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Effect of Fire-retardant Fillers on the Flammability and Mechanical Properties of Wood Polymer Composite

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Abstract—*Wood based polymer composite (WPC) has significant benefit on environment and now a days it is widely used for various applications like paneling, flooring, fencing and automobile components. The tendency to catch fire by these composites prohibits its wide application in industries. In order to reduce its flammability fire retardants (FR) additives like montmorillonite, manganese dioxide, zinc borate (ZB), stannic oxide, and ammonium polyphosphate (APP) are normally added in these composites. The compatibility of these additive with matrix material is a critical parameter which affect the mechanical properties. APP releases phosphoric acid when exposed to heat, which creates a char layer that can act as a barrier between the flame and the underlying material. In this study different ratio of APP and silica gel has been mixed as a FR additive in the mixture of wood husk and epoxy resin at different ratio. Effect of different ratio of APP and Silica gel on mechanical properties of composite fabricated by hand layup and vacuum assisted resin transfer moulding (VARTAM) has been compared. Furthermore, the fire-retardant properties of the developed composites were analyzed using the fire-retardant test. At the temperature of 1000°C for 30 sec the weight loss of the material was observed. It was observed that combination of APP and silica have synergetic effect on reduction of flammability. The mechanical properties of the composites decrease with addition of flame retardants. It was noticed that when composite is fabricated without mixing fire retardant the tensile, flexural and impact strength is higher.*

Keywords: *natural fiber reinforced composites, fire retardant, flame test, tensile strength, impact strength, wood husk, silica gel.*

1. INTRODUCTION

Due to the increasing concern about the negative impact of traditional manufacturing practices on the environment, many industries are now seeking to adopt more sustainable practices by using eco-friendly materials that are derived from renewable sources. This has led to a growing interest in natural fibers as a substitute for conventional materials in the production of composite materials that can be used in various applications.

The use of natural fibers in biobased composite materials is becoming more popular because these materials are derived from renewable feedstocks and have a lower environmental impact than their conventional counterparts. These natural fibers can be obtained from a variety of sources, such as agricultural waste, recycled materials, and other renewable sources [1]. Overall, the use of natural wood fiber reinforced polymer composites represents a sustainable and cost-effective solution for industries looking to reduce their environmental impact while maintaining high levels of performance and durability in their products [2].

One of the major problems associated with the natural fiber reinforced composite is its tendency to get deteriorate by temperature. The presence of cellulose in the plant and hydrocarbon in the polymers used to fabricate composite enhances it flammability. At temperature greater than 500ºC the polymers release combustible gases which is major drawback prohibiting its wide application in industries. Furthermore, the flammability also reduces the creep strength and load bearing capacity. Therefore, development of flame-retardant natural composites with good mechanical properties will help to promote the engineering application in more eco-friendly manner [3].

Wood primarily contains cellulose. During thermal decomposition the cellulose produces dehydrocellulose which further decompose to produce char and other volatile components. The polymer used as matrix change its molecular structure during initial heating and later on the breaking of molecular bond takes place producing volatile products. When heat is continuously applied on natural fiber reinforced polymer composite these volatile components can establish a combustion cycle thus making the composites unsuitable for engineering and other structural application.

Since both natural fiber and polymer ignites very easily therefore addition of fire-retardant (FR) material may be feasible solution to reduce the flammability of the natural fiber reinforced polymer composites. These FR materials helps to break the chain reaction in a combustion cycle. FR may be additive or reactive in nature. Additive FR are physically added in the composite during its fabrication whereas the reactive FR act by altering the structure of polymer. Magnesium hydroxide ($Mg(OH)_2$) and aluminum hydroxide ($Al(OH)_3$) reduces the flammability by producing water during reaction. Ammonium polyphosphate (APP) is more effective FR which prohibits the fire due to its decomposition into polymeric phosphoric acid and ammonia. APP produces swollen multicellular char which further reduces the flammability of polymers.

