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Synthesis and Characteristics of Acetylated Corn Cob Powder/ Unsaturated Polyester Composite

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K E Y W O R D S

Natural fiber, Corn cob, Untreated natural fiber, Treated natural fiber, Acetylated corn cob powder, Composite material, Cellulose acetate composite.

A B S T R A C T

In this research, the composite material was prepared from untreated and treated corn cob powder by acetylation process and the physical and mechanical properties have been studied. Cellulose is extracted using the Kürchner-Hoffer method and then acetylated. Untreated corn cob powder and Acetylated corn cob powder were mixed with unsaturated polyester resin in different concentrations (0, 1, 2, and 3 wt. %). The Mechanical and the physical test results showed that there is an enhanced in mechanical properties (Tensile, Impact, Hardness, and Bending) with the treatment (Acetylation) by increasing treated corn cob powder mass fraction (1, 2, and 3wt. %) respectively.

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1. Introduction

Cellulose is regarded as one of the generality numerous biopolymers on earth. Cellulose produced each year about (1.5 × 1012) tons. Thus, it shows up a biodegradable resource for raw materials and a massive amount of renewable [1]. Cellulose is mightily used due to its' lightweight, environment-friendly, and load-bearing in engineering applications [1, 2]. Cellulose, which containing composites may have more benefits among many industries, due to cellulose availability, and an improved CO2 balance with the environment in comparison with the composites made up of manufacture material, and good mechanical properties of cellulose composites [3-11]. Cellulosic composites are friendly environment materials and they are economically, also minify the pollution generation. Also, they are completely degradable fibers and natural resins to develop green composite materials. Petroleum products, In the past (synthetic polymers, resins, etc.) were generally used in composites, but nowadays researchers focusing more on green materials essentially cellulosic. Cellulosic fibers are used to replace synthetic fibers as reinforcement to make the products environmentally friendly [12]. The developers of bio green and green composites of composite applications have been because of increasing benefit in sustainable material concepts, environmental awareness. All that led to removing the chemical incompatibility between matrix and reinforcement by utilizing cellulose for both matrix and reinforcement [13]. The ingrained chemical incompatibility between hydrophilic cellulose and hydrophobic polymer matrix leads to mechanical strength and low stiffness of

composite due to an inactive stress transfer under load, which on the other hand happens because of weak interfacial bonding between the cellulosic and biopolymer components, especially in the case of thermoplastic biopolymers [14-16]. The chemical compatibility of the polymer matrix and cellulose can be improved by chemical treatments of the matrix or fiber, alkaline, and acetylation which provide different degrees of improvement. Also, can be increased Interfacial bonding by using cellulose in Nanosized forms such as cellulose whiskers, micro fibrillated cellulose, and bacterial cellulose, which they all provide an increase in surface area per volume [12, 17–23].

2. The aim of this study

The main objective of the work is to strengthen the unsaturated polyester resin with natural fibers (corn cob powder) which treated with three types of treatments (solvent treatment, nitric acid treatment, acetylation). The physical and mechanical properties of the prepared samples were used to evaluate the performance of the three treatments. The results were compared with untreated corn cob powder/ unsaturated polyester composite.

3. Materials and Methods

I. Preparation process

1- Removal corn grains: corn cob contains grains. It is required to remove manually to remain the cob free of grains, which is consider as a waste material, then dry it after making sure it clean and free from grains.
2- Cutting the sample (Corn Cob): After the removing process of the grains and drying the cob, corn cob was cut it into small cubes in order to prepare it for the next step the grains.
3- Grinding & Sieving: After cutting the sample into small pieces, then move on to the next step, grinding the small pieces cob as powder by using the electrical powder grinder. After grinding the sample into powder, sieving it to make sure it's the desired size, which is 0.45 mm as shown in Figure 1.

