

Oil-Water Adsorptive Properties of Chemically Treated Sugarcane Bagasse

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Abstract

Among the contaminants plaguing our waters today, oil remains one of the most pervasive and challenging contaminant to remove. Oil pollution occurs not only through factory discharge, but also by accident spills from the fuel of the vehicle or the transportation of oil. Sugarcane bagasse (SB) is an abundant agricultural by-product containing almost half of cellulose and one quarter of lignin. After chemical treatments SB can be modified their hydrophobicity leading to improve its oil adsorptive properties. In a column experiment containing 1 g of SB, oil was able to be adsorbed from oil and water mixture by, from the highest to the lowest uptake, AASB, ASSB, NSB, SSB and BSB with the average oil adsorption capacity of 13.0 mL/g, 11.25 mL/g, 10.50 mL/g, 9.0 mL/g and 8.75 mL/g respectively. The results were concurrently meeting the result of material characterisation using FTIR, where acetic acid treated SB (AASB) consists of high lignin leading to high hydrophobicity. On the other hand, BSB showed the lowest oil adsorption capacity and more hydrophilic due to the lowest amount of lignin present in SB. The result showed a potential use of natural material of SBs with high lignin content to tackle oil spill in water environment.

Keywords: cellulose, hydrophobicity, lignin, oil spill, sugarcane bagasse, water

1. Introduction

Due to increasing oil consumption, oil pollution of water has steadily increased (Hussein et al., 2009). It is a global concern as one of the main sources of water contamination is oil spills or oily waste waters. It imposes a serious damage on the environment and the economics. Oil pollute all water sources such as seas, oceans, rivers or groundwater (Behnood et al., 2013). Oil spills are caused by many factors such as discharge of crude oils from tankers, ships, offshore platforms; accidental spill in pipe lines or production process; spill of other oily waste waters such as produced water and ballast water (Behnood et al., 2013). When oil is spilled into a marine environment, it is subject to several processes including spreading, drifting, evaporation, dissolution, photolysis, biodegradation and formation of water-oil emulsions (Annunciado et al., 2005). Plants and animals will be contaminated and some will be unable to survive. In general, oil spills occur in oceans, estuaries, rivers, lakes, ponds, or on land, they can affect many living things such as plants, invertebrates, fish, birds, and mammals.

Several clean-up techniques have been used to tackle oil spillage, including use of dispersants, mechanical tools and in-situ burning of oil on water (Allen, 1998). However, those clean-up practices are not efficient enough and will post environmental effects. Conversely, adsorption is becoming more common in removing organics dissolved in water (Okiel et al., 2011) due to its promising results and lower operation cost (Abdul et al., 2012; Kavitha & Namasivayam, 2007). Synthetic sorbents such as polypropylene have good hydrophobic and oleophilic properties as they have high oil adsorption capacity (Jarre et al., 1979), but they are unable to biodegrade (Choi & Cloud, 1992).

In recent years, agricultural by-products are being increasingly used as oil adsorbents due to their low cost and biodegradability. They include rice husk ash (Vlaev et al., 2011), barely straw (Hussein et al., 2009) and peat-based sorbents (Cojocaru et al., 2011) as well as sugarcane bagasse (SB) from the production of cane sugar. Even though SB is produced in huge abundance every year, only a small proportion of it is reused as biofuel and pulp and paper

