Morphological Study of SiO_2 Biocomposite

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Abstract **- Industries are pursuing towards biocomposite technologies by using plant fibres as the reinforcement materials. There are presence of silica in the plant fibres and generally act as a defense mechanism for the plants against herbivores. Silica biocomposite will be prepared by mixing silica fiber along with linear low-density polyethylene (LLDPE) and undergo grafting via extrusion in an extruder. This study is done to study the morphology of SiO_2 biocomposite and the effect of SiO_2 towards the biocomposite strength. The results are obtained by performing scanning electron microscopy (SEM) analysis with variation of different SiO_2 insertion based on the effect of cutting techniques and the surface of the sample being rubbed by sand paper.**

Keywords**:** Linear low-density polyethylene, silicon dioxide, morphological study, grafting, biocomposite

I. INTRODUCTION

Since ancient times, biocomposites is already known and utilized by humankind. However, the use of these things were completely outran in twentieth century by the use of synthetic polymers in industry [1]. Recently, the interest on biocomposite materials has shown an increment due to the potential of being substitute to conventional materials used in manufacturing industries. Biocomposite materials are produced with natural fibers or natural resins instead of the synthesized ones. The bio-based fibers are usually obtained from plants or animals [2]. Basically, biocomposites are used to supplant or preserve non-renewable energy sources and petrochemicals. It has been well documented that biocomposites have potential benefits for example, minimal effort, great thermal properties, low thickness and non-rough preparing. Thus, it is essential to put to use these biocomposites and assimilate them into biodegradable polymer and improving its properties as well as reducing the production cost [3].

As per ecological concerns and budgetary issues, natural fibers have turned out to be intriguing and captivating these days to be used as a modern material and auxiliary material for restoring of structures. Oil palm fiber is one of the large agriculture waste. Oil palm empty fruit bunch fiber (OPF) is a natural fiber, which is discovered a great deal in tropical regions. Researchers have used OPF fiber with many types of resins such as epoxy, polypropylene, polyester, and phenol formaldehyde [4]. The fuse of these oil palm fibers significantly enhances the mechanical performance of phenol–formaldehyde resin. The resultant composite item will be a financially perceptive and value-added substitute for regular building materials, which can go about as a superior substitute for wood in building industry [5].

There are embedded silica bodies at the surface of oil palm fiber [6]. Silica is known to ease different wellsprings of worry in plants including herbivory, pathogens, dry season, and substantial metal poisonous quality. Silica accumulation by plant such as grass is regularly viewed as an adjustment for increased herbivore pressure. The testimony of silicon into epidermal cells of grass species is accepted to be a basic part that plants use as a defend against vermin and natural burdens. Silica bodies are a standout amongst the most solid structures in grass tissues that stay as particles in the dirt even after all other common parts of plant have normally rotted or corrupted [7].

Morphology is part of science that relates with the study of the form and structure of organisms or substances and their specific structural features. It can be seen that with the morphological study, the characteristic for the certain substances can be understood and can be proved. Through SEM analysis, the presence and the shape as well as the arrangement of silica bodies are shown and determined. The image gotten from the SEM shows different arrangement of silica inside oil palm empty fruit bunch (OPEFB) fiber, where the arrangement are random or aligned with different weight percent of silica inside the related fiber [8]. Morphological study can be done to achieve the qualitative objective. There is a study where the arrangement of the silica bodies have an effect towards the strength of the biocomposite [9].

II. METHODOLOGY

2.1 Materials

LLDPE and SiO_2 are the main components of the biocomposites that will be used in this research. Both silica and LLDPE are bought from the supplier. Silica will be used as the reinforcing agent or fiber to reinforce the matrix, which is the LLDPE. It has the density of 2.65 g/cm^3 and it is insoluble in terms of its solubility. The melting point and the boiling point is 3110°F and 4046°F respectively. LLDPE acts as the matrix that will hold the fibers in place. It has a density of 0.91-0.94 g/cm^3. Other chemicals include maleic anhydride (MAH) and dicumyl peroxide (DCP). The anhydride has density of 1.32 g/cm^3, and melting point from 51 to 53°C. The peroxide has density of 1.56 g/mL and melting point from 39 to 41°C. These two chemicals were involved in graft copolymerization of fibers as initiator and monomer respectively.

