



Effects of Fibers and Fillers on Mechanical Properties of Thermoplastic Composites

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ABSTRACT

Thermoplastic copolyester elastomer (TCE) and Polyoxymethylene (POM) filled polytetrafluoroethylene (PTFE) composite, reinforced with short glass fiber (SGF) and different shape microfillers such as short carbon fiber (SCF), silicon carbide (SiC) and alumina (Al_2O_3) were prepared by melt mixing method using twin screw extruder followed by injection moulding. Mechanical properties such as tensile, flexural and impact strengths were studied. The mechanical properties test results show that short glass fiber obviously improves the strength of TCE and POM filled PTFE composites and different shape ceramic fillers decreases the tensile and bending properties of TCE filled PTFE composite, but different shape microfillers exhibits a synergistic effect on tensile and bending properties of POM with PTFE simultaneously. Hybrid composites have shown lower tensile strength and strain with increase in fiber/filler content. POM composites exhibited better tensile (strength of 75.78 and modulus of 1769.1 MPa respectively) and flexural (strength of 116.2 and modulus of 5697 MPa respectively) properties. TCE composites showed moderate elongation at break and better impact strength as high as 80 J/m (with 20 wt % glass fiber content) compared to POM composites.

Keywords: TCE/POM filled with PTFE hybrid composites, Fibers and fillers, Mechanical properties

1. INTRODUCTION

The use of polymeric materials has become very popular in engineering applications. As a result, a continuous improvement in fiber/matrix, development of innovative fabrication technologies, advanced polymeric composite offers possibilities for major leaps in design, manufacturing, energy conservation, product utility and diversity. Glass fibers are the reinforcement agent most used in thermoplastic based composites, as they have good balance between properties and costs. However, their final properties are mainly determined by the strength and stability of the polymer-fiber interphase. Fibers do not act as an effective reinforcing material when the adhesion is weak. Many efforts have been done to improve polymer-glass fiber adhesion by compatibility enhancement. The most used techniques include modifications in glass surface, polymer matrix and/or both.

Although the thermoset showed superior tensile, shear and compressive strengths, several deficiencies were uncovered. With the epoxy-based

composites, namely inferior performance in damage tolerance, hot/wet stability and high manufacturing cost associated with the conventional hand-layup fabrication process. Significant progress has been made for improving the fracture toughness property of the thermoset systems, but other problems associated with hot/wet stability, recyclability and environment protection issues and manufacture cost remained unresolved. These needs have drawn attention to the potential use of thermoplastic matrix system. Polymer based composites is very common in situations where combinations of good properties are required. It is often found that such properties are not attainable with a single polymer alone. The methods of polymer modification include copolymerizing, reinforcing and blending. Strengthening of polymers with fibers and fillers in augmented proportions reveals unlimited possibilities of producing materials with variable properties. These advantages have led to its rapid use in applications of polymers.

Thermoplastic copolyester elastomer (TCE) is a unique material combining the strength and

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processing characteristics of engineering plastics with the flexibility of elastomers. It offers highly consistent performance over a wide operating temperature range with very low variation in properties between low and high temperature extremes. They are novel constructional polymers, which are physically cross linked materials made up of a thermoplastic and an elastomer. Applications include flexible couplings, ski boots, gears, pressure hose lines, coverings for wire and optical fiber cables [1].

Polyoxymethylene (POM) is a crystalline polymer with very high crystallinity, strong, stiff and tough engineering material with lower coefficient of friction. However, POM is notch sensitive material. It is denser than polyamide but in many respects their properties are similar and they can be used for the same types of engineering application. A factor which may favour acetal in some cases is its relatively low water absorption [2].

Polytetrafluoroethylene (PTFE), an engineering plastic, has been widely used in industrial fields because of its excellent thermal stability, good solvent resistance and low-friction coefficient. However, its application is greatly limited by its poor mechanical properties and difficult to process [3]. A lot of research has been made to improve the mechanical properties by means of incorporation of PTFE with various neat polymers/fillers, such as fibers, fine particles, whiskers.

