ANALYSIS OF HYBRID COMPOSITES OF 3-D WOVEN CARBON FIBRE AND PARTICULATES REINFORCED ULTRA-HIGH DENSITY POLYETHYLENE USING SCANNING ELECTRON MICROSCOPY

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ABSTRACT

The composite materials based on renewable raw materials from agriculture and biomass is more used, because these products significantly compensate for the use of fossil fuels and reduce the greenhouse gas emissions in comparison with the petrol-based traditional materials. The composite from the 6cm of (3-D) woven mat Carbon Fibre and particulates: 0.075µm Metal Chips (Mild Steel), 0.075µm of Aluminium Silicate (Glass), 0.040µm Alumina (Ceramic) were the reinforcements while the smelted ultra-high density Polyethylene was the matrix. The percentage by weight of Polyethylene (matrix) was varied from 44wt% to 76wt% for optimization. The samples specimen of composites were produced for Mechanical property tests ; Tensile Strength of (10x15x100)mm^3, Impact Strength of (10x10x100)mm^3, Flexural Strength of (30x30x100)mm^3, and Hardness Strength of (30x30)mm^2. The experimental results showed that the highest Tensile Strength of 113MPa, Impact Strength of 2.855J, Flexural Strength of 122.517MPa and Hardness Strength of 67Hv were obtained. The Scanning Electron Microscopy was carried out to observe the fibre arrangements of 76wt%, 68wt%, 60wt%, 52wt% and 44wt% of the composite from the cut section. The result showed that 44wt% of resin in the composite has highest mechanical strength.

KEY WORDS: Composite, SEM, Tensile, Impact, Flexural and Hardness Strength

1 INTRODUCTION

Natural fibers in polymers comes with several challenges, such as excessive absorption of water and low thermal properties, which must be overcome in order to manufacture materials with properties comparable with the traditional composite materials. The synthetic resins have the disadvantage of a restriction of processing due to the high viscosity at melting, phenomenon which occurs at injection molding, and the final product is hard to be recycled. This disadvantage can be removed by using a vegetal oil resin-based thermorigid-biological matrix, which, being biodegradable, does not require a polymerization process [1] Bio-polymers are obtained from renewable resources and, during recent years, they have acquired a higher importance compared with petrol-based polymers. Among the most used vegetal resins are Sandarac, Copal and Dammar. These natural resins are insoluble in water, but are mildly soluble in oil, alcohol, turpentine and partially in petrol. With certain organic solvents, they form solutions usable as coverage Polymers 2020, 12, 1649; doi:10.3390/polym12081649 www.mdpi.com/journal/polymers Polymers 2020, 12, 1649 2 of 20 polish. These polishes have been used since the middle ages by people with the purpose of embellishing and protecting paintings, musical instruments or furniture. A disadvantage for the natural varnishes is given by the fact that it cannot form viscous resins (see for example the works. A

