

Comparative Study of Mechanical Properties of Multiwall Carbon Nanotubes and Functionalized Multiwall Carbon Nanotubes/Poly Aryl Ether Ketone Nanocomposites

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Abstract: The present study deals with the effect of Multiwall Carbon Nanotube (MWCNT) and its functionalized (carboxyl and amine) MWCNT on the mechanical properties of Poly Aryl Ether Ketone (PAEK) polymer composites. The incorporation of nanomaterials into polymer matrices has garnered considerable attention due to the potential for enhancing material performance. The existing literature lacks in providing a thorough understanding of the intricate impacts of MWCNTs on PAEK composites, motivating the need for this research.

The study examines the varied concentrations of MWCNT and functionalized (carboxyl and amine) MWCNT as 0.25, 0.5 and 0.75 weight percentages. These composites were prepared using a twin-screw extruder via the melt compounding method. The specimens were meticulously prepared through injection molding, adhering to American Society for Testing and Materials (ASTM) standards. Samples were assessed for tensile strength, impact strength, flexural strength, heat deflection temperature, hardness, and density. The key findings reveal a significant and gradual improvement in tensile strength, impact strength, flexural strength, and heat deflection temperature up to a 0.5% filler loading, followed by a reduction at higher concentrations of filler in PAEK matrices. Remarkably, the most notable improvements are observed with amine functionalized MWCNT. These findings highlight the potential of functionalized MWCNTs in enhancing the mechanical properties of PAEK polymer composites. The implications of this study offer valuable insights for material scientists, engineers, and industries aiming to enhance the performance of PAEK-based materials. The identified results provide advanced polymer composites for specific applications.

Keywords: Poly Aryl Ether Ketone, Multiwalled Carbon Nanotube, Functionalized Multiwalled Carbon Nanotube, Nanocomposites, Fourier transform infrared spectroscopy.

1. INTRODUCTION

PAEK-based polymer composite materials (PCMs) have gained the attention by many researchers due to their various unique properties like lightweight, corrosion resistance, chemical resistance, etc. The PAEK-based polymer composites are very useful for industrial and automotive applications to manufacture gears, bearings, seals etc.

The molecular chain-ring structure containing ether and ketone in the PAEK chain, coupled with its semi-crystalline thermoplastic nature, confers chemical inertness, biocompatibility, and thermal stability [1, 2]. When compared, the well-known high-performance polymer Poly Ether Ether Ketone (PEEK) exhibits higher structural strength and stiffness. Meanwhile, PAEK is acknowledged for its superior mechanical properties than PEEK. The use of PAEK in additive manufacturing facilitates the development of products with intricate customized structures and functionalities

[3]. Low wear resistance and higher thermal expansion of PAEK prevent its application in various demanding areas like subsea connectors, bio-implants and high-performance composites. Increases in hardness, impact strength and storage modulus have been reported with the addition of boron carbide and multiwalled carbon nanotube in PAEK [4, 5]. A polymer composite was formed through the combination of PAEK with glass fiber as a reinforcement and graphite as a lubricant. The investigation revealed that the composites with the small particle size exhibited superior properties [6]. The incorporation of hexagonal boron nitride (hBN) and thermo-graphite in polyether ketone (PEK) and PAEK has shown noteworthy enhancement in their tribological properties [7–9]. The synergistic effect of these additives has contributed to the formulation of composite materials that exhibit superior and more desirable characteristics in frictional behavior and wear resistance properties. Mica is a naturally occurring mineral with inherent

strength, durability and suitable filler for effective reinforcement in the polymer matrix. The mica in the PAEK composites has proven to be a judicious enhancement in their mechanical properties [10]. There is an increase in the interfacial interaction and bonding with the addition of carbon fiber in PEEK polymer [11]. The incorporation of montmorillonite (MMT) clay into PAEK resulted in enhanced mechanical properties and it positively influenced the material's structural characteristics, such as improvements in its overall strength, durability, and other mechanical attributes [12]. The introduction of aluminum oxide (Al_2O_3), aluminum nitride particles (AlNp), and phenyl trimethoxy silane treated silica oxide (SiO_2) into PEEK resulted in an improvement in thermal stability, crystallinity, and storage modulus of the material, indicating improved stiffness and mechanical strength.