The reinforcing fibers of advanced polymer composites are responsible for their high strength and stiffness. However, these can be fulfilled only if sufficient stress transfer from fiber to matrix and vice versa can take place by a proper bonding between the two constituents. This means that physical and to some extent chemical compatibility is required between fiber and matrix. Therefore, the structure and properties of the fiber-matrix interface play a major role in the mechanical and physical properties of composite materials. The most important fibers in current use are glass, carbon, aramid, etc. Of the fibers, glass has a relatively low stiffness; however, its tensile strength is competitive with the other fibers and its cost is dramatically lower. This combination of properties is likely to ensure that glass fibers remain the most widely used reinforcement for high-volume commercial polymer matrix composites applications.

Fillers, in the form of particulates and short fibers are often added to polymeric materials to enhance their processability and mechanical compound properties, as well as to reduce material costs. Filler behavior in the compound was affected by factors

like the particle size distribution, surface area, shape and surface chemistry [4]. Silicon carbide (SiC), a well-known ceramic material, is the only chemical compound of carbon and silicon. It increases the abrasion resistance and has unique physical properties such as superior resistance to chemicals and high-temperature, high-electron mobility, excellent thermal/heat conductivity and superior mechanical properties [5, 6], which permit possible various applications such as nanodevices in harsh environments. Aluminium oxide (Al_2O_3) is used for its better hardness and strength. In addition to the increase in strength, it improves the fatigue properties. Also reduces coefficient of thermal expansion and improves chemical inertness, improves hardness and abrasion resistance. Improved strength retention at elevated temperatures has also been cited in literature [7].

Over the decades, it has been a focus of research for enhancing the mechanical properties of polymers by incorporating fibers and fillers and research articles updating the state of art of polymer based composites for structural applications. Several investigations on mechanical characterization of polymer matrix and their composites reinforced with fibers and fillers have been carried out. Bijwe et al. [8] investigated the role of adding PTFE (PTFE of 0, 7.5, 15, 22.5 and 30 wt. %) into polyetheretherketone (PEEK) to form blends on mechanical properties. They found that 30 wt. % PTFE into PEEK showed the maximum impact strength and other mechanical properties are diminished. Zhao Rong-guo et al. [9] reported that in PTFE/PA6 and PTFE/PA66 blends, increment of PTFE content reduces tensile strength, flexural strength, and impact strength of the blends. Chiang and Huang [10] studied the various properties of the blends of POM with up to 20 wt% chemically surface-treated polytetrafluoroethylene (CPTFE) and compared with those of POM/PTFE blends. The mechanical properties of POM/PTFE blends decrease with increasing PTFE content, but tensile strength and Young's modulus of POM/CPTFE blends are more than 2 times higher than that of the POM/PTFE blends. Loon [11] in his research work discussed the investigation of Chiang et al. and Palanivelu et al. Chiang et al. have investigated that the tensile strength, modulus, density and crystallinity decreased by increasing the thermoplastic polyurethane (TPU) concentration in POM blends. Palanivelu et al. have investigated that the tensile and flexural strength of the blends decreased with increasing TPU concentration. However, impact strength increased with increasing TPU concentration in POM blends. Sakai et al. [12] investigated the flexural properties of glass fiber reinforced POM. They concluded that the bending strength and

