Comparative study of the Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) of Bio-synthesized Magnesium Oxide Nanoparticles Using Rubber (Hevea braziliensis), Awolowo (Chromolaena odorata), and Oil palm (Elaeis guineensis) leaves Extract

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Abstract

Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) were used to assess the thermal stability of bio-synthesized MgO nanoparticles (MgONPs). TGA revealed that Rubber leaf MgONPs had a multi-step degradation process: initial weight loss at 220°C-325°C, significant loss at 330°C-625°C, with 24.36% residue. Palm Oil leaf (POLE) MgONPs showed initial loss at 300°C-460°C, further decomposition up to 575°C, and 25.5% residue. Awolowo leaf MgONPs had a single degradation phase from 480°C to 790°C, with 28.9% residue, indicating higher stability. DTA analysis showed exothermic peaks at 240°C, 360°C, and 600°C for Rubber leaf MgONPs; 470°C and 560°C for POLE; and 590°C and 660°C for Awolowo leaf MgONPs, highlighting Awolowo's superior thermal stability

1.0 INTRODUCTION

Nanoparticles continue to remain an important and diverse class of materials with applications in all major areas of the economy, including manufacturing, medicine, and energy, with new products finding their way to market [1]. The main classes of nanoparticles utilized in products include carbon-based (nanotubes and fullerenes), metallic (including gold, silver, iron, and copper), metal oxide (MgO, ZnO, TiO_2, CeO_2, SiO_2 among others), and quantum dots [2].

The synthesis and analysis of nanoparticles have become crucial in various scientific fields due to their unique properties and diverse applications. Among the various nanoparticles, magnesium oxide (MgO) has gained significant attention for its roles in catalysis, antibacterial activity, and as an additive in refractory materials [3]. However, traditional methods of synthesizing MgO nanoparticles often involve hazardous chemicals and high energy consumption. This necessitates the exploration of greener synthesis routes, such as bio-synthesis, which utilizes natural resources and offers a more sustainable and environmentally friendly approach [4].

Bio-synthesis of nanoparticles leverages plant extracts, which contain a variety of phytochemicals such as flavonoids, alkaloids, and tannins, acting as reducing and stabilizing agents [5]. In this study, the focus is on the bio-synthesis of MgO nanoparticles mediated by the leaves of Rubber (Hevea brasiliensis), Awolowo (Chromolaena odorata), and Oil palm (Elaeis guineensis). These plants are chosen due to their rich phytochemical content and abundance in tropical regions, making them ideal candidates for green synthesis.

The Rubber tree (Hevea brasiliensis) are primarily known for latex production, but the leaves contain various bioactive compounds [6]. Similarly, Awolowo (Chromolaena odorata), a medicinal plant, is rich in secondary metabolites with potential applications in nanoparticles synthesis [10]. Oil palm (Elaeis guineensis), primarily cultivated for oil production, has leaves that are often considered waste but possess valuable phytochemicals [8]. By utilizing these plant extracts, the synthesis of MgO nanoparticles can be achieved in an eco-friendly manner.

The analysis of these bio-synthesized nanoparticles is critical to understanding their properties and potential applications. Some of the most common methods for nanoparticles analysis include microscopy, spectroscopy, elemental characterization, and particle sizing methods. According to Ikhouria et al. [7], these techniques provide comprehensive information about the nanoparticles' size, shape, morphology, elemental and structural characteristics, and particle sizes.

Microscopy methods such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are essential for visualizing the sample's size, shape, and morphology [10]. SEM provides insights into particle sizes, morphology, and the degree of agglomeration or networking [11], while TEM reveals the internal structure and crystallinity of the particles[12]. Spectroscopy methods like Raman spectroscopy, UV/Visible spectroscopy, and Fourier transform infrared spectroscopy (FTIR) are used to analyze the nanoparticles' electronic structure, surface chemistry, and chemical groups [13]. Raman spectroscopy is particularly useful for determining sizes for very small particles and identifying order/disorder within the structure [14]. Elemental characterization techniques, including the use of X-ray fluorescence Spectroscopy provide detailed information about the elemental composition of the nanoparticles [15]. Particle sizing methods such as dynamic light scattering yield information about the size and shape of the particles [16].

