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Preparation and Analysis of New Hybrid PAEK Nano-Composites Containing MWCNT, Inorganic and Organic Nano-Particles

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Abstract:
In modern era light weight material with complex shape and high material properties is required for advanced applications at room temperature. In this concern, the experimental study analysis to observe the effect of different nanofiller material like GO, MWCNT, NH2 and COOH modified MWCNT as organic nanofiller and WS2 and MoS2 as inorganic nanofiller reinforced in pure PAEK. This experimental study focuses on incorporation of organic and inorganic nanofillers separately and combine as hybrid nanofiller nanocomposite. On preparation of PAEK nano-composite and hybrid nanocomposite the comparative study of mechanical properties like tensile, flexural and impact is carried out. The nano-composites were prepared by melt compounding method. Scanning Electron Microscopy confirm the homogenous dispersion and alignment of nanofilers. The hybrid nano-composites show highest tensile strength as compared to the organic and in-organic nanofiller nanocomposite. As the percentage of MWCNT increases from 0.2 to 1 wt% , the properties of nano-composites decreases due agglomeration tendency of MWCNT that results in low load transfer capacity of nanocomposite, therefore agglomeration tendency of MWCNT required to be controlled for preparation of nano-composite and this is achieved by reinforcing Hybrid nanofillers to pure PAEK matrix. However, organic filler shows 75 MPa to 83 MPa tensile properties and which is lower compared to in-organic and hybrid filler due agglomeration tendency of organic nanofiller confirmed by SEM analysis.

Keywords: Multi-wall Carbon Nano Tubes, In-organic filler, Organic filer, tensile strength, flexural strength, Impact strength.

