Research Article

Effect of Borax-Boric Acid Treatment on Fire Resistance, Thermal Stability, Acoustic, and Mechanical Properties of Mycelium Bio Composites

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Abstract
Mycelium biocomposite materials have been established as a sustainable alternative to polystyrene in single use applications like packaging. However only little investigations are done on improving their resistance to fire and heat, which can find use in newer applications. This paper focuses on the development and characterization of a mycelium-based sawdust-coir pith biocomposite material treated with a combination of fire-retardant compounds (borax and boric acid). The outcomes of fire resistance tests, such as flammability, flame penetration and rate of burning demonstrated a significant improvement in values with respect to untreated samples. However, samples having 30% boron compounds by weight in it exhibited the best fire resistance properties. The thermal analysis of treated samples indicated that the presence of fire-retardant chemicals has not significantly affected their thermal stability. The glass transition temperature (Tg) of treated mycelium composite material was found to be 212.75 °C against a value of 207.78 °C for untreated samples. The fire retardant treated mycelium composite samples having 30% boron by weight in it, exhibited an average sound absorption coefficient of 0.38 compared with a sound absorption coefficient of 0.29 for polyurethane foam. The prepared mycelium biocomposite has a self-extinguishing nature and exceptional fire resistance capabilities with an LOI value of 50%. The mechanical testing revealed that the presence of fire-retardant chemicals has significantly improved the flexural properties. However, only a marginal increase was visible in the compression strength of mycelium biocomposites.

Keywords: Acoustic insulation, Biocomposites, Borax, Boric Acid, Fire Resistance, Mycelium

1 Introduction

Sound insulation characteristic of a material is an important aspect of construction materials, especially in places where loud sounds and echoes occur, such as movie theatres, airport terminals, or engine rooms in ships. Sound insulation is achieved by preventing sound waves from penetrating and is quantified by the sound transmission loss expressed in decibels as the difference between incident and permeated sound [1].

The amount of energy removed from a sound wave as it passes through a material is measured as sound absorption. Sound absorbers are porous materials in which sound propagates through a network of interconnected pores, dissipating acoustic energy through thermal and viscous effects. Sound absorption in polymeric materials, on the other hand, is the conversion of sound waves into heat energy [2]. Polymeric materials like polyurethane and polyethylene are commonly used for sound insulation and acoustic treatment applications. These petroleum-derived polymeric materials often end up in landfills or recycling yards where several instances are being reported of heaps of these inflammable materials catching fire by accident. These materials are extremely hazardous to the environment and human health when they are not recycled or carefully disposed [3], [4].

The production of petroleum-derived plastic materials has been on the rise at a rapid pace since its discovery and world production increased from two million tonnes in the 1950s to a little over 380 million tonnes in 2018. It has been reported that approximately 3% of all the non-biodegradable plastic products produced each year are dumped in various water bodies. The widespread use of plastic products post World War II, has resulted in the generation of around 8.3 billion metric tonnes of plastic products and about 6.3 billion tonnes of this completed its useful life and became waste material until 2015. Of this waste plastic material, about 12% is incinerated and only 9% is recycled. The rest end up in landfills or the environment, where they will remain indefinitely because they do not decompose easily. In the year 2017 alone, the global production of plastic is around 350 million tonnes. The current estimates project that 12 billion tonnes of waste plastic will end up in landfills by the year 2050 [5]. Despite several initiatives, it is evident that more plastics are thrown away than collected and recycled. Even recycling would not be a practical option because making new plastics is less expensive than recycling. This means that recycling plastic would not continue to be an effective approach to reducing plastic in the actual world. An obvious way to reduce plastic waste is to promote the use of biodegradable plastic products and incentivize recycling of non-biodegradable plastic products, along with a decrease in global production and use of virgin plastic as a raw material.

