

Characterization of Biomass-Reinforced Biopolyol-Based Polyurethane Foams

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ABSTRACT

In this study, polymer composites comprised of polyurethane (PU) foam and two kinds of biomass (coco peat and sphagnum) were prepared. PU industry heavily depends on the petroleum industry. Coco peat and sphagnum are renewable resources. Their use as a reinforcing filler to increase PU foam's renewable content is yet to be explored. The PU foam was produced via a reaction between 2,4-diphenylmethane diisocyanate and palm kernel oil-based polyol (PKOP). The filler varied from 0–1.2 g. Fourier-transform infrared spectroscopy, morphological analysis, and determination of the mechanical properties of PU foam composites were conducted to identify the effect of the fillers on the PU foam. The results showed that sphagnum has more impact than coco peat on cell structure and the apparent density of the PU foams. The foam cell lost its regular hexagonal 'bee-hive' structure, and the apparent density increased to approximately 30% more than the reference sample at 1.2 g filler loading. The PU foam filled with 1.2 g coco peat exhibited significantly improved mechanical properties, with 120% increase in tensile strength compared to the reference sample of the PU foam.

Keywords: Coco Peat, Polyurethane Foam, Palm Kernel Oil-Based Polyol, Sphagnum.

1. INTRODUCTION

Polyurethane (PU) is a hetero-chain polymer produced by the reaction between isocyanates and polyols, and its main chain contains carbamate. Polyurethane's insulation, waterproof, and mechanical properties [1] have led to its wide use in many industries: furniture [2], electronics [3], textile [4], medical treatment [5], insulation [6], construction and building [7], automobile [8], and aerospace [9]. PU foam is the most common PU product which is polymerized from polyols and isocyanates under the action of the foaming agent and catalyst. It can be used as thermal insulation material, bumpers, bedding, and seat cushioning [10-13].

At present, the PU industry heavily depends on petrochemicals; namely, the polyester or polyether polyols and isocyanates [14]. Petroleum is a non-renewable resource with limited reserves and high prices. From mining to refining, pollution would be caused by petroleum at every stage [15]. It contributes to air pollution, water pollution, and many diseases. Due to the rapid depletion of crude oil reserves, concerns about the ecological environment, uncertainties around oil price and supply, and the preference of various government organizations for

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sustainable development, the chemical industry has been prompted to adopt sustainable raw materials, especially renewable resources to manufacture products.

1.1 Biopolyol-Based PU Foam

Bio-polyols derived from renewable materials rather than petrochemicals can directly replace petroleum-based polyol because they have similar structures and properties. More and more petroleum polyols are being replaced by bio-polyols derived from vegetable oil to produce PU foam, such as castor oil [16], soybean oil [17], canola oil [18], and so on. Malaysia is a big producer of oil palm which can produce two kinds of vegetable oil (palm oil and palm kernel oil). Palm kernel oil is mainly used for non-edible purposes. Therefore, polyol synthesized from palm kernel oil was selected as a renewable bio-polyol in this research. The palm kernel oil-based polyol (PKOP) is a diol-based refined, bleached, and deodorized polyol synthesized through direct polycondensation and transesterification methods as described in a study conducted by Badri et al. [19].

1.2 Biomass as Reinforcement Fillers

Other than using bio-based chemical raw material to synthesize products, adding biomass as reinforcement fillers in PU foam is another way to increase the renewable content. Empty fruit bunch [20], hemp stem [21], nutshell [22] as natural original fillers in PU foam were researched in the past. These biomass fillers had effects on PU foams' microstructure, mechanical properties, and so on. Coco peat and sphagnum were selected due to its availability and wide application in agriculture. At present, there are no studies reporting the usage of coco peat fiber or sphagnum fiber in PU foams as fillers.

