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Research Article

THERMAL CHARACTERIZATION OF WALNUT SHELL AND KAOLIN ENHANCED POLYPROPYLENE COMPOSITES

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Keywords

Walnut Shell,
Kaolin,
Bio-based Polypropylene Composites,
Synergistic Effect of Fillers,
Thermal Properties.

Abstract

Composite materials for natural-based organic and inorganic fillers have become an innovative class of materials that have attracted attention in recent years. Improving the properties of Polypropylene (PP), which is one of the most widely used thermoplastics due to its advanced mechanical properties, chemical resistance and processability advantages, and reducing the usage rates and environmental impacts of petroleum-based polymers have led to the increasing use of natural-based fillers. In this study, the applicability of walnut shell (W), an agricultural waste, together with kaolin (K), a natural clay mineral, in PP matrix was investigated and the synergistic effects of natural and inorganic fillers were characterised by thermal analyses. Differential scanning calorimetry (DSC) results revealed that kaolin acted as an effective nucleating agent, increasing the crystallization temperature of PP from 115.7 °C to 122.2 °C and enhancing the degree of crystallinity from 48.2% to 53.7%, while the melting temperature remained nearly constant (165–167 °C) for all compositions. Thermogravimetric analysis (TGA) and thermomechanical test results showed that hybrid W/K filled PP composites exhibited improved thermal and dimensional stability, with the highest thermal onset temperature reaching 426.1 °C and reduced coefficient of linear thermal expansion (CLTE) values down to 86.6 μm/m·K, indicating synergistic effects of filler hybridization on thermal resistance and viscoelastic performance. It was observed that the combination of two natural-based fillers with PP can improve the thermal properties of the composites and also has the potential to reduce the negative impact on the environment.

CEVİZ KABUĞU VE KAOLİN KATKILI POLİPROPİLEN KOMPOZİTLERİN TERMAL KARAKTERİZASYONU

Anahtar Kelimeler

Ceviz Kabuğu,
Kaolin,
Biyo Esaslı Polipropilen Kompozitler,
Dolgu Maddelerinin Sinerjik Etkisi,
Termal Özellikler.

Öz

Doğal esaslı organik ve inorganik dolgu malzemeleri için kompozit malzemeler son yıllarda dikkat çeken yenilikçi bir malzeme sınıfı haline gelmiştir. Gelişmiş mekanik özellikleri, kimyasal dirençleri ve işlenebilirlik avantajları nedeniyle geniş bir kullanım alanına sahip olan termoplastiklerin başında gelen Polipropilen (PP) polimerinin sahip olduğu özellikleri daha da iyileştirmek, petrol esaslı polimerlerin kullanım oranlarını ve çevresel etkilerini azaltmak gibi amaçlar, doğal esaslı dolgu maddelerinin kullanımlarının giderek artmasına sebep olmuştur. Bu çalışmada tarımsal bir atık olan ceviz kabuğunun (W) doğal bir kil minerali olan kaolin (K) ile birlikte PP matrisinde kullanılabilirliği incelenmiş, doğal ve inorganik iki dolgu maddesinin sinerjik etkileri termal analizler ile karakterize edilmiştir. Diferansiyel taramalı kalorimetrik analiz (DSC) sonuçları, kaolinin etkili bir çekirdeklenme ajanı olarak hareket ettiğini, PP'nin kristalleşme sıcaklığını 115,7 °C'den 122,2 °C'ye yükselttiğini ve kristallik derecesini %48,2'den %53,7'ye yükselttiğini, erime sıcaklığının ise tüm bileşimler için neredeyse sabit kaldığını (165–167 °C) ortaya koymuştur. Termogravimetrik (TGA) ve termomekanik analizler, hibrit W/K dolgulu PP kompozitlerin gelişmiş termal ve boyutsal stabilite sergilediğini, en yüksek termal bozunma başlangıç sıcaklığının 426,1 °C'ye ulaştığını ve doğrusal ısıl genleşme katsayısı (CLTE) değerlerinin 86,6 μm/m·K'ye düştüğünü göstermiştir. Bu da dolgu hibridizasyonunun termal direnç ve viskoelastik performans üzerinde sinerjik etkileri olduğunu göstermektedir. Kullanılan iki doğal esaslı dolgu malzemesinin PP ile kombinasyonunun, kompozitlerin termal özelliklerini geliştirebildiği, aynı zamanda çevre üzerindeki olumsuz etkilerini de azaltabilme potansiyeline sahip olduğu görülmüştür.

