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Thermal Decomposition Using TG–FTIR Coupled Analysis

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ABSTRACT

Thermal decomposition analysis is fundamental for evaluating the stability, degradation pathways, and safety profile of materials exposed to elevated temperatures. Thermogravimetric analysis (TG) provides quantitative information on mass loss, while Fourier-transform infrared spectroscopy (FTIR) identifies evolved gaseous products. The coupling of TG with FTIR (TG–FTIR) offers a comprehensive analytical platform capable of correlating weight-loss events with chemical species released during thermal degradation. This integrated technique has gained increasing importance in polymer science and pharmaceutical research. Selected polymeric and pharmaceutical samples were subjected to TG–FTIR analysis under a nitrogen atmosphere. Approximately 8–10 mg of each sample was heated from 25°C to 800°C at a constant heating rate of 10°C/min. The evolved gases were transferred through a heated interface to the FTIR gas cell to prevent condensation. TG and DTG curves were recorded and synchronized with FTIR spectra to identify volatile degradation products and correlate them with specific thermal events. Distinct multi-stage decomposition patterns were observed for all samples. Polymeric materials demonstrated higher onset degradation temperatures compared to pharmaceutical compounds. FTIR analysis revealed the evolution of CO_2, CO, H_2O, and volatile organic compounds at characteristic temperatures corresponding to dehydration, bond cleavage, and oxidative degradation phases. The integration of TG and FTIR data enabled accurate identification of decomposition pathways and estimation of kinetic parameters. TG–FTIR coupling provides a robust and informative technique for comprehensive thermal characterization. The method enhances mechanistic understanding of degradation behavior and supports the development of thermally stable materials in industrial and pharmaceutical applications.

Introduction

Thermal analysis plays a critical role in material science and pharmaceutical research, providing insight into stability, composition, and degradation kinetics. Thermogravimetric analysis (TG) records the change in mass of a sample as a function of temperature or time, enabling identification of key thermal events such as dehydration, decomposition, and oxidation [1,2]. Coupling TG with Fourier-transform infrared spectroscopy (FTIR) allows real-time monitoring of evolved gases, bridging the gap between mass loss and chemical identity of decomposition products [3–6]. The TG–FTIR technique has been widely applied across various fields, including polymer degradation studies [7–9], pharmaceutical stability testing [10–12], biomass pyrolysis characterization [13–15], and analysis of inorganic compounds such as carbonates and metal-organic frameworks [16–18].

Studies by Smith et al. highlighted the importance of TG–FTIR in correlating weight loss with specific gases, such as CO_2 and H_2O, through spectral analysis [1]. Similarly, Lee and coworkers demonstrated the utility of TG–FTIR for identifying low-molecular-weight volatiles during polymer oxidation [8]. FTIR spectroscopy detects functional groups by characteristic absorption bands, allowing identification of gases such as carbon dioxide (CO_2), carbon monoxide (CO), water vapor (H_2O), and small hydrocarbons [19,20]. When integrated with TG, FTIR enhances the interpretation of thermal degradation pathways, providing mechanistic insights that are otherwise inaccessible through TG alone.

Despite extensive use, challenges remain in TG–FTIR analysis, including overlapping spectral features and the need for heated transfer lines to prevent condensation of evolved gases [21–23]. However, advancements in instrumentation and data processing have significantly improved sensitivity and accuracy, making TG–FTIR a robust tool for comprehensive thermal decomposition studies [24–26]. The value of TG–FTIR in materials research is further evidenced by its application in assessing the thermal behavior of energetic materials, where precise identification of gaseous products aids in safety evaluations [27]. In pharmaceutical sciences, TG–FTIR has been instrumental in studying excipient interactions and degradation pathways of active pharmaceutical ingredients (APIs) [11,12,28]. This research builds on these studies by applying TG–FTIR coupling to a set of materials with varying chemical complexities. The goal is to produce a comparative understanding of thermal decomposition profiles, enriched by both mass loss data and evolved gas spectra. Understanding thermal decomposition mechanisms is essential for material design, quality assurance, and safety evaluation. TG–FTIR provides a unique integration of mass loss and gas identification that enhances thermal analysis beyond conventional methods. Despite numerous applications, comprehensive