Coco peat is a spongy particle produced after the extraction of fibres from the coconut shell and is widely used as soil-less planting material in tropical regions [23]. It is a mix of short fibres (2–13% in total) and coarse-to-fine cork particles [24, 25]. A coconut shell weighs approximately 400 g and consists of 75% fibre and 25% cork. Coconuts also contain about 100 g of coco peat [26]. Coco peat has high water retention and has traditionally been used to improve the physical and chemical properties of soil. The addition of coco peat to agricultural soil can improve the effective nutrient content, infiltration rate, water retention, hydraulic conductivity, and total porosity of the soil [27]. If all the coconut husk in the world were used to produce fibers, about 8 million tons of coco peat would be produced each year, but most of the coconut husks are not yet processed and utilized [24]. Each year about 5,280 kg per hectare of coconut waste can be obtained in Malaysia [28]. Coco peat is a source of lignocelluloses, consisting of lignin, cellulose, and hemicellulose.

Sphagnum is a genus of over 300 species of bryophyte that is highly adapted to acidic, cold, waterlogged, and extremely nutrient-poor environments commonly called as peat moss [29]. Sphagnum can retain water 15 times to 20 times its own dry weight [30]. Most of the peat moss (probably less than 30 species) grows in the north temperate zone and northern regions, while in other latitudes, peat moss can grow, but it usually grows at high altitudes, such as the snowy mountains of Australia, the Andes mountains of Chile, and the tops of equatorial mountains in Africa [31]. An example of a single species with isolated distribution is *S. junghuhnianum* ssp growing in the mountains of Malaysia, Japan, and China [31]. The application of sphagnum in horticulture is mainly focused on the cultivation of orchids, which satisfy special growth medium requirements such as good aeration and sufficient water retaining capacity [32]. The annual yield of sphagnum can reach 2.5-4.5 tons per hectare [33]. Sphagnum is similar to coco peat and also contains large amounts of lignin, cellulose, and hemicellulose [34].

This study examines the changes of physical properties (mechanical, apparent density, and microstructure) and chemical properties (Fourier-transform infrared spectroscopy, FTIR) of

bio-polyol PU reinforced with biomass fillers. The comparison was made between coco peat and sphagnum reinforced PU respectively.

2. MATERIAL AND METHODS

2.1 Raw Materials

The PKOP used in this study was supplied by UKM Technology Sdn Bhd, UKM/MPOB Station, Pekan Bangi Lama, Malaysia. Petroleum-based polyol (Voranol 4240), crude MDI (2, 4-methylene diphenyl diisocyanate), and the catalyst (33LV) were purchased from Cosmopolyurethane (M) Sdn Bhd, Port Klang, Malaysia. Coco peat and sphagnum were purchased from the local horticultural market.

2.2 Specimen Preparation

The PU foam was produced by a one-step foaming method by reacting component A (polyol, H₂O, 33LV) with component B (crude MDI) at room temperature. Coco peat and sphagnum were respectively dried and crushed, then sieved through a 100 µm sieve to obtain an even powder. Component A, consisting of a mixture of petroleum-based polyol (Voranol 4240), palm kernel oil-based polyol, water, and 33LV, was mixed at 500 rpm for 30 s. The fillers were added to component A in varying amounts: 0.3 g, 0.6 g, 0.9 g, and 1.2 g. Component A was then stirred for 10 s at 1,500 rpm prior to reaction with component B. The mixture was moulded in a stainless steel mould and demoulded after 20 mins. The samples were conditioned for 16 h before testing for apparent density and conducting FTIR analyses. The composition of each sample is shown in Table 1, followed by the characterisation process as stated in 2.3.

Table 1 The composition of each sample

Sample No	Component A	Component B	Filler content
0	10 g	6.5 g	0 g
A1	10 g	6.5 g	0.3 g
A2	10 g	6.5 g	0.6 g
A3	10 g	6.5 g	0.9 g
A4	10 g	6.5 g	1.2 g
B1	10 g	6.5 g	0.3 g
B2	10 g	6.5 g	0.6 g
B3	10 g	6.5 g	0.9 g
B4	10 g	6.5 g	1.2 g

2.3 Characterization

2.3.1 Morphological Observation

A Dino-Lite handheld digital microscope (AM413T5) and the DinoCapture 2.0 software were used to observe changes of the microstructure and morphology in PU foams at 500× magnification.