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THERMAL CHARACTERIZATION OF WALNUT SHELL AND KAOLIN ENHANCED POLYPROPYLENE COMPOSITES

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Highlights

- The synergistic effects of walnut shell (W) and kaolin (K) in polypropylene (PP) matrix were investigated.
- The presence of W increased the maximum degradation temperature.
- Thermal stability increased with increasing W content.
- The synergistic effect affected the viscoelastic properties.
- The presence of W and K caused a small increase in crystallinity.

Graphical Abstract

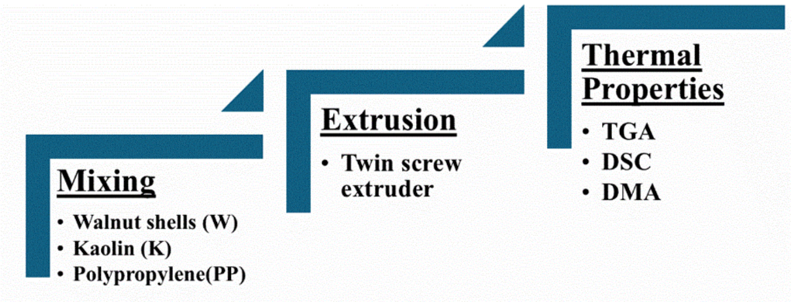

Figure. Flowchart of this study

Purpose and Scope

What are the reason(s) for writing the paper or the aims of the research?
The aim of this study was to investigate the synergistic effects of the combined use of inorganic and organic fillers on the thermal properties of polypropylene.

Design/methodology/approach

How are the objectives achieved? Include the main method(s) used for the research. What is the approach to the topic and what is the theoretical or subject scope of the paper?
In the present study, composite development studies were carried out using twin screw extruder in order to investigate the effects of organic and inorganic based natural fillers in PP matrix. DSC, TGA and DMA analyses were carried out to investigate the thermal properties of walnut shell (W) and kaolin (K) filled PP based composite materials obtained by extrusion method. The present study covers PP-W/K additives, their thermal behaviour, thermal stability and temperature dependent changes in viscoelastic properties.

Findings

What was found in the course of the work? This will refer to analysis, discussion, or results.
As the presence of W and K in the composite structure increases, a general increase in crystallinity ratios is observed, along with an increase in maximum degradation temperatures. Increases in storage and loss moduli were also observed with the addition of W/K to PP.

Originality

What is new in the paper? State the value of the paper and to whom.
In this study, the synergistic effects of using kaolin without any purification process and walnut shells, which are the most common agricultural waste in our country, in PP matrix on thermal properties were investigated. The benefits to be gained from this hybrid use and study will be of special value to our country and the world.

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1.Introduction

Composite materials have been the materials of choice for many years in a wide range of industrial applications from automotive to aviation, packaging to defence industry due to their advanced properties such as strength, stiffness and lightness (Feng et al., 2024). However, social and economic developments that have gained momentum in recent years have dangerously increased the rate of consumption of available resources, increasing ecological risks and the global energy crisis have led to widespread studies on bio-based, more environmentally friendly material groups (Pritchard, 2004). The production of environmentally friendly materials, also called green composites, has received significant attention due to growing environmental concerns, demand for sustainable applications and the use of agricultural waste as a low-cost filler for environmentally friendly polymer composites (Karagöz et al., 2024). In particular, reinforcements and fillers obtained from agricultural wastes, which do not have efficient disposal strategies and do not have any added value, are seen as an important potential for the creation of environmentally friendly green composites (Singh et al., 2023). These agricultural waste materials such as walnut shells, almond shells, hazelnut shells and pistachio shells can contribute to the properties of the composite structure while providing environmental and economic benefits due to their sustainability, widespread availability and low cost potential (Çelik et al., 2021; Kuram, 2022).