Thermal analysis methods, which measure the properties of the sample as a result of changes in temperature or heat flow, are often used to provide quick information on nanoparticles in manufacturing or laboratory settings [17]. Techniques such as thermogravimetric analysis (TGA) and differential thermal analysis (DTA) are critical for understanding the thermal stability and composition of bio-synthesized nanoparticles. TGA measures the amount and rate of weight change in a material as a function of temperature or time, offering insights into decomposition patterns [18]. DTA complements TGA by detecting exothermic and endothermic transitions, providing a deeper understanding of the thermal properties and stability of the synthesized nanoparticles [19].

The purity and composition of nanoparticles is vital for both their practical applications and for maintaining quality control in manufacturing processes [20]. The wide array of production methods introduces significant challenges, particularly regarding potential impurities or residues that can influence nanoparticles properties. For instance, nanoparticles synthesized through solution-based methods, which involves precipitation from chemical reactions. By-products or residues may become trapped within or adsorbed onto the nanoparticles [21]. In applications such as drug delivery, bio-reduction precipitation technique can encapsulate components of the suspension solution within the nanoparticles themselves

Thermogravimetric analysis (TGA) serves as a crucial technique for assessing nanoparticles composition. TGA measures the sample's weight change as it is heated, offering insights into thermal stability and decomposition patterns [17–22]. This method allows researchers to quantify the mass percentage of specific components present in the nanoparticles mixture, aiding in the identification and quantification of impurities or encapsulated materials [23]. However, a primary challenge in TGA analysis of nanoparticles lies in isolating them from their surrounding solution or matrix. Techniques like centrifugation or other separation methods are often necessary to extract nanoparticles accurately for analysis[24]. Once isolated, heating the nanoparticles sample reveals temperature transitions indicative of oxidation, which are compared to those of pure components to determine chemical composition [25]. TGA plays a pivotal role in nanoparticles analysis by providing essential information on purity, composition, and thermal properties. This capability is crucial for understanding and optimizing nanoparticles production processes, ensuring their suitability for diverse applications in fields ranging from medicine to materials science [26]. This research aims to explore the thermal behavior of MgO nanoparticles synthesized using the extracts of Rubber, Awolowo, and Oil palm leaves. Through a comprehensive TGA/DTA analysis, this study seeks to provide insights into the thermal stability, composition, and potential applications of these bio-synthesized nanoparticles and contribute to the growing body of knowledge on green synthesis methods and their applications in nanotechnology.

2.0 MATERIALS/ METHODOLOGY

Chemicals utilized in this study were magnesium nitrate hexahydrate ($Mg(NO_3)•6H_2O$), sodium hydroxide, double-distilled water, freshly collected Awolowo, Oil palm and rubber leaves. The chemicals were obtained from Sigma Aldrich and were used without further purification

2.1 Sample Preparation

The Awolowo, Rubber and Oil palm leaves were collected fresh. The leaves were properly washed with distilled water to remove any impurities such as sand and dust. The samples were then dried at room temperature till the leaves were properly dried. The dried leaves were blended into fine powder and stored in an air tight container for further use.

2.2 Preparation of leaves extracts

10 grams of each distinct powdered tropical foliage (such as Awowolo, Rubber, and Oil palm leaves) were boiled in 200 mL of distilled water in a 500 mL beaker individually, and agitated at a temperature of 70°C for approximately 30 minutes. The resultant solution was initially filtrated using cotton wool before employing filter paper (Whatman No.1). The acquired extract was preserved in the refrigerator until needed.

2.3 Bio-synthesis of MgO nanoparticles

The green synthesis of magnesium oxide (MgO) nanoparticles using Awowolo, Rubber, and Oil Palm leaves serves as source of phytochemicals for the reduction of magnesium salt into magnesium oxide nanoparticles, follows similar procedure described by Ikhuoria et al. [26, 27]. 2.5 g of magnesium nitrate $Mg(NO_3)_2$ was dissolved in 80 ml of an aqueous extract derived from the leaves in a 250 ml conical flask. The solution was thoroughly mixed for two hours at a constant temperature of 65°C using a magnetic stirrer. To this mixture, 20 ml of 0.1 M NaOH was gradually added drop by drop, adjusting the pH to 8 to facilitate the rapid formation of colloidal particles. The mixture was continuously stirred for an additional hour and then allowed to settle undisturbed. Once the colloidal suspension settled, the supernatant was discarded, and the suspension was collected. To separate the MgO nanoparticles from the solvent, the suspension was centrifuged at 10,000 rpm for 10 minutes. The resulting nanoparticles were then washed repeatedly with distilled water to neutrality dried in an oven at 80°C for six hours. Finally, the dry magnesium oxide nanoparticles were crushed into a powder and stored in an airtight container for further study and use.

