Research Article

Dynamic Mechanical Analysis and Three-Body Abrasive Wear Behaviour of Thermoplastic Copolyester Elastomer Composites

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Various amounts of short fibers (glass and carbon) and particulate fillers like polytetrafluoroethylene (PTFE), silicon carbide (SiC), and alumina (Al_2O_3) were systematically introduced into the thermoplastic copolyester elastomer (TCE) matrix for reinforcement purpose. The mechanical properties such as storage modulus, loss modulus, and Tan δ by dynamic mechanical analysis (DMA) and three-body abrasive wear performance on a dry sand rubber wheel abrasion tester have been investigated. For abrasive wear study, the experiments were planned according to L_{27} orthogonal array by considering three factors and three levels. The complex moduli for TCE hybrid composites were pushed to a higher level relative to the TCE filled PTFE composite. At lower temperatures (in the glassy region), the storage modulus increases with increase in wt.% of reinforcement (fiber + fillers) and the value is maximum for the composite with 40 wt.% reinforcement. The loss modulus and damping peaks were also found to be higher by the incorporation of SiC and Al_2O_3 microfillers. The routine abrasive wear test results indicated that TCE filled PTFE composite exhibited better abrasion resistance. Improvements in the abrasion resistance, however, have not been achieved by short-fiber and particulaute filler reinforcements. From the Taguchi's experimental findings, optimal combination of control factors were obtained for minimum wear volume and also predictive correlations were proposed. Further, the worn surface morphology of the samples was discussed.

1. Introduction

In recent times, there has been a remarkable growth in large scale production of fiber and/or filler-reinforced thermoplastic polymer matrix composites for engineering applications. Engineering polymers and polymer reinforced with fibers and particulate fillers are widely used in design. Polymer composites are often required to move in contact with hard abrasive particles as counterface. Some practical situations are sleeve bearings, marine stern bearings, pipe and chute liners used to transport abrasive materials, rotor and powder mixers, blades and components in agricultural and earth moving equipment, and impellers in pumps handling industrial fluids [1]. In general, it is the mechanical load carrying capacity and specific wear rate of the composites that determine their acceptability in practical applications. Different types and amounts of fiber reinforcement and/inorganic fillers are known to improve many physical and/or mechanical properties of polymers, and their potential in improving the abrasive wear resistance is worth discovering [2]. Abrasive wear is the most important among all the forms of wear because it contributes almost 64% of the total cost of the wear [3]. It is caused due to hard particles or hard protuberances that are forced against and move along a solid surface [4].

Several researchers have observed the significant changes in the mechanical properties like storage and loss moduli and Tan δ of polymers reinforced with fibers and particulate fillers [5–10]. Jin et al. [5] conducted the study on dynamic

mechanical analysis (DMA) of multiprocessed multiwalled carbon nanotube/polymethylmethacrylate (PMMA) composites to determine storage and loss moduli and Tan δ. Hassan et al. [6] investigated the dynamic mechanical property of injection moulded glass fiber/polyamide 66 (PA66) composite. They determined the Tan δ of the virgin and filled material under dry and 50% relative humidity (RH) and wet condition. Huang et al. [7] reported the modulus of polytetrafluoroethylene (PTFE) filled silica composites and concluded that increase in silica content in the composite reduces the modulus. However they have not reported the three-body abrasive wear behaviour in the study. Suresha et al. [8] investigated the effect of organomodified montmoril-lonite (oMMT) content in different wt.% filled with epoxy nanocomposites on glass transition temperature (T_g), storage and loss moduli and Tan δ along with three-body abrasive wear tests. They concluded that the T_g, storage, and loss moduli were decreased with increasing of oMMT filler loading. However, higher oMMT filler addition has shown minimum wear volume loss and improved mechanical properties. Thongsang et al. [9] studied the dynamic mechanical and tribological properties of fly ash silica (FASi)/precipitated silica (PSi) filled natural rubber (NR) composites by varying the total silica contents in hybrid NR composites. They concluded that a 75% PSi fraction to be introduced into FASi/PSi-filled NR compounds in order to achieve optimal mechanical properties and wear resistance. However the above investigations have not reported the relationship between the dynamic mechanical properties and wear resistance of the composites. Brostow et al. [10] demonstrated a link between dynamic mechanical properties and scratch/wear resistance on various thermoplastics like polycarbonate (PC), polypropylene (PP), polystyrene (PS), PTFE, styreneacrylonitrile (SAN), acrylonitrilebutadienestyrene (ABS), santoprene, surlyn, polyethersulfone (PES), and low density polyethylene (LDPE), when plotting Tan δ as a function of penetration depth or residual depth, a parabolic dependence is observed. However, a clear relationship with storage and loss moduli has not been found.

A number of studies on polymers and their composites subjected to abrasive wear have been reported [11–32] in literature. Budinski [11] investigated abrasion resistance of twenty one types of polymers and reported polyurethane having better abrasion resistance over other polymers. Briscoe et al. [12] reported abrasive wear of polyetheretherketone (PEEK) filled PTFE and PTFE filled PEEK. Sole and Ball [13] investigated the effect of fillers such as $CaCO_3$, $BaSO_4$, and fly ash on abrasive wear behaviour of PP. They reported that the addition of mineral fillers to the PP matrix decreases the wear resistance under severe abrasion conditions. Liu et al. [14] reported the abrasion resistance of ultrahigh molecular weight polyethylene (UHMWPE) with and without quartz particles as reinforcement under three-body abrasion conditions. Three-body abrasive wear of polymer based composites [15–21] has been extensively studied and considerable progress has been made in understanding the complex tribological process. Many researchers have reported that, the abrasive resistance of polymeric materials depends upon the intrinsic mechanical properties of the materials as well as on the test method used [17, 18].

It is well known that, by the addition of fillers or fibre reinforcement into most of the thermoplastic composites will improve the mechanical properties, however the same does not augment the wear resistance if the wear mechanisms are highly abrasive in nature [21]. However the findings of Bahadur et al. [22–27] revealed that the wear rate was considerably reduced by the addition of CuO and CuS to PTFE, CuS, CuF_2, CaO, and PbS to Nylon 11, and CuO, CuS, and CuF_2 to PEEK. Contrary to the above observations, they also found that wear rate increased when the polymers were filled with particulate materials such as BaF_2, CaF_2, ZnF_2, SnF_2, ZnS, SnS, ZnO, and SnO [22, 26]. Ravi Kumar et al. [28] conducted three-body abrasive wear behavior on PA66/PP blend and nanoclay (NC) + short carbon fiber (SCF) filled PA66/PP. They found that PA66/PP with NC + SCF exhibited highest wear rate than that of PA66/PP blend. Bijwe et al. [29] examined the neat polyetherimide (PEI), PEI filled with SGF and PTFE, molybdenum disulphide (MoS_2), and graphite in various wear modes. They reported that neat PEI exhibits better resistance under three-body abrasion. These findings have revealed that the fibers and fillers are detrimental for three-body abrasive wear behaviour and are in good agreement with the above listed findings [21, 22, 26].

