kaolin powder by Thermogravimetric Analysis (TGA) and Fourier Transform Infrared (FTIR) spectroscopy. In accordance with data presented through TGA analysis the sample produced a total weight loss of 5.071%, most weight loss in the temperature range of 400–800 °C because that is when kaolinite fully dehydroxylates to metakaolin. Dehydroxylation is important to enhance the thermal stability as well as significantly enhance the chemical inertness of metakaolin to enable it to be utilized at elevated temperatures. FTIR spectroscopy validated kaolinite phase with a sharp Al–OH bending absorption at 925.87 cm^{-1} and validated no detectable impurities of carbonates or organics. This significantly provided the researchers with confidence regarding mineralogical purity and structural integrity of the tested kaolin material.

Due to this singular ability to compare to literature, the authors assumed that their low weight loss of the kaolin sample also showed a very pure kaolin sample with very few volatile contents; a positive attribute for greater thermal performance in terms of application as a sustainable insulation and refractory. The overall findings suggest that the kaolin powder investigated possesses good thermal stability, workable chemical composition, and promise for limited industrial uses. It possesses the ability to maintain structural stability upon exposure to thermal cycling, high purity composition, and numerous engineering projects have some structural consideration to be suitable starting material for manufacturing thermal insulation bricks and refractory ceramics, among other inclusion project that are heat-resistant. Continuous study of potential minor alterations to, or necessary combinations of kaolin may be investigated to reflect whether either, mechanical strength or thermal conductivity of kaolin may be altered to suit future industrial needs. Taken in totality, these findings establish the potential of this kaolin powder for sustaining energy efficient and long term industrial processes, and advances environmentally friendly and economical thermal management needs.

V. ACKNOWLEDGEMENTS

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A. Appendix A: CIPET Test Report – Kaolin Powder Characterization

This Appendix contains a comprehensive summary of the official laboratory test report (Report No. 142 dated 03.06.2025) prepared by the Central Institute of Petrochemicals Engineering & Technology (CIPET) Raipur on chemical and thermal characterization of the kaolin powder test sample under study in the present research work through Thermogravimetric Analysis (TGA) and Fourier Transform Infrared (FTIR) spectroscopy.

The report is in summary format as follows in the next three sections, Part A – Sample Particulars, Part B – Additional supporting information, and Part C – Experimental Results.

Part A: Sample Particulars

S No	Parameter	Details
1	Sample Name	Kaolin Powder
2	Grade/Type/Class/Size	Not specified
3	Brand Name	Not specified
4	Batch No. & Date of Manufacture	Not specified
5	Physical Appearance	Fine white powder
6	Quantity Supplied	~50 grams
7	Packaging Details	Sealed plastic container (airtight, moisture-protected)
8	Sample Condition at Receipt	Free-flowing, no visible agglomeration or contamination
9	Sample Storage	Desiccator maintained at ambient temperature prior to testing
10	Sample Received Date	01.05.2025
11	Duration of Testing	01.05.2025 – 03.05.2025
12	Laboratory Reference	CIPET, Raipur – Material Testing Division

Part B: Supplementary Information

S No	Parameter	Details
1	Sampling Procedure	Sample supplied directly by the submitting research team (no field sampling involved)
2	Pre-treatment of Sample	Preheated to 80 °C for 1 hour to remove adsorbed surface moisture (as per ASTM guidelines)
3	Supporting Documents	(i) TGA thermogram (22 °C – 1000 °C) (ii) FTIR spectral plot (4000–400 cm^{-1})
4	Analytical Instruments Used	*TGA:* TA Instruments (Platinum HT Model) with N_2 purge (20–50 ml/min) *FTIR:* DTGS detector with KBr pellet method

