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Original Paper

Production and investigation of mechanical properties of ash filled thermoset composites

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ABSTRACT

Fly ash is the mineral residue from the combustion of organic or inorganic materials, sourced from coal, biomass, volcanic activity, and industrial processes. Its composition and properties vary depending on the source and combustion conditions. Ash-filled thermoset-based composites are innovative materials created by incorporating ash into polymer matrices, enhancing mechanical properties and reducing costs while supporting environmental sustainability. These composites are widely used in industries such as construction, automotive, electronics, and packaging. In this study, fly ash obtained from hazelnut tree wood combustion was used as a filler in polyester resin, a commonly preferred material in composites. The composites were prepared by mixing specific proportions of ash with resin and hardened using the open casting method with Teflon molds. Mechanical properties were evaluated through tensile, Izod impact, hardness, and density tests, while microstructural analysis of fractured surfaces was performed via scanning electron microscopy. As a result of the study, it was evaluated that fly ash can be used as a filler in polyester-based composites. Such natural wastes are environmentally friendly, reduce waste management problems, and can provide added value by using them as fillers in composites.

1. INTRODUCTION

The second synthetic thermoset resin discovered after phenolic resin in the early 1940s was unsaturated polyester (UPE) resin. UPE is composed of an unsaturated polyester, a monomer, and an inhibitor [1]. UPE resins are one of the most popular thermosetting matrices used in advanced polymeric composite structures due to their optimized properties and cost parameters, good processing properties, specific physical properties, reasonable price and ability to cure under normal temperature and pressure [2]. It is easy to use in almost all molding-production processes [3]. The curing of UPE resin is a free radical polymerization, i.e. the cured resin changes from a liquid state to a rigid, cross-linked molecular structure that becomes insoluble and infusible, and is highly exothermic in nature [2]. Polyester resins can be broadly classified into two main categories based on their compositions and applications: general-purpose polyester resin and specialty polyester resin [1]. General-purpose resins are made from low-cost raw materials and used without modification. The main criteria for these resins are that they can be offered at a competitive price and provide a network structure with acceptable physical properties within a reasonable time frame. For general purpose UPE resins, material cost is a primary consideration over performance. In contrast, specialty polyesters are formulated with carefully selected raw materials to enhance the properties and performance of the resin, often at a higher cost. Specialty resins are used when high mechanical strength and resistance to chemicals and corrosion are required [4]. Areas of use can be listed as automotive, aviation, marine systems, railway, construction and electrical sectors [5].

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Fly ash (FA) is an industrial waste generated from power plants fueling many manufacturing processes [6]. It is a solid, fine-grained material resulting from the combustion of pulverized coal in power station furnaces. The term FA is not applied to the residue extracted from the bottom of boilers. FAs are notably high in SiO_2, Al_2O_3, and Fe_2O_3, and include other oxides like CaO, MgO, MnO, TiO_2, Na_2O, K_2O, and SO_3 [7]. The composition of FA varies depending on its source, allowing for diverse applications, particularly in civil engineering and construction. Due to its rich elemental content, it is also used as a nutrient source in the soil. FA polymer composites are being developed for various applications, and it functions as an adsorbent for capturing certain pollutants from the air and purifying water. While FA is utilized in many fields, its primary application remains in the construction sector. Utilization of FA as an additive component in polymer composites has received increased attention recently [8]. The unused FA is disposed of in ash ponds and landfills, posing a significant environmental threat and leading to severe health issues such as skin cancer and lung cancer. To address this problem, researchers have proposed utilizing the unused FA as a particulate filler in a polymer matrix for cost-effective and high-volume applications. Reducing the particle size of FA can enhance its surface area by up to 100 times, resulting in improvements in properties such as tensile strength, tear resistance, and modulus in composites. Additionally, FA offers advantages such as excellent processability, thanks to its predominantly spherical shape, and a low cost [6]. In the literature, some studies have been done on the preparation of UPE composites with FA. Rebeiz et al. [9] investigated the effect of adding sand and FA on the strength properties of polyester mortar (PM). The results showed that adding FA and PET waste significantly improves PM quality while reducing costs. Moreover, using these waste materials in PM contributes to energy saving and addresses an environmental issue. Rohatgi et al. [10] use hollow FA particles, known as cenospheres, a low-cost byproduct of coal combustion, as filler in a polyester matrix material. The results show an increase in specific modulus of over 110% with only a slight decrease in strength, indicating significant weight-saving potential in structures using polyester/FA syntactic foams. Zahi [11] incorporated surface-treated FA particles, ranging from 0 to 50 wt%, as a filler in UPE. The findings revealed that the most effective reinforcement was achieved with particles averaging 60 µm in diameter, with micro-hardness increasing as the FA particle content rose. The best performance in terms of impact strength and Modulus of Elasticity was observed with 20-40 wt% of particles. Additionally, the results indicated that FA is a promising filler for enhancing the mechanical properties of the UPE matrix. In this study, it is aimed to evaluate hazelnut wood ash, which is traditionally evaluated as waste, as a filler material in UPE matrix composite materials. The use of lignocellulosic, carbon-rich wastes such as hazelnut wood ash in composites has the potential to reduce material costs, improve mechanical and physical properties and contribute to sustainable production principles. Therefore, the effect of hazelnut wood ash additive on the mechanical properties of UPE composites was investigated in the study. Since there are limited studies in the literature on ash addition as a filler to UPE and no data on the use of hazelnut wood ash in polyester resin matrix, this study will add new data to the literature and contribute to the development of the field of FA-reinforced UPE composites.

