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A Comparative Evaluation of Mechanical and Ablative Properties of Ceramic/Phenolic Composites filled with different fillers

M. Sasikumar [1], R. Balaji [2]

[1,2]*School of Mechanical and building Science (SMBS), VIT University, Chennai Campus, India.*

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- ✓ Back Face Temperature

[1]msasikumar@msn.com
[2]baluji.r@gmail.com

Abstract

Ceramic Fibre Reinforced Polymer composites are attractive materials for various engineering applications, particularly for high temperature application such as a heat shield. In this study, Alumina/Silica based ceramic fibre reinforced phenolic resin composites filled with different fillers such as Yttria-stabilized zirconia, microsilica and cenosphere were prepared using traditional hand layup technique. Three different fillers were filled with phenolic resin in various filler concentrations. Mechanical characterizations such as tensile and compressive were carried out to quantify the effect of fillers on the mechanical properties of the filled composites. An enhancement in mechanical properties of the filled composites was found as concentrations of filler content increased. However, beyond certain concentrations, further addition of the fillers showed a reduction in mechanical properties of the filled composites. Ablation behaviors of the filled composites were investigated by oxyacetylene ablation test in terms of the linear ablation rate, mass ablation rate and back face temperature. Experimental results showed a favorable ablation resistance with the addition of all the micro sized fillers. In comparison with microsilica filled ceramic/phenolic composites, the phenolic matrix embedded with the yttria-stabilized zirconia particles significantly improved the ablative properties of the filled composites in terms of mass ablation rate and linear ablation rate but not when compared to cenosphere filled composites. The result of the mechanical and the ablation characterization of different fillers filled composites were compared and discussed in detail.

1. Introduction

The Composite material is an advanced engineering material composed of two or more different materials or phases (matrix phase and reinforcing phase), which combined together with well-defined interfaces. The resulting composites have bulk properties, which significantly differs from individual constituents of the composites. Out of various polymer matrix composites, Phenolic based polymer matrix composite is widely used in high temperature application such as Aerospace industries, because of its unique thermal resistance.

Phenolic resin is a class of thermoset resin generally used for high temperature application. Phenolic resin is an engineered high polymer, which is formed by either step-growth polymerization reaction in presence of catalyst, either acid or base. The high cross-linking in cured phenolic resin gives its hardness, good thermal stability and chemical resistance. The curing behaviour of phenolic resins is totally different from another kind of thermoset resins such as epoxies, due to the fact that water is being generated during the cure reaction.

Phenolic-based polymer composite offers many favourable properties such as high thermal, corrosion and creep resistance and excellent fire/smoke/smoke toxicity properties. Phenolic composites exhibit good dimensional stability over a wide range of temperature. At the same time, phenolic composite showed merely a moderate mechanical strength and it needs high shaping pressure during manufacturing [1]. For high temperature application such as Thermal Protection System (TPS), the candidate polymer composites should have good mechanical strength with high thermal stability.

It is necessary to augment the mechanical properties, especially mechanical properties and ablation properties of the phenolic composites for potential application for high temperature application. Several approaches such as the addition of fillers, binders, etc., have been made to improve the structural and the ablation properties of the phenolic composites. Particularly to increase the mechanical properties of phenolic composites various reinforcing fillers such as Organic Tannin [2,3], Montmorillonite Clay [4], Zirconium [5,6], NanoSilica [7,8], Carbon Nanotubes (CNT) [9,10] etc, are added. In a work, Navaneethakrishnan et al. fabricated a natural coconut shell particulate (CSP) reinforced composites and characterized for mechanical properties. The addition of CSP increases the mechanical properties of the composites such as tensile, flexural and impact energy absorption capabilities [11]. Similarly, in another work, Bhaskar et al. studied the effect of coconut shell particles on physical and mechanical properties of epoxy-based composites [12]. The density and tensile strength of the filled composite continuously decrease with the increase of filler concentration. Aigbodion et al. did thermal degradation study to find the effect of orange peels ash (OPA) on the thermal properties of the high-density polyethylene composites by means of thermogravimetric analysis and found that the addition of OPA considerably increases the thermal degradation temperature of the composites [13]. In another work, Gowthami et al. investigated the effect of silica on the thermal and mechanical properties of sisal fibre reinforced polyester composites [14]. The experimental characterisation revealed that the addition of silica considerably increases the thermal and mechanical properties of the sisal/polyester composites. In a series of our previous work, we investigated the effect of cenosphere as filler on mechanical behaviour, ablation behaviour and thermal stability of the phenolic composites and found that the addition of micro-sized filler increased the thermal and the ablation behaviour of the ceramic/phenolic composites significantly [15-17] and also investigated the effect microsilica on mechanical properties of phenolic composites [18].

The aim of this study is to quantify and compare the effect of three different filler on the mechanical and ablative properties of the filled phenolic composite. Ceramic reinforced with phenolic resin composites were fabricated with the various percentages of three different fillers such as yttria-stabilized zirconia, microsilica and cenosphere using traditional hand lay-up technique. Mechanical properties such as tensile strength and compressive strength are investigated using experimental tests. Ablation behaviour is characterized by the oxyacetylene gas burner in terms of back face temperature, linear ablation rate and mass ablation rate [19]. The results are compared and discussed within the subject of the effect of three different fillers on the mechanical and the ablation properties of the filled composites.

2. Materials and methods

In this present study, Phenolic resin and Ceramic woven fibre are used as matrix and reinforcement materials because of unique thermal stability and good chemical resistance. The physical properties of the phenolic resin and ceramic woven bi-directional fibre are tabulated in Table 1.

Table: 1 Properties of Ceramic Woven Fibre and Phenolic resin

Reinforcement: **Ceramic Woven Fibre**	Matrix: **Phenolic Resin**
Density : 2.7 g/cc GSM: 200gsm Constituents: Alumina (<99%) Thickness: 0.6mm Color: White	Density : 1.2 g/cc Constituents: Phenol and Formaldehyde Curing Temperature: 160˚C Color: Dark red

In the current study, yttria-stabilized zirconia is used as one of the micro-sized filler to enhance the properties of phenolic composites. Yttria-stabilized zirconia (YSZ) is a white ceramic crystalline oxide of zirconium fully stabilized with yttrium oxide. The properties of yttria-stabilized zirconia are listed in table 2. Silica fume is an amorphous material made up of glassy spheres of silicon dioxide and it is used as one of the fillers in the study. Silica fume and also called as microsilica is collected as a by-product of the Carbo-thermic reduction of high-purity quartz with carbonaceous materials in electric arc furnaces during the production of silicon and ferrosilicon alloys. The properties of microsilica are listed in Table 2. Cenospheres are unique free-flowing powders composed of hard-shelled, hollow, minute spheres are used as one of the fillers in the study. It is recovered as an industrial by-product from the thermal power plant during the combustion process, which arises along with flues of gases. The properties of cenosphere are listed in Table 2.

