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Fabrication and Flammability Assessment of Hybrid Composite Material Reinforced with Natural Fibers

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Abstract

The utilization of natural fibers in composites has surged owing to their sustainable advantages. Natural fibers, categorized by their origin into plant, mineral, and animal fibers, have garnered significant attention. This study focuses on flax and wool fibers as representatives of natural fibers, paired with epoxy as the matrix material. The properties of flax and wool fibers were reviewed and found that wool has good fire retardancy property while flax is good in mechanical strength. The hybridization approach employed a combination of flax and wool fibers in the fabrication process of a polymer matrix composite, utilizing the hand layup technique. Four specimen were prepared while keeping the total fiber content constant (5%). Two samples were exclusive of flax and wool while rest two were hybridized. The flammability characterisation was conducted through thermogravimetric analysis using vertical burn test setup. The advantage of both fibers were taken in order to make the composite suitable for high end applications. SEM analysis served to characterize both the fibers and matrix in terms of their morphology and composition.

Keywords: Natural fiber, wool fiber, flax fiber, hybrid composite, mechanical properties, fire retardancy

INTRODUCTION

The word 'composite material' refers to those materials which are made up of two or more chemically and physically distinct materials, which has the advantage of their own set of qualities from both materials [1]. The utilization of naturally produced materials in composites is nothing new. Around 1500 BC, Mesopotamians and Egyptian builders and artisans utilized the natural reinforcement for mud bricks, ceramics, and boats [2]. Plant, mineral, and animal fibers are the three types of natural fibers, depending on their origins. However, animal fibers such as hair, silk, and chicken feather, wool have not been used as a reinforcing phase. Wool is a fiber that has been widely used as a raw material for clothing since ancient times. However, there is growing interest in creating new composites that replace synthetic fibers with wool fibers in engineering applications. The reason behind this is wool's unique chemical composition and thermal insulation property. It is composed of a protein called keratin, which contains the leading five elements oxygen, nitrogen, hydrogen, sulphur, carbon, and other elements [3].

Wool fiber is made up of cuticle and cuticle cells that are connected by the cell membrane complex. Epicuticle, exocuticle, and endocuticle layers make up the cuticle. A protein matrix and outer fatty acid monolayer comprise the epicuticle, which envelopes each cuticle cell of the wool fiber. Because fatty acid makes the epicuticle hydrophobic, the wool surface becomes

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hydrophobic. Fatty acid makes the epicuticle hydrophobic, and therefore wool surface becomes hydrophobic. So, even after being hydrophilic, the wool surface has water-repellent characteristics [4]. Wool fiber is mainly obtained from sheep, goats, qiviut (muskoxen), bison, Yak, rabbits, and camelids (Bactrian camel, Arabian camel, Llama, Vicuña, Alpaca, Guanaco). Properties of these different wools vary like their source origin. Wool from alpacas is considerably warmer and lighter than wool obtained from sheep. Depending on the quality of the fiber, alpaca wool thickness ranges from 12 to 29 μm. Alpaca fiber is generally white in appearance; however, it also exists in black and brown tints. The Cashmere goat, also known as the pashmina goat, produces a luscious soft wool fiber. It is not as strong as fine wool. Strong alkalis and high temperatures are damaging to it. To be termed cashmere, the fiber must have a diameter of less than 18.5 μm and a length of at least 3.175 cm.

The thin and soft wool fiber obtained from angora rabbits is called angora wool fiber. Like other natural wool, Angora wool has strong heat retention and water absorption property and it is six times warmer than sheep's wool due to the hollow core of angora hairs. The thickness of good quality Angora fiber ranges from 12-16 μm. Qiviut wool is a high-priced smooth fiber derived from the underwool of muskox, which lies beneath the longer outer wool. They have a 5 to 8 cm length and a diameter of 15 to 20 μm. It is eight times warmer than wool obtained from sheep and eight times softer than cashmere wool. It also does not shrink when submerged in water[5]. Wool obtained from sheep is commonly used due to its availability and cost. Most other wool is dangerous to animal life, such as angora wool from Angora rabbit.