solution to remove this disadvantage is to use hybrid resins obtained by combining more elements, out of which at least one is organic and at least one is synthetic. It must be mentioned that most tests to obtain such resins have taken place in the industry of polishes [2] Hybrid resins represent an environment friendly alternative in comparison with synthetic resins. Besides the hybrid resins, the use of natural reinforcing materials instead of synthetic fibers has developed very much with a view to manufacturing biodegradable composite materials [3]. In comparison with synthetic fibers, natural fibers are characterized by biodegradability and renewability. Among the most used natural fibers, the following can be mentioned: kenaf, hemp, flax, jute, henequen, pineapple leaves, sisal, wood, herbage, corn stem, coconut, etc. According to [1], the specific strength and specific moduli of some natural fibers are comparable with those of the glass fiber. Instead, the ultimate tensile strength, Young's modulus, density are lower in comparison with those of the glass fiber. The mechanical features, such as density, fiber diameter, tensile strength, Young's modulus, elongation at break, for natural fibers (flax, hemp, jute, kenaf, ramie, nettle, sisal, henequen, abaca, palm oil, cotton, coconut) and synthetic fibers (glass fiber, carbon, Kevlar) are found in [3]. Among these natural fibers, flax, due to its higher tensile properties, has been applied widely as reinforcement material for polymeric composites. Flax fibers have some advantages in comparison with glass fiber, because these are less dense, renewable, combustible and have a relatively low price. [4] compared the inorganic fraction and the mechanical properties of three nanofilled composites with four universal hybrid and two microfilled composites. The degrees of conversion of materials photopolymerized using halogen and LED units were also investigated using Scanning Electron Microscopy (SEM). Three nanofilled (Supreme, Grandio and Grandio flow), four universal hybrid (point-4, Tetric ceram, Venus, Z 100) and two microfilled (A 110, Durafill VS) composites were used. The morphology of the filler particles was determined using Scanning Electron Microscopy (SEM). The microfilled composites exhibited by far the lowest mechanical properties. [5] determined the effect of the different surface treatments on the bond strength of PEEK composite materials. He evaluated the effect of different surface treatments on the bond strength between polyetheretherkotone (PEEK) composite materials and each of two different luting cements. One hundred specimens were randomly divided into five groups (n = 20/group) as follow: (a) no treatment (b) 98% sulfuric acid (c) 9.5% hydrofluoric acid (d) argon plasma treatment and (e) sandblast with 50µm Al_2O_3 particles. Each group was divided into two subgroups of different cements: Rely X^{TM} Unicem and SEM Bond/ Clearfil AP- X^{TM}. The cements were bonded onto the specimens. All specimens were stored in distilled water at 37^{o} for 24hour. The surfaces bond strength value of using SE Bond/Clearfil AP- X^{TM} was higher than that of sing Rely X^{TM} unicem with the same surface conditioning method (P less than 0.05). The use of SE Bond/Clearfil AP- X^{TM} after the surface conditioning of PEEK composite material treated with sulfuric acid or argon plasma can be recommended as an effective bonding method. [6] carried out acoustic emission to characterize failure mechanisms in laminated composite materials subjected to end notch flexure test which stimulate mode II delamination. The phase angle of Hilbert transform was used to analyze the extracted acoustic emission signals in three main stages during the loading i.e. the initiation, the maximum nearby and the stage where the crack has propagated to the middle of the specimens. A scanning electron microscope was used to observe the cracked surfaces. The results showed good applicability of the proposed acoustic emission based method for characterization of the damage mechanisms in the laminates. There was also a good agreement between the scanning electron microscopic images and the achieved results. [7] worked on in-situ SEM study of transverse cracking and delamination in laminated composite materials. He investigated into how altering transverse crack densities can influence the growth of delamination. Novel in-situ SEM micromechanical testing and acoustic damage detection techniques were coupled and used to determine transverse crack initiation loads, transverse crack density and local micro-delamination lengths for a member of cross-ply laminated. The laminates were loaded in a four-point bending mode to induce crack opening direct stresses on the tension side. To examine the effect of combined direct and shear stresses, the laminates were also loaded in three-point bending mode, the suitable comparisons between both the bending modes allowed for the influence of the shear stress to be isolated. It was found that the lower transverse crack densities meant that the micro-delamination which initiated at the ply boundary required significantly greater stress to fully coalesce as the distance between transverse cracks was greater. Once micro-delamination had initiated, its length was found to be linearly related to the load applied. [8] carried out reinforcement of experimental