Figure 1: Corn cob as powder

After the preparation steps of the sample, which included removal of grains, cutting, drying, grinding, and sieving then processes of extraction cellulose and isolate it from extracts and lignin and that done by the following processes:
1- Using Soxhlet extractor to extract cellulose from corn cob powder with the solvents to remove the extractor and the solvents are Benzene: Ethanol with Volume ratio 1:1 and the process has been done for 7 Hours
2- Take the extracted corn cob powder from the flask of Soxhlet extractor and spread it on a tray and leave to dry at ambient temperature for 24 Hours.
3- Using Kürchner-Hoffer method to extract Cellulose by reaction (5gm) of cellulose with Ethanol: Nitric acid with volume ratio 4:1 for 1 hour and repeat the process three times to get white cellulose.
4- The obtained cellulose was then filtered through filter paper.
5- The obtained cellulose was washed with hot deionized water and dried overnight at 70 °C for 1hour in order to remove excess water, and then obtained cellulose as shown in Figure 2.

Figure 2: Cellulose

II. Acetylation process

Cellulose acetate is one of the commercially important cellulose derivatives, cellulose acetate is produced by the following processes:

1- Reaction (10gm) of cellulose that extracts from corn cob with (50ml) of glacial acetic acid and (100ml) of Toluene and (0.5ml) Perchloric acid, mixed them in a flask and put them on a magnetic stirrer for 1 hour. The reaction mixture became dense and the mixing process became difficult.

2- After 1 hour of mixing with a magnetic stirrer, add (100 ml) of de-ionized water to deposit of cellulose acetate that formed during the previous process and then filtering it.

3- Wash cellulose acetate with (100 ml) of hot de-ionized water for 4 times to remove the remaining ethanol from the previous step and then filtering it after making sure the cellulose acetate free from ethanol.

٤- Drying the filtered cellulose acetate in the oven at 40 °C for 8 hours.

٥- Grinding cellulose acetate as a powder with an electrical powder grinder as shown in Figure 3.

Figure 3: Cellulose acetate.

III. Casting process

Casting is a manufacturing process in which a liquid material is poured into a mold. Mold contains a cavity of the desired shape, the liquid is poured into the cavity and left to solidify and the solidified part is known as a "casting," casting materials are usually metal or polymers or mixing two or more components together; for examples plaster, epoxy, concrete, and clay. The casting mould has a tensile, impact, bending and thermal mould specimens. The mould has been made from silicone mould and they are free from defects.

IV. Preparation of composites

Untreated and Acetylated corn cob was prepared by mixing it with unsaturated polyester resin in a beaker. The mass fractions (0, 1, 2, and 3 wt. %) were used. The following formula was used in the preparation of composites:

$$vf = \frac{ms/ps}{\frac{ms}{ps}+mm/pm} \qquad (1)$$

Where:

m_s: the mass of powder corn cob (g)

m_m: the mass of matrix (g)

V_f: the powder corn cob mass fraction
P_m and p_s: the density of matrix and powder corn cob, respectively (g/cm^3)
The mixture was stirred continuously for 5 minutes, after which the hardener was added to the mixture with slow mixing for 5 minutes too until the mixture became homogenous. The mixture then poured into the mould and left for 24 hours at room temperature to solidify. After that, the cast was placed in an oven at 50 °C for 3 hours. Finally, the cast was left for 72 hours at room temperature. This process is important to the best coherency and reveals residual stresses. Figure4 shows the different samples of composite material prepared in this work.

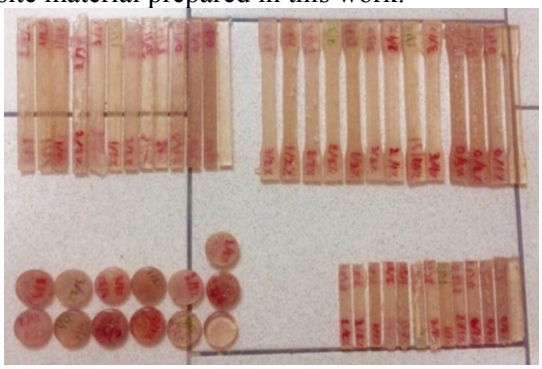

Figure 4: Composite samples after casting.

V. Tests

Mechanical properties tests (Tensile, Bending, and Hardness).
The tensile test was performed according to the international standard (ASTM D638-87b). The tensile test was carried out at room temperature by utilizing the universal tensile instrument type (LARYEE) with a capacity load (50 KN). The strain rate (speed of crosshead) was 2mm/min and the tensile load was applied gradually until fracture of the sample occurs. The length of the specimen used is (165 mm) with thickness of (7 mm). The tensile test was performed by a universal testing machine (UTM) as shown in Figure 5, with a loading capacity of 50 KN and a head speed of (5 mm/min).