The recent advancements in the bioprocessing of materials have resulted in the emergence of a novel class of materials developed using natural processes and sourced from biomatter. Mycelium biocomposite materials formed by using *Pleurotus ostreatus* fungal mycelium as a binder on substrates like plant or agricultural waste are found to be an effective replacement for foam materials derived from petroleum products [6], [7]. Mycelium is a fast-growing vegetative portion of a fungus that can be grown on biological and agricultural wastes which acts as a binder to form biocomposites. These mycelium biocomposite materials are organic, renewable, and environmentally friendly in nature. Mycelium swiftly forms self-assembling connections that result in a robust and biodegradable substance [8], [9]. Mycelium can be grown in a variety of shapes and sizes for numerous applications. Mycelium biocomposite products can be easily decomposed after their useful life, and it is less expensive when produced on a large scale. Mycelium biocomposite materials are largely made from agricultural waste materials, which are generally combustible in nature [7] and thus possess a risk of catching fire unless they are treated with fire retardant chemicals. Mycelium biocomposite materials with improved fire resistance could possibly replace sound insulation materials like polyurethane and polyethylene to be used inside buildings where fire safety is a primary concern.

Mycelium is used to fabricate various materials from surfboards to flower pots and lamp shades. The application also extends to the construction industry where mycelium biocomposites were developed for building insulation applications [10], [11]. Mushroom bricks were developed and used to construct a tower, 40-foot high, which is the tallest structure built of mycelium bricks. Other applications include the development of panel boards for insulation and replacement fiber boards for construction. Myco-board can potentially replace engineered wood products like plywood containing toxic compounds like urea-formaldehyde. Insulation boards help to create a thermal barrier that makes the buildings more energy-efficient. Mycelium foams can be used in place of expanded polystyrene (EPS) to protect packaged goods from shocks [6], [7]

The mycelium biocomposite panels made from saw dust-coir pith substrates were found to be effective at absorbing sound [12]. The acoustic absorption rate of mycelium samples was evaluated with various substrates and the results exceeded 70–75% at a frequency of 1,000 Hz. It is reported that the audio frequency spectrum of biocomposite samples made from switch grass and sorghum shows the highest absorption rate when the substrate is composed of 50% switch grass and 50% sorghum [13]. Compared to materials like EPS, mycelium biocomposite materials demonstrated a significantly higher flexural strength. Unlike many other insulation materials, such as polyurethane, this material is biodegradable. Since the substrates needed to cultivate mycelium are not expensive, they can be produced at very low costs [14]. Mycelium biocomposite materials made from hemp and straw as substrates have exhibited densities ranging from 57–99 kg/m^3 and thermal conductivities between 0.04 and 0.08 W/mK, which makes them a natural choice for insulation applications. This means they can be used in the same way as conventional commercial thermal insulation products, such as glass wool and polystyrene insulation in addition to natural insulation materials like sheep wool and kenaf [15].

In fire retardant treatments, buffers based on boric acid-borax have been found to reduce thermal degradation greatly. Due to their substantial flame-retardant characteristics, borax and boric acid are particularly convenient to employ as flame-retardant compounds. Due to their thermal resistance property, they have been widely used in fire-resistant fields. However, Borax tends to reduce the flame spread, but it can promote smoldering and glowing, while Boric acid suppresses glowing and catalyzes dehydration and other oxygen-eliminating reactions [16]–[18].

The engineering applications of mycelium bio-composite materials are presently constrained, but there is untapped potential for enhancing their utility by integrating fire-resistant properties into the material [14]. However, there has been a scarcity of reported research dedicated to enhancing the fire resistance of mycelium bio-composite materials, particularly used for acoustic insulation applications. The development and characterization of a fire-resistant mycelium-based panel for acoustic insulation is the main focus of this study. When creating bio-composite materials, sawdust and coir-pith are utilized as the substrate, and chemical additives are added to boost the material's fire resistance. Numerous tests are performed, including the tests for flammability, flame penetration, burning rate, compressive and flexural strength, limiting oxygen index, and acoustic impedance. The characteristics of both treated and untreated materials are compared to analyze the effect of fire-retardant treatment.

2 Materials and Methods

2.1 *Materials*

A 3:2 blend of sawdust and coir-pith is selected as the matrix. Compared to coir-pith, sawdust encourages faster growth. However, lignin found in coir-pith prevents microorganisms from decomposing it as easily as sawdust, giving it a far longer shelf life than sawdust [7], [12]. The trial-and-error method is used to determine the 3:2 ratio [19]–[21]. The physical and chemical properties of raw materials used to develop the biocomposite samples are shown in Table 1. Sawdust serves as the primary source of nutrients for the mycelia in this matrix, therefore reducing the quantity of sawdust leads to a decreased growth rate, while a reduction in the amount of lignin affects the shelf life [7].