2. MATERIAL AND METHODS

In this study, polyester resin developed for general purpose composite applications were used. In addition, Methylethylketone peroxide (MEK-Peroxide), which is widely used in resins such as polyester and vinylester, was preferred as a hardener. FA was used as a filler material and the mechanical properties of the composite were examined. The ash used as a filler material in composite production was obtained from hazelnut wood burned in home stoves using the traditional method (Fig. 1). The burning process was carried out in a controlled manner. The resulting ash was stored at room temperature and protected from moisture. The reason for using ash as a filler material is to eliminate the damage it causes to the environment and to examine its effect on the polyester based composite material. The samples consist of polyester resin and ash filler. A certain amount of hardener was used for each sample. Specimens were prepared with 5%, 10% and 15% ash filler, by weight. This stage is critical to ensure that the resin and ash interact well. The prepared mixture was poured into a Teflon mold. After the casting process, the mixture was kept in the mold for 12 hours to cure on its own, then the samples were removed from the Teflon mold and the excess on the edges of the mold were smoothed. With this process, the samples were ready for standard mechanical tests. Separate samples were prepared as 5 for tensile tests and 5 for impact tests for all filling ratios (0%, 5%, 10%, 15%). The mechanical properties of the specimens were examined by performing tensile test at 5 min/mm tensile speed according to ISO 527 standard, Izod impact test with 5.4 J Izod hammer on notched specimens and hardness test with Shore D device. SEM analysis of the fractured surfaces was also performed.

