

# A SUSTAINABLE APPROACH OF THERMOPLASTIC FILMS FROM TACCA LEONTOPEALOIDES STARCH REINFORCED WITH BIOCHAR RICE HUSK

A. M. Mohd Amin<sup>1, a</sup>, S. Mohd Sauid<sup>2, b</sup>, M. Musa<sup>3, c</sup>,  
K. H. Ku Hamid<sup>4, d</sup>

\*Corresponding author :  
aeesyamardhiah@gmail.com

<sup>1,2,3,4</sup>Faculty of Chemical Engineering, Universiti Teknologi  
MARA 40450 Shah Alam, Selangor, Malaysia

<sup>a</sup>aeesyamardhiah@gmail.com, <sup>b</sup>suhaila1706@gmail.com,  
<sup>c</sup>mohibah@salam.uitm.edu.my <sup>d</sup>kuhalim@salam.uitm.edu.my

## Abstract

The objective of this study is to investigate the properties of thermoplastic starch (TPS) films derived from *Tacca leontopetaloides* and biochar rice husk (RH) in chemical, morphological and water absorption. The films properties were characterized by FT-IR, SEM and water uptake test. In this study, the films were prepared by varying the glycerol content (v/v) at 5%, 10% and 15% through solution casting method and sulphur vulcanization method to produce TPS. While, TPS reinforced with bio-filler was further blended with 5wt% of biochar RH using the identical methods to produce TPS-BChr films. From the experiment, it was found that the varying amount of glycerol significantly affect the chemical structure, morphology and water absorption properties of films. FT-IR had indicated that increasing of glycerol content was displaced the peak of TPS and TPS-BChr. It had also confirm the existence interaction of biochar composite onto the TPS film. Besides, the morphological study of TPS and TPS-BChr had showed smoother surface, homogenous and good distribution with increasing of glycerol content. In water uptake analysis, increasing of glycerol tended to increase the affinity of water due to the hydroscopic properties. Though, addition of biochar RH had lowered the water uptake because biochar has aliphatic group that induced TPS-BChr more hydrophobic. From the results revealed that films with glycerol content at 10% has superior combination to blend with *Tacca leontopetaloides* starch and had demonstrated the good properties with addition of biochar as bio-filler.

**Keywords:** *Tacca leontopetaloides*; Thermoplastic starch; Biochar; Glycerol

## 1.0 INTRODUCTION

Biobased plastics from renewable resource approach a vastly interesting field for various applications in packaging and agricultural. The prominent renewable resource use for bioplastics production is starch. Starch has receiving a lot of

attention because it has natural carbohydrate storage in plant that form intracellular granules [1]. The intracellular granules is composed by two molecules; amylose and amylopectin which has formed in linear polysaccharides and branched

polysaccharides respectively. These two components are able to improve the biobased plastic properties to be flexible and digestible [2]. Starch has been considered as an excellent alternative to substitute or replace the synthetic polymer due to its abundance, low cost, renewable and biodegradable [3]. Furthermore, the approach of starch in bioplastic production is relevant for various conventional processes such as melt-mixed, extrusion, compression and molding. This is because, starch is able to conditioned under heat and shear to transform the granule of molecules into polymeric amorphous phase with suitable plasticizer to produce thermoplastic starch (TPS) material [4]. Many research works have devoted to study the blend of starch as polymer-based material to reveal the ductility and biodegradability of the material [5]. Previously, much works have used starch to produce TPS from food crops such as corn, sago, potatoes and cassava to evaluate their properties and functionalities [4], [6]-[8]. Despite that, no study has investigate *Tacca leontopetaloides* as starch based polymer for TPS production.

