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Experimental Study on the Influence of Carbon Black Nano Particles on Ablative Properties of Carbon/Phenolic and Silica/Phenolic Composites

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ABSTRACT

This experimental study investigates the impact of Carbon Black Nanoparticles (CBNP) on the ablative properties of two commonly used composite materials in aerospace applications: Carbon/Phenolic and Silica/Phenolic composites. Neat and Nanofillers added laminates of these two composite types were prepared using a hand layup process followed by curing in an autoclave at 170 °C temperature and 5 bar pressure. Physical properties such as density, fibre content, degree of cure, and glass transition temperatures of all four types were found. Mechanical tests were conducted to observe the change in Interlaminar Shear Strength (ILSS), Flexural Strength (FS), and Flexural Modulus (FM) of the laminates due to the addition of fillers. Oxy-acetylene torch tests were carried out on all classes of laminates to study the influence of these new class of fillers on the ablative properties of composites such as Liner Ablation Rate (LAR) and Mass Ablation Rate (MAR). A heat flux of 835 W/cm² was applied for the ablation test in this experiment. The results indicated that the addition of CBNP (5 wt %) significantly enhances the ablation properties of Carbon/Phenolic composites (LAR & MAR reduce by 60 % & 62 % respectively), while it degrades for Silica/Phenolic composites (LAR & MAR increase by 17 % & 27 % respectively.

Keywords: Rayon-based carbon fabric; Silica fabric; Phenolic resin; Hand lay-up technique; Autoclave; Oxy-acetylene flame, LAR & MAR

1. INTRODUCTION

Aerospace launch vehicles can experience temperatures as high as 2500 °C in the atmosphere[1]. A Thermal protection system (TPS) is a crucial entity in safeguarding structures from high heat flux, high temperatures, and physical erosion. Polymer Composite materials are preferred as TPS over traditional metals/alloys in applications where extreme heat is present, like in rocket nozzles or spacecraft re-entry vehicles, because they protect the systems by sacrificing a layer of material through a controlled "ablation" process, effectively acting as a sacrificial heat shield while protecting the underlying structure from excessive heat. These ablative polymer composites are indispensable to the aerospace industry due to their capability to withstand varied hyperthermal environments[2].

1.1 Ablation Mechanism

The ablation mechanism involves a series of complex reactions when heat flux is applied to the specimen. The ablator decomposes to produce gases and a solid carbonaceous char as residue. Forming a protective layer on the composite surface is crucial for minimizing heat transfer into the inner layers. Traditional composite materials tend to create weak and brittle char layers, which are quickly removed by mechanical shear forces. A good ablator should resist mechanical erosion caused by high-velocity aerodynamic shearing under harsh conditions.

Among the ablative composites, phenolic resin-based composites are particularly suitable as ablative TPS due to their excellent mechanical and thermal properties and hence are employed as ablative liners to resist thermal and erosive environments[3]. When exposed to high temperatures, phenolic composite materials form a carbonaceous layer known as "char," which radiates heat and acts as an insulating barrier, protecting the underlying base material. As one layer of char is eroded by the combustion gases, another layer is produced to safeguard the virgin material.

The efforts to improve the ablative properties of thermal protection systems (TPS) have introduced a new class of nano-fillers. These fillers influence the mechanical and thermal properties of the materials, augmenting the overall performance of thermal liners and reducing their weight, which in turn improves the performance of launch vehicles. Within a polymer, nanofillers can create an inter-phase zone between reinforcement and matrix, yielding unique thermo-mechanical and thermo-physical properties that significantly alter the behaviour of the composite[4]. Nanomaterials have garnered attention due to their small size and unique surface and quantum tunnelling effects. Given that small additions of these nanofillers do not significantly change the overall density, they are used in composites designed for re-entry vehicles[5]. One of

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the advantages of polymer nanocomposites is that they give several properties to the primary polymer[6] both mechanical and ablative advantages. It was reported by researchers that, near to nano-particle surface, there is an inter-phase zone where the polymer chains are entangled with each other and create a zone with different properties compared with the neat material and it can influence the thermal stability too[7-8].

Carbon/phenolic and silica/phenolic composite materials are suitable for their ability to operate at extremely high temperatures for prolonged periods, hence utilized as ablative materials for rocket nozzles and motor casings. Rayon-based carbon fibre is preferred for ablative composites over PAN-based carbon fibre because of its significantly lower thermal conductivity, which is crucial for heat dissipation in high-temperature applications. When exposed to high temperatures, carbon phenolic composites experience a significant change in heat flow pattern primarily due to the pyrolysis of the phenolic resin, which transforms into a char, leading to a shift in heat flow pattern to a combination of conduction, radiation, and ablation depending on the temperature range and exposure duration. Similarly, High silica glass fibre/phenolic resin composites have the characteristics of ablation resistance and good thermal insulation performance; thus, they are often used in the heat-insulating system of many systems. Silica fabric combined with phenolic resins is used in thermal structures due to its resistance to high temperatures, chemicals, and abrasion.