2.4 TGA/DTA CHARACTERIZATION OF MgONPs

The thermal behavior and stability of biosynthesized Magnesium Oxide nanoparticles (MgONPs), capped by phytochemicals from Oil Palm, Rubber, and Awolowo leaf aqueous extracts, were investigated through Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA). The experiments were conducted using a Shimadzu Q50 TGA thermal analyzer under a nitrogen atmosphere, with a heating rate of 20°C per minute. Nitrogen was chosen as the horizontal flow gas to prevent additional mass loss due to oxidation of the sample material [28]. This research provides a comparative analysis of the TGA/DTA results for the MgONPs synthesized using the three different tropical leaf extracts.

3.0 RESULTS /DISCUSSION

Table 1
TGA/DTA results of Oil palm, Awolowo and Rubber leaf mediated MgONPs

S/N	Temp. (°C)	TGA (%wt)	DTA (°C/mg)	TGA (%wt)	DTA (°C/mg)	TGA (%wt)	DTA (°C/mg)
		Oil palm leaf		Rubber leaf		Awolowo leaf	
1	200.00	77.13	0.011	88.72	0.016	88.14	0.022
2	250.00	77.35	0.013	84.61	0.030	88.14	0.023
3	300.00	77.38	0.014	82.70	0.014	88.14	0.021
4	350.00	72.60	0.015	76.03	0.020	88.14	0.021
5	400.00	68.39	0.020	64.85	0.064	88.16	0.023
6	450.00	50.63	0.028	64.30	0.017	88.13	0.026
7	500.00	44.72	0.028	86.70	0.018	88.10	0.043
8	550.00	34.81	0.042	48.53	0.021	70.32	0.063
9	600.00	34.53	0.072	44.76	0.028	55.80	0.054
10	650.00	34.06	0.013	40.84	0.006	37.26	0.039
11	700.00	34.06	0.014	36.50	0.003	39.26	0.031
12	750.00	34.06	0.014	35.43	0.003	36.41	0.024
13	800.00	34.06	0.014	35.45	0.004	35.97	0.023
14	850.00	34.06	0.010	35.31	0.005	34.50	0.023

Table 1 show the TGA/DTA results of Oil palm, Awolowo and Rubber leaf mediated MgONPs, it provides critical insights into the thermal stability and composition of materials. This is crucial to ensure materials designed for high-temperature applications remain stable under those conditions. Even slight decomposition at expected operating temperatures can lead to material failure over repeated use, compromising the reliability and longevity of devices made from such materials [27]. The thermal behavior and stability of MgO nanoparticles capped with phytochemicals from Rubber, Oil Palm, and Awolowo leaves exhibit distinct degradation patterns and exothermic peaks, reflecting the unique composition of phytochemicals from each plant source. Rubber and Oil Palm leaf MgO nanoparticles (Figs. 1 and 2) showed multi-step degradation, which can be attributed to the layered nature of phytochemical decomposition. This behavior aligns with the findings of Venkatachalam et al., [28], who reported similar multi-step degradation in Moringa-synthesized MgO nanoparticles. The multi-step process suggests a sequential breakdown of various phytochemicals, each decomposing at different temperature ranges, thereby providing staggered thermal stability. The single-step degradation profile observed in Awolowo leaf MgO nanoparticles (Fig. 3) indicates a more uniform composition of phytochemicals, which decompose more cohesively at higher temperatures. This pattern resembles TGA results described by Mrig, et al., [29] where a continuous weight loss was observed without distinct multi-step phases.