Pleurotus ostreatus mycelium from oyster mushrooms acts as the binder and reinforcement for the composite. The temperature at which oyster mushrooms can survive is significantly higher than the temperature at which other species of mushrooms can survive. Additionally, oyster mushroom spawns produce a crop with a longer shelf life, which makes them quite popular among mushroom producers. The bio-composite is impregnated with boron compounds to make it more fire resistant [22], [23].

Table 1: Physical and Chemical properties of materials used

Raw Materials Used	Physical Properties					Chemical Properties			
	Average Particle Size (Micron)	Moisture Content (%)	Specific Gravity	Water Retention (%)	Porosity (%)	Lignin (%)	Cellulose (%)	Hemi-Cellulose (%)	C:N Ratio
Saw dust	300	10.8	0.14	50	84	29.3	41.58	32.81	325
Coir Pith	750	14.3	0.12	500	60	34.23	27.28	19.76	118

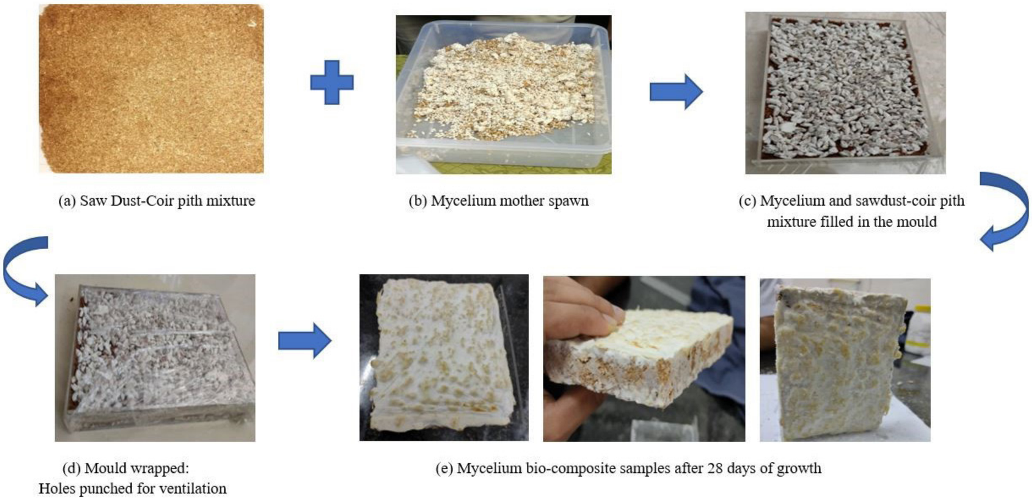


Figure 1: Preparation process flow of the mycelium biocomposites.

2.2 *Processing of raw materials*

Mycelium is grown from mother spawns of the highest quality *Pleurotus ostreatus* mushroom. The tissue is removed from the neck of the mushroom and is allowed to grow on a medium, where the mother spawn is cultured. Sawdust is a waste product and was obtained from a sawmill and high-quality coir-pith is procured. A transparent acrylic mold was used for accurate monitoring of the growth of mycelia. Needles, dishes, and molds used in the process are cleaned and sterilized with rubbing alcohol. After being filled with the matrix and reinforcement materials, the acrylic glass mold is covered but with proper ventilation to keep the humidity necessary for the growth of the mycelia [7], [12].

The sawdust and coir-pith mixture are sterilized in an autoclave, where pressurized steam at 121 °C is circulated for about 20 min to destroy the presence of any bacteria, spores, and germs that are resistant to boiling water and detergent. Additionally, a hot air oven that acts as a dry heat sanitizer is used to sterilize the molds.

