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Thermal and Flammability Characterization of LDPE Based Wood Plastic Composites Reinforced With Variable Size Glass Fiber Powders

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Research Article

Abstract − Wood plastic composites (WPCs) are commercially utilized in building and infrastructure applications; however, their inherent flammability remains a significant concern. While continuous fiber has been investigated to improve thermal behaviour, the effects of fine particulate inorganic fillers on thermal diffusion pathways remain relatively limited. This study aims to evaluate the potential of enhancing the thermal stability and fire resistance of low-density polyethylene (LDPE) based WPCs by incorporating micro-sized glass fiber powder as a reinforcing filler option. WPC panels were manufactured using Scots pine wood flour, LDPE powder, and a maleic anhydride grafted polyethylene (MAPE) coupling agent, with varying concentrations (5% to 30%) and lengths (100 µm and 200 µm) of glass fiber powder. The thermal behavior of the composites was evaluated using thermogravimetric analysis (TGA), while ignition resistance was assessed via the limiting oxygen index (LOI) test.TGA results revealed that glass fiber powder reinforcement improved the thermal stability of the WPCs by contributing to a physical barrier effect, particularly shifting the onset degradation temperature (T_{onset}) to higher values as filler content increased. Formulations containing 200 µm fibers demonstrated enhanced shielding ability at intermediate concentrations, which is suggested to originate from a potentially higher aspect ratio of the larger particles within the polymer matrix. The final inorganic residue increased from 2.59% in the control WPC to approximately 31.03% in the 30% reinforced variations. Correlating with these findings, LOI values rose from 18% for neat LDPE and 23% for the control WPC to a value of 24% for the reinforced composites. The results suggest that the glass fiber powder can serve as a cost-effective additive for improving the thermal stability and ignition resistance of WPCs.

Keywords − *Wood plastic composites, glass fiber powder, thermogravimetric analysis, limiting oxygen index, fire retardancy, thermal stability.*

Farklı Boyutlardaki Cam Elyaf Tozu ile Takviye Edilmiş DYPE Esaslı Odun Plastik Kompozitlerin Termal ve Yanıcılık Karakterizasyonu

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Araştırma Makalesi

Öz − Odun plastik kompozitler (OPK), inşaat ve altyapı uygulamalarında yaygın olarak kullanılsa da yapısal açıdan yanıcılıkları önemli bir sorun teşkil etmeye devam etmektedir. Termal davranışı iyileştirmek için sürekli elyaflar araştırılmış olsa da ince partikül formundaki inorganik dolguların termal difüzyon yollarına etkileri nispeten sınırlıdır. Bu çalışma, alternatif bir takviye dolgu maddesi olarak mikron boyutunda cam elyaf tozu kullanımının, düşük yoğunluklu polietilen (DYPE) bazlı OPK'ların ısıl kararlılığını ve yangın direncini geliştirme potansiyelini değerlendirmeyi amaçlamaktadır. Kompozit paneller; dolgu malzemesi olarak sarıçam odun unu, termoplastik bağlayıcı olarak DYPE kullanılarak üretilmiştir. Takviye dolgu maddesi olarak farklı konsantrasyonlarda (%5-%30) ve uzunluklarda (100 µm ve 200 µm) cam elyaf tozu kullanılmıştır. Dolgu malzemeleri ve termoplastik polimer arasındaki bağlanmayı iyileştirmek için uyumlaştırıcı ajanı olarak maleik anhidrit aşılı polietilen (MAPE) kullanılmıştır. Kompozitlerin termal davranışları termogravimetrik analiz (TGA) ile, tutuşma dirençleri ise limit oksijen indeksi (LOI) testi ile değerlendirilmiştir. TGA sonuçları, cam elyaf tozu takviyesinin bir fiziksel bariyer etkisi oluşturarak OPK'ların ısıl kararlılığını iyileştirdiğini ve özellikle dolgu içeriği arttıkça bozunma başlangıç sıcaklığını (T_{onset}) daha yüksek değerlere ötelediğini ortaya koymuştur. 200 µm uzunluğundaki elyafları içeren formülasyonların ara konsantrasyonlarda sergilediği daha yüksek başlangıç sıcaklıkları, büyük partiküllerin en-boy oranına bağlı potansiyel olarak daha etkin kalkanlama kabiliyeti ile ilişkili olabileceği düşünülmektedir. Nihai inorganik kalıntı miktarı, kontrol grubu OPK' da %2,59 iken, %30 takviyeli varyasyonlarda yaklaşık %31,03'e yükselmiştir. Bu bulgularla paralel olarak LOI değerleri; saf DYPE için %18 ve kontrol grubu OPK için %23 iken, takviyeli kompozitlerde %24 seviyesine ulaşmıştır. Bu sonuçlar, cam elyaf tozunun OPK'ların ısıl kararlılığını ve tutuşma direncini geliştirmek için maliyet etkin bir katkı maddesi seçeneği olabileceğini göstermektedir.