Figure 5: Tensile test device.

The hardness test was performed on specimens with standard dimensions, by using (Shore D) device. As shown in Figure 6, the samples were disks with diameter 40 mm and height 5 mm, with applying 60 kg load on five different areas of the sample and take the average load.

Figure 6: Hardness test device.

The bending test was carried out according to the international standard (ASTM D 790 -03) as shown in Figure 5, The samples were measured according to the standard dimensions of 96 mm and 10 mm width and height of 4.8 mm.

Physical properties tests (Density, Thermal conductivity, and water absorption). In the density test, the composite samples were prepared according to the ASTM standard (D-792) and sample weights were measured according to the Archimedes method as shown in Figure 7 by accurate balances kind: PS 360/C/1 device.

The water absorption test was conducted by soaking the weighed specimens in a beaker containing distilled water at room temperature for 24 hours, the excess water on the surface of specimens was removed using a soft cloth. Then the samples were weighed using an electronic balance that shown in Figure 7.

From the difference of the final and initial weights, the percentage of water uptake was calculated by using the following expression:

$$\text{Water Absorption (\%)} = \frac{m2-m1}{m1} * 100\% \quad (2)$$

Where:

m_2: is the mass of the sample after soaking

m_1: is the mass of the sample before soaking

Figure 7: Density test device.

The thermal conductivity of the material was calculated by using "Lee's Disk method" as shown in Figure 8, In which heat is transferred from Heater to the disc that follows until it reaches the last disk, it is possible to measure the temperature of the three disks (T_A, T_B, T_C) by using the thermometers inside them.

The thermal conductivity of a sample in the form of a disk with diameter 40 mm and height 5 mm is then measured from the following equation:

$$K\left(\frac{T_B - T_A}{d_s}\right) = c\left[T_A + \frac{2}{r}\left(d_A + \frac{1}{4} \times d_s\right)T_A + \frac{1}{2r} \times d_s \times T_B\right] \quad \textbf{(3)}$$

As (e) represents the amount of heat passing through the disk per second it is calculated from the following equation:

$$IV = \pi \times r^2 (T_A - T_B) + 2\pi \times r \times e\left[d_A \times T_A + d_s \times \frac{(T_A + T_B)}{2} + d_B \times T_A + d_C \times T_C \right] \quad \textbf{(4)}$$

Where:
T_A, T_B, T_C: Disks temperature (A, B, C)
I: the current
r: disk radius
d: disk thickness
v: supply voltage

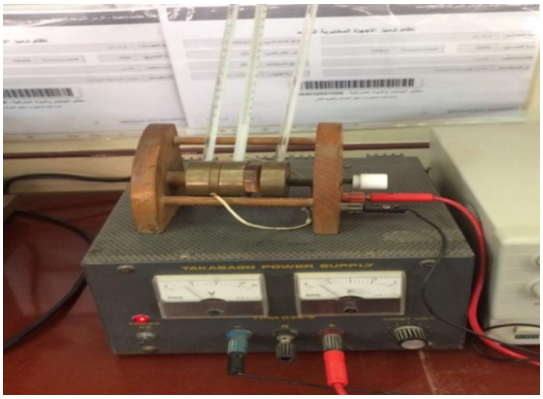

Figure 8: Thermal conductivity test device.

Microstructure and spectroscopy properties tests (SEM) were used to determine the composite properties. The surface morphology of the fracture surface of the tensile specimen was examined by Oxford Instruments scanning electron microscope device provided by Aztec that shown in Figure 9, the sample was cut into dimensions (0.5* 0.5* 0.5 cm) to fit into the device.

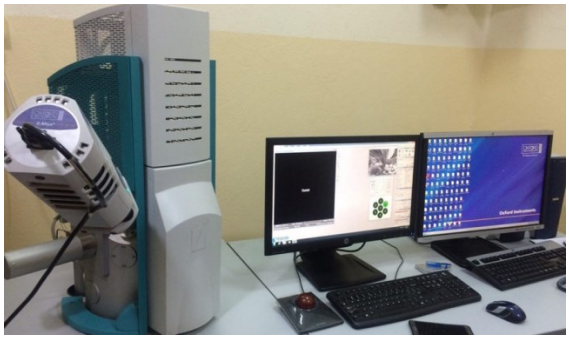

Figure 9: SEM test device.