2.3.2 Fourier-transform Infrared Spectroscopy (FTIR)

The Fourier transform infrared spectrometer (Thermo Scientific™ Nicolet™ iS5 FTIR Spectrometer) is used to measure the infrared spectrum of samples. The FTIR measurements using the iD5 ATR Accessory with diamond crystals were taken in the 4000–650 cm⁻¹ range scanning 20 times. The resolution was set to 4 cm⁻¹. The foam sample was placed between the

crystal and the metal tip, and the fixing load was enough to ensure an adequate contact between the sample and the ATR crystal.

2.3.3 Apparent Density

The apparent density of the PU foam was determined according to the ATSM 3574 standard and calculated using the volume and mass of samples. The physical volume of specimens were measured. Apparent density was calculated according to the following equation.

$$\text{Apparent density} = \frac{M_{\text{sample}}}{V_{\text{sample}}} \quad (1)$$

2.3.4 Tensile Test

The tensile test was carried out by the universal testing machine (INSTRAN 5566), according to the ATSM 3574 standard. The flaky sample was cut to a standard shape by the D421 die. The test speed was fixed at 500 mm/min and the load was 10 kN. The mean value of tensile stress was recorded.

3. RESULTS AND DISCUSSION

3.1 Microstructure of Foam Composites

The cellular structure and morphology of the reference sample were observed parallel and perpendicular to the foaming direction as shown in Figure 1. It can be seen from Figure 1 that the cells are a mix of open cells and closed cells. Closed cells have a thin and transparent PU membrane between the cell structures, which hinders the permeability of air and liquids in the foams. The size distribution of the foam cells is relatively uniform, with an average cell size of 0.021 mm^2 . Viewing from the perspective perpendicular to the foaming direction, the dominant cells are in circular shape. However, when viewed from the perspective parallel to the foaming direction, the cell shape is oval (with the cell size ratio of the long axis to the short axis of 9:5). The reference sample (labelled 0) exhibits anisotropy foam due to gravity and uneven temperature and pressure [35] during foaming process. The morphological study observed by Hamilton *et al.* [36] also shows anisotropy foam formation.

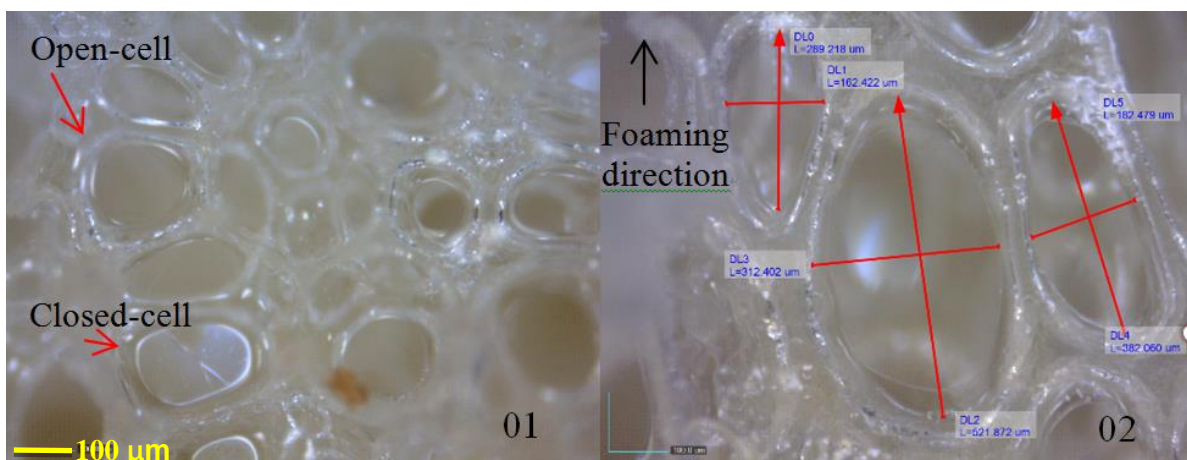


Figure 1. Micrographs of the reference sample from perpendicular (01) and parallel (02) perspective to the foaming direction.