The incorporation of these specific fillers demonstrates a synergistic effect on PEEK, reinforcing its thermal and mechanical properties for applications requiring enhanced performance under challenging conditions [13–15]. The addition of nano-silicon dioxide and modified graphene oxide into the PEEK polymer composite led to a substantial improvement in its mechanical properties. This composite formulation demonstrates a promising avenue for advancing the mechanical capabilities of PEEK [16]. PEEK, PEK, and Poly Ether Ketone Ketone (PEKK) polymer materials are extensively studied by scientists due to their outstanding material properties. The incorporation of hBN and basalt fibers into PAEK has resulted in an enhancement of the material's performance with a small amount of these fillers. The addition of hBN contributes to improved thermal conductivity and lubricity, while basalt fibers showed improved strength and stiffness [17, 18].

Carbon nanotubes, because of their inherent advantages such as high aspect ratio, low density and superior mechanical and thermal properties are suitable fillers for enhancing the properties of polymer composites [19, 20]. MWCNT has several concentric nanotubes nested within one another. It has very good thermal, electrical and mechanical properties [21]. The remarkable dispersion effect achieved by incorporating PAEK-grafted-MWCNTs filler into the PEEK material improved the results on electromagnetic shielding and conductivity in PAEK-grafted-

MWCNTs/PEEK PCMs [22]. PEEK composites were developed by incorporating MWCNTs as conductive fillers and showed improvement in electrical conductivity, mechanical properties, and thermal stability [23]. The study of injection molding-based foaming process of polyamide 6 (PA6)/MWCNTs nanocomposites, exploring morphological properties, tensile strength, hardness, mean cell size, and density, and reveals successful development with well-dispersed MWCNT, improved microcellular structure and it showed notable enhancements in strength [24, 25].

The synergistic effect of an MWCNT/ nanoclays hybrid was investigated for high-impact polystyrene (HIPS) nanocomposite foam material, resulting in enhancement in glass transition temperature and improvements in cell density, showcasing enhanced properties compared to individual MWCNT and nanoclays [26]. Nanocomposite foams polymeric material, specifically acrylonitrile butadiene styrene (ABS) and MWCNT, showed a remarkable reduction in skin depth, leading to microwave absorption enhancement and it was made superior by foaming and adding MWCNT in ABS polymer matrix material [27]. Chemical treatment is an emerging technique to achieve good dispersion of carbon nanotubes and the interfacial adhesion between the polymer matrix and MWCNT. This process not only fosters better dispersion but also plays a pivotal role in enhancing overall interfacial adhesion [28–30].

Nevertheless, there is a gap in available studies on the investigation of modified nanomaterials such as MWCNT for the development of PAEK-based nanocomposites. The current study focuses on the synthesis and assessment of the tensile strength, impact strength and flexural strength are the mechanical properties of PAEK-based PCMs including MWCNT and functionalized (carboxyl and amine) MWCNT nanofiller in different concentrations. It also includes the study of density, hardness, heat deflection temperature; Fourier transforms infrared spectroscopy (FTIR) and scanning electron microscope (SEM) images of these PCMs. The functionalized MWCNT has additional functionality including dispersibility and ability to attach on binding sites and thereby realise enhanced performance of the resulting nanocomposites.

2. EXPERIMENTAL PROCEDURES

2.1. Materials

The PAEK 1200G a semi-crystalline granular polymer material for injection molding process is procured from Gharda Chemicals Ltd, Panoli, Gujarat, India. The density, melting temperature and glass transition temperature of PAEK material are 1.3 g/cc, 152°C, and 372°C respectively. The powdered nanomaterial MWCNT, carboxyl functionalized MWCNT and amine-functionalized MWCNT of surface area 110-350 m²/g was obtained from Adnano Technologies Pvt Ltd, located in Shimoga, Karnataka, India. The outer diameter is 10-30 nm, the inner diameter 5-10 nm and the length is 10 µm with a purity 99%.

2.2. Preparation of Composites

The MWCNT and functionalized MWCNT nanofillers are mixed and dispersed in PAEK using a high-speed mechanical mixer for 15 minutes to get a uniform mixture at concentrations of 0.25, 0.5 and 0.75 wt.% respectively.

