

MECHANICAL AND PHYSICAL PROPERTIES OF THERMOPLASTIC CORN STARCH

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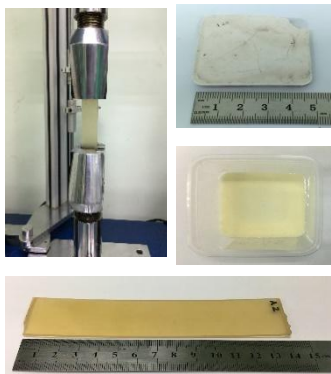
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Graphical abstract



Abstract

The awareness to produce biodegradable composite has increased rapidly because of non-toxic and reachable. However, fully biodegradable composite production still low due to the matrix used in the composite is not biodegradable. Thus, this paper presents the study on mechanical and physical properties for the mixtures of corn starch (CS) with different weight percentages of glycerol as thermoplastics corn starch (TPCS) matrix. The selected glycerol contents were at 30, 35 and 40 wt%. The mixtures of CS and different weight percentages of glycerol were made using hot compression moulding at 165°C for 15 minutes to produce the TPCS samples. The mechanical and physical properties were done: the tensile test, hardness test, water absorption test, moisture content test and microstructure analysis under the Scanning Electron Microscopes (SEM). Incorporating 30 wt% loadings of glycerol has increased the tensile strength and hardness. The results show that the addition of higher than 30 wt% loadings of glycerol has decreased the tensile strength and hardness of the TPCS. The physical test results for 30 wt% loadings of glycerol for water absorption test and moisture content show the lowest value than other TPCS samples. However, the density value for all wt% loadings of glycerol does not offer much difference. It reveals that 30 wt% loadings of glycerol in the mixture of CS have shown a good interaction in the TPCS mechanical properties. Based on this finding, the TPCS has huge potential to be used as a matrix to develop a fully biodegradable composite.

Keywords: Thermoplastic, corn starch, glycerol, biodegradable, composite

Abstrak

Kesedaran untuk menghasilkan komposit terbiodegradasi meningkat dengan cepat kerana tidak beracun dan mudah diperolehi. Walau bagaimanapun, pengeluaran komposit terbiodegradasi sepenuhnya masih rendah kerana pengikat yang digunakan dalam komposit tidak terbiodegradasi. Oleh itu, kajian ini mengemukakan sifat mekanikal dan fizikal bagi campuran kanji jagung (CS) dengan peratusan berat gliserol yang berbeza sebagai pengikat kanji jagung termoplastik (TPCS). Kandungan gliserol yang dipilih adalah pada kadar berat 30, 35 dan 40%. Campuran CS dan peratusan berat gliserol yang berbeza dibuat dengan menggunakan mampatan panas pada suhu 165°C selama 15 minit untuk menghasilkan sampel TPCS. Sifat mekanik dan fizikal dilakukan: ujian tegangan, ujian kekerasan, ujian penyerapan air, ujian kandungan kelembapan dan analisis struktur

mikro di bawah Pengimbas Elektron Mikroskop (SEM). Kemasukkan 30% berat gliserol telah meningkatkan kekuatan tegangan dan kekerasan. Hasil kajian menunjukkan bahawa penambahan gliserol yang melebihi 30% berat telah menurunkan kekuatan tegangan dan kekerasan TPCS. Hasil ujian fizikal untuk 30% berat gliserol untuk ujian penyerapan air dan kandungan kelembapan menunjukkan nilai terendah daripada sampel TPCS yang lain. Walau bagaimanapun, nilai ketumpatan untuk semua berat gliserol tidak memberikan banyak perbezaan. Ini menunjukkan bahawa 30% berat gliserol dalam campuran CS telah menunjukkan interaksi yang baik dalam sifat mekanik TPCS. Berdasarkan penemuan ini, TPCS berpotensi besar untuk digunakan sebagai pengikat untuk membuat komposit yang dapat terbiodegradasi sepenuhnya.

Kata kunci: Termoplastik, kanji jagung, gliserol, biodegradasi, komposit

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1.0 INTRODUCTION

Due to increased human populations, plastic products from petroleum-based polymers are developing rapidly and used in vast engineering applications such as automotive, furniture and packaging industries [1]–[6]. The disposal of these plastic products from petroleum-based polymer becomes a significant issue and contributes to environmental pollution [3], [7], [8]. In addition, global warming and oil depletion are the reasons that attracted attention from researchers in various fields to investigate the production of biopolymer materials [9]–[12]. There is increasing interest from researchers to utilize the available natural resources to develop a more environmentally friendly polymer to overcome the pollution of the environment, global warming, and oil depletion issues and replace conventional materials. However, this kind of biopolymer materials primary problem is that it is not suitable for a long-term application [13], [14].