Researchers have published the influence of several types of nanofillers when added to Phenolic resins in ablative composites. Some of these researches include the influence on Carbon/Phenolic composites when added with nanofillers like Nano silica[9], carbon nanotubes (CNTs)[10-11], Multi-walled Carbon Nanotubes (MWCNTS)[12-13], Graphene[14], Nano clay[15], CNF[16], SiC[17] etc and found that there are significant influences on the composite performance. Carbon Black Nano Particles (CBNP) are particularly popular as nanofillers in phenolic resin-based ablative composites because of their compatibility with phenolic resin[18]. A study by L. Asaro*, et al.* investigated carbon/phenolic linear erosion rate and the highest insulation index, which indicates the effectiveness of the char in protecting the virgin material[19]. Aravind Jithin*, et al.* explored the potential for improving silica/phenolic composites by adding fillers to enhance their performance in extreme thermal environments[20]. Their research specifically examined the effects of nanocarbon black particles as fillers.

2. SELECTION OF MATERIALS

The following materials-Resin, Reinforcement and Nanofillers are used in this experiment.

2.1 Resin

In this experimental study, Resole-type SIL grade phenolic resin is utilized as the matrix material. Two different solvents are employed to dilute the phenolic resin: ethylene glycol and 1-propanol. Ethylene glycol is chosen for the manufacturing of ablative samples because it promotes the formation of a gel phase during the curing process of the phenolic resin. This gel phase is beneficial for ensuring a homogeneous distribution of the resin within the carbon felt and helps prevent the agglomeration of nanoparticles. The viscosity of the resin is between 100 cps and 150 cps at 30 °C. It chars when heated beyond 250 °C and continues to do so until temperatures reach 500 °C. During this process, the resin completely transforms into amorphous carbon, forming an ablative surface that acts as a heat shield, wearing away in a controlled manner to protect inner surfaces. This characteristic is highly desirable for use in rocket nozzles and reentry vehicles.

2.2 Reinforcement for Carbon/Phenolic Laminates

Rayon-Based Carbon Fabric (RBCF) is used to manufacture Carbon/Phenolic composite laminate. Rayon fibres are transformed into carbon fibers with diameters ranging from 5 microns to 10 microns through processes of stabilization, carbonization, and graphitization to achieve the desired properties. RBCF is utilized as an ablative material in aerospace applications and rocket nozzles, serving as heat shielding due to its high thermal resistance. The selected fabric is an 8-hardness satin weave, contains 94 % carbon by weight, 255 GSM, and has a warp-breaking strength of 9.1 kg/inch and a weft-breaking strength of 6.8 kg/inch.

2.3 Reinforcement for Silica/Phenolic laminates

SIL-grade silica fabric is utilized in the production of the second type of laminate. This high-silica glass fibre cloth contains over 96 % weight of high-purity silica. It has a softening temperature of 1700 °C, exhibits near-zero thermal expansion, and demonstrates good chemical inertness. The chosen fabric is an 8-hardness satin weave,

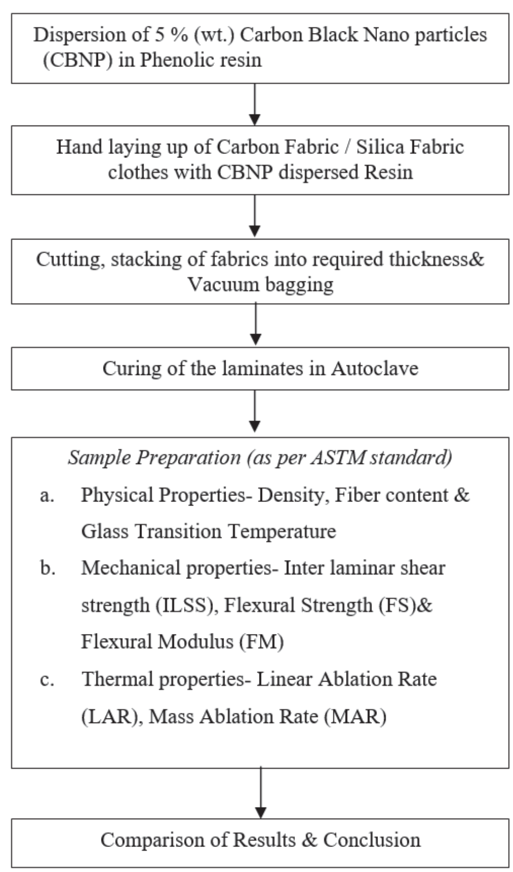


Figure 1. Flow chart of the experiment.

with a warp-breaking strength of 18 kg/inch and weft-breaking strength of 10 kg/inch

2.4 Filler Material

Carbon Black Nano Particles (CBNP), which consist of 99 % carbon, are in sizes from 50 nm to 500 nm with a surface area of approx.1050 m2/g, are used as fillers in both types of laminates: Carbon/Phenolic/CBNP and Silica/Phenolic /CBNP. The aim is to study how these fillers affect the properties of the resulting composites, with a primary focus on their ablative characteristics. These nanoparticles are spherical in shape and black in colour, with a density of 2.267 g/cm^3, a melting point of 3550 °C, and a boiling point of 3825 °C. They are integrated into composite materials due to their unique high surface area-to-mass ratio, which contributes to their excellent strength.