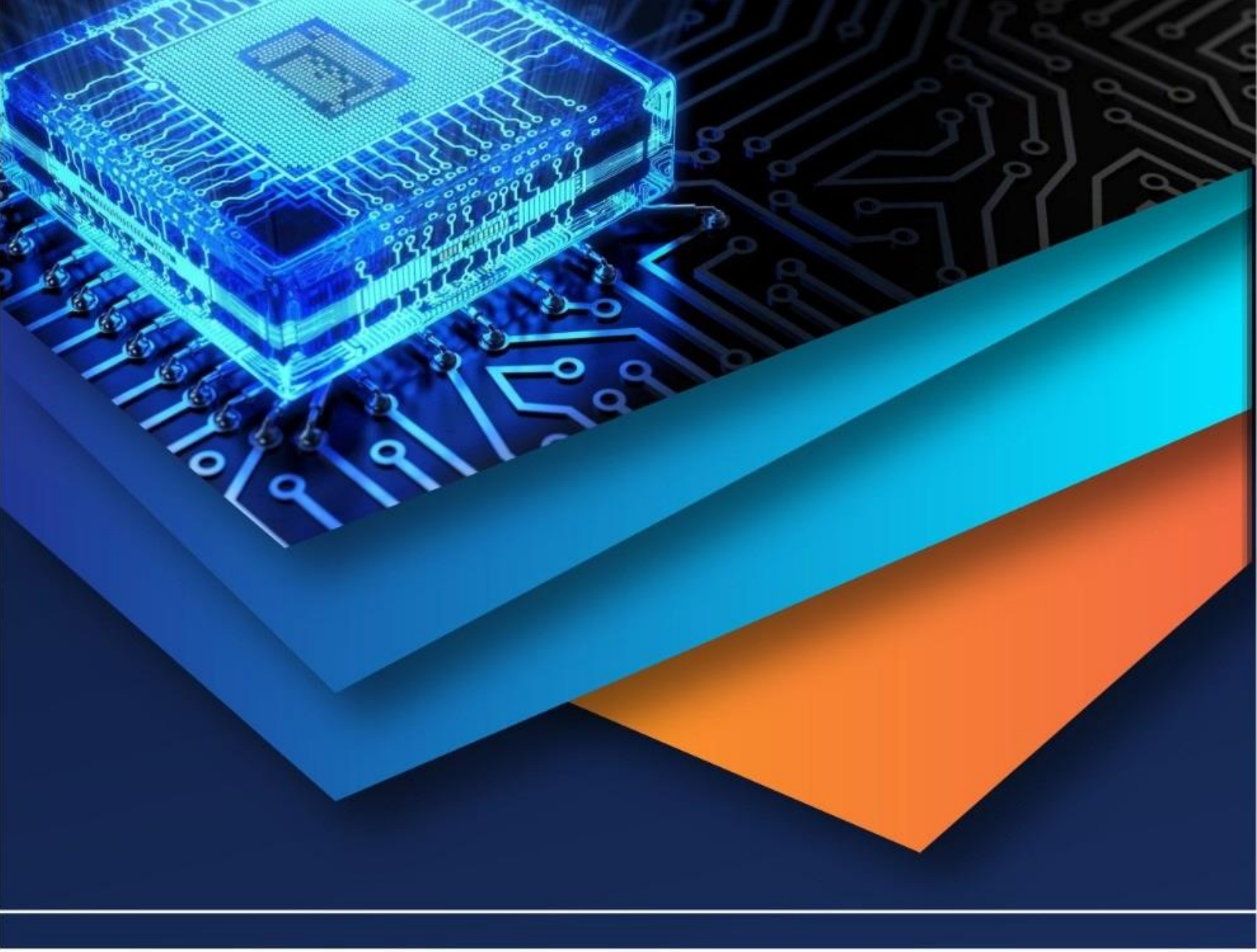
5	Deviation from Test Methods	None reported
6	Subcontracting	TGA performed at NABL-accredited subcontract laboratory; FTIR conducted in-house at CIPET
7	Sample Retention Policy	Retained for 3 months post-report issuance (until 03.09.2025), after which disposal as per laboratory SOP

Part C: Experimental Test Results

S No.	Test	Unit	Standard Method	Result Obtained
1	TGA Analysis	% weight change	ASTM E1131	Weight loss: 5.071% (22–1000 °C) Major loss: 4.354% (400–800 °C, dehydroxylation → metakaolin) Residue @1000 °C: 94.930%
2	FTIR Analysis	—	ASTM E1252	925.87 cm^{-1}: Al–OH bending (kaolinite) 1984 & 2149 cm^{-1}: Overtone bands 1400–1600 cm^{-1}: No impurities Range: 4000–400 cm^{-1} Resolution: 4 cm^{-1}

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