Anahtar Kelimeler − *Odun plastik kompozitler, cam elyaf tozu, termogravimetrik analiz, limit oksijen indeksi, yangın geciktiricilik, ısıl kararlılık.*

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1. Introduction

A composite material has a structure that consists of at least two macroscopically identifiable materials that work together to achieve a better result (Nijssen, 2015). The definition of the composite material actually refers definition of WPCs. WPCs are materials composed of two main components, polymers and lignocellulosic fillers (Kim et al., 2010; Klyosov 2007). While thermosetting resins like phenol formaldehyde were initially used in industrial applications of WPCs, thermoplastics such as polypropylene, polyethylene (PE), and polyvinyl chloride have been widely utilized in production for many years (Ashori, 2008; Panthapulakkal et al., 2006). Lignocellulosic materials have been used as fillers, including wood and agricultural waste, and are annual plants that are renewable, biodegradable, accessible, inexpensive and of low density, as well as being environmentally friendly (Cavus and Mengeloglu, 2020).

WPCs are widely preferred both indoor and outdoor applications such as flooring, cladding, door and window frames, fencing, indoor and outdoor furniture, docks, bridges and railway sleepers, and playground equipment. Moreover, due to ecofriendly approaches, increasing demand and needs, their application areas are continuously expanding (Smith and Wolcott, 2006; Schwarzkopf and Burnard, 2016; Ashori, 2008; Clemons, 2002). The WPC market share was reported to be 7.97 billion USD in 2024, and it is projected to grow at a compound annual growth rate (CAGR) of 11.6% from 2025 to 2030 (URL-1, 2025).

The advantages of WPCs over wood are their workability, they can be moulded easily to produce any desired shape and their resistance to both decay and moisture. They can also be made from 100% waste. However, two key mechanical properties compare unfavorably to cut wood; their strength and stiffness are inferior (Pilarski and Matuana, 2006). Most research has focused on improving WPCs physical, mechanical, and weathering durability. WPCs are also more vulnerable to fire than wood alone, as plastics were made from flammable hydrocarbon materials (Caulfield et al., 2005). The high flame sensitivity of polymer restricted WPC application, mainly indoors and outdoors, due to higher heat during thermal degradation. Heat or temperature differentiations affect WPCs thermal properties (Guo et al., 2019).

Fire behavior of WPCs is a major concern over the decade and much effort has been devoted to assessing and investigating the applications of fire-resistant techniques in reducing their fire hazards. To improve fire performance of WPCs, several fire retardants materials (boric acid, zinc oxide, guanylurea phosphate) are added to the WPC (Zong et al., 2020). Compounds that are efficiently used for fire applications may either consist of a single or combined elements such as bromine, phosphorus, and chlorine (Mokhena et al., 2021; Stark et al., 2009). The use of natural mineral fillers, such as perlite, vermiculite, and expanded perlite and vermiculite (Wang et al., 2016; Szadkowski et al., 2020; Lanzón et al., 2022, Diler et al., 2024), and binders, such as gypsum, geopolymers, and starch, are also effective and have significant effects on flame retardancy of bio based composite panels and wood (Manzello et al., 2007; Bumanis et al., 2020).