Tris Nonylphenyl phosphite (TNPP) of 0.02 wt.% was incorporated as an antioxidant. The compounding was carried out with a twin-screw extruder having a screw diameter of 26 mm and L/d ratio of 40:1. The temperature zones of the twin screw extruder varied from 380°C to 400°C and the screw speed was 300 rpm. The strands were prepared via extrusion and chopped by cutter and samples were prepared in granular form. Preheating of materials at 220°C temperature for 3 hours, was carried out before injection molding to eliminate moisture content and ensure the mixture was thoroughly dried.

The injection molding machine (Make-R.K. Techniques, Mumbai, India, 40 tonnage capacities) was utilized to prepare testing samples, using granular PAEK and its composite materials, as per ASTM standards (fig. 1). The injection time was set at 10 sec and holding time was 8 sec.

The temperatures of injection molding machines were set at 380°C, 385°C, 390°C and 400°C for temperature zone1, zone 2, zone 3, and zone 4 respectively, spanning from feed zone to nozzle and the mold temperature was maintained at 400°C temperature.

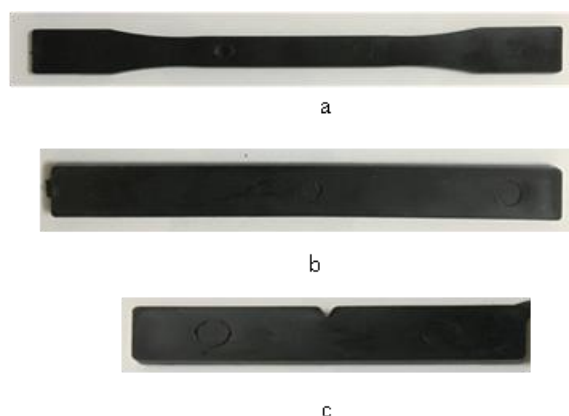


Fig. 1. Injection molded samples for (a) tensile testing, (b) flexural and HDT testing, (c) impact tests.

2.3. Characterization

The samples were analyzed for tensile properties such as tensile strength and elongation at break utilizing a universal testing machine (UTM), with a cross-head speed set at 50 mm/min, adhering to the ASTM D-638 standards. The impact strength was assessed on the impact tester (Make-International Equipments, Mumbai, India) using notched specimens following the ASTM D 256 standards. The test involved maintaining a 22 mm distance for the hammer to the pendulum, positioned above the clamping vice. The flexural strength was measured using a universal testing machine (LR50K, Lloyd Instruments UK) according to ASTM D790. Durometer -Shore D hardness tester, DIN-53505 was used for hardness testing and tests are taken as per ASTM D2240-05 standards. The heat deflection temperatures of samples were obtained by placing the 150 gm weights on the upper end of the loading rod to achieve significant stress in the samples using heat deflection temperature equipment (Make-International equipments, Mumbai, India). The density of the composites was determined using the standard ASTM D792 method, employing Archimedes' principle and a digital weighing balance machine. Five test samples of each composite were examined, and the average value of these results was taken into consideration. SEM analysis was carried out on the Zeiss Supra 55 VP SEM machine. The digitized images of the samples were recorded and studied.

3. RESULTS AND DISCUSSION

3.1. Tensile Properties

Fig. 2 shows the variation in tensile strength of MWCNT and functionalized (carboxyl and amine) MWCNT reinforced PAEK composites. The tensile strength increases with the addition of nanofillers up to a loading of 0.5 wt%, followed by, the tensile strength experiencing a decline with a further enhancement in nanofiller loading. The Increase in the tensile strength can be linked to the reinforcing effect of MWCNT in the polymer composites, due to its excellent tensile strength, stiffness, and thermal properties etc. [31, 32]. Also, there is strong adhesion of functionalized MWCNT nanofiller with PAEK polymer having a high ratio of area to volume and it contributed to excellent interfacial interaction and bonding between MWCNT and PAEK composites [33–35]. For higher loading of nanofiller, there is a decrease in the tensile properties of PCMs due to agglomeration [36]. Hence, functionalized MWCNT and PAEK composites showed better tensile strength than MWCNT and PAEK composite materials.