products (Rowell & Keany, 1991). SB properties can also be modified in physical, biological, chemical and photochemical processes (Tronc et al., 2006). SB mainly consists of cellulose, hemicellulose, and lignin with the average content percentages of 40%, 30% and 20% respectively (Sun et al., 2003). In the adsorption of oil, hydrophobicity of the adsorbent is a key factor as it is a measure of repulsion of polar substances of water and attraction of non-polar substances of oil. On the other hand, hydrophilicity is the measure of attraction of polar substances and repulsion of non-polar substances. In SB, the hydrophobic functional groups are found on lignin and the hydrophilic groups are the hydroxyl groups found on cellulose and hemicellulose. Hence, in order to improve the oil adsorption capacity of SB, pretreatment of the material can be done. Different pretreatment methods applied to SB may alter the cellulose crystallinity of or selectively remove the hemicellulose and lignin in the lignocellulosic substrate. The methods include physical process such as milling (Chang & Holtzapple, 2000; Gharpuray et al., 1983) and irradiation (Taherzadeh & Karimi, 2008; Lafitte-Trouqué and Forster, 2002), physical-chemical methods such as using ammonia explosion (Gollapalli et al., 2002) and acids and bases (Betancur & Pereira, 2010; Deschamps et al., 1996; Sun et al., 1995), and biological methods such as using bacteria and fungi (Kurakake et al, 2007; Srilatha et al., 1995). Acids hydrolyse hemicellulose and minor amounts of lignin (Geddes et al., 2010; Rocha et al., 2011) while bases hydrolyse hemicellulose and lignin (Zhang & Lynd, 2004; Jackson, 1977). Acetylation is also another method used to treat lignocellulosic material where hydrophobicity of the material is increased by substituting hydrophilic hydroxyl groups with hydrophobic acetyl groups. The reaction is usually carried out by heating the material with acetic anhydride, with or without the presence of a catalyst (Rowell, 1983).

The objective of the work is to investigate an oil adsorption from SB with various chemical treatments. Characterisation of the samples was first conducted to identify crystallinity, crystallite size and presence of various functional groups. Column adsorption experiments were then conducted with the SB samples using water only and a mixture of oil and water.

2. Methodology, Equipment and Materials

2.1 Pre-Treatment of SB to Prepare NSB, SSB, BSB, ASSB and AASB

An estimated 5 kg of SB was collected from a hawker centre using a plastic bag. After random cleaning, approximately 30 g of SB was weighed using a four digits weighing balance. The samples were rinsed using tap water to ensure free from any dust or fine particles. Up to 1 L of deionised water was used to further clean the samples. The SB was placed on an aluminium tray and dried in an oven at 103°C overnight to fully remove water moisture. The SB was collected the next day and cut into smaller pieces using scissors and blenders. A uniform size of SB was stored after sieving using ASTM sieve between 2.0 mm - 850 µm. The sieved SB was labelled as natural SB of NSB. NSB was chemically treated to prepare other SB variants of SSB, BSB, ASSB and AASB after being overnightly soaked in 0.5 M of sodium chloride, sodium hydroxide, sulphuric acid and acetic acid solutions respectively. The morphology change of SB was investigated using FTIR. Each sample was then rinsed with 8 L deionised water each and dried in the oven at 103°C overnight. All the samples were stored in labelled containers.

2.2 Characterisation of SB using SEM, FTIR and XRD Analytical Instruments

The SB samples' surface was scanned using SEM (JEOL- 6400) to understand the morphology of the samples. Material characterisation was conducted using the available analytical instruments of FTIR and XRD to determine various functional groups including lignin existence, existence of cellulose and size of crystalline cellulose from NSB, SSB, BSB, ASSB and AASB samples. The dried samples were grinded and embedded in KBr pellets. IR spectra of the samples were recorded by using IRPrestige-21 FTIR spectrophotometer (Shimadzu). The spectra were recorded in the transmission band mode in the range of 4000 - 400 cm⁻¹. Crystallinity of the samples was determined by X-ray diffraction using D8 ADVANCE diffractometer (Bruker). The diffraction spectrum was taken by the θ - 2 θ method. Samples were scanned from 2 θ = 10° to 70° with a step size of 0.02°. The samples' crystallinity was determined as the percentage as follows:

$$CrI = \frac{[I_{002} - I_{am}]}{I_{002}} \times 100\% \quad (1)$$

Where, I_{002} = the maximum intensity of the (002) lattice diffraction (2 θ \approx 22°), I_{am} = the intensity at 2 θ \approx 15 - 18° (amorphous)