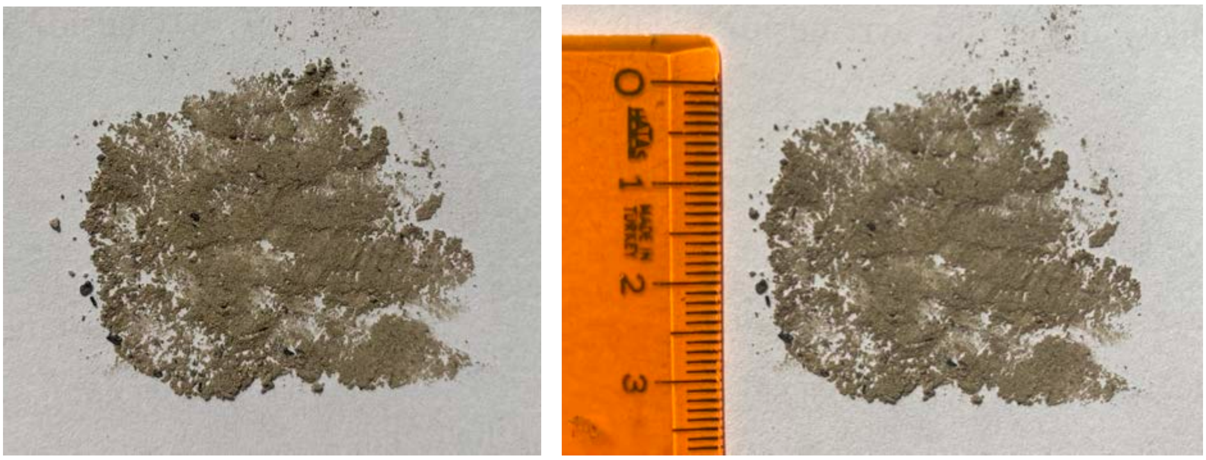

Figure 1. Hazelnut wood ash burned in traditional home stoves

3. RESULTS AND DISCUSSIONS

In this study, the usability of waste ash added to general purpose polyester resin as a filler in polyester-based composites was investigated. For this purpose, composite samples were prepared, and their mechanical properties were examined. As seen in Fig. 2, Shore D hardness test results show that there was a slight increase in the surface hardness of the composites at first and then a significant decrease depending on the increase in the FA additive ratio. While the polyester resin sample without additive had a Shore D hardness value of 68, this value increased to 69 with 5% FA additive; however, it decreased to 67 and 62 Shore D values at 10% and 15% additive ratios, respectively. It is thought that FA additive at low rates increases hardness by acting as a filler in the resin matrix, reducing voids and making the structure more compact. On the other hand, when the additive ratio exceeded 5%, the particles were not distributed homogeneously, showed a tendency to aggregation (clumping) and sufficient bonding was not formed at the matrix and particle interface, causing a noticeable decrease in hardness. In addition, high additive ratios may lead to an increase in microscopic voids within the composite structure and a weakening of mechanical integrity, causing the surface to show lower resistance to the measuring device. As the FA content increases, the decrease in hardness means that the mechanical properties of the material, such as load-carrying capacity and impact resistance, also decrease.