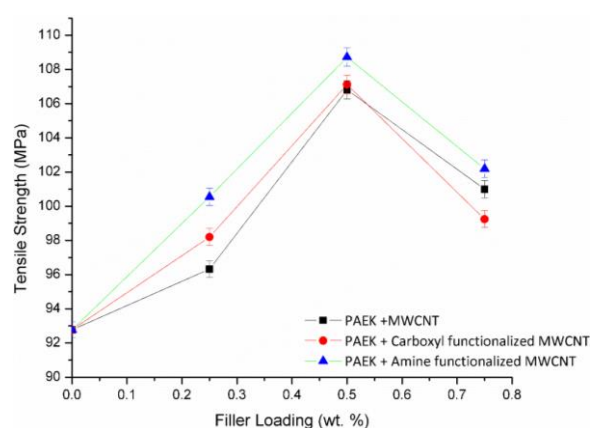


Fig. 2. Plot of Tensile strengths vs. filler loading for PAEK based PCMs.

3.1.1. Elongation at break

Fig. 3 illustrates the variation in elongation at break (%) of MWCNT and functionalized MWCNT reinforced PAEK composites. The elongation at break increases with the addition of nanofillers up to a loading of 0.5 wt%, subsequently, there is a reduction in it with a further increase in nanofiller loading. There is an enhancement in the elongation at break due to the ductile nature of composites at lower nanofiller loading and a decline in the elongation at break at higher filler loading can be attributed to an increase in brittleness with further addition of

nanofillers [37, 38]. Also, the presence of MWCNT nanofiller restricts the segmental mobility which adversely affects the elongation behavior of polymer matrix material at higher nanofiller loading [39].

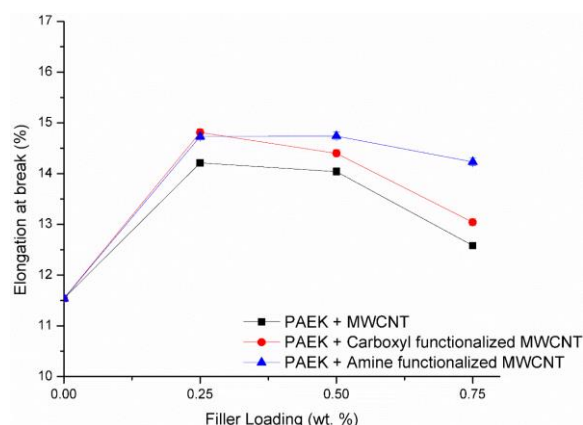


Fig. 3. Plot of Elongation at break vs. filler loading for PAEK based PCMs.

3.2. Impact Properties

Fig. 4 illustrates the variation in impact strength of MWCNT and functionalized MWCNT reinforced PAEK composites. It showed enhancement in impact strength with an increase in nanofiller loading up to 0.5 wt.% loading, subsequently, there is a reduction in it with a further increase in nanofiller loading. Increase in the impact strength can be linked to better interfacial bonding between matrix material, nanofiller and uniform dispersion of nanofiller in composite material at lower filler loading. The nanofiller such as MWCNT interaction with the matrix allows the composites to absorb the fracture energy and thus it contributes to enhancing the impact strength [40]. Functionalized MWCNT has excellent interfacial bonding at lower wt.% loading of nanofiller due to surface functionality [32]. Hence, there are better impact properties at 0.5wt% functionalized nanofiller loading in PAEK composites than pristine MWCNT reinforcement in PAEK composites.

However, there is a decrease in impact strength at higher concentrations, which can be attributed to an increase in brittleness and compromising the toughness [41]. Inadequate adhesion of nanofiller at high concentration and possible agglomeration of high surface area MWCNT also act in the direction of reduction of impact strength.

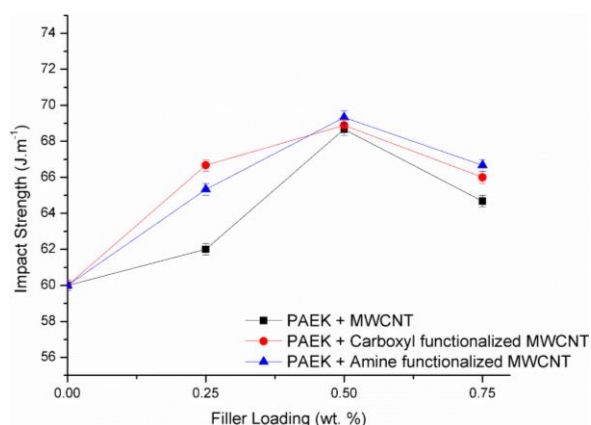


Fig. 4. Variation of Impact Strengths as a function of filler loading for PAEK based PCMs