The TGA analysis of MgO nanoparticles synthesized from rubber leaves (Fig. 2) indicates a multi-step degradation process. Initially, a weight loss of 9.09% occurs between 220°C and 325°C, which can be attributed to the evaporation of surface-adsorbed moisture and low-molecular-weight phytochemicals [32]. This -initial phase suggests the removal of volatile components weakly adhered to the nanoparticles surfaces. Following this, a significant weight loss of 50% is observed within the temperature range of 330°C to 625°C. This substantial reduction corresponds to the decomposition and evaporation of stabilizing phytochemicals or biomolecules acting as capping agents on the nanoparticles [33]. The breakdown of these organic components accounts for the major weight loss in this phase. After complete thermal degradation, a residue of 24.36% remains, primarily consisting of stable inorganic MgO, which withstands high temperatures without further degradation. In comparison with MgO nanoparticles derived from oil palm leaves (Fig. 1) exhibit a different thermal degradation profile. The initial weight loss is more pronounced at 33.33%, occurring between 300°C and 460°C. This stage likely represents the decomposition of the initial layer of phytochemicals and the removal of adsorbed water. A further weight loss of 34.61% is observed from 460°C to 575°C, indicating the breakdown of more complex and stable organic components. The remaining residue after thermal degradation is 25.5%, closely aligning with the residue observed for rubber leaf MgO nanoparticles. This similarity suggests a comparable proportion of stable inorganic MgO in the final product, despite differences in the degradation stages

The thermal behavior of MgO nanoparticles from Awolowo leaves (Fig. 3) deviates notably from the other two samples. There was no weight loss observed from 200°C to 480°C and from 790°C to 900°C, indicating the stability of the nanoparticles within those temperature ranges. The degradation occurs primarily in a single, extended phase between 480°C and 790°C, resulting in a continuous weight loss of about 59%. Unlike the multi-step degradation seen in rubber and oil palm leaf nanoparticles, Awolowo leaf nanoparticles undergo a prolonged, single-phase degradation process. The residue left after thermal degradation is 28.9%, slightly higher than the residues for the other two samples. This higher residual content indicates a greater proportion of stable inorganic MgO, suggesting that the nanoparticles from Awolowo leaves may contain more thermally stable components. Magnesium oxide itself (MgO) is a stable compound that has a high melting point (approximately 2800°C) and does not undergo significant decomposition under typical TGA conditions (up to 900°C). Therefore, in TGA studies of MgO nanoparticles, the weight loss observed is predominantly due to the decomposition of the organic stabilizing agents rather than the MgO core itself [34].

DTA thermal analysis methods provide insights into the energy release associated with various physical and chemical transformations in materials. When analyzing MgO nanoparticles synthesized from different leaf sources, the exothermic peaks reveal distinct energy release patterns, reflecting the unique composition and interactions within each type of nanoparticles [35]. The MgO nanoparticles synthesized from rubber leaves exhibit exothermic peaks at 240°C, 360°C, and 600°C (Fig. 2). These peaks indicate distinct stages of energy release associated with specific transformations within the nanoparticles. The exothermic peak at 240°C likely corresponds to the combustion or oxidative degradation of surface-adsorbed organic molecules and low-molecular-weight phytochemicals. The peak at 360°C suggests a further stage of energy release, possibly due to the breakdown of more stable organic components or capping agents on the nanoparticles surfaces. The final exothermic peak at 600°C indicates the energy release from the decomposition of remaining complex organic molecules or the transformation of inorganic components within the nanoparticles [36]s. In contrast, MgO nanoparticles derived from oil palm leaves (Fig. 1) display exothermic peaks at 470°C and 560°C (Fig. 1). The exothermic peak at 470°C signifies a significant energy release phase, likely due to the combustion of initial organic layers or phytochemicals associated with the nanoparticles. This peak occurs at a higher temperature compared to the initial exothermic peaks of rubber leaf nanoparticles, suggesting the presence of more thermally stable organic compounds in the oil palm leaf-derived nanoparticles [37]. The subsequent peak at 560°C indicates an additional energy release phase, possibly associated with the breakdown of more complex and stable organic components. The fewer number of exothermic peaks compared to rubber leaf nanoparticles suggests a more simplified degradation process.