The biopolymer materials resources, which are environmentally friendly polymers, are natural plants such as soy, cellulose and starch [15]. Among these biopolymer materials, starch is one of the most versatile, inexpensive, non-toxic, biodegradable and sustainable materials available [16], [17]. Zou *et al.* [18] reported that starch, a heterogeneous material, contains two microstructures: amylose and amylopectin. Amylose has a long linear chain structure of α -1,4 linked glucose units, whereas amylopectin has a large molecular weight and highly branched structures consists of much shorter chains of α -1,4 chains linked by α -1,6 bonds [19]. Most starches contain 20 to 30% amylose and 70 to 80% amylopectin [20]. The amylopectin ratio to amylose in starch significantly influences the functional properties of the starch [21]. Cassava, corn, potato and sago and rice are the most familiar forms of starch used to produce biopolymer [22].

Corn is also known as Maize is a large grain plant. Some corn production is used for corn starch (CS), corn syrup and other corn products such as animal feed and corn ethanol [23]. CS has (72%)

amylopectin and (28%) amylose content. The amylose content of CS is more than cassava (17%) and potatoes (25%) [24]. Based on the literature [25], it is known that the degree of polymerization efficiency depends on the amylose content of the starch [18]. Since it is simple, fast and cost effective, blending with other materials is an alternative method to diversify starch properties. In plasticizers such as glycerol, water or sorbitol and heat, starch undergoes spontaneous destructurization and creates homogeneous melt formation, namely Thermoplastic Starch (TPS) [26][27]. Although TPS accomplishes environmentally friendly characteristics, it retains some limitations in its application due to physical and mechanical characteristics [2], [13], [28].

Several studies have investigated the characterization plasticization effect of glycerol on corn starch to prepare the thermoplastic corn starch films [27], [29]–[34]. However, all these studies were focus on the developing thermoplastic corn starch by using the solution casting method in producing thin-film samples which have the limitations on their potential application. In this study, the thermoplastic corn starch was developed using the hot press method to produce sheets plate samples. It should be noted that the corn starch and glycerol used in this study were not chemically treated or modified, which would lead to the development of more environmentally friendly and cheaper production processes.

This study aimed to study the effect of glycerol mixture with corn starch on mechanical and physical properties and morphological. Different weight percentage ratios (wt%) were used to examine the effect of corn starch mixture with glycerol. Various experimental methods such as tensile test, hardness test, density, moisture content, water absorption, and SEM analysis have been used to describe the properties of the mixture. This may be an initial step to promote further research using this method for the fabrication of thermoplastic corn starch.

2.0 METHODOLOGY

2.1 Materials

Corn starch powder and glycerol (Q-rec G4018-1-2500) were obtained from Polyscientific Enterprise Sdn. Bhd.

2.2 Sample Preparations

The samples were prepared using high speed mechanical stirrer model AM300S-P to mix CS with glycerol to produce a mixture of CS and glycerol. In order to achieve the best properties from the mixture of CS and glycerol, different wt% loadings of glycerol were added into the CS. As tabulated in Table 1, 20, 25, 30, 35, 40,45 and 50 wt% loadings of glycerol were added into CS. However, the mixtures for 20 and 25 wt% loadings of glycerol were maintained in the powder form and most brittle as shown in Figure 1. Whereas the 45 and 50 wt% loadings of glycerol were produced in liquid form as shown in Figure 2. It is understood that both powder and liquid forms are not suitable to be made into sheets plate for testing such tensile, hardness and many more. As such the 30, 35 and 40 wt% loadings of glycerol were selected for further testing.