1. Introduction

Polyaryletherketones (PAEK) is a high performance thermoplastic which is in a semi-crystalline structure. PAEK possess unique combination characteristics like fair thermal stability in organic environment with better mechanical properties (such as strength and toughness), chemical inertness, solvent resistance, good fire resistance and electrical performance over a wide range of commercial and industrial applications [1]. Further, the glass transition temperature and melting temperature of PAEK are 147 °C and 310 °C respectively; in addition to that they have higher density than other PEEK thermoplastics [2]. However, higher density of PAEK leads to better robustness in structure which is used in aerospace industry, energy field, chemical industry, and medical field also it having good machinability offer to making complex engineering shape. Besides, these thermoplastics have poor wear resistance, also low heat dissipation rate makes them to excessive heat expansion results reduces the material performance in real filed application. Hence in the last decades large number of researchers takes efforts to improve the PAEK properties by incorporated nano reinforcement nanoparticles, like SiO_2, Al_2O_3, SiC and glass fiber [1-3]. Among in that carbon nanotubes is widely used as reinforcement to enhance the PAEK properties called as polymer nano composites. This carbon nanotube has small dimensions, tube like shape, rigidity to prompt the mechanical, electrical and thermal properties of PAEK. Therefore, these unique properties of nano-composites used in to manufacture automobile and aeronautical parts, thermal interface materials, and packing where higher flexibility design required for higher performance at cheaper cost [4]. In this regard Satyanarayana and Hubner reported dispersion of carbon nanotube (CNT) in various thermoplastics through different methods [5]. The article includes the properties, synthesis and different modification methods on CNT along with fabrication methods. The effect of dispersion of CNT is studied for different properties like mechanical, thermal and electrical [5].Bangarusampath et al. [6] manufactured PEEK/MWNT nanocomposite by melt blending with a twin screw extruder and compression molding techniques. A semi-crystalline PEEK 151 grade with molecular weight of 27 kg/mol was used as the matrix and nano composites were prepared at different weight percentage of MWCNT 2, 5, 10 and 17 wt. Experimental results seen that a smaller region the MWNTs well-dispersed in the PEEK due to the shear forces applied during melt mixing. In addition to that thermal and mechanical properties of PEEK nano composites were linearly increases with MWNT percentage. Diez-Pascual et al. [7] fabricated PEEK nanocomposite using SWCNT under different weight percentage using melt-mixing in micro-extruder. They observed that thermal and mechanical properties of PEEK nano composites increased due to efficient dispersion of SWCNT. Also, as increases the SWCNT upto 0.1 % larger crystalline size of nano composite was seen than the raw matrix but beyond 0.1% of SWCNT tough for higher crystalline structure due to polymer chain diffusion. Kuo et al. [8] developed PEEK composites with a reinforcement of nano-sized silica and alumina (Al_2O_3) by varying weight percentage. As the weight percentage of silica and alumina increases in the matrix hardness of nano composites also increases but loss of tensile ductility. Also it was observed that there was no chemical reaction occurred between non-organic nano particle mix and matrix. Rong et al. [9] investigated effect of carbon nanotubes on the mechanical properties and crystallization behavior of PEEK. As an increasing the nanotube loadings increased tensile strength, and more improvement of tensile strength observed in f-CNTs/PEEK composites with good interaction between f-CNTs and PEEK. It was also found that f-CNTs/PEEK composites show better mechanical properties in comparison with CNTs/PEEK composites. Because good compatibility for PEEK and SPEEK with f-CNTs because of their similar structure. Results show that load was efficiently transferred from matrix to f-CNTs. Song et al. [10] prepared PEEK composite using sandwich structure technique in which of SWCNT paper layer and PEEK placed one by one. They observed that 40% of Young’s modulus and 4% failure strength increased of PEEK composite as compared to pure PEEK. Lu-qi Lui et al. [11] compared mechanical strength and stiffness of multi-wall carbon nanotube (MWCNT) dispersed in thermoset epoxy and thermoplastic PMMA. The nanocomposites are manufactured by solution mixing method. Deng et al. [12] reported multi-walled carbon nanotubes reinforced in PEEK by melt blending under various percentage of MWCNT such as 0, 6.5, 9, 12 and15 wt%. The experimental results observed that as compared to pure PEEK, an addition of MWNTs increased in the elastic modulus and the yield strength. The tensile modulus of the pure PEEK matrix at room temperature was 4GPa and increased by 88% to 7.55 GPa for composite containing 15 wt% MWNTs. Naffakhet.al [13] prepared PEEK/INT-WS_2 (inorganic nanotube-tungsten disulphide) nano-composite by melt mixing method. They studied thermo-gravimetric analysis and concluded that use of INT-WS_2 in PEEK increases thermal stability of nano-composites and improve thermo-mechanical properties. Kalin et al. [14] studied preparation of PEEK/WS_2, CNT and grapheme nanopowder (GNP) by ultrasonic mixing then solution blending followed by hot pressing. In this work effects of WS_2, CNT and GNP are compared for hardness and tribological properties. Higher hardness was observed in case of PEEK/WS_2 whereas high wear rate seen in PEEK/GNP combination. Toshio et al. [15] investigated nonlinear stress-strain behavior of CNT dispersed (PEEK) composite. They prepared PEEK/CNT master batches containing 0, 9, 15 wt. % CNT using a twin-screw extruder. An experimental result shows that many CNTs in the CNT/PEEK composite are aligned longitudinally by FIB observations. Although the stress-strain behavior of PEEK is almost linear up to 1.5% strain, the stress-strain curves of CNT/PEEK composites exhibit considerable nonlinear and hysteresis behaviors from the extremely low strain (<0.1%) under both tensile and compressive loading.

From the above literature it was observed that polymer matrix (PAEK) material property was improved by adding CNT or organic and in-organic reinforcement besides most of the authors were used organic, SWCNT as nano filler for enhancing properties also few of the authors were also used MWCNT as reinforcement. But the development strategies to incorporate MWCNTs homogenously into PAEK matrix is not addressed and agglomeration tendency of organic nanofillers especially about MWCNTs is not discussed. Also, comparative study between pristine MWCNT and f-MWCNT on properties of nano composite is not discussed in the earlier literature. Therefore, the main aim of the study is to develop the PAEK nano composite along with PAEK Hybrid nanocomposite using standard process technique for sucessful MWCNT reinforcement under pristine and functionalized condition. Also to study the effect of different weight percentage of MWCNT, organic and in-organic filler separately and combine on mechanical properties of resulting nanocomposite material.

Experimental details

Materials

In this study PAEK 1200 P (Poly Aryl Ether Ketone) used as polymer matrix in powder form was supplied by Gharada Chemical Ltd. Mumbai under the trade name of G-PAEK (See Fig. 1a). The PAEK was in unreinforced structure with low viscosity. The typical characteristics of PAEK are tabulated in Table 1.