Moreover, some studies have also been carried out to improve the thermal stability of WPC with the reinforcement of synthetic fibers. Rasana et al., (2019) stated that glass fibers act as a thermal shield and inhibit degradation. The degradation of carbon fibers above 600 °C, which is higher than polyethylene and wood, improves the thermal stability of WPCs (Eibl, 2017; Guo et al., 2019b; Yao et al., 2018). As in previous studies, the thermal degradation of WPCs was retarded with the reinforcement of glass and carbon woven fabrics and short fibers (Durmaz et al., 2021, Durmaz and Aras, 2022). Moreover, some studies also dealt with improving the thermal stability and fire performance of fibers with aerogels (He et al., 2022; Zhang et al., 2023).

While conventional continuous or woven synthetic fibers significantly improve the properties of composites, their high processing costs and matrix incorporation difficulties restrict their broader industrial use. To address these drawbacks, micronized mineral fillers and short/powdered synthetic fibers have emerged as promising alternatives. Glass fiber powder stands out among these alternatives due to its inherent non-combustibility, exceptionally high degradation temperature (exceeding 1000 °C) (Han et al., 2017), and lower economic footprint compared to woven glass fabrics. In polymer-based systems, fine particulate inorganic fillers can fill the interfacial gaps between the wood flour and the hydrophobic thermoplastic matrix, thereby optimizing heat transfer path disruptions. Recent studies have highlighted that short or powdered glass fibers act as an efficient physical barrier that disrupts the continuous pathway of heat and oxygen diffusion into the matrix interior (Durmaz and Aras, 2022; Turkmen, 2024). Moreover, particulate inorganic reinforcements limit the mass loss rate of volatile organic compounds by stabilizing the char structure during exposure to high temperatures (Durmaz et al., 2023a). Nevertheless, data on the usability of these materials in WPC production is quite limited in the literature.

High thermal stability and fire resistance are essential for the expansion of indoor and outdoor applications of WPCs. The main objective of this study was to improve the thermal stability and fire resistance of LDPE based WPCs by incorporating micro-sized glass fiber powder (100 µm and 200 µm in length) as a cost-effective reinforcing filler. Based on the premise that fine inorganic particulates can disrupt heat transfer networks and hinder volatile mass transfer, it was hypothesized that variations in both filler loading and aspect ratio would systematically alter the degradation kinetics and ignition pathways of the composite matrix. For this purpose, the effect of glass fiber powders used in various concentration ratios (5% to 30%) on the WPCs thermal degradation parameters (T_{onset}, T_{endset}, T_{deg} and final residue) and flammability metrics were systematically investigated via Thermogravimetric analysis (TGA), Differential Thermogravimetry (DTG), and Limiting Oxygen Index (LOI) testing,

2. Material and Methods

2.1. Materials

Commercially sourced Scots pine wood flour (Pinus sylvestris L.) with a mesh size of 20-80 was utilized as the lignocellulosic filler. The thermoplastic matrix consisted of low-density polyethylene (LDPE) powder (Ucar Plastic, İzmir, Türkiye) characterized by a melt flow index (MFI) of 22 g/10 min (190°C/2.16 kg) and a density of 0.919 g/cm³. Due to the inherent polarity of wood and glass fiber particulates, achieving strong interfacial compatibility with the nonpolar LDPE matrix is challenging, often leading to poor wettability and inferior mechanical properties (Lu et al., 2000; Matias et al., 2000; Durmaz et al., 2023). To mitigate this, maleic anhydride grafted polyethylene (MAPE; Licocene PE MA 4351 Fine Grain) was incorporated as a coupling agent. This specific MAPE, with a softening point of 123°C and a density of 0.99 g/cm³, is highly effective for PE based composites (Durmaz et al., 2023). Additionally, two type of glass fiber powders were employed as reinforcing fillers: G100 (Ø13–100 µm length, target density 1.25-1.45 g/cm³) and G200 (Ø13-200 µm length, target density 0.67-0.77 g/cm³).