Walnut shells, which are easily obtained as a by-product of the walnut processing sector and released in high quantities, are an important group of materials characterised as agricultural waste. Walnut, known as Juglans regia L., is the second most consumed nut in the world after almonds (Çevik and Avşar, 2024; Zhuang et al., 2024). During the harvesting and processing of walnuts, by-products such as the shell (endocarp), which accounts for about 67% of the total fruit weight, are released and more than 1.6 million tonnes of walnut shell waste is produced worldwide each year (Albatrni et al., 2022; Li et al., 2024). Walnut shells, which consist of 25.6% cellulose, 52.3% lignin, 22.1% hemicellulose and 3.6% other components by weight, can be used as lignocellulosic fillers in green composites due to this content (Pirayesh et al., 2012). Lignocellulosic fillers offer indisputable advantages over conventional reinforcing materials such as low density, low cost, recyclability, stiffness and acceptable strength (Joseph et al., 1992; Singha and Thakur, 2009). The use of lignocellulosic fillers can solve the problem of agricultural waste disposal and create a high economic value. In recent years, some researchers have studied the use of walnut shells as fillers in composite materials made of polymers such as thermosetting epoxy resins and polylactic acid (PLA) (Orue et al., 2020; Salasinska et al., 2018). These studies have shown that walnut shells as fillers can significantly increase the tensile strength of composite structures and improve the stiffness and stiffness of the materials.

The development of a bio-based composite structure by adding natural fillers such as walnut shells to a thermoplastic structure such as Polypropylene (PP), which has low density, advanced thermal, mechanical and electrical properties, easy to process, preferred in various industrial applications such as automotive, packaging, aviation, food, etc., is important in terms of further increasing sustainability by adding renewable, biodegradable ingredients to composites (Karian, 2003). In composite development studies with PP, inorganic fillers such as calcium carbonate (Jakubowska and Klozinski, 2015), talc (Wang et al., 2013), fiber glass (Etcheverry et al., 2013) have been used for years. Although the end of the life cycle of the composites used today is of great importance to protect our environment, the use of bio-based additives alone for this purpose may not be sufficient in terms of their compatibility in the polymer matrix and the performance of the composite. For this reason, the use of bio-based fillers in combination with an inorganic additive material is more meaningful in terms of composite performance, and it is known that mechanical and thermal properties can be improved especially in PP-based composites using natural-based clay structures (Hasegawa et al., 1998; Zhou et al., 2005).

In this study, it is aimed to use kaolin, which is widely used in various industrial applications due to its impressive thermal and mechanical properties, together with walnut shell powder in PP matrix. Kaolin, a white, soft clay, has properties such as providing a smooth surface finish, reducing shrinkage and cracking during processing, increasing impact strength and improving the resistance of polymers to weathering and chemical effects (Prasad et al., 1991). Kaolin minerals are known to provide protection against extreme operating temperatures, corrosion, corrosive chemicals, lubricants, vehicle fluids and UV rays (Chaka et al., 2017). Studies have shown that kaolin improves the tensile and flexural strength and impact strength of thermoset-based polymer composites, while its effectiveness in increasing thermal resistance has also been observed (Chaka et al., 2017; Li et al., 2019; Prakash et al., 2015; Şen, 2024). With these studies, kaolin-based thermoset composites can be used in various applications in the automotive industry, including electrical parts, powertrain and transmission components and products exposed to the external environment.

PP-based composites with walnut shell and kaolin additives can exhibit superior performance, especially in areas such as durability, thermal stability and biodegradability. The use of walnut shell, which is rich in lignocellulosic content, together with kaolin, which has proven properties, has the potential to be suitable for use in many industries such as automotive, construction, packaging and consumer products in terms of both sustainability and environmental aspects and the performance of composites. For this reason, in this study, it was aimed to evaluate the thermal properties of the composites obtained by using walnut shell and kaolin additives in PP matrix and the changes in the properties were examined.