Table 1 Typical property of PAEK and MWCNT

PAEK properties	
Glass transition temperature (°C)	152-154
Melting temperature (°C)	370-372
Density (g/cm^3)	1.30
MWCNT properties	
Outer diameter (nm)	10-30
Inner diameter (nm)	5-10
Length (μm)	5
Bulk density (g/cm3)	0.04
CNT content	95-99%

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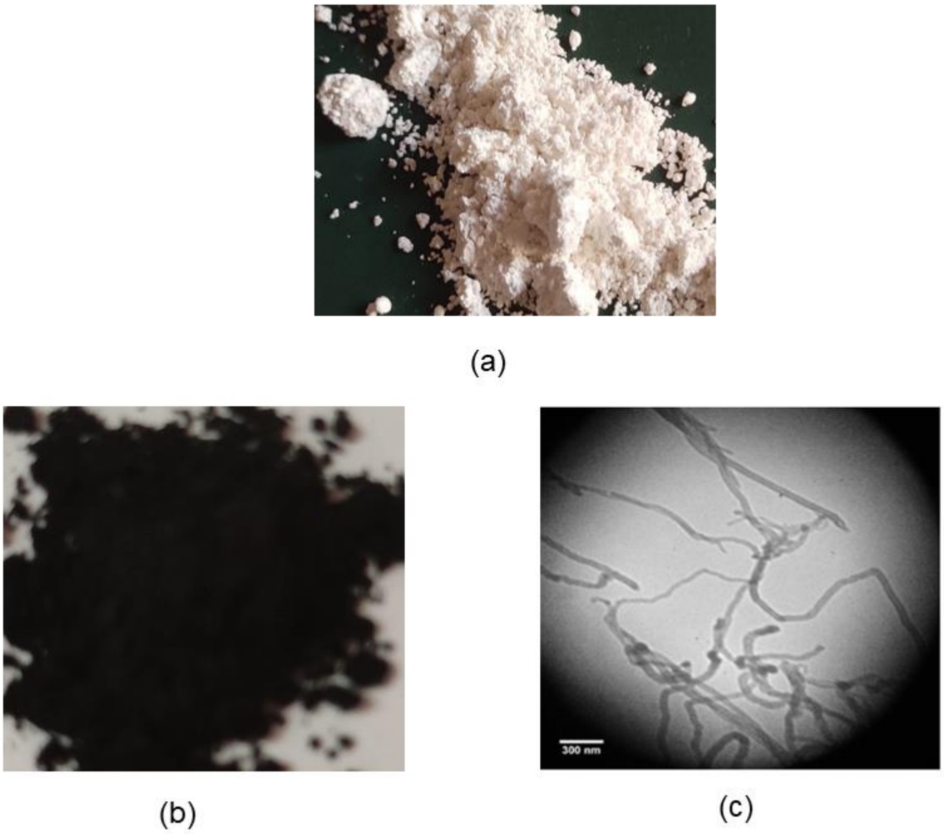

Figure 1 (a) Pure PAEK, (b) Carbon nano tube, and (c) morphology of PMWCNT TEM image

Carbon nanotubes (density: 0.04 g/cm^3, outer diameter= 10 to 30 nm), Graphene oxide nanosheets (density: 0.05 g/cm3, average thickness: 1 to 2 nm), and Molybdenum disulfide (MoS_2) (particle size: 80 to100 nm, Density: 4.8 g/cm^3) was purchased by Ad-nano Technologies Pvt. Ltd., Shimoga. . Tris (nonylphenol) phosphite, TNPP, was used as a stabilizer and antioxidant agent in processing PEK polymer nanocomposites.