2.2. Production of WPC

Experimental design of the panel manufacturing that conducted in this study was given in Table 1. To attain a moisture content of less than 2%, wood flour was oven dried at 103±2◦C until constant weight achieved. prior to panel manufacturing. All the components were dry mixed by a mechanical mixer at 1200 rpm and then with a rotary drum blender (30 to 40 rpm) for 5 minutes to be obtained a homogeneous mixture (Durmaz et al., 2020; Durmaz et al., 2022). Then the mixture was laid to a metal draft frame which placed between two aluminum caul plate. Wax paper was used to prevent the draft from sticking to the aluminum caul plates. The draft was hot pressed at 170°C under 100 bar for 15 min (CemilUsta SSP 125) and then allowed to cool slowly inside the press. The pressure was 2.3-2.5 N/mm^2. The target density of the draft was 1 g/cm^3. At the end of the process, the manufactured panels were trimmed to dimensions of 500 mm × 500 mm × 4 mm and conditioned according to ASTM D618-21.

Table 1. The composition of the manufactured WPCs
Tablo 1. Üretilen ahşap plastik kompozitlerin bileşimi

Groups	Glass Fiber Powder 100 µm (%)	Glass Fiber Powder 200 µm (%)	Wood Flour (%)	LDPE (%)	MAPE (%)
Neat-LDPE	-	-	-	99	1
Control WPC	-	-	50	49	1
G100-5	5	-	45	49	1
G100-10	10	-	40	49	1
G100-15	15	-	35	49	1
G100-20	20	-	30	49	1
G100-25	25	-	25	49	1
G100-30	30	-	20	49	1
G200-5	-	5	45	49	1
G200-10	-	10	40	49	1
G200-15	-	15	35	49	1
G200-20	-	20	30	49	1
G200-25	-	25	25	49	1
G200-30	-	30	20	49	1

2.3. Test Procedures

2.3.1. Thermogravimetric Analysis (TGA) Test

Thermogravimetric analysis (TGA) was employed to monitor sample weight changes as a function of temperature and time, while differential thermogravimetry (DTG) was derived from the first derivative of the TGA curves. Thermal stability was assessed using a Perkin-Elmer STA 6000 thermogravimetric analyzer (USA). Prior to testing, WPC samples were ground using an IKA grinder equipped with a 1 mm sieve. The analysis was performed using approximately 4-5 mg of powdered samples placed in aluminum pans. The specimens were heated from 30 °C to 600 °C at a constant rate of 10 °C/min under a nitrogen atmosphere maintained at a flow rate of 20 ml/min. To ensure reproducibility, two independent measurements were performed for each group and representative curves were presented.

2.3.2. Limiting Oxygen Index (LOI) Test

The flammability of the WPCs was evaluated using the Limiting Oxygen Index (LOI) test, a widely recognized method for characterizing the fire behavior of materials using small scale specimens. The LOI value represents the minimum oxygen concentration required to support sustained flaming combustion. Tests were conducted using a Dynisco LOI analyzer (Franklin, USA) in accordance with ASTM D 2863-19. For each group, five specimens (127 mm × 12.7 mm × 4 mm) were positioned vertically in the center of a glass chimney and exposed to a controlled O_2 and N_2 gas mixture. Following atmospheric stabilization within the tube, the specimens were ignited with a pilot flame and monitored for a burning duration of 180 s. or a propagation length of 50 mm. Higher LOI values denote enhanced flame retardancy, whereas lower values indicate higher susceptibility to ignition.

2.4. Statistical Analysis

Statistical differences among the variables were evaluated using analysis of variance (ANOVA) followed by post-hoc Duncan's multiple range test at a $p < 0.05$ significance level. This statistical approach was utilized to determine the significance of the variations the experimental dataset.

3. Results and Discussion

3.1. TGA Analysis

The thermal degradation behavior of the manufactured WPCs was evaluated by TGA analysis. To ensure reliability and experimental reproducibility, the measurements were performed duplicate, and representative curves are presented in Figure 1 and 2. The corresponding thermal degredation parameters are summarized in Table 2.