Pettarin et al. [30] reported the wear behavior of high molecular weight high density polyethylene (HMWHDPE)/ MoS_2 composites under dry sliding and abrasive wear conditions. They concluded that, incorporation of MoS_2 to HMW-HDPE improves its sliding and abrasive wear performance with a content of MoS_2 for minimum wear rate around 10 wt.%. Tong et al. [31] found that the coefficient of friction of UHMWPE was increased with the addition of wallastonite fibers, while the wear resistance of the composites was the highest when the fiber content was about 10 wt.%. Voss and Friedrich [32] studied the sliding and abrasive wear behavior of short glass and carbon fiber reinforced PEEK composites at room temperature. They concluded that, the addition of SGF to PEEK showed marginal improvement in the abrasion resistance. The latter investigated reports [30–32] revealed that the incorporation of some type of fibers and fillers had improved the abrasion wear resistance of the polymer composites. These findings are in good agreement with the earlier investigation reports [22–27]. The above findings are in contrary, which had made the researchers to investigate the synergistic effect of the fibers and fillers on tribomechanical performances of the newly designed polymer based composites.

Design of experiment (DOE) is a technique to obtain the maximum amount of conclusive information from the minimum amount of work, time, energy, or other limited resources [33]. Taguchi's DOE approach has produced a unique and powerful quality improvement discipline that differs from the traditional process. It has been successfully applied for parametric appraisal in dry sliding wear study of polymer composites [34–37]. Ramesh and Suresha [37] investigated the optimization of tribological parameters in abrasive wear mode of carbon-epoxy (C-E) hybrid composites. They implemented Taguchi's DOE, Analysis of Variance (ANOVA) to determine the significance of control factors influencing wear and grey relational grade (GRG) to optimize the tribological

TABLE 1: Source and supplier details of materials used in the present investigation.

Polymer/filler	Designation	Source and supplier
Thermoplastic copolyester elastomer	TCE	DSM and M/s Gargi Enterprises, Bangalore.
Polytetrafluoroethylene	PTFE	Du Pont Co. Ltd.
Short glass fibre	SGF	Fine Organics, Mumbai
Short carbon fibre	SCF	Fine Organics, Mumbai
Silicon carbide	SiC	Carborundum Universal Ltd, Chennai, India.
Alumina	Al_2O_3	Triveni Chemicals, Gujarat, India.

TABLE 2: Mechanical properties of TCE + PTFE filled hybrid composites.

Sl. number	Matrix material (wt.%)	Fiber (wt.%)	Filler (wt.%)	Hardness (H) (Shore D)	Tensile strength (S) (MPa)	Tensile elongation (e) (%)
1	TCE (85)	—	PTFE (15)	60.8	30.58	18.9
2	TCE (68)	SGF (20)	PTFE (12)	72.8	57.84	16.36
3	TCE (60)	SGF (17.5) + SCF (2.5)	PTFE (10) + SiC (5) + Al_2O_3 (5)	69.7	30.16	9.87

parameters having multiple response. They concluded that filler loading and abrasive particle size have more significant effect on the specific wear rate of the composite and are therefore ranked first and second.

Efforts have been made by researchers to understand the dynamic mechanical characteristics and abrasive wear behaviour of the polymer based composites.

From the above discussion, it is obvious that the mechanical properties and tribological behaviour of thermoplastic blend system is significantly affected by fibers and fillers. Moreover, the abrasive wear performance of thermoplastics is very poor with the addition of fibers and or fillers. The mechanism that leads to the poor abrasion performance of thermoplastic composites has not been fully understood yet. Attempts to reinforce both fibers and fillers into thermoplastic copolyester elastomer (TCE) + polytetrafluoroethylene (PTFE) composite have not been reported so far. TCE as matrix and PTFE, SiC, and Al_2O_3 fillers are good choice since for bearing applications both high modulus and high strength are desirable. Hence, in the present research article, TCE + PTFE composite with short fibers and fillers have been taken up for investigation with the intention of characterizing them for their dynamic mechanical analysis and three-body abrasive wear mode.

2. Experimental Details

2.1. Materials. Thermoplastic copolyester elastomer (TCE), commercially available as Arnitel EM 740 in the form of granules is the matrix material in the present investigation. Short E-glass fiber (SGF) was selected as reinforcement. Polytetrafluoroethylene (PTFE), short carbon fiber (SCF), silicon carbide (SiC), and alumina (Al_2O_3) were selected as micronscale fillers. The average diameter of the SGFs was approximately 12 μm with an average fiber length of about 4 mm. The average diameter of SCFs was approximately 7 μm with an average fiber length of about 100 μm. The particle size of PTFE

was about 5 μm and around 5 to 10 μm for SiC and Al_2O_3. The sources and suppliers of these materials are listed in Table 1.

2.2. Preparation of TCE Composites. The polymer granules and fillers were dried at 75°C for 10 h in an oven before compounding. Selected compositions were mixed and extruded in Barbender corotating twin-screw extruder (make: CMEI, model: 16CME, SPL, and chamber size 70 cm^3). The mixing speed of 100 rpm was maintained for all the compositions. The extrudates from the die were quenched in cold water and then pelletized. In the melt blending, the temperature profile of the extrusion were zone 1 (200°C), zone 2 (210°C), zone 3 (220°C), zone 4 (240°C), and zone 5 (260°C), respectively. The extrudates of the compositions were pelletized using pelletizing machine. The pellets of the extrudates were predried at 100°C in vacuum oven for 24 h and injection moulded in a reciprocating screw injection moulding machine (Windsor, 50 T) to produce test specimens. The processing temperature at zone 1 (220°C) and zone 2 (250°C) in injection moulding machine barrel was maintained, respectively. The mould temperature was maintained at 35°C. The details of the composites fabricated for present investigation are given in Table 2.

2.3. Dynamic Mechanical Analysis. DMA is a technique, which is used to study the stress, temperature, and frequency of the material when it is subjected to the small deformation by sinusoidal load. It is used to measure the stiffness and damping in terms of moduli and Tan δ. It was carried out using TA instruments DMA Q 800 Dynamic Mechanical Analyzer with specimen dimension of 20 mm × 12 mm × 1.6 mm. The measurements were carried out in a three-point bending mode of the equipment and the corresponding viscoelastic properties were determined as a function of temperature. The temperature range was −60°C to 150°C at a heating rate of 5°C min^{-1} under nitrogen flow. The samples were scanned at a frequency of 1 Hz.
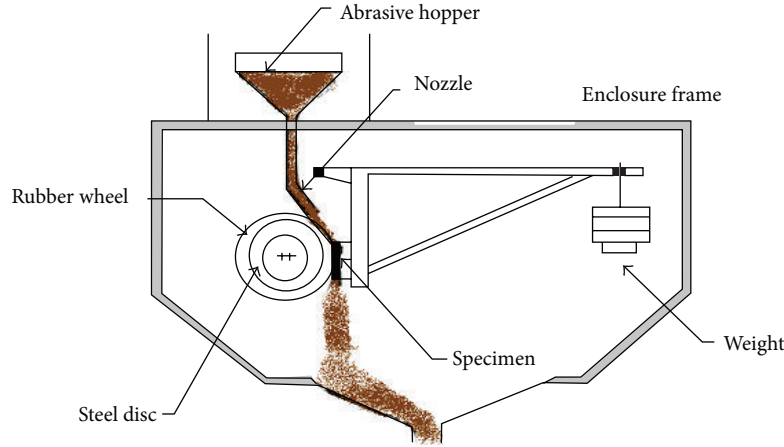
FIGURE 1: Schematic diagram of dry sand rubber wheel abrasion tester.