A new promising starch resource, *Tacca leontopetaloides* or known as Polynesian arrowroot is a starchy food material has wildly grow in tropical regions of Africa, South-East Asia and also distributed in the Pacific island region [8]-[9]. *T. leontopetaloides* starch belongs to Dioscoreaceae family has underground tuber, relatively hard and potato-like. *T. leontopetaloides* starch is an essential food medium to serve as food source for living people in Pacific island because consists of  $\pm 88.07\%$  of carbohydrates [8]. In ancient history, tuberous of *T. leontopetaloides* is popular as indigenous traditional medicines in India to cure the stomach ailment as it is containing of taccalin based bitter flavonoid, highly nutritious and good for dietary [9]-[11]. In contrast, *T. leontopetaloides* starch has potential as starch-based to produce TPS film because it has amylose and amylopectin components which about 22.5% and 77.5% respectively and has similar composition with corn starch [9]. In Malaysia, *T. leontopetaloides* plant is native and mainly found in east coast of Peninsular Malaysia and not being exploited nor harvested because *T. leontopetaloides* starch is not staple food source among Malaysians [10]-[11]. In previous literature, *T. leontopetaloides* starch was used for floor mat application. This author had revealed that *T. leontopetaloides* starch has highly incorporated with rubber to produce thermoplastic elastomer (TPE) [11]. Therefore, this study opted *T. leontopetaloides* as a starch-based material to produce the reproducibility of TPS films.

However, the solely native of starch can caused the TPS to less elastic and brittle. Hence, plasticizer played an important role to transit the rigidity of starch material into flexible, improve the gelatinized

process, and avoid the formation of pores or crack in polymeric matrix. In TPS film, gelatinization process with addition of plasticizer is important to ensure the thermoplasticity of film. The thermoplasticity is formed under heat and shear conditions. Plasticizer helps to shift the rigidity of starch granule to a molten state of plastic and become a desired product [12]. The example of plasticizers that commonly used are water and polyol such as glycerol and sorbitol [13]. Besides that, the present of electrical charged from acid solution helps to induce TPS to dissolve better. The addition of acetic acid is practical to denature the molecule of starch during heated. Acetic acid reacts with plasticizers can form hydrogen ions and acetate ions in the solution state. The ions present will bonded with starch molecules to break the amylopectin polymers as it has an extensive branching and high degree of entanglement [14]. The broken of amylopectin structures allow the long linear of amylose to exhibit strong interaction and making the plastic more ductile. Besides that, acetic acid has larger molecular weight and has relatively strong intermolecular interaction to tight the film structure [15]. The process of acid disturbing the starch molecule is so-called as acid hydrolysis [16].

The use of agricultural waste as reinforcing material in bioplastic composite is interesting to enhance the properties of TPS. Due to some disadvantageous of starch based thermoplastic such as lack of strength, low water resistance, and thermal instability has encouraged the use of reinforcing material in TPS. In previous literature, biofiller from natural cellulose fibers are widely used to reinforce TPS [17]. However, the used of fillers exhibit slow degradation due to high cellulosic structural and might hindered the molecules [18]. Thus, biochar from renewable resource and carbonaceous material is a novelty approach for various composite polymer. Biochar is generated from thermo-chemical process and has converted the organic wastes to the elemental carbon. The process is so-called as pyrolysis [19]. Biochar is extensively used for catalysis [20] and environmental remediation [21]. Biochar has attractive features such as high surface area, high carbon content, high porosity, hydrophobic nature, and sustainable as composite [17]-[18]. Due to this, biochar has high potential as bio-filler in polymer composites. Up to date, the study of biochar in polymer is recently developed. For instance, in recent work by Das et al. (2015), wood polymer composite added with biochar denoted as wood polymer biochar composites (WPBCs) was developed. This biopolymer was compared with conventional wood polymer composites (WPC) to investigate the mechanical, chemical, thermal and microscopic properties [22]. Other, biochar from natural fibers engineered with Nylon 6 had been reported by Mohanty et al. (2015) to investigate the tensile, flexural and impact of Nylon 6 onto biochar

composites [23]. Despite of this work, none has focused on the investigation onto the TPS polymer reinforced with biochar from rice husk (RH) as a bio-filler. RH is an agriculture waste that abundantly available, cheap and sustainable to nature. Besides that, the used of biochar from RH also can lessen the generation of agricultural wastes. Furthermore, this research work has investigated the application potential of biochar in other area than used in catalysis, soil remediation or contaminant removal.