2.2 *Synthesis of LLDPE composites:*

The process of grafting copolymerization was done by means of extrusion. The process was done by using the twin-screw extruder with a specific reaction zone temperature in the extruder. The materials were mixed and melt-blended in a twin-screw extruder at 170°C and a screw speed of 20 rpm. The model used is HAAKE™ Rheomax CTW 100 OS. Then, the extruded fibers were grinded. After the grinding, the grinded fibers were compressed into a film with specific thickness through the usage of Hydraulic Hot Forming Press Machine QC-602A (Cometech Testing Machine Co., Ltd.). The grinded, extruded mixture is hot-pressed at 170°C onto 1mm thick sheets for 10 minutes under pressure of 2000 psi with residence time at 20 minutes.

Before running the extruder, the substances needed and related such as the amount of fiber, anhydride and peroxide were weighed beforehand according to the formulation. The total of weight of sample of mixture of LLDPE, peroxide, maleic anhydride and

silicon dioxide is 100g. The function of peroxide as initiator is because of its viability to present long chain branches in LLDPE. The formulation is shown in Table 1. The biocomposite of LLDPE-g-MA/SiO_2 was prepared in three different weight percent (wt%) which are (93,2,4,1), (91,4,4,1) and (89,6,4,1) that represent weight percent of LLDPE, SiO_2, peroxide and maleic anhydride. This study is attempting to imitate or mimic the composition of SiO_2 in oil palm mesocarp fiber (OPMF).

Table 1: Formulation of all sample of LLDPE-g-MA/SiO2

Sample	Weight percent (%)			
	LLDPE	SiO_2	Peroxide	MA
1	93	2	4	1
2	91	4	4	1
3	89	6	4	1

2.3 *Morphological Analysis*

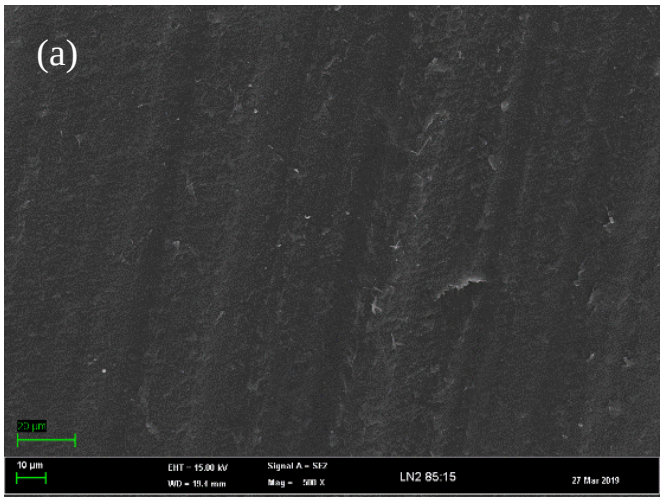
The surface morphology of the SiO_2 biocomposites were investigated by using scanning electron microscopy and an energy dispersive X-ray analyzer (EDX or EDA). SEM provides detailed high-resolution images of the sample by rastering a focused electron beam across the surface and detecting secondary or backscattered electron signal. EDX is used to provide elemental identification and quantitative compositional information. SEM/EDX is a secondary and backscattered electron images, elemental composition for sample sizes up to 200mm diameter and 80mm depth.

III. RESULTS AND DISCUSSION

3.1 *Effect of Different Cutting Techniques*

LLDPE biocomposites mixed with various composition of SiO_2 were successfully fabricated by grafting. The amount of SiO_2 mixed was 5wt%, 10wt%, and 15wt%. The samples that are going to be analysed are cut in half. The samples are cut through these two techniques. The first technique is cut the sample half by using the Universal testing machine for tensile stress by pulling the sample until it tear apart into half and the second technique is by immersing the sample in liquid nitrogen in a vacuum flask until it becomes hard and break it into half.

Figure 1(a) shows the surface of the sample that is cut by liquid nitrogen while Figure 1(b) shows the surface of the sample that is cut through tensile stress. In Figure 1(a), it can be seen that the surface of the sample is smoother than the surface of the sample that is shown in Figure 1(b). Figure 1(b) also shows that the surface became stretched and a little bit curly. The surface is stretched due to tensile stress activity, as tensile stress is the amount of stress that causes the material to elongate.