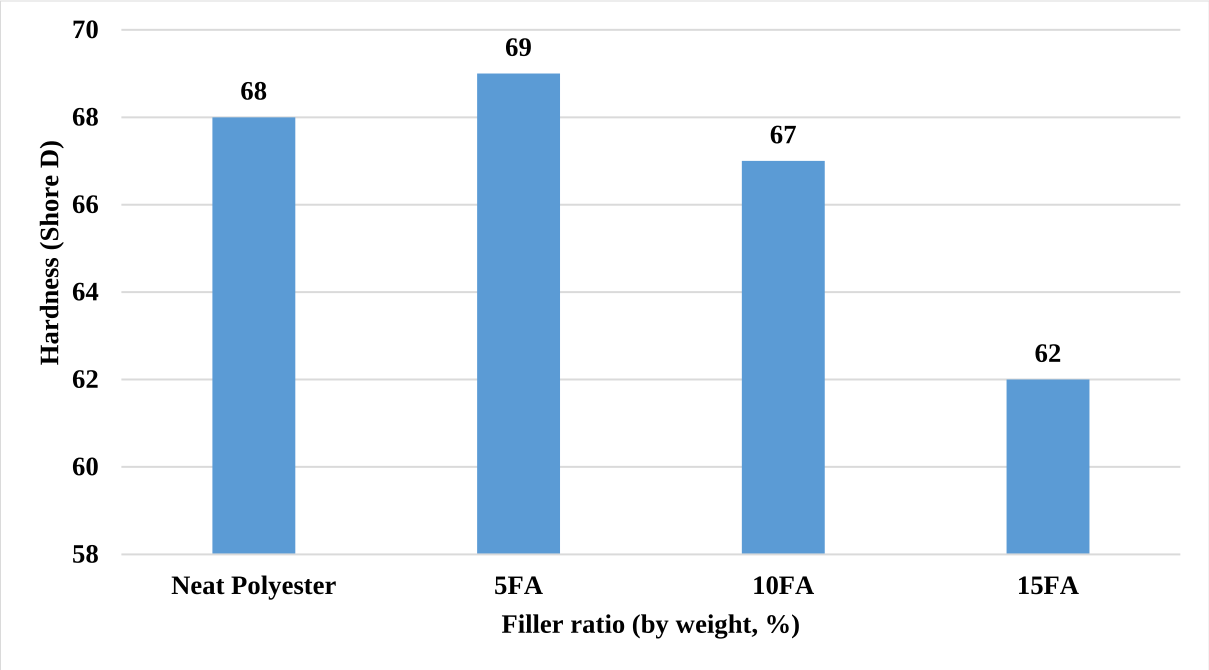

Figure 2. Hardness properties of FA filled polyester resin composites

As seen in Fig. 3, a significant decrease was observed in the impact strength of UPE composites as the FA contribution rate increased. The polyester sample without the additive showed the highest impact strength with 2.40 kJ/m². When the FA contribution was increased to 5%, this value decreased to 2.05 kJ/m², to 1.70 kJ/m² with 10% contribution and to 1.20 kJ/m² with 15% contribution. One of the main reasons for this decrease is that the rigid and inorganic structure of the fly ash particles reduces the ductility of the matrix and makes the composite more brittle. In addition, the insufficient strength of the interface bonds between the FA particles and the UPE causes stress to not be effectively transferred through the particles during impact, thus facilitating fracture. With the increasing FA ratio, the weakening of the matrix-filler interaction and the inhomogeneous distribution of the particle density increase stress concentrations and facilitate crack formation. Especially at 15% additive rate, the weak areas within the material increased because of the clustering of particles and fracture occurred in these areas at the time of impact. As a result, it was determined that the fly ash additive negatively affected the impact resistance and that high additive rates significantly reduced the toughness properties of the composite [12].