Table 1 Appearance of mixture corn starch and glycerol in different weight percentage (wt%)

Corn Starch (%)	Glycerol (%)	Appearance
50	50	Liquid
55	45	Liquid
60	40	Sheets Plate
65	35	Sheets Plate
70	30	Sheets Plate
75	25	Powder
80	20	Powder

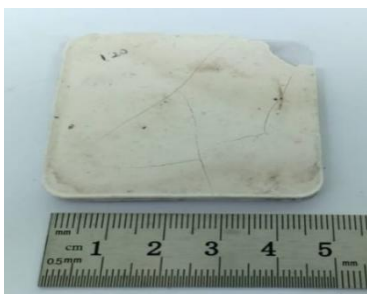


Figure 1 Sample in powder form

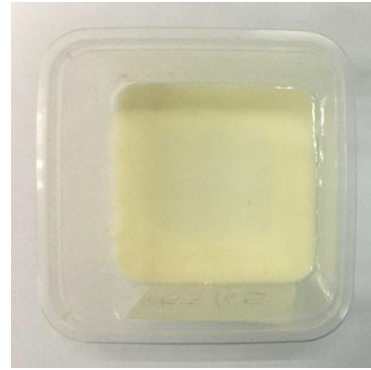


Figure 2 Sample in liquid form

In order to produce a sheets plate with a thickness of 3 mm, the mixtures of CS and glycerol needed to pass through a thermo-pressed. For this purpose, the mixtures were preheated for 15 minutes and pressed for 15 minutes at the temperature of 165 °C under the load of 20 kg/cm² by using Go-tech (GT-7014-A30) hydraulic thermo-press. The same process had been utilized for the preparation of other types of CS mixtures [9]. In this study, five samples were prepared for each wt% loading of glycerol.

2.3 Tensile Testing

Tensile tests were carried out in compliance with ASTM D 3039: Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials. The same sample size had been utilized in the literature [1]. The tests were repeated five times where the specimen dimension was 140 mm x 13 mm x 3 mm as shown in Figure 3. The test was conducted using Universal Testing Machine (Model HZ-1003) controlled with 1 kN load and operated at constant crosshead speed tests of 1 min/mm.

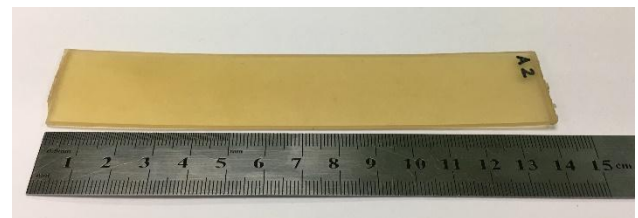


Figure 3 Sample for tensile testing

2.4 Hardness Testing

The hardness of the specimens was measured by using a micro-Vickers hardness tester (Model HMV-G21DT). Hardness tests were conducted according to ASTM E384. The indentation depth was measured on five samples (n=5) for each mixture and the average value was calculated. Due to the materials hardness characteristic appearing to shift at the

edges of the samples, the test was conducted at least 12 mm away from the boundary [35][36].

2.5 Density

A digital electronic densimeter (MD-300S) was used to measure the density of the sample. Five samples for each mixture were used and the average value was calculated. The Density tests were conducted according to ASTM D792.

2.6 Moisture Content

Five samples ($n=5$) with a dimension of $10 \times 10 \times 3$ mm (length \times width \times thickness) for each mixture were prepared to determine the moisture content. All samples were heated to 105°C in the oven for 24 hours. The sample weight before, M_i , and after, M_f , the heating was calculated in order to determine the moisture content [13]. The level of moisture content was calculated with Eq. (1).

$$\text{Moisture content \%} = \frac{M_i - M_f}{M_i} \times 100 \quad (1)$$

2.7 Water Absorption

In order to release existing moisture, five samples ($10 \times 10 \times 3$ mm) were dried in an air circulating oven at $105 \pm 2^\circ\text{C}$ for 24 hours and then submerged in water at room temperature ($23 \pm 1^\circ\text{C}$) for 0.5 and 2 hours, as suggested by [37]. The samples were weighed before, W_i and after immersion, W_f and the water absorption of the samples were determined with Eq. (2).

$$\text{Water content \%} = \frac{W_f - W_i}{W_i} \times 100 \quad (2)$$

2.8 Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) model JEOL, JSM 6010 PLUS/LV with an acceleration voltage of 8 kV, was utilized to observe tensile fracture surface morphology. The specimen was carried out with platinum coating before being observed under SEM because the sample was nonconductive material.

3.0 RESULTS AND DISCUSSION

3.1 Tensile Properties

Figure 4 shows the effect of glycerol addition on tensile strength. The tensile strength of corn starch blended with glycerol shows significant improvement with decreasing glycerol loading (wt%). Based on different glycerol ratios, the decreasing glycerol loading tends to cause higher strength of thermoplastic CS. The highest strength around 1.43 MPa is obtained by adding 30 (wt%) glycerol to the corn starch. When

glycerol 40 (wt%) is loaded to a higher weight, the tensile strength is slightly reduced to 0.47 MPa and this observation similar to another study on the effect of glycerol [38]. According to Zhang *et al.* [39], thermoplastics starch has low tensile properties, typically below 6 MPa in tensile strength. Based on previous studies, the value of tensile strength for Cassava starch (1.4 - 1.6 MPa)[40], Pea starch (1.4 - 5.8 MPa)[41], Potato starch (3 MPa)[42] and Rice starch (3.2 MPa)[43].

