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Fabrication of Conductive Polymer Fiber Composite for Effective Transmission of Electromagnetic Wave in the Porous Medium

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Abstract

Interest has been generated in the use of conductive polymer fibers composite because of its environmentally kindly and conductive properties. PVDF is normally used as insulation on electrical wires, because of its blend of flexibility low thermal conductivity, low weight, high chemical corrosion resistance, and heat resistance. PVDF is a specialty plastic used in applications requiring the highest purity, as well as resistance to solvents, PVDF polymer composite filled with synthesis of MWCNT were fabricated by spinning techniques, conducting polymer composite is extensively demanded in advance application most especially in electromagnetic wave propagation. This research focus on the enhancement of polymer carbon fiber with thermal stability for better performance in electromagnetic wave propagation in the porous medium. The thermal stability of polymer composite properties was measure and evaluated, morphology of the fabricated polymer fiber was studied via field TEM, Emission scanning Electron Microscope (FESEM), it was revealed from the stimulation that P orbital above the fermi level for MWCNT is from 0 to 16eV and for PVDF+MWCNT is from 0 to 19eV. It was revealed from the result that incorporation of Carbon-based material increases the thermal stability of the material which improved its performance for all application. From the result obtain it was discovered that electric permittivity of polymer carbon fiber increased from 0.6×10^{10} at to 9×10^{10} (S/m).

1. Introduction

The conductivity of electrical conducting polymer result from mobile charge carriers introduced into the π -conjugated system that is formed by the continuous overlap of extended and delocalized P-orbitals along the polymer chain's backbone. However, conducting polymers without additive generally exhibit very low conductivity at room temperature. Their conductivity can be varied by adding an additive to change the charge carrier density on the polymer backbone. It has been documented that the conductivity of those polymers ranges from 10^{-10} S/cm (an insulator) to about 10^{-5} S/cm (a semiconductor) to greater than 10^4 S/cm (a metal), depending on the additive concentration. Hence, it

is imperative to know why conductive polymers can alternatively behave as a metal, semiconductor, or insulator [1-3]. Since the discovery, CNTs became the first choice in many applications [4, 5]. They exhibit great potential, and their possible uses are constantly expanding due to their very large aspect ratio, high rigidity, and high resistance to traction, virtual absence of thermal expansion, excellent electrical conductivity, and good thermal conductivity. These characteristics make them compellingly useful in the preparation of nanocomposites [5]. Electromagnetic (EM) waves entering a shielding conductor attenuate exponentially. The depth at which the EM field decreases to 1/e of the incident value is called the skin depth (δ), and for highly conductive materials, such as metal, it is given by equation (1) [6].

$$\nabla \times \bar{H} = \bar{J} + \epsilon \frac{\partial \bar{E}}{\partial t} \quad (1)$$

At a specific frequency, high conductivity and permeability are imperative for well shielding. Thus, metal has been frequently used for electromagnetic interference (EMI) shielding due to its high conductivity. Carbon based materials such as graphite, carbon fiber, carbon black [5-7] and conducting polymers [8-10] are good candidates due to their virtual high conductivity and lighter weight, as well as their application to traditional fabrication processes. Carbon nanotubes (CNTs) are a promising EMI shielding candidate [11-17] due to their lightweight, high conductivity, and exceptionally high mechanical properties. In the case of highly conductive single-walled carbon nanotube (SWCNT) film, 10 nm thicknesses gives more than 20 dB at 10 GHz and over 50 dB can be easily achieved over 10 μ m thickness [12]. It was recalled that high aspect ratio of CNT filler has advantage compared to carbon nanofiber and carbon black in term of conductivity due to their lower percolation limit in polymer composite. In the EMI to maximize the electrical conductivity and mechanical properties, higher CNTs loading in electromagnetic interference shielding is preferred. Nevertheless, most of CNT composite with polymer matrix has maximum loading around 40 wt.% [13]. Higher loading will decrease the mechanical properties due to agglomeration. Unlike other CNT composites, Poly (vinylidene fluoride) (PVDF) is a fluoropolymer comprising of the monomer unit CF_2-CH_2 . Additionally, it is a polymorphic, semi-crystalline polymer showing at least four crystal phases at different processing conditions [1,2]. It has an exceptional chemical stability and a large dipole moment of 9.8×10^{-30} cm, perpendicular to the polymer chain [3,4]. This results in piezo-, pyro-, and ferroelectric characteristics due to a polar crystal phase it can build. This phase is the β phase with an all-trans conformation in orthorhombic unit cells. It can be formed from the non-polar α phase. The most stable α phase forms from melt crystallization at temperatures below 160°C and can be transformed into the β phase by application of mechanical stress at temperatures below 100°C [2]. PVDF polymer composites can achieve a high concentration of CNTs and high conducting nanotube networks to further improve EMI shielding effectiveness (SE). SE is the sum of the effectiveness of all attenuating mechanisms and EM losses measured in decibels (dB) and expressed by equation (2) [18].