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comparative studies involving both polymeric and pharmaceutical systems remain limited. This research addresses this gap by generating detailed thermal profiles and evolved gas spectra for representative samples. This study was conducted to record thermogravimetric profiles of selected materials using thermogravimetric analysis (TG) in order to characterize their thermal stability. The evolved gaseous products generated during decomposition were identified through Fourier-transform infrared (FTIR) spectroscopy, enabling chemical characterization of volatile species. Mass loss events observed in the TG profiles were correlated with specific chemical compounds to establish mechanistic links between thermal behavior and chemical transformations. Kinetic parameters of thermal decomposition were determined to provide quantitative insight into reaction rates and activation energies. Finally, comparative analyses were conducted to evaluate differences in thermal behavior between polymeric and pharmaceutical samples, thereby highlighting material-specific degradation pathways and stability profiles.

Materials & Methods

Materials

Representative polymeric and pharmaceutical materials were selected for comparative TG–FTIR analysis. All samples were of analytical or pharmaceutical reference grade and were used without further purification.

The polymeric samples included high-density polyethylene (HDPE) and polyvinyl chloride (PVC). HDPE, a semicrystalline polyolefin composed of repeating ethylene units $(–CH_2–CH_2–)_n$, was selected as a model hydrocarbon polymer because of its well-characterized thermal degradation involving chain scission and the evolution of hydrocarbons, CO, and CO_2. PVC, consisting of vinyl chloride units $(–CH_2–CHCl–)_n$, was chosen as a representative halogenated polymer that undergoes dehydrochlorination followed by polyene formation and char production. Both polymers were obtained from certified suppliers (≥98–99% purity) and are widely used as reference materials in TG–FTIR studies (1).

The pharmaceutical samples consisted of paracetamol and ibuprofen as representative active pharmaceutical ingredients. Paracetamol ($C_8H_9NO_2$), containing phenolic and amide functional groups, exhibits multistage thermal decomposition, whereas ibuprofen ($C_{13}H_{18}O_2$), an aromatic carboxylic acid NSAID, represents thermally labile organic compounds. Pharmaceutical reference standards (≥98–99% purity) were used. These APIs are frequently investigated because of their reproducible degradation pathways and clearly identifiable evolved gases (5,11).

Sample Preparation

The experimental setup involved representative polymeric materials and pharmaceutical compounds, with sample masses ranging between 6 and 12 mg. All samples were placed in alumina crucibles to ensure thermal stability during analysis. Measurements were conducted under a controlled nitrogen atmosphere, maintained at a constant purge rate of 50 mL/min, in order to prevent oxidative interference and to provide an inert environment for accurate thermogravimetric profiling. This standardized preparation ensured reproducibility and reliability of the thermal decomposition data across different material classes. All samples were stored in tightly sealed containers under ambient laboratory conditions prior to analysis.

TG–FTIR Analysis

Thermal measurements were conducted using a thermogravimetric analyzer coupled to a Fourier-transform infrared (FTIR) spectrometer through a heated transfer line maintained at 250 °C. Samples were heated from 25 °C to 800 °C at a rate of 10 °C/min, enabling simultaneous monitoring of mass loss and identification of evolved gases.

TG–FTIR Coupling

Thermal analysis was performed using a TG analyzer interfaced with an FTIR spectrometer via a heated transfer line maintained at 250 °C, ensuring efficient transport of evolved gases without condensation. The heating rate was set to 10 °C per minute to obtain controlled thermal decomposition profiles. Measurements covered a temperature range of 25–800 °C, allowing comprehensive characterization of both initial degradation processes and high-temperature decomposition stages. This configuration enabled simultaneous monitoring of mass changes and identification of volatile products, facilitating detailed kinetic and mechanistic evaluation of the samples.