Zang et al. [1] studied natural wood fiber reinforced polymer composites for high performance and durability. They found that polypropylene (PP) is an excellent choice for these composites due to its lightweight, water and chemical resistance [1]. Madyaratri et al. [2] found that stricter environmental regulations are driving the adoption of biobased composite materials [2]. Jeencham et al. [3] found that modifying the surface of the filler material is an effective approach to improving adhesion to the binder and enhancing flame retardancy in bio composites [3]. Giancaspro et al. [4] explored the potential of using an inorganic matrix to improve the fire resistance and strength of composite materials made from industrial by-products such as sawdust. Anna et al. [5] observed that the surface treatment of cellulose fibers can improve the compatibility between the fibers and the PP matrix, leading to better mechanical properties and FR performance. Li and He [6] demonstrated the potential of using ammonium polyphosphate (APP) as an effective flame retardant (FR) for LLDPE-WF composites. The addition of magnesium hydroxide (MP) or pentaerythritol (PER) to APP did not significantly improve the FR performance, indicating that APP alone is sufficient to provide flame retardancy. Jogendra et al [7] investigated the fire-retardant properties of carpet waste polymer composite. Price et al. [8] also found that the flammability of natural fiber composites is a significant drawback in engineering applications and the dripping of molten material from these composites during combustion is an additional safety concern. When natural fiber composites are exposed to fire, they release a significant amount of heat and smoke, which can contribute to the spread of the fire and create poor visibility [9].

Mouritz and Gibson observed that when natural fiber composites are exposed to high temperatures, the softening and creep behavior of the fiber reinforcement and polymer matrix can cause buckling and failure of load-bearing composite structures [10]. Azwa et al. [11] observed that addition of FR reduces the mechanical properties of fabricated composites. Therefore, it is important to strike a balance between fire resistance and mechanical performance. Price and Horrocks [12] proposed that the structure of polymer matrix plays a crucial role in the thermal decomposition of composites. Polymers with inherent flame resistance, such as phenolic and polyimide resins, can yield char during combustion, which helps to protect the material from further burning. Matko et al. [13] conducted a study on the flammability of different NFRC using APP as a FR. Their findings showed that the addition 20% APP in polyurethane composites with either corn shell or wood flake as a filler increases the flame retardancy.

Above literature survey reveals that most of the work related to fire retardancy of NFRC has been conducted for hand layup prepared NFRC composite using polypropylene as matrix. Very few studies related to effect of FR in epoxy based NFRC with wood husk as reinforcement has been reported. In this study, epoxy based NFRC using wood husk as reinforcement has been prepared at different composition with both hand layup and VARTM technique. Fire retardancy test and mechanical characterization of the fabricated composite has been performed as per the ASTM standards.

2. MATERIAL AND METHOD

To fabricate the fire-resistant composite material mango wood husk were used as a reinforcement and epoxy (resin) as a base(matrix) material. APP and silica gel were added as a FR material. Mango's wood has high strength due to very dense grain particle which imparts high strength. Mango wood husk was extracted through crusher machine. Epoxy LY556 resin with the property of density:1.16 g/cm^3, flexural strength: 25.29 MPa, Tensile strength: 11.14 MPa was mixed with hardener in the ratio of 10:1. Since longer chain APPs are more resistant to water and heat therefore long chain APP were added as FR for composite fabrication.

NaOH is used for the chemical treatment of the mango wood husk. The fundamental alkaline treatments were utilized for surface adjustment of fibers. Mango wood husk was treated with 5% of sodium hydroxide in 2 liters of distilled water for 6 hours and after that it was washed with water and dried at 25 degrees centigrade.

2.1 Hand Layup method

The epoxy resin (LY 556) was mixed with hardener (HY 951) in the ratio of 10:1. The dried mango wood husk was added in the mixture and stirred at slow speed. The mixture was poured into wooden mould of dimensions $200 \times 100 \times 6 \quad mm^3$. Hand roller was used to remove the air bubbles. Load of 25 kg was applied on the mould and fabricated composite was left to cure for 24 hours. The specimen is also post cured at room temperature.