To our best knowledge, no study has investigated the effect of plasticizer-acid on *T. leontopetaloides* reinforced with biochar from rice husk. In this paper, we report a study on the sustainable approach of starch-based *T. leontopetaloides* to develop TPS and TPS reinforced with biochar (TPS-BChr) by varying the glycerol content and added with the acetic acid to promote the chemical modification of starch which differing from the standard glycerol-plasticized of TPS. The performance of TPS and TPS-BChr were investigated by using Fourier Transform Infrared (FT-IR), Scanning Electron Microscopy (SEM) and water uptake test. With proper blending treatment, TPS and TPS-BChr were developed through solution casting method and sulphur vulcanization method using two-roll machine. In this paper also has investigated the properties of biochar RH in pH, ash and volatile content, elemental composition, surface area and morphological.

## 2.0 EXPERIMENTAL

### Materials

*T. leontopetaloides* starch was collected from Mersing Johor, Malaysia as starch-based of TPS film. Glycerol, acetic acid, sulphur were purchased from Merck (M) Sdn Bhd. Biochar rice husk was manufactured by Sendi Enterprise in Sekinchan, Selangor to use as bio-filler.

Table 1. TPS denotation and compositions

Thermo-plastic Starch Film Denotation	Distilled Water (mL)	<i>T. leontopetaloides</i> Starch (g)	Glycerol (%)	Acetic Acid (%)	Sulphur (%)	Biochar RH (%)
TPS5%	80	20	5	10	-	-
TPS10%	80	20	10	10	-	-
TPS15%	80	20	15	10	-	-
TPS5%-BChr	80	20	5	10	0.2	5
TPS10%-BChr	80	20	10	10	0.2	5
TPS15%-BChr	80	20	15	10	0.2	5

### Preparation of Thermoplastic Films

Starch based thermoplastic films (TPS)s were prepared by adding *T. leontopetaloides* starch with various amount of glycerol according to formulation and denotations as stated in Table 1. 5% (v/v) acetic acid was dissolved in deionized water until the solution mixture boiled. Then, *T. leontopetaloides* starch was added to the solution and formed starch solution which heated at 90 °C under constant stirring for 30 min. The glycerol was periodically added until solution completely gelatinized at 85 °C to 90 °C. The gelatinized mixture was casted onto the polyacrylic plate and was dried in ventilated oven at 45 °C until constant weight. The dried films were peeled off from the polyacrylic plate. Then, dried films were vulcanized with sulphur using two roll-mill machine to form TPS. The speed of two roll-mill was adjusted between 5 rad/s to 10 rad/s and the vulcanized temperature was maintained at 50°C. While, TPS-BChr was prepared by accordingly added with 5wt% of biochar from rice husk. The sheets of TPS and TPS-BChr were rolled until uniform thickness of films obtained. All experiments were repeated in triplicates.

### Biochar Rice Husk Characterization

The determination of moisture content, ash content and the volatile matter of char were carried out according ASTM D1752-84. The moisture content was conducted by measuring 100g of biochar RH and dried overnight in conventional ventilated oven at 110 °C. Then, dried biochar RH was weight to measure the weight loss of char. The ash content was determined by measuring the weight loss 1g of char that undergoes combustion in muffle furnace under 750 °C. The final weight of char was calculated that determine the ash content. Then, the similar procedure was used to determine the volatile matter with different combustion temperature at 950°C. The pH of biochar also was determined by measuring the dilution of 10g of char in 100mL of distilled water (1:10, w:v ratio) using MP220 pH meter. Then, the elemental composition of C, H, and N was determined using an elemental analyzer (Thermo Finnigan EA-1112, Thermo Fisher Scientific Inc., MA, USA). The surface area of the biochar was also obtained from adsorption-isotherm using Brunauer Teller (BET) method. Table 2 shown the characteristic of biochar derived from agricultural waste of rice husk.