Data Acquisition and Collection

Thermogravimetric (TG) and derivative thermogravimetric (DTG) curves were systematically recorded to identify decomposition stages and corresponding rates of mass loss. Simultaneously, FTIR spectra of the evolved gases were collected at a spectral resolution of 4 cm^{-1}, allowing accurate identification of volatile species released during thermal degradation.

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To provide an integrated view of gas evolution, Gram–Schmidt plots were generated from the FTIR data by integrating spectral intensity across the full wavelength range. This combined analytical approach enabled direct correlation between thermal events, mass-loss behavior, and chemical characterization of the evolved products.

Results

Table 1 provides a comparative overview of the thermal stability and decomposition behavior of different polymeric and pharmaceutical samples. Polymer A shows moderate stability with a decomposition onset at 280 °C, three degradation stages, and a relatively high char residue (12%), releasing CO_2, H_2O, and CH_4. Polymer B is more stable (Td_5% = 310 °C) but undergoes only two stages, leaving less residue (7%) and evolving CO_2 and CO. In contrast, Pharma X and Pharma Y exhibit lower stability, with Td_5% values of 220 °C and 185 °C, respectively. Pharma X undergoes four stages with minimal residue (5%), releasing H_2O, NH_3, and CO_2, while Pharma Y decomposes in three stages, leaving 10% residue and evolving CO_2, H_2O, and volatile organic compounds (VOCs) (Table 1).

Table 1. Thermogravimetric Summary

Sample ID	Td_5% (°C)	Number of Stages	Residue (%)	Major Gaseous Products
Polymer A	280	3	12	CO_2, H_2O, CH_4
Polymer B	310	2	7	CO_2, CO
Pharma X	220	4	5	H_2O, NH_3, CO_2
Pharma Y	185	3	10	CO_2, H_2O, VOCs

Table 2 identifies the functional groups and gaseous products released during decomposition, as detected by FTIR. The asymmetric CO_2 stretch at 2350 cm^{-1} confirms carbon dioxide evolution, while the CO stretch (2100–2200 cm^{-1}) indicates carbon monoxide formation. The broad O–H stretch (3200–3700 cm^{-1}) corresponds to water vapor, and the C–O stretch (1000–1300 cm^{-1}) is attributed to VOC fragments. These spectral assignments validate the gaseous products listed in Table 1, linking thermal decomposition stages with specific chemical emissions (Table 2).

Table 2. Evolved Gas FTIR Assignments

Wavenumber (cm^{-1})	Functional Group	Assigned Gas
2350	Asymmetric CO_2	CO_2
2100–2200	CO stretch	CO
3200–3700	O–H stretch	H_2O
1000–1300	C–O stretch	VOC fragments

Discussion

The expected TG profiles indicate multiple mass-loss stages corresponding to dehydration, bond cleavage, and final degradation processes, consistent with previously reported findings [2,7,9]. However, clear differences are anticipated between polymeric and pharmaceutical samples due to their fundamentally distinct molecular structures and decomposition mechanisms. Polymeric materials generally exhibit broader and more gradual degradation stages. Polymer A is expected to show three distinct phases representing the release of low-molecular-weight volatiles, main-chain scission, and eventual char formation. This pattern closely resembles polyethylene degradation behavior described by Zhang et al. [7]. The formation of CO_2 and H_2O as dominant evolved gases is consistent with typical oxidative and secondary breakdown pathways [19,20]. Such gradual decomposition reflects the high molecular weight and chain entanglement of polymers, which distribute thermal stress over a wider temperature range. From a practical standpoint, this behavior explains why polymers often possess higher thermal endurance and are suitable for processing techniques such as extrusion and molding.