3. MANUFACTURING OF TEST SAMPLES

In this experimental study, the following types of laminates are prepared using a hand lay-up process followed by vacuum bagging and curing in an autoclave.

The following Flow chart depicts the steps followed in the experiment.

3.1 Hand Layup

Initially, phenolic resin is stored separately in one container. In another container, phenolic resin is mixed with 5 % (wt.) carbon black nanoparticles (CBNP), which are added and dispersed using a high-power stirrer (Fig. 2(a)). The stirring process promotes a homogeneous distribution of the nanoparticles and prevents agglomeration.

For the preparation of neat and nano filler-added laminates, two separate lengths of both carbon fabric and silica fabric are cut and laid on the prepared surfaces. For the neat laminates, phenolic resin is used to impregnate the fabrics. In contrast, for the nano filler-added laminates, phenolic resin that is dispersed with CBNP) fillers is utilized. Then the resin is applied to the fabrics using brushes and rollers (Fig. 2(b)) and is allowed to cure until the desired tackiness is achieved. The fabrics are then cut into required sizes for placing in moulds prior to vacuum bagging.

3.2 Vacuum Bagging & Autoclave Curing

The fabrics are cut into square shapes measuring 300 ×300 mm and stacked in a mould to achieve a final thickness of approximately 4 mm. The moulds are then vacuum-bagged to eliminate any voids (Fig. 2(c)). Four moulds are prepared, each containing different stacks of fabrics: Caron/Phenolic, Carbon/Phenolic/CBNP, Silica/Phenolic, and Silica/Phenolic/CBNP. These moulds are appropriately labelled and placed in an autoclave (Fig. 2(d)), where heat and pressure are applied simultaneously. The curing cycle lasts for seven hours and involves subjecting the composites to 90 °C for 1 hour, 120 °C for 2 hrs, 150 °C for 1 hour, and 170°C for 2 hrs, followed by cooling to 90 °C over the last hour. 5 bar pressure and of 0.9 bar vacuum are maintained. Once the laminates are removed from the autoclave chamber, they are sent for specimen preparation for testing (Fig. 2(e)).

3.3 Designation of the laminate samples:

All the test samples are designated as per following labels.