Table 2. Characteristics of biochar rice husk (RH)

Moisture % (w/w)	pH	Volatile Matter (%)	Ash (%)	C (%)	H (%)	N (%)	BET (m <sup>2</sup> /g)
8.15 ±0.13	8.20 ±0.04	13.64 ±2.31	48.2 ±4.6	26.23 ±3.33	6.88 ±1.77	0.28 ±0.03	186.99 ±8.43

### Fourier Transform Infrared Spectroscopy (FT-IR)

Fourier transform infrared (FT-IR) analysis was recorded on a Perkin Elmer FT-IR spectroscopy. Samples were analyzed by scanning the wavelength between  $4000\text{cm}^{-1}$  and  $515\text{cm}^{-1}$ . The infrared spectroscopy detects the vibration characteristics of chemical functional groups in the sample.

### Morphological Analysis

The morphology of tested sample was examined by using Hitachi TM 3000 scanning electron microscopy operated at 15kV. The samples were small cutting to examine under electron beam. Magnification used to examine the native *T. leontopetaloides* starch and raw of biochar RH were at 1.5 $\times$  of magnification. The tested samples of TPS films and TPS-BChr films were at 1.0 $\times$  of magnification.

### Water Uptake Test

The casted samples (size: 50mm $\times$ 30mm $\times$ 1mm) were immersed in water at room temperature. Then, the samples were removed at specific intervals, excess water on the surface of the samples was gently blotted with tissue paper, and the weight for each sample was recorded. This process was repeated at several time intervals. The water

absorption ratio was calculated by using the equation (Eq.1).  $X_t$  indicates the percentage of water absorption in interval hour  $M_x$  indicates the weight of the samples in the  $X_t$  hour;  $M_o$  indicates the initial weight of the samples;

$$X_t = (M_x - M_o) / 100\% \quad (1)$$

## 3.0 RESULTS AND DISCUSSION

### Fourier Transform Infrared Spectroscopy (FT-IR)

FTIR spectroscopy is a method to analyze the intermolecular interactions of molecules. In this study, FTIR spectroscopy was used to observe the functional group present that shifts the absorption peak into specific regions due to the interaction between TPS with glycerol, acetic acid and biochar RH. FTIR spectrum of TPS and TPS-BChr are depicted in Fig. 1. As illustrated in Fig. 1, all spectrum waves of TPS films had showed the range of absorption peak between  $3500\text{cm}^{-1}$  to  $3250\text{cm}^{-1}$  that indicates the bond of -OH, hydroxyl group. While, the band at approximately between  $2950\text{cm}^{-1}$  to  $2920\text{cm}^{-1}$  were characterized by the stretching of carboxylic acid (C-H) from  $\text{CH}_2$  and  $\text{CH}_3$  and also had assigned the vibration of alcoholic group from glycerol [24].