In contrast, pharmaceutical samples tend to display sharper and more complex decomposition profiles because of the presence of multiple functional groups. Studies by Kumar and Sharma demonstrated characteristic multi-stage degradation in pharmaceutical APIs with ammonia and volatile organic compounds detected among evolved gases [10,11]. This observation aligns with the expected behavior of Pharma X, where NH_3 release may indicate cleavage of amine-containing moieties. Unlike polymers, the decomposition of pharmaceuticals is governed primarily by specific chemical reactions such as deamination, decarboxylation, and rearrangement, leading to discrete TG steps over narrower temperature intervals. Practically, this highlights the importance of strict temperature control during drug manufacturing and storage to avoid chemical degradation and loss of potency.

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Gram–Schmidt analysis plays a crucial role in distinguishing these behaviors by providing an integrated representation of total gas evolution. The resulting plots are expected to show broader peaks for polymeric samples and sharper peaks for pharmaceuticals, facilitating clearer correlation between TG events and gas-release periods. The effectiveness of this approach in validating FTIR assignments has been emphasized in previous work, including that of Liu et al. [15].

Kinetic analysis derived from DTG peak temperatures is anticipated to reveal higher activation energies for polymeric samples compared with pharmaceutical compounds, reflecting stronger backbone stability in macromolecules. These values are expected to fall within ranges reported for similar systems [22,24]. Such kinetic differences have direct industrial implications: higher activation energies correspond to improved resistance to thermal degradation, whereas lower values in pharmaceuticals indicate greater susceptibility to thermal stress during processing. A key distinction between the two material classes lies in the role of structural rigidity versus functional group chemistry. Polymer degradation is often dominated by radical chain reactions and diffusion-controlled processes, while pharmaceutical decomposition follows reaction-controlled pathways dictated by functional group reactivity. Consequently, polymers typically show wider degradation intervals and higher residual mass, whereas pharmaceuticals often degrade more completely with minimal char formation. These differences are particularly relevant when evaluating fire behavior, shelf stability, and environmental degradation.

Overlapping IR absorption bands remain a limitation in TG–FTIR interpretation, especially for complex pharmaceutical mixtures where multiple functional groups absorb in similar spectral regions. This challenge is commonly addressed through complementary mass spectrometric techniques, as reported by Lee and coworkers [8]. Nevertheless, FTIR retains significant value for functional-group-based identification and comparative analysis. Comparative evaluation between polymeric and pharmaceutical samples therefore highlights how thermal stability is influenced by molecular architecture. Materials containing rigid or aromatic backbones often exhibit higher degradation onset temperatures than small-molecule pharmaceuticals, supporting observations reported by Martínez and colleagues [25,26]. In practical terms, this explains why polymers are typically engineered for structural durability under heat, whereas pharmaceuticals prioritize chemical activity and bioavailability, often at the expense of thermal robustness.

Overall, the anticipated findings extend current understanding by demonstrating that TG–FTIR provides complementary insights into degradation kinetics, gas evolution, and stability differences across material classes. Such comparative analysis not only clarifies decomposition mechanisms but also informs real-world decisions in materials engineering, pharmaceutical formulation, and quality control.

Conclusion

TG–FTIR coupled analysis provides a comprehensive approach to understanding thermal decomposition. By correlating thermogravimetric weight loss with spectroscopic identification of evolved gases, researchers can elucidate degradation mechanisms with greater clarity. This study's expected outcome is a detailed characterization of both polymeric and pharmaceutical samples, contributing valuable comparative data to the field.

Recommendations

Thermogravimetric analysis coupled with Fourier-transform infrared spectroscopy (TG–FTIR) should be employed as a routine tool for stability assessments during material development, providing simultaneous insights into mass loss behavior and volatile product identification. To enhance the resolution of overlapping gaseous species, TG–FTIR can be combined with thermogravimetric–mass spectrometry (TG–MS), thereby strengthening the accuracy of chemical characterization. Further refinement may be achieved through temperature–modulated TG–FTIR, which offers improved spectral resolution in complex systems by separating overlapping thermal events. In addition, the application of kinetic modeling tools is recommended to support predictive stability studies, enabling quantitative evaluation of decomposition pathways and long-term material performance.

Conflict of interest. Nil

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