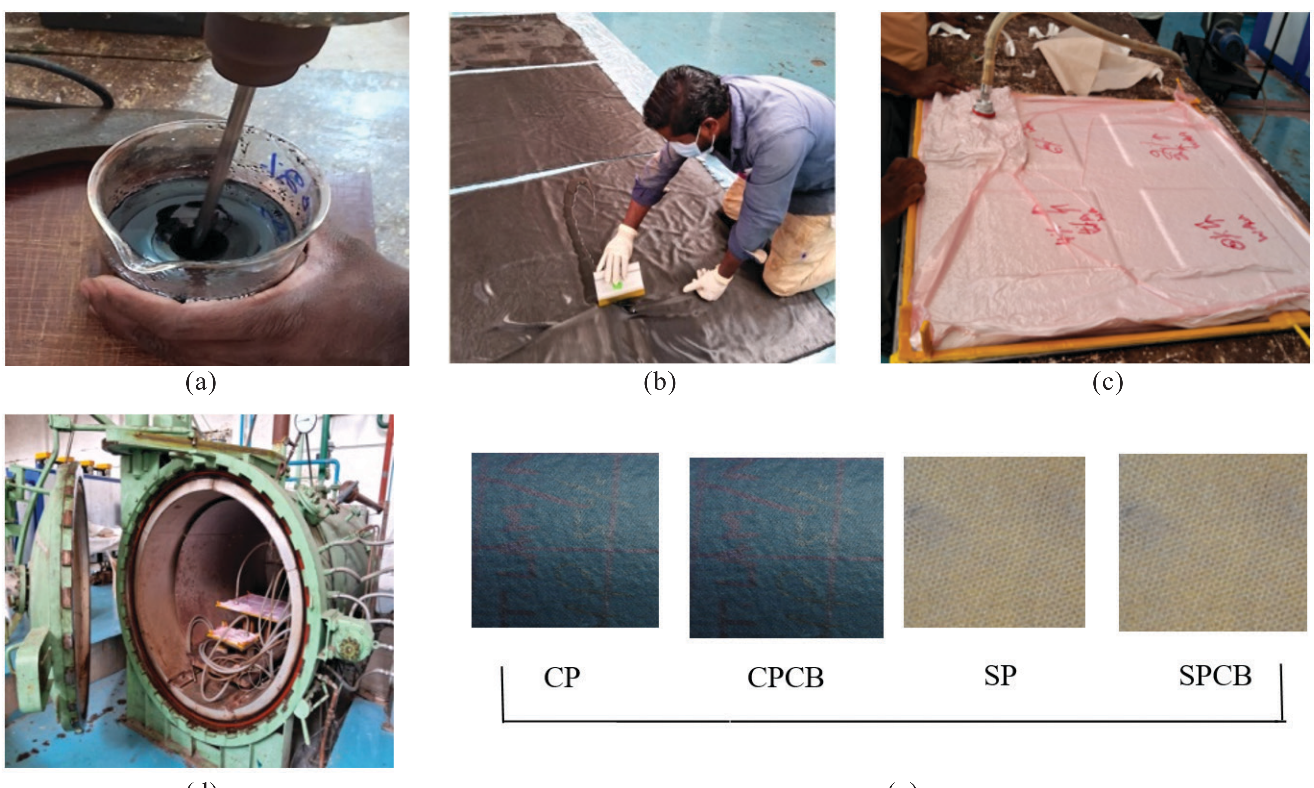


Figure 2. Manufacturing of laminates, (a) Dispersion of CBNP in resin; (b) Wetting of fabric with resin; (c) Vacuum bagging; (d) Curing in auto clave; and (e) Cured laminates.

- **CP :** Carbon/ Phenolic without filler (neat)
- **CPCB :** Carbon/Phenolic with 5 % (bywt.) Carbon Black nano particles (CBNP)
- **SP :** Carbon/ Phenolic without filler (neat)
- **SPCB :** Silica/Phenolic with 5 % (by wt.) Carbon Black nanoparticles (CBNP).

4. TESTING OF SAMPLES

The laminates of all the four types of composites are sent for preparation of samples for conducting physical, mechanical, and ablation tests according to ASTM standards.

4.1 PHYSICAL PROPERTIES

4.1.1 Density

The density of the composite samples is evaluated according to the ASTM D-792 standard. As per the test procedure, small samples of each laminate are prepared and weighed both in air and in water separately. Using Archimedes' principle, the density of each composite is calculated and recorded in Table 1.

4.1.2 Fibre Content

The fibre content of the composites is determined by measuring the resin content. For carbon fabric laminates, Nitric Acid Digestion Method (ASTM D-3171) is used for measuring resin content. In this method, the weights of the carbon fabric-based laminates are first measured in air. The samples are then placed in Nitric acid for complete digestion of the resin), after which the weights of the remaining fibres are measured. The difference in weight indicates the resin content.

For silica fabric laminates, the samples were weighed and then kept in a Muffler furnace at a temperature of 600 °C for 1 hr to ensure that the resin completely evaporated. Again, the difference in weight after this process reflects the resin content.

Once the percentage of resin content is measured, the percentage of fibre is calculated. The values of Fiber content are recorded in Table 1. It is observed that the incorporation of nanofillers enhances the bonding between the fibers and the matrix in both carbon fabric and silica fabric composites.

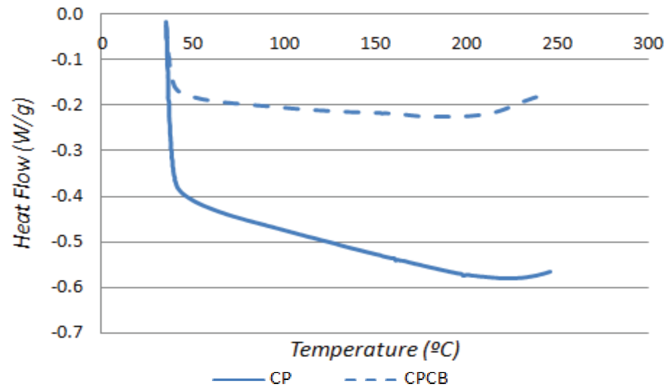
4.1.3 Ultrasonic Test

Ultrasonic Testing (UT) is the most widely used non-destructive inspection method for examining composites for evaluation of their structural integrity without damaging the material. All four types of composite laminate samples were subjected to UT testing, and it was observed that no internal defects like voids, delaminations, and porosity were detected.