2.3 *Mycelium bio-composite samples preparation*

A mixture of sawdust and coir-pith in the ratio of 3:2 by weight is mixed and sterilized in an autoclave at 120 °C, at a pressure of 103.4214 kPa (15 psi) for about 15 min. This process deactivates any microbes in the mixture that might retard the growth of mycelia. The transparent acrylic mold is sterilized for 15 min in a hot air oven at 100 °C and additionally by rubbing with alcohol. The mold is first filled with a thin layer (≈ 1.5 mm) of oyster mushroom mother spawn and then with a layer (≈ 3 mm) of sterilized coir-pith and sawdust mixture. Repeat this step until the required thickness is achieved. A hard-outer covering is required for the structural stability of biocomposite samples, which is achieved by filling the last layer with mycelium spawn. The filled mold is then kept in a dark room at room temperature for 28 days, all the while ensuring proper ventilation and humidification [7], [12].

During these days, the growth of mycelium is monitored and the required humidity level is maintained by spraying water on the samples. After the growth was complete, samples were subjected to the fire-retardant chemical treatment. Figure 1 shows the process flow of mycelium biocomposite preparation. mycelium bio-composite samples were separated from the mold after a growth of 28 days. Further growth of mycelia was inhibited by drying the samples in a hot air oven.

The samples were soaked in distilled water before adding them with 10%, 20%, and 30% by weight of Borax-Boric acid powder in a ratio of 4:3. (30% of Borax-Boric acid is found to be the maximum amount that can be added after which no more powder gets adsorbed to the sample). The sample is dried at a temperature of 140 °C for 20 min in a hot air oven. Moisture is removed from the samples through drying which in turn makes it light in weight. Drying of the samples also will inhibit the further growth of mycelia.

2.4 *Methods*

2.4.1 Thermogravimetric analysis

Thermogravimetric Analysis (TGA) measurements were performed as per ASTM E1131 on treated and untreated samples at a heating rate of 10 °C/min using a Thermogravimetric analyzer (Perkin Elmer STA 6000) using air as the environment. Thermal decomposition of each sample occurred in a programmed temperature range of 40–550 °C. Using TGA, the continuous temperature changes and weight loss were noted and analyzed.

2.4.2 Differential scanning calorimetry

Both treated and untreated samples were subjected to Differential Scanning Calorimetry (DSC) analysis as per ASTM D3418 using a Differential Scanning Calorimeter (METTLER STAReSW 8.10) with air as the atmosphere at a heating rate of 10 °C/min. The temperature range of each sample was programmed to be between 30 and 570 °C for all measurements.

2.4.3 Fire resistance test

To characterize the fire resistance of mycelium bio-composite samples, a set of three experiments were performed following IS 1734: part 3 (1983). They are flammability test, flame penetration test, and rate of burning test. It was then followed by the Limiting oxygen index test based on ASTM D2863.

Figure 2 shows the mycelium biocomposite material samples being tested for flammability. In the flammability test, for a specimen size of 125 × 125 × 16 mm, the time between the ignition of the lower specimen and the ignition of the higher specimen is recorded. Whereas, for flame penetration test the specimen is rotated at 75 revolutions per minute so that the flame's center depicts a circle with a diameter of 25 mm. It is timed as to how long it takes the flame to pierce the specimen's thickness. Figure 3 shows the mycelium biocomposite material samples being tested for flame penetration.

The rate of burning test uses a sample size of 100 × 12.5 × 16 mm as the test specimen. It is ignited, and the amount of time needed to lose 10% of its mass is noted. For a comparison, the period between 30–70% decrease in mass is used. Figure 4 shows the mycelium biocomposite material samples being tested for rate of burning. All three tests were conducted as per IS 1734: part 3 (1983).

Figure 2: Flammability test.

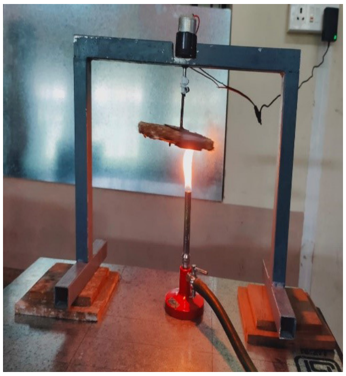


Figure 3: Flame penetration test.

Figure 4: Rate of burning test.

(a) Compression test (b) Flexural test

Figure 5: Mechanical testing of the samples for a) compression test and b) flexural test.

Limiting oxygen index test, was conducted on test specimens with the dimensions 150 × 50 × 10 mm. Then, in accordance with ASTM D2863, the minimal oxygen concentration in an oxygen-and-nitrogen mixture to sustain the flammability of mycelium biocomposite samples for 15 s was assessed.