By using the traditional hand-lay-up technique, the ceramic/phenolic composites are fabricated by incorporating the micro-sized yttria-stabilized zirconia, microsilica and cenosphere as fillers. In this study, the filler weight fraction is varied from 5 to 20% to quantify the effect of three different filler on the ablation and the mechanical behaviour of the filled composites. The fabricated 7 layers of laminated composites are placed in hot air woven at a temperature of 140˚C at a very slow rate of heating (2˚C per minute) for curing and again post curing is done at 180˚C for about two hours.

Table: 2 Properties of Yttria-stabilized zirconia, Microsilica and Cenosphere.

Properties	Yttria-Stabilized Zirconia (YSZ)	Cenosphere	Microsilica
Density	2.3g/cc	0.85g/cc	0.7g/cc
Constituents	Zirconia and yttrium oxide	Silica and alumina	Silica (<95%)
Particle Size	40 µm(avg)	150 µm(avg)	5 µm(avg)
Color	White	Light grey	White

3. Characterisation and Testing

An experimental characterization such as tensile strength and compression strength is done to analyse the effect of three different fillers on the mechanical behaviour of the filled composites. Mechanical test such as tensile and compression are done at ambient condition using UTM (Shimadzu's Autograph AG-X Plus 50K Universal testing machine), according to ASTM procedures D3039 [22] and D3401 [23] respectively. The optical image of unfilled and three different filler filled composites are shown in Fig 1.

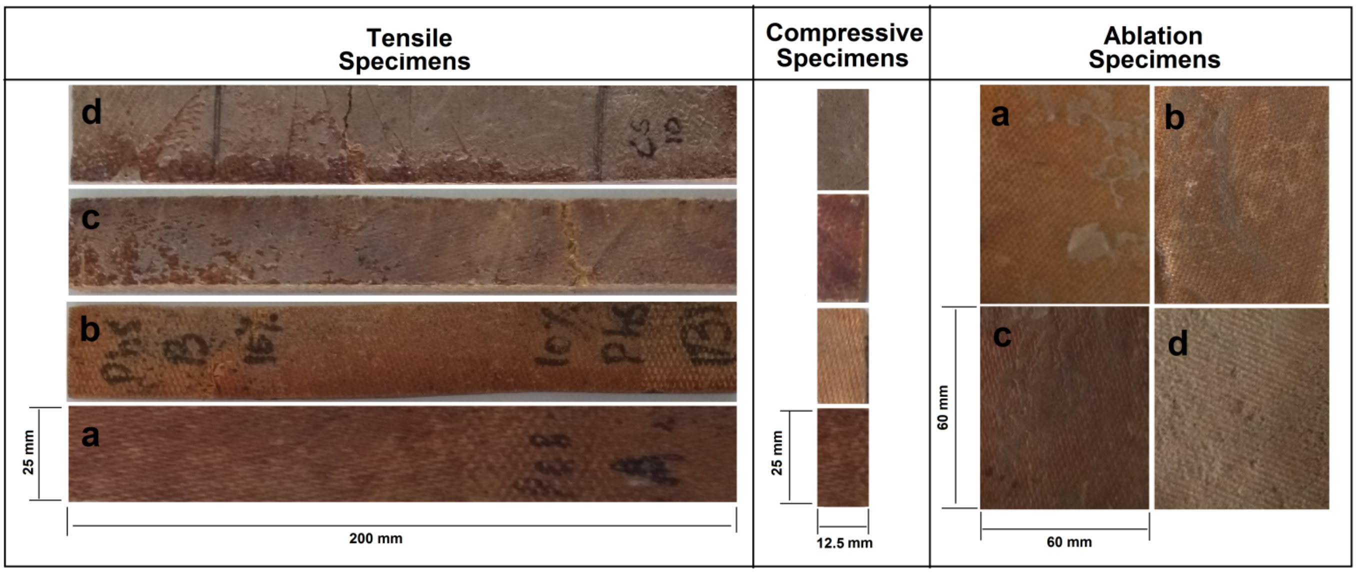

Fig 1. Optical image of unfilled and filled Composite specimens (a. Unfilled composites, b. Microsilica filled composites, c. Yttria-stabilized zirconia filled composites, d. Cenosphere filled composites)

In this study, the severe hyperthermal environment for the ablation test is stimulated using an oxyacetylene flame system to evaluate the effect of three different fillers on the ablation properties of the filled ceramic/phenolic composites according to ASTM E285-80 [24]. This standard test method covers the screening of ablative materials to determine the relative thermal insulation effectiveness when tested as a flat panel in an environment of a steady flow of hot gas provided by an oxyacetylene burner. In the current study, hot combusted gases from the oxyacetylene flame system are narrowed normal to the composite sample throughout the ablative test time of 120s. The composite specimen is kept at 20mm away from the torch nozzle during the entire study. Two thermocouples are used to measure the front and back face temperature of the composites. The ablation behaviour of the filled composites is characterised for mass ablative rate, liner ablative rate and thermal insulating effectiveness. The insulating effectiveness is determined from the back face temperature of the filled composites, which was measured during ablation test.

The ablation parameters such mass ablation rate and linear ablation rate are calculated using following formula [20,21]: Mass ablation rate (**ARM**) = (m1 – m2) / t

Linear ablation rate (**ARL**) = (d1 – d2) / t

where ARM is Mass ablation rate (g/s), m1 and m2 are mass of specimen measured before and after ablation test and t is ablation time (s), ARL is the Linear ablation rate (mm/s), d1 and d2 are thickness of specimens before and after ablation test (mm).

4. Result and discussion

4.1. Tensile behaviour

The tensile test was done to clarify the effect of three different fillers on the tensile strength of the ceramic/phenolic composites. The tensile strength of the phenolic composite embedded with various concentrations of yttria-stabilized zirconia, microsilica and cenosphere are tabulated in Table 3. At a first glance of the experimental reports of filled composites, the result indicated that up to a certain percentage, the addition of fillers to the ceramic/phenolic composites increased the tensile strength, similarly like previously reported work with coconut shell particulate (CSP) reinforced polymer composites [11].