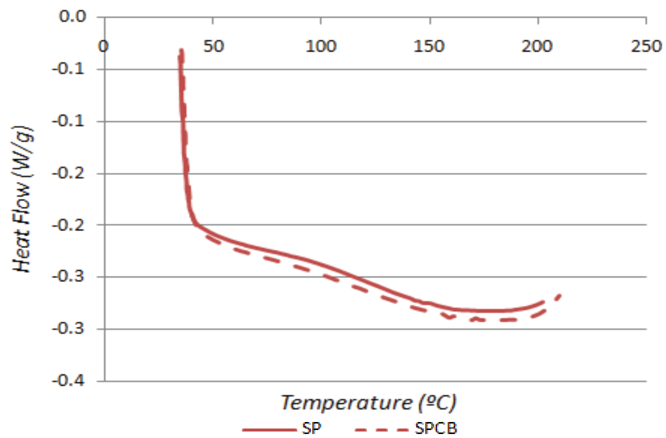
4.1.4 Glass Transition Temperature (Tg)

It is the temperature at which the transition of a material from a glass-like rigid solid to a more flexible, rubbery state takes place. A Differential Scanning Calorimeter (DSC 2920) instrument is used to measure the heat of the reaction and the glass transition temperature (Tg). Samples weighing between 3 to 8 mg are encapsulated and placed in the DSC, while an empty aluminium sample serves as a reference. The temperature is gradually increased from room temperature to 250 °C at a rate of 20 °C per minute. The rate of heat generation from the sample is measured over time, and the Tg is observed on the resulting graphs (Fig. 3(a) & 3(b)).

The Physical properties of all four types of composite laminates are recorded in Table 1.



(a)



(b)

Figure 3. Heat flow vs glass transition temperature, (a) CP vs CPCB laminates; and (b) SP vs SPCB laminates.

Table 1. Physical properties

Sample	Density (g/cc)	Fibercontent (%)	Tg (ºC)
CP	1.36	67.4	165.95
CPCB	1.34	72.1	164.9
SP	1.51	70.05	149.7
SPCB	1.49	71.12	159.74

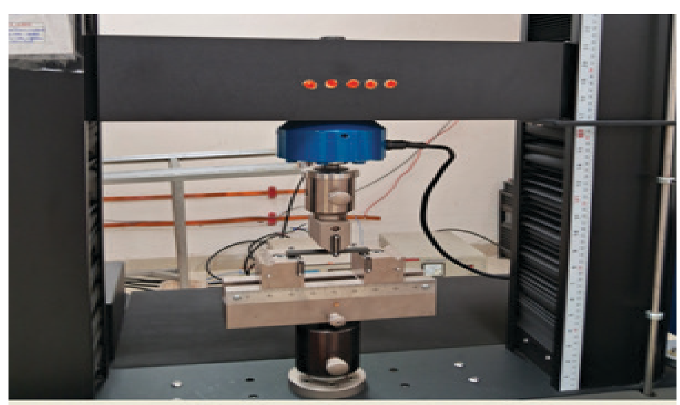
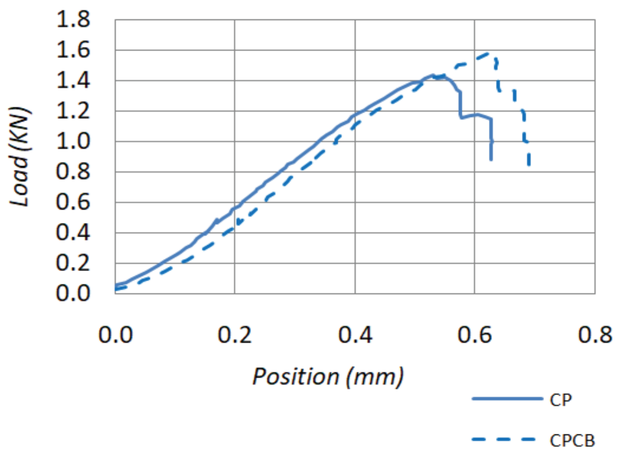
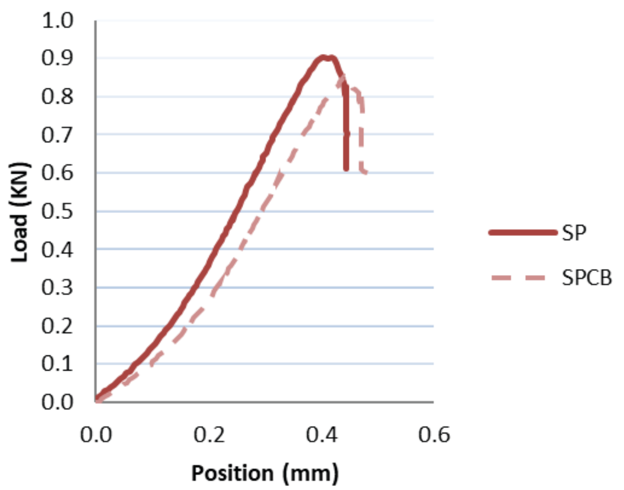


Figure 4. Set up for ILSS test.