In addition, the higher tensile strength of thermoplastic CS is due to two main reasons. Firstly, the effect on amylose content in the corn starch itself and secondly is the added amount of plasticizer (glycerol) into the mixture. The amylose content in corn starch (28%) is larger than others such as potato (20–25%), rice (20%), cassava (17%) and waxy rice (5%) [44]. Prachayawarakorn *et al.* noticed that the mixtures higher amylose content resulted in a higher degree of polymerization [44]. They found that higher amylose content in rice starch contributed to higher tensile strength than waxy rice [45]. Besides that, Prachayawarakorn *et al.* also concluded that the amount of plasticizer affected the tensile strength. This conclusion was obtained by comparing other studies using different amounts of plasticizer/starch ratios such as 50:50 [45], 65:35 [46] and 70:30 [47].

Development of TPS with lower loading (wt%) of glycerol produces greater strength and rigidity in conjunction with low ductility. The higher amount of plasticizing usage decreases the strength due to the plasticizing effect, which reduces the strong intramolecular in the starch chains [48]. This observation can be explained based on the SEM results where low glycerol presents a more rigid surface compared to high glycerol. Furthermore, the different manufacturing methods and parameters used are other factors that may influence the dissimilarity of the mechanical characteristics as the results obtained.

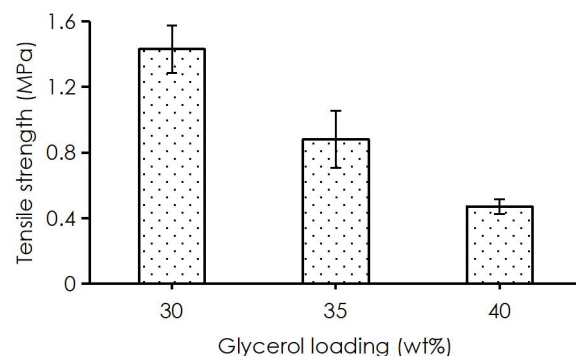


Figure 4 Tensile strength of corn starch blended with glycerol

The effect on elongation of glycerol addition in CS was demonstrated in Figure 5. The elongation of corn starch blended with glycerol showed significant

improvement with increasing glycerol content. The lowest elongation was obtained from the 30 wt% of glycerol loading which is 10.92%. At higher glycerol loading (40 wt%), the elongation was increased to 14.96%. According to the results, elongation at break and tensile strength were inversely correlated. The less plasticizing effect imparted by glycerol in the corn starch blend will be due to the sample becoming brittle or less flexible. These findings are in agreement with previous studies [20], [29], [48]–[50]. The increase in elongation is due to plasticizers (glycerol) decrease the intermolecular bonds between amylose-amylopectin of the starch and thus, substitute them with hydrogen bonds. Such disruption and reconstruction of starch molecular chains reduce the rigidity and enhances flexibility [31], [48], [51].

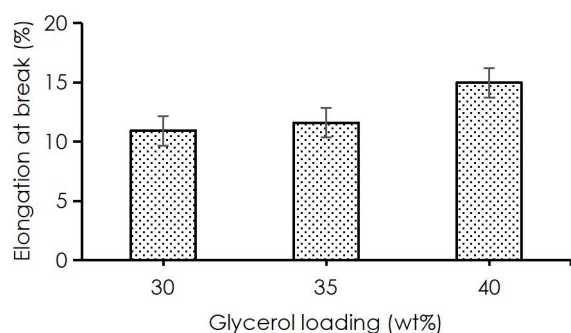


Figure 5 Elongation at break of corn starch blended with glycerol

3.2 Hardness

Table 2 shows the hardness test data results for three different compositions of cornstarch blended with glycerol. This table clearly shows that the hardness increases when the loading (wt%) of glycerol decreases for all samples. The obtained hardness result for loading (wt%) of 40 glycerol has the lowest value with an average of 2.404 from the five samples. Meanwhile, the loading of 30 g with% glycerol has the highest value of hardness, which is 5.8785. Hardness is related to the strength and toughness of the composite [1]. This result shows a significant change with the obtained tensile strength as in Figure 1, where the loading (wt%) of 30 glycerol also shows the highest value. This is caused by the lower glycerol usage that produces higher strength and stiffness along with low ductility. This finding is in agreement with previous studies that reported the increasing content of glycerol caused the hardness to decrease [52].