4. Results and Discussions

I. Tensile test

The values of tensile strength of pure unsaturated polyester resin, unsaturated polyester resin reinforced by untreated and acetylated corn cob powder are illustrated in Figure 10. This figure shows the effect of the addition of untreated and acetylated corn cob powder with different mass fractions (0, 1, 2, and 3wt. %) on the tensile strength of unsaturated polyester resin/corn cob powder composites. As shown in Figure 10, the tensile strength of all samples decreases with increasing the mass fraction of corn cob powder in comparison with pure unsaturated polyester resin. The highest tensile strength for acetylated corn cob powder/ unsaturated polyester resin composite was (30, 27.8, and 24.3 Mpa) for (1, 2, and 3wt. %) mass fractions respectively.

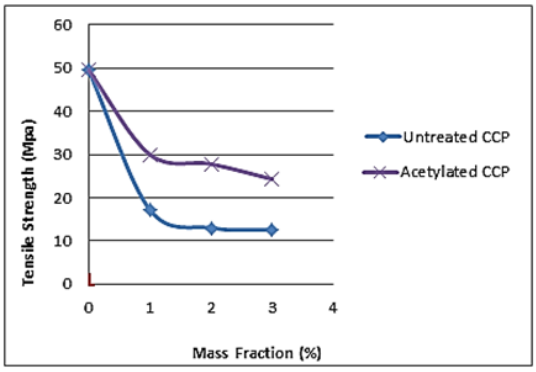

Figure 10: Tensile strength of Unsaturated Polyester Resin reinforced by different mass fractions of untreated and acetylated Corn Cob Powder.

II. Bending test results

The effect of the addition of untreated corn cob powder and acetylated corn cob powder to the unsaturated polyester resin with different mass fractions (0, 1, 2, and 3wt. %) on the flexural strength of the prepared composite materials are shown in Figure 11. From Figure 11, it can be noted that the flexural strength of untreated and acetylated corn cob powder/ unsaturated polyester resin composite decrease with increasing the volume fraction of corn cob powder in comparison with pure unsaturated polyester resin. Also, it is observed from the Figure 11 that the unsaturated polyester resin reinforced with an acetylated corn cob powder has higher values of flexural strength in comparison with unsaturated polyester resin reinforced by untreated corn cob powder. The reason behind such behavior is related to the good physical bonding between acetylated corn cob powder and the unsaturated polyester resin matrix and the bad wettability between untreated corn cob powder and unsaturated polyester resin matrix which have the lowest values of flexural strength. The highest flexural strength of acetylated corn cob powder/ unsaturated polyester resin composite was (3.1, 2.3, and 1.48 Mpa) for (1, 2, and 3wt. %) mass fraction, respectively. The improvement in flexural strength for acetylated corn cob powder/ unsaturated polyester resin composite in comparison with untreated corn cob powder/ unsaturated polyester resin composite were (72%, 10%, and 35%) for (1, 2, and 3wt. %) mass fractions respectively.

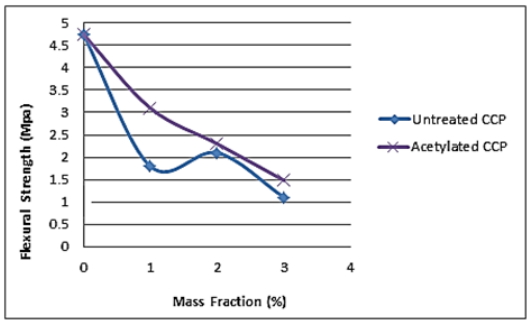

Figure 11: Flexural Strength of Unsaturated Polyester Resin reinforced by different mass fractions of untreated and acetylated Corn Cob Powder

III. Hardness test results

The hardness values of pure unsaturated polyester resin, unsaturated polyester resin reinforced by untreated and acetylated corn cob powder are illustrated in Figure 12. As shown in Figure 12, the hardness of the acetylated corn cob powder decreases with increasing the mass fraction of corn cob powder in comparison with pure unsaturated polyester resin. The reason behind such behavior is attributed to the formation of the strong structure of treated corn cob powder/ unsaturated polyester resin composite materials that depending on the formation of a powerful interface bonding between corn cob powder and unsaturated polyester resin, this leads to increasing the hardness of the treated/ unsaturated polyester resin composite materials. The highest hardness of the acetylated corn cob powder/ unsaturated polyester resin composite were (84.2, 86, and 89 Mpa) for (1, 2, and 3wt. %) mass fractions, respectively. The improvement in hardness for acetylated corn cob powder/ unsaturated polyester resin composite in comparison with untreated corn cob powder/ unsaturated polyester resin composite were (7%, 8.3%, and 9.2%) for (1, 2, 3wt. %) mass fractions, respectively.