2.2 Vacuum Assisted Resin Transfer Molding (VARTM)

VARTM can produce composite structures with fewer voids compared to hand layup method and improve the quality of the final composite structure. In this method pressure difference is created at the inlet and outlet so that fibers can be introduced into the resin. In this process a mold of desired size is prepared in which the fabric/ reinforcement is laid off. After this the mold is sealed and vacuum is created to infuse the resin so that composite can be obtained after curing.

3. TESTING

3.1 Fire Retardant Testing

Fire retardant tests are designed to evaluate the ability of materials to oppose ignition, prevent the spread of flames, and limit the amount of smoke and toxic gases that are produced during a fire. One commonly used fire-retardant test is the ASTM D635 test, which is the standard process to test the surface burning characteristics of material. This test evaluates by the testing of the flame spread and the smoke developed by the material when it exposed to a heat source. The fire retardant test is a type of experiment designed to determine the ability of a material to resist ignition and slow down or prevent the spread of fire. The horizontal fire setup is a common method used for this test, in which the specimen is placed horizontally and subjected to a 30-second torch ignition, followed by a 30-second period of observation to evaluate the extent of self-burning. The results of this test can help determine the fire safety and performance characteristics of a material, so that materials can be appropriately used for various applications.

Fig. 1: Fire Retardant test

Fig. 2: Char formation

3.2 Mechanical Testing

A tensile test, also referred to as a tension test, is a mechanical test that is used to determine the strength and ductility of a material. Tensile test involves pulling a sample of the material in opposite directions, along its longitudinal axis, until it breaks or fractures.

A flexural test, also referred to as a bend test or a transverse test, is a mechanical test used to determine the strength and stiffness of a material under bending loads. In this test, a sample of the material is supported at both ends, and a load is applied to the center of the sample, causing it to bend. The amount of force required to cause the material to bend is measured, along with the amount of deflection, or deformation, that occurs in the material as a result of the applied load. This information can be used to determine the material's flexural strength, modulus of elasticity, and other properties that are important for understanding its behavior under bending loads. Impact test is used to assess a material's ability to absorb energy and resist fracture or deformation when exposed to sudden shocks or impacts.

The fabricated samples of composites were characterized for all the above three mechanical testing. Tensile test was conducted according to ASTM D638 with the feed rate of 5 mm/min and flexural test was conducted according to ASTM D790 at the feed rate of 2mm/min. Tensile and flexural test were conducted by using Instorn-1195 universal testing machine of capacity 100 KN. Impact test is conducted by using Zwick impact testing machine and sample is prepared according to ASTM D256.

4. RESULTS AND DISCUSSION

Table 1 and Fig.3, depicts that weight percentage composition of APP play an important role in developing retardant property of composite material. As the weight % of APP in composite material increase, the mass loss of composite material reduces which indicates that APP material resist the fire in better way.

Table 1: Mass loss (%) of composite sample with different composition

Method	Sample	Initial Wt. (gram)	Final Wt. (gram)	Loss (%)
Hand Layup	Epoxy=60%, WH=40%.	15.490	14.340	7.4241
Hand Layup	Epoxy=60%, WH=15%, APP=25%	17.350	17.140	1.2104
Hand Layup	Epoxy=60%, WH=25%, APP=15%	17.220	16.800	2.4390
Hand Layup	Epoxy=60%, WH=15% APP=20%, Silica gel=5%	18.430	18.00	2.3332
VARTM	Epoxy=60%, WH=15%, APP=20%, Silica gel=5%	15.00	14.690	2.0667
VARTM	Epoxy=60%, WH=15%, APP=15%, Silica gel=10%	14.660	14.350	2.1146
VARTM	Epoxy=60%, WH=10%, APP=25%, Silica gel=5%	13.250	13.00	1.8868