$$\nabla \times \bar{E} = -\mu \frac{\partial \bar{H}}{\partial t} \quad (2)$$

$$\bar{J} = \sigma \bar{E} \quad \sigma: \text{Conductivity } (\Omega/m)$$

$$\nabla \times \bar{H} = \sigma \bar{E} + \epsilon \frac{\partial \bar{E}}{\partial t} \quad (3)$$

Conducting polymers have gained a significant attention over the last epochs by researchers for their strong potential as alternatives to their inorganic counterparts; different researchers considered

conducting polymers in the late 1970s to be intractable and insoluble, when piezoelectricity of Polyvinylidene fluoride (PVDF) was discovered by Dr. Heiji Kawai in 1970. Conducting polymer have been studied continuously including polypyrrole (PPy), polyaniline (PANI), polythiophene (PT), poly(3,4-ethylenedioxythiophene) (PEDOT), trans-polyacetylene, and poly(p-phenylene vinylene) (PPV) [19]. In general, CPs possesses alternating single (σ) and double (π) bonds, and these π -conjugated systems lend the CPs their inherent optical, electrochemical, and electrical/electronic properties. It is known that the parameters that most affect the physical properties of CPs are their conjugation length, degree of crystallinity, and intra- and inter-chain interactions [20]. Conductive polymers composite is extensively demanded in advance application most especially in electromagnetic wave propagation. As effective nanoscale reinforcement, carbon nanotubes (CNTs) have attracted great interests in the field of conducting polymer nanocomposites. These nanocomposites should possess good mechanical properties, excellent electrical and thermal conductivities, which are considered useful attributes for many applications in the electronics industry. However, the high aspect ratio and the flexibilities of CNTs along with the van der Waals forces between them cause CNTs to be severely entangled in close packing upon synthesis. Furthermore, the chemically inert nature of CNTs leads to poor dispensability and weak interfacial interactions with polymer fibre. In this study, conducting carbon nanotube polymer fiber composite was design, characterize and analyse for effective propagation of electromagnetic wave in the porous medium.

2. Electromagnetic Wave Equation of a Conducting Material

Conductive polymers or, more precisely, intrinsically conducting polymers (ICPs) are organic polymers that conduct electricity [21]. Such compounds may have metallic conductivity or can be semiconductors. The biggest advantage of conductive polymers is their process ability, mainly by dispersion. Conductive polymers are generally not thermoplastics, *i.e.*, they are not thermoformable. But, like insulating polymers, they are organic materials. They can offer high electrical conductivity but do not show similar mechanical properties to other commercially available polymers. The electrical properties can be fine-tuned using the methods of organic synthesis [22] and by advanced dispersion techniques [23]. Below equation will illustrate electromagnetic wave equation in a conducting medium.

$$\nabla \times \nabla \times \bar{E} = -\mu \frac{\partial}{\partial t} (\nabla \times \bar{H}) = -\mu \frac{\partial^2 \bar{E}}{\partial t^2} - \mu\sigma \frac{\partial \bar{E}}{\partial t} \quad (4)$$

$$\nabla \times \nabla \times \bar{E} = \nabla(\nabla \cdot \bar{E}) - \nabla^2 \bar{E} \quad (\text{Vector identity})$$

$$\nabla(\nabla \cdot \bar{E}) - \nabla^2 \bar{E} = -\mu\sigma \frac{\partial \bar{E}}{\partial t} - \mu \frac{\partial^2 \bar{E}}{\partial t^2} \quad (5)$$

$$\text{But } \nabla \cdot \bar{D} = \rho$$

$$\epsilon \text{ is constant, } \nabla \cdot \bar{E} = \frac{1}{\epsilon} \nabla \cdot \bar{D}$$