Table 2. TGA values of WPCs
Tablo 2. Ahşap plastik kompozitlerin TGA değerleri

Groups	Stage	T_{onset} °C	T_{endset} °C	$T_{deg.}$ °C	Weight Loss (%)	Weight onset (%)	Weight endset (%)	Residue (%)
Neat-LDPE	1st	307	497	469	99.735	99.825	0.090	-0.51
Control WPC	2nd	203	382	356	32.941	97.460	64.519	2.59
	3rd	389	499	473	51.124	64.004	12.88	
G100-10	2nd	207	386	358	29.117	97.674	68.557	12.76
	3rd	386	500	470	51.273	68.557	17.284	
G100-20	2nd	219	384	358	21.028	98.591	77.563	22.94
	3rd	384	499	472	51.121	77.563	26.442	
G100-30	2nd	230	385	357	15.001	99.218	84.217	31.03
	3rd	385	500	470	50.269	84.217	33.948	
G200-10	2nd	216	383	359	26.118	97.805	71.687	14.94
	3rd	391	499	498	52.299	71.081	18.782	
G200-20	2nd	225	383	359	21.743	98.276	76.533	21.95
	3rd	387	500	474	51.376	76.368	24.992	
G200-30	2nd	230	381	358	15.007	98.622	83.615	30.38
	3rd	386	497	470	49.785	83.247	33.462	

All samples exhibited a progressive weight loss across three distinct thermal degradation stages as temperature increased (Table 2). The first stage, observed between approximately 50 °C and 200 °C, was associated with the evaporation of absorbed and bound moisture from the wood flour, resulting in a minor weight loss of approximately 3-4%. Although the encapsulation of wood fibers by the polymer matrix restricts water absorption, residual humidity remains within the lignocellulosic structure and is released during this initial phase (Durmaz et al., 2023b). This drying stage proceeded up to the second stage, which corresponds to the main degradation region of the wood components. Lignocellulosic cell wall constituents decompose at different temperature ranges; the thermally unstable hemicellulose initiates degradation first above 170-180 °C, followed by cellulose above 225 °C, and finally lignin above 275 °C (Jarukumjorn and Suppakarn, 2009; Mengeloglu and Kabakci, 2008; Rowell, 2012). In contrast, the third stage was dominated by the thermal decomposition of the polymer matrix; as shown in Table 2 and Figure 1, neat-LDPE displays nearly no weight loss up to 300 °C, representing its typical degradation onset, followed by a major degradation step driven by macromolecular cracking and random chain scission reactions up to 500 °C (Dai et al., 2021).

The glass fibers are manufactured at high temperatures exceeding 1000 °C, meaning their degradation temperatures are significantly higher than both wood and polymer. Consequently, the initial degradation temperature (T_{onset}) of the WPCs systematically shifted toward higher values with increasing glass fiber powder content. For instance, while the Control WPC exhibited a T_{onset} of 203 °C, the values rose to 230 °C for the formulations containing 30 wt% glass fiber powder. Furthermore, a prominent reduction in the main wood decomposition stage (2nd stage) weight loss was monitored, dropping from 32.94% in the Control WPC to 15% in the G100-30 formulation, while the maximum degradation temperatures (T_{deg}) remained highly stable around 356-359 °C for all composite groups. This thermal retardation effect varied depending on glass fiber powder size; the T_{onset} values of WPCs containing 200 µm glass powder were higher at intermediate concentrations compared to those with 100 µm. This behavior indicates that the larger particle size is likely to improve the physical barrier effect, potentially establishing a tortuous pathway that suggestively slows down the heat transfer and delays the overall thermal degradation process. Moreover, the DTG profiles in Figure 2 clearly illustrate that the peak height of neat LDPE is nearly twice that of the WPCs, demonstrating its rapid volatilization rate, whereas the glass fiber powder reinforced groups show lowered degradation velocity rates and stabilized degradation profiles.