1.1 Preparation of PAEK/MWCNT composite

In this work, PAEK polymer based nanocomposites and Hybrid nanocomposites were prepared by using melt-mixing method. Because this method helps to provide uniform dispersion of filler in to the pure polymer. Various PAEK nanocomposites samples were prepared by variation in nanofiller contents such as by weight 1% COOH, 1% NH2, PMWCNT (Pristine), Pure PAEK, 1% WS2, 1% MoS2, 0.5 % COOH + 0.5% WS2, 0.5% GO+ 0.5 % WS2, 0.5% GO+ 0.5 % MoS2, 0.2% COOHMWCNT+ 0.8 % WS2, 0.8% COOHMWCNT+ 0.2 % WS2. The value of percentage of nano filler has been selected based on the past literature. PAEK with nanofillers is subjected for melt-compounding using Coperion ZSK 26 (Germany) twin-screw extruder. Extrusion process carried out at rotational speed of 60 rpm with maximum barrel temperature was 400 °C. In this extruder there are 10 temperature zones available with flow rate of 12 kg/hr. All compositions viz. polymer and nanofiller were pre-dried in vacuum oven at 120 °C for 6 hr. Before extrusion 0.2 wt% TNPP added as an anti-oxidant in each PAEK nanocomposite batch. Further polymer and required amount of nanofiller mixed in Neoplast high speed mixer about 5 minutes. After melt-mixing of PAEK and nanofiller in twin screw extruder the PNC strands passed through blower for cooling and palletized by Lunarmech palletizer. Injection-molded samples (according to ASTM standards) were prepared using ARBURG 320C ALLROUNDER injection-moulding. The parameters of injection-moulding maintained for all the compositions were as such injection flow 40 cm^3/s, melt temperature 400 °C divided into 5 zones, mold temperature 190 °C, injection time 5 second, holding time 10 second and cooling time 20 second. The barrel diameter of injection moulding machine is 30 mm.

1.2 Characterization

The prepared nano-composites have various characterizations done such as tensile, flexural and impact. In the tensile characterization, tensile properties such as yield strength (σ_y), Young's modulus (E_t), stress at break (σ_b) and elongation at break (ε_b) were tested and calculated by using Universal Testing Machine (UTM Make: Hounsfield Tinius Olsen). The magnitudes of tensile strength, Youngs modulus and stress at break were obtained from stress-strain curve which provided by UTM. In addition to that toughness of the nano composite was obtained from stress-strain curve. For the tensile characterization the specimen was mold (prepared) as per ASTM D 638 having length 165 mm, width 12.6 mm, thickness 3.3 mm and gauge length 80 mm. Whereas, tensile specimens were obtained by injection molding. However the flexural strength measured by using 3 point flexural method machine (Make: Instron corporation) was carried in the Gharada Chemicals Mumbai. It was automated materials testing machine system were sample rates points per sec was 10 and full scale load range was 50 kN. The Izod impact testing was performed on Izod testing apparatus (Make: Tinius Olsen Model 892) by following a standard procedure and impact strength (toughness) calculate based on the reading of dial. However, to measure the impact strength of nano composite a notch was made middle of the test specimen. The dimensions and tolerances of test specimen were prepared as per standard ASTM D 256 were 63 X 12.6 X 3.3 mm. After successfully completion of nano composite characterization scanning electron microscopic analysis (SEM) has been carried on (Make: SEM EVO 18) for the impact tested fractured specimen. In the SEM analysis the fracture surfaces of impact specimen were observed as well as the agglomeration of the filler materials. For SEM analysis sample should be in electrical conductive nature but PAEK is a polymeric material leads to low electrical conductivity hence gold sputtering has been carried out on samples for ten minutes before the SEM study for SEM analysis.

2. Results and Discussion

2.1 Tensile Properties

The mechanical behavior of PAEK polymer is strongly decided by the contribution of each component and morphology developed during compounding and the interfacial adhesion between the phases. In addition to that elongation at break and toughness plays important role to monitor the adhesion between phases and tensile strength is related to the morphology, domain size and size homogeneity. Various tensile properties such as tensile strength, Young's modulus (Et) and elongation at break (εb) calculated by using universal testing machine (UTM) with crosshead speed of 5 mm/min.

Tensile property of PEEK composite under the different filler composition is as shown in Fig.1. It is observed that tensile strength of pure PAEK was showed in white color (no color fill) bar whereas

PAEK nano composite tensile properties was shown by colored bar. It was noticed that an organic filler nano composite tensile strength was less than the pure PAEK and its tensile strength reduces from 93 MPa to 75 MPa this is attributed to the agglomeration of organic nano filler as well as not reinforcing to the pure PAEK. Besides, these organic nanofillers possess good mechanical properties but having strong affinity of nano particles to each other which caused by strong Vander walls forces of attraction leading to limits their use in PAEK polymer as nanocomposites. Further, due to vander wall forces they form an agglomeration results deteriorates the mechanical properties of PAEK material. Also, large scale agglomeration of organic filler result nano composites become more brittle and load transfer capacity was reduced. However, in case of hybrid nano filler showed higher