The average size of the crystallites was calculated from the Scherrer equation, which was based on the width of the diffraction patterns obtained in the X-ray reflected crystalline region. In present study, the crystallite sizes were determined by using the diffraction pattern obtained from the 002 (*hkl*) lattice planes of samples using the following formula:

$$D_{(hkl)} = \frac{K\lambda}{B_{(hkl)}} \cos \theta \quad (2)$$

Where, (hkl) = the lattice plane, $D_{(hkl)}$ = the size of crystallite, K = the Scherrer constant (taken as 0.9), λ = the X-ray wavelength (0.154 nm), $B_{(hkl)}$ = the FWHM (full width half maximum) of the measured hkl reflection, 2θ = the corresponding Bragg angle (reflection angle)

2.3 Experimental Set Up using Acrylic Column

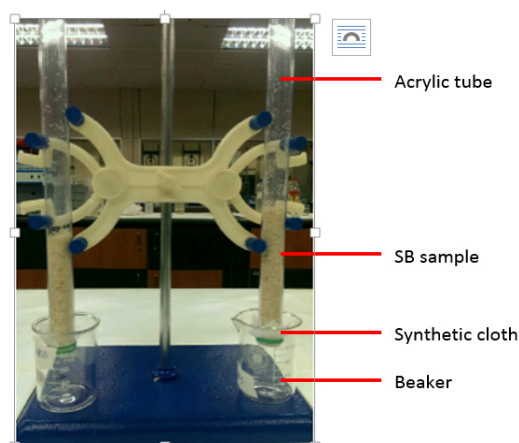


Figure 1. Experiment set up

A column experiment set up was prepared as shown in Figure 1. A fixed mass of 1.0 g NSB, SSB, BSB, ASSB and AASB were weighted respectively using a four digits weighing balance. Each sample was then placed into a 40 cm long transparent acrylic column with internal diameter of 1.8 cm and compacted by applying a constant weight on the sample. Two sets of experiments were conducted with the adsorbates consisting of water only and the adsorbate consisting of a mixture of used vegetable oil and water in a ratio 1:1. The total volume of 100 mL was used in both experiments. To prepare a fully dispersed of oil and water mixture a metal spatula was used in stirring the mixture in a 500 mL beaker. The adsorbate was poured into the column with SB sample and excess time was given for the mixture to pass through the column. All the filtrate was then collected and measured using 100 mL measuring cylinder.

2.4 Determination of Oil and Water Adsorption of SB Samples

The adsorption capacity of adsorbent was measured separately. Water adsorption was the expression of the volume of water that could be adsorbed after water passing through the column, either from water only or a mixture of water and oil. Oil adsorption was the expression of the volume of oil that could be adsorbed after a mixture of water and oil passing through the column. Both were expressed in mL of water per g of SB or mL of oil per g of SB, in the case of the water and oil mixture used.

$$\text{Water adsorption (mL/g)} = [(W_i - W_f) / M] \quad (3)$$

$$\text{Oil adsorption (mL/g)} = [(O_i - O_f) / M] \quad (4)$$

Where W_i = Initial volume of water (mL), W_f = Final volume of water (mL), O_i = Initial volume of oil (mL), O_f = Final volume of oil (mL), M = Mass of SB in the column (g)

3. Results and Analysis

3.1 SB Characterisation using SEM, XRD and FTIR

Figure 2(a) shows physical appearance of various SB samples with the related SEM surface images shown in Figure 2(b) to 2(f).