From the micrographs of the PU foams filled with coco peat and sphagnum (Figure 2), it can be observed that the addition of fillers affects the cell structure formation. The cell size and cell struts' thickness of the PU foam increases as the filler content increases. As can be seen from the images of samples A1 (coco peat) and B1 (sphagnum), a few filler particles are embedded in the cell struts, and the number, size, and morphology of cells are similar to that of the reference sample (shown in Figure 1). Cell growth is controlled by the foam matrix stiffness, gas diffusion rate, and gas loss rate [37]. With the increase of filler content from 0.6 g to 1.2 g (shown in images A2, A3, A4, and B2, B3, B4), more and more filler particles form agglomeration. Furthermore, the number of cells in a given area decreases, the cell size distribution increases, and the cell shape is distorted. This indicates that the gas does not diffuse well in the matrix and the gas loss rate increases, resulting in cell dysplasia. Bai-Liang Xue et al. [38] and Mosiewicki et al. [39] observed similar results in their studies. Their studies found that the increase of fillers led to the destruction of the foam morphology and structure when reinforcing PU with wood powder and lignin.

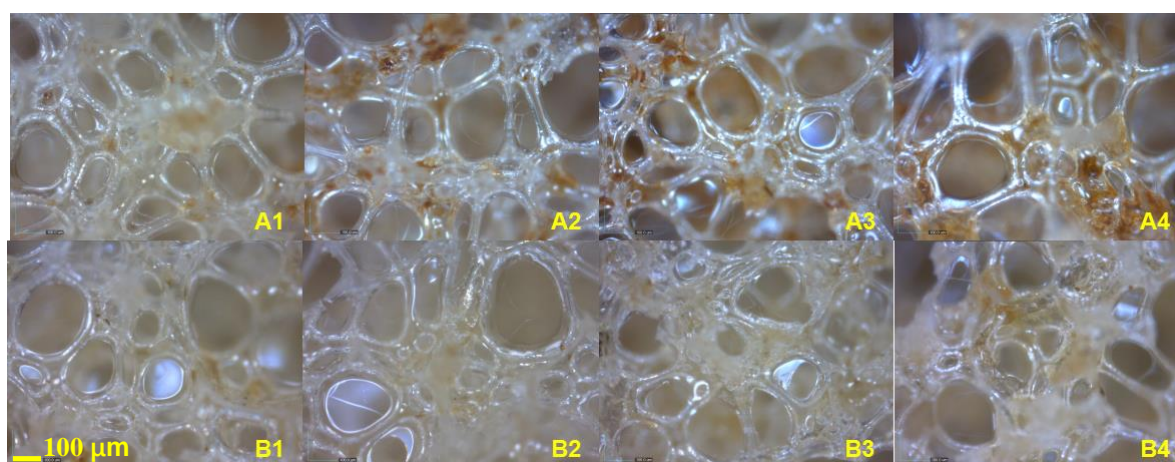


Figure 2. Micrographs of the PU foam filled with coco peat (A) and sphagnum (B) from the perpendicular direction.