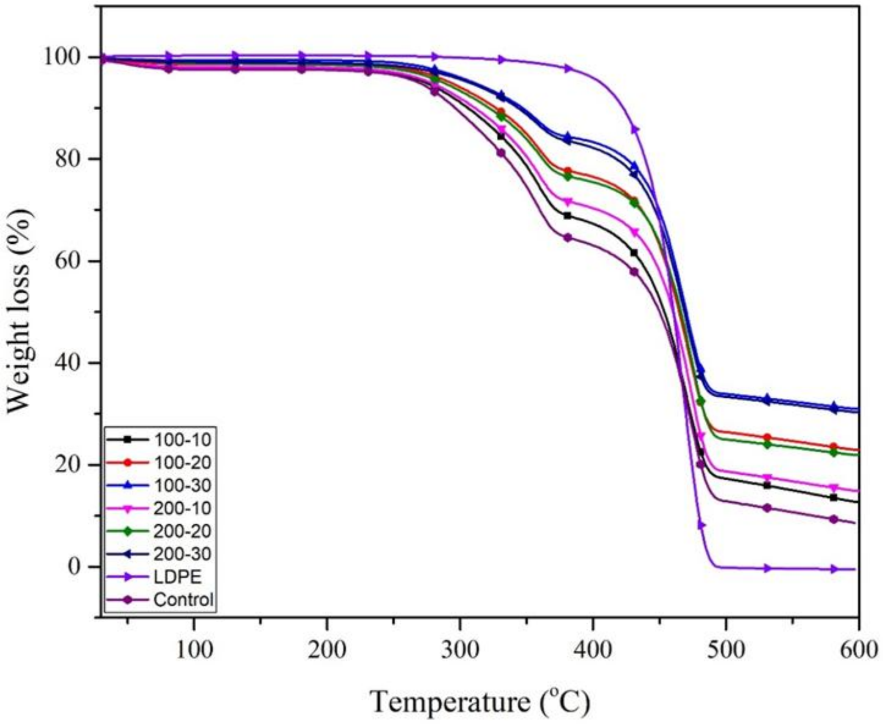

Figure 1. The TGA thermograms of WPCs
Şekil 1. Ahşap plastik kompozitlerin TGA termogramları

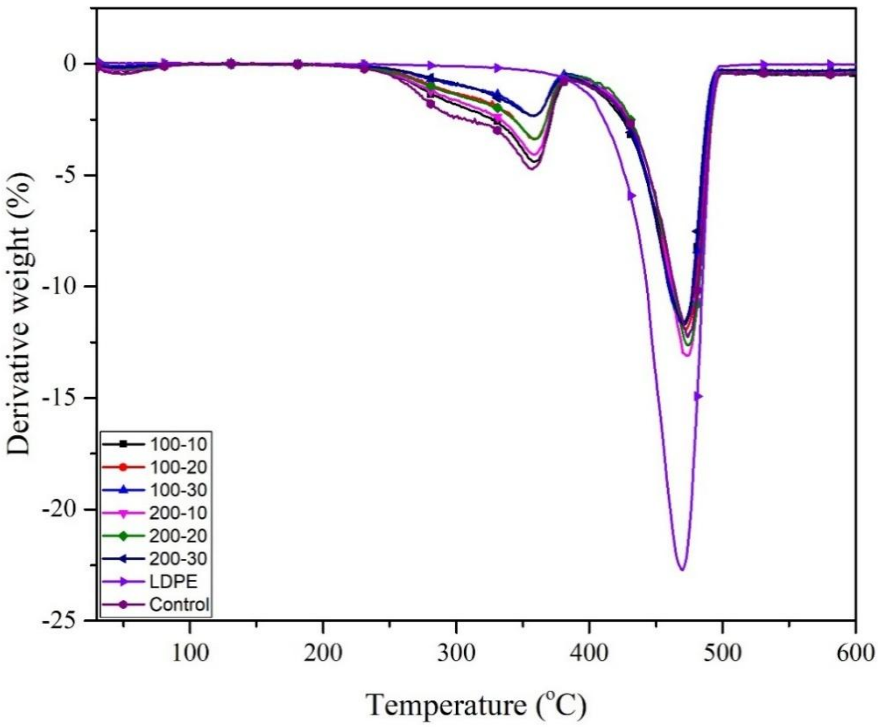

Figure 2. The DTG thermograms of WPCs
Şekil 2. Ahşap plastik kompozitlerin DTG termogramları