TABLE 3: Test parameters considered for three-body abrasive wear test.

Parameters	Test conditions
Load	40 N
Rotational speed of rubber wheel	200 rpm
Diameter of rubber wheel	228.6 mm
Abrasive particles	Silica sand, angular, 212, 425, and 600 μm
Size of the specimen	76 mm × 26 mm × 6 mm
Total test duration	5.25, 10.25 and 15.25 min
Abrading distance	752 m, 1468 m, 2185 m.
Sand flow rate	255 ± 5 g/m.

TABLE 4: Control factors and levels used in the three-body abrasive wear tests.

Control factors	Levels		
	1	2	3
PTFE + particulate filler content (composition) (A), in (%)	15	32	40
Abrasive sand particle size (B), in (μm)	212	425	600
Abrading distance (C), in (m)	752	1468	2185

Also, correlation of wear data with mechanical properties was carried out and the correlation coefficient can be calculated by the following equation:

$$r = \frac{n\left(\sum xy\right) - \left(\sum x\right)\left(\sum y\right)}{\sqrt{\left[n\left(\sum x^2\right) - \left(\sum x\right)^2\right]\left[n\left(\sum y^2\right) - \left(\sum y\right)^2\right]}}, \quad (2)$$

where r is the correlation coefficient, n is the number of observations, x is the independent variable (in this case, reciprocal of mechanical properties), and y is the dependent variable (in this case, it is wear volume of the composites).

2.5. Worn Surface Morphology. After wear test, the worn surface morphology of the composites was carried out using E-SEM, Quanta 200 model scanning electron microscope instrument with a voltage of 20 kV.

2.6. Design of Experiments. Designs of experiments were applied on three-body abrasive wear behaviour of polymer composites [34–39]. In this research article, three factors, that is, filler content/composition (A), abrasive particle size (B), and abrading distance (C) each at three levels, are considered in this study in accordance with L_{27} (3^{13}) orthogonal array. The control factors and their levels are listed in Table 4.

The experimental observations are further transformed into signal-to-noise (S/N) ratios. There are three categories of quality characteristic in the S/N ratio of the Taguchi method: the lower the better, the higher the better, and the nominal the better. These can be calculated as logarithmic transformation

2.4. Three-Body Abrasive Wear Tests. Three-body abrasive wear studies of TCE with and without SGF and their composites were studied on a dry sand/rubber wheel abrasion test rig as shown in Figure 1. The detailed test procedures were discussed elsewhere [8, 28]. After conducting a set of routine experiments, the final test conditions were implemented and noted in Table 3. The weight loss due to wear was noted down. Volume loss (Δv) due to wear and the specific wear rate (K_s) were calculated after the test using the following equations:

$$\Delta v = \frac{\Delta m}{\rho}\ \left(\text{mm}^3\right)$$

$$K_s = \frac{\Delta v}{F_N D}\ \left(\text{m}^3/\text{Nm}\right), \quad (1)$$

where Δv is the volume loss due to wear, Δm is the mass loss of the specimen, ρ is the experimental density of the sample, F_N is the applied load, and D is the abrading distance. Three replicates of wear tests were performed for each sample to minimize data scattering and the average of the three replicate test results is reported as the specific wear rate in this work.

of the loss function. Wear volume loss (Δv) as "lower is better" performance characteristic is selected and is determined by below given equation:

$$S/N = -10 \log \frac{1}{n} \left(\Sigma y^2 \right), \tag{3}$$

where "n" is the number of observations and "y" is the observed data.

A statistical ANOVA was performed to identify the control factors that are statistically significant. With the S/N ratio and ANOVA analysis, the optimal combination of three-body abrasive wear parameters is predicted to acceptable level of accuracy.

3. Results and Discussion

3.1. Dynamic Mechanical Analysis. The results of dynamic mechanical analysis were discussed in the following sections.

3.1.1. The Effect of Fiber and Filler Loading on Storage Modulus, Loss Modulus, and Tan δ. A dynamic mechanical analysis (DMA) was used to evaluate dynamic elastic (both storage and loss) moduli and Tan δ of the fiber and particulate-reinforced TCE + PTFE composite materials. The storage modulus, loss modulus, and Tan δ of TCE hybrid composites are plotted against temperature and demonstrated in Figure 2. These data provides insight into the stiffness of the composites, representing the elastic nature of the material [40]. Figure 2 shows the plots of storage modulus and loss modulus, against temperature in the range from −60 to 140°C for the various wt.% (15, 32 and 40) of TCE composites. It is shown from Figure 2 that dynamic elastic modulus values (both storage and loss) in the glassy region have increased with the increasing fiber and filler loading. However, the composite containing combination of fibers and fillers with 40 wt.% (17.5% GF + 2.5% CF + 5% SiC + 5% Al_2O_3) shows remarkably higher storage and loss moduli.

In general, there are three physical states of polymer materials: glassy region, glass transition region, and rubbery region. In the glassy region, the movement of chain segments is mostly "frozen" due to tight packing, so the material is prone to store more energy; this results in a high storage modulus [41]. The storage moduli of hybrid TCE + PTFE composites are high at −60°C and decreases as the temperature increases. This is because the components become more mobile and lose their close packing arrangement, leading to a very noticeable decrease of storage modulus in the glass transition region. As a result, in the rubbery region there is no significant change in storage modulus. This can be attributed to a highly entangled state of the polymer macromolecules [42]. The stiffening effect of SCFs and ceramic fillers like fine SiC and Al_2O_3 (<10 μm) particles on the TCE + PTFE composite is stronger than that of the SGFs for temperatures below −60°C, which can be ascribed to the shape of different fillers. Also, TCE reinforced with 40 wt.% fiber and fillers had higher values of storage modulus as compared to those with 32 wt.% fibers + fillers and 15 wt.% fillers. This is because of the reinforcement fibers and fillers, leading to a strong and
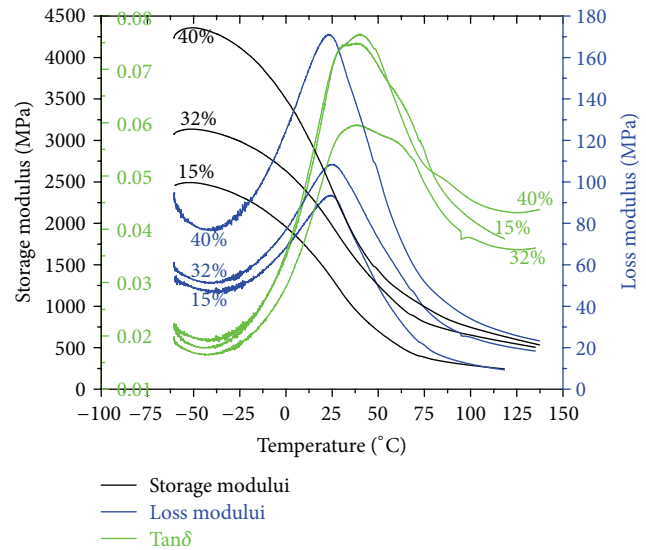

FIGURE 2: DMA thermogram depicting the storage modului, loss modului, and Tan δ of TCE + PTFE filled hybrid composites.

stiff interface between fiber/filler and polymer molecules [43]. In addition, TCE hybrid composites filled with micron sized filler particles possess higher storage modulus than those with large particles (fibers) and greater surface area occupied by the fillers, resulting in the effective matrix-filler interactions. The loss modulus peaks of TCE hybrid composites filled with 40 wt.% fiber + filler content become broader than those with 32 wt.% fiber + filler and 15 wt.% filler content, because of hindrance of the molecular motion.