Since there is no net charge within a conductor the charge density is zero (there can be charge on the surface), we get

$$\nabla \cdot \bar{E} = \frac{1}{\epsilon} \nabla \cdot \bar{D} = 0$$

Then using eqn. (5) we get

$$\nabla^2 - \mu\epsilon \frac{\partial^2}{\partial t^2} = 0 \quad (6)$$

The wave eqn. for the electric field

$$\nabla \times \vec{B} = \mu\epsilon\vec{E} + \mu\sigma\vec{E} \quad (7)$$

By equating $\nabla \times \vec{E} = -\vec{B}$ into equation (7) will arrived at wave equation for electric field in a conducting media

$$\nabla^2 \vec{E} - \mu\sigma\vec{E} - \mu\epsilon\vec{E} = 0 \quad (8)$$

3. Experimental Details

3.1 Chemical and instruments.

Poly (vinylidene fluoride) (PVDF) pellet was purchased from sigma-Aldrich (63103) St. Louis USA with 99% purity without further purification, 1-methyl-2-pyrrolidinone (NMP) reagent plus of 99% purity was also purchased from Sigma-Aldrich international GMBH C/O Aldrich chemical co LLC, 600 North Teutonia Avenue Milwaukee, WI 53209 USA. With Mw: 99.13g/mol, Mp: -24°C (lit), bp: 202°C (lit), bp: 81-82°C/10mmHg (lit), Fp: 1.4 (lit), Vapor pressure: 0.2gmmHg (20°C) and assay 99%, smith electric stirrer and the polymer were used as received [24].

3.1.1 Synthesis of Single and Multi-Wall Carbon Nanotube through CVD

This present study used high temperature technique to synthesis MWCN by dispersion of 2g ferrocene into 500ml of methanol as the feedstock, this was sonicated for about 1hrs before placing it for production figure 1 illustrate the schematic diagram of Chemical Vapor Deposition (CVD) system and figure 2A and B show image of morphology of carbon nanotube.

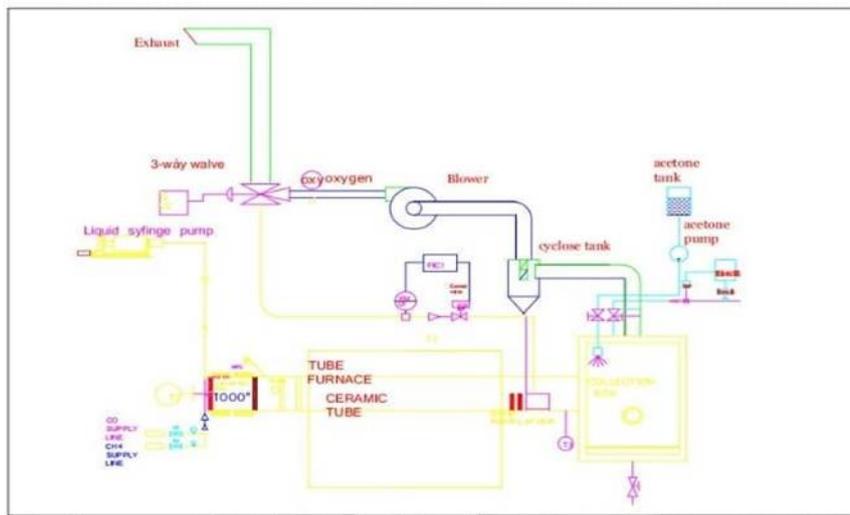


Fig: 1 Schematic diagram of the CVD system.