3.2 Fourier-Transform Infrared Spectroscopy (FTIR)

Figure 3 showed the FTIR spectra of samples 0, A3, and B3. The transmission peaks appeared at the same wavenumber (offset less than 1 cm^{-1}). This indicates that the addition of the filler did not affect the formation of the PU and that the filler did not react with the raw materials. The chemical structure of PU is determined by the main characteristic absorption bands of urethane groups (shown in Figure 3): N-H stretching vibrations at $3,499\text{ cm}^{-1}$ (free N-H) and $3,299\text{ cm}^{-1}$ (hydrogen-bonded N-H), N-H bending vibrations at $1,510\text{ cm}^{-1}$, C=O stretching vibrations at $1,730\text{ cm}^{-1}$, C-N stretching vibrations at $1,598\text{ cm}^{-1}$, and C-O stretching vibrations at $1,220\text{ cm}^{-1}$ [40-43]. In addition, the peaks at $1,538\text{ cm}^{-1}$ are related to the stretching vibrations of C=C bonds in benzene rings of MDI, and peaks associated with the vibrations of C-H in benzene rings of MDI were observed at $1,308\text{ cm}^{-1}$ (bending), 816 cm^{-1} (1,4 disubstituted benzene wagging), 766 cm^{-1} (1,2 disubstituted benzene wagging) [41, 44, 45]. The bands of $1,455\text{ cm}^{-1}$, $1,373\text{ cm}^{-1}$, and $1,343\text{ cm}^{-1}$ probably resulted from -CH₂- bending in the backbone of MDI [46].

However, the intensity of the peak at $1,731\text{ cm}^{-1}$ in the spectral curves (A3, B3) of foam reinforced with coco peat (76.4%) or sphagnum (78.3%) is weaker than that of the reference sample (75.7%). According to the Lambert-Beer law, absorbance is related to the extinction coefficient, light path, and concentration. Due to the limitation in determining extinction coefficient and the optical path, the concentration cannot be quantitatively compared; however, a semi-quantitative comparison can be made using absorbance. This result indicates that as the carbonyl content of the characteristic group of PU decreases, so does the concentration of PU

decreases. The reduction of carbonyl content characteristic peak is attributed to the reaction of isocyanate with moist contained in coco peat or sphagnum instead of hydroxyl in polyol, resulting in a decrease in the urethane concentration. The same phenomenon was observed by Członka, Bertino, and Strzelec [47].

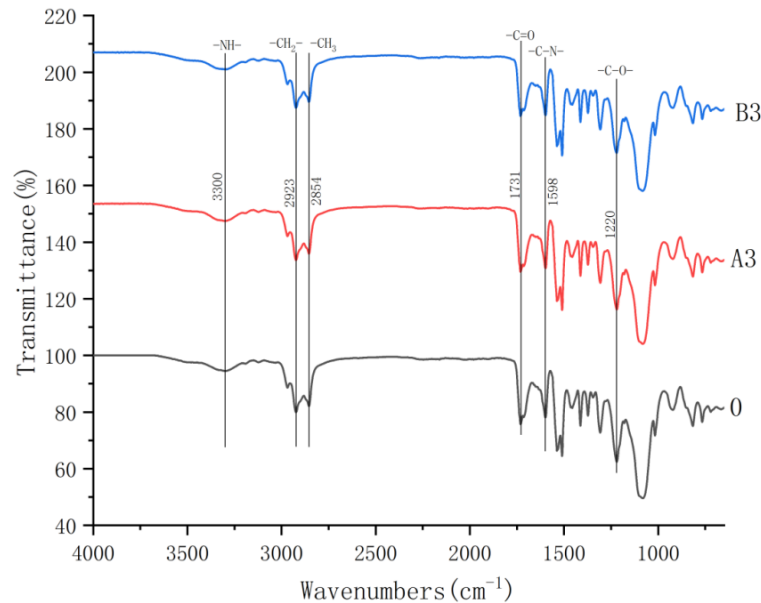


Figure 3. FTIR spectra of PU foam samples (0, A3, B3).