The ratio of loss modulus to storage modulus is referred to as internal damping or the Tan δ. Generally, the major contributing causes of composite damping are (a) the nature of the matrix and filler; (b) the nature of the interphase; (c) frictional damping due to slippage in the unbound regions between the filler and the matrix interface, or delamination, and (d) energy dissipation in the area of matrix cracks. The damping peak usually occurs in the glass transition region and is associated with the movement of side groups or low molecular-weight units and molecular chains within the rubbers. Therefore, the higher the damping peak, the greater the degree of molecular mobility [44]. It can be seen from Figure 2, that the incorporation of 32 wt.% fiber + filler loading into the TCE composite decreased the damping characteristics of the composites, causing a lower and broader damping peak. This could be explained because the fiber/filler acted as a barrier to the mobility of polymer chains, which restricted the segmental motions of polymer through the strong interaction between fiber/filler and polymer molecular chains, indicating the reinforcing nature of filler with polymers [43]. Moreover, there was a reduction of the damping peak for 15 wt.% filler in TCE composites and hence a greater improvement in the fiber reinforcements. The effect of the fiber + filler wt.% in TCE composites on DMA results is summarized in Table 5.

TABLE 5: Dynamic mechanical properties of TCE + PTFE filled hybrid composites.

TCE + (fiber + filler wt.%)	Storage modulus (MPa)	Loss modulus (MPa)	tan δ	Glass transition (T_g), °C
85 + (15)	2505.41	93.72	0.0750	24.65
68 + (12 + 20)	3151.16	109.17	0.0600	24.65
60 + (20 + 20)	4381.81	171.26	0.0768	23.56

3.2. The Effect of Fiber and Filler Loading on Wear Volume and Specific Wear Rate. The wear volume of TCE + PTFE and its composites filled with SGFs and particulates (carbon, SiC, and Al$_2$O$_3$) abraded with three different silica sand abrasive particle size of 212, 425, and 600 μm, to a distance of 752, 1468, and 2185 m and for a fixed load of 40 N is listed in Table 6. It can be seen that the wear volume increases with increase in abrading distance, size of abrasives, and increasing in fiber and particulates loading. It is also evident from Table 6 that PTFE particles as reinforcement into TCE enhanced the abrasion resistance. However, the incorporation of SGFs increased the wear volume of TCE + PTFE composite. Furthermore, the reinforced effect of hard particles, namely, SiC and Al$_2$O$_3$ are less effective in SGFs reinforced TCE + PTFE composite. In the case of TCE + PTFE composite with 20 wt.% SGFs, the wear volume was approximately increased by 173% than that for TCE + PTFE composite. It should be noted that the increase in wear volume was greater when SGFs, carbon, SiC, and Al$_2$O$_3$ were used in combination. The wear volume for TCE + PTFE was 130.8759 mm^3 under a load of 40 N, while that of hybrid TCE composite was 396.8096 mm^3. The TCE + PTFE composite show an increase in wear resistance by 170 and 225% with comparison to that of TCE + PTFE + SGF reinforced and hybrid TCE composites, respectively. Meanwhile, adding micron sized ceramic particles (10 wt.%) into SGFs reinforced TCE + PTFE drastically increased the wear volume by 303%. Obviously, there is an adverse effect of ceramic fillers on the enhancement of abrasion resistance of TCE filled PTFE. This could be due to the abrasive nature of glass fibers and hard particles into the soft segment, that is, TCE filled PTFE composite. Better adhesion was found between TCE and PTFE, as it exhibited higher wear resistance to abrasive wear. Addition of SGF, carbon fiber, SiC, and Al$_2$O$_3$ increased the concentration of fiber and filler contents in the TCE + PTFE composites, which is detrimental to abrasive wear behaviour. In addition, it can be seen that the wear volume of TCE + PTFE and its composites increased with increase in particle size of the silica sand used as abrasives.

The specific wear rate decreases with increasing abrading distance and increases with the particle size of the abrasive. As seen in Figures 3(a)–3(c), when the silica sand particles are of coarse grade (600 μm), the specific wear rate starts to decrease drastically with increase in abrading distance. Higher specific wear rate was noticed for TCE hybrid composite compared to TCE + PTFE composite.

TCE + PTFE + SGF and TCE hybrid composites showed excessive wear after 1468 m abrading distance, respectively, in the experiments and the wear debris corresponded was in the form of large slices (Figure 4) under a load of 40 N with 212 μm silica sand abrasives. It was found that glass fiber reinforcement could effectively reduce the wear volume of TCE + PTFE composite with comparison to that of ceramic filler filled TCE hybrid composite. By comparison, SGF + TCE + PTFE had slightly better wear resistance than TCE hybrid composite.

For abrasion of polymeric composites, many attempts have been made to understand the tribo behaviour of various materials at several operating parameters, and the efforts continue. Three-body abrasion of polyester composites reinforced with randomly distributed short glass fibres has been studied by Chand et al. [39, 45], and it was found that increasing the size of the abrasive particles and the applied load worsened the wear property of the composite. Meanwhile, increasing the sliding velocities reduced the wear rate, and increasing the volume fraction of the glass fibres, according to the high energy theory, improved the wear properties, whereby a higher energy was required to facilitate the failure in the fibrous regions. The wear mechanism was predominated by matrix failure, pitting, cracks, and grooves. Harsha et al. [46, 47] studied the effect of fibres, solid lubricants, and applied load on the three-body abrasive wear characteristics of polyaryletherketone (PAEK) composites and showed that the ketone/ether ratio highly influenced the wear resistance of the composites especially at a higher load (12 N). Surprisingly, the addition of fillers and/or fibres to the PAEK worsened its three-body abrasive wear characteristics. Moreover, carbon fibres provided an inferior support for the PAEK composites as compared to glass fibres. Filler addition to the two phase composite showed worsening of the abrasion resistance. The results of the present work are also consistent with the literature findings [46, 47].

3.3. Correlation of Wear Data with Mechanical Properties. The hardness and tensile properties of TCE composites are shown in Table 2. The primary reasons for adding fillers or reinforcing fibers to polymers is to improve their mechanical properties, but the effects on wear rate are not invariably beneficial. According to Lancaster [48], the product of "S" and "e" (where "S" is the ultimate tensile strength and "e" the ultimate elongation) is a very important factor which controls the abrasive wear behaviour of composites. Reinforcement with the fibers/fillers increases the tensile strength ("S") of neat polymer, they usually decrease ultimate elongation to break ("e") and hence product "Se" may become smaller than that of neat polymer. Hence, reinforcement usually leads to deterioration in the abrasive wear resistance. Various theories have been proposed to interpret the abrasive wear performance of the composites. According to Ratner et al. [49] the wear rate of material is inversely proportional to the product of "Se". In the present investigation, the relationship between wear volume versus $(e)^{-1}$, $(He)^{-1}$ (where H is the hardness of

TABLE 6: Correlation coefficient of wear volume as a function of e^{-1}, He^{-1}, and Se^{-1} of TCE + PTFE hybrid composites.