composite materials based on amorphous calcium phosphate with inert fillers. He examined the influence of the addition of glass fillers with different sizes and degrees of silanization percentages to remineralizing composite materials based on amorphous calcium phosphate (ACP). Three ACP based materials: O-ACP (40wt% ACP, 60wt% resin), Ba-ACP (40wt% ACP, 50wt% resin, 10wt% barium-glass) and Sr-ACP(40wt% ACP, 50wt% resin, 10wt% strontium-glass) were compared to the control material, resin modified glass ionomer. The fillers and composites were characterized using Scanning Electron Microscopy (SEM). Flexural strength and modulus were determined using three-point bending test. The addition of barium-glass fillers (35.4(29.1-42.1))MPa, (Medium (25-75%)) had improved the flexural strength. The mixture strontium-glass did not have any effect on flexural strength, but significantly improved its flexural modulus. In corporation of barium-glass fillers to the composition of ACP composites contributed to the improvement of flexural strength and modulus. [9] evaluated water sorption, flexural properties, bonding properties, and elemental composition of photopolymerizable resin-impregnated fibre-reinforced composite (FRC) materials (everstick C & B and BR-100) (FBD). Bar shaped specimens (2x2x25)mm were prepared for water sorption and flexural strength testing. The specimens (n=6) were polymerized either with a hand light-curing unit for 40s or, additionally, in a light-curing oven for 20mins and stored in water for 30days. Water sorption was measured during this time, followed by measurements of flexural strength and modulus. A shear bond strength test was performed to determine the bonding characteristic of polymerized FRC to composite resin luting cement (panavia-F), (N=15). The cement was bonded to the FRC substrate and the specimens were thermocycled 5000 times (5 to 55^{o}C) in water. SEM/EDS were analyzed to evaluate the elemental composition of the glass fibres and the fibre distribution in cross-section. SEM/EDS analysis revealed that fibres of both FRC materials consist of the same oxides (SiO_2, CaO, and Al_2O_3) in ratios. The distribution of fibres in the cross-section of specimen was more evenly distributed in everstick C & B than in BR-100. [10] investigated microstructure and tensile properties of porous natural coir fibre for use in composite materials. X-ray microtomography in SEM (SEM-CT) and SEM image analysis were used to examined the fibres, microfibril angles and fibre porosity. Mechanical properties of coir fibres were determined by performing fibre tensile tests, in which an integrated optical strain mapping system is used to define fibre strain for producing more reliable values of E-modulus and strain at failure elementary fibres and a lacuna at the centre. The elementary fibre microfibrils with a large misorientation with respect to the elementary fibre axis. Coir fibres appear to have a high porosity of 22 to 30%. The high microfibrillar angle in the coir fibres leads to the low stiffness in fibre direction and to high elongation to failure to reorientation of the microfibrills under tensile loading. [11] carried out research on Nylon-wood fibre and Polypropylene-wood fibre composite material were manufacture without any additive to determine the effects of wood fibre on the mechanical properties of the different composites. The raw materials used were eucalypt hard wood fibre, Nylon fibre obtained from stockings, and polypropylene (PP) pellets. A hot press technique was used to manufacture the composite materials, and improvements in the manufacturing method are suggested. Tests were carried out on the manufactured boards to determine tensile strength and modulus of elasticity. Fracture surfaces were examined using scanning electron microscopy (SEM) to investigate failure mechanisms. An increase in tensile strength and modulus of elasticity was observed in wood fibre/nylon matrix composites, indicating that interfacial bonding occurred between these two phases. Bundles of wood fibres with internal voids prevent achieving maximum mechanical properties. The tensile strength of the PP based composites decreased significantly with increasing wood fibre content. In this research, the Scanning Electron Microscopy was carried out to observe the fibre morphological arrangements in the composite from the cut section of the laminate for the determination of the mechanical properties of the composite.

2 MATERIAS AND METHOD

In this research, the composite from the 6cm of (3-D) woven mat Carbon Fibre and particulates: 0.075µm Metal Chips (Mild Steel), 0.075µm of Aluminium Silicate (Glass), 0.040µm Alumina (Ceramic) were the reinforcements while the ultra-high density Polyethylene was the matrix. The percentage by weight of Polyethylene (matrix) was varied from 44wt% to 76wt% to obtain the optimal value. The Scanning Electron Microscopy was carried out to observe the fibre morphological arrangements in the composite from the cut section of the laminate for the determination of the mechanical properties of the composite.

2.1 Materials

The following materials were used for the production of the hard body armour protection: 6cm of 3-D woven Carbon Fibre, Metal Chips (mild steel), Glass-Aluminium Silicate (Al_2SiO_5), Ceramic-Alumina (Al_2O_3), Ultra-High Density Polyethylene (Resin), Silicon Wax, Petroleum Jelly, Gel coat material and other miscellaneous materials.

3-D Woven Carbon Fibre

It was obtained from Nigerian Air Force, Air Force Base Kaduna. It is the dense carbon fibre with three dimensional woven fibre for high mechanical properties performance

Metal Chips (Mild Steel)

It was sourced from Air Force Institute of Technology Kaduna Central Workshop. The chips was taken to Chemical Engineering for Milling using Ball Milling Machine to particle sizes of 0.03mm in ABU Zaria. The Chips was further subjected to mechanical impact force using piston, in Building Technology Department of the same Institution which reducing the particle sizes to 0.075µm.

Aluminium Silicate (Al_2SiO_5)

It was sourced from Kaduna Metropolis where the particle sizes were reduced using hammer to the particle sizes of 0.01mm. The particle sizes were further reduced using Ball Milling Machine to 0.07.5 µm from ABU Zaria Kaduna.