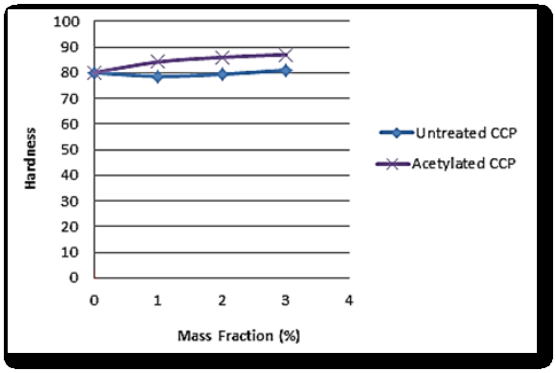

Figure 12: Hardness of Unsaturated Polyester Resin reinforced by different mass fractions of untreated and acetylated Corn Cob Powder

IV. Density test results

The density values of pure, untreated, and acetylated corn cob powder/ unsaturated polyester resin composite are illustrated in Figure 13. As shown in the figure, the density of all samples decreases with increasing the mass fraction of corn cob powder. Also, the density of acetylated corn cob powder/ unsaturated polyester resin composite is higher than that for untreated corn cob powder/ unsaturated polyester resin composite. This result can be attributed to the good wettability and uniform distribution of corn cob powder in the case of acetylated corn cob powder. The highest density of acetylated corn cob powder/ unsaturated polyester resin composite material was (1.243, 1.24, and 1.237 g/cm^3) for (1, 2, and 3wt. %) mass fraction, respectively. The percentage of increase in density for acetylated corn cob powder/ unsaturated polyester resin composite materials in comparison with untreated corn cob powder/ unsaturated polyester resin composite were (0.65%, 0.5%, and 0.45%) for (1, 2, and 3wt. %) mass fraction, respectively.

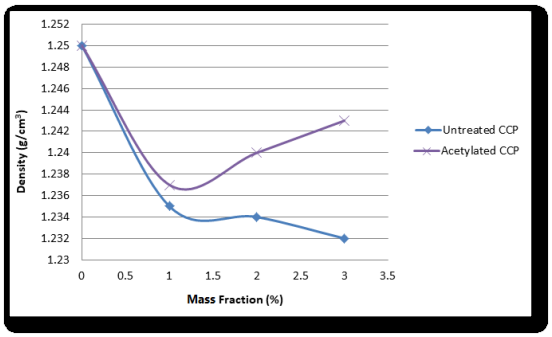

Figure 13: Density of Unsaturated Polyester Resin reinforced by different mass fractions of untreated and acetylated Corn Cob Powder.

V. Water absorption test results

As shown in Figure 14, the water absorption percent increases with increasing the mass fraction of corn cob powder and acetylated corn cob powder/ unsaturated polyester resin composite materials.
Figure 14, also shows that the water absorption percent of acetylated corn cob powder/ unsaturated polyester resin samples. This is due to the hydrophilic nature of corn cob powder, the affinity of corn cob powder towards the moisture, and also may be due to the high moisture absorption level of natural fibers in the polymer matrix that result from polar hydroxide groups in the fiber.
The lowest water absorption percentages for acetylated corn cob powder/ unsaturated polyester resin composite materials are (1.3%, 1.5%, and 1.7%) for (1, 2, and 3wt. %) mass fractions of corn cob powder, respectively.
The percentages in the reduction of water absorption percent of acetylated corn cob powder/ unsaturated polyester resin samples in comparison with untreated corn cob powder/ unsaturated polyester resin samples are (61%, 42%, and 19%) for (1, 2, and 3wt. %) mass fractions, respectively.