2. Material and Method

2.1. Materials

PP with the trade name Buplen was supplied by Lukoil Petrochemical Company/Russia (density: 0.91 g/cm^3 and melt flow index: 7 g/10 min). Raw kaolin were supplied by Dimbos Tarım Ürünleri in Türkiye. Walnut shells were collected locally from Bursa, Marmara region, washed, dried and ground into powder form. Maleic anhydride (MA) with the brand name Lushan PR-3C was supplied from Guangzhou Lushan New Materials.

2.2. Preparation of W/K Filled PP Composites

The sample codes and compound ratios used for W/K filled PP composites are shown in Table 1. A co-rotating intermeshing twin screw extruder (Leistritz 27 MAXX) with 11 zones, 27 mm screw diameter and L/D of 48 was used to produce W/K filled PP composites. The zone temperatures were set as 200, 200, 205, 210, 210, 215, 220, 220, 210, 210, 205, 200 °C; the screw speed and power were set to 400 rpm and 50 kg/h, respectively. After exiting the die, the polymer strands were cooled in a water bath and then cut into pellets using a pelletiser. Test samples were then produced using a Bole/BL90EK model injection moulding machine.

Table 1. The ratios and sample codes of W/K filled PP composites.

Sample Code	PP (wt. %)	MA (wt. %)	K (wt. %)	W (wt. %)
PP	100	0	0	0
PP-K	89	1	10	0
PP-W	89	1	0	10
PP-1K3W	89	1	2.5	7.5
PP-1K1W	89	1	5	5
PP-3K1W	89	1	7.5	2.5

2.3. Thermal Characterizations of W/K Filled PP Composites

2.3.1. Differential Scanning Calorimetry (DSC) Analysis

DSC analyses of W/K filled LDPE composites were carried out using TA Instruments/DSC250 brand/model device to investigate melting and crystallization behaviors and crystallinity percentages in accordance with Equation 1. Tests were carried out in the temperature range of 20 °C to 200 °C with a heating rate of 10 °C/min.

$$X_c = \frac{\Delta H_m x100}{\Delta H°_m(1-w)} \tag{1}$$

While the ΔH_m value in Equation 1 represents the melting enthalpy of PP, the $\Delta H°_m$ value represents the melting enthalpy of the completely crystalline form of the polymer, and this value is accepted as 209 J/g in the literature (Tuna and Akkoyun Kurtlu, 2024). The w in the equation represents the weight ratio of the polymer in the composite.

2.3.2. Thermogravimetric Analysis (TGA)

TGA analysis of PP and the W/K filled PP composites were conducted with a TG analyzer (TA Instruments Inc., Discovery SDT 650). The analyses were conducted at a heating rate of 10 °C/min from room temperature up to 600 °C with a heating rate of 10 °C/min in a nitrogen atmosphere.

2.3.3. Dynamic Mechanical Analysis (DMA)

Dynamic mechanical properties of PP and W/K filled PP composites were analyzed using a dynamic mechanical analyzer Discovery DMA 650 (TA Instruments) in air atmosphere. Tests were performed using a dual cantilever in multi frequency strain modulus mode. The temperature was swept from 30 °C to 120 °C with a heating rate of 3 °C/min. The specimen dimension was 3 x 12.80 x 60 mm^3. The coefficient of linear thermal expansion (CLTE) tests were performed by DMA device with a penetration clamp with 6 mm probe under 1 N load between 30 °C and 90 °C at air atmosphere with heating rate of 3 °C/min.

All comparisons in this study are based on trend analysis of experimental results obtained under identical conditions, and no statistical significance analysis was performed.