Ceramic- Alumina (Al_2O_3)

It was sourced from Nigerian Research Institute of Metals and Mineral resources (Malani) in Kaduna. The material was milled with Ball Mill Machine in Chemical Engineering Department to the particle sizes of 0.04µm. Ultra-High Density Polyethylene is the resin used for the reinforcements of the composites. It was bought from the National Institute of Leather and Polymer Technology Zaria. It was smelted using two roll mill after ten (10) minutes for proper mixing of the composites.

Sample Preparation of the Materials for Mechanical Test

The percentage weight of aluminium silicate, alumina, metal chips (mild steel), polyethylene and carbon fibre each are measured with a digital weighing balance. The required pellets of polyethylene were smelted and the quantities of these particulates were added to the TwoRoll Mill and rolled for ten (10) minutes for proper mixing of the particulates. The composites were layed into the mould and were placed in a compressive machine at a holding temperature of 120^{o}C for 15 minutes. The laminates produced is cooled in the air for about 30minutes before the mechanical test was carried out.

2.2 Methods

Mechanical Test

Table 1. List of Equipment

S/N	Equipment	Specification	Manufacturer and location of equipment
1	Electronic Universal Testing Machine (Tensile)	WDW-100KN 190536	(China)Metallurgy/Material Engineering Depart. ABU Zaria
2	Charpy Impact Testing machine	HD96QD (15J & 25J)	(Britain) Metallurgy and Material Engineering Depart. ABU Zaria
3	Vickers Hardness Testing Machine	ASTM E140	Mechanical Engineering Department ABU Zaria
4	Universal Materials Testing machine (Flexural)	Nr. 261	(Britain) Mechanical Engineering Department ABU Zaria
5	Mill Roller	KERA b.v	(Holland)Chemical Engineering Department ABU Zaria

6	Industrial sieve	40 & 75mm micron standard	Building Technology Department ABU Zaria
7	Dynamic Mechanical Analysis (DMA)	DMA242E_Artemis	(NEZTCH GERMANY) Mechanical Engineering Department ABU Zaria
8	Crushing Machine	Masch Nr.70992	(Holland)Chemical Engineering Dept. ABU Zaria.
9	Scanning Electron Microscope (SEM)	PRO:X Phenonm World. (MVE01570775) 800-07334	(Switzerland) Central research laboratory, Umaru Musa Yar'adua University Katsina

Sample Specimens for Mechanical Test

The samples specimen for the preparation of bending strength is (30 x 30 x 100) mm^3, the tensile strength is (100 x 15 x 10) mm^3, the impact strength is (100 x 10 x 10) mm^3 and finally, the compressive strength is (30 x 30) mm^2.

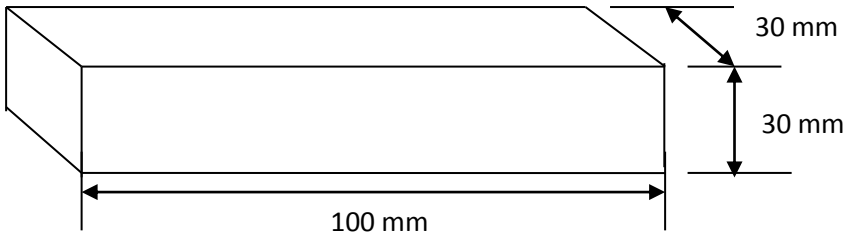

Figure 1. Sample specimen for bending strength

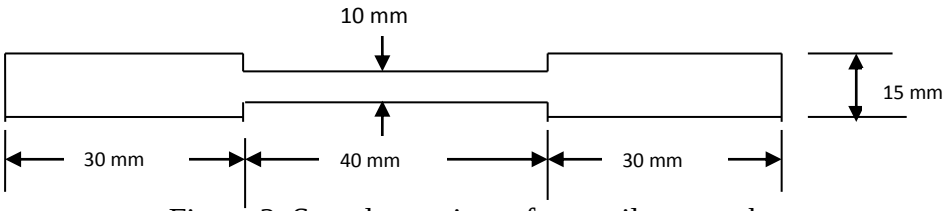

Figure 2. Sample specimen for tensile strength.