3.3 Apparent Density

The density of the polymer foam depends on the amount of the foam network the foam matrix density, and the intracellular gases density [22]. The effect of filler addition with different loading on the apparent density of PU foam is shown in fig. 4. As can be seen from curve A, the apparent density of PU foam filled by coco peat fluctuated between 90 kg/m³ and 100 kg/m³ with the increase of the filler loading. All the apparent densities of the reinforced PU foam were lower, as compared with the unreinforced PU foam (100 kg/m³). When the coco peat content was 3 g, the apparent density of the reinforced PU foam declined to 92 kg/m³. The apparent density subsequently increased to 100 kg/m³ (0.9 g). The apparent density of the B samples, showed slight decrease to 99 kg/m³ when 0.3 g of sphagnum was loaded, as compared to the reference sample. The apparent densities was further increased, when more sphagnum were added.

As the raw materials of all the samples were the same and the weight of the filler was a variable, there are two major factors contributing to the changes in apparent density, namely moist content and filler loading effect. The presence of moist content in the coco peat and sphagnum reacts with the isocyanate in the foaming process, thus generating more CO₂ and increasing the foam volume [48]. The addition of fillers in PU foams resulting in an increase of water content is consistent with the results of the FTIR shown in Figure 3, in which a trace of water was detected. This water content would lead to a decrease in apparent density. The increase in apparent density may be due to the viscosity of component A rising as the filler loading increases [49]. The high initial viscosity of the foaming system makes the expansion of the reaction mixture more difficult. Some studies [50, 51] have shown that the increase of the viscosity of the reaction system will increase the foaming time. This leads to more gas loss through open cells instead of foaming. This increase in viscosity leads to an increase of the apparent density. For coco peat, the change of apparent density was caused by a combination of these two reasons.

For sphagnum, the apparent density increased after 0.3 g filler loading, which can be attributed to the dominant effect of increasing viscosity as more fillers are added to the PU foam.

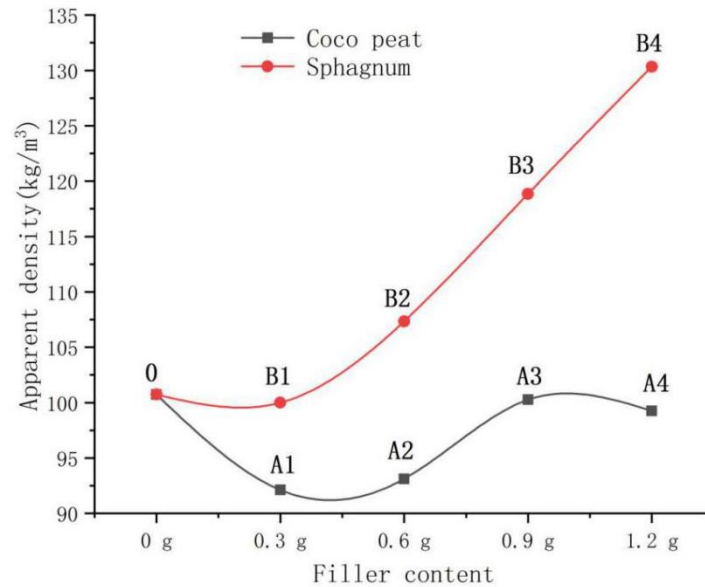


Figure 4. Apparent density of PU foam samples with different filler loading (0, A1, A2, A3, A4, B1, B2, B3, and B4).

3.4 Tensile Property

The Figure 5 shows the influence of different filler loading on the tensile strength of PU foams. As shown in curves A (coco peats) and B (sphagnum), the tensile strength increased continually along with the increase of filler loading. The tensile strength of PU foam filled with 1.2 g coco peat increased to 0.62 MPa, which was 120% higher than that of the reference sample (0.28 MPa). The tensile strength of PU foam filled with 1.2 g sphagnum rose to 0.56 MPa, doubled the reference sample (0.28 MPa). The strength of composite materials mainly depends on the volume fraction and the strength of composite materials and the filler-matrix interfacial bonding [52, 53]. Furthermore, the density, cell structure and size, anisotropy, and cell wall thickness are also factors affecting the mechanical properties of foam [50]. The increase in the PU foam's tensile strength are aligned with the increase of the filler loading. The improvement in tensile strength is attributed to the increase of cell strut's thickness when filler loading is increased, as shown in Figure 2. Thickening of struts structure allowed better load distribution when force is applied.