Finally, the MgO nanoparticles from Awolowo leaves show exothermic peaks at 590°C and 660°C (Fig. 3). These peaks are indicative of the energy release associated with the decomposition of highly stable organic molecules or phytochemicals. The exothermic peak at 590°C suggests the breakdown of organic components that are more resistant to thermal degradation [38]. The higher temperature of this peak compared to those observed in rubber and oil palm leaf nanoparticles indicates that the organic materials associated with Awolowo leaf-derived MgO nanoparticles are more thermally stable. The subsequent peak at 660°C represents another stage of energy release, potentially due to the transformation of remaining organic materials or interactions between organic and inorganic components within the nanoparticles.

The TGA results reveal distinct thermal degradation behaviors for MgO nanoparticles derived from different leaf sources. Rubber leaf MgO nanoparticles show an initial low-temperature weight loss followed by a significant mid-temperature loss, indicative of the presence of both volatile and stable organic components [39]. In contrast, oil palm leaf nanoparticles exhibit a pronounced initial weight loss, suggesting a larger quantity of volatile components, followed by a secondary loss of more stable organics. Awolowo leaf MgO nanoparticles differ by undergoing a single, prolonged degradation phase, resulting in a higher residual content of stable inorganic MgO. Furthermore, the exothermic peaks observed in MgO nanoparticles synthesized from different leaf sources reveal distinct thermal behaviors and energy release patterns. Rubber leaf MgO nanoparticles exhibit multiple exothermic peaks at lower temperatures (240°C, 360°C, and 600°C), indicating the presence of various stages of energy release associated with the degradation of both volatile and stable organic components [40]. Oil palm leaf nanoparticles, with exothermic peaks at 470°C and 560°C, suggest the presence of more thermally stable organic compounds, resulting in a simplified degradation process compared to rubber leaf nanoparticles. Awolowo leaf MgO nanoparticles, showing exothermic peaks at 590°C and 660°C, reflect the highest thermal stability among the three types, indicating the presence of highly stable organic materials [41].

These variations in exothermic peaks highlight the influence of the source plant's biochemical composition on the thermal and energy release characteristics of the synthesized nanoparticles. Understanding these differences is crucial for tailoring the properties of MgO nanoparticles for specific applications, particularly those requiring precise thermal management and stability, such as in photocatalytic studies [42, 43]. This comparative analysis of exothermic peaks shows the importance of selecting appropriate leaf sources to optimize the performance characteristics of MgO nanoparticles for various industrial and technological uses.

3.2 CONCLUSION

The comparative TGA/DTA analysis of bio-synthesized magnesium oxide (MgO) nanoparticles derived from Rubber (Hevea brasiliensis), Awolowo (Chromolaena odorata), and Oil Palm (Elaeis guineensis) leaves provides critical insights into their thermal stability and degradation behavior. TGA analysis revealed distinct thermal degradation profiles for the MgO nanoparticles from different leaf sources. Rubber and Oil Palm leaf nanoparticles exhibited multi-step degradation, suggesting the presence of various phytochemicals decomposing at different temperature ranges. In contrast, Awolowo leaf nanoparticles demonstrated a single, extended degradation phase, indicating a more uniform composition of phytochemicals and superior thermal stability. DTA analysis further highlighted unique energy release patterns corresponding to the thermal decomposition of organic components. The multiple exothermic peaks in Rubber leaf nanoparticles indicated complex decomposition stages, while the fewer peaks in Oil Palm leaf nanoparticles suggested a simpler degradation process with more thermally stable compounds. The high-temperature exothermic peaks in Awolowo leaf nanoparticles confirmed the presence of highly stable organic materials, reinforcing their superior thermal stability. These findings emphasize the importance of carefully selecting plant sources based on their phytochemical composition to tailor the thermal properties of MgO nanoparticles. Such knowledge is crucial for optimizing nanoparticles for specific high-temperature applications, where stability and reliability are paramount. Generally, this study contributes to the advancement of sustainable nanomaterial synthesis by demonstrating the potential of using tropical foliage extracts to produce MgO nanoparticles with tailored thermal properties. The insights gained from this research pave the way for further exploration and application of bio-synthesized nanoparticles in various industrial and technological fields, including catalysis, materials science, and environmental remediation.