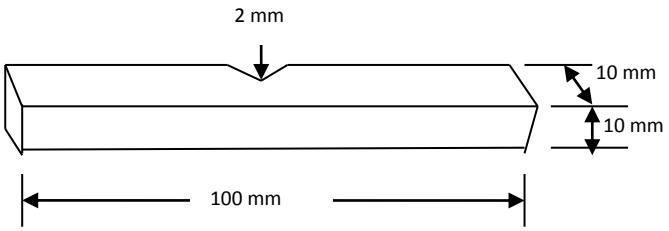

Figure 3. Sample specimen for impact strength.

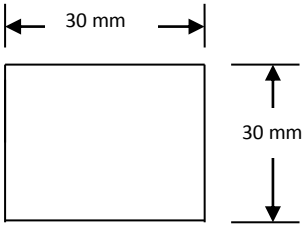

Figure 4. Sample specimen for compressive strength

Tensile Test

The tensile test was carried out using Universal Electronic Testing Machine, WDW-100KN 190536. The test samples were placed and gripped in between the jaws of the machine. The load was

gradually applied through a hydraulic press until deformation occurred and the result was displayed digitally [12]. Ultimate tensile strength stresses were calculated using equations 1 and 2.

$$\sigma = \frac{P}{A} \quad (1)$$

$$A = \frac{\pi d^2}{4} \quad (2)$$

Impact Test

The impact test was carried out on the Charpy impact testing Machine in accordance to HD96QD (15J & 25J). The machine consists of tops between which the test sample is inserted. With the specimen positioned in the vice, a blow was stroked by two released pendulums in opposite directions from their calibrated standard height to fracture off the specimen. The energy retained was determined by the height rise of the pendulum as indicated by the dial gauge. The difference between the energy input and energy retained gave the energy absorbed [13].

Hardness Test

The hardness values of the hybrid fibre composites were determined in accordance with the ASTM E140 standard using the Vickers Hardness testing machine present in the shell office, department of Mechanical Engineering A.B.U., Zaria. The machine consists of a digital display reading HV and HB.,0.42cm steel ball indenter with a load of 0.3kgf. The specimen prepared to a good finish of perfectly horizontal surface were indented. The depth of indentation indicated the hardness of the laminates [14].

Flexural Test

The machine used for the flexural test is a universal tester of model Nr. 261. It has a hydraulic press that actuates the pressure of the machine to grip the load and the result of the force is displayed digitally on the machine of the composite materials. The Universal Material Testing Machine is situated in Mechanical Engineering Department, ABU Zaria [15].

3 RESULTS AND DISCUSSION

3.1 Scanning Electron Microscopy (SEM)

Scanning electron microscope produced images of a sample by scanning the surface with a focused beam of electrons. The electron beam is scanned in a raster scan pattern, and the position of the beam is combined with the intensity of the detected signal to produce an image. The PRO:X: Phemonm World was used, the sample was first cleaned with methanol in order to remove dirt from the surface. The computer was switched on and the software for the microscope was opened, then the Digital microscope was then powered and set to the required magnification of 300μm. The sample was then placed on the sample slit and the lens was focused on the sample. The setting was done so that it was not blurred and the images were taken. The result of the bond between the reinforcements and resin in the composites were snapped from the cut section as shown below.