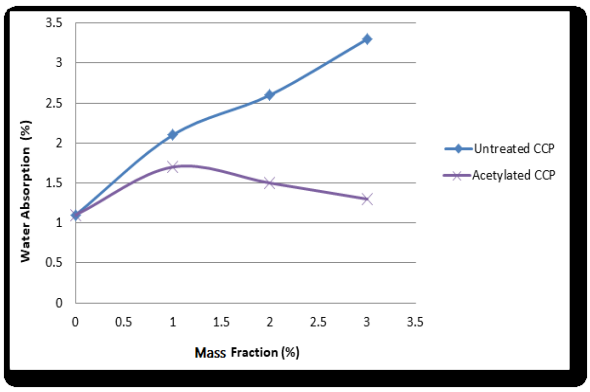

Figure 14: Water absorption of Unsaturated Polyester Resin reinforced by different mass fractions of untreated and acetylated Corn Cob Powder

VI. Thermal conductivity test result

As shown in Figure 15, the thermal conductivity increases with an increase in the mass fraction of corn cob powder for acetylated corn cob powder. The reason behind such behavior is attributed to the interaction of the treated corn cob powder with the chains inside the unsaturated polyester resin matrix to prevent the insulating influence of the unsaturated polyester resin matrix through forming pathway and allowing the heat transmission from one side of samples to another which mean an increase in thermal conductivity. The thermal conductivity of acetylated corn cob powder/ unsaturated polyester resin composite materials is high, this may be attributed to that acetylated corn cob powder samples have much interaction between corn cob powder and unsaturated polyester resin chains. The highest thermal conductivity is for acetylated corn cob powder/ unsaturated polyester resin composite (1.04, 1.08, and 1.13 W/m. °C) for (1, 2, and 3wt. %) mass fractions, respectively. The percentage of increase in thermal conductivity for acetylated corn cob powder/ unsaturated polyester resin composite in comparison with untreated corn cob powder/ unsaturated polyester resin composite is (30%, 42%, and 55%) for (1, 2, and 3wt. %) mass fractions, respectively.

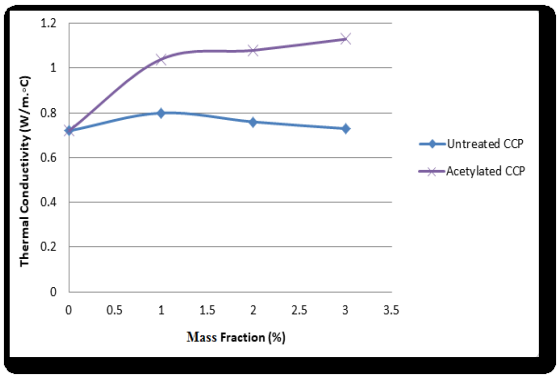

Figure 15: Thermal conductivity of Unsaturated Polyester Resin reinforced by different mass fractions of untreated and acetylated Corn Cob Powder.

VII. Scanning Electron Microscope (SEM)

Figure 16 shows the SEM micrograph of the tensile fracture surface of unsaturated polyester resin in magnification (2.05k x), Figure 17 shows the SEM micrograph of the tensile fracture surface of untreated corn cob powder in magnification (198x), and Figure 18 shows the SEM micrograph of the tensile fracture surface of the acetylated corn cob powder unsaturated polyester resin composites in magnification (500x) in (3 wt. %) mass fraction of all samples. Figure 16 shows very little pull out of polyester resin, this is due to the tensile fracture surface of unsaturated polyester resin. Figure 17 shows a splitting between the polyester resin and untreated corn cob powder. Also, it observed that some matrix material adheres to surface of pulling out the fibers indicating a good bond and interfacial adhesion between the matrix and the fiber, this is due to incompatibility between natural fiber and unsaturated polyester resin due to the hydrophilic nature of natural fiber while polyester

resin has hydrophobic nature, therefore, it is important to treat the natural fiber to enhance interaction between matrix and reinforcement [24]. The value of fracture is generally lower for treating fiber (Acetylated corn cob powder) composite as shown in Figure 18 when compared with untreated fiber composites.