3. Results and Discussion

3.1. DSC Analysis Results of W/K Filled PP Composites

The cooling and second heating thermograms obtained in the DSC analysis of PP and W/K filled PP composites are presented in Figure 1. Table 2 consists the details of the crystallization temperature (Tc), melting temperature (Tm), supercooling temperature (ΔT=Tm-Tc), crystallization enthalpy (ΔHc), melting enthalpy (ΔHm) and crystallization degree (Xc) that were obtained from these thermograms. As can be seen in Table 2, the Tc of PP is 115.7 °C, while PP-K has the highest Tc at 122.2 °C. It is evident that the addition of W powder does not have a significant effect on Tc. The Tc values of the hybrid composites range from 116.7 °C to 118.3 °C. The Tc in PP-K composites indicates that kaolin functions as a nucleating agent, promoting earlier crystallisation due to its rigid, inorganic structure (Jikan et al., 2010; Leong et al., 2004; Li et al., 2019). The Tm values demonstrate consistency across all samples, ranging from 165 to 167 °C, thereby suggesting that the presence of fillers does not induce substantial alterations in the melting behaviour of PP. This finding is consistent with the hypothesis that Tm is predominantly influenced by molecular structure of the polymer rather than the quantity of filler content (Guessoum et al., 2012; Li et al., 2019). The ΔT is indicated by the degree of nucleation in the material. It is evident that as these values decrease, the polymer is increasingly influenced by nucleation. The present study lends support to the hypothesis that a decrease in this value is observable as the amount of kaolin in the matrix increases (Şen et al., 2024; Wang et al., 2020). The Xc value of PP indicates the lowest level of crystallinity (48.2%), while all other filled compositions demonstrate higher levels of crystallinity. The composition with the highest crystallinity is PP-1K3W, as 53.7%.

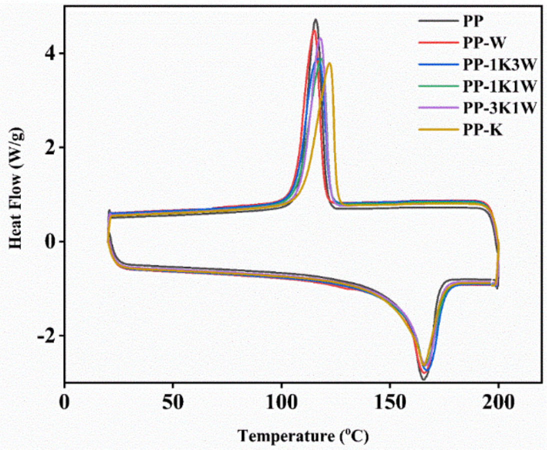

Figure 1. DSC thermograms of W/K filled PP composites.

Table 2. DSC analysis results of W/K filled PP composites.

Sample Code	Tc (^{o}C)	ΔHc (J/g)	Tm (^{o}C)	ΔHm (J/g)	ΔT (^{o}C)	Xc (%)
PP	115.7	100.2	165.5	100.7	49.8	48.2
PP-K	122.2	95.3	165.4	95.0	43.2	51.1
PP-W	115.1	101.1	165.7	97.0	50.6	52.1
PP-1K3W	116.7	102.6	166.9	99.9	50.2	53.7
PP-1K1W	118.3	96.9	166.2	95.3	47.9	51.2
PP-3K1W	117.9	97.5	166.6	94.1	48.7	50.6

3.2. TGA Analysis Results of W/K Filled PP Composites

The TGA thermograms of PP and W/K filled PP composites are presented in Figure 2 and the data obtained from these thermograms for the temperature of 5% mass loss (T5), the temperature of 50% mass loss (T50), the maximum degradation temperature (Tmax), the mass loss between 30 °C and 600 °C (Wloss), and the residue mass at 600 °C are listed in Table 3. The thermograms of W/K filled PP composites demonstrate a single region of mass loss. The T5 of PP commences at 408.6 °C, while PP-K and PP-W exhibit markedly diminished thermal stability (291.7 °C and 396.1 °C, respectively). The hybrid composites demonstrate enhanced T5 values ranging from 409-426 °C. It is noteworthy that PP-1K1W exhibits the highest T5 value at 426.1 °C. The T50 and Tmax values of PP are 450.4 °C and 451.0 °C, respectively, and the addition of W powder generally increases these temperatures (PP-W: T50 = 456.0 °C, Tmax = 459.9 °C). As demonstrated in extant literature, an increase in the Tmax values of natural fiber filled thermoplastic materials has been reported in the presence of natural fibers (Atagür et al., 2022; Sever, 2020). The hybrid composites show similar or slightly better thermal stability compared to PP-W, with PP-K showing the lowest values, indicating reduced thermal stability. As illustrated in Table 3, complete decomposition of PP is evident. The residue amounts of PP-K and PP-W composites were found to be 7.5% and 3.4%, respectively. Analysis revealed that the inorganic ash content was directly proportional to the kaolin content in the hybrid composites. It has been reported in the literature that the thermal degradation of K is a dihydroxylation reaction between 450 °C and 750 °C, and they reported that there was no change in the mass due to the absence of a gas phase in the decomposition reaction at later temperatures (Ptáček et al., 2011, 2010). In the composites examined in these studies, the release of water in the presence of kaolin may have accelerated the thermal degradation of the PP matrix. In this case, the presence of inorganic fillers leads to a decrease in T50 and Tmax values by modifying the thermal degradation pathway of the PP matrix, whereas the higher residue values are associated with the non-decomposing inorganic fraction remaining after thermal treatment. (Chaka et al., 2017; Venkatesan et al., 2023).