Sample code	e^{-1}	Wear volume (mm³) Abrasive size (μm)									Correlation coefficient (r)
		212 μm Abrading distance (m)			425 μm Abrading distance (m)			600 μm Abrading distance (m)			
		752	1458	2185	752	1458	2185	752	1458	2185	
TCE + PTFE	0.05291	46.78832	114.8905	140.073	49.8540	87.88321	122.488	55.82044	77.73723	130.8759	
TCE + PTFE + SGF	0.06112	94.98305	136.0678	196.5424	95.9322	166.2373	239.1864	103.0508	164.678	226.7119	0.700026
TCE hybrid composite	0.10194	111.0429	189.816	261.1043	184.6626	304.2331	374.2945	175.3374	296.2577	396.8098	
	He^{-1}										
TCE + PTFE	0.00087	46.78832	114.8905	140.073	49.8540	87.88321	122.488	55.82044	77.73723	130.8759	
TCE + PTFE + SGF	0.00084	94.98305	136.0678	196.5424	95.9322	166.2373	239.1864	103.0508	164.678	226.7119	0.648937
TCE hybrid composite	0.00145	111.0429	189.816	261.1043	184.6626	304.2331	374.2945	175.3374	296.2577	396.8098	
	Se^{-1}										
TCE + PTFE	0.00173	46.78832	114.8905	140.073	49.8540	87.88321	122.488	55.82044	77.73723	130.8759	
TCE + PTFE + SGF	0.00106	94.98305	136.0678	196.5424	95.9322	166.2373	239.1864	103.0508	164.678	226.7119	0.550661
TCE hybrid composite	0.00336	111.0429	189.816	261.1043	184.6626	304.2331	374.2945	175.3374	296.2577	396.8098	

(a)

(b)

(c)
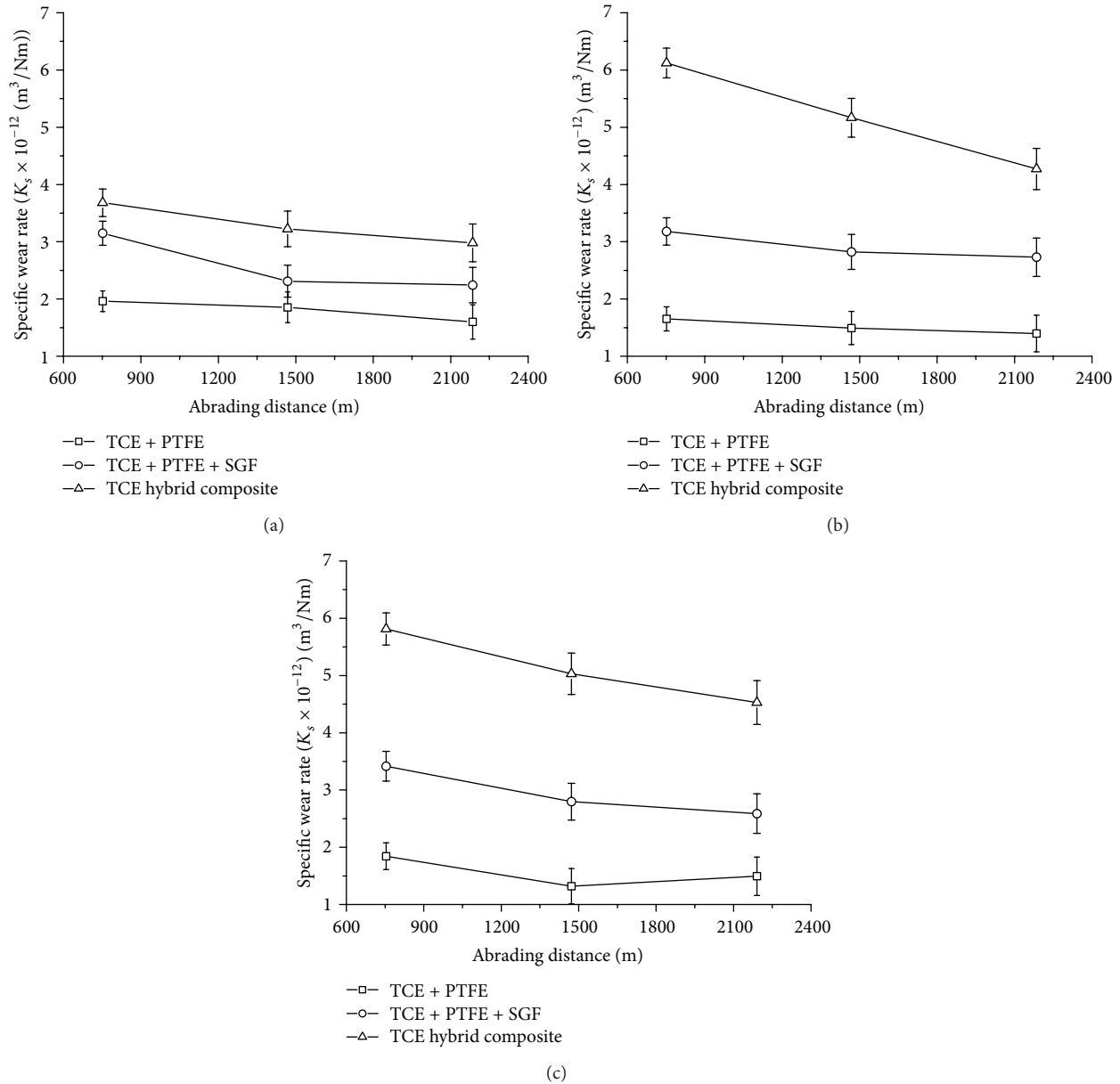
FIGURE 3: (a) Effect of abrading distance on K_s of TCE + PTFE filled hybrid composites at 40 N (212 μm silica sand). (b) Effect of abrading distance on K_s of TCE + PTFE filled hybrid composites at 40 N (425 μm silica sand). (c) Effect of abrading distance on K_s of TCE + PTFE filled hybrid composites at 40 N (600 μm silica sand).