(a)



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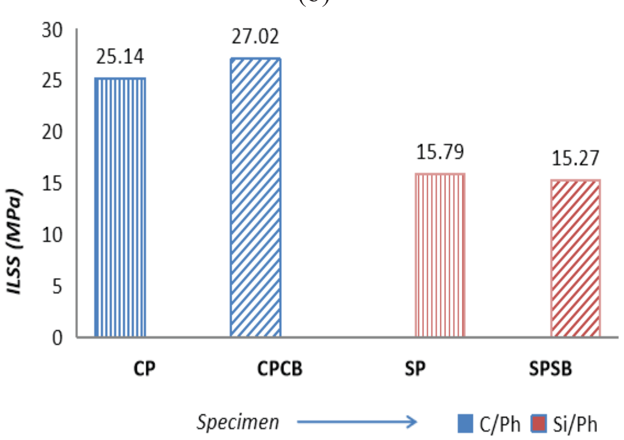


(c)

Figure 5. Influence of CBNP on ILSS, (a) CP vs CPCB laminates; (b) SP vs SPCB laminates; and (c) C. Inter Laminar Shear Strength (ILSS).

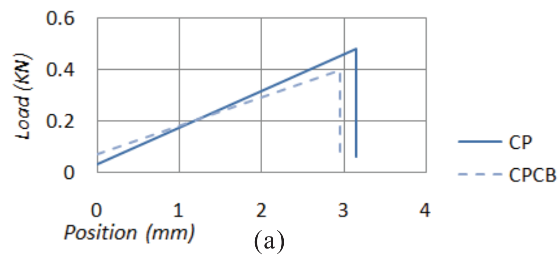
4.2 Mechanical Properties

Mechanical tests are conducted to analyse the influence of the CBNP filler on ILSS, FS & FM.

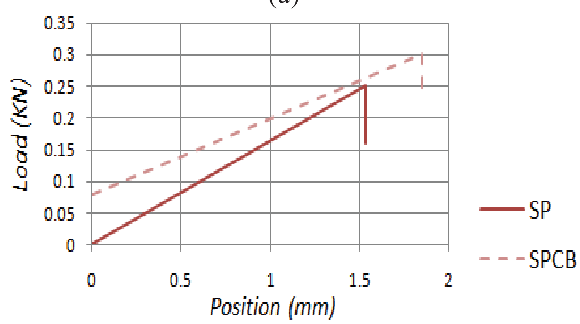
4.2.1 ILSS Test

Interlaminar Shear Strength (ILSS) is a crucial material property for the design of laminated composite structures subjected to transverse loads. ASTM D-2344 standard is

Figure 6. Set up for flexural test.



(a)



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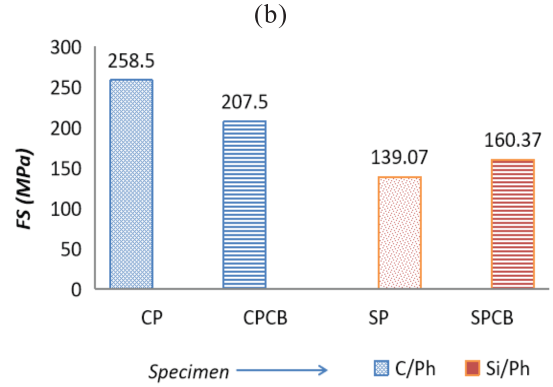

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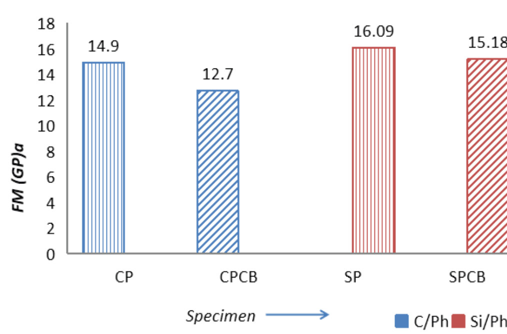

(d)

Figure 7. Influence of CBNP on FS & FM, (a) Load Vs position (CP vs CPCB); (b) Load Vs position (SP vs SPCB); (c) Flexural strength; and (d) Flexural modulus.

followed for conducting the three-point bending test (Fig. 4) and is conducted on an ADMET universal testing machine. In this test, specimens measuring 4x10x40 mm are used, with a span length of 16 mm, and a load of 1 kN is applied. The load versus position graph is shown for all samples of composites (Fig. 5(a) & 5(b)) and the influence of the nanofiller on ILL is shown in graph (Fig. 5(c))

The inter-laminar shear stress is calculated by using the Eqn. 1:

$$\tau_{max} = \frac{3P}{4bt} \tag{1}$$

where P = Load
b = Breadth of the specimen
t = thickness of the specimen

4.2.2 Flexural Test

The flexural tests are conducted in accordance with ASTM D790 using a 3-point loading method. Six sample specimens, each measuring 10 mm x 4 mm x 80 mm, are prepared for the test. The specimens are tested using an ADMET universal testing machine (see Fig. 6), equipped with a 1 kN load cell and a cross head speed of 1.7 mm/min.