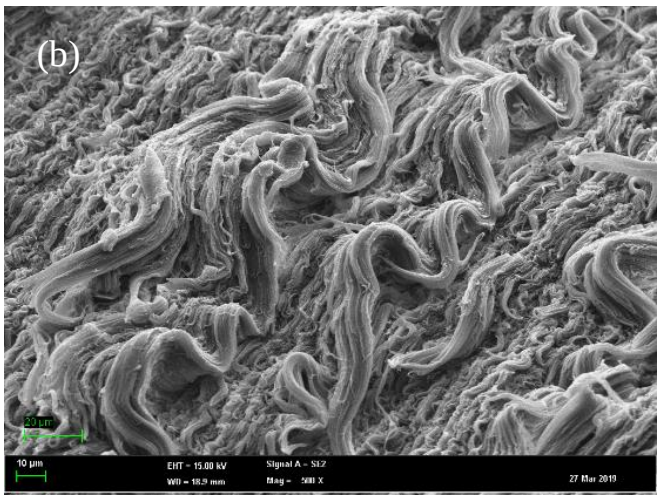


Fig. 1: SEM micrographs of (a) sample that is cut by liquid nitrogen and (b) sample that is cut due to tensile stress

Silica acts as the natural fiber in this composite as silica is present in the fiber of the plant that often used as the reinforcing fiber in the composite. Silica is often deposited in the form of amorphous silica gel ($SiO_2.nH_2O$) in most plants [10]. Figure 2 shows the surface of three different sample of different content of silica that is cut using liquid nitrogen, which is 5wt%, 10wt% and 15wt% respectively. In (a), the surface looks the roughest compared to the other two while (c) looks the smoothest and (b) stands in the middle. Silica can be seen dispersed in the matrix and are fairly distributed. Silica are often presents in the fiber in the form of small granules that acts as a defense mechanism for plants and also helps reduce many abiotic and biotic stresses to the plants [11].

Figure 3 shows the surface of the samples that is cut due to tensile stress. Both (a) and (b) show different morphology between each other and different from the morphology pattern in Figure 2. In Figure 3(a), the surface of the sample looks like gravel and they are agglomerate. In addition, in Figure 2(a) and 2(b), both show agglomeration at their silica deposits. The agglomeration of the fibers indicates that the sample or the composite has weak interfacial bonding [12]. Reinforcing polymer with natural fiber does resulting in increased of both flexural and tensile strength. As the fiber weight increases, the strength of the resulting composite is also increases. However, the deterioration of properties will happen if the fiber content is increased in excess amount and attributed to excessive clustering of fiber that will cause debonding under mechanical load [13].

The mechanical strength depends on the adhesion between the polymer layer and an adjacent inorganic dielectric such as silicon dioxide. Areas of poor interfacial adhesion such as at corners or other sharp features of the structure or any part that is associated with the damage processes in adjacent layers may initiate debonding in the structure between the fibers and also may lead to fracture on the surface [14]. Fractures can also be observed in Figure 3(a). Figure 3(b) shows that the matrix of the sample is stretched due to tensile stress. The flexibility and plasticity of the matrix because it is a type of geotextile and plastic make it stretches and elongates instead of breaks and fractures when the sample is passed its yield stress and undergoes plastic deformation.

Moreover, the fibers are less evenly dispersed as shown in Figure 2 and the stress will less evenly distributed and may cause the reduction in mechanical strength of the composite. The decrement in tensile strength per unit percentage increase becomes smaller as fiber loading amount is increased [12].