the composite material), and $(Se)^{-1}$ with different abrading distance and varying abrasive particle sizes was studied. Composite with 40 wt.% fiber + filler in TCE matrix exhibits lower "S" and "e" values but moderate "H" value showed higher specific wear rate and wear volume with least resistance to three-body abrasion wear. Composite with 32 wt.% fiber + filler in TCE matrix possesses higher "S" and "H" values with moderate "e" value, exhibited moderate specific wear rate, wear volume, and resistance to three-body abrasion wear. Composite TCE + PTFE with lower "S" and "H" values but higher "e" value showed lower specific wear rate, wear volume, and excellent resistance to three-body abrasion wear in the study group. The decrease in elongation in TCE + PTFE

reinforced with SGF composite and TCE + PTFE hybrid composites is due to the fiber reinforcement and inclusions of filler particulates which results in deterioration of abrasive wear performance. Hence, the fiber/fillers were detrimental to the abrasive wear resistance of ductile polymers, which reduces toughness. Table 6, shows the correlation coefficient values of wear volume as a function of e^{-1}, He^{-1}, and Se^{-1} of TCE composites with different abrading distance and abrasive particles size. The term reciprocal of elongation $(e)^{-1}$ ranked first in correlation with correlation coefficient value of 0.700026, followed by $(He)^{-1}$ and $(Se)^{-1}$ with correlation coefficient values of 0.648937 and 0.550661, respectively, for the composites studied. With this, it can be concluded
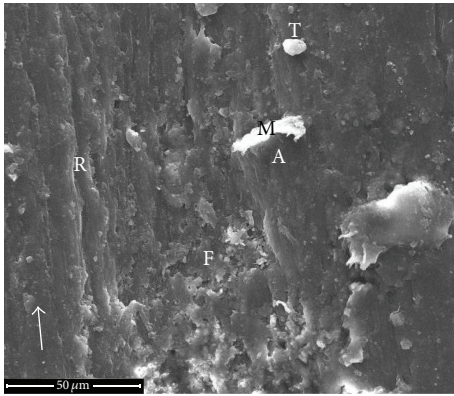
FIGURE 4: Scanning electron micrograph of TCE + PTFE composite abraded with sand particle size of 212 μm, to a distance of 2185 m and at load of 40 N.

FIGURE 5: Scanning electron micrograph of TCE + PTFE with SGFs composite abraded with sand particle size of 212 μm, to a distance of 2185 m and at a load of 40 N.

that ultimate elongation to break (e) influences the three-body abrasive wear behaviour of the composite compared to ultimate tensile strength (S) and hardness of the composite (H) in the present investigation.

3.4. Worn Surface Morphology. Abrasive wear occurs generally by three different mechanisms, that is, microploughing, microcutting, and microcracking [45]. Worn surfaces of materials were examined to find out predominant wear mechanisms. SEM photomicrograph of damaged surface for TCE + PTFE composite is shown in Figure 4. The deep grooves in the abrading direction (indicated with an arrow) are formed due to the microcutting action by sharp abrasive particles which are seen on the surface. Displacement of material had occurred by action of rolling and sliding of abrasive particle leading to the formation of furrows (F) and forming ridges (R) in transverse direction, similar to ploughing process. Material is deformed under load through ploughing and cutting with subsequent application of load. The deformed material (M) can be seen which is adhering to abraded surface (A) resulting in the ductile nature of the matrix material. PTFE particulates (T) can be seen on the surface. The worn surface provides evidence of rolling action of abrasive particles which is dominant in the abrasion process compared to sliding action. The deep furrows and wear damage are due to the rolling action of abrasive particles. Formation of ridges and shallow grooves are least, and this may be due to the sliding action of abrasive particles. Figure 5 shows SEM micrograph of TCE + PTFE reinforced with SGF composite. It was noticed that SGF are completely embedded (E) in the TCE matrix. This is due to TCE, as a low melting polymer, and composites are processed by injection moulding. Hence, skin of TCE matrix is formed on the SGF fibers. The material damage can be observed which is due to the formation and propagation of cracks (C) at the surfaces. The wear debris (D) attached to surface in the form of thin layers indicates the grooves formation, which is due to the ductile nature of the matrix. Fibers pull out or fiber damage is absent and indicates good adhesion between fiber and matrix material. The cracks are visible in the abrading
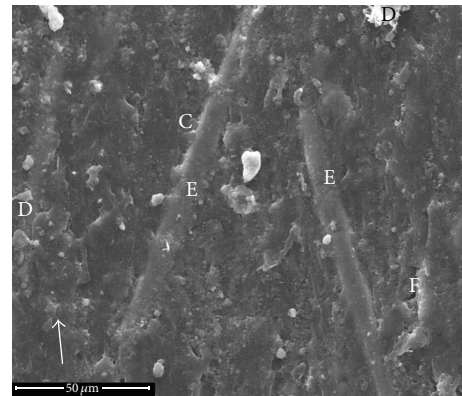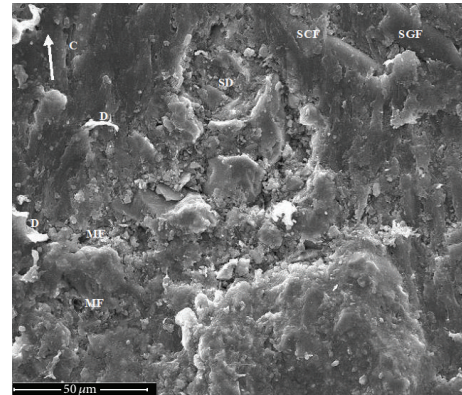

FIGURE 6: Scanning electron micrograph of TCE + PTFE + SGF filled with SCF, SiC and Al$_2$O$_3$ hybrid composite abraded with sand particle size of 212 μm, to a distance of 2185 m and at a load of 40 N.

direction (indicated with an arrow) which is due to the microcutting by the abrasive particles. The worn surface also indicates the ploughing action by the abrasive particles in the wear direction. Figure 6 shows the damaged surface of TCE + PTFE, SGF, SCF, Al$_2$O$_3$, and SiC filled hybrid composite. Severe damage (SD) can be seen in the micrograph which resulted in higher wear volume loss. Fiber damage and removal can also be seen which has resulted in the higher specific wear rate of the composite in the study group. Cracks, furrows, and ridges can be seen which result due to ploughing and cutting action of abrasive particles. Microfillers (MF) are loosely debonded to the matrix material which resulted in the formation of pits. The sharp edges of microfillers turned out to be abrasive particles and hence increase the wear volume. Wear debris in the form of thin film adhering to the surfaces indicates the toughness of the matrix material.

3.5. Statistical Analysis of Wear. The analysis was made using the software MINITAB 16, specifically used for the DOE applications. Table 7 presents the plan of experiments with output results using L_{27} orthogonal array. Abrasive wear

TABLE 7: Experimental design using L_{27} orthogonal array.

Exp. Number	Filler content (wt.%) (PTFE + particulate)	Sand size (μm)	Distance (m)	Weight loss (g)	Wear volume (mm³)	Specific wear rate ×10⁻¹² (m³ N⁻¹ m⁻¹)	S/N ratios (dB)
1	15	212	752	0.0641	46.78832	1.555463	−33.4027
2	15	212	1468	0.1574	114.8905	1.956582	−41.2057
3	15	212	2185	0.1919	140.073	1.602666	−42.9271
4	15	425	752	0.0683	49.85401	1.657381	−33.9540
5	15	425	1468	0.1204	87.88321	1.496649	−38.8781
6	15	425	2185	0.1678	122.4818	1.401393	−41.7614
7	15	600	752	0.0762	55.62044	1.849084	−34.9047
8	15	600	1468	0.1065	77.73723	1.323863	−37.8126
9	15	600	2185	0.1793	130.8759	1.497436	−42.3372
10	32	212	752	0.1401	94.98305	3.157681	−39.5529
11	32	212	1468	0.2007	136.0678	2.317231	−42.6751
12	32	212	2185	0.2899	196.5424	2.248769	−45.8691
13	32	425	752	0.1415	95.9322	3.189235	−39.6393
14	32	425	1468	0.2452	166.2373	2.831016	−44.4146
15	32	425	2185	0.3528	239.1864	2.736687	−47.5747
16	32	600	752	0.152	103.0508	3.425893	−40.2610
17	32	600	1468	0.2429	164.678	2.804461	−44.3327
18	32	600	2185	0.3344	226.7119	2.593957	−47.1095
19	40	212	752	0.181	111.0429	3.691587	−40.9098
20	40	212	1468	0.3094	189.816	3.23256	−45.5667
21	40	212	2185	0.4256	261.1043	2.987463	−48.3363
22	40	425	752	0.301	184.6626	6.139048	−45.3276
23	40	425	1468	0.4959	304.2331	5.181082	−49.6641
24	40	425	2185	0.6101	374.2945	4.282546	−51.4643
25	40	600	752	0.2858	175.3374	5.829037	−44.8775
26	40	600	1468	0.4829	296.2577	5.04526	−49.4334
27	40	600	2185	0.6468	396.8098	4.540158	−51.9716