Table 2 Hardness test for the mixture of CS and glycerol based on the ratio of CS and glycerol

Sample	Thermoplastics CS		
	70/30	65/35	60/40
1	6.1726	4.8747	2.0716
2	6.0392	4.6877	2.4445
3	5.2577	4.6997	2.1770
4	6.0130	4.9910	2.8068
5	5.9100	4.9131	2.5230
Average	5.8785	4.8332	2.4046

3.3 Density

Table 3 shows the density of corn starch blended with glycerol. In general, a slight increment of density is evidenced by decreasing the glycerol loading (wt%). The mixture of 60/40 loading (wt%) shows the lowest density compared to other mixtures with 1.375 g/cm³. Moreover, the mixture with the lowest glycerol loading (wt%) shows the highest density value. This finding can be associated with the structure network between the corn starch and glycerol itself, which affect the density value. In addition, the amylose and amylopectin molecules chain inside the CS influences the density value. Amylopectin chain forming a double helical crystalline structure and amylose present as a helical complex with the lipids in the starch granule [53]. The orientation of amylose and amylopectin molecules inside a starch granule is very compact, and there is no space for water or any other molecule to enter [54]. Therefore, the increase in corn starch content increases the content of amylose and amylopectin. An increase in the corn starch content causes the increment of the density value. It can be said that the higher loading (wt%) of corn starch can affect the density of the mixture. These results are similar to the observed in the literature by other studies [55], [56].

Table 3 Physical properties of corn starch/glycerol

Thermoplastics CS	Density (g/cm ³)	Moisture Content (%)
70/30	1.404	24.60
65/35	1.396	27.22
60/40	1.375	28.77

3.4 Moisture Content

The moisture content of the corn starch blended with glycerol is shown in Table 3. Generally, the moisture content results slightly decrease when the loadings (wt%) of glycerol are decreased. This indicates that the moisture content results are directly proportional to glycerol loading (wt%) [27]. However, further incorporation of 40 (wt%) of glycerol in corn starch has shown the highest moisture content value among other mixtures due to corn starch and glycerol

behaviour, which are hydrophilic materials. Meanwhile, the moisture content for loading of (wt%) 65/35 slightly decreases to 27.22% from the highest of 28.77%. Furthermore, the composition for loading (wt%) 70/30 has shown the lowest moisture content, which is only 24.6%. In discussing the hydrophilic behaviour, the mixture of corn starch and glycerol becomes more hydrophilic with an increased plasticizer (glycerol). Therefore, similar studies have shown that the moisture content increases when adding more plasticizers [38], [56].

3.5 Water Absorption

Studies indicate that bio-based materials are more vulnerable to water; thus, these materials must be tested in this study to assess the water absorption characteristics of the blended corn starch and glycerol [7]. Figure 6 indicates the water absorption potential of the mixture after 0.5 hours and 2 hours of immersion. It shows that a longer immersion time leads to more excellent water absorption for all mixtures due to the higher amount of water that the materials can absorb. By a complete half hour of immersion, it's possible to decide that the mixture of 70/30 ratio yields the lowest possible value on water uptake. Further incorporation of glycerol in corn starch loading (wt%) of 70/30 ratio continues to show lower water uptake than other mixtures after 2 hours of immersion. In contrast, the highest water uptake is shown by the mixture of 60/40 loading (wt%) ratio. This ratio continually shows the highest reading after 0.5 hours until 2 hours of immersion than other mixtures. The difference in water uptake between the 70/30 mixture and the 60/40 mixture is more evident after 2 hours of immersion, which are 66.03% and 40.43% respectively. It relates to the weak corn starch molecule interactions, which are dominant at higher plasticizer concentrations, resulting in a less dense starch network and structure. This observation is also reported by previous authors [48].