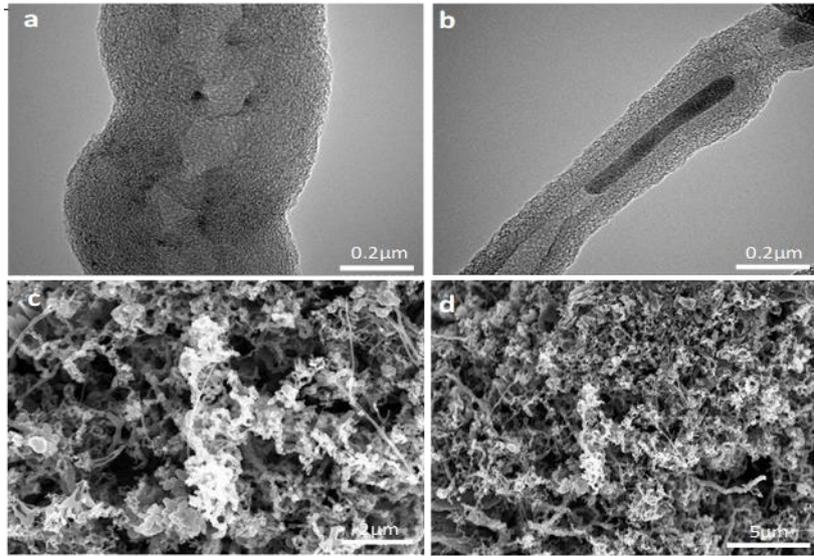


Figure: 2. (a and b) TEM images, (c and d) FESEM of MWCNT

3.1.2 Preparation of carbon nanotube polymer fibre composite

The PVDF polymer fibres were prepared by solvent induced phase separation through the spinning equipment. The PVDF pellet was dissolved in 1-methyl-2-pyrrolidinone (NMP) followed by using electric stirrer at temperature between 60°C – 70°C until the solution became homogeneous. Then 0.5g, 1g, 2g and 3g of MWCNT was dispersed into 20g, 30g and 40g respectively of ethanol and then added into 200ml of polymer dope solution, it was stirred again at 150rpm using electric stirrer and degas for about 5hrs. Spinning solution with different polymer concentrations (20 to 25wt. %) were formulated. All the polymer solution prepared were clear and homogeneous at room temperature. Water was used as the external coagulant; the spinneret with orifice diameter/inner diameter of the tube 0.6mm was used. In spinning polymer fibres, the take up velocity (8-9m/min) and the air gas was in the range of 5 to 25cm. Figure 3 show the formation of the fibre produced with different weight percent.

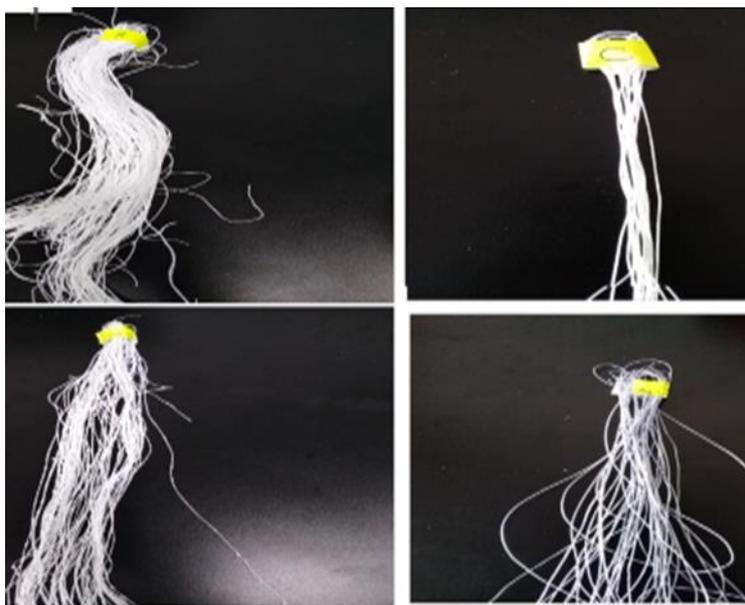


Fig. 3. Fibre orientation with a) 0.05, b) 0.1, c) 0.2 And d) 0.3 weight % of carbon materials.

Table 1: Air gas, pump speed length of extrudent and draw ration of the fiber.

Sample	AG (cm)	Pump Speed (cm/Min)	V1 (cm/min)	V2 (cm/min)	D.R
1	10	16	1061.0331	1697.652959	1.6
2	10	24	1061.0331	2546.479439	2.4
3	10	32	1061.0331	3395.305919	3.2
4	5	16	1061.0331	1697.652959	1.6

Table 2: Dry phase residence and fiber diameter

Sample	T res.(s)	(rpm)	Sample diameter (mm)
1	0.43499	16	0.34
2	0.332639	24	0.3
3	0.269279	32	0.21
4	0.217495	16	0.18

4. Results and Discussion

Because of their light weight, low cost, and oxidation resistance, polymer conductive composites based on carbon fillers (such as carbon black, carbon nanotubes, graphite, graphene, carbon fibre, metal coated carbon fibre, and so on) have been found to be superior to metals in many electrical and electronic applications. These carbon fillers are utilized in the insulating polymer matrix as fibre, flake, particle, and filament to improve the electrical conductivity, mechanical properties, and electromagnetic interference (EMI) shielding efficacy of the composites. Figures 4 and 5 show the band structure and density of state electromagnetic wave propagation versus MWCNT loading (wt. percent) of clean PVDF and PVDF–MWCNT composites.