Wool fiber has excellent thermal insulation properties, and it is due to the presence of nitrogen (15-18%) and water content that leads to interference of combustion. Its limiting oxygen index (LOI) is also very high (about 25) among other natural and synthetic fibers. It means wool requires a high minimum oxygen concentration to maintain combustion after igniting the material. Briefly, wool needed more oxygen to burn, making it suitable for applying fire safety. Furthermore, wool fiber is drip-resistant. It does not melt or stick to the skin because of the dehydration reactions and cross-linking of sulfur-containing amino acids present, which aids in char formation during a fire [6]. The heat evolved, flame spread and smoke evolved index are all zero, making the wool flammability index value very low. The lower the value, the better the flammability index. In addition, wool's cell membrane structure, which is cross-linked highly, swells and provides an insulating layer that hinders flame propagation, so it creates char in an intumescent fashion. This self-extinguish property is used in high fire risk environments such as Formula 1 driver's clothes and astronaut's cloth [7]. Several researchers used the self-extinguishing property of wool and tried to form a composite. Guna et al. investigated the wool-polypropylene (PP) composite containing waste wool for applying green buildings and automobiles. The inherent self-extinguishing property of wool provides high flame resistance and obtained the highest fire retardant rating of UL 94 V-0.

No other natural fiber reinforced polypropylene composite had a rating of V-0 [8]. Kim and Bhattacharyya used ammonium polyphosphate (APP) and talc as a flame retardant in wool reinforced polypropylene composite. Ammonium polyphosphate's (APP) high nitrogen content obstructs oxidation, and talc forms the char that boosts fire retardancy. Their study found that with APP into wool reinforced PP composite, the char formation increases and brought down the mass-loss rate. 30% wool reinforced PP shows a 23.4% decrease in the peak heat release rate (PHRR), and with the addition of APP by 20%, PHRR comes down by 74.4% [9]. Fiore et al. studied the wool's reinforcement effect on thermal insulation and mechanical properties of cement composite and obtained that as we increase the amount of wool in composite, its thermal conductivity decreases or thermal insulation property increases [10]. Conzatti et al. added maleinized polypropylene (MAPP) compatibilizer in wool reinforced polypropylene composite and investigated its effect on fiber-matrix adhesion. Thermal stability improves with MAPP compatibilizer due to better interaction between wool fiber and polypropylene [11]. Subasinghe et al. studied the thermal performance of wool/kenaf/polypropylene composite. Flame retardant characteristics improve with the addition of wool. PP/Kenaf/APP/Wool system forms more residual char than other composite samples [12]. Das et al. studied the mechanical

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and flammability performance of the biochar/wool hybrid PP composite. Adding wool into biochar increases the LOI values and delays the early onset of ignition. The hybridization of wool and biochar effectively maintains the flexural strength and improves tensile and flexural moduli while significantly improving flammability [13,14]. A suitable hybridization of fibers minimizes the disadvantage of individual and maximizes the advantages [15,16].

In this research, sheep wool's suitable flame retardant property is investigated with the hybridization of flax fiber. Flax fiber is a plant fiber that comes under bast fiber. It is extracted from the epidermis of the stem of the flax plant[11] . A similar composite was fabricated with wheat gluten bioplastic in which lansol was used to improve the fire retardancy while biochar was used for mechanical strength[17]. Flax fiber is lustrous, soft, flexible, and more robust than cotton but less elastic. It is due to the crystalline structure of flax that makes them stronger and stiffer. Pectin, hemicellulose, lignin, and cellulose are the constituents of flax fiber. Each fiber of flax is composed of concentric cell walls at the microscopic level. The lumen is located at the center of the crude fiber, the small open channel in the concentric cylinder. The outermost layer is known as the primary cell wall. Its function is to coat the secondary walls. The fiber's strength is determined by the secondary wall (S2). It is the thickest layer of all and contains many crystalline cellulose micro-fibrils[12] .