modulus increased as the fiber content increases. However, the effects of increasing fiber volume fraction on mechanical properties were larger than the effect of increasing crystallinity. Naga Mahendra Babu [13] investigated the effect of glass fiber and fillers (red mud+SiC) addition into polyetheretherketone (PEEK) composite and concluded that SGF+PEEK improves the flexural properties. Addition of red mud+SiC to SGF reinforced PEEK composites improves the flexural strength, flexural modulus and tensile strength of the material. Mamoor et al. [14] investigated the effect of short glass fiber (SGF) on mechanical properties of polymethyl methacrylate (PMMA) and styrene butadiene rubber (SBR) vulcanizates and concluded that increases in concentration of SGF causes increase in tensile strength and decrease in elongation at break. Jian and Tao [15] investigated the mechanical properties of polyphenylene sulphide/carbon fiber (PPS/CF) composites and polyamide 6 (PA6) filled PPS/CF composites. They concluded that 25 % wt CF in PPS significantly improved the flexural properties of composite. They also revealed that 6 % wt. PA6 in CF/PPS composite exhibited better flexural properties than that of CF/PPS composites. Kushwaha et al. [16] investigated the mechanical properties of nickel coated carbon fiber reinforced polycarbonate composites and concluded that there was considerable improvement in tensile and flexural properties, but had no effect on the shore hardness values of the composite. Hua et al. [17] investigated the effect of CF and nano silica (nano-SiO₂) on the flexural and tribological properties of POM composites. They concluded that combination of 3 vol % nano-SiO₂ and CF in POM offers an optimized composite material with excellent mechanical and tribological properties. Shoufan Cao et al. [18] reported the effect of basalt fibre in ultra-high molecular weight polyethylene (UHMWPE). Increase in basalt content led to decrease in toughness and increase in strength, hardness and creep resistance. Alhareb and Ahmad [19] reported that the incorporation of aluminium oxide (Al₂O₃)/zirconium oxide (ZrO₂) into PMMA managed to improve that fracture toughness, tensile modulus and flexural properties of denture base composite materials. Tarawneh et al. [20] reported that the tensile strength, tensile modulus and impact strength are improved significantly while sacrificing high elongation at break by incorporating multi-walled carbon nanotubes as filler in thermoplastic natural rubber (TPNR) compared to pure TPNR. Sreekanth et al. [21] investigated the role of inorganic fillers namely mica and fly ash added to the polyester thermoplastic elastomer (TPE). They concluded that there is a significant increment in the flexural strength and modulus with an increase in the filler

concentration. The impact strength decreases with concentration of filler due to the reduction of elasticity of material due to filler addition and thereby reducing the deformability of matrix and its ability to absorb deformation energy.

The effectiveness of reinforcement essentially depends on the adhesion between matrix and fiber, so this is a key factor in determining the final properties of the composite material, particularly its mechanical properties. The fiber-matrix adhesion is confined to a region known as interphase, where stress-transfer occurs. The interphase is defined as the tridimensional region, located between the fiber and the matrix. It is considered as a transition region or third phase with its own characteristics, corresponding neither to fiber properties nor to matrix ones.

In spite of the fact, that the polymer composites are used in structural applications, no data are reported on the influence of PTFE in TCE and PTFE in POM composites with SGF and other inorganic particulate fillers viz. short carbon fiber (SCF), silicon carbide (SiC) and alumina (Al₂O₃). Keeping this in view, a series of TCE/PTFE and POM/PTFE composites with silane treated short fibers and ceramic fillers were investigated for their mechanical properties and the results are compared to determine suitable applications. Efforts were made to study the role of fiber and ceramic fillers inclusion on the relevant strength properties.

2. EXPERIMENTAL

2.1. Materials

A thermoplastic co-polyester elastomer (TCE) and POM were considered separately as a matrix material in this study. PTFE was selected as particulate filler to form a polymer composite. Silane treated short glass fibers were used as reinforment. Short carbon fibers, SiC and Al₂O₃ were selected as micro-scale fillers. The average diameter of the short glass fibers was approximately 12 µm with an average fiber length of about 4 mm. The average diameter of the carbon fibers was approximately 7 µm with an average fiber length of about 100 µm. The particle size of PTFE was about 5 to 15 µm and around 5 to 10 µm for SiC and Al₂O₃. The source and selected properties of these materials are listed in Table 1.