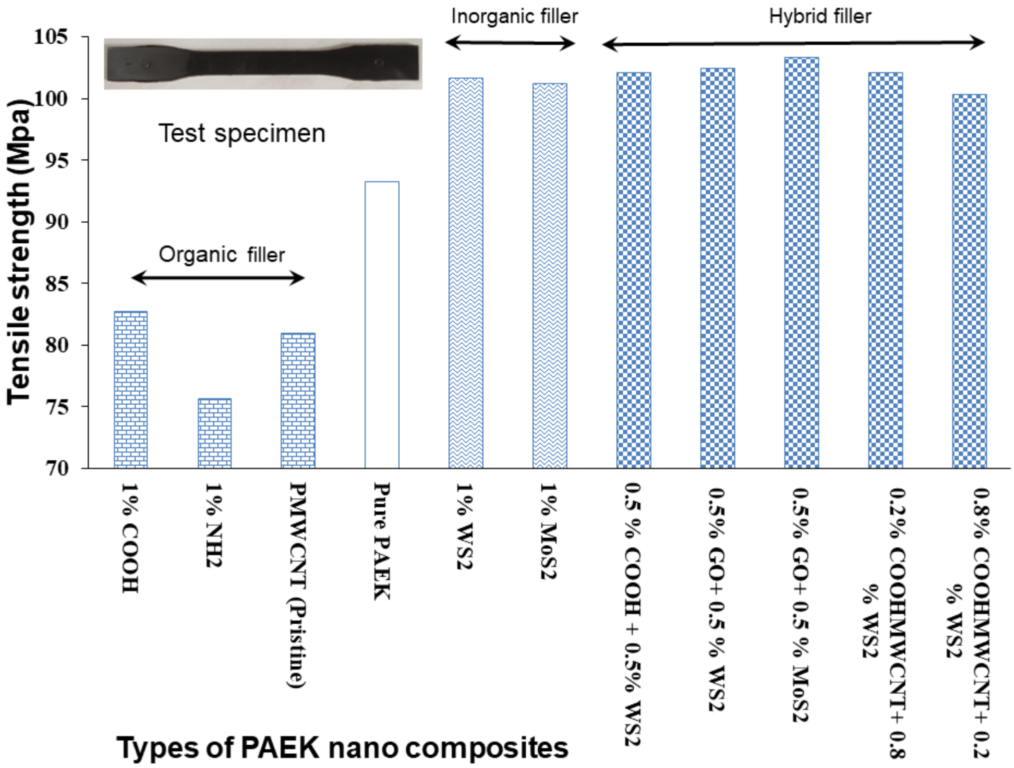

Figure 2 Effect of nano filler on tensile strength of PEEK nano composite

Table 2 Elongation and Modulus value of PEEK composite

Composition	Modulus (MPa)	Elongation break (%)
1% COOH	4363.104	3.022
1% NH2	4327.737	2.596
PMWCNT (Pristine)	4365.55	2.816
Pure PAEK	4254.095	3.561
0.2% CBMWCNT+ 0.8 % WS2	4167.404	5.217
0.8% CBMWCNT+ 0.2 % WS2	4252.03	4.312
1% WS2	4362.152	4.049
1% MoS2	4238.14	9.896
0.5 % COH + 0.5% WS2	4167.404	5.217
0.5% GO+ 0.5 % WS2	4202.648	12.615
0.5% GO+ 0.5 % MoS2	4149.217	12.793

tensile properties than the pure PAEK as well as organic and inorganic filler and its tensile strength increased from 93 MPa to 103 MPa. This is due to the extent of dispersion and uniform distribution of hybrid nanofiller in the pure PAEK. The hybrid nanofillers disperse homogeneously which leads to form better mechanical interlock as well as good reinforce with PAEK matrix and bonding with polymer molecules. This helps to even distribution of load as compared to other cases of polymer nanocomposites. Besides, in case of hybrid nanofiller 0.5% GO and 0.5% MoS_2 and 0.5wt% COOH MWCNT and 0.5wt% WS_2shows higher tensile strength as compared to other hybrid nano filler. The probably reason behind that when organic and inorganic are mixed to each other in PAEK composite there dispersion rate due to higher mobility of molecule which is found to be improved tensile strength besides, they having better inherent physical properties and similar result obtained in COOHMWCNT and WS_2. In case of COOH group is improve their dispersion rate within the polymer matrix due to random interruption of the structural regularity of the PAEK chain and strong the inter and intra-molecular interaction associated carboxyl group. It also depicts that in case of inorganic filler the tensile strength is higher than the pure PAEK and organic filler and lower than hybrid filler because its having better dispersion as compared to organic. Further, elongation at break is an indicator for the toughness of the materials a better enhancement in elongation at break is observed in case of hybrid nano filler due to high tensile strength. In case of organic filler elongation deteriorate and it is lower than the pure PAEK. But Elastic modulus of nano composite is found to be continuously decreasing in case of hybrid nanofiller. At 1wt% COOH organic filler shows highest modulus as compared to other composition. This may be due to agglomeration of filler is generally unwanted phenomenon but that leads to improve elastic modulus of material.