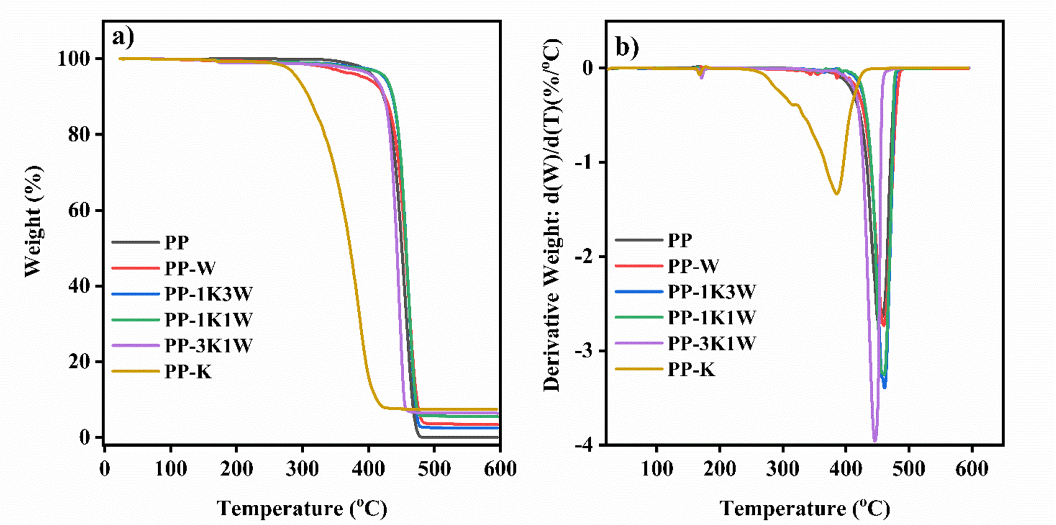

Figure 2. TGA thermogram of W/K filled PP composites.

Table 3. TGA analysis results of W/K filled PP composites.

Sample Code	T5 (°C)	T50 (°C)	Tmax (°C)	Wloss (%)	Ash content (%)
PP	408.6	450.4	451.0	100.0	0.0
PP-K	291.7	372.2	385.4	92.5	7.5
PP-W	396.1	456.0	459.9	96.6	3.4
PP-1K3W	424.3	457.1	461.2	97.5	2.4
PP-1K1W	426.1	457.0	460.6	94.5	5.5
PP-3K1W	409.1	442.9	446.1	93.6	6.4