2.2. Specimen preparation

2.2.1. Compounding

Before compounding the polymer granules and fillers were dried at 75°C for 10 h in an oven. Selected compositions were mixed and extruded in Barbender co-rotating twin-screw extruder (Make:

Table 1. Characteristics of materials used for the study.

Polymer/Filler	Designation	Density (g/cm ³)	Source and Supplier
Thermoplastic			DSM and M/s Gargi
Copolyester Elastomer	TCE	1.29	enterprise, Bangalore.
Polyoxymethylene	POM	1.42	Du Pont Co. Ltd.
Polytetrafluoroethylene	PTFE	2.16	Du Pont Co. Ltd.
Short glass fibre	SGF	2.50	Fine organics Mumbai
Short carbon fibre	SCF	1.74	Fine organics Mumbai
Silicon carbide	SiC	3.21	Carborundum India Ltd.
Alumina	Al ₂ O ₃	3.95	Triveni groups

Table 2. Constituents of the polymer material composite system for present study.

Material code	Matrix (% wt)	Fiber (% wt)	Fillers (% wt)	Measured Density (g/cm ³)
T ₁	TCE (85)	-----	PTFE (15)	1.38
T ₂	TCE (85)	SGF (20)	PTFE (15)	1.60
T ₃	TCE (85)	SGF (20) + SCF (2.5)	PTFE (15)	1.79 +Al ₂ O ₃ (6.25)

Table 3. Constituents of the polymer material composite system for present study.

Material code	Matrix (% wt)	Fiber (% wt)	Filler (% wt)	Measured Density (g/cm ³)
A ₁	POM (85)	-----	PTFE (15)	1.47
A ₂	POM (85)	SGF (20)	PTFE (15)	1.67
A ₃	POM (85)	SGF (20) + SCF (2.5)	PTFE (15) SiC (6.25) + Al ₂ O ₃ (6.25)	1.84

Table 4. Mechanical properties of TCE and POM composites

Property	Composites					
	T ₁	T ₂	T ₃	A ₁	A ₂	A ₃
Duro hardness (Shore D)	60.8	72.8	69.7	69.9	75.2	73.7
Tensile strength (MPa)	30.58	57.84	30.16	46.97	75.78	44.48
Tensile modulus (MPa)	624.4	797.8	693.8	832.5	1769.1	936.6
Elongation at break (%)	18.9	16.36	9.87	17.77	17.13	12.9
Flexural strength (MPa)	41.0	80.7	51.49	79.0	116.2	81.2
Flexural modulus (GPa)	1.068	2.528	3.038	2.85	4.75	5.70
Deflection at bending (%)	21.10	16.54	9.08	17.84	8.92	12.96
Izod impact strength (J/m)	60.0	80.0	40.31	42.0	32.0	38.0

CMEI, Model: 16CME, SPL, chamber size 70cm³. The compositional details of each material are

presented in Table 2 and 3. The mixing speed of 100 rpm was maintained for all the compositions. The melt blending, temperature profile of the extrusion are 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 in pelletizing machine.

2.2.2. Injection moulding

The pellets of the extrudates were pre-dried at 100°C in vacuum oven for 24 h and injection moulded in a reciprocating screw injection

moulding machine (DGP Windsor, 50T) to produce tensile, flexure, and impact test samples. The processing parameters for zone 1 (220°C) and zone 2 (250°C) respectively. The mould temperature was maintained at 35°C .

2.3. Density test

Density test was conducted as per ASTM D792 using METTLER AE 200 densometer. The specimen is weighed in air, and then weighed when immersed in distilled water at 23°C using a sinker and wire to hold the specimen which was completely submerged. Density was calculated.

2.4. Hardness

Hardness of the polymer blend and their composite materials were tested as per ASTM D2240 (type M) using HP-E II series digital Durometers. Durometers has patented pressure mechanism, which ensures a constant contact pressure according to standards, eliminating measuring errors caused and influenced by tilting or slope contact. The measured value will be shown on the display.