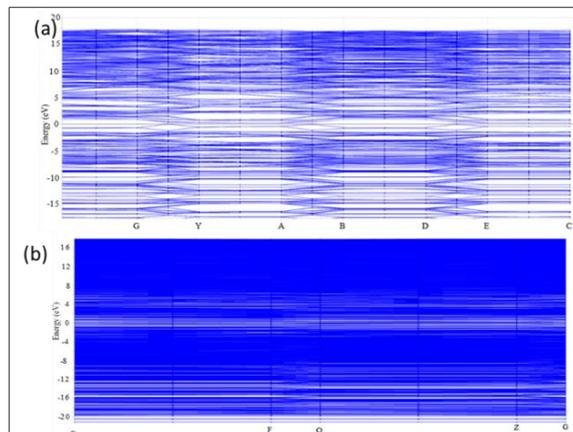


Fig. 4. Band structure of a) MWCNT b) MWCNT with PVDF Polymer.

Figure 4 shows the reaction mechanism of energy band gap between the carbon nanotube without polymer fiber and with polymer fiber it was clear from the result that the band gap of polymer fiber with MWCNT has higher electron volts. According to Fig. 4, the electrical conductivity of composites rises as MWCNT loading increases. The increase in electromagnetic shielding from zero to 0.05 wt. percent MWCNT is minor, whereas a sharp rise in conductivity is seen with a shift in filler content from 0.05 to 0.3 wt. percent. However, a slight increase in DC conductivity is found when the concentration of PVDF–MWCNT composites is increased from 0.05 to 0.3 wt. percent.

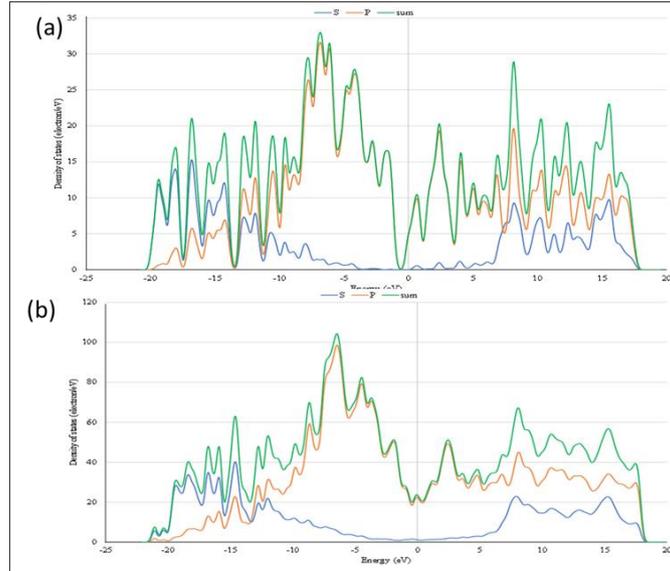


Fig. 5. Partial Density of States (PDOS) of the a) MWCNT b) MWCNT with PVDF Polymer.

As a result, the percolation threshold/limit for PVDF matrix is 0.3 weight percent of MWCNT loading (equal to volume fraction, 0.003). It indicates that at 0.3 wt. percent MWCNT loading, a continuous conductive network of CNTs forms inside the polymer matrix, resulting in a significant increase in composite conductivity (Table 3). However, following the construction of a continuous conducting network, the marginal change in conductivity for an increase in MWCNT from 0.05 to 0.3 wt. percent is seen. This indicates that during percolation, a continuous conducting wire is produced through an insulating polymer matrix to make it conductive; after percolation, only the number of such wires increases.

Table 3: Energy ranges, total orbital and partial density of state for P orbital for MWCN and MWCN/PVDF

<i>Electronic properties of nanomaterial</i>	<i>MWCN</i>	<i>PMWCN</i>
Sum of orbital energy range above fermi level (eV)	-18 to 16	-22 to 19 (eV)
Maxmum sum of orbital of PDOS across fermi level (e's/eV)	21	27 (eV)
Maxmum sum P orbita PDOS across fermi level (e's/eV)	17	27 (eV)
Maximum sum of P orbital above fermi level	0 to 16	0 to 20 (eV)

4.2 Raman Spectroscopy of carbon nanotube polymer fiber

We confirmed that CNTs inserted in polymer fiber may attain several roles, comprising use as enhancement, orientation detectors as well as molecular sensors around structural defects and reinforcements. Raman spectroscopy to carbon nanotube base composite materials is being reported recently for its improvements in the application. This method was used to detect carbon nanotubes, access their dispersion in polymer, evaluate nanotube/matrix interactions and detect polymer phase transitions [25]. Raman spectroscopies provide distinctive information about vibrational and electronic properties of CNTs, it was also used to identify materials through the characteristic's vibrations of certain structures. It can also be used to determine the orientation of CNTs in polymer matrices or within the CNT bundles [25]. From the figure 6, D Raman band of carbon nanotube and polymer fiber composite was identified from 1133 and 1429 cm^{-1} has a linear dependence on laser excitation energy.