Declarations

Conflict of interest:

Non applicable

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Author Contribution

Author ContributionsGodfrey Osatohanmwen Otabor: Conceptualization, Methodology, Formal Analysis, Writing – Original Draft.Esther Uwidia Ikhuoria: Supervision, Project Administration, Writing – Review & Editing.Joshua Osaretin Onaifo: Review & Editing.Hilary Ikhazuagbe Ifijen: Review & Editing.Aiyevbekpen Clinton Ehigie:Analysis

Data Availability

All data used to support the findings of this study are included in this article.

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Plates

Plates 1 to 3 are available in the Supplementary Files section

Figures

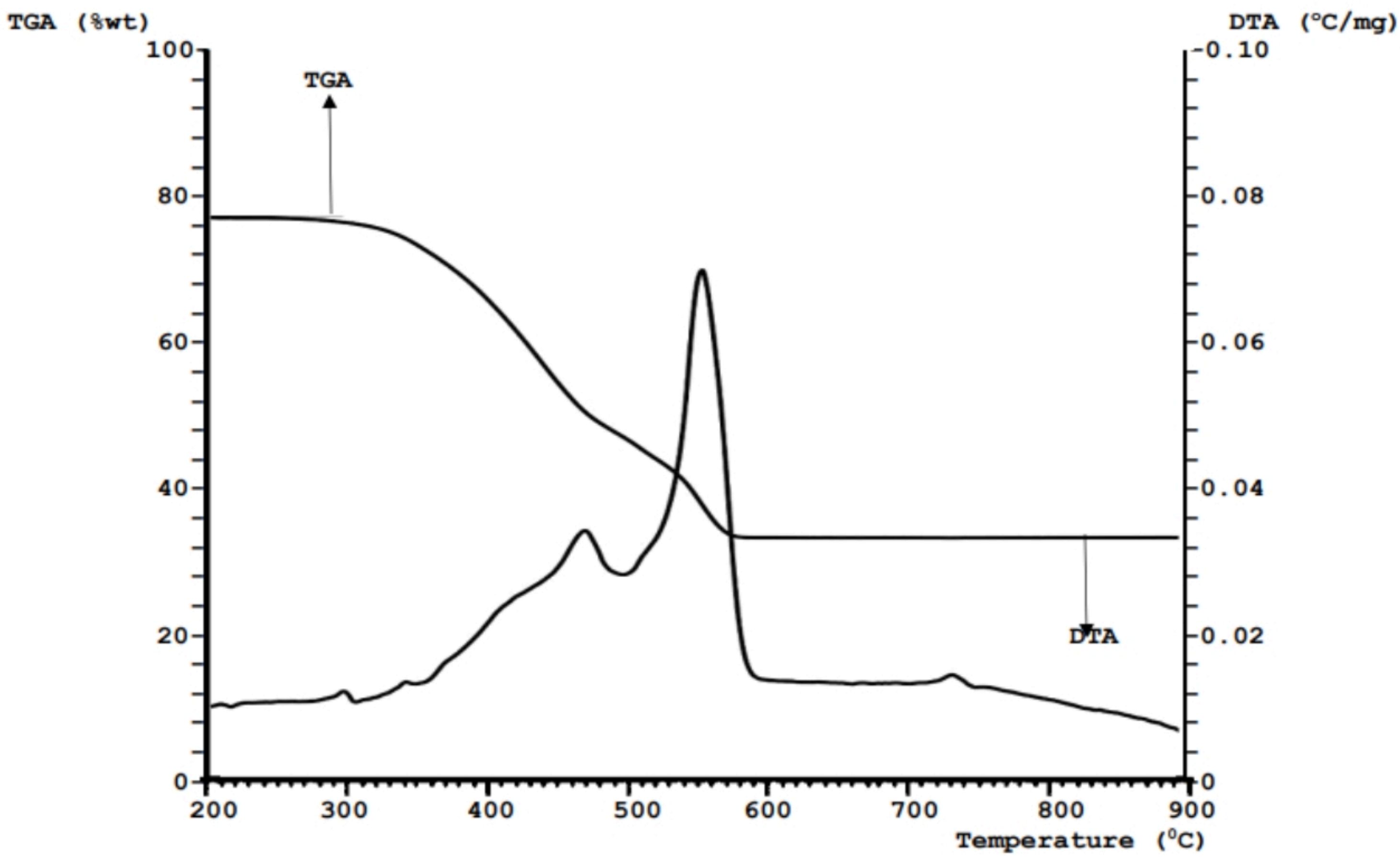