2.5. Tensile test

The tensile properties were performed according to ASTM D638- Type 1 using Universal testing machine (Lloyds, capacity 1-20 kN). Testing speed was set at 5 mm/min and carried out at room temperature and specimen dimensions are 165mm x 19 mm x 3.3 mm as shown in Figure 1. Tensile modulus and strength were evaluated from the load-displacement curve.

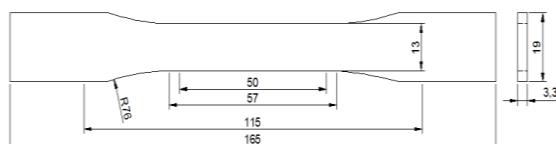


Figure 1: Tensile test specimen dimensions

2.6. Flexure test

The flexural properties were determined by three-point bending test and were performed in accordance with ASTM D790-Type B using Universal testing machine (Lloyds, capacity 1-20 kN). The span length was set at about 50 mm. Testing speed was set at 2 mm/min and carried out at room temperature and specimen dimensions are 125 mm x 12.5 mm x 3.3 mm as shown in Figure 2. Flexural modulus and strength were evaluated from the load displacement diagrams.

2.7. Impact test

Izod impact strength was performed according to ASTM D256-Type A using INSTRON (Impact hammer with a mass of 1.3 kg) impact testing machine at the striking rate of 3.2 m/s. A notch of

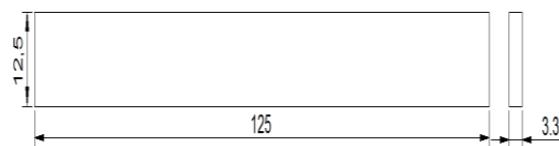


Figure 2: Flexural test specimen dimensions

2.5 mm width with an included angle of 45° was generated on the sample in the thickness 3.3 mm and width and length of the sample was 12.5 and 63.5 mm respectively (Figure 3).

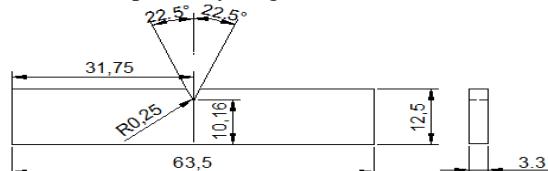


Figure 3: Impact test specimen dimensions

3. RESULTS AND DISCUSSION

3.1. Density

TCE/PTFE and POM/PTFE composites with varying content of SGF, SCF, SiC, Al_2O_3 and MoS_2 in wt. % are summarized in Table 2 and 3. TCE/PTFE and POM/PTFE composite exhibits a density of 1.38 and 1.47 g/cm^3 respectively which is slightly greater than that of pure TCE and POM. This is due to the presence of PTFE in TCE/PTFE and POM/PTFE composite. Bijwe et al. [8] reported that inclusion of PTFE in PEEK increases the density. All other samples, i.e. TCE/PTFE and POM/PTFE composites exhibit higher density value respectively. The presence of fibers, microfillers and MoS_2 makes TCE/PTFE and POM/PTFE hybrid composite a denser material among their respective groups in this study. Figure 3, depicts the graph comparing the density of both TCE and POM composites. POM/PTFE composites are denser than TCE/PTFE composites in the range of 6 % to 3 % approximately.