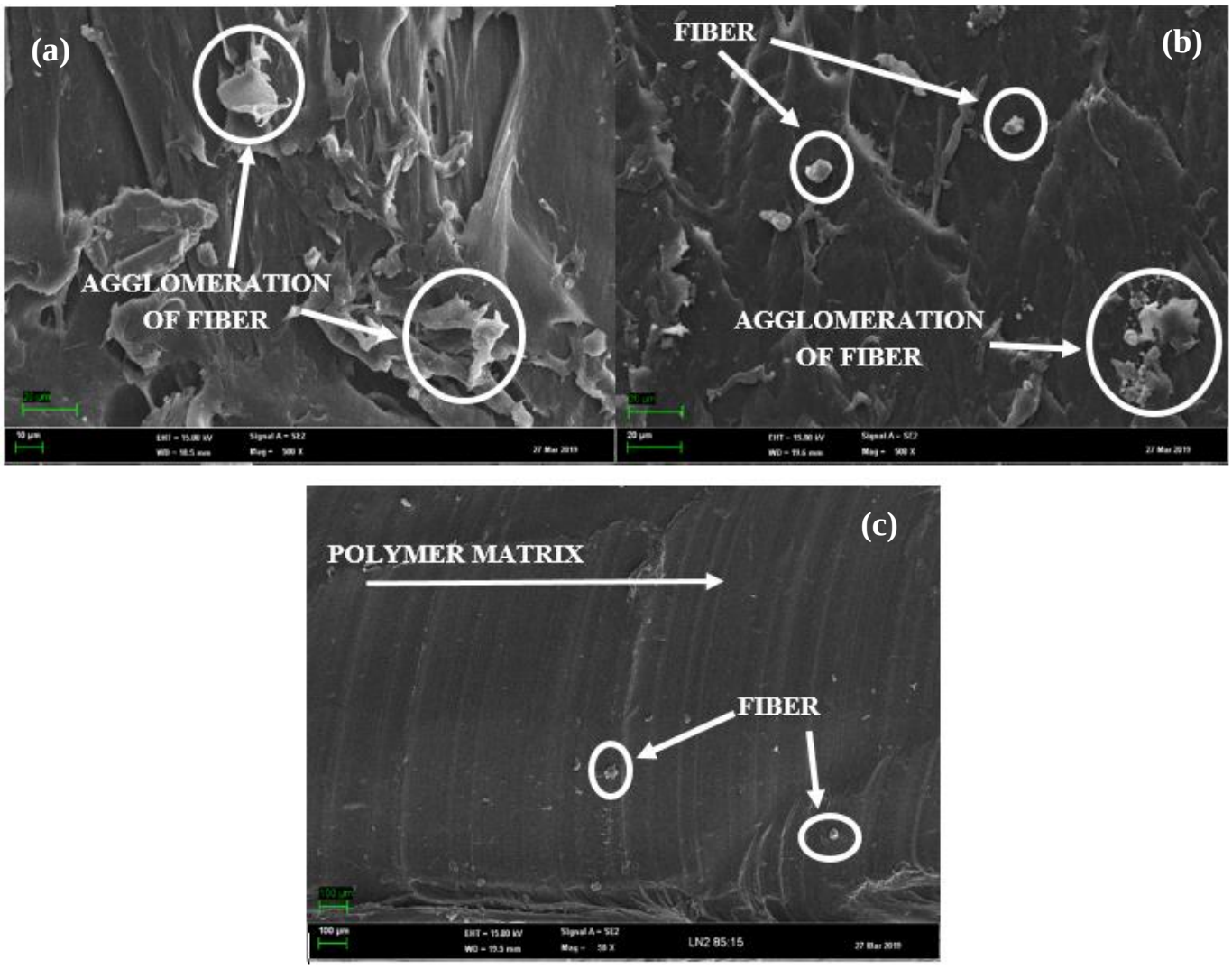


Fig. 2: SEM Micrographs of sample with different content of silica (a)5wt% (b)10wt% and (c)15wt%