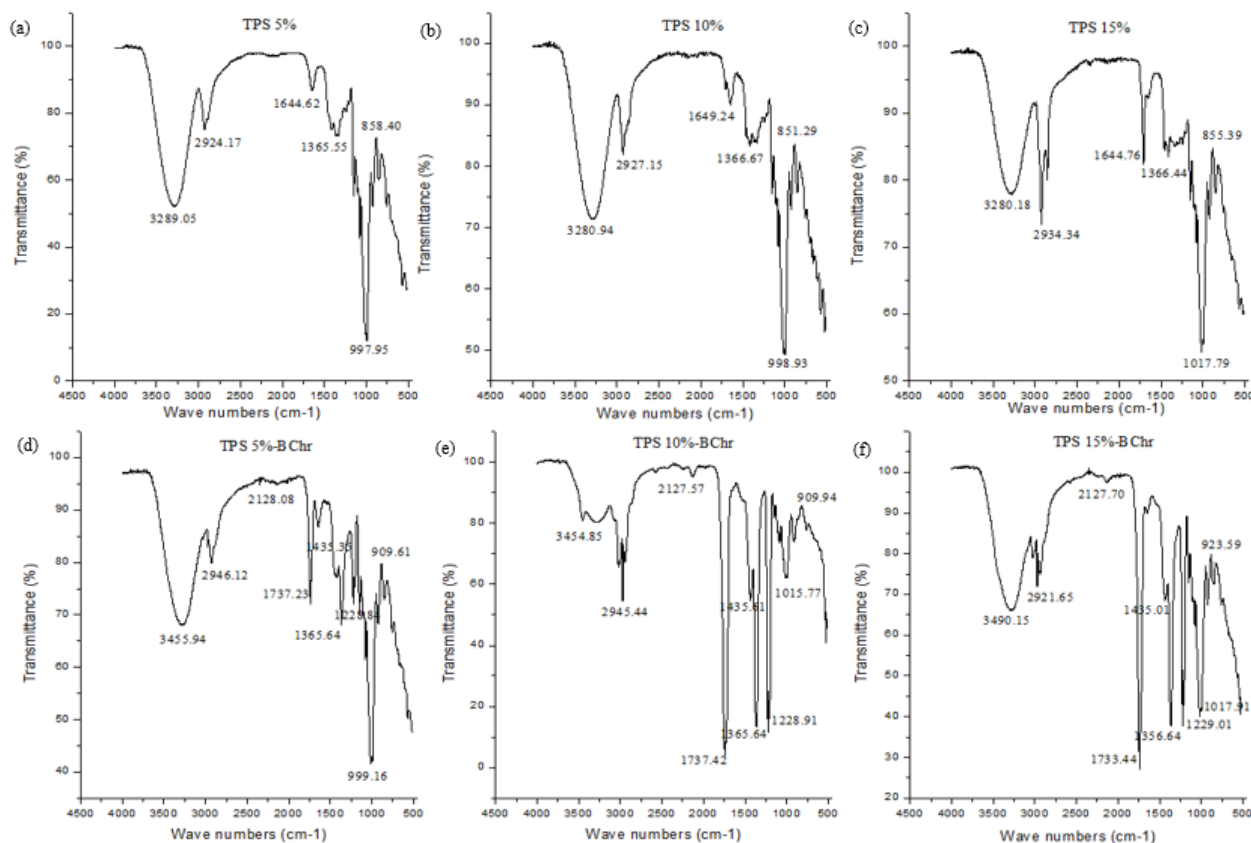


Fig. 1 FT-IR spectra of TPS and TPS-Bchr containing glycerol content (v/v): 5%, 10% and 15%.

In TPS films, the peak of spectrum had shifted to indicate the interaction of hydrogen bond when the plasticizer was gradually added. According to Ahmad et al, the lower number in absorption peak was resulted by the strong interaction of hydrogen bond [4]. Besides that, the band of -OH has shifted is also due to the carbon double bond to oxygen (C=O) from aldehyde group in native starch [9]. This was attributed by the reduction wavelength of -OH bond when glycerol content was increased. In Fig. 1 shown that the wavelength spectrum peak of Fig. 1(a to c) had decreased at approximately from 3289.05  $\text{cm}^{-1}$  to 3280.94  $\text{cm}^{-1}$  and to 3280.18  $\text{cm}^{-1}$  because the bond of starch was disturbed by the increment of glycerol content. Meanwhile, in Fig. 1 (d to f) have similar trend in chemical structures as the spectrum peaks had decreased with increasing of glycerol content at approximately between 3455.94  $\text{cm}^{-1}$  to 3454.85  $\text{cm}^{-1}$  to 3290.15  $\text{cm}^{-1}$ . Then, the spectrum peaks were shifted to other region between 1740  $\text{cm}^{-1}$  to 1640  $\text{cm}^{-1}$  that indicates the esterified acetic acid which assigned the bond of  $\text{COO}^-$  in all films.