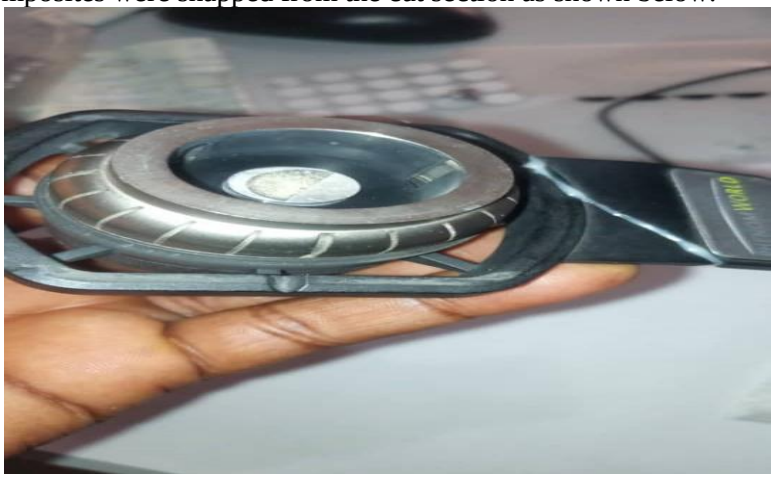

Plate 1. SEM PRO: X: Phemonm World

Plate 2. Demonstration for the Operation of SEM PRO:X:Phenonm

3.2 SEM of 44wt% Polyethylene in Composite

The SEM revealed that the percentage by weight of (44wt%) polyethylene has the reinforcement arranged in non-uniform direction. It was discovered that irregular pattern of the arrangements of the carbon fibre in the composite gave stronger bond with the resin and higher strength of the composite. This means that the impact load can easily be transferred from the resin to the reinforcement. In figure 5, the resin, particulates and carbon fibre represented as A, B and C were clearly shown, and there was no default during the production stage.

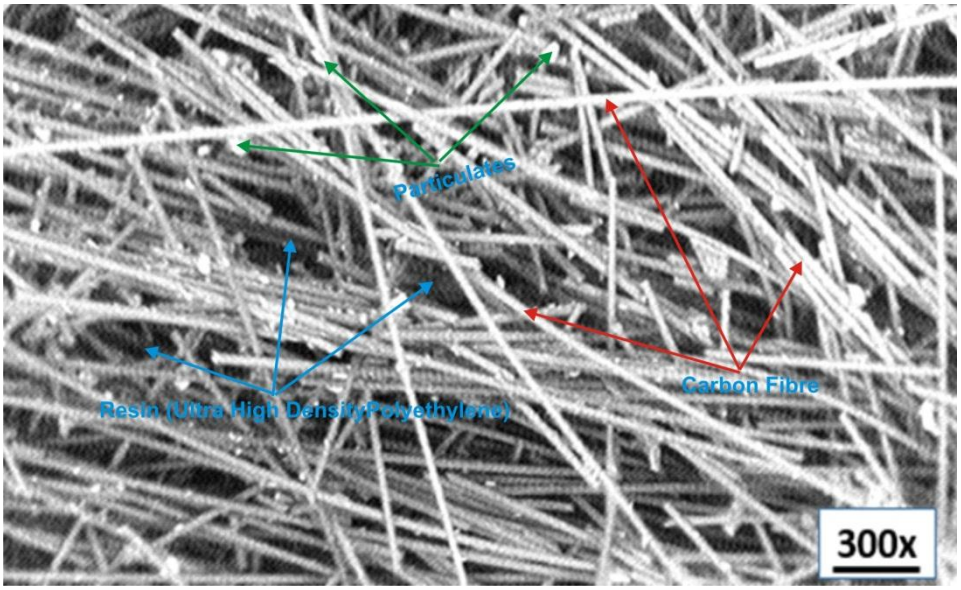

Figure 5. SEM of 44wt% of Polyethylene in the composite

3.3 SEM of 52wt% Polyethylene in Composite

The arrangements of Carbon fibre in the composite was almost on the same direction. It means that 52wt% polyethylene can transferred impact load from the resin to the reinforcement but as that of composite of 44wt% polyethylene. The resin, particulates and the carbon fibre represented as A, B and C were clearly shown in figure 6 using SEM, and there was no default during the production stage.