2.2 Flexural strength

The flexural strength of different nano-composite is illustrates in Fig. 3. Highest flexural

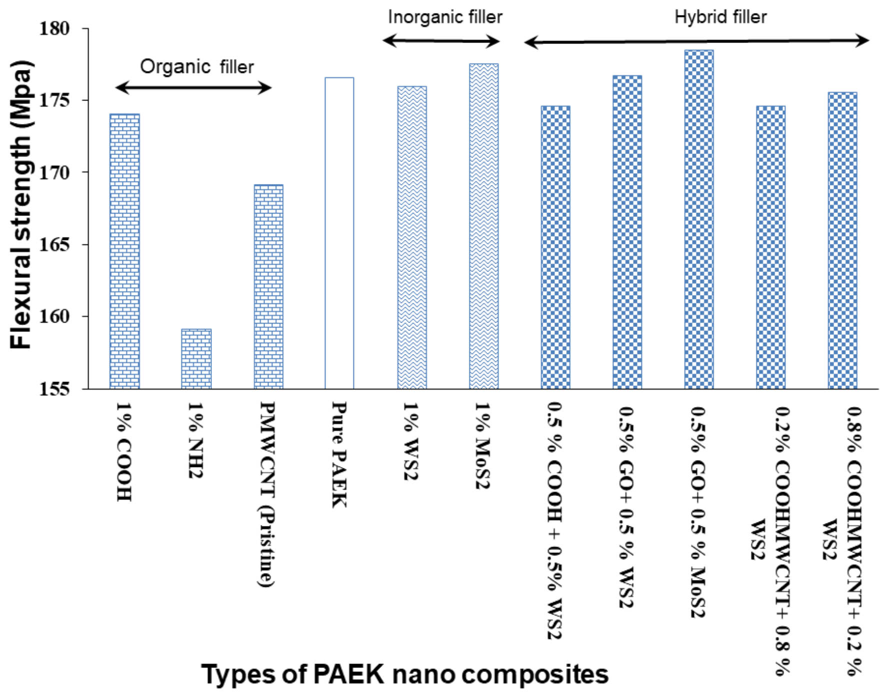

Figure 3 Effect of nano filler on flexural strength of PEEK nano composite

strength was observed in case of hybrid filler (0.5% GO and 0.5% MoS_2). This might be due to the ductile of MoS_2 and brittle GO easily and better interface in the PAEK matrix.

2.3 Impact strength

The impact test results of nano filler for PAEK composites under various nano filler is as shown in Fig. 4. It depicts that an addition of hybrid filller in PAEK matrix improves impact strength. This may be uniform dispersion of nanofiller in matrix due to better bonding occurs between matrix and nanofiller which increases the strength leads to increasing the impact strength. Further, in case of organic filler decrease the impact strength as compared to the pure PAEK due to reduction of toughness of composites due to formation of agglomeration. Agglomeration of fillers acts as a stress concentrated areas leads to deteriorate impact strength.