3.3. DMA Analysis Results of W/K Filled PP Composites

DMA was performed between temperatures of 30 °C and 120 °C in order to evaluate the effect of W/K content on the viscoelastic behaviour of the PP matrix. The thermograms of the temperature-dependent change of the storage modulus (SM), loss modulus (LM) and tan delta, obtained as a result of DMA analysis, are given in Figure 3. A closer examination of the SM thermograms for 10% K, 10% W, and hybrid composites reveals an increase in SM with the incorporation of K and W within the PP matrix material. This increase can be attributed to the presence of K and W, along with their movement within the PP polymer chain (Atagür et al., 2022). Conversely, an increase in temperature was observed to result in a decrease in SM, a phenomenon that was observed across the entire range of PP and W/K filled PP composites. This decline can be attributed to the enhanced thermal motion of the side chain segments within the polymer, which is directly proportional to the increase in thermal energy (Mittal et al., 2019). As demonstrated in Figure 3b, an examination of the temperature-dependent LM changes of PP composites reveals a decline in temperature-dependent LM values across all samples. However, while the decline is minimal between 30 °C and 50 °C, it is observed to be more pronounced between 70 °C and 120 °C. The opposite trend is evident in Figure 3c. This phenomenon can be attributed to the α phase transition that occurs between 50 °C and 60 °C (García-Martínez and Collar, 2022; Hansen et al., 2019).

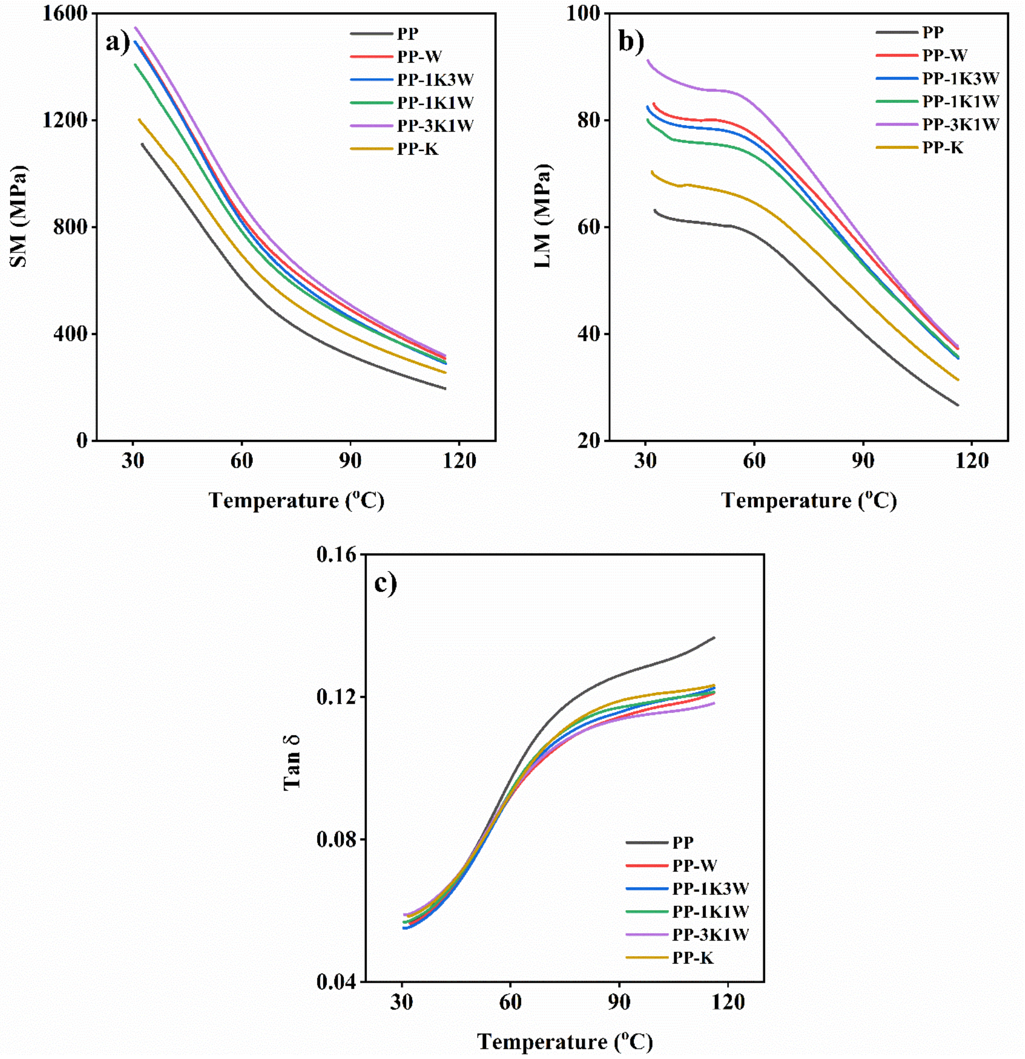

Figure 3. DMA thermogram of W/K filled PP composites.