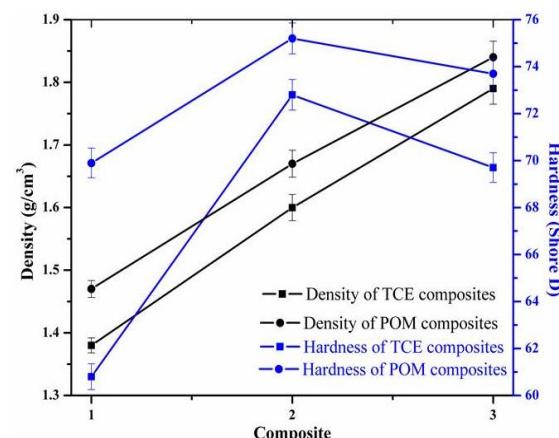


Figure 4: Comparison of density and hardness of TCE and POM composites

3.2. Hardness

Table 4 and Figure 4 summarize the hardness (Shore-D) values of TCE/PTFE and POM/PTFE composites. Inclusion of PTFE to TCE and POM reduced the hardness. Bijwe et al. [8] reported that addition of PTFE into PEEK composite reduces the hardness value. The results for the TCE/PTFE and POM/PTFE composites revealed that the hardness increases with addition of short fibers and inorganic fillers weight fraction, and reaches its maximum amount compared to the hardness of the TCE/PTFE and POM/PTFE which is equal to 60.8 and 69.9 respectively. This increase in hardness is attributed to increasing wettability or bonding (interaction) between the matrix, fibers and the filler particles.

3.3. Tensile properties

Tensile properties provide information about the behaviour of the material when it is subjected to stretching or pulling force before it fails. In this work, the mechanical properties such as tensile, flexure and impact were used to evaluate the strength and toughness of TCE and POM filled PTFE and their hybrid composites. The characterization of the composites reveals that inclusion of short fibers and particulate fillers has very strong influence on the mechanical properties of composites. By incorporating short glass fibers into the TCE/PTFE and POM/PTFE composites, synergistic effects, as expected were achieved in the form of modified tensile and flexural properties.

Tensile properties of TCE/PTFE and POM/PTFE composites are shown in Figure 5. Inclusion of PTFE particles into neat TCE and POM has deteriorated the tensile properties by 12 % and 23 % respectively, but addition of SGF into composites has improved the tensile strength by 40 % in TCE/PTFE composites and 20 % in POM/PTFE composites. The addition of SiC and Al_2O_3 in the composites deteriorated their tensile properties by 48 % in case of TCE/PTFE and 41 % in case of POM/PTFE, both reinforced with SGF. Microfillers used in this study, cause maximum reduction in the composite's tensile strength and elongation at break. It may occur due to the poor interface bonding between the matrix, microfillers and fibers resulting in poor adhesion of particles and fiber pull-out, which are not good to transfer the tensile stress. Another reason is that the corner points of the irregular shaped particulates result in stress concentration in the polymer composites. However, POM/PTFE composites exhibits better tensile properties than that of TCE/PTFE composites, except elongation at break due to tensile load.

3.4. Flexural properties

Flexural properties provide information about the behaviour of material in bending. The tested result of flexural strength and flexural modulus values of TCE/PTFE and POM/PTFE composites with varying content of SGF, SCF, SiC and Al_2O_3 varying in wt. % is shown in Figures 6 and Table 4. The TCE/PTFE and POM/PTFE composite showed poor flexure mode properties. This is due to the addition of PTFE as second matrix material, which reduces the flexural properties because of poor compatibility. Many of researchers have showed that the incorporation of fibers increased both flexural strength and modulus [20, 21]. The result in the investigation reveals that TCE/PTFE and POM/PTFE with SGF exhibits better flexural properties. This may be due to uniform distribution of SGF and strong adhesion of polymer matrix material to the SGF. The applied load penetrates the matrix material and transferred around the fiber instead of penetrating it. These results in fiber rupture. Both TCE/PTFE and POM/PTFE composites with fibers and micro fillers exhibited better flexural modulus. This composition could act as very good reinforcing filler in the TCE/PTFE and POM/PTFE composites. Also, the stresses are received by these fillers, hence, which lead to moderate deflection. However POM/PTFE composites exhibited better flexural strength and modulus than that of TCE/PTFE composites, except the deflections due to bending load.