responses and calculated signal to noise (S/N) ratio are shown in Table 7. The overall mean for the S/N ratio of the wear volume was found to be −43.1912 dB. Figures 7 and 8 graphically denote the effect of the control factors on the wear volume. Process parameter settings with the highest S/N ratio always gave the optimum quality with minimum variance. The graphs show the change of the S/N ratio when the setting of the control factor was changed from one level to the other. The best wear volume was at the higher S/N ratio values in the response graphs. From the graph, it is clear that control factor combination of A_1, B_1, and C_1 gives minimum wear volume. Thus minimum wear volume for the developed composite materials is obtained when the filler content (A), abrasive particle size (B), and abrading distance (C) are at the lowest level. This may be due to the factor that the PTFE content in the composite as fillers tends the abrasive particles to slip during abrasion process, resulting in low specific wear rate of the composite.

The S/N ratio response is presented in Table 8. The S/N ratio delta values of A, B, and C are 8.93, 1.40, and 7.39, respectively. The strongest influence on the wear volume loss

TABLE 8: Response data for S/N ratios under three-body abrasion.

Level	Composition (A)	Sand size (B)	Distance (C)
1	−38.58	−42.27	−39.20
2	−43.49	−43.63	−43.78
3	−47.51	−43.67	−46.59
Delta	8.93	1.40	7.39
Rank	1	3	2

was exerted by factor A, followed by factors C, and B, respectively. The interaction effects of control factors plot for S/N ratio is shown in Figure 8. Interactions do not occur when the lines on the interaction plots are parallel and strong interactions occur between control factors when the lines cross. Observation of Figure 8 yields an interaction between filler content versus sand particle size and abrading distance versus sand particle size. In order to justify the interactions, statistical analysis (ANOVA) was carried out.
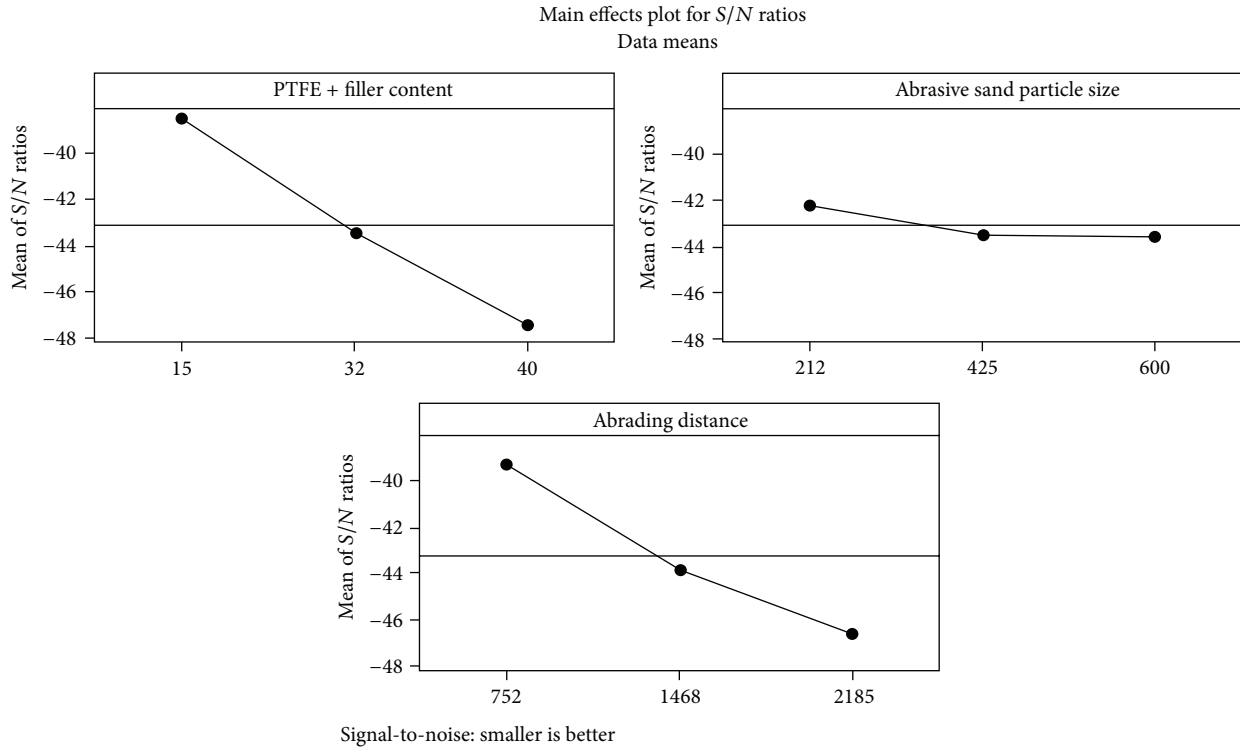
Main effects plot for S/N ratios
Data means

FIGURE 7: Main effects plot for S/N ratio of TCE composites.

TABLE 9: ANOVA data of TCE + PTFE filled hybrid composites under three-body abrasion.

Source	DF	Seq SS	Adj SS	Adj MS	F	P value	P (%)
Composition (A)	2	120998	120998	60499	262.20	0.000	52.46
Abrasive sand size (B)	2	8294	8294	4147	17.97	0.001	3.59
Abrading distance (C)	2	76247	76247	38123	165.23	0.000	33.05
Composition × sand size ($A \times B$)	4	13681	13681	3420	14.82	0.001	5.93
Sand size × distance ($B \times C$)	4	642	642	161	0.70	0.616	0.27
Composition × distance ($A \times C$)	4	8934	8934	2234	9.68	0.004	3.90
Error	8	1846	1846	231			0.80
Total	26	230642					100

S = 15.1899, R-Sq = 99.20%, and R-Sq = 97.40%.
DF: degree of freedom, Seq SS: sequential sum of squares, Adj SS: adjusted sum of squares, Adj MS: adjusted mean squares, F: variance, P value: test statistics, and P (%): percentage of contribution.