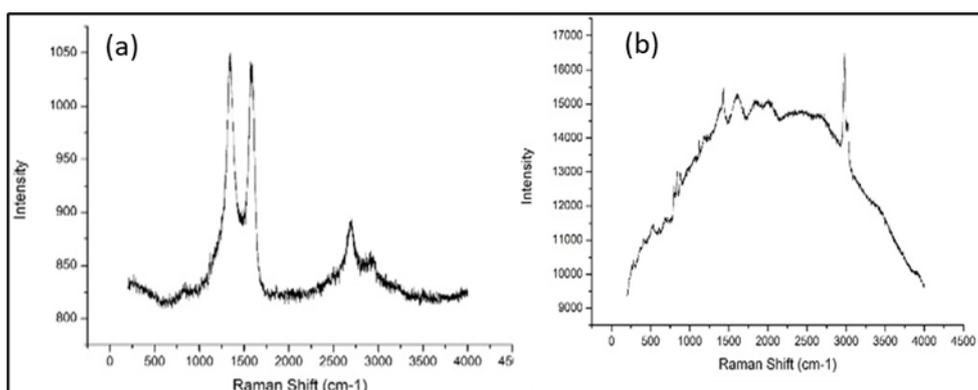


Fig. 6. Raman Spectra of a) MWCNT and b) MWCNT with polymer fiber.

It was stated that this band is activated in the first order scattering process of Sp^2 carbons by the existence of in-plane substitution hetero-atoms, vacancies, grain boundaries or other defects (D stands for defects). The D-band and its second order overtone G' -band (also called the 2D band) are observed in the 1250 - 1450 cm^{-1} and 2500 - 2900 cm^{-1} regions, respectively. The D-band scattering involves one-phonon emission while the G' -band scattering involves emission of two phonons. The causes of these two bands both involve a double resonance Raman process. The D-band scattering consists of one-elastic and one-inelastic scattering process, in which the elastic scattering arises from defects (such as vacancies, impurities and hetero-atoms) in the crystal. On the other hand, the G' -band is due to two-inelastic scattering process, in which the two emitted phonons possess vectors of $+q$ and $-q$, respectively. The momentum constant is therefore automatically preserved in the G' -band scattering and no defect is required to observe the G' -band.

4.3: Thermal stability of the Carbon filler using DSC Analysis

DSC analysis is broadly used for investigation the polymeric material to determine their thermal transition and stability. This is used to compare the transition between materials, although the transition does not uniquely identify the composition. The complementary techniques such as IR spectroscopy may be completed by the composition of unknown materials. For most polymer the melting points and glass transition temperature are available from standards accumulations, this technique can show polymer degradation by lowering of expected melting point [26]. It was observed from the figure 7 that the composite is free of void, also indicate that both the fiber and the matrix behave as perfect linear elastic materials and there is perfect bonding between the fiber and the filler which show the stability of the materials. It was revealed that effect of significant improvement of thermal

stability by incorporation of CNT was investigated in CNT reinforced on the PVDF polymer composite, as increase in CNT contents from 0.05w% to 0.3w% however improvement the thermal stability by incorporating CNT into PVDF polymer. The peak was both endothermic and exothermic

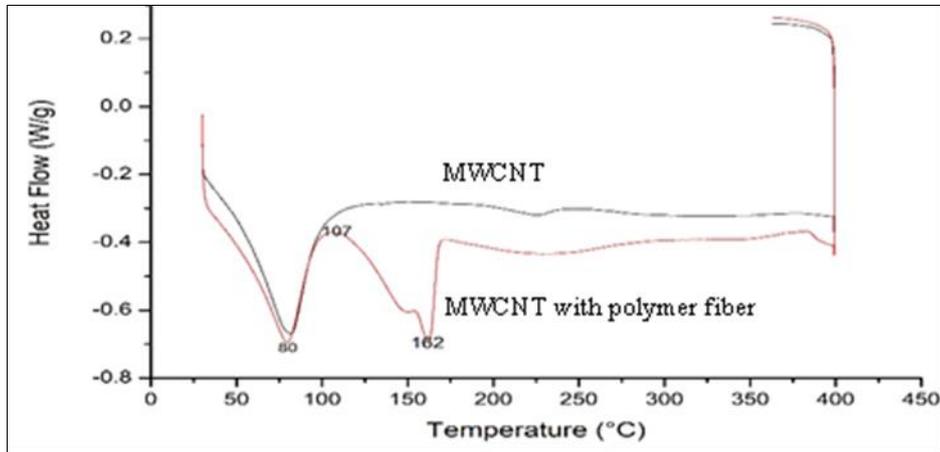


Fig. 7. DSC analysis of MWCNT powder and MWCNT with Polymer fiber.