3.4. Coefficient of Linear Thermal Expansion (CLTE) Analysis Results of W/K Filled PP Composites

The coefficient of linear thermal expansion (CLTE) of composites has been measured both as a function of volume fraction and of filler concentration and temperature (Dey and Tripathi, 2010). The thermograms obtained as a result of the CLTE analysis of W/K filled PP composites are given in Figure 4. The CLTE values of the composites are provided in Table 4. As illustrated in Figure 4, PP exhibits a higher CLTE (126.4 μm/m.K) within the lower temperature range (35-60 °C). Conversely, a significant decline in CLTE (47.7 μm/m.K) is observed at higher temperatures (75-97 °C). This behaviour is distinct from that exhibited by the filled composites, suggesting a potential phase transition or crystallinity change in PP around its softening point. A transition was also identified in the thermogram of PP in Figure 3b, consistent with the findings of Rukmini et al. (Rukmini et al., 2013). PP-K exhibits diminished CLTE at reduced temperatures (86.6 μm/m.K) in comparison to PP, signifying enhanced dimensional stability. Conversely, PP-W demonstrates elevated CLTE values across the entire temperature range in contrast to PP-K, indicating that W powder is less efficacious in constraining polymer chain mobility (Idumah and Hassan, 2016). The hybrid compositions manifest intriguing synergistic effects. The hybrid composition PP-1K3W exhibits the highest overall CLTE (133.1 μm/m.K) across the entire temperature range, while PP-1K1W demonstrates intermediate behaviour and PP-3K1W provides the optimal dimensional stability among the hybrid composites.

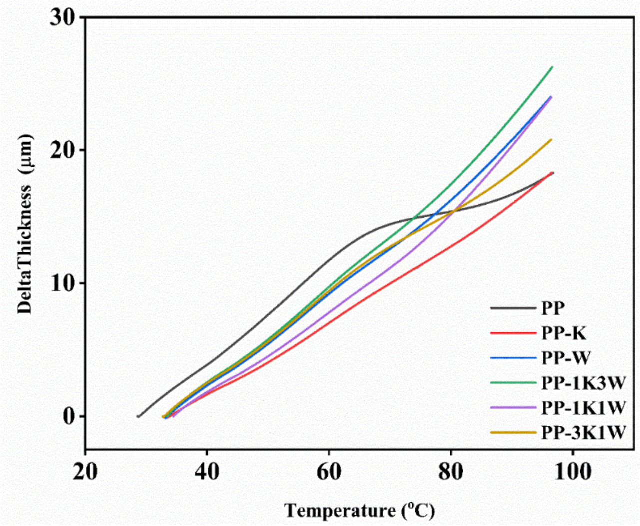

Figure 4. CLTE thermograms of W/K filled PP composites.

Table 4. CLTE values of W/K filled PP composites.

Sample Code	**CLTE (µm/m.K)**		
	35-60 °C	**75-97 °C**	**35-97 °C**
PP	126.4	47.7	85.2
PP-K	86.6	109.0	98.1
PP-W	111.3	152.0	123.9
PP-1K3W	115.3	167.9	133.1
PP-1K1W	97.1	171.9	122.9
PP-3K1W	111.4	104.3	107.9

4. Conclusion

The addition of fillers modifies the crystallization and melting behavior of PP composites. Kaolin acts as a nucleating agent, increasing Tc and Xc, while walnut shell powder enhances crystallization enthalpy. Hybrid composites demonstrate the potential for tailored thermal and mechanical properties by varying filler ratios. TGA analysis demonstrated that the thermal stability of composites incorporating K was lower than that of those incorporating W. In the context of DMA analysis, it was observed that the SM and LM values of filled materials exceeded those of PP material. The values of the CLTE prior to and subsequent to the PP phase transition temperature exhibited a precise inverse relationship to the alterations observed in the filled materials.

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Conflict of Interest

No conflict of interest was declared by the authors.

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