2.4.4 Mechanical strength test

The compressive properties of the material are evaluated using an ASTM D-695 compressive strength test. The samples are made in the shape of cylinders 10 × 10 cm. These samples are placed in between two compressive plates that are parallel to the surface and are then compressed at a uniform rate. The maximum load and stress-strain values are then recorded after the specimen has been uniformly compressed.

To measure the flexural strength, test specimens with dimensions 150 × 50 × 10 mm were employed. A 3-point bend test is performed by ASTM D 790 for a span length of 80 mm. The specimens were deformed monotonically to failure displacement controlled at 5 mm/min. The rate of bending and span length ensured that the samples behaved elastically when the load was applied. When the load is gradually applied, the beam surface away from the load is under tension while the surface closer to the load is under compression.

2.4.5 Acoustic impedance test

The acoustic impedance and sound absorption coefficient of mycelium biocomposite samples were found as per the standard ISO 10534-2. Transfer-function method is used to determine the sound absorption coefficient and the impedance of the samples. The ratio of absorbed sound energy to incident sound energy is known as the sound absorption coefficient. While acoustic impedance is the ratio of the rate of particle flow across a surface to the pressure over a unit area of a sample in a sound wave. Four frequencies (250, 500, 1000, and1600 Hz) were selected to determine the corresponding change in the sound absorption coefficient.

3 Results and Discussion

3.1 *Thermogravimetric analysis*

Figure 6 represents the TGA curves of boric acid-borax treated and untreated mycelium biocomposite samples. Thermal stability analysis of both types of samples revealed an identical curve line when weight loss % and temperature were analyzed. The thermal stability of samples has not been affected by fire retardant treatment. Figure 6 shows the weight loss versus temperature curve from TGA that shows a steep decline for the first weight loss for untreated samples between 80–100 °C, which is attributed to the dehydration of samples; [23] however the boric acid-borax treated samples exhibited slightly less weight loss during the temperature range between 80–100 °C, which indicated that the boric acid-borax treated samples are marginally more thermally stable after the treatment.

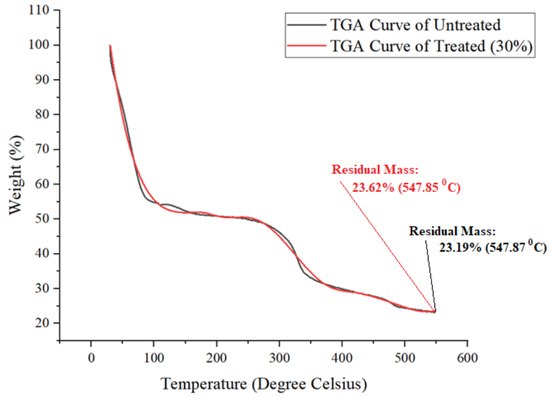


Figure 6: TGA vs. Temperature graph.

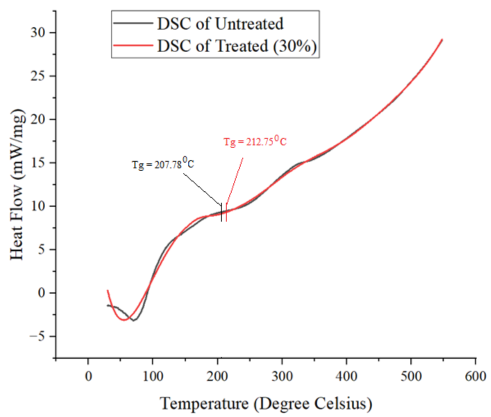
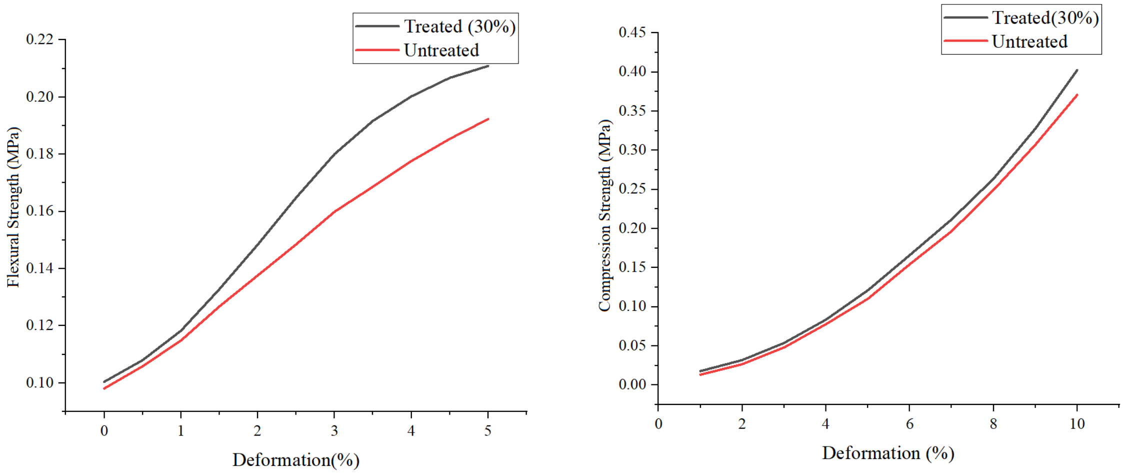