3.5.1. ANOVA and the Effects of Control Factors. ANOVA is a statistical design method used to break up the individual effects from all control factors. The percentage contribution of each factor is employed to measure the corresponding effect on the quality characteristics. The ANOVA with wear volume results are listed in Table 9. This analysis was undertaken for a level of significance of 5%, that is, for level of confidence 95%. The last column of the table indicates the order of significance among control factors and interactions. It could be observed from Table 9 that the control factor A (P = 0.0000) has greater static influence of 52.46%, C (P = 0.0000) has an influence of 33.05%, and B (P = 0.001) has an influence of 3.59% on wear volume of the material system under study. However, the interaction between A versus B (P = 0.001), B versus C (P = 0.616), and A versus C (P = 0.004) has

an influence of 5.93%, 0.27%, and 3.90%, respectively, which show less significance of contribution on wear volume.

The present analysis shows that three-body abrasive test parameters and their interactions have both statistical and physical significance (percentage contribution is greater than error) in the wear behaviour of TCE composites. But interaction between B versus C has statistical significance but do not have physical significance [50], since error associated is more than percentage contribution of these interactions, which is evident from the ANOVA results.

3.5.2. Confirmation Experiment. The confirmation experiment is the final step in the DOE process. The purpose of the confirmation experiment is to validate the conclusions drawn during the analysis phase [51, 52]. The estimated S/N ratio
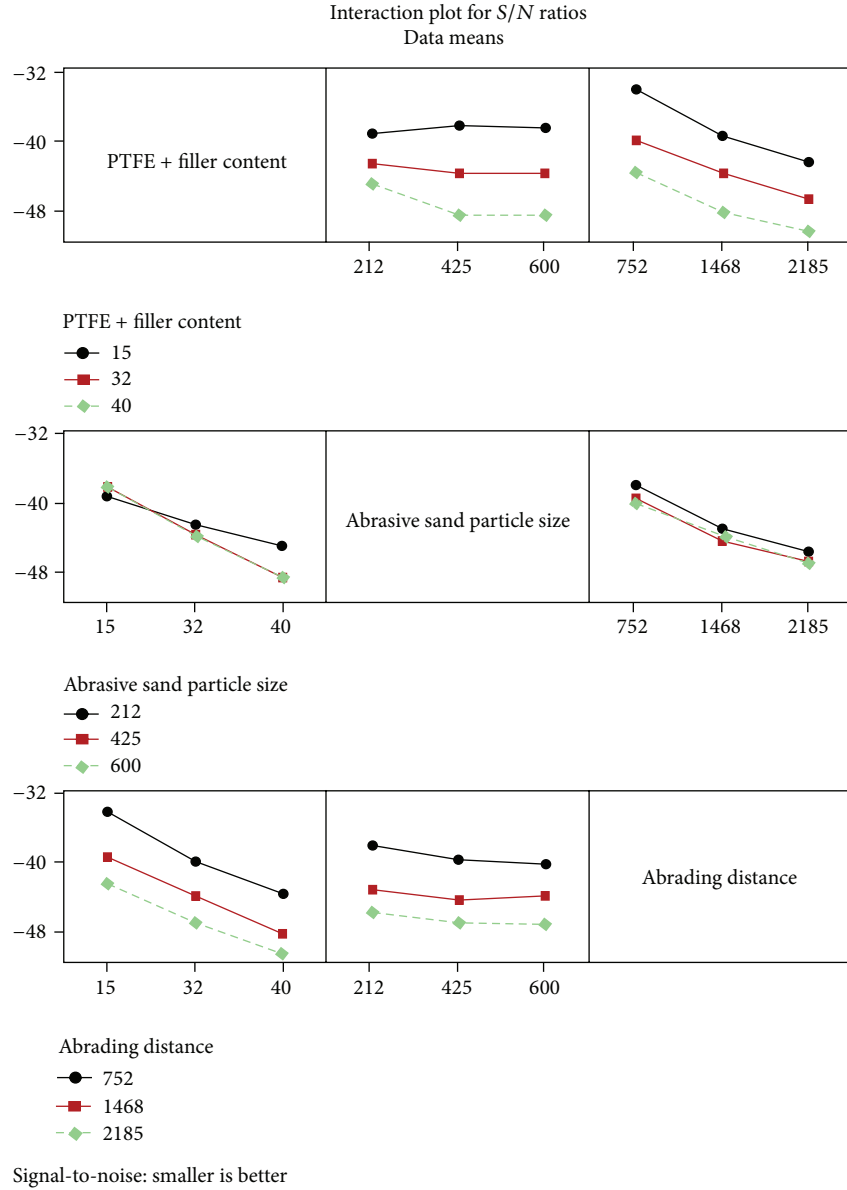
Figure 8: Interaction plot for S/N ratio of factors of TCE composites.

for specific wear rate using the optimum level of parameters can be calculated with the help of the following predictive equation [52, 53]:

$$\overline{\eta}_{\mathrm{opt}} = \overline{T} + \sum_{j=1}^{k} \left(\eta_j - \overline{T} \right); \quad j = 1, 2, \ldots, k, \qquad (4)$$

where $\overline{T}$ = overall experimental average of S/N ratio, η_j = mean of the S/N ratio at the optimum parameter level, and k = number of main design parameters that significantly affect the wear rate of TCE composites.

The results of experimental confirmation using optimal wear parameters and comparison of the predicted specific wear rate with the actual specific wear rate using the optimal specific wear parameters are shown in Table 10. The improvement in S/N ratio from the starting level to optimum level is 0.5513 dB. The wear volume is reduced by 6.15%. Therefore the wear volume performance is improved by using Taguchi method.

4. Conclusions

The three-body abrasive wear behavior of the TCE composites was studied as a function of particulate filler content, sand size, and abrading distance. The results were obtained as per Taguchi's L_{27} orthogonal array and the following conclusion can be drawn from the present study.

Table 10: Confirmation test for wear volume.

Level	Initial process parameter A_1 B_2 C_1	Optimal process parameter		Improvement in the result
		Prediction A_1 B_1 C_1	Experimental A_1 B_1 C_1	
S/N ratio (dB)	−33.954	−34.3611	−33.4027	0.5513 dB
Wear volume mm^3	49.85401	66.6763	46.78832	6.15%

(1) The storage and loss moduli of TCE + PTFE composite increased with the addition of fiber reinforcement and inclusion of ceramic fillers to short fiber reinforced TCE + PTFE (hybrid) composite. However, TCE + PTFE hybrid composite showed lower glass transition temperature.

(2) The TCE + PTFE composite showed better abrasion resistance as well as lower specific wear rate as compared to TCE + PTFE reinforced with SGF and ceramic fillers filled hybrid composites.

(3) Specific wear rate is more in TCE hybrid composite than that of TCE + PTFE composite. The carbon fiber, ceramic fillers, namely, SiC and Al_2O_3 were found to be detrimental to the abrasive wear performance. Glass fiber reinforcement to TCE + PTFE provides better abrasion resistance as compared to hybrid composites.

(4) The results indicated that the factor (A_1) that is composition exerted the greatest effect on the wear, followed by the abrading distance (C_1) and abrasive particle size (B_1). The effect of combined factors on composition and sand size ($A \times B$) on wear performance played a vital role.

(5) Design of experimental approach by Taguchi method enables to analyze the wear behavior successfully which has been influenced by number of parameter like filler content, sand size, and abrading distance.

(6) Correlation between the wear volume and selected mechanical properties emerged from the present investigation.

(7) SEM studies of the worn surfaces support the involved mechanisms during the abrasion process.

Conflict of Interests

The authors declare that there is no conflict of interests with any financial organization regarding the material discussed in this paper.

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