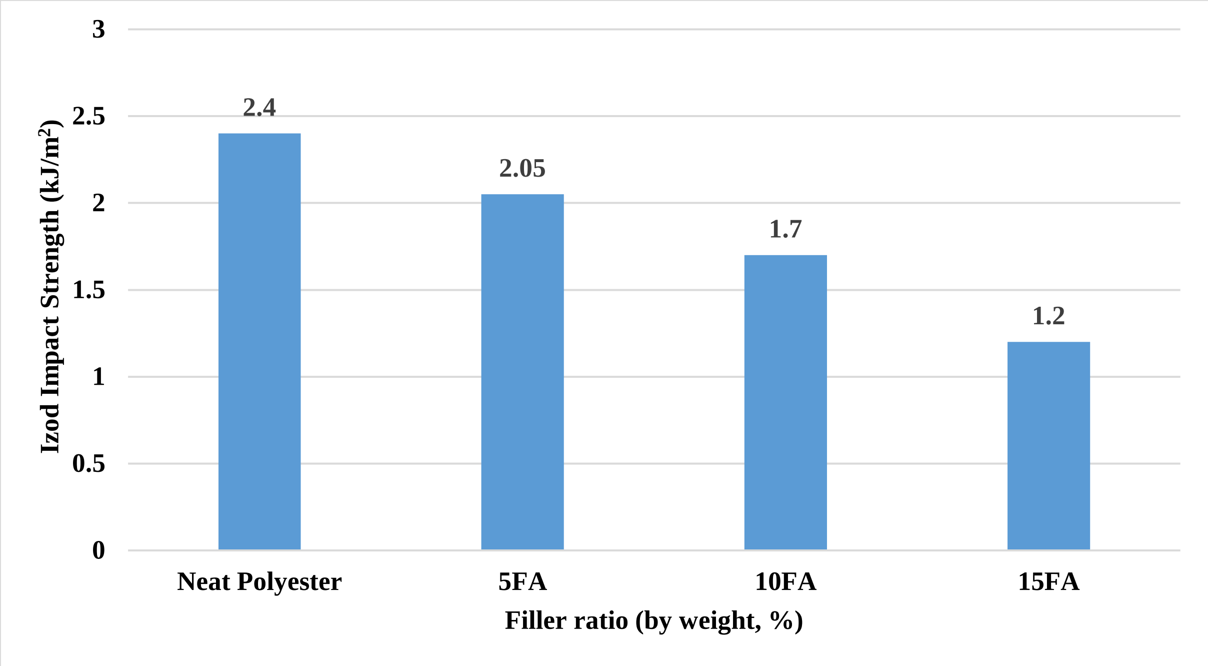

Figure 3. Izod impact strength of FA filled polyester resin composites

Fig. 4 shows the tensile strength of samples with different FA ratios. From these results, it can be concluded that increasing the FA content can increase the tensile strength up to a certain point, but the strength decreases with further increase in this content. It can be observed that a FA content of 5% is optimal for this composite material, because at this percentage the tensile strength reached its maximum value. Higher percentages, such as 10% and 15%, negatively affected the mechanical properties of the material, reducing the strength. Such a trend may be due to the difficulty of uniform distribution of FA in the composite, weakening of the phase interface or other microstructural defects [13, 14].

In Fig. 5, the effects of adding FA additive to the polyester matrix at different weight ratios on the material density are analyzed. The first column shows the density of pure polyester at approximately 1.23 g/cm^3, reflecting the baseline density value of the matrix without additives. With the addition of FA at 5%, the density increased to 1.25 g/cm^3. This increase shows that the additive has a positive effect on the density of the material and that the filler is well integrated into the matrix. When the FA content was increased to 10%, the density reached the highest value of 1.28 g/cm^3. This result indicates that increasing the amount of filler increases the material density and suggests that a certain amount of additive fills the voids in the material and creates a more compact structure. However, when the FA content was increased to 15%, the density decreased to 1.23 g/cm^3. This decrease indicates that high filler ratios can lead to undesirable microstructural changes in the material matrix or that filler-matrix interactions exceed the optimum level after a certain point, creating a negative effect. Furthermore, the overloading of the filler may have made its homogeneous distribution within the matrix difficult, resulting in the formation of voids or weak regions in the structure. Based on this graph, it is thought that the reason for the decrease in mechanical properties is due to the lack of homogeneous mixing.