3.2. LOI test

The flame retardancy of the WPCs was quantitatively assessed using the LOI test, which identifies the minimum oxygen concentration required to sustain the combustion of a material. Evaluating ignition resistance is crucial for WPCs, as the flammable nature of the thermoplastic matrix often limits their use in building and infrastructure applications. The experimental LOI values for the neat LDPE, the control WPC, and the reinforced with glass fiber powder variations are summarized in Figure 3., while the physical condition of the samples at the end of the test were given in Fig. 4. These results illustrate the transition from a highly flammable neat polymer to a more flame-resistant composite system through the synergistic effect of wood fibers and mineral reinforcements.

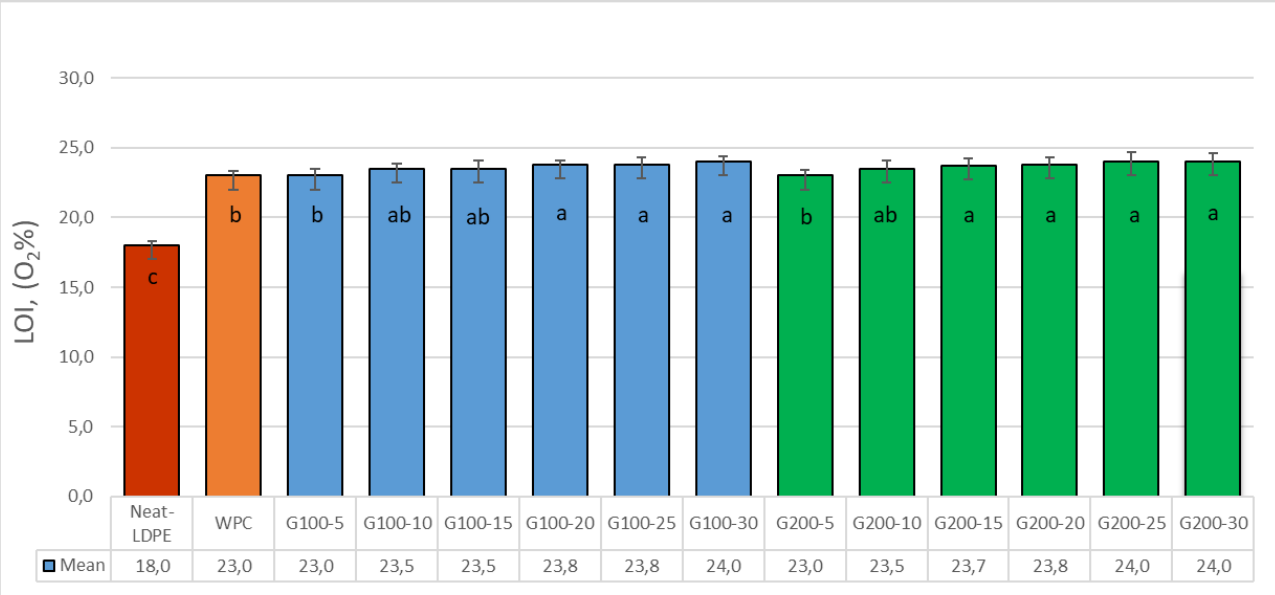

Figure 3. LOI values of WPCs

Şekil 3. Ahşap plastik kompozitlerin LOI değerleri

The fire performance of the WPCs, as characterized by the LOI test, demonstrates a robust correlation with the thermal stability and char forming characteristics observed in the thermogravimetric analysis (TGA). This integrated assessment reveals that the incorporation of glass fiber powder systematically alters the ignition pathways of LDPE based WPCs. While neat LDPE exhibits extreme flammability with a baseline LOI of 18% and undergoes complete thermal decomposition, polyethylene transforms into CO, CO_2, and H_2O volatile gases during the decomposition process (He et al., 2012). The addition of wood flour in the Control WPC increases the LOI to 23% by utilizing the natural char forming capacity of lignocellulosic fibers, which are known to reduce the mass lose rate through surface carbonization (Borysiak et al., 2006; Kozlowski and Wladyka-Przybylak, 2008).