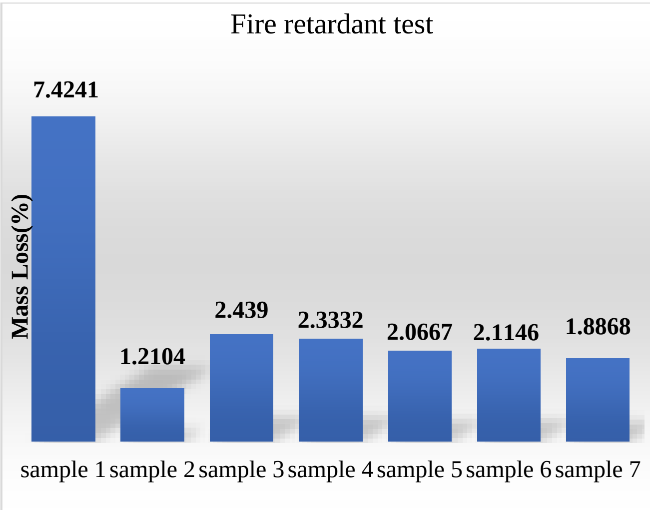

Figure 3: Results of flammability test

From Table 2 and Fig.4, it is observed that the maximum tensile strength is obtained when sample is fabricated through VARTM method at the weight percentage of wood husk (15%), APP(20%) and silica gel (5%).

Table 2: Flexural strength of composite for different composition

Method	Sample	Peak Stress (MPa)	Peak Load (kN)
Hand Layup	Epoxy=60%, Wood husk=40%, APP=0, Silica gel=0	20.117	0.128
Hand Layup	Epoxy=60%, Wood Husk=15%, APP=25%	17.389	0.084
Hand Layup	Epoxy=60%, Wood Husk=25%, APP=15%,	28.746	0.198
Hand Layup	Epoxy=60%, Wood Husk=15%, APP=20%, Silica gel=5%.	18.498	0.087
VARTM	Epoxy=60%, Wood Husk=15%, APP=20%, Silica gel=5%.	28.8	0.08
VARTM	Epoxy=60%, Wood Husk=15%, APP=15%, Silica gel=10%	22.0	0.086
VARTM	Epoxy=60%, Wood Husk=10%, APP=25%, Silica gel=5%.	16.38	0.076

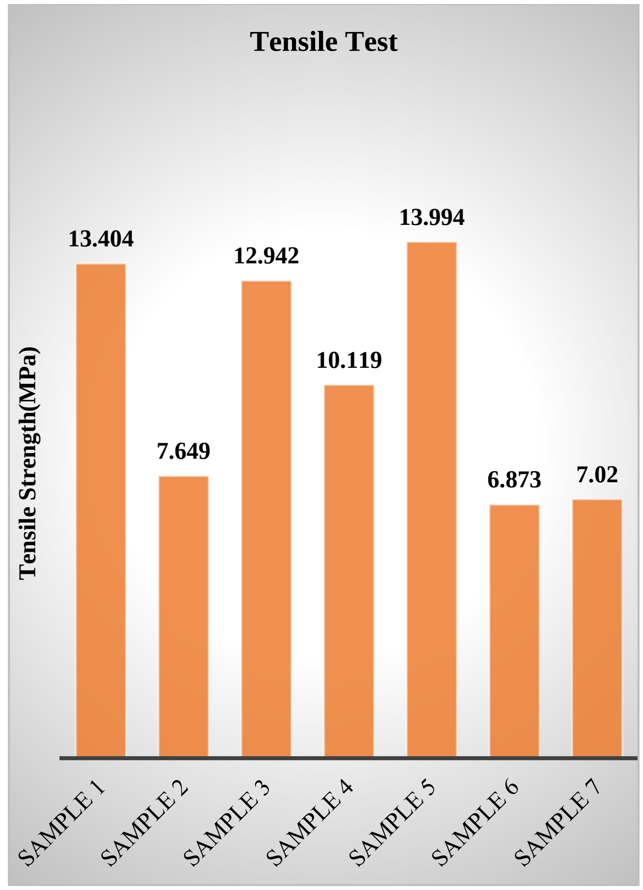

Figure 4: Tensile strength of composite

From the table 3 and fig.5 it is observed that the maximum flexural strength is obtained when sample is fabricated through VARTM method at the weight percentage of wood husk, APP and Silica gel with 15%,20%,5%.