MATERIALS AND METHODS

Fabrication of composite

Wool and flax fiber were taken for reinforcements which are shown in Figure. 1. Flax fiber is one of the most utilized natural fibers in structural applications [18]. The high strength-to-weight ratio of flax makes it an excellent alternative to synthetic fiber in applications like automobile, aircraft parts, construction, and sports products. However, the issue associated with flax fiber-reinforced composite is thermal degradation. The safety of people from fire is an essential factor. That's why to gain the benefit of mechanical properties of flax fiber composite without compromising on the thermal aspect. Wool fiber is incorporated, which is a naturally flame-resistant fiber. The physical properties of both fibers are given in Table 1 [19,20]. Araldite LY 556 with hardener HY 951 was used as a matrix. The properties of these material are given in Table 2 [21,22]. The hand lay-up technique was used to fabricate hybrid and non-hybrid composite with a constant fiber content of 5%. Blending ratios for the various sample of composite are mentioned in Table 3. Araldite and hardener were mixed in a 10:1 ratio by weight and stirred manually. Then the required dry fiber was weighed on a weighing machine and dipped and blended in the matrix solution uniformly. The fiber and matrix amalgam is placed inside the female mold and spread uniformly. A mold sheet was applied to the amalgam, and a roller was used to extend the mixture more uniformly to avoid void formation. The male part of the mold was placed over the female part, and pressure was applied for 45 minutes using a load of around 5 KN. Finally, the sample was left untouched for 48 hours for curing at room temperature. Three sample were prepared for each fiber –matrix composition of composite and results were prepared as an average of three. The step-by-step procedure of fabrication of composite is shown in Figure. 2.

Table 1. Physical properties of fibres.

Properties	Wool fiber	Flax fiber
Elongation at a break (%)	25-40	2.7-3.5
Aspect ratio	8000	170
Specific gravity	1.28-1.32	1.54

Table 2. Properties of matrix material used

Araldite (LY-556)		Hardener HY951	
Appearance	Clear, pale yellow liquid	Appearance	Clear liquid
Density @ 25°C	1.15 gm/cm^3	Density @ 25°C (ISO 1675)	0.98 gm/cm^3
Flash Point ISO 2719	>200°C	Flash Point (DIN 51758)	110°C
Viscosity @ 25°C (ISO 12058-1)	10000-12000 mPa.s	Viscosity @ 25°C (ISO 12058)	10-20 mPa.s

Figure 1. (a) Wool fiber (b) Flax fiber

Figure 2. Step-by-step fabrication of hybrid natural fiber-reinforced polymer composite.

Table 3. Blend ratios of samples of composite.

Fiber–Matrix Composition of Composite	Sample Name	Wool wt%	Flax wt%	Epoxy wt%
Wool Epoxy	W5	5	-	95
Flax Epoxy	F5	-	5	95
Wool + Flax +Epoxy (Hybrid reinforcement)	WF32	3	2	95
Wool + Flax +Epoxy (Hybrid reinforcement)	WF23	2	3	95

Experimentation

Vertical burn test

Various burning features are measured by the vertical burn test of a sample in an upright posture, such as the likelihood to ignite and fire spread. A blue flame was ignited to the base of the samples using a Bunsen burner while it was held vertically, as shown in Figure. 3. The self-extinguishing times were determined after removing the test flame [23]. This test provides three UL94 V-rating: V-0, V-1, or V-2. The sample will get a rating of V-0 if the burning stops 10 seconds after removing the burner and drips of flame are not allowed. In V-1 and V-2 ratings, flame stops 30 seconds after removing the burner, and flaming drips are also acceptable in V-2 ratings [18]. UL-94 is a specification of material flammability that fits industrial needs, published by Underwriters Laboratories Inc. This specifies materials and their hazards and provides methodologies for testing each material. It is a laboratory-scale test that uses visual observations to evaluate the flammability ratings of materials like plastics [24].

Figure 3. Vertical burn test setup.

Thermogravimetric analysis

Thermogravimetric analysis (TGA) is a kind of thermal analysis in which variations of chemical and physical characteristics in the form of mass in materials are assessed as a time function (with constant mass loss and/or constant temperature) or as a function of rising temperature (with heating rate as constant). Changes in the mass of the sample due to various thermal phenomena (reduction, desorption, decomposition, vaporization, oxidation, absorption, and sublimation) are investigated, when the specimen was subjected to a temperature change. It is utilized to analyze the loss of volatile and gaseous products during the reaction in composites, thermosets, elastomers, fibers, thermoplastics, and other materials.