FT-IR spectra also solely observed that the films of Fig. 1 (d to f) had spectrum peaks at approximately between 1228.84  $\text{cm}^{-1}$  to 1229.01  $\text{cm}^{-1}$  which revealed the functional group of biochar due to the vibration of lignocellulosic structure. It were also showed strong bond with wavelength spectrum peak at approximately 1435  $\text{cm}^{-1}$  while TPS showed in Fig. 1 (a to c) had showed lower band at approximately 1365  $\text{cm}^{-1}$  which assigned the bond of anyhydroglucose ring in native starch. Besides that, these spectrum peaks also indicates the bond of S=O due to the addition of sulphur in all films [25]. Then, the wavelength spectrum of films in Fig. 1 (a to c) were distinct shifted with the increment of glycerol content which vibrates at approximately between 997.95  $\text{cm}^{-1}$  to 998.93  $\text{cm}^{-1}$  to 1017.79  $\text{cm}^{-1}$ . The identical trend of spectrum shifting also reported onto films in Fig. 1 (d to f) which shifted the band between 999.16  $\text{cm}^{-1}$  to 1015.17  $\text{cm}^{-1}$  to 1017.91  $\text{cm}^{-1}$ . This was ascribed by the bonding of aromatic and alkene compound from the structure of native starch. Meanwhile, the vibration of spectrum was less shifted between 858.40  $\text{cm}^{-1}$  to 923.29  $\text{cm}^{-1}$  due to the strong of anyhydroglucose ring of starch. The phenomenon of FT-IR spectra reveals that the *T. leontopetaloides* starch, glycerol-acid were compatible and the addition of biochar as filler in the formulation was good complement to show the better interaction between these materials upon blending.

### Morphological Analysis

SEM microphotograph is presented in Fig. 2 to observe the morphology of TPS and TPS-BChr films to reveal the dispersion, homogeneous and appearance of pore present. The morphology of the

film is significantly contributed to the breaking up of polymer chains during water uptake testing. In Fig. 2 (a) shows the granules structure of starch powder and Fig. 2 (b) shows the biochar rice husk with the presence of impurities and other constituents of fiber as lignin and hemicellulose. In Fig. 2 (c to d) show the morphology of TPS5% and TPS5%-BChr respectively. Sheet of film in Fig. 2 (c) had indicated weak adhesion due to less amount of glycerol used to interrupt the granule starch. This had led to the formation of large pore and poor dispersion onto the surface of TPS5% film. Likewise, the film shown in Fig. 2 (d) reinforced with biochar composite also had poor adhesion, poor dispersion and the presence of many pores on the surface of film. The some pores present on the surface of TPS5%-BChr is also due to the fiber pullout that cause the roughness and uneven surface [22]. Thus, this had confirmed that low amount of glycerol at 5% (v/v) was not adequate to break the interaction of native starch and had resulting the roughness surface and less homogenous under SEM.

Conversely, the raise of glycerol content attributed to good adhesion and better dispersion as the polymer molecules of carbohydrates completely released and melted the granular structure due to high amount of plasticizer used during plasticization [26]. The uniform homogenous and well dispersion of films were clearly illustrated in Fig. 2 (e to f) which represent the TPS 10% and TPS10%-BChr. It can be seen that the films showed almost no single of starch granule present on the surface of films when 10% of glycerol amount were added. In TPS10%, the addition of 10% glycerol was effectively reduced the size of pore on the surface of film. Meanwhile, TPS10%-BChr film had showed evenness surface with less pores present on the surface of film when the biochar was added. In addition, the film shows that starch and biochar has good miscibility without the present of fiber pullout. From our observation, the adequate amount of plasticizer onto the film during heat and shear process had forced the char to break the fiber and enhance the interfacial adhesion of film. Meanwhile, the similar trend was obtained in the films of TPS15% and TPS15%-BChr which depicted in Fig. 2 (g to h). Both films were showed the homogenous surface with well dispersion. The good interfacial adhesion also showed on both surface as the higher amount of glycerol at 15% (v/v) was added. Liu et al. reported that the addition of plasticizer is significantly useful to disrupt the starch matrix in water solution to yield the good homogeneity of TPS [27]. Moreover, the increment of plasticizer content in the formulation of TPS also assists in easier process-ability and enhances the disruption of starch molecules [28].