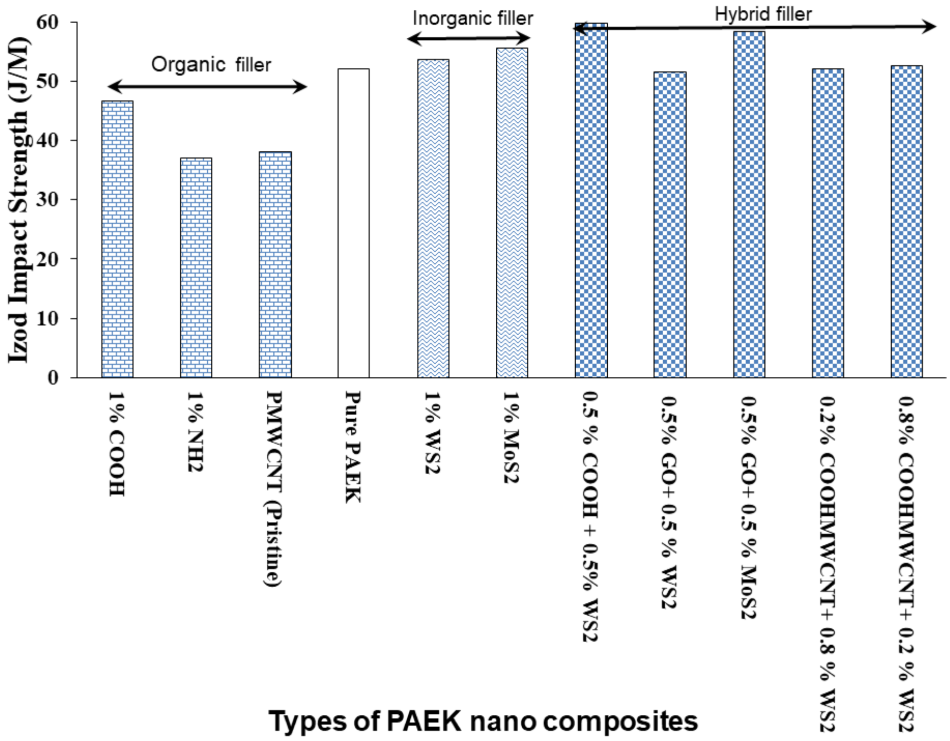

Figure 4 Effect of nano filler on Impact strength of PEEK nano composite

2.4 Microstructural (SEM) analysis

When the multiwall carbon nanotube, in-organic nanofiller and organic nanofiller were added to the base material of PAEK then morphology of nano-composites analysed by Scanning Electron Microscopy (SEM). The impact tested fractured samples have been used for SEM analysis. The SEM images of PAEK nano-composites as shown in Fig. 5. It can be observed from SEM images that the more homogenous dispersion is observed where the combination of organic and inorganic fillers is used. Fig 5 (a) shows SEM of impact tested specimen of pure PAEK. The Fig 5 (a) depicts the co-continuous morphology and smooth surface without any defects in pure PAEK