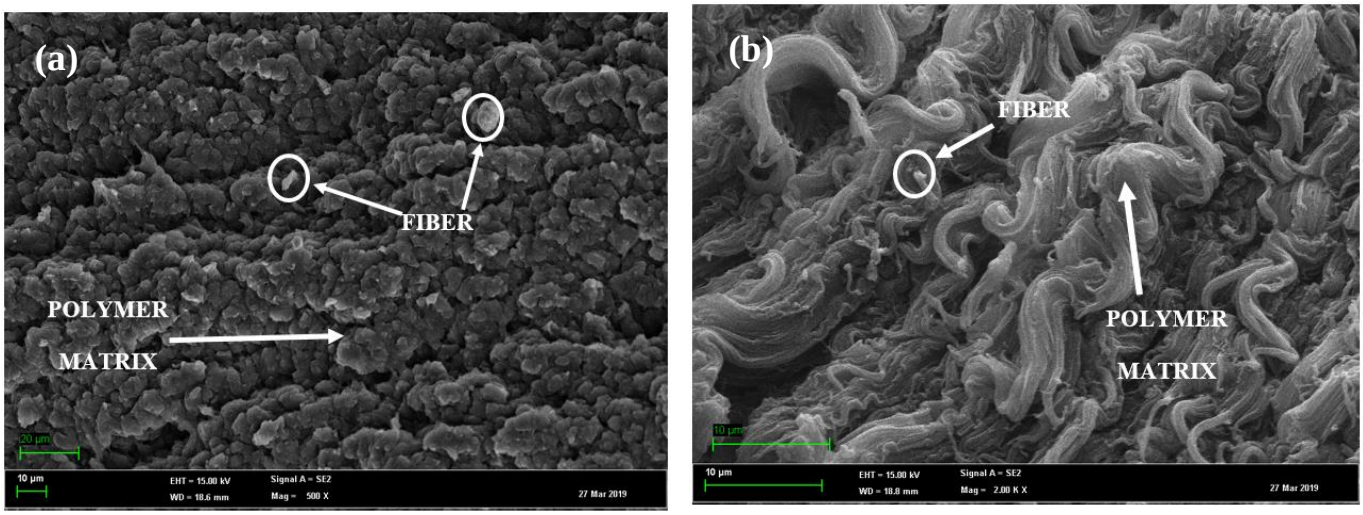


Fig. 3: SEM micrographs of the surface due to tensile stress (a)5wt% and 10wt% (b)15wt%

3.2 *Effect of Sand Paper*

SEM observation are also done on the cut surface of the sample that is rubbed with sand paper for three different content of silica as well, which is also 5wt%, 10wt% and 15wt%. Before the samples are rubbed, they are also cut by using liquid nitrogen.

Figure 4 shows the surface of the three different samples that are rubbed with sand paper. All of the figures below exhibit parallel lines throughout the polymer matrix while the fiber maintain its form and some have slight changes due to the rubbing of the sample surface against the surface of the sand paper. The known form of silica in natural fiber is granule form and it can be seen that some of the granules in the figures below are stretched, expanded and even undergo agglomeration but not changing most of the granules form as the polymer matrix did.

Furthermore, it can be deduced that the silica granules have irregular shapes as they have sides and angles of any length and size. Agglomeration can only be clearly seen in Figure 4(a) and (b). As mentioned previously above, agglomeration of fibers means that the sample has weak interfacial bonding [12]. The interfacial strength refers to the strength of the bond between the matrix phase and the dispersed phase and usually for polymer matrix, high interfacial strength is desired. Figure 4(c) did not display such agglomeration. This may due to having the highest amount of silica fiber load than the other two and thus can be deduced that the sample that has 15wt% of silica has the highest flexural and tensile strength based on these morphologies observations.

Another objective of the research is to study the SiO2 effect towards the biocomposite strength. High mechanical and interfacial strength is usually desirable for polymer matrix. The interfacial strength refers to the strength of the bond between the matrix phase and the dispersed phase. The other part of the experiment is conducted by rubbing the surface of the sample with sand paper. The sample is cut beforehand using liquid nitrogen. The study is also done by observation of the SEM micrographs. This part of experiment is also carried out for three different load of fibers in the composites (5-15wt%). Agglomeration is displayed in the results and indicates that the composites have weak interfacial bonding thus have interfacial strength. The sample that has 15wt%, which is the highest content of silica exhibits the least sign of