3.3. Flexural Properties

Fig. 5 illustrates the variation in flexural strength of MWCNT and functionalized MWCNT reinforced PAEK composites. The enhancement in flexural strength with an increase in nanofiller loading up to 0.5 wt.% loading, subsequently, a slight decrease in it with a further increase in nanofiller loading. The increase in flexural strength can be attributed to strong interfacial bonding between PAEK and functionalized MWCNT, resulting in better stress transfer [28]. The decrease in the flexural strength at higher filler loading can be linked to the probability of agglomeration of nanofiller at higher weight fraction [39].

The MWCNT and functionalized MWCNT incorporation increases the flexibility of polymer composites up to a certain concentration of nanofiller loading, then decreases due to its brittle nature at higher nanofiller loading [37, 42].



Fig. 5. Variation of Flexural Strengths with filler loading for PAEK based PCMs

3.4. Hardness

Fig. 6 illustrates the variation in hardness values of MWCNT and functionalized MWCNT reinforced PAEK composites. The enhancement in hardness with an increase of nanofiller loading up to 0.5 wt.% loading, subsequently, decrease in it with further increase in the loading. Due to the surface modification of functionalized MWCNT nanofiller a better dispersion is obvious that results in higher hardness values for functionalized MWCNT reinforced PAEK composites than MWCNT reinforced PAEK composite material. The presence of MWCNT enhances the toughness of composites hence it contributes to enhancing the mechanical properties of composites at lower nanofiller loading [31]. The hardness values of composites are impacted by nanofiller void content, weight fraction, and interfacial bonding between nanofiller and polymer matrix [43].

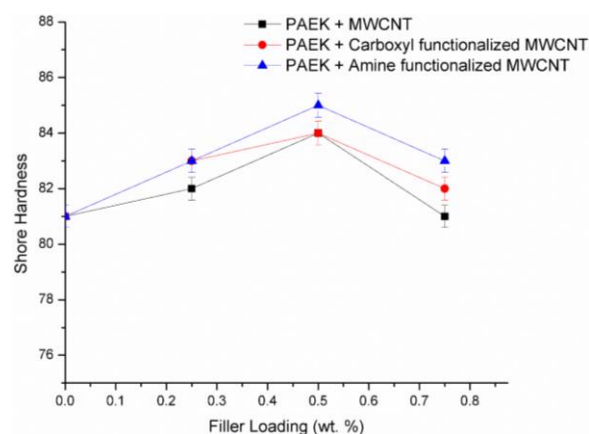


Fig. 6. Variation of Hardness of PAEK based PCMs with filler loading

3.5. Heat Deflection Temperature (HDT)

Fig. 7 illustrates the variation in heat deflection temperatures of MWCNT and functionalized MWCNT reinforced PAEK composites. There is an enhancement in heat deflection temperatures with an increase in nanofiller loading up to 0.5 wt.% loading, as the addition of MWCNT, improves the stiffness and rigidity and stiffness of polymer composite [44]. There is a decrease in heat deflection temperature at higher filler loading.

3.6. Density of Composites

Fig. 8 shows the variation in the density values of MWCNT and functionalized MWCNT reinforced

PAEK composites as a function of the incorporation of filler. The composite density depends on the proportionate content of the matrix and nanofiller material [45]. As expected there is also an increase in density with an increase in nanofiller loading. The maximum increase in density is observed for the amine-functionalized MWCNT-incorporated PAEK matrix.

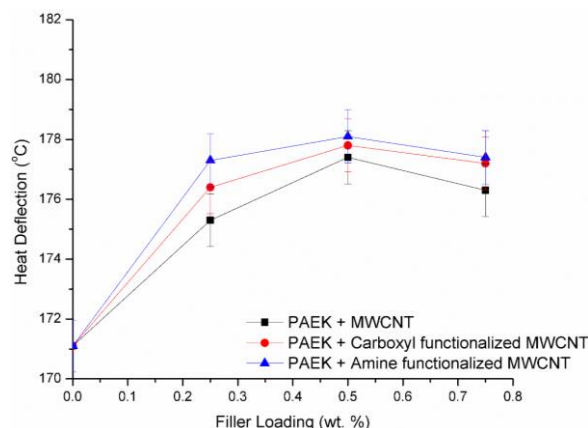


Fig. 7. Variation of Heat deflection temperatures of PAEK based PCMs with filler loading.