3.5. Impact strength

It is the ability of the material to resist the fracture under stress applied at high speed. The specimens are deformed within a short time and therefore exposed to high strain rates. Impact strength of TCE/PTFE blend and their composites are shown in Figure 7 and also in Table 4. SGF reinforced TCE/PTFE composites have the highest impact strength among all the composites. This increase is due to fiber-related energy dissipation mechanisms, such as fiber debonding, pull out, bridging and fracture, which induce plastic deformation of the

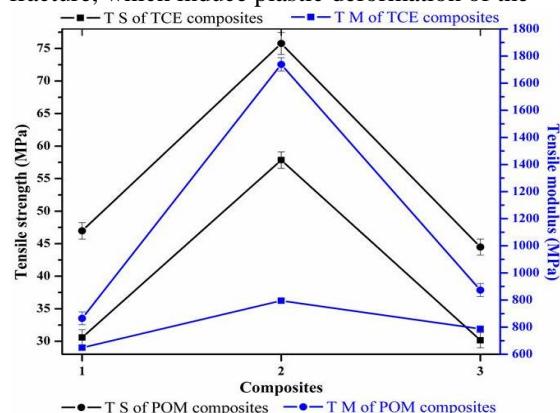


Figure 5: Comparison of tensile properties of TCE and POM composites

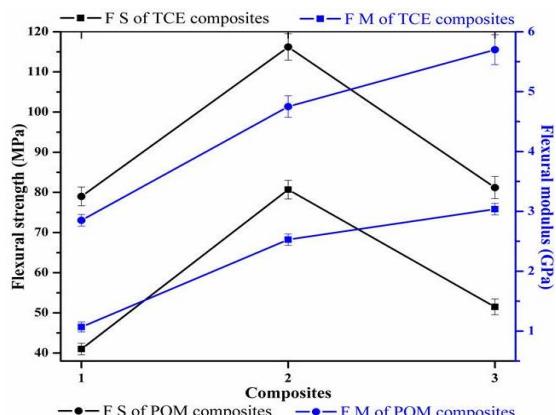


Figure 6: Comparison of flexural properties of TCE and POM com

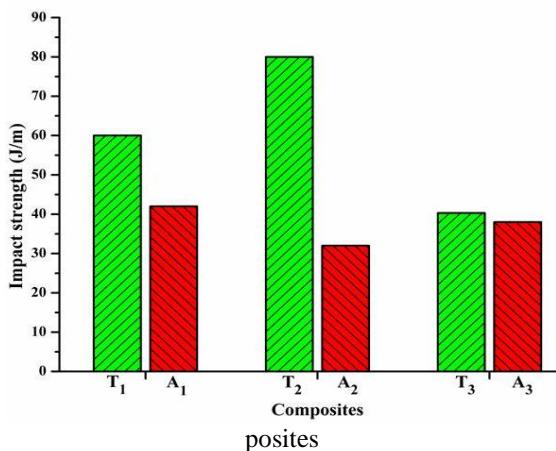


Figure 7: Comparison of impact strength of TCE and POM composites

polymeric matrix before failure. Bridging and fiber fracture are likely to occur as a consequence as a set of glass fibers with length longer than the critical value for effective reinforcement, while debonding and fiber pull out are expected to occur as the result of a set of glass fibers with length shorter than the critical value. However, POM/PTFE composites exhibited poor impact strength and this is due to its high hardness in nature (82 HRC) than that of TCE/PTFE composites.

4. CONCLUSION

- Short glass fiber (20 wt. %) as reinforcement, increased the mechanical strength of neat matrix material except the impact strength in case of POM/PTFE composites.
- POM/PTFE composites showed better hardness, tensile and flexural properties than TCE/PTFE composites except the elongation/deflection at break due to load.
- TCE/PTFE composites showed better impact strength as high as 80 J/m than any of the POM/PTFE composites.

- Composites filled with short glass/carbon fiber, SiC, and Al₂O₃ composites, exhibited improved flexure strength and modulus.
- Mechanical properties of both the composites were discussed and POM/PTFE composites proved to be better than TCE/PTFE composites in terms of tensile and flexural load bearing capacity. TCE/PTFE composites proved to be better than POM/PTFE composites in terms of impact energy a capacity.

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