Figure 7: Heat Flow vs. Temperature graph.



(a) Flexural Strength (b) Compression Strength

Figure 8: Stress vs. Deformation graph for a) flexural strength and b) compression strength.

3.2 *Differential scanning calorimetry*

The thermal stability of both treated and untreated mycelium biocomposite samples characterized by DSC is shown in Figure 7. The heat flow versus temperature graph shows nearly identical exothermic and endothermic reactions.

The fire-retardant treatment of samples had little effect on the sample's exothermic and endothermic reactions. This behavior of the material indicated the self-extinguishing nature of the newly developed mycelium biocomposite material and this became more obvious when the results of the limiting oxygen index test were analyzed.

3.3 *Compression test and flexural test*

Figure 8 shows the compressive strength and flexural strength of treated and untreated mycelium biocomposite samples to deformation on a relative average basis.

Results of compression strength tests show that treatment with fire retardant chemicals has no significant negative effects on compression strength. Nonetheless, a slight increase in compression strength was evident from the results.

The results of the flexural test revealed that the fire-retardant chemical treatments have no significant negative impact on the flexural strength of mycelium composites. However, treated samples exhibited a modest increase in flexural strength when compared to untreated ones.

Table 2: Results of fire resistance tests

Test as per IS 1734 (part 3) 1983	Results (min)				Prescribed Value as per IS 5509:2000 [16]
	Weight percentage of Boron in samples (%)				
	0 (Untreated)	10	20	30	
Flammability (Time required for the second specimen to ignite after the first specimen gets ignited)	25.5	31	37	44.5	More than 30 min
Flame penetration (Time required for the flame to penetrate from bottom surface to top surface)	35	52.5	64	79	More than 15t/6 = 40 min (where, thickness of sample, t = 16 mm)
Rate of burning (Time required to lose weight from 70% to 30%)	17.5	24	31.5	45.5	More than 20 min

The presence of mycelium is responsible for the adhesion of the substrate materials and it grows into a dense composite structure. When fire retardant chemicals like borax-boric acid powder are added to the samples, it further increases the densification and mechanical properties like compressive strength and flexural strength are slightly improved

3.4 *Fire resistance and limiting oxygen index*

Fire retardant chemical treatment has considerably increased the fire resistance properties of mycelium biocomposite samples. The consolidated results of fire resistance tests confirmed that the samples having 30% boron compounds by weight exhibited the best fire resistance properties. The consolidated results of the fire resistance measurement values are shown in Table 2.

It is evident from the results that the untreated samples exhibited lower values than required to pass the tests for all three tests, flammability, flame penetration and rate of burning. Whereas, treated mycelium biocomposite samples conformed to the acceptance parameter for all three tests. The best values were observed for the samples having a weight percentage of 30% boron in them.

Limiting Oxygen Index (LOI) measures the minimum concentration of oxygen in a mixture of oxygen and nitrogen to support the flammability of materials for 15 s (as per ASTM D2863). Materials are classified as flammable if their Limiting Oxygen Index (LOI) value is less than 21% and any material with a value of more than 21% is considered a self-extinguishing material since it cannot burn under ambient conditions without the assistance of an external energy source [23]. Materials with a higher LOI rating have better fire-retardant qualities [23], [24].