The reinforcement with glass fiber powder in increments from 5% to 30% progressively elevates these values for both the G100 and G200 series, peaking at 24%. According to the statistical analysis in Figure 3, the variations containing 20% to 30% glass fiber powder reached the peak statistical group (“a”), demonstrating a minor yet statistically significant enhancement compared to the Control WPC (“b”). Although this increase confirms a statistical significance, the practical improvement in the absolute LOI value remains relatively modest (from 23% to 24%). This positive trend is quantitatively supported by TGA findings in Table 2 where final inorganic residue reached approximately 31.03% in the 30% reinforced formulations compared to 2.59% in the Control WPC. As can be seen by the tested specimens in Figure 4, this stable residue provides macrostructural evidence suggesting that the inorganic particles assist the carbonaceous char skeleton during combustion.

Figure 4. Post-test macrostructural photographs of WPC specimens after the LOI test
Şekil 4. LOI testi sonrasında WPC numunelerinin test sonrası makroyapısal fotoğrafları

This cohesive surface layer is suggested to exhibit a shielding performance, which may restrict the continuous diffusion of atmospheric oxygen and suppress the mass transfer of flammable volatile gases toward the flame zone, thereby increasing the oxygen concentration required to sustain combustion (Devi and Maji, 2012; Xu et al., 2019; Durmaz et al., 2023a). Notably, the G200 series (200 µm) reached the statistical peak group ("a") at a lower threshold concentration (15%) compared to the G100 series (100 µm), which required a 20% concentration to enter same group. This behavior suggests that the larger particle size provides a more effective shielding ability, which is attributed to the higher aspect ratio of the larger particles that enhances the physical barrier efficiency within the polymer matrix. Consequently, the synergy between the high inorganic residue content and the stabilized char structure is suggested to contribute to stabilizing thermoplastic matrix, supporting of glass fiber powder as a cost-effective reinforcement option for WPCs.

4. Conclusion

This study investigated the potential of utilizing different lengths and concentrations of glass fiber powder to enhance the thermal stability and fire performance of LDPE based wood plastic composites. Based on the experimental results and statistical evaluations, the following conclusions can be drawn:

- The incorporation of glass fiber powder provides a modest but systematic enhancement in the thermal stability of WPCs, which is suggested to be associated with a physical barrier effect that delays the onset of lignocellulosic degradation.
- TGA results confirmed that the final inorganic residue reached up to 31.03% in the 30% reinforced formulations compared to 2.59% in the control WPC, reflecting the systematic retention of the mineral phase at high temperatures.
- Particle size plays a critical role in thermal performance; the 200 µm glass fiber powder (G200 series) exhibited higher onset temperatures compared to the 100 µm series at intermediate concentrations, which is suggested to originate from a potentially more efficient physical barrier performance associated with the higher aspect ratio of the larger particles.

- The ignition resistance of the composites, as measured by the LOI, increased from a baseline of 18% for neat LDPE and 23% for the control WPC to a statistically significant yet practically modest value of 24% with the addition of 30% glass fiber powder.

- Post-test macrostructural evaluations confirmed that the stable inorganic residue integrated with the carbonaceous char skeleton, which may potentially contribute to the structural integrity of the protective surface layer during combustion.

Overall, these findings establish glass fiber powder as a viable and cost-effective reinforcement for optimising the thermal stability and flame behaviour of building grade WPCs. For future research, it is highly recommended to investigate microstructural char morphology via scanning electron microscopy (SEM) and perform bench-scale flammability tests, such as cone calorimetry, to further elucidate the underlying flame retardant mechanisms.

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