Table 3. Tensile strength of the unfilled and three different filler filled phenolic composites

Materials	Unfilled Composites (N/mm^2)	5% filled Composites (N/mm^2)	10% filled Composites (N/mm^2)	15% filled Composites (N/mm^2)	20% filled Composites (N/mm^2)
Microsilica	49.81	62.58	66.93	69.82	68.31
Yttrai-Stabilized Zirconia	49.81	61.36	64.61	66.95	65.03
Cenosphere	49.81	58.02	63.5	61.72	60.06

With 5% and 10% of yttria-stabilized zirconia content dispersed in the composites increased the tensile strength by 23% and 30% respectively, when compared to the unfilled composites, which can be observed from Fig 1. Microsilica filled composite also showed a similar increment of about 26% and 34% for 5% and 10% filler concentration, respectively as like cenosphere filled composites, which showed in Fig 2.

In all the cases, the significant enhancement in the tensile strength was inferred by the action of uniform dispersion of micro-sized filler content in phenolic composites as shown in SEM image (Fig 3). These three micro-sized fillers can exhibit greater interactions with phenolic resin and ceramic fabric, resulting in a good dispersion in the composites [16]. These uniformly dispersed filler contents in the phenolic composites enhance the load transfer efficiency of the phenolic resin, which directly improves the load carrying ability of the filled composites. Above the same phenomenon was noticed previously by the addition of cenosphere with high-density polyethylene composites [25].

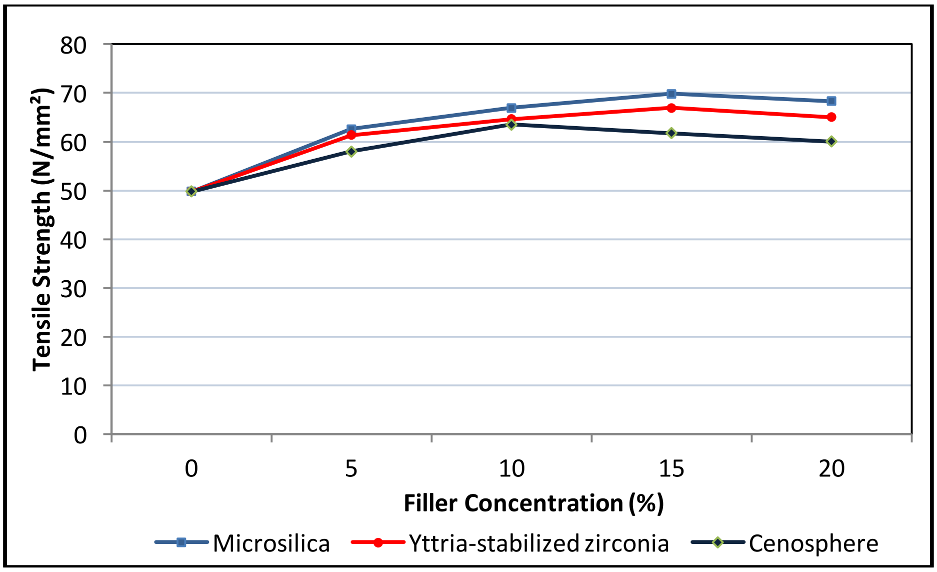

Fig: 2 Effect of fillers on the tensile behaviour of the composites

For 15% concentration, yttria-stabilized zirconia and microsilica filled composites showed a further increase in tensile strength of the composites, which is about 34% and 40% respectively, when compared to the unfilled composites. However, the composite loaded with 15% of cenosphere showed a reduction in tensile strength when compared to 10% concentration of cenosphere, but not significantly less when compared to the unfilled ceramic/phenolic composite as shown in Fig 2. Reduction in tensile strength can be due to two actions, poor wettability and poor adhesive. The addition of these micro-sized fillers increased the viscosity of the filled matrix, which makes the resin hard to be impregnated into the woven fibre. Thus, more concentration of cenosphere directly affects the wettability of the fibre. Again, the addition of more volume of cenosphere to the phenolic resin reduces adhesive strength of the resin with woven fabric.

When comparing to the composite loaded with 15% of both yttria-stabilized zirconia and microsilica, cenosphere filled composite showed a reduction in tensile strength in this filler concentration as can be observed from Fig 2. It is inferred by the particle size of the fillers. The viscosity of the modified phenolic resin changes with respect to the size and concentration of fillers. Previously it was revealed that the particle size and wall thickness of the cenosphere play an important role in the properties of the composites [26,27]. It was reported in

Table 2 that the average size of cenosphere particles is much larger than the size of microsilica and yttria-stabilized zirconia particles. The addition of this larger sized cenosphere decreases the flow-ability of the filled resin to a great extent when compared to smaller sized filler filled phenolic resin. Thus, the additions of this comparably larger sized cenosphere make the filled composites to fail earlier when compared to the other filler composites. For 20% concentration, all the filler-loaded composites showed a decrease in tensile strength when compared to the 15% filler concentration, but not less than unfilled composite, as in Fig 2.