### Water Uptake Analysis

Due to the hydrophilic characteristic of the TPS matrix, the water uptake test is an essential



parameter to deduce the applicability TPS films. Both TPS and TPS-BChr with different concentration of glycerol were immersed in water for 15 hours. Hence, the water uptake results are illustrated in Figure 3. As

shown in Figure 3, TPS and TPS-BChr immersed in water were characterized by three parts; Part I represent the fast absorption zone which

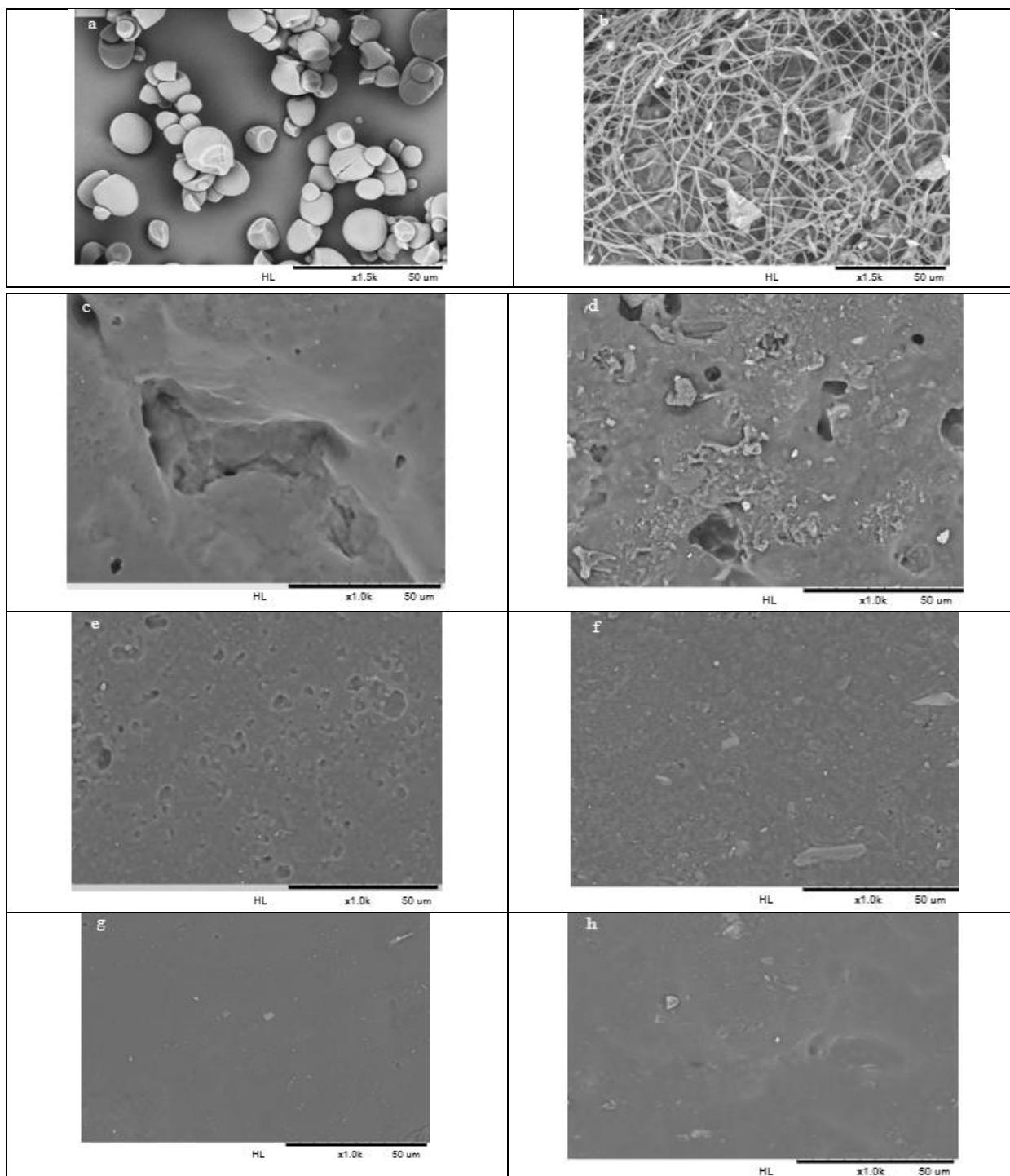


Fig. 2 SEM micrographs of native (a) Tacca starch (b) biochar RH and TPS surface of: (c) TPS5% (d) TPS5%-BChr (e) TPS10 (f) TPS10%-BChr (g) TPS15% and (h) TPS15%-BChr

was at the first two hours; Part II represent the steady increment zone of water uptake which was at the next eight hours; and Part III indicates the slow absorption zone at the last five hours. During experiment, it was observed that at the last five hours, all films were completely swollen and disintegrated.