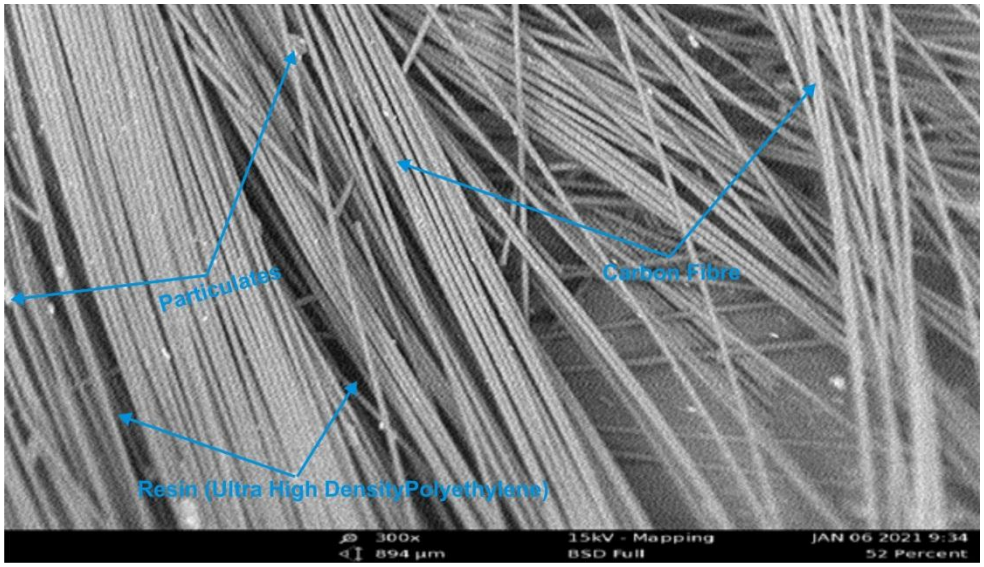

Figure 6. SEM of 52wt% Polyethylene in the composite

3.4 SEM of 60wt% Polyethylene in Composite

The arrangements of Carbon fibre in the composite was dense and not uniformly arranged but remember that the higher the non-uniform arrangement of the carbon fibre, the higher the mechanical strength. It means that impact load can easily be transferred from the resin to the reinforcement in 60wt% polyethylene in the composite. In figure 7, SEM showed the distributions of resin, particulates and carbon fibre as indicated as A, B and C respectively and there was no default during the production stage.

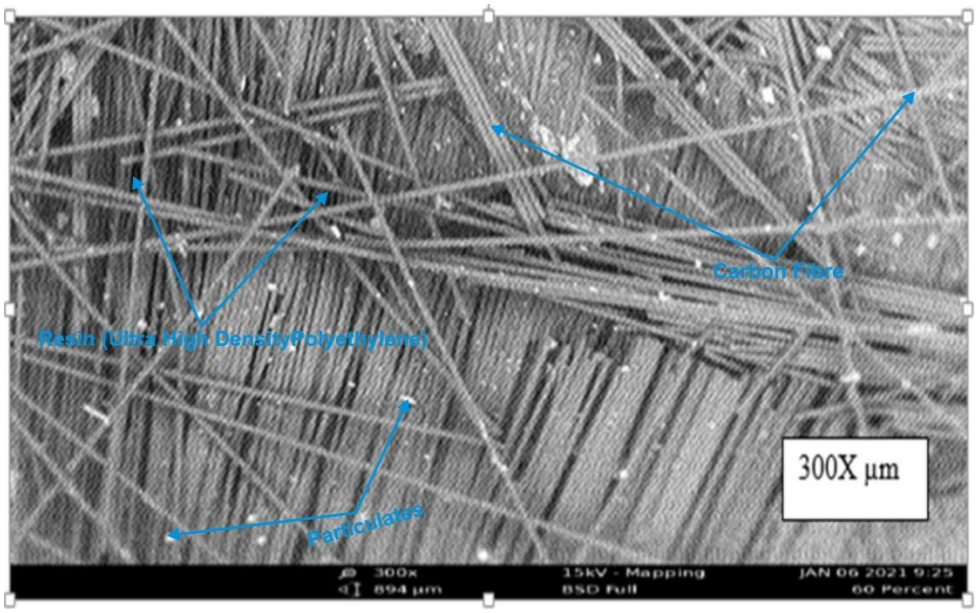

Figure 7. SEM of 60wt% Polyethylene in the Composite

3.5 SEM of 68wt% Polyethylene in Composite

The arrangements of Carbon fibre in the composite was dense and uniformly arranged. In figure 8, SEM clearly showed the presence of the A, B and C which means that resin, particulates and carbon fibre were randomly distributed. The impact load from the resin can easily be transferred to the reinforcement. There was no default during the production stage for 68wt% polyethylene in the composite.