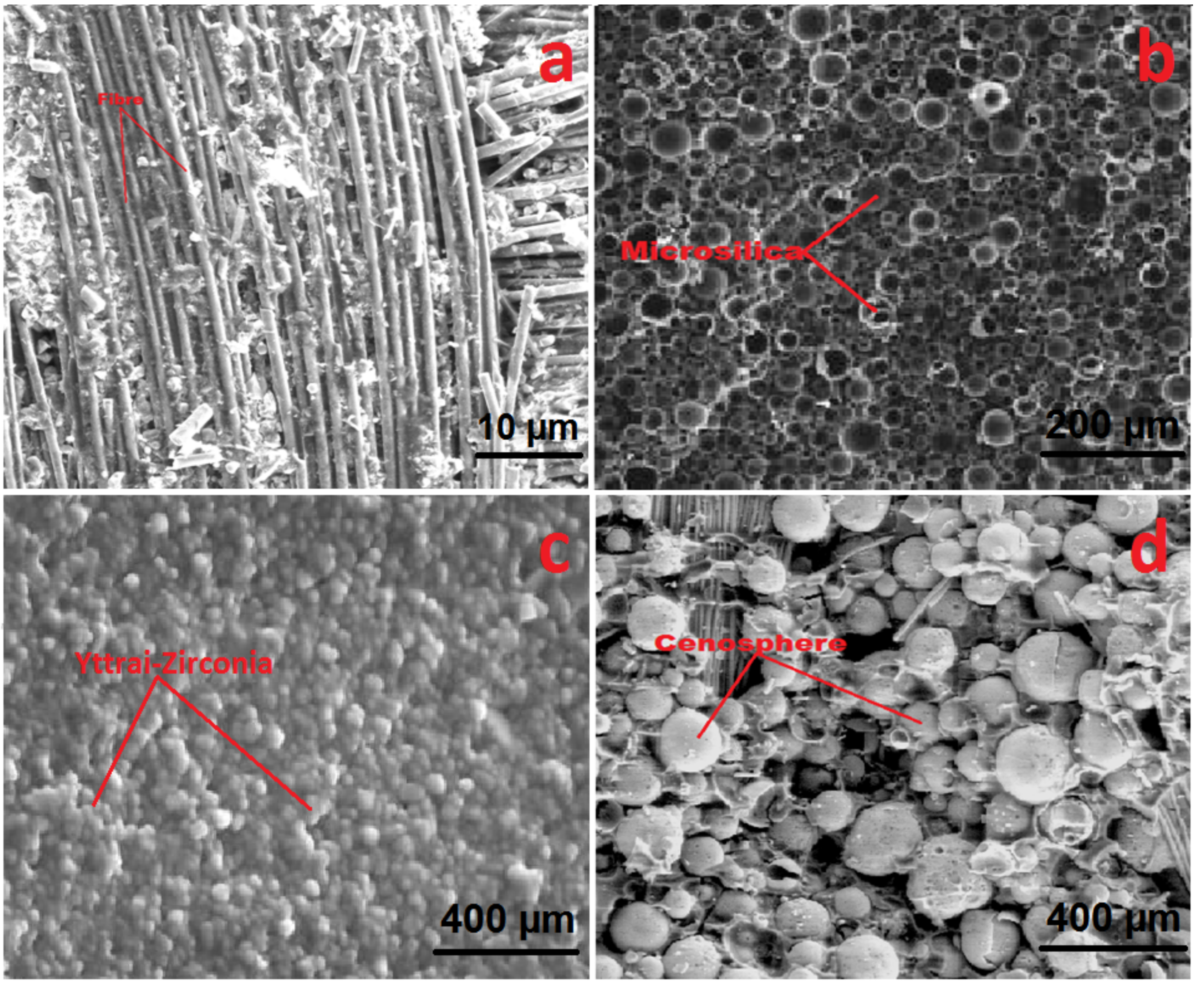

Fig 3. SEM image of unfilled and fillers filled composites (a: Unfilled composites, b: 20% microsilica filled composite, c: 20% yttria-zirconia filled composite, d: 20% cenosphere filled composite)

Overall, the composites filled with microsilica showed improved tensile strength than the composites filled with both yttria-stabilized zirconia and cenosphere. It is due to again particles size of the fillers. Microsized microsilica dispersed more uniformly in the phenolic composites than the hollow spherical cenosphere which in turn uniform dispersion of microsilica showed improved tensile properties than the other filled composites. From Fig 2, it can be observed that the tensile strength of the filled composite increased significantly for finer particles than larger particles. Hence, the addition of the fillers with the composites increased the tensile strength of the filled composites, again beyond the certain concentration the further addition of fillers decreases the tensile strength of the filled composites.

4.2. Compressive behaviour

Like the tensile test, to clarify the effect of different fillers on the compressive strength, the compressive test was done on the filled composites. The compressive test results of various concentrations of filler loaded composites are tabulated in Table 4. It is observed from the graphical representation (Fig 4), the addition of all the fillers with ceramic/phenolic composites increased the compressive strength of the filled composites.

For 5% and 10% of all the fillers loaded composites showed an increase in compressive strength of the composites as same like tensile strength which can be inferred from the Fig 4. The composites filled with 5% and 10% of microsilica showed an increase of about 25% and 37%, respectively, when compared to the unfilled composites and yttria-stabilized zirconia loaded composites showed an increase of about 22% and 34% for 5%

and 10% filler concentration respectively, whereas 5% and 10% of cenosphere loaded composites showed an increase of about 34% and 47% respectively. The increase of compressive strength of the fillers loaded composites are due to the uniform dispersion of micro-sized filler particles in the composites. These micro-sized particles have greater interaction with both matrix and fibre, which directly increased the load transferring behaviour of the phenolic resin. Thus, the addition of micro-sized fillers increased the compressive strength of the filled ceramic/phenolic composites.

Table 4. Compressive strength of the unfilled and three different filler filled phenolic composites

Materials	Unfilled Composites (N/mm^2)	5% filled Composites (N/mm^2)	10% filled Composites (N/mm^2)	15% filled Composites (N/mm^2)	20% filled Composites (N/mm^2)
Microsilica	17.47	21.94	24.01	25.85	24.90
Yttria-Stabilized Zirconia	17.47	21.32	23.41	24.65	23.09
Cenosphere	17.47	23.52	26.03	25.25	23.83

Again, like in the case of tensile property, the composites filled with 15% of microsilica and yttria-stabilized zirconia showed an increase in the compressive strength. However, the composite filled with 15% of cenosphere showed decreases in the compressive strength when compared to 10% of cenosphere filled composites. It is due to the same phenomenon as discussed in the tensile strength that is due to poor wettability and poor adhesion. The additions of all the fillers increase the viscosity of the phenolic resin, which results in poor wettability of woven reinforcement. Also, the addition of filler concentration reduces the contact surface of the phenolic resin with ceramic reinforcement, which directly affects the mechanical strength of the filled composites. The composites filled with 20% filler concentration showed a decrease in compressive strength when compared to the unfilled composites, but not less than the compressive strength of the unfilled composite.

Unlike tensile properties, the composites filled with cenosphere exhibit improved compressive behaviour than the composites loaded with microsilica and yttria-stabilized zirconia, as seen in Fig 4. It can be inferred from the two facts that the compressive behaviour of raw cenosphere particles is superior to microsilica and yttria-stabilized zirconia and other the nature of the cenosphere particles was hollow, which was lacking in other two fillers since they are solid particles. It is previously known that the average compressive strength of the cenosphere particles is around 3500psi. These two qualities of cenosphere particles directly influence the compressive behaviour of the cenosphere filled composites and make the cenosphere filled composites to perform better than the other two fillers filled composites. From Fig 4, it is inferred that microsilica filled composites showed improved compressive strength when compared to yttria-stabilized zirconia filled composites and again this is due to the particles of the filler, same like tensile strength. Hence, the addition of fillers increases the compressive strength of the filled composites and again beyond the certain concentration, further the addition of fillers decreases the compressive strength of the filled composites.