Figure 1

TGA/DTA spectra of Oil palm leaf *(Elaeis guineensis*) (MgONPs) sample

B

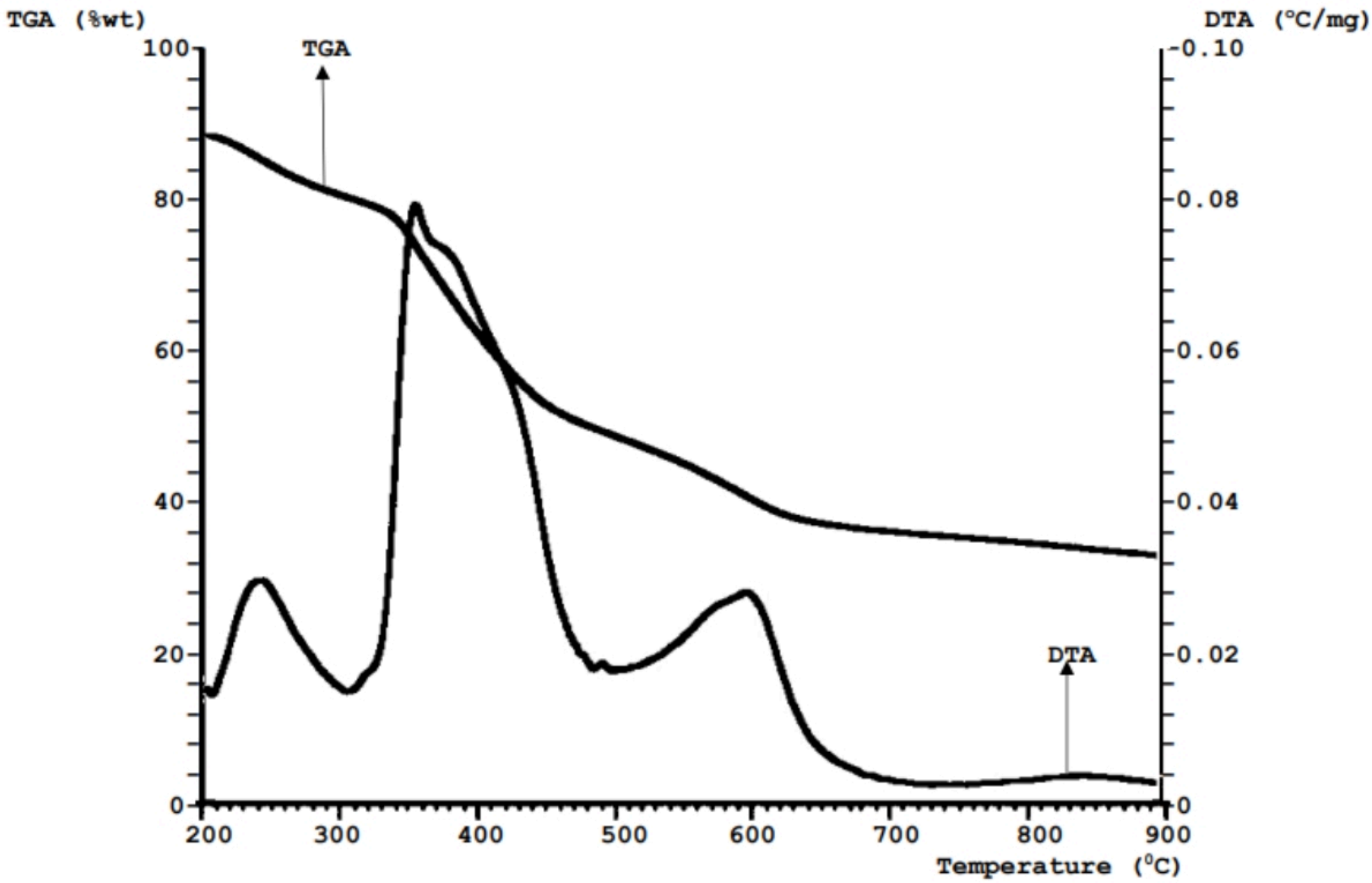

Figure 2

TGA/DTA spectra of Rubber leaf (*Hevea braziliensis*) (MgO)sample

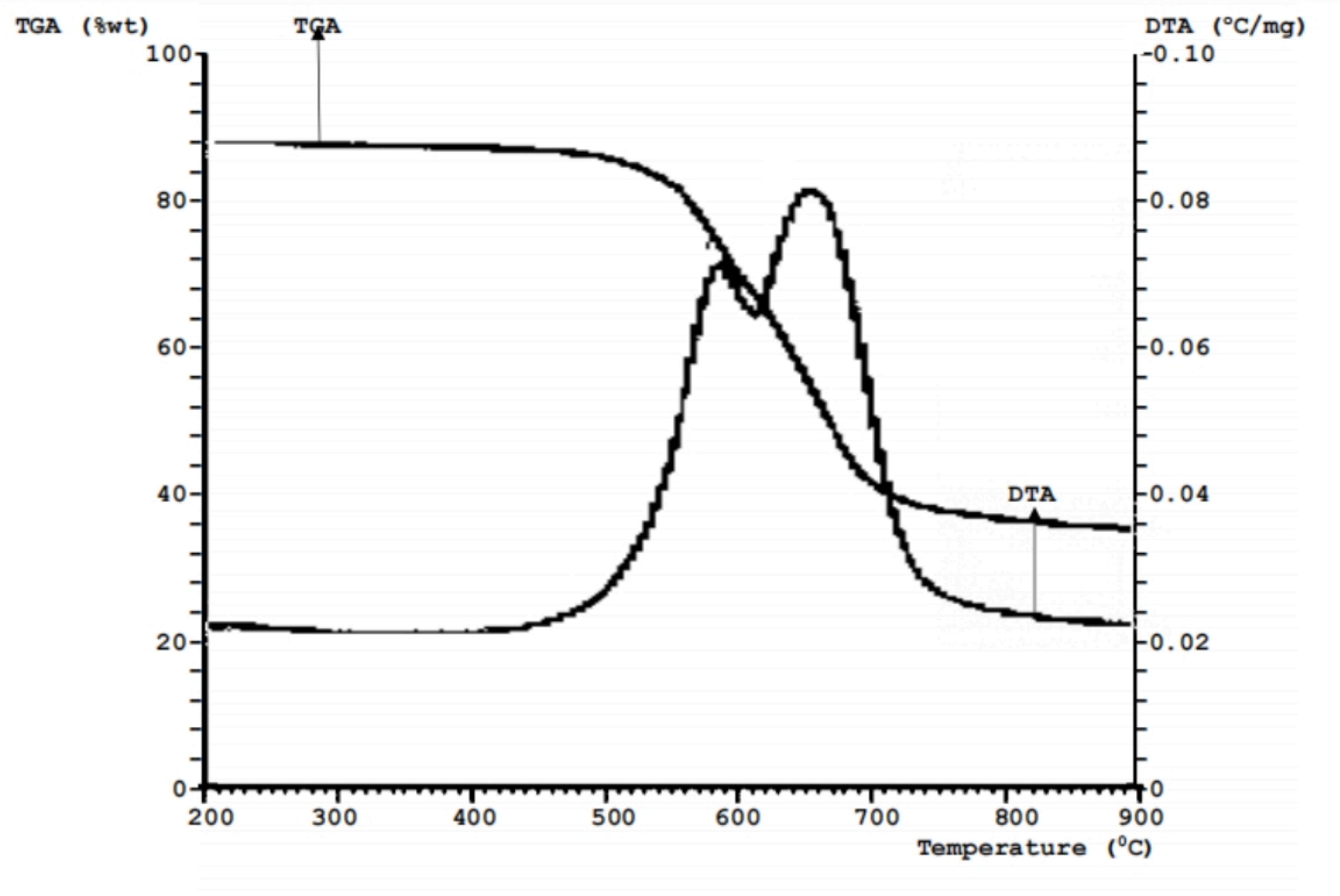

Figure 3

TGA/DTAspectra of Awolowo leaf MgONPs sample

Supplementary Files

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