Flexural strength (FS) is determined from the Eqn. 2:

$$F.S. = 3.P.L./2.b.e.^2 \tag{2}$$

Where:
F. S. = Flexural strength, *MPa*
P : Rupture load, *N*
L : Support span, *m*
b : Width of specimen, *m*
e : Thickness of specimen, *m*

Graphs for load versus position graphs, Flexural strengths & Flexural modulus are also generated.

The average mechanical test results from the six specimens in each category of the composites tested for mechanical properties are summarized in Table 2.

Table 2. Mechanical properties

Sample	ILSS (MPa)	FS (MPa)	FM (GPa)
CP	25.14	258.5	14.9
CPCB	27.02	207.5	12.7
SP	15.79	139.07	16.09
SPCB	15.27	160.37	15.18

4.3 Thermal Tests

Ablation is an erosive phenomenon that results in part of the material being removed by combined thermo-mechanical, thermo-chemical and thermo-physical influences due to a combustion flame or other heat source at high temperature, pressure and velocity. To evaluate the ablative properties of composites, an Oxy-Acetylene test bed (OTB) is used for simulating hyper thermal environment characterized by high heat flux and elevated temperatures and conducting the tests as per ASTME 285 standard, as shown in Fig. 8. In this test set up, temperatures of up to 3000 °C can be simulated. Literature indicates that the addition of nanofillers can significantly

Figure 8. Oxy acetylene test bed.

influence the mechanical properties of the char, as well as its thermal conductivity and pyrolysis processes. In this test, both the Linear Ablation Rate (LAR) and Mass Ablation Rate (MAR) of the test samples are determined. Linear ablation rate is the erosion in thickness of the composite laminate per unit time (mm/s), while Mass ablation rate is the reduction in mass per unit time (g/s). For the experiment, three samples of 100 mm x 100 mm size from each laminate type were prepared.

The ablative test specimen is held in the fixture of the OTB firmly. The torch is positioned at 20 mm from the sample surface to achieve the desired incident heat flux of 835 W/cm². During the approach of the torch, the samples are shielded with insulating panels to prevent undesired pre-heating. The specimen surface is exposed to the flame until the thickness is completely burned through, and the time for this process is recorded. After the exposure time ends, the samples are rapidly cooled using a stream of carbon dioxide gas to avoid post-test oxidation phenomena.

4.3.1 Linear Ablation Rate (LAR) /Erosion Rate

LAR, or the erosion rate (in mm/sec) of the composite, is determined when exposed to high temperatures. The thickness of each sample is measured at the beginning of the test. The specimen is then placed in the test setup and subjected to heat flux until a throughhole is formed. The time taken for piercing is recorded. The erosion rate (mm/s) is calculated by dividing the recess thickness by the time taken to pierce. The average erosion rate of the three samples is recorded in the table for each type of composite laminate. Graph for Linear ablation rates of each type of composite is depicted in Fig. 9(a).

4.3.2 Mass Ablation Rate (MAR)

The Mass Ablation Rate (MAR) measures the mass loss of materials due to erosion when exposed to high temperatures and high-velocity flames. Initially, the samples are weighed, and the values are recorded. After the samples are exposed to

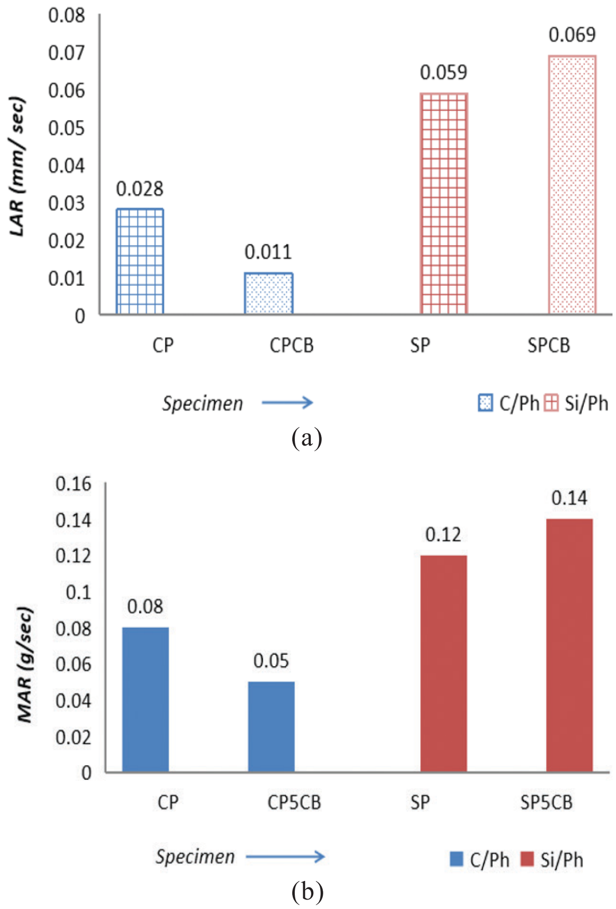

Figure 9. Influence of CBNP on ablation properties, (a) Linear ablation rates; and (b) b. Mass ablation rates.