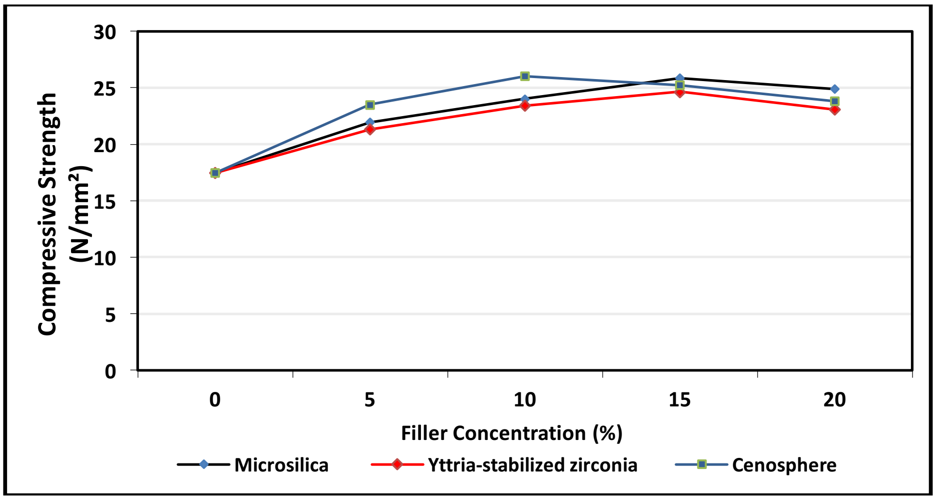

Fig 4. Effect of the fillers on compressive behaviour of the filled composites

4.3. Ablation behaviour

The ablation test is carried out to evaluate the effect of different fillers on the ablative properties of the filled phenolic composites. The ablation behaviour of the filled composites is characterised in terms of Mass Ablative Rate, Linear Ablative Rate and thermal insulation effectiveness by means of back face temperature.

Overall, experimental results of the ablation test revealed that the ablation behaviour of the filled composites significantly improves with the addition of three different filler. The ablative degradation of the composites generally happened by two actions. One is the mechanical denudation, which is referred to as the denudations of the surface materials of the composites under the high pressure and shearing forces of ablative gases during the ablation process, and other is chemical erosion which is related to oxidation and sublimation of the surface materials of the composites [15].

Table 5. Linear Ablative Rate of the filler filled ceramic/phenolic composites

Filler	Filler Weight Fraction (%)	Before Test	After Test	Linear ablation rate (mm/s)
		Thickness (mm)	Thickness (mm)	
No filler	0	6.03	4.87	0.00967
MS	5	6.13	5.09	0.00865
MS	10	6.38	5.43	0.00793
MS	15	6.59	5.75	0.00696
MS	20	6.71	6.00	0.00589
ZYS	5	6.21	5.20	0.00831
ZYS	10	6.44	5.53	0.00754
ZYS	15	6.63	5.87	0.00628
ZYS	20	6.79	6.16	0.00522
CS	5	6.31	5.34	0.00808
CS	10	6.53	5.67	0.00717
CS	15	6.75	6.02	0.00608
CS	20	6.88	6.30	0.00483

Linear ablative rates of the composites filled with various concentrations of three different fillers are numerically calculated using ablation time and the thickness of specimens measured before and after the exposure of flame and are reported in Table 5 and plotted in the graphical representation as shown in Fig 5. For unfilled ceramic/phenolic composites, recorded linear ablative rate is about 0.00967 mm/s. The addition of 5% filler concentration of microsilica, yttria-stabilized zirconia and cenosphere with the phenolic composites decreased the linear ablative rate of about 10%, 14% and 16% respectively when compared to the unfilled composites as shown in Fig 5. The significant reduction in the linear ablative rate is due to the presence of micro-sized fillers, which form a network of uniformly distributed filler layers in the filled composites. This filler layer act as a protective covering over the surface of the filled composites. Consequently, these protective layers protect the residual composites from the hot combusted gases, which directly reduce the internal ablation. Similarly, further addition of all the fillers considerably reduced the linear ablation rate of the filled composites, which is shown in Fig 5. Graphical representation of the linear ablative rate revealed that linear ablative rate decreased with the increase of filler concentration, in other words, both, linear ablation rate and filler concentration is indirectly proportional to each other. A similar phenomenon of reduction in ablation rate with an increase of filler concentration is observed with different filler such Organoclay [28], Silicone Rubber [29] and mesoporous silica particles [30].

From the graphical representation of the linear ablative rate, Fig 5 it is observed that the composites filled with cenosphere showed improved ablation behaviour in terms of the linear ablation rate than the composites filled with microsilica and yttria-stabilized zirconia. For higher filler concentration, cenosphere filled composites showed a maximum reduction of about 49% less than the linear ablative rate of the unfilled composites, yttria-stabilized zirconia filled composites showed a reduction of about 46%, whereas maximum reduction of about 39% is recorded for microsilica filled composites. From Fig 5, it is inferred that cenosphere filled composite showed a maximum reduction in linear ablation rate. It is due to the thermal conductivity of the fillers. Microsilica and yttria-stabilized zirconia have a thermal conductivity of about 0.28 W/mK and 0.21 W/mK respectively, whereas micro-sized cenosphere has a thermal conductivity of about 0.11 W/mK, which is lesser than the other two fillers. The addition of cenosphere forms an effective filler protection layer than the other filled composites, which directly improves the ablative behaviour of cenosphere filled composites.

Hence, in terms of the linear ablative rate, the addition of all the filler improves the ablation behaviour of the filled composites, however, for particular filler concentration, the effect fillers is in the order of, cenosphere filler showed maximum effect, yttria-stabilized zirconia showed moderate effect and microsilica least effect on linear ablative behaviour on filled composites. Like the linear ablation rate, the mass ablation rate is numerically calculated to clarify the effect of fillers on the ablation behaviour of the filled composites.

The mass ablative rate of various concentrations of filler loaded composites is reported in Table 6. It is observed from the graphical representation of the mass ablation rate, that the addition of all the three fillers decreased the mass ablation rate of the filled composites, which in turn enhanced the ablation behaviour. The mass ablation rate of the composites embedded with various concentrations of three different fillers is shown in Fig 6.