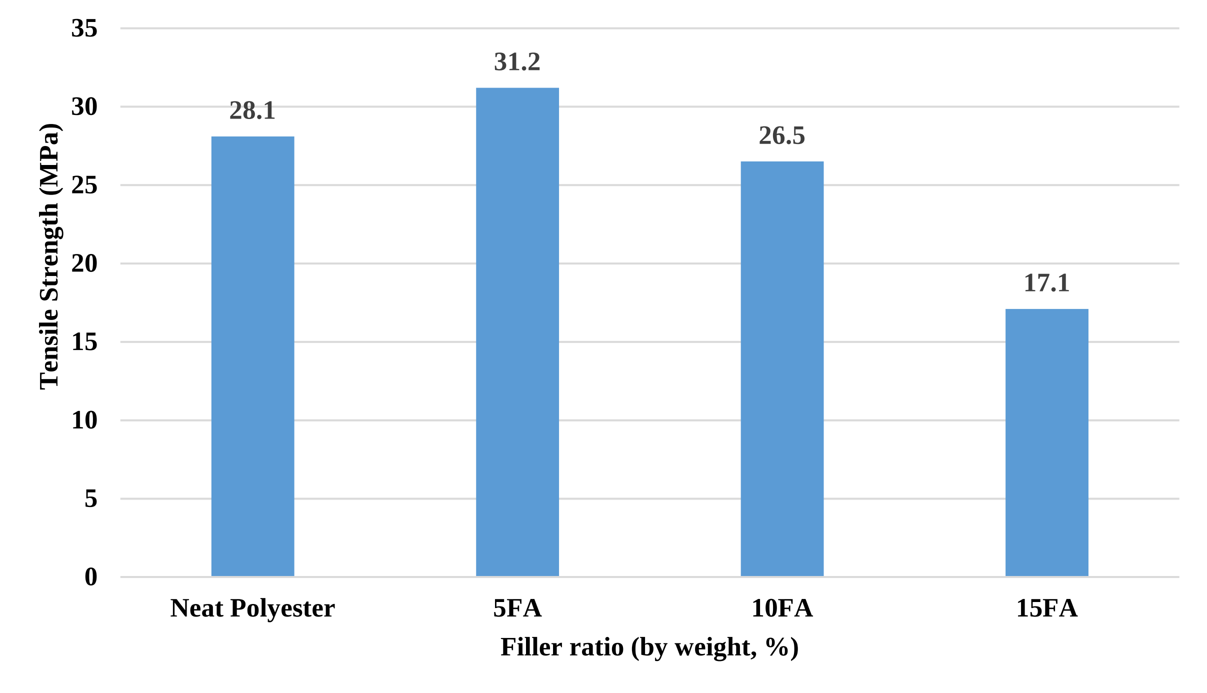

Figure 4. Tensile strength of FA filled polyester resin composites

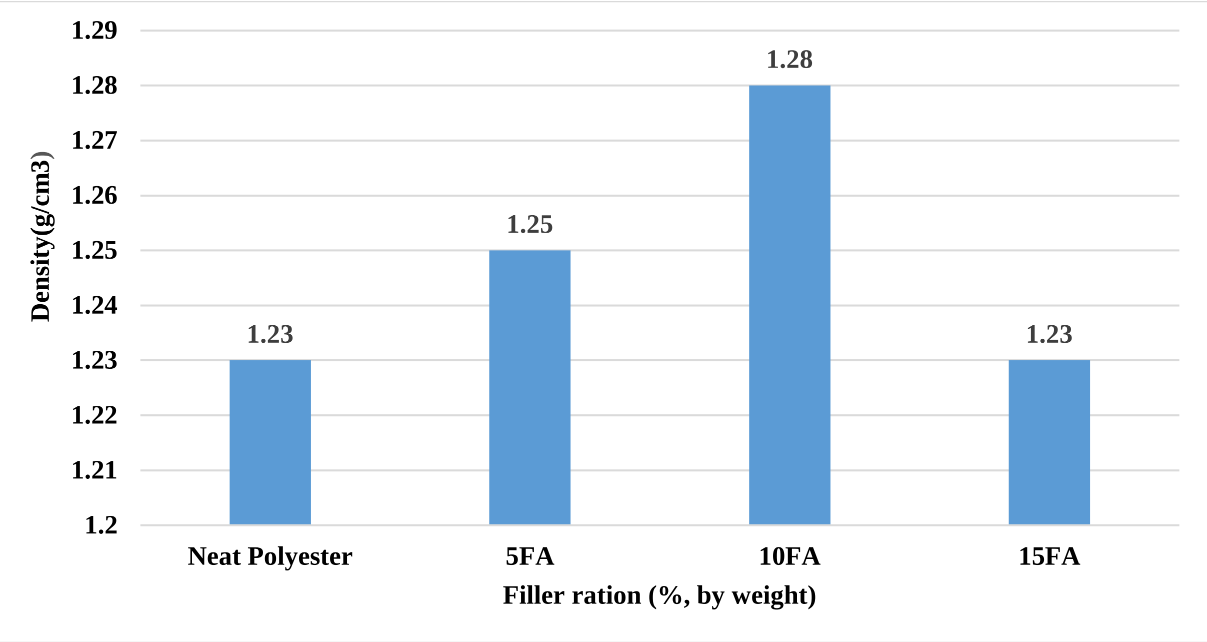

Figure 5. Density of FA filled polyester resin composites

The SEM images shown in Fig. 6 comparatively examine the fracture surface morphologies of pure polyester resin and FA-added polyester resin composite. Fig. 6(a) shows a typical brittle fracture surface of the pure polyester resin. The microstructural features of the polymer are shown in detail with a scale bar of approximately 10 microns. The apparent roughness on the surface indicates that the crack propagates with sudden and limited plastic deformation. This homogeneous surface suggests that the material does not contain significant structural defects at the macro scale. Fig. 6(b) shows the fracture surface of the 10% FA-filled polyester resin composite with a scale bar of 2 microns. On this surface, ash particles dispersed in the resin matrix are clearly observed. The differences in the morphology of the matrix around the ash particles and the tendency of the cracks to deviate or block around the particles indicate that the additive material significantly changes the fracture mechanism. Increased local surface roughness suggests that more energy is absorbed during crack propagation, and therefore there is a potential for increased toughness in the composite. In an ideal situation, particles would be distributed homogeneously, with the aim of providing the desired properties evenly throughout the entire material. However, in this image, areas where particles are clustered can also be observed, indicating that properties may differ in some areas of the material. Such heterogeneities can create weak points in the structure of the material, thus negatively affecting mechanical performance [15].

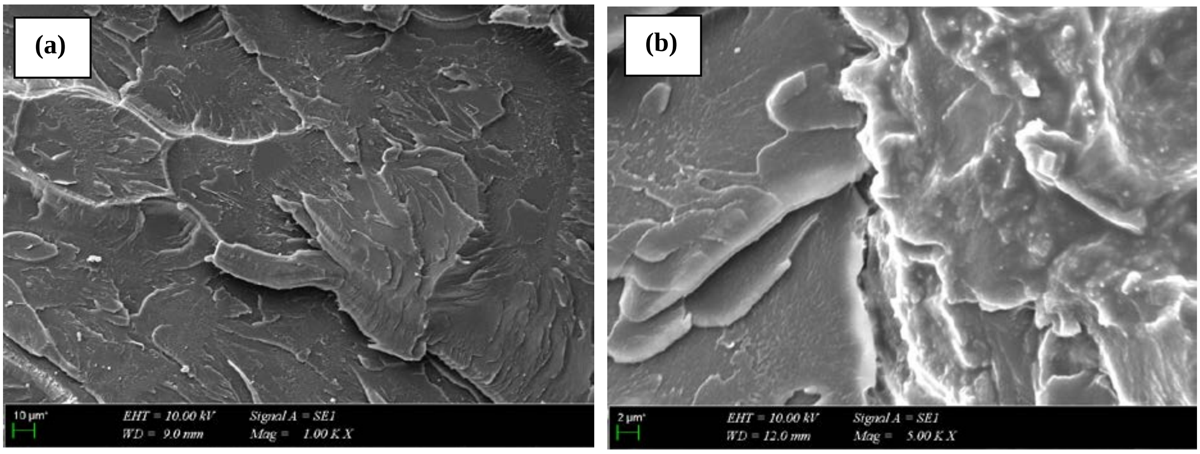

Figure 6. a) SEM image of neat polyester and b) 10% FA-filled polyester