the flame and experience piercing from the high temperature, their mass is measured again. The difference in mass is then divided by the time taken to burst the thickness, which gives the Mass ablation rate (g/sec). The average mass loss rate of the three samples is recorded in the table for each type of composite laminate. Graph for Mass ablation rates of each type of composite is depicted at Fig. 9(b).

The average values of Linear ablation rate and Mass ablation rates of all the four types of laminates are recorded in Table 3.

Table 3. Ablative Properties

Sample	LAR (mm/s)	MAR (g/s)
CP	0.028	0.08
CPCB	0.011	0.05
SP	0.059	0.11
SPCB	0.069	0.14

5. ANALYSIS OF RESULTS

Based on the results obtained from the tests, the following summary highlights the influence of carbon black nanofiller (CBNP) on the properties of carbon/phenolic and silica/phenolic composites..

5.1 Physical Properties

- It is observed from Table 1 that there is not a significant difference in the densities on account of the addition of CBNP nanofillers. As brought by literature, since the density of composites is not influenced by nanofillers, there will be negligible effect on weight
- There is slight increase in Fiber contents (2-6 %) of both types of composites. It is attributed to the strong bondage of the nano fillers with fibers at interface zones.
- Regarding Glass Trnsition temperature (Tg), which is an important physical property of polymer composites, there is a negligible change for Carbon/Phenolic composites when CBNP fillers are added because carbon black has a slightly higher resistance to softening at elevated temperatures, while it is increased by 7 % for Silica Phenolic composites with CBNP, this is because the carbon black particles disrupt the molecular interactions and soften at a lower temperature within the polymer matrix.

5.2 Mechanical Properties

- The results shown in Table 2 indicate that when added in specific percentages, CBNP can influence the ILSS for both types of composites. For Carbon/Phenolic composites, the addition of nanofillers enhanced the ILSS by 7.5 %, conversely, there was a 3 % decrease for silica fabric composites
- b) The analysis of Flexural strength reveals a decrease of 19 % in for carbon phenolic composites when dispersed with CBNP fillers. In contrast, silica phenolic composites show an increase of 15 %. Flexural Modulus is reduced by 14 % and 5 % for CPCB & SPCB composites which is attributable to the agglomeration of the nanofillers.

5.3 Ablation Properties

- For CPCB composites, both the linear and mass ablation rates showed significant improvements. As seen in Fig. 8(a) & Fig. 8(b), the addition of CBNP reduces both the linear ablation rate (LAR) and Mass ablation rate (MAR) for carbon/phenolic composites by 60 % & 62 %, respectively. This significant reduction in ablation rates suggests improved thermal resistance because the addition of CBNP nanofillers into Carbon/Phenolic composites is significantly influencing the formation of char and its ability to withstand severe thermal environment
- In contrast, for SPCB composites, the addition of CBNP nanofillers led to deterioration in both the linear and mass ablation rates, with increases of 17 % & 27 % respectively. This is attributed to the formation of Silica oxide during ablation which speeds up the consumption of the composite and decreases ablation resistance.

6. CONCLUSION

The experiments outlined above determine the influence of CBNP at 5 % (wt.) on carbon/phenolic and silica/phenolic composites, with a particular emphasis on their ablative properties. It was observed that the addition of Carbon Black Nanofillers (CBNP) significantly enhances the thermal resistance of carbon fibre/ silica composites due to the strength provided to the char by the fillers in severe thermal conditions.

In contrast, for the silica/phenolic composite, after the addition of CBNP, ablation resistance decreased due to the formation of Silica Oxide during the ablation process, which is responsible for faster consumption of the composite.

The improved ablative properties of carbon/ phenolic composites with CBNP suggest their potential use in high-temperature applications such as rocket nozzle liners. However, despite the Silica/phenolic composite's ability to withstand high-temperature environments, the addition of CBNP showed deterioration in ablative properties. The study was conducted under controlled laboratory conditions, which may not fully replicate real-world aerospace environments. Further testing under simulated atmospheric conditions is recommended. This suggests that further research is needed to optimize their composition for similar uses.

Future studies may be explored for understanding the effect of varying percentages of CBNP on ablative properties of the ablative composite materials which are essential for aerospace applications as Thermal Protection Structures (TPS).

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His contribution to the current study include: conceptualization, planning of experiments, guidance in manuscript drafting, analysis of the results and discussions on concluding.