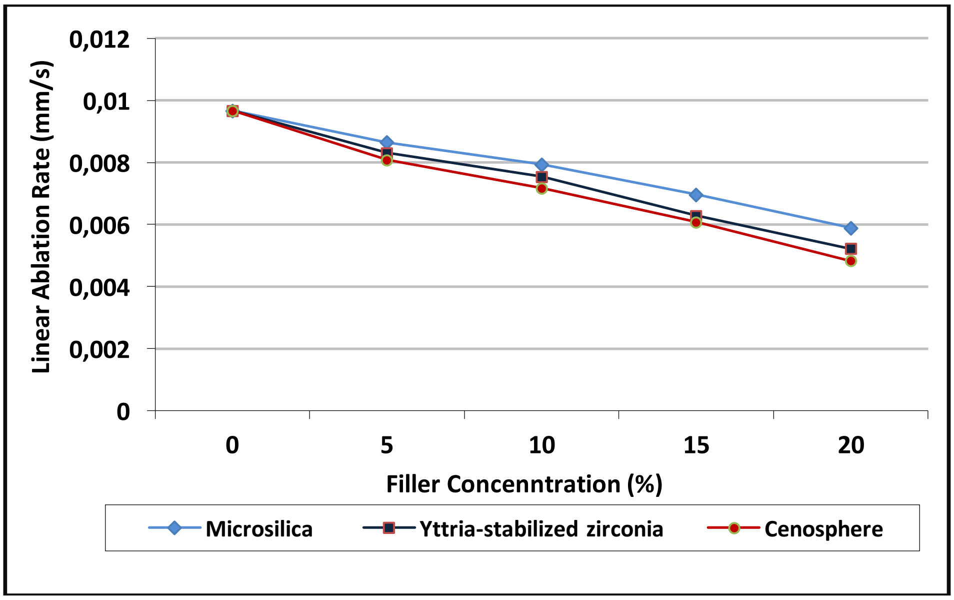

Fig 5. Effect of the fillers on Linear Ablative Rate of the composites

Table 6. Mass Ablative Rate of the filler filled ceramic/phenolic composites

Filler	Filler Weight Fraction (%)	Before Test	After Test	Mass ablation rate (g/s)
		Mass (g)	Mass (g)	
No filler	0	53.92	42.05	0.09892
MS	5	54.29	43.45	0.09017
MS	10	56.03	45.55	0.08733
MS	15	58.46	48.92	0.07925
MS	20	60.23	51.77	0.07052
ZYS	5	57.92	47.36	0.08804
ZYS	10	60.11	50.14	0.08309
ZYS	15	63.06	52.04	0.07518
ZYS	20	65.58	57.63	0.06627
CS	5	55.86	45.52	0.08617
CS	10	57.74	47.94	0.08167
CS	15	59.17	50.37	0.07333
CS	20	63.84	56.11	0.06442

From the graphical interpretation of the mass ablative rate, it is inferred that the filler concentration is indirectly proportional to the mass ablative rate of the filled composites, as shown in Fig 6. The significant reduction in the mass ablative rate is inferred by two actions. One is the uniform dispersion of the filler in phenolic composites, which forms a layer of fillers and other is the formation of this filler layer, acts as protecting layer, consequently, it insulates and protects the residual composites from the server hot gases during the ablation process.

In terms of the mass ablative rate, the cenosphere filled composites showed improved ablation behaviour when compared to yttria-stabilized zirconia and microsilica filled composites. Here also, like in case of linear ablation rate, the effect fillers on mass ablative behaviour on filled composites is in the order of, cenosphere filler showed maximum effect, yttria-stabilized zirconia showed the moderate effect and microsilica least effect for particular filler concentration. The variation in terms of mass ablative behaviour directly relied on the thermal conductivity of the filler loaded in the filled composites. On the whole, the mass ablative rate decreased as a function of filler concentration in the ceramic/phenolic composites.

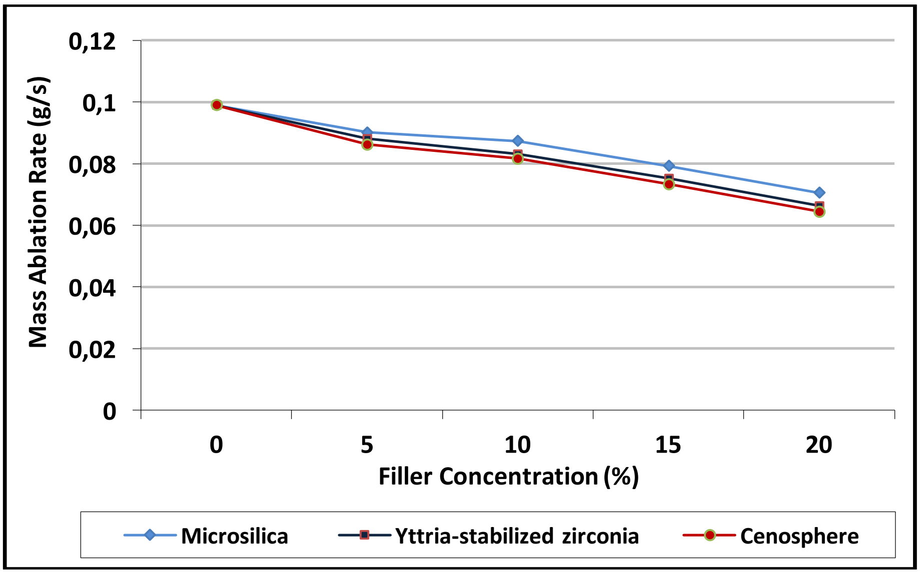

Fig 6. Effect of the fillers on Mass Ablative Rate of the composites

The thermal insulating effectiveness of the filled composites is determined from back face temperature measurements. The back face temperatures of the composite filled with various concentrations of three different fillers such cenosphere, yttria-stabilized zirconia and microsilica with respect to time are shown in Fig 7, Fig 8 and Fig 9 respectively.

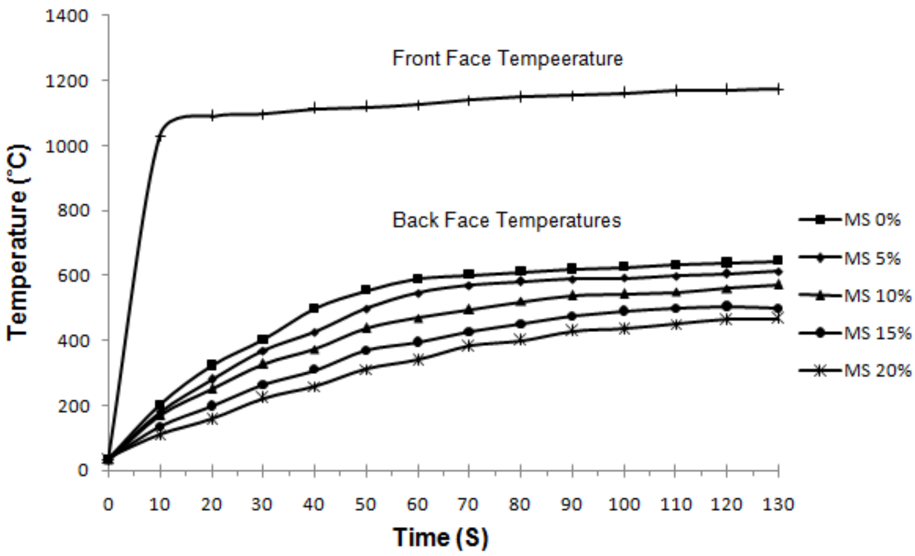

Fig 7. Back face temperature of various concentrations of Microsilica filled ceramic/phenolic composite

The graphical interpretation of temperature distribution showed that the addition of fillers considerably reduces the back face temperature of the filled composites, which in terms increases the insulation the filled composites. It indicated that the addition of fillers forms an insulating layer, which increases the insulating effect of the filled composites significantly under harsh testing environment.