4.4 In-situ Rheology graph (at 80°C) for four polymer dope solution with different weight percent of CNT

All nanomaterial's display high viscosity at low shear rate and reduce to a low viscosity with high shear rate, due to the occurrence of the number of particles, resulting in increasing interactions between particles which then leading to an overall increase resistance with fluid layer. Nanomaterials show Newtonian behaviour with an increase in shear rate where the interaction between particles become weaker and is broken down. Rheology viscometer was used to determine the viscosity of the polymer composite with the addition CNTs in different ratio figure 8. The result shows the concentration of the carbon nanotube alter the viscosity of the dope solution, it was revealed that the increasing in temperature lead to decrease in viscosity and M4 show higher viscosity due to the high concentration of CNTs figure 9.

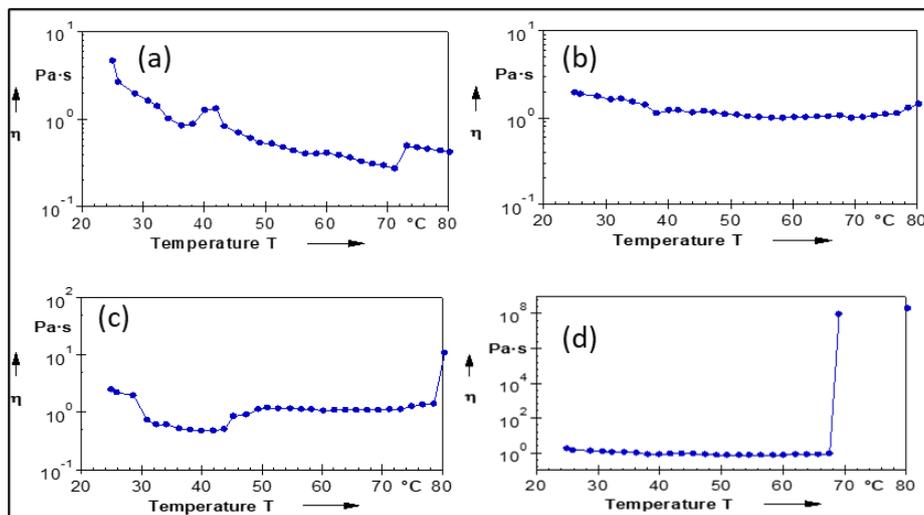


Fig. 8. Viscosity versus temperature of MWCNT with polymer dope solution with ratio of a) 0.005w% b) 0.01w% c) 0.02w% and d) 0.03w%.

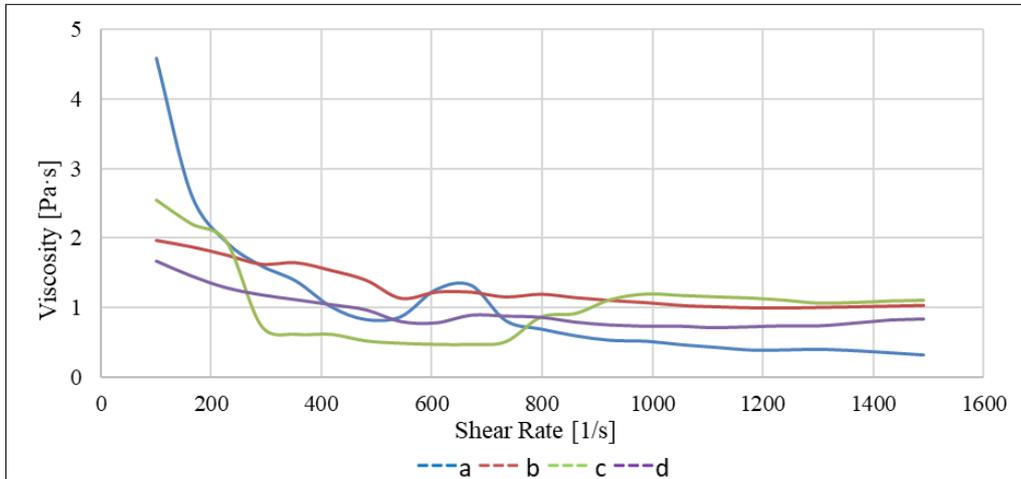


Fig. 9. Viscosity versus shear rate of MWCNT with polymer dope solution with ratio of a) 0.005w% b) 0.01w% c) 0.02w% and d) 0.03w%.