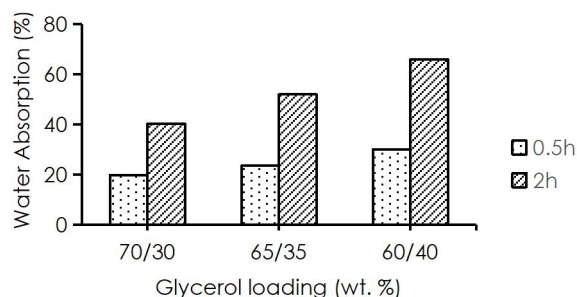


Figure 6 Water absorption for corn starch/glycerol

3.6 Morphological Properties

A scanning electron microscope (SEM) was used to analyze the surface morphology. The scanning electron microscopy allows a description of the corn

starch structure's behavior with different glycerol ratios. It is known that starch granules break down and form a continuous phase with glycerol in the thermoplastic CS [13]. Figures 7.1, 7.2 and 7.3 show that the corn starch and glycerol formed a homogenous surface with minimum clusters or agglomeration. After incorporating glycerol into corn starch, the blends are shown to have a smooth surface with small pores and cavities. This indicates good interaction between corn starch and glycerol with a minimum cluster of 40 wt% loadings of glycerol. However, the mixture for 35 wt% loading glycerol clearly shows visible connecting clusters while the number of clusters increases for the 30 wt% of glycerol in the mixture. This result indicates that the mixture with higher loading (wt%) of glycerol shows a minimum of clusters rather than the lowest loading (wt%) of glycerol and this observation is aligned as mentioned in previous literature [20]. This phenomenon is due to the glycerol used in this research in liquid form, so when the higher loading (wt%) of glycerol, the homogeneous composition is easy to form. In addition, the passage that exists between clusters is due to the processing technique that is used to mix the corn starch and glycerol.

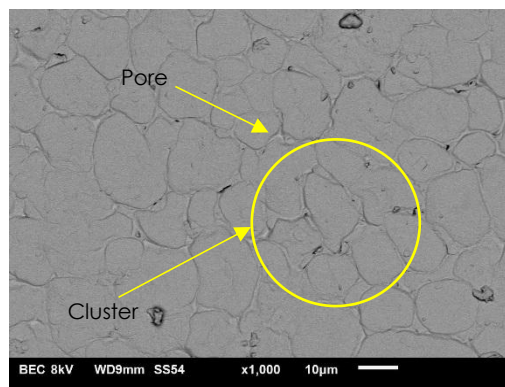


Figure 7.1 30 wt% of glycerol

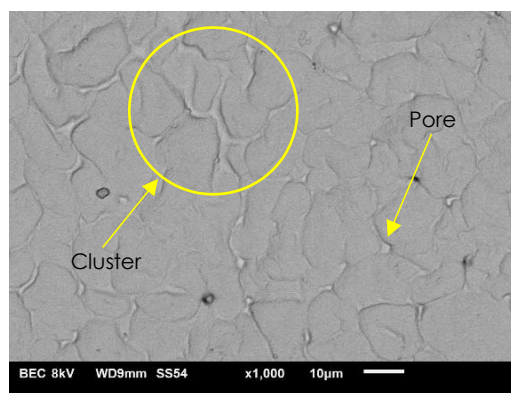


Figure 7.2 35 wt% of glycerol

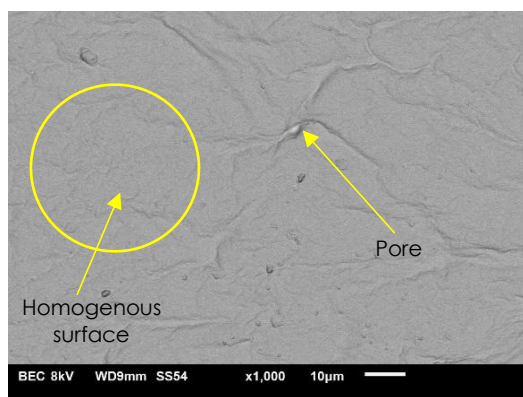


Figure 7.3 40 wt% of glycerol

4.0 CONCLUSION

In this study, biopolymer fillers or matrix from mixed corn starch and glycerol were fabricated with the laboratory mixer and hot press machine. The findings show that corn starch and glycerol are well-suited. The introduction of glycerol (up to 30 wt%) able to increase the mechanical properties of corn starch such as tensile strength and hardness. Compared to other mixtures, corn starch blended with 30 wt% glycerol displays the highest tensile strength value. Meanwhile, corn starch mixed with 40 wt% glycerol frequently shows the lowest tensile properties (tensile strength), hardness, and physical properties. Based on this indicator, corn starch blended with 30 wt% glycerol has vast potential to be biopolymer matrix. It can reinforce natural fiber from various sources such as pineapple leaf fiber, kenaf, banana leaf, bamboo, coconut, etc. to form a fully biodegradable composite.

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