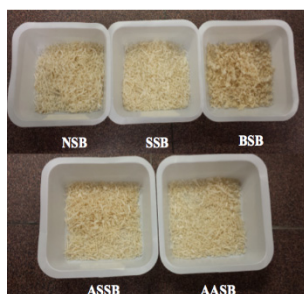


Figure 2(a). Physical appearance of SB samples

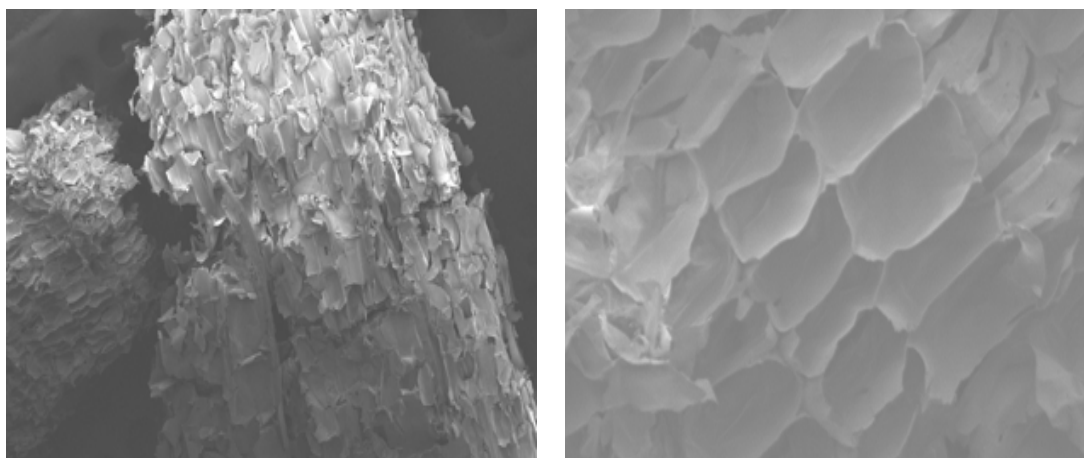


Figure 2(b). SEM image of NSB sample magnified 25 times (left) and 200 times (right)

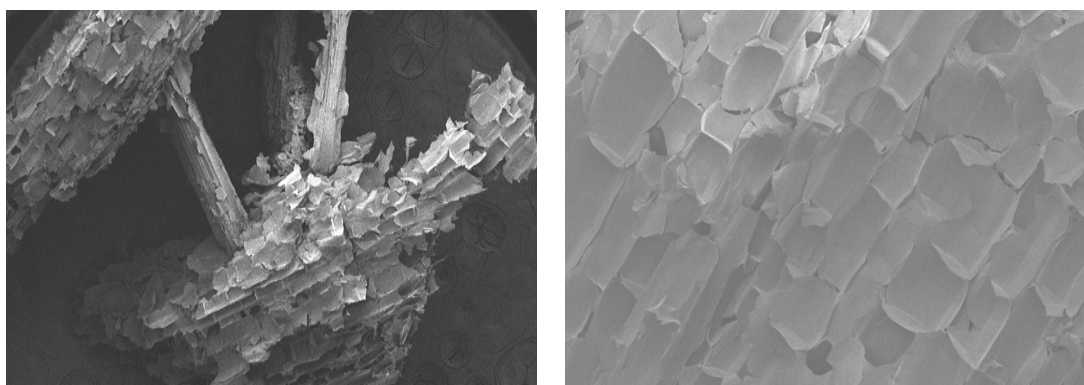


Figure 2(c). SEM image of SSB sample magnified 25 times (left) and 100 times (right)