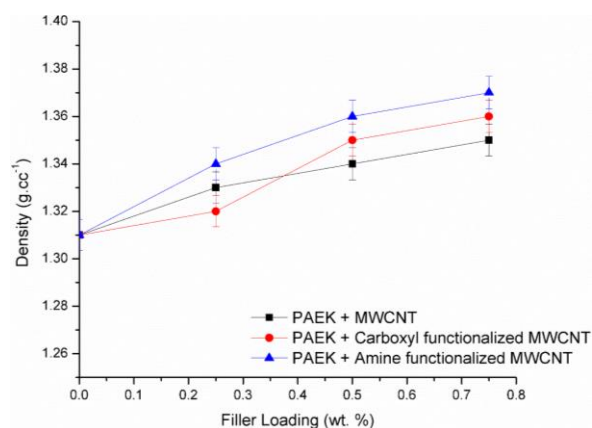


Fig. 8. Variation of Densities of PAEK based PCMs with filler loading

3.7. Fourier transforms infrared spectroscopy

A representative FTIR spectrum is shown in fig. 9. The major peaks present in the FTIR spectra of PAEK can be seen at 1648, 1585 and 1305 cm^{-1} , respectively. This can be attributed to the stretching of the C=O bond, benzene ring plane R–O–R, and stretching vibration spectrum of R–CO–R benzene ring. The CH bending of the aryl ketone structure in the plane of the benzene ring is observed at 1158 cm^{-1} . The 925 cm^{-1} peak is observed due to symmetric stretching of

R–CO–R [46].

The carboxylated MWCNTs show four major peaks, at 3728, 3425, 2361, and 1560 cm^{-1} , respectively. The peaks at 3728, 3425, and 2361 cm^{-1} are associated with free hydroxyl groups, stretching of –O–H associated with carboxyl groups and the O–H stretching originates from the present strongly hydrogen-bonded –COOH group respectively [47]. The peaks are low in intensity in the presence of major peaks of PAEK in the nanocomposite, because of the low concentration of modified MWCNT, which are modified to a very low extent. The peak at around 1560 cm^{-1} , assigned to carboxylate ions, is merged with the 1585 cm^{-1} peak of PAEK.

Similarly, the amine-functionalized MWCNT is associated with FTIR peak at 3290, 1293, and 1063 cm^{-1} assigned as –NH stretching and –C–N stretching respectively. However, with the same argument, the peaks are very small, and some remain merged with the PAEK peaks.

3.8. Scanning Electron Microscope Images

The SEM images were recorded for the fractured interface of the nanocomposite samples. Fracturing was achieved by immersion in liquid nitrogen, followed by breaking and utilizing the interface for SEM scanning. It is evident from the SEM images that there are agglomerates in PAEK/MWCNT nanocomposite indicating poor distribution of nanotubes. The carboxyl modification is found to have maximum dispersion with traces of agglomerate. With amine modification, the improvement in a dispersion of modified MWCNT is improved. This result directly influences the properties of the nanocomposites.

4. CONCLUSIONS

There is an increase in tensile strength, elongation at break, impact strength, flexural strength, hardness, and heat deflection temperatures up to 0.5 wt% nanofiller loading in PAEK, followed by a decrease with higher nanofiller loading.

The increase in mechanical property was maximum for amine functionalized MWCNT reinforced PAEK based PCM, and it was higher at any given nanofiller loading in comparison to MWCNT and carboxyl functionalized MWCNT reinforced PAEK composites.