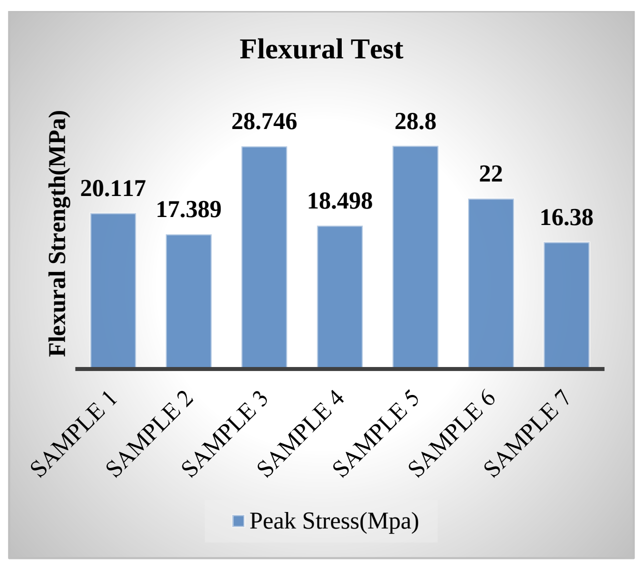

Figure 5: Flexural strength of composite

From the table 4 and Fig.6 it is observed that the impact strength is maximum when sample is prepared without APP and Silica gel.

Table 4: Impact strength of composite

S.NO	Sample (Composition)	Impact Strength (KJ/M^2)
Hand Layup	Epoxy=60%, Wood husk=40%,	4.860983
Hand Layup	Epoxy=60%, Wood Husk=15%, APP=25%,	4.489735
Hand Layup	Epoxy=60%, Wood Husk=25%, APP=15%,	4.68957
Hand Layup	Epoxy=60%, Wood Husk=15%, APP=20%, Silica gel=5%.	4.358462
VARTM	Epoxy=60%, Wood Husk=15%, APP=20%, Silica gel=5%.	4.358462
VARTM	Epoxy=60%, Wood Husk=15%, APP=15%, Silica gel=10.	4.4411537
VARTM	Epoxy=60%, Wood Husk=10%, APP=25%, Silica gel=5%.	4.755914