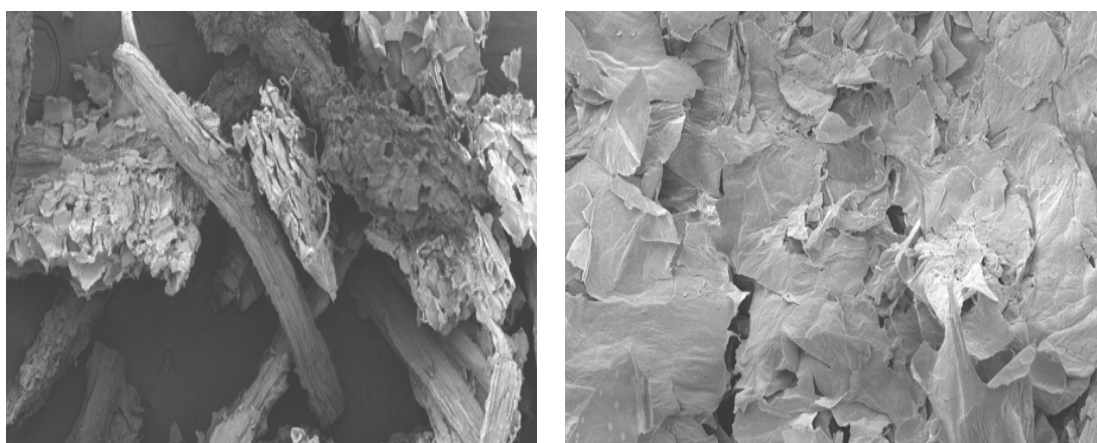


Figure 2(d). SEM image of BSB sample magnified 25 times (left) and 300 times (right)

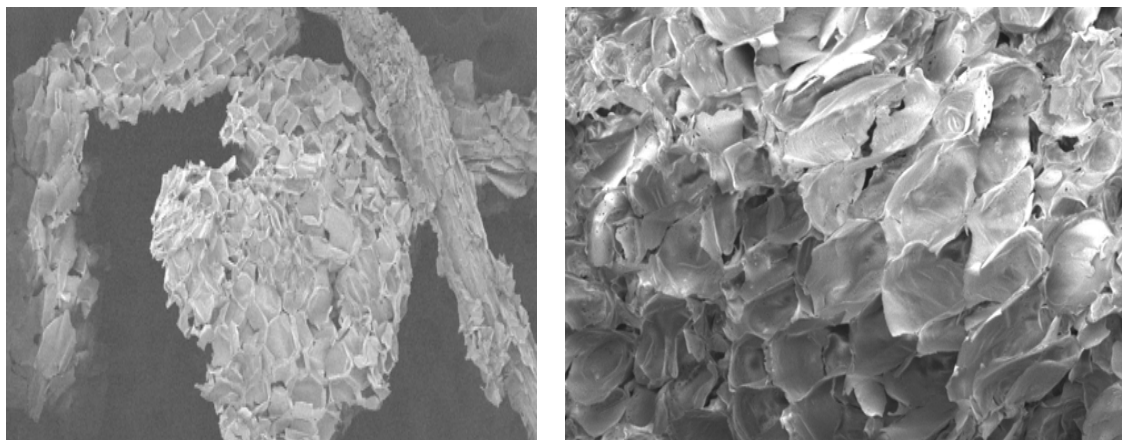


Figure 2(e). SEM image of ASSB sample magnified 25 times (left) and 200 times (right)