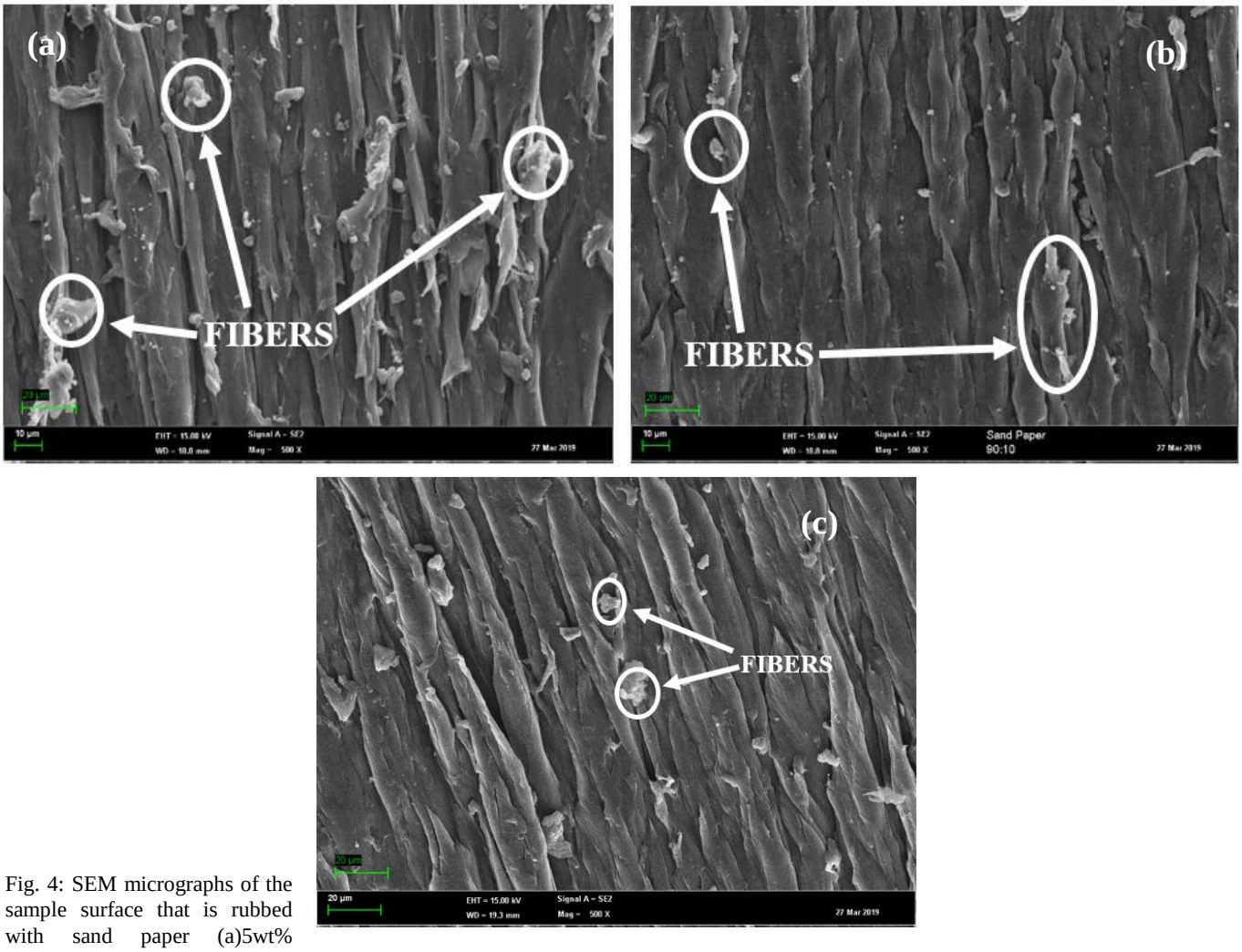


Fig. 4: SEM micrographs of the sample surface that is rubbed with sand paper (a)5wt% (b)10wt% and (c)15wt%

agglomeration. Even though the samples are rubbed with sand paper and the matrix of the samples undergo changes, the fiber granules in the polymer matrix maintain their shapes and only have slight changes.

In conclusion, it was evident that the sample that has 15wt% of silica has the highest flexural and tensile strength based on these morphologies observations. As the fiber weight increases, the strength of the resulting composite is also increases.

IV. CONCLUSION

This experimental study was conducted to achieve several objectives. The first objective of this study is to study the morphology of SiO_2 biocomposite. Silica acts as the natural fiber that reinforces the composites. In this study, LLDPE biocomposites loaded with an amount of fibres 5 to 15wt% were successfully fabricated by melt-blending technique in an extruder in the presence of MAH and DCP as monomer and initiator respectively. The characterization of SiO_2 biocomposites to be determined in this study is the observation of the surface of the sample that is cut through these two techniques, which is cut by using liquid nitrogen and cut due to tensile stress. The study was done from the observation of the SEM micrographs. The surface of the sample that is cut due to tensile stress exhibits a rougher and stretched surface compared to the sample that is cut by using liquid nitrogen.

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