Figure 3 shows that TPS and TPS-BChr with high glycerol content have strong affinity to attract more water molecules compared to lower concentration. Maran et al. stated that the starch and glycerol hold

strong hydrophilic capacity and induce the high affinity of water molecule [29]. In contrast, the addition of biochar in the formulation had significantly reduce the affinity of water uptake as shown in Figure 3 Oisik et al. [30] postulated that when biochar is in contact with water it induced the aliphatic group which adjacent to the pores to re-organized the structure in order to minimize the direct uptake of water molecules. It can be concluded that TPS-BChr has relatively lower affinity of water compared to TPS.

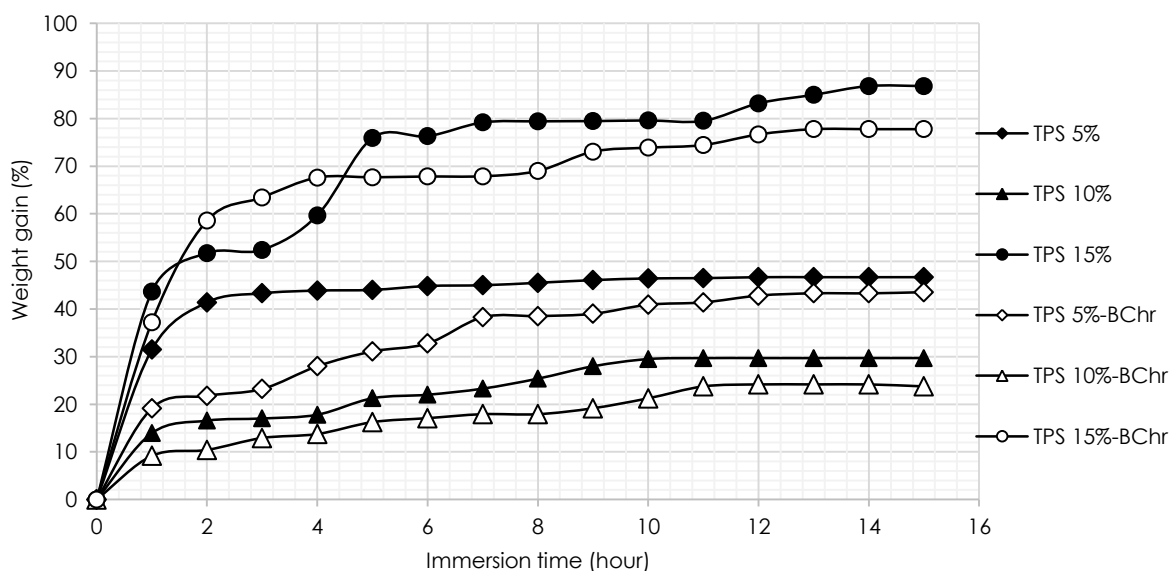


Fig. 3 Graph for water uptake analysis of TPS and TPS-Bchr containing glycerol content (v/v): 5%, 10% and 15%.

#### 4.0 CONCLUSION

In the current study, the TPS films derived from *T. leontopetaloides* starch reinforced with biochar RH were successfully prepared via solution-casting and sulphur vulcanization methods. It can be concluded the properties of TPS films were greatly affected by the chemical modification of glycerol-acid and biochar RH. From the finding, the increment of glycerol content induced the strong hydrogen bond and showed the intermolecular structure between starches with blending components. In morphological analysis, TPS films and TPS-BChr at high glycerol content had improved the homogeneity and also reduced the pores or cracks on the surface of films. However, the excessive of hydrogen bond caused the instability films that induce the wettability of films to attract more water molecules. The blends of starch with biochar to produce a novel biopolymer is very significant. Also, it has suggested

the need for extensive research on agricultural waste for more commercial benefits in the future.

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