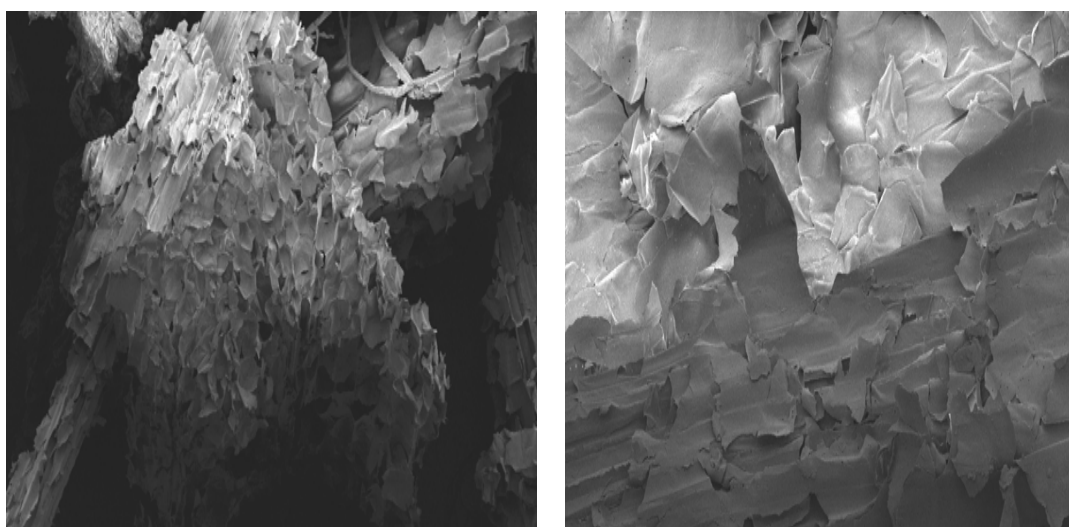


Figure 2(f). SEM image of AASB sample magnified 25 times (left) and 140 times

Figure 2(a) showed that all the SB samples have similar physical appearances except for BSB which was darker yellow and has a greater tendency to stick to each other. BSB also had a higher density compared to the rest of the samples. The surface texture and shape of each SB sample could be differentiated using SEM. Figure 2(b) showed that NSB is flaky and rough surface with regular rectangular honeycomb structure of cellulose in its surface as magnified 25 to 220 times. Similar surface characteristic was also shown in Figure 2(c), after NSB experiencing sodium chloride treatment. Square shapes and more irregular honeycomb pattern of cellulose was shown in Figure 2(e), after experiencing sulphuric acid treatment. Figure 2(d) and 2(f) showed that SB surface has rougher surface due to oxidation process of sodium hydroxide and acetic acid. More irregular pattern and collapsing honeycomb cellulose structure could be seen too. Chung et al (2011) measured the BET surface area of untreated SB of 0.9 m²/g could be improved up to 60% after acetic acid treatment.

Table 1. Density, crystallinity and crystallite size of SB samples

Sample	Density (g/cm ³)	Crystallinity (%)	Crystallite size (nm)
NSB	0.0342	29.038	3.300
SSB	0.0342	37.014	3.321
BSB	0.0513	65.329	3.192
ASSB	0.0342	54.848	3.100
AASB	0.0342	59.359	3.069

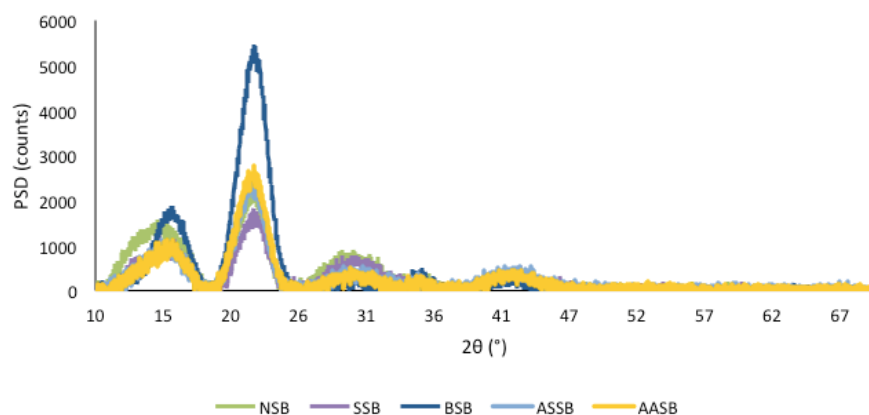


Figure 3. XRD spectra of SB samples

With reference to the XRD spectra of SB samples in Figure 3, the diffraction spectrum at 15 - 18° and 22° represent cellulose in the amorphous and crystalline forms respectively. As expected the simple pre-treatments of SB samples was not expected to produce nano crystallite sizes but to change the hydrophobicity of SB samples. With the removal of lignin by NaOH base, the plant cell walls in BSB lose their structural integrity and cause the cellulose to collapse into an amorphous form as it was clearly shown in SEM scan in Figure 2(d). BSB had a much higher crystallinity as compared to the rest of the SB samples. In Table 1, BSB showed the highest crystallinity of cellulose *CrI* (1), followed by AASB, ASSB, SSB and NSB. The high density of BSB is in agreement with its high crystallinity. According to the Scherrer's equation (2), the order of crystallite sizes were shown as NSB, SSB, BSB, ASSB and AASB. The effect of crystallite size of cellulose on oil adsorption capacity for the SB samples may be partly co-related.