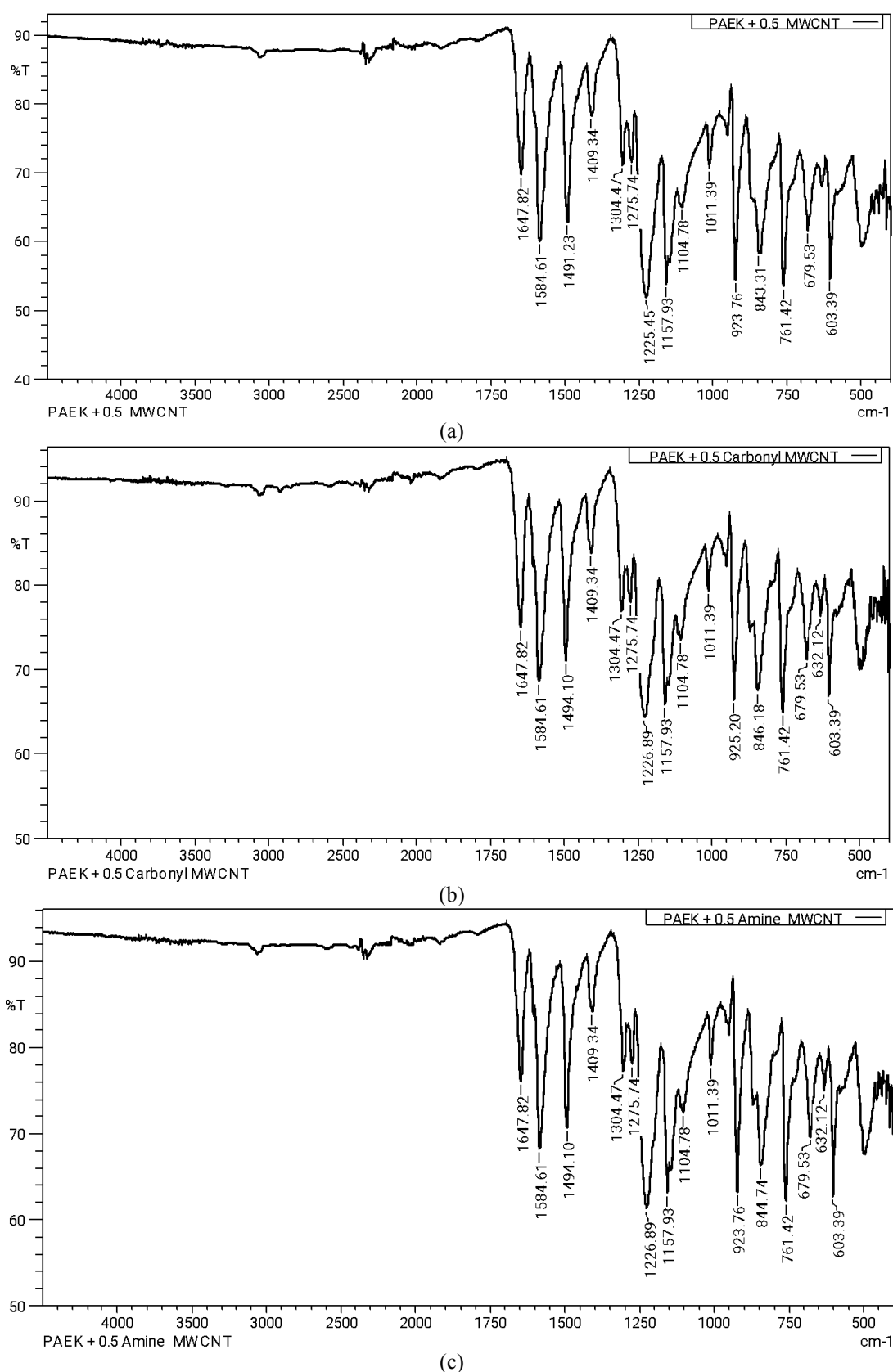


Fig. 9. FTIR spectroscopy results: PAEK and 0.5% MWCNT PCM FTIR spectroscopy image (a); PAEK and 0.5% carboxyl functionalized MWCNT PCM FTIR spectroscopy image (b); PAEK and 0.5% amine functionalized MWCNT PCM FTIR spectroscopy image (b)