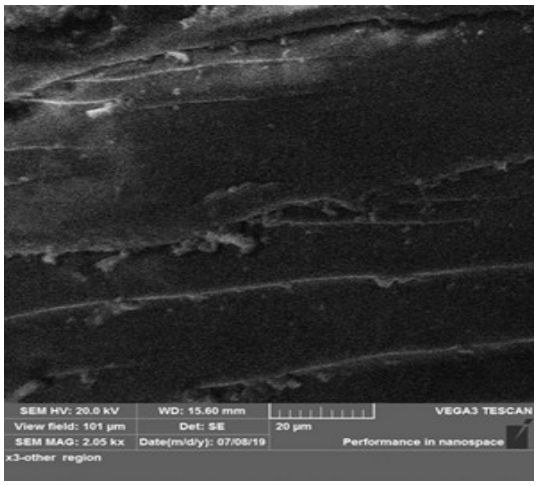

Figure 16: SEM micrograph of the tensile fracture surface of unsaturated polyester resin.

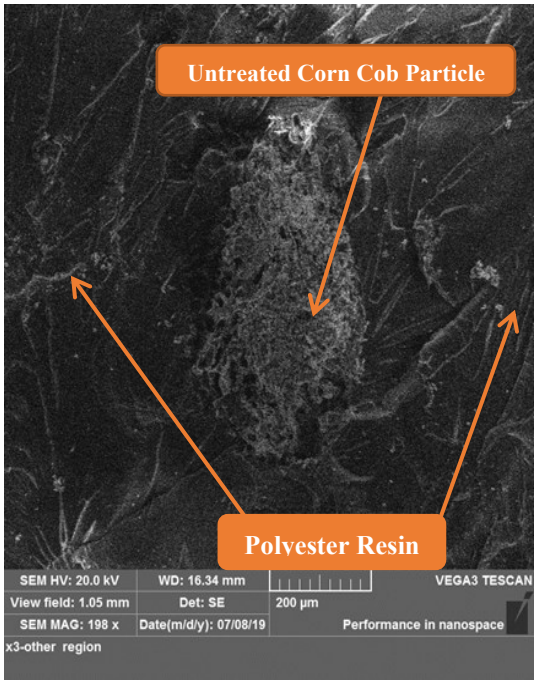

Figure 17: SEM micrograph of the tensile fracture surface of untreated corn cob powder unsaturated polyester resin composite.

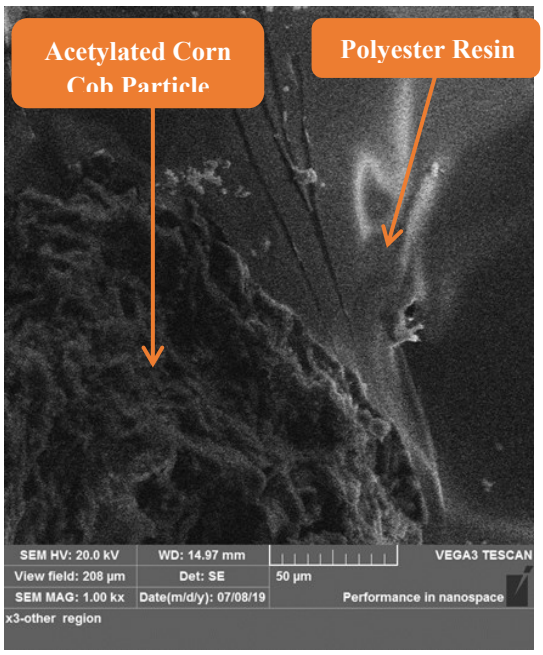

Figure 18: SEM micrograph of the tensile fracture surface of acetylated corn cob powder unsaturated polyester resin composite

5. Conclusions

1- Mechanical properties test results showed that there is an enhance in mechanical properties (Tensile, Impact, Hardness, and Bending) due to the treatments (Solvent treatment, Nitric acid treatment, and Acetylation) by increasing the treated corn cob powder mass fraction (1, 2, and 3wt. %), respectively.

2- Physical properties testing results (Density, Water absorption, and Thermal conductivity) showed that there is a decreasing in density during each treatment (Solvent treatment, Nitric acid treatment, and Acetylation) by increasing treated corn cob powder mass fraction (1, 2, and 3wt. %) respectively, while there is increasing in thermal conductivity and water absorption for each treatment and by increasing in mass fraction

3- SEM showed that as increasing the chemical treatment to natural fiber, compatibility between matrix and reinforcement increased.

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