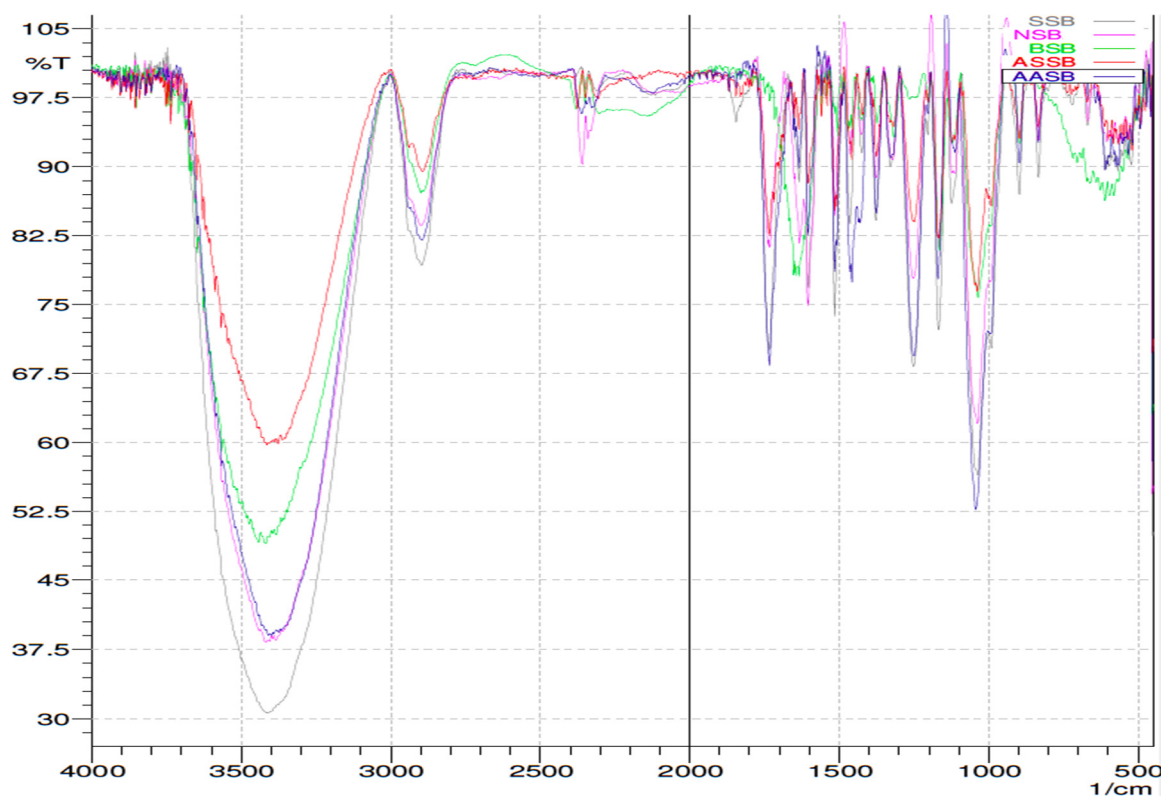


Figure 4. FTIR spectra of SB samples

Figure 4 showed FTIR spectra of SB samples with the bands associated with the hydroxyl O-H group, lignin and acetyl group. The bands at 3400 cm^{-1} and 2910 cm^{-1} are assigned to O – H stretching and C – H stretching respectively in all five SB samples. For O-H stretching, SSB has the highest intensity, followed by similar values from NSB and AASB. The second lowest intensity is BSB and the lowest intensity is ASSB. The O-H trend for BSB and ASSB could have been due to the partial and significant removal of hemicellulose respectively. The lower removal of O-H groups in AASB compared to ASSB could have been due to the fact that acetic acid is a weaker acid than sulphuric acid. For 1600 cm^{-1} and 1510 cm^{-1} , they represent the aromatic skeletal vibrations. Generally, SSB shows the highest intensity of lignin, followed by AASB, NSB, ASSB and finally BSB. For acetyl group, 1750 cm^{-1} represents the C = O bond, 1380 cm^{-1} represents the – C – CH₃ – bond and 1240 cm^{-1} represents the – C – O – bond. For all the acetyl associated bands, AASB has one of the highest intensities. Hence, it is possible that some acetylation could have taken place. Band at around $1040 - 1060\text{ cm}^{-1}$ is assigned to C – O (primary alcohol) which the AASB is having the lowest intensity at this band.