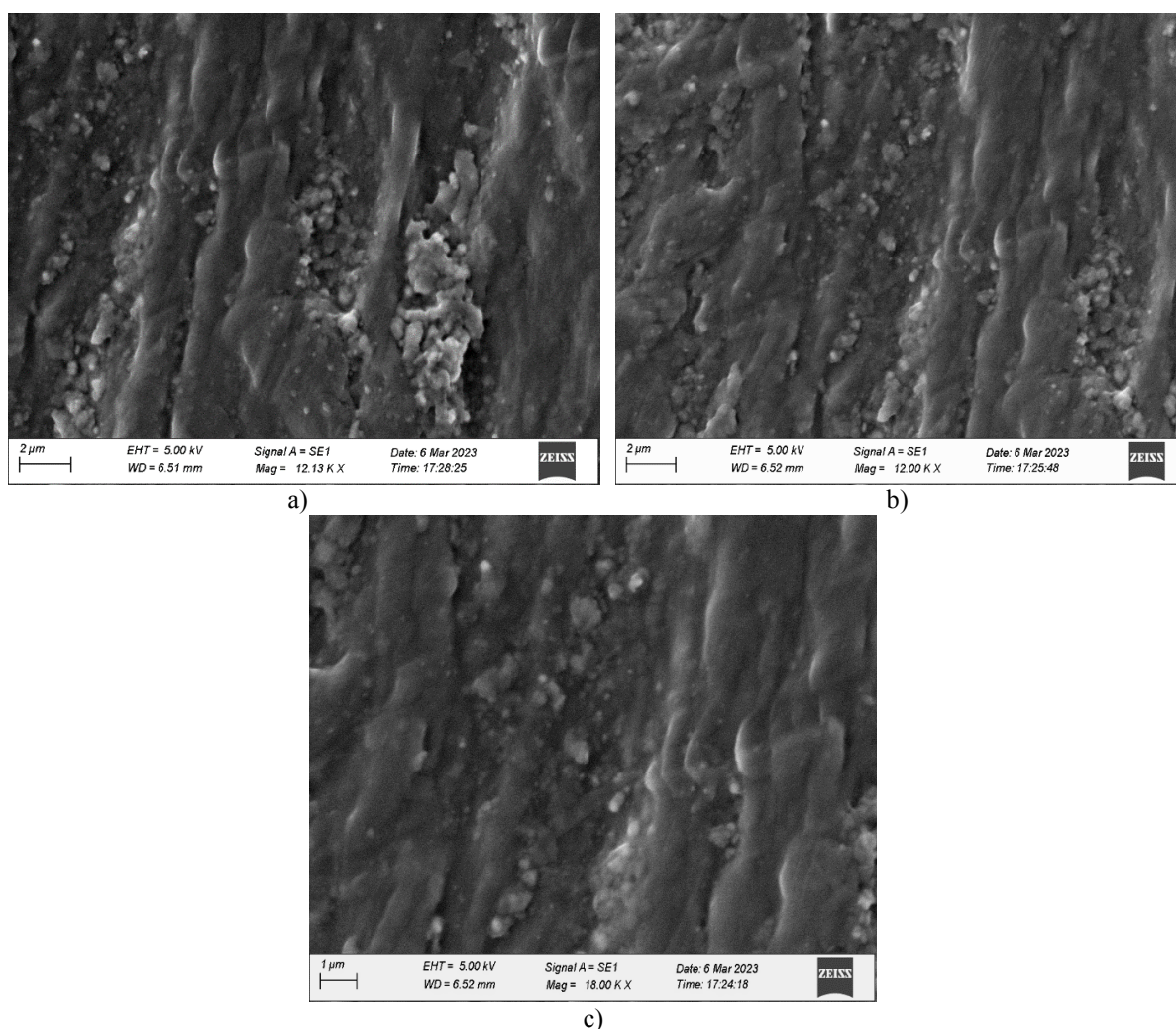


Fig. 10. SEM images: PAEK and 0.5% MWCNT PCM SEM image (a); PAEK and 0.5% carboxyl functionalized MWCNT PCM SEM image (b); PAEK and 0.5% amine functionalized MWCNT PCM SEM image (c)

Functionalized MWCNT-reinforced PAEK composites showed better mechanical properties than non-functionalized MWCNT-reinforced PAEK composite materials. As the nanofiller concentration increased in the PAEK matrix, density values also increased. With the incorporation of 0.5 wt.% amine functionalized MWCNT in PAEK, increments in tensile strength were observed up to a maximum of 17.19%, elongation at break up to 27.72%, impact strength up to 15.55%, flexural strength up to 22.75% and heat deflection temperatures up to 4.09%, respectively.

COMPETING INTEREST

The author(s) declared no potential conflicts of interest concerning the research, authorship, and/or publication of this article.

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