Table 3: Limiting oxygen index of treated and untreated samples

Sample Specifications	LOI (O_2%)
Untreated samples	50
Treated samples (30% boron)	50

The LOI values for treated and untreated samples are shown in Table 3. The machine had a maximum capacity level of testing in an atmosphere of 50% Oxygen during the process. Both the samples did not ignite even when the test was conducted in the presence of maximum oxygen.

Therefore, the values of LOI were approximated to 50 in both cases. Since the fire retardant treated sample's LOI value is 50% which exceeds 21%, it can be considered as a fire-resistant material with self-extinguishing capabilities. However, it is equally interesting to note that even untreated samples exhibited a similar performance, making fire retardant treatment of samples not at all significant to LOI.

3.5 *Acoustic impedance*

The results shown in Figure 9 indicate that polyurethane foam samples have a poorer sound absorption coefficient than the treated mycelium bio-composite samples. Mycelium biocomposite samples having 30% boron in it has an average sound absorption coefficient of 0.38, compared to polyurethane foam of 0.29.

Fire retardant treated mycelium biocomposite

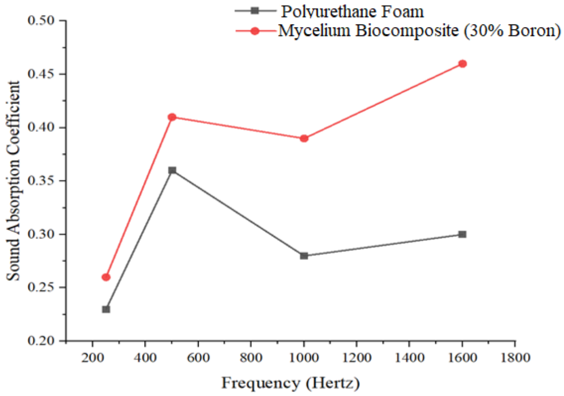


Figure 9: Result of impedance test.

material is an ideal choice for sound insulation applications (Table 4). Treated samples exhibited a better sound absorption coefficient than untreated samples. An average sound absorption coefficient above 0.2, is considered as a minimum requirement for a material to be a sound insulator [25], [26]. Treated mycelium biocomposite materials exhibited an excellent sound absorption coefficient to be used for sound insulation applications.

Table 4: Sound absorption coefficient at different frequencies

Sl. No	Frequency (Hz)	Polyurethane Foam	Mycelium Bio Composites	
			Treated (30% boron)	Untreated [12]
1	250	0.23	0.26	0.17
2	500	0.36	0.41	0.19
3	1000	0.28	0.39	0.33
4	1600	0.30	0.46	0.22

4 Conclusions

Mycelium is an environmentally friendly bio-composite material that easily decomposes as it is discarded to the environment after its useful life. Mycelium biocomposite based acoustic panels have the potential to replace plastic based acoustic panels that are widely used today. The results of an acoustic impedance test reveal that the treated biocomposite samples have an average impedance value of 0.38 and polyurethane foam has an average impedance value of 0.29. Therefore, this material is an effective sound absorber. Derived from completely organic materials like sawdust and coir-pith, it is a combustible material by nature. Treating the biocomposite with boron compounds has significantly increased its fire resistance. By conducting tests like flammability test, flame penetration test, rate of burning test, it was found that the best values were observed for samples having a weight percentage of 30% boron in them. TGA and DSC tests revealed that the thermal stability and exo/endothermic reactions of untreated and treated samples stay the same and have not varied much by treatment with chemicals. With an LOI value of 50%, biocomposite is self-extinguishing and has superior fire resistance. Compression strength test and flexural test reveal that fire retardant chemical treatment has not deteriorated the mechanical properties of treated samples. However, treated samples showed a slight increase in strength than untreated samples.

Author Contributions

T.A.: investigation, reviewing and editing; R.C.R: conceptualization, investigation, methodology, writing original draft; K.M.P: research design, writing—reviewing and editing; E.J.: research design, writing—reviewing and editing. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest

The authors declare no conflict of interest.

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