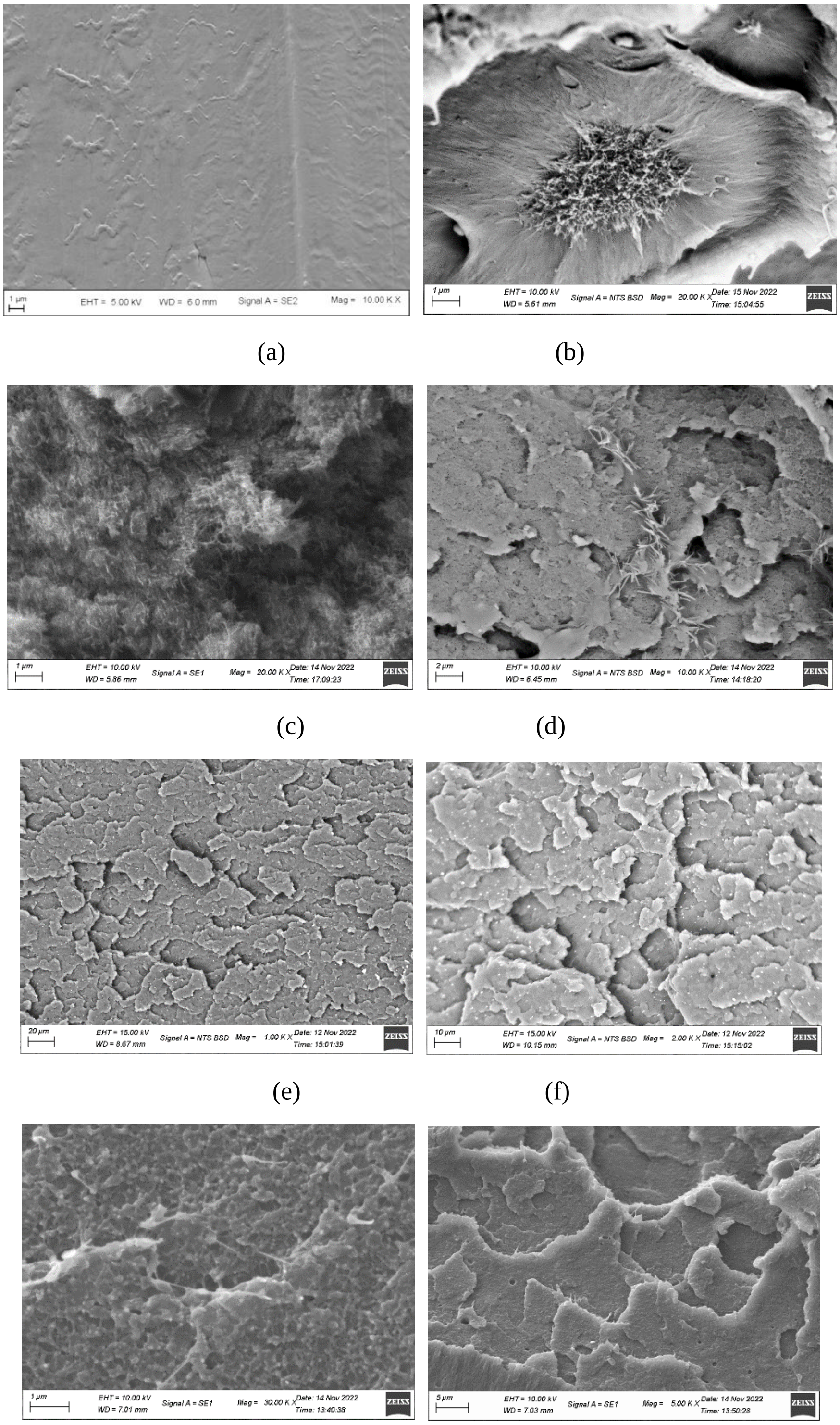

Figure 5 SEM analysis of PAEK composite

Fig 5 (b), (c) and (d) show SEM of PEK reinforced with MWCNT, NH_2 and COOH nanocomposite where agglomeration of MWCNTs is clearly seen. This may happens because of strong agglomeration tendency of MWCNTs due to Vander Walls forces of attraction. In order to reduce these forces, the modified MWCNTs in combination with inorganic nanofillers like WS_2 and MOS_2 are planned to reinforce in PAEK.

Fig. 5 (e) and (f) depicts incorporation of WS_2 and MOS_2 in pure PAEK, both the inorganic nanofillers show homogenous dispersion in polymer matrix. Fig. 5 (g) and (h) depicts incorporation of WS_2 and MOS_2 along with COOH MWCNT and GO in pure PAEK which changes polymer structure significantly. Fig. 5 (g) and (h) depicts that there is more uniform dispersion of hybrid nanofillers results in rough surface that produces good physical bonding in matrix-nanofiller leads to intercalation of nanofiller and polymer. This produces surface to rough with lots of irregularities. As the rough surface produces crack path which when deviated along the original plane that results in increment in crack area which also increases energy for crack propogation, shows more mechanical strength in hybrid nanocomposite compared to pure PAEK

3. Conclusions

In this experimental study successful incorporation of the organic and inorganic nanofiller along with combination of organic and inorganic nanofiller i.e hybrid nanofiller in pure PAEK at room temperature is carried out and examined. The uniform dispersion of hybrid nanofiller in base material is observed and the behaviour of tensile, compression, flexural properties of nano-composite is compared with organic and in-organic filler under various percentage of filler. Organic filler shows lower tensile properties compared to hybrid nano filler. With 0.5 wt% of MWCNT in combination with 0.5 wt% of WS2 as hybrid nanofiller shows higher overall mechanical properties due to uniform dispersion of MWCNT into PAEK using melt compounding process. As there is increase in the percentage of MWCNT in PAEK matrix, that decreases the material properties due to accumulation of MWCNT which results in poor load transfer capacity. In case of in-organic nanofiller WS_2 shows better material properties at 1 wt% loading but in order to take the advantage of excellent mechanical properties of organic filler MWCNT, the combination of both i.e organic and inorganic nanofiller as hybrid nanofiller is superior.

The experimental result suggest that COOH modified MWCNT in combination with WS_2 shows better material overall properties as compared to MWCNT and modified MWCNT used individually at same filler weight persantage.

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