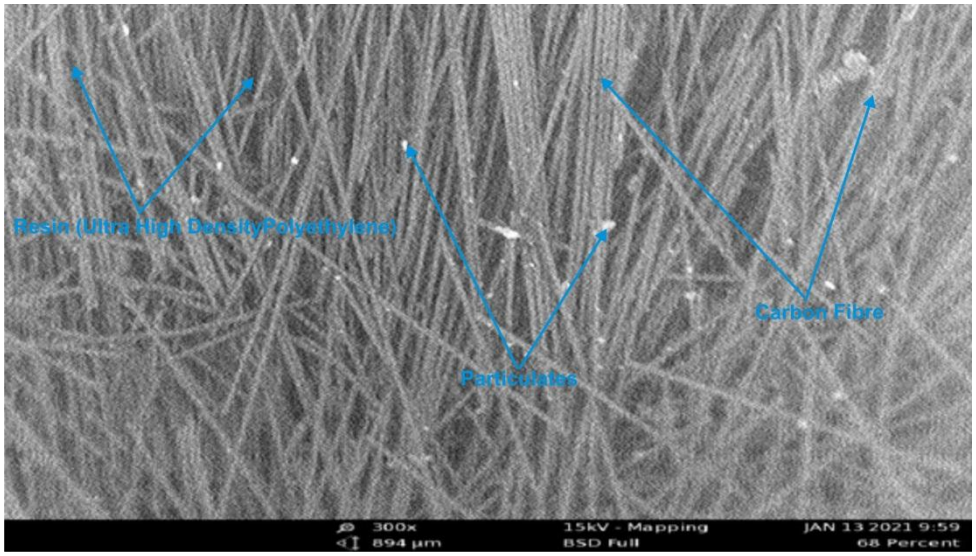

Figure 8. SEM of 68wt% Polyethylene in the Composite

3.6 SEM of 76wt% Polyethylene in Composite

The arrangements of Carbon fibre in the composite was dense but not uniformly distributed. Symbol A, in figure 9 shows that the reinforcements are not dense at that point therefore, the ultra-high density polyethylene occupied the space which means that transfer of impact load from the resin to the reinforcement will be low and it is termed as default during production stage. B and C were distributed as determined by SEM. It means that 76wt% polyethylene in the composite has high mechanical strength but lower than 44wt% of polyethylene in the composite.

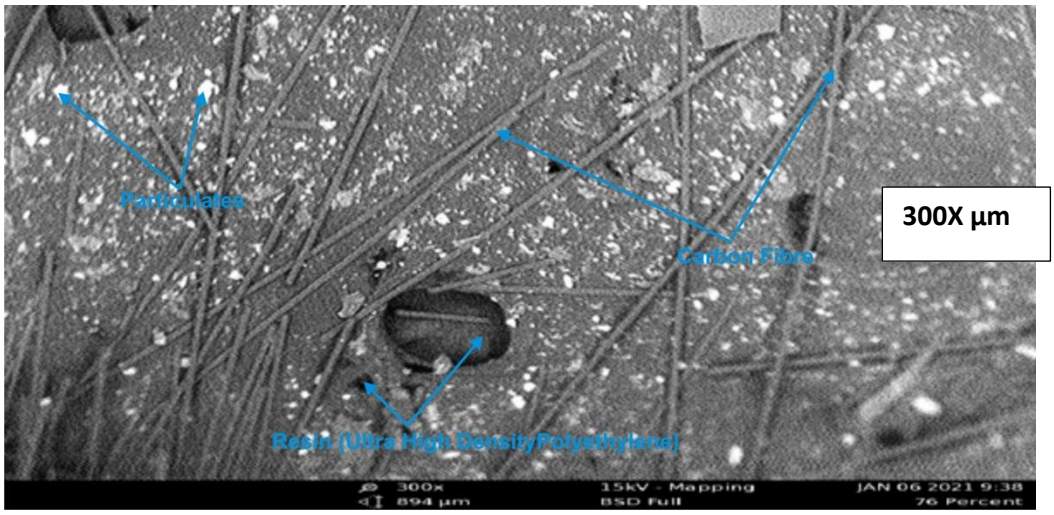

Figure 9. SEM of 76wt% Polyethylene in the Composite

4 CONCLUSIONS

The experimental results showed that from the composite, the highest Tensile Strength was 113MPa, Impact Strength was 2.855J, Flexural Strength was 122.517MPa and Hardness Strength was 67Hv. The Scanning Electron Microscopy was carried out to observe the fibre arrangements of 76wt%, 68wt%, 60wt%, 52wt% and 44wt% of the composite from the cut section. The result showed that 44wt% of resin in the composite has highest mechanical strength. the bonding arrangement of the fibres in the matrix from the cut section, showed that the percentage by weight of 44wt% resin in the composite has the highest randomly arranged pattern of the carbon fibres in the matrix, which means that the the higher the non-uniformly arranged carbon fibre in the composite, the higher the mechanical strength. The percentage by weight of 76wt% resin in composite has production defaults, that can been seen from SEM. It has cracks and holes which affects the mechanical strength of the composite.

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