Thermogravimetric analyzer STA 6000 (Perkin Elmer), which has the ingenious Saturn. A sensor for high accuracy was used. Parameter used during the test are mention in Table 2.

Table 2. Parameter used in thermogravimetric analysis.

Sample	**5 mg in powder form**
Environment	Nitrogen
Temperature range	Room temperature - 600°C
Temperature ramp rate	10°C
Flow rate	20 ml/min

Scanning electron microscope (SEM)

The SEM analysis was used to study the topography of materials on Nova Nano FE-SEM 450 (FEI). It offers ultra-high-resolution characterization and analysis and exact, accurate manometer-scale data. The gold coating was needed for the analysis to make the specimen conductive. The materials should be conducive for analysis on SEM.

RESULTS AND DISCUSSION

Vertical burn test

W5 specimen and WF32 specimen attain the UL94-V2 rating among all the samples. Other samples did not get any rating as they do not meet the UL94 standard. Therefore, not rated (NR).

Table 3. Flammability ratings of the composites.

Sample	**Time to self-extinguish after ignition (sec)**	**Dripping**	**UL 94 Rating**
W5	<30	Drips	V2
F5	>30	Drips	NR
WF32	<30	Drips	V2
WF23	>30	Drips	NR

Figure 4. Residual samples after vertical burn test: (1) F5 specimen, (2) W5 specimen, (3) WF32 specimen, (4) WF23 specimen

The images of samples after the vertical burn test are shown in Figure. 4. Flame self-extinguishes in less than 30 seconds in W5 and WF32 specimens. However, dripping on a cotton ball is noted. As a result, it was rated as UL94 V2 rating. The charring potential of wool is the rationale for the V2 rating. It prevents the entry of oxygen, which is required for combustion.

Thermogravimetric test analysis

Thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG) were used to explore the thermal degradation of composites. The thermal stability assessment is based on significant weight loss at a specific temperature. The TGA curves of the composites are shown in Figure. 5. Due to the matrix (epoxy) disintegration, considerable weight loss occurs during the thermal deterioration of composites in the temperature range of 260°C-480 °C. Finally, beyond 500°C, temperature char residue formation was detected. W5 specimen shows a maximum residual of 16.902 wt% which is more than the flax reinforced composite, F5. It is due to the charring ability of wool fiber [25]. The TGA curve and Table 4 show that after the W5 specimen, WF32 char residue is the highest (16.043 wt %) among all the hybrid fabricated composites. Char development served as shielding and reduced the release of combustible volatiles, which also helped in resisting the polymer thermal degradation. Char impedes the further entry of oxygen essential for burning [26].

Table 4. Char development of composites.

Composite sample	Char residue at 600°C (wt %)
W5	16.902
F5	15.093
WF23	15.197
WF32	16.043

F5 specimen, a flax-reinforced epoxy composite, decomposes more slowly than W5 wool-reinforced epoxy composite. It is because of flax fiber's interfacial solid adhesion to the matrix. Strong interfacial bonding requires more energy to break apart the matrix and fiber under the effect of heating compared to weak bonding in the case of wool-reinforced composites [9][26].