3.2 Water adsorption onto SB samples

Adsorption of water measures the hydrophilicity of the material. The higher the water adsorption, the more hydrophilic the material is. Table 2 showed the results of the water adsorption experiments indicating that NSB is the most hydrophilic, followed by SSB, AASB, ASSB and BSB. A duplicate of volume measurements using two different batches of SB column were conducted as shown as V1 and V2 in Table 2.

Table 2. Adsorption of water onto 1 g of NSB, SSB, BSB, ASSB and AASB

SB types	Initial volume (mL)	Water filtrate volume (mL)			Average water adsorption (mL/g)
		V1	V2	Average V	
NSB	100	84.0	85.0	84.5	15.5
SSB		84.5	86.0	85.3	14.7
BSB		88.0	89.0	88.5	11.5
ASSB		85.0	88.0	86.5	13.5
AASB		87.0	85.0	86.0	14.0

These results are generally in line with the intensity of O-H groups in different SB types, with NSB and SSB having the greatest amount of O-H and ASSB and BSB having the least amount of O-H.

3.3 Adsorption of Oil and Water Mixture onto SB Samples

The average water adsorption from adsorption of oil and water mixture (Table 3) shows a similar trend as that of adsorption of water alone in Section 3.2. Adsorption of oil measures the hydrophobicity of the material. The higher the oil adsorption, the more hydrophobic the material is. For oil adsorption, AASB shows the greatest hydrophobicity, followed by ASSB, NSB, SSB and BSB. This is generally in agreement with the material characterisation where AASB has one of the highest lignin and acetyl group content while BSB has the lowest lignin content. AASB may be the most hydrophobic sample among the SB samples. A duplicate of volume measurements using two different batches of SB column were conducted as shown as V1 and V2 in Table 3.

Table 3. Oil adsorption onto 1 g of NSB, SSB, BSB, ASSB and AASB in the mixture of oil and water

SB types	Water and oil mixture (mL)	Water filtrate volume (mL)			Oil filtrate volume (mL)			Avg. water adsorption (mL/g)	Avg. oil adsorption (mL/g)
		V1	V2	Avg vol	V1	V2	Avg vol		
NSB	100	40.0	39.0	39.5	40.0	39.0	39.5	10.50	10.50
SSB		37.0	38.0	37.5	41.0	41.0	41.0	12.5	9.0
BSB		41.5	42.0	41.75	41.5	41.0	41.25	8.25	8.75
ASSB		39.5	39.0	39.25	38.5	39.0	38.75	10.75	11.25
AASB		40.0	43.0	41.5	38.0	36.0	37.0	8.5	13.0

4. Conclusion

The presence of hydrophilic hydroxyl groups and hydrophobic lignin in all SB samples has enabled them to adsorb water and oil. However, a greater amount of hydroxyl groups in NSB and SSB have resulted in higher water adsorption while a greater amount of lignin groups in AASB have resulted in higher oil adsorption. The result of column experiment indicated that only AASB may be identified as the most hydrophobic sample and BSB the least hydrophobic sample. FTIR analysis showed that simple chemical treatment of soaking SB in acetic acid overnight was able to substitute some hydroxyl groups with acetyl groups. Although ASSB has the second highest oil adsorption after AASB, use of sulphuric acid for chemical treatment may be more environmentally unfriendly than acetic acid. Hence, AASB is the most suitable material for oil adsorption in aqueous environments.

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