In other words, these uniformly dispersed filler embedded in the filled composites acts as an effective thermal barrier, which considerably prevents the heat transfer deep into the composites and consequently it offers effective insulation to the virgin material beneath the ablated surface of the filled composites. For all the micro-sized filler, the maximum insulation achieved for maximum filler concentration, as shown in Fig 7, Fig 8 and Fig 9. The filler concentrations in the filled composites behave like a heat dissipater and it offers a better dissipating network for heat flow in random directions. Because of the nature of the filler particles, it dissipates the heat uniformly throughout the layer, which reduces the temperature concentration in the filled composites. Hence, the filler concentrations in the filled composites act as an additional heat barrier.

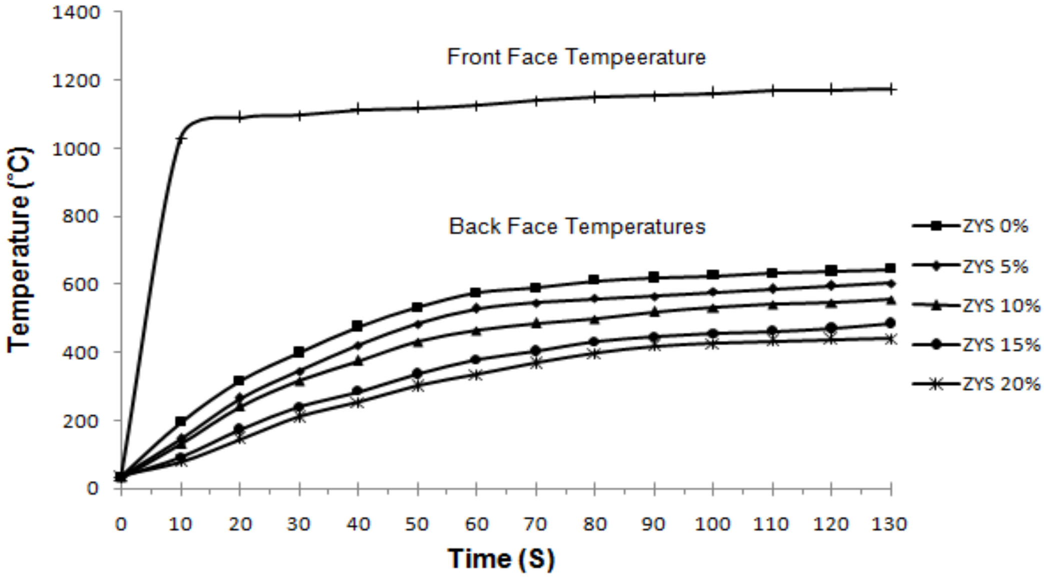

Fig 8. Back face temperature of various concentrations of Yttria-stabilized zirconia filled ceramic/phenolic composites

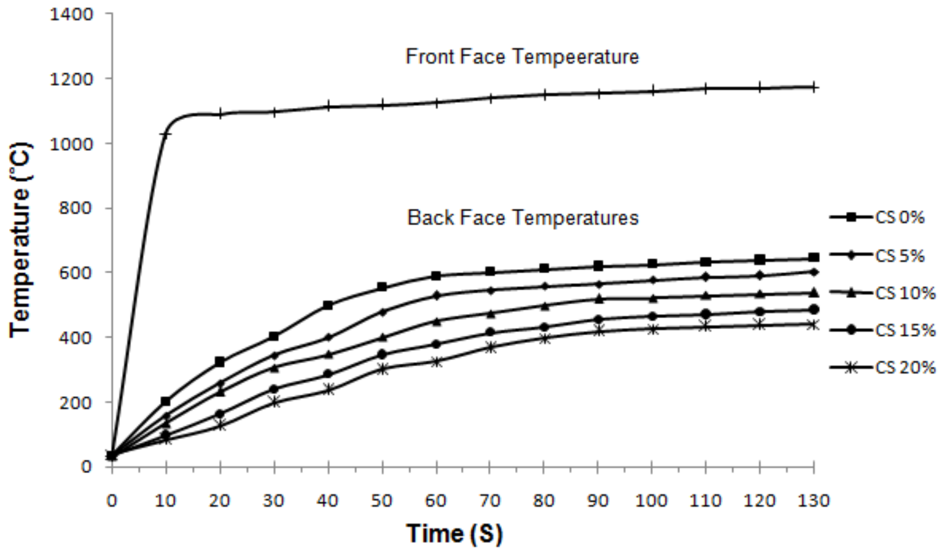

Fig 9. Back face temperature of various concentrations of Cenosphere filled ceramic/phenolic composite [16]

The post-ablation morphology of the composites filled microsilica, yttria-stabilized zirconia and cenosphere are shown in Fig 10. It is observed from the post-ablation figures, the refractory charred layer formed over the ablated surface of the filled composite, additionally protects the internal residual of the composites from the further degradation during intensive ablation as shown in SEM image (Fig 11). Improved thermal insulating effect of the filled composites is also evidenced by the lower depth penetration of the degradation zone (char zone) along the thickness, which is shown in cross-section image of the ablated surface (Fig 10). It can be observed from the cross-sectional image of the ablated surface, that when compared to neat composite, the effective thickness of the charred insulating zone (char depth) of the filled composites has significantly reduced with an increase of filler concentration.

The filler content dispersed in the phenolic resin act as a mechanical stabilizer, which holds the ablated char layer and protects the internal residual from hot shearing gases, as observed from the post ablated surface optical (Fig 10) and SEM (Fig 11) of the filled composites. Thus, filler particles dispersed in the ceramic/phenolic composites reduce the erosion process consequently enhanced the ablation behaviour of the filled composites. On the whole, the addition of three different fillers such as cenosphere, yttria-stabilized zirconia and microsilica, improved the ablation behaviour of the composites. Maximum improvement in the ablation behaviour in terms of linear ablative rate, mass ablative rate and back face temperature, is achieved for maximum filler concentration. However, cenosphere filled composites showed overall superior behaviour, and then yttria-stabilized zirconia and microsilica filled Ceramic/Phenolic composites.