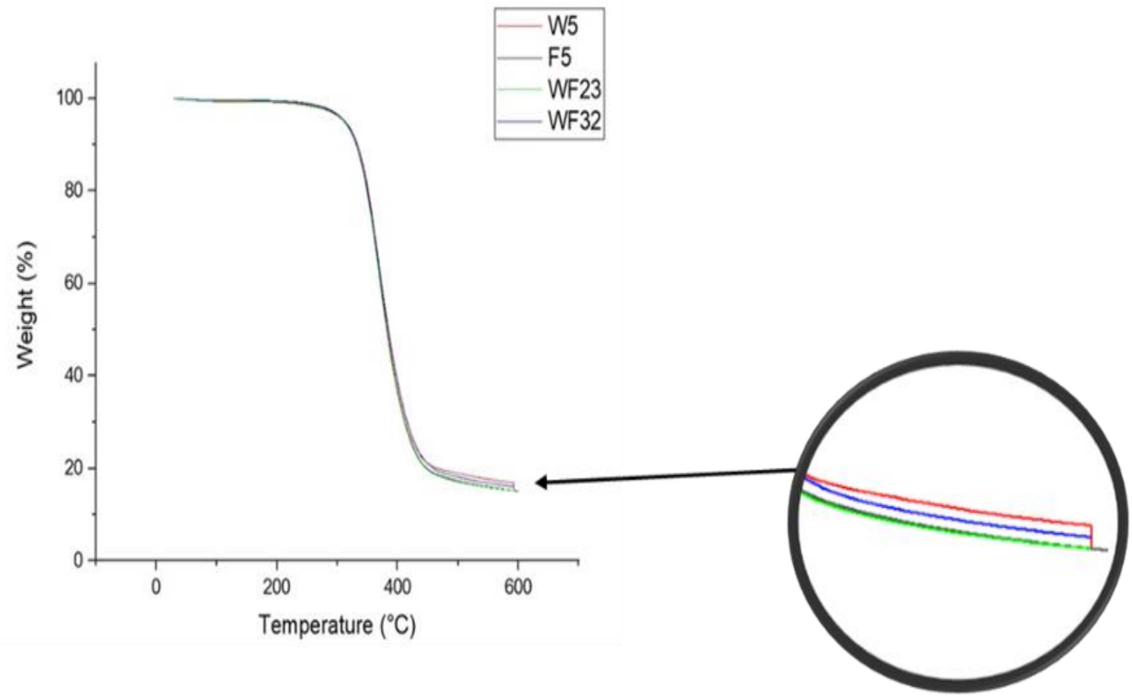

Figure 5. Thermogravimetric curves for various types of composites.

DTG curve shows the derivative weight loss of the composites. Figure. 6 shows a relatively higher derivative weight loss for the F5 specimen. WF32 revealed a minimal derivative weight loss among all the hybrid composites.

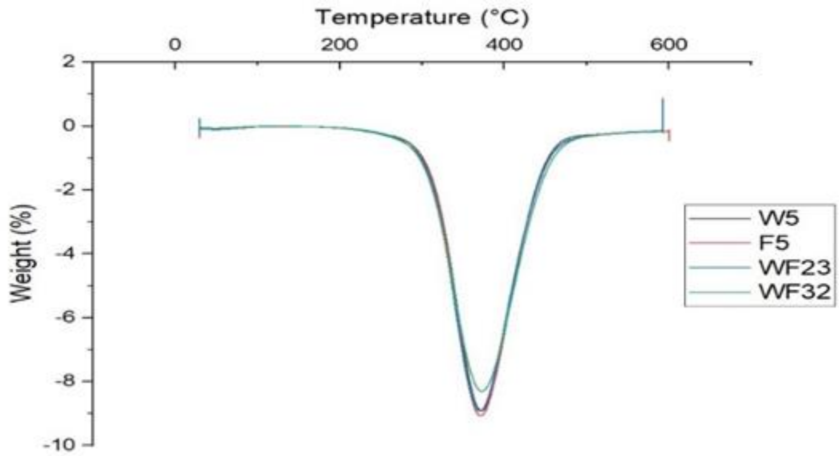

Figure 6. Derivative thermogravimetric curves of composites.

SEM Analysis

The SEM image revealed the distribution and bonding pattern of fibers in matrix as shown in Figure. 7(a-d). It also shows the flaws and defects of the composite. The intergranular brittle fracture was observed in SEM analysis of the fractured cross-sections of the tensile test specimens. It is owing to the composite's amorphous nature that is responsible for creating the brittle fracture [9]. It was observed that wool fibers have a bigger diameter than flax fiber, as shown in Figure. 7 (d).