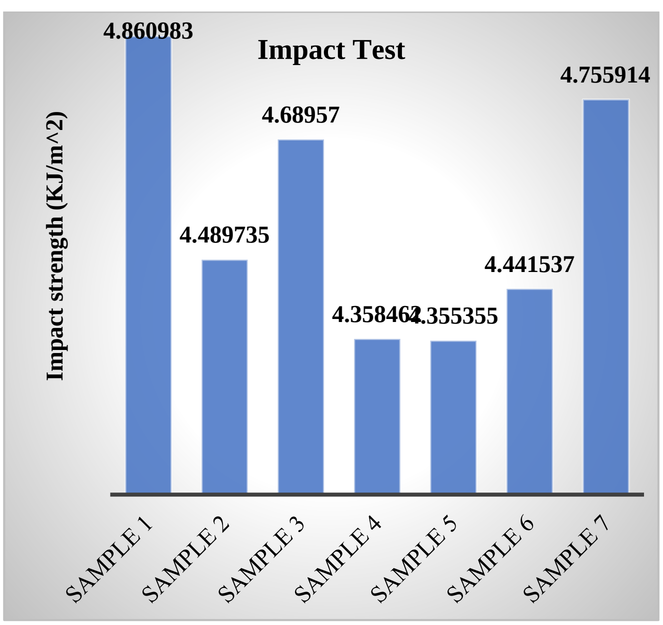

Figure 6: Impact strength of composite

Table 5: Tensile strength of composite

S.O	Sample (Composition)	Peak Stress (MPa)	Peak Load (KN)
Hand Layup	Epoxy=60%, WH=40%,	13.404	2.469
Hand Layup	Epoxy=60%, Wood Husk=15%, APP=25%,	7.649	1.1
Hand Layup	Epoxy=60%, WH=25%, APP=15%,	12.942	2.167
Hand Layup	Epoxy=60%, WH=15%, APP=20%, Silica gel=5%.	10.119	1.443
VARTM	Epoxy=60%, Wood Husk=15%, APP=20%, Silica gel=5%.	13.994	1.951
VARTM	Epoxy=60%, Wood Husk=15%, APP=15%, Silica gel=10.	6.873	0.96
VARTM	Epoxy=60%, Wood Husk=10%, APP=25%, Silica gel=5%.	7.02	0.838

5. CONCLUSION

In order to evaluate the effectiveness on fire retardants materials in reducing the flammability of natural fiber reinforced composites, wooden husk reinforced epoxy-based composites were fabricated using the hand layup and VARTM method. The composition of composites was changed by varying the quantity of wooden husk, silica gel and APP whereas the epoxy was kept constant at 60 wt%. The impact of these components on the mechanical strength of the fabricated composites was also analyzed. The major finding of the study are as follows:

a. Epoxy based NFRC prepared by hand layup method with Epoxy 60 wt %, wood husk 15 wt % and 25% APP 25 wt% has the highest flame retardation capability and the weight loss due to burning is lowest. But the mechanical property of this composition is not good.
b. When a high percentage of APP is added as FR in the composite it reduces the flexural strength of fabricated composite. Composite containing 60% epoxy, 15% wood husk and 25 % APP has a tensile strength of 17.389 MPa. It is also observed that addition of silica gel further reduces the tensile strength to 16.38 MPa.
c. Addition of APP up to 25 wt % reduce the flexural strength from 20.117 MPa to 17.389 MPa when the composite was fabricated by hand layup method.
d. In case of hand layup method, the composite with composition of Epoxy 60% wooden husk (25% and APP (15%) yield impact strength of 4.68957 kJ/m^2 which higher compared to other samples containing APP. Addition of

silica gel up to 5% decreases all the three mechanical characteristics.

e. Composite prepared by VARTAM method with the composition of Epoxy=60%, Wood Husk=15%, APP=20%, Silica gel=5% yields better results in terms of all mechanical properties as well as fire retardation.
f. Increase of only APP increases the fire retardancy but decreases the flexural, tensile and impact strength.

6. FUTURE SCOPE

Fire retardant composites have a promising future as they offer a lightweight, durable, and high-performance alternative to traditional materials such as steel and concrete. The demand for fire retardant composites is increasing across various industries, including aerospace, construction, automotive, and defense, due to their unique properties, such as high strength-to-weight ratio, corrosion resistance, and fire resistance.

In the aerospace industry, fire retardant composites are used to build lightweight aircraft components, such as wings, fuselages, and landing gears, to reduce fuel consumption and increase flight range. In the construction industry, they are used for the development of fire-resistant structures, walls, and panels that can withstand extreme temperatures and prevent the spread of fire. Moreover, in the automotive industry, fire retardant composites are used to produce lightweight and fuel-efficient vehicles that comply with safety standards. In the defense industry, fire retardant composites are used to manufacture lightweight and durable armored vehicles, ships, and aircraft that can withstand high-velocity impacts and fire hazards.

The future scope of fire-retardant composites is vast, as there is a continuous need for materials that offer improved safety, durability, and performance. As new technologies and manufacturing processes emerge, the production of fire-retardant composites is expected to become more cost-effective, enabling broader applications across various industries.

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