4. CONCLUSIONS

In this study, the usability of general-purpose polyester resin as a filler by adding ash was examined. The results revealed that the ash content significantly affects the material's performance, particularly in terms of hardness, impact resistance, and tensile strength. The hardness tests showed that while the addition of 5% ash slightly improved the material's hardness, further increases in ash content (10% and 15%) resulted in a noticeable decline in hardness. This suggests that although ash can initially enhance the material's rigidity, excessive ash content weakens the overall mechanical structure. Similarly, impact resistance tests indicated a gradual decrease as ash content increased, confirming that higher ash percentages reduce the material's ability to absorb impact energy. Tensile strength tests demonstrated that 5% ash filler produced optimal results, enhancing the material's strength. However, higher ash ratios led to a decline in tensile strength, due to challenges in distributing ash particles uniformly within the polyester matrix. The scanning electron microscope (SEM) analysis confirmed the presence of heterogeneous regions where ash particles clustered, which could explain the weakening of the composite at higher ash levels. In conclusion, this study demonstrates the potential of ash as a filler material in polyester composites, with optimal mechanical properties observed at a 5% ash ratio. While ash offers cost and environmental benefits, its impact on mechanical performance must be carefully managed to avoid degradation of the material's properties at higher concentrations. Further research is recommended to explore methods for improving the uniform distribution of ash particles and to assess the long-term performance of ash-filled composites in various applications.

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