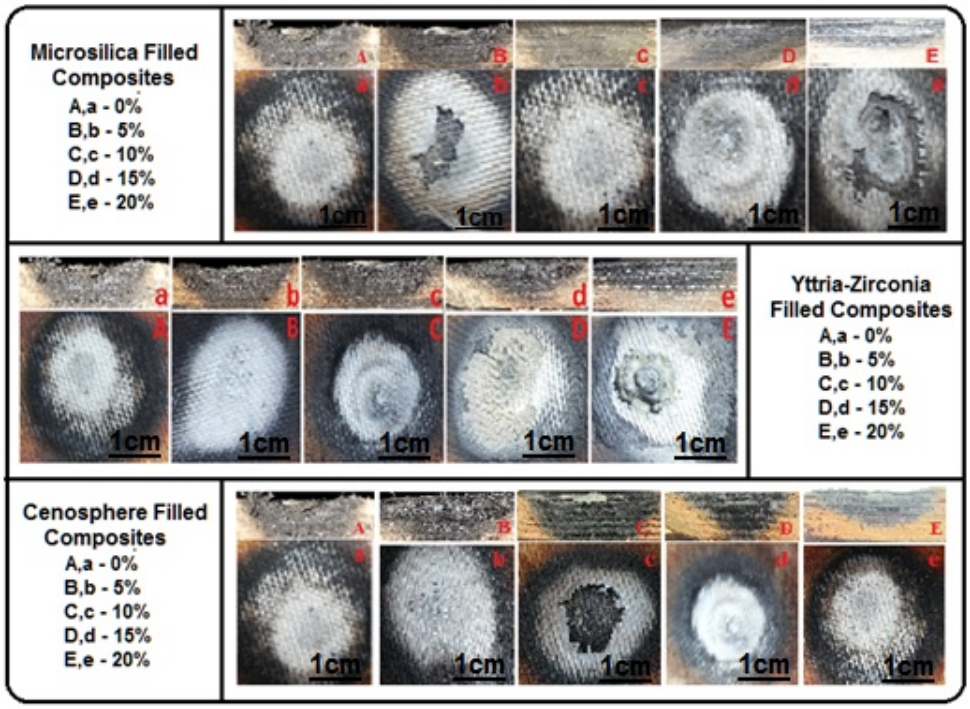

Fig 10. Post-ablation tested surface of three different fillers filled composites (a: Unfilled composites, b: 5% filled composite, c: 10% filled composite, d: 15% filled composite, e: 20% filled composite)

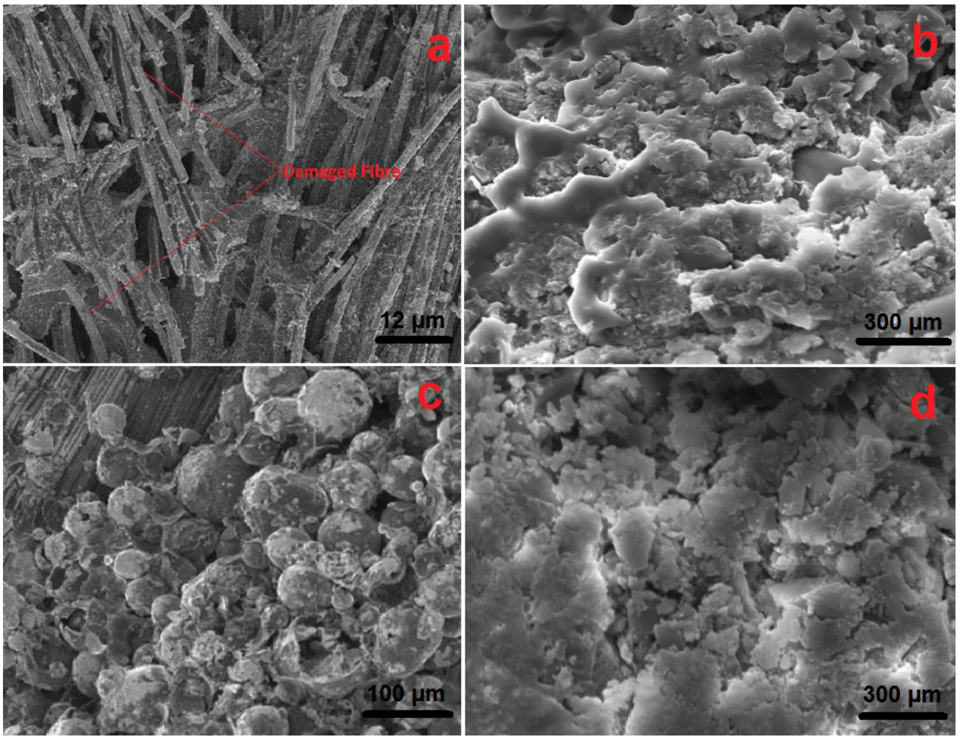

Fig 11. SEM image of ablated surface of the composites (a: Unfilled composites, b: 20% microsilica filled composite, c: 20% cenosphere filled composite, d: 20% yttria-zirconia filled composite)

5. Conclusion

In this study, mechanical characterizations were done to clarify the effect of fillers on the mechanical properties of the filled composites. The addition of three different fillers enhanced the tensile strength and compressive properties of the filled composite. However, beyond certain filler concentration, further addition of fillers reduced the mechanical strength of the filled composite. When compared to the neat composites, yttria-stabilized zirconia and microsilica filled composites showed an increase in the mechanical strength of the filled composite up to 15% of microsilica concentration. However, in case of cenosphere filled composites enhancement was noticed only up to 10% addition of cenosphere content. After exposure to the oxyacetylene flame, the deep differences in terms of an ablative behaviour as the function of the fillers concentration are clearly visible. The ablative rate of three different micro-sized fillers filled composites decreases with the increase in filler concentration. However, cenosphere filled composites showed better ablation behaviour than the other two fillers filled composites. The filler concentration embedded in the composites acted as a mechanical stabilizer and thermal barrier, consequently, hold and prevent the material denudation. Thus, these lightweight, inexpensive fibre reinforced composites compounded with micro-sized fillers can be used as potential high temperature materials for rocket motor nozzle and thermal protection system.

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