A comparison of coco peat and sphagnum shows that the tensile strength of all coco peat reinforced PU foam samples were higher than that of the samples reinforced by sphagnum. The main components of sphagnum are lignin (14.3%), cellulose (17.64%), and hemicellulose (22.2%) [54]. However, the proportions of cellulose and lignin in coco peat are higher than those of sphagnum [55]. Cellulose has a high tensile strength and lignin contributes to strength properties [56, 57]. Subsequently, the tensile strength of PU foam filled with coco peat was higher than that of sphagnum.

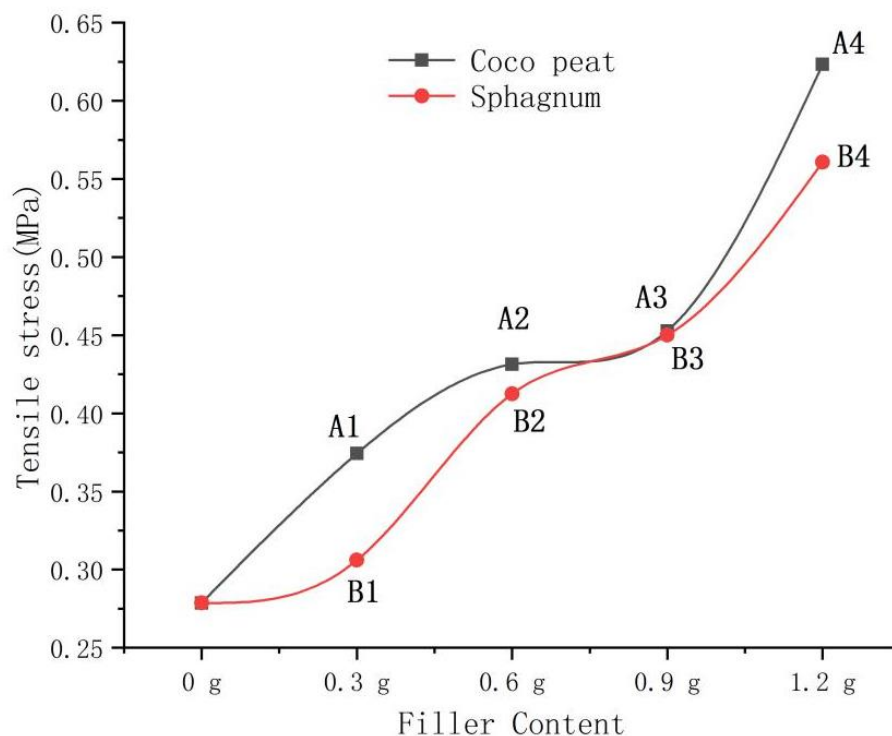


Figure 5. Tensile strength of PU foam with different filler loading (0, A1, A2, A3, A4, B1, B2, B3, and B4).

4. CONCLUSION

In general, with the increase in filler loading, the cellular structure of PU foam becomes distorted and the cell size distribution becomes wider in both coco peat and sphagnum samples. The apparent density of PU foam reinforced with 1.2 g sphagnum increased to 130 kg/m³. However, coco peat has less influence on the physical appearance of the PU foam. The FTIR spectra indicated that the addition of filler did not affect the chemical chain structure of the PU foam, although the presence of moisture was traced in the filler, which might react with isocyanates. The addition of coco peat or sphagnum increased the tensile strength of PU foams. Compared to the reference sample with a tensile strength of 0.28 MPa, the tensile strength of PU foam filled by coco peat at 1.2 g filler loading reached 0.62 MPa. The addition of sphagnum at 1.2 g filler loading resulted in a tensile strength of 0.56 MPa. The PU foam reinforced with coco peat exhibited better tensile property than PU foam reinforced with sphagnum.

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