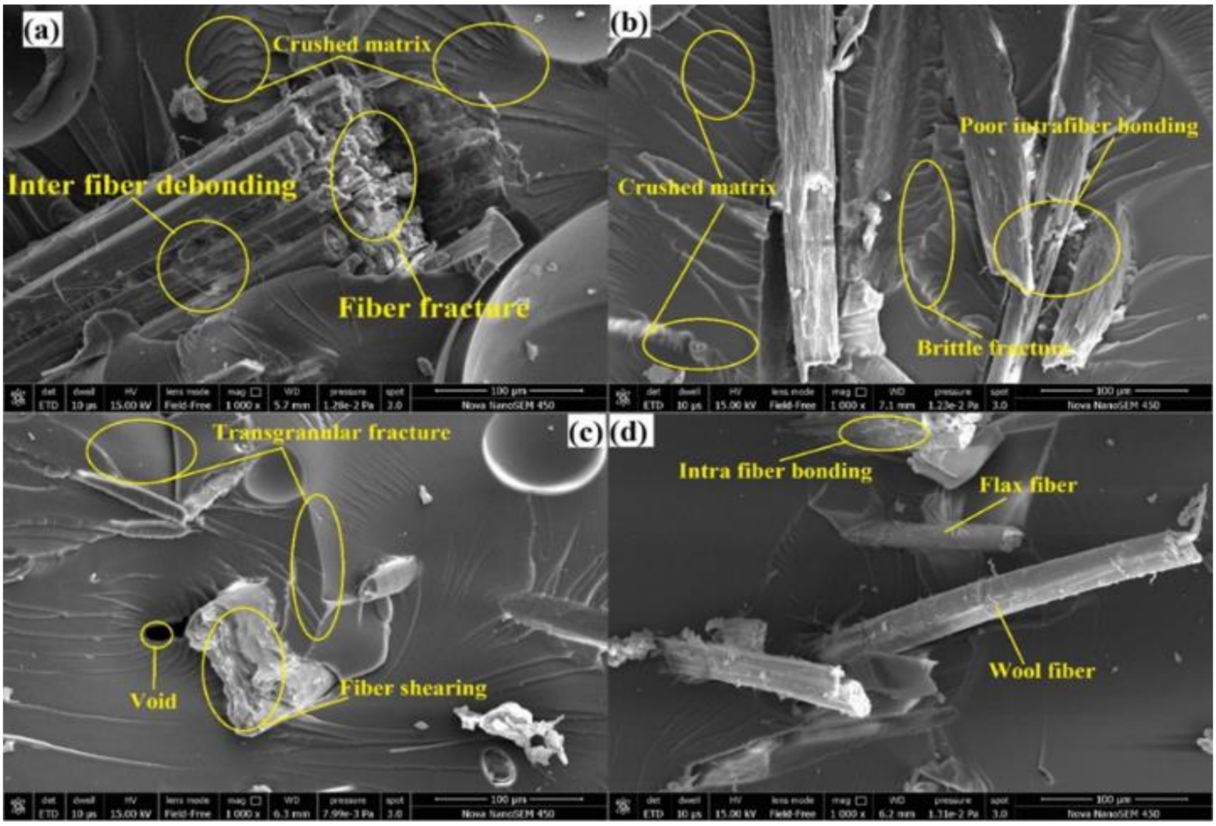

Figure 7. SEM images of tensile fractured surface of various types of composites: (a) F5, (b) W5, (c) WF32, (d) WF23 composites.

CONCLUSION

- W5 and WF32 specimen were rated as UL94 V-2 with a self-extinguished flame under the vertical burn test. V2 rating is due to the charring ability of wool, which impedes the oxygen supply.
- TGA of the fabricated sample shows that wool-based composite W5, WF23, and WF32 have more char residue than the F5 specimen. Wool incorporation in epoxy/flax systems outperforms individual flax reinforced composite (F5) during residual char production, improving flame retardant properties.
- SEM analysis was used for the distribution pattern of fibers and flaws of composite.
- Characterization results may be compared by fabricating the wool and flax hybrid composites using different fabrication techniques.
- Flammability tests, such as the cone calorimeter test, can be conducted to investigate the hybrid composite for economic viability in industrial applications.

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