4.5. Electrical Conductivity of the polymer fiber composite

The Carbon nanotube polymer fibre composite consists of intrinsic resistance and the constant resistance by the tunnelling effect between the next CNTs. Then electrical conduction only goes through from one CNT to another in non-conductive polymer matrix.

$$R_{CNT} = R_i + R_c \quad 9$$

Where R_i is an intrinsic resistance of a CNT and R_c is a contact resistance between CNTs. The estimation of conductance of a CNT is difficult since the conductance of a CNT depends on many parameters, such as the number of current carrying shells, number of conducting channels for each shell, inter-shell interactions, and the number of metallic shells [27]. In this study, a CNT is considered as a conducting medium, and thus its intrinsic resistance can simply be calculated as equation 10 and figure 10 illustrate the result of the conductivity at different CNT ratio:

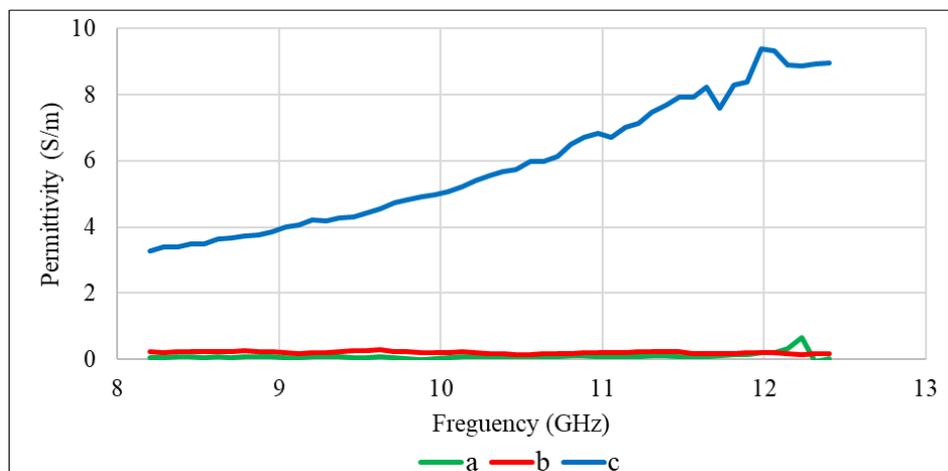


Fig. 10. Electrical permittivity of MWCNT polymer fibre of (a) 0.005w% b) 0.01w% c) 0.02w% and d) 0.03w%.

$$R_i = \frac{4L}{\pi D^2 \sigma_i} \quad 10$$

Where σ_i is the electrical conductivity of a CNT L the length, and D the diameter of the CNT, respectively. The contact resistance of CNTs dispersed in a polymer matrix also varies in a wide range [28, 29]. The contact resistance between CNTs in the polymer matrix should be higher than that without the matrix since a thin nonconductive polymer film exists between CNTs, which results in a dramatic increase in the contact resistance of the composites. Therefore, for a CNT/polymer composite, once R_i and R_c for a single CNT are obtained, the effective electrical conductivity of the CNT can be calculated by

$$\bar{\sigma}_{cnt} = \frac{1}{1 + R_c/R_i} \quad 11$$

5. Conclusion

The polymer fibres composite was extruded by spinning techniques and conductive polymer carbon polymer fiber with diameters in range of about 80-500nm. It appeared that the increase in concentration of carbon nanotube increase the conductivity of the material, the stimulation of MWCNT using CVD shows the sum of orbital range above the fermi level between -18- 16 ev and the maximum sum of P orbital above fermi level is between 0 to 16ev. The nanomaterial -display high viscosity at low shear rate and it reduce the viscosity at high shear rate which reveal from the result that the concentration of the CNTs in the dope solution alter the viscosity of the materials, MWCNT was able to increase the conductivity of the fiber from 0.6×10^{10} (S/m) to 9×10^{10} it was also reveal from the result that CNTs concentration significantly improved the thermal stability of the polymer carbon nanofiber which is suitable in different applications and this polymer fiber serve as the wave propagation to activate nanoparticles in the porous media.

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