



# Synthesis and Characterisation of Pyridinium-Based Ionic Liquid as Activating Agent in Rubber Seed Shell Activated Carbon Production for CO<sub>2</sub> Capture

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## ARTICLE INFO

### Article history:

Received 16 December 2020

Received in revised form 13 March 2021

Accepted 20 March 2021

Available online 14 April 2021

### Keywords:

Activated carbon (AC); Ionic Liquid (IL); 1-butylpyridinium bis (trifluoromethylsulfonyl) imide [C<sub>4</sub>Py][Tf<sub>2</sub>N]; Nuclear Magnetic Resonance (NMR); Fourier Transform Infrared Spectroscopy (FTIR); Rubber seed shell (RSS)

## ABSTRACT

The use of an activating agent in chemical activation of activated carbon (AC) production is very important as it will help to open the pore structure of AC as adsorbents and could enhance its performance for adsorption capacity. In this study, a pyridinium-based ionic liquid (IL), 1-butylpyridinium bis (trifluoromethylsulfonyl) imide, [C<sub>4</sub>Py][Tf<sub>2</sub>N] has been synthesized by using anion exchange reaction and was characterized using few analyses such as <sup>1</sup>H-NMR, <sup>13</sup>C-NMR and FTIR. Low-cost AC was synthesized by chemical activation process in which rubber seed shell (RSS) and ionic liquid [C<sub>4</sub>Py][Tf<sub>2</sub>N] were employed as the precursor and activating agent, respectively. AC has been prepared with different IL concentration (1% and 10%) at 500°C and 800°C for 2 hours. Sample AC2 shows the highest S<sub>BET</sub> and V<sub>T</sub> which are 392.8927 m<sup>2</sup>/g and 0.2059 cm<sup>3</sup>/g respectively. The surface morphology of synthesized AC can be clearly seen through FESEM analysis. A high concentration of IL in sample AC10 contributed to blockage of pores by the IL. On the other hand, the performance of synthesized AC for CO<sub>2</sub> adsorption capacity also studied by using static volumetric technique at 1 bar and 25°C. Sample AC2 contributed the highest CO<sub>2</sub> uptakes which is 50.783 cm<sup>3</sup>/g. This current work shows that the use of low concentration IL as an activating agent has the potential to produce porous AC, which offers low-cost, green technology as well as promising application towards CO<sub>2</sub> capture.

## 1. Introduction

Over the few past years, the emission of carbon dioxide, CO<sub>2</sub> in our environment kept increasing day by day and thus causing global warming and climate change. Human activities such as the combustion of fossil fuels are one of the major contributors to the emission of CO<sub>2</sub> into the environment. This issue has gained attention among researchers to study for CO<sub>2</sub> capture technologies such as absorption, adsorption, cryogenic and membrane separations. Among these, the most conventional method for the emission of CO<sub>2</sub> in the environment is amine-absorption using

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MEA, DEA and MDEA as solvents. However, this technology has a few disadvantages such as high heat capacity and energy consumption, corrosion in equipment used and caused foaming [10,13,20,25]. Therefore, the new evolution had attempted to overcome this problem. As a result, the development of environmentally friendly, low capital and operating cost and high thermal stability has been given much attention in group researchers. Adsorption techniques by using solid adsorbent from biomass such as activated carbon (AC) is one of the promising alternatives to increase the CO<sub>2</sub> adsorption.

AC is widely used and applied in many applications such as adsorption of heavy metals, decolorization, purification, catalysis, catalytic support, electrochemical capacitor and gas storage [8,12]. AC has many advantages such as low cost, highly porous structure, firm and stable in shape, has a broad surface area with high adsorption capacity, organized macro, meso and microporous structure and a high degree of surface reactivity [8,22]. Besides, biomass-based AC offers high their abundantly available, cheap, renewable, easy processing, high volume applications, less abrasive to equipment, low cost for waste disposal and reduces waste produced [12]. There are many literature studies used biomass as their precursor in converting into AC such rice straw matter, olive stones, nutshells, coffee grounds and rubber seed shell (RSS) and many more [3,4,11,12,17,21,31]. AC can be produced via physical or chemical activation. Traditionally, chemical activation by using excess of chemical agents such as ZnCl<sub>2</sub>, H<sub>3</sub>PO<sub>4</sub> and KOH before undergoes carbonization step under inert condition at temperature between 500°C to 800°C is much more preferred due to its offer may advantages such as gives high surface area, more porous structure and high surface adsorption capacity [1,4,8,16]. Besides their advantages, they also have disadvantages such as chemical used is not environmentally friendly and can cause corrosion towards the equipment used [2].

Recently, ionic liquid (IL) have been applied in CO<sub>2</sub> application. IL have been reported exhibiting good attractive properties such as have melting point less than 100°C, not flammable which means not easily to burn, good ionic conductivity, have negligible low vapour pressure, have a high stability both chemically and thermally and acts as good solvent with widely varying polarity such as polar and non-polar [14,24,37]. Additionally, IL also can be considered as a safe, clean and environmentally friendly solvent and also known as “green solvents” [26,33,37]. IL is mainly composed of cations and anions and these combinations can be tuneable depending on the specific chemical applications. Previously, CO<sub>2</sub> capture by using imidazole-based IL pairing has extensively used with different anions. Anion nature plays a primary role in CO<sub>2</sub> solubility. The fluorinated anion such as Tf<sub>2</sub>N, PF<sub>6</sub> and BF<sub>4</sub> will give a higher affinity towards CO<sub>2</sub> capture than other anions such as N(CN)<sub>2</sub> and NO<sub>3</sub> [32]. Despite high CO<sub>2</sub> solubility, it also has a major problem such as the viscosity of IL itself thus limit the mass transfer of between CO<sub>2</sub> gas and IL [15].

Besides, IL itself also can be as precursors to produce porous carbon [23]. However, the use of IL as an activating agent in the production of AC using biomass as a precursor are still very scarce. Herein, we present pyridinium-based IL, 1-butylpyridinium bis (trifluoromethylsulfonyl) imide, [C<sub>4</sub>Py][Tf<sub>2</sub>N] as an activating agent to produce porosity on the surface of AC. Besides, this pyridinium-based IL offers low cost and toxicity, high thermal stability and biodegradable and high CO<sub>2</sub> solubility compared to imidazolium-based IL [34]. Anion like Tf<sub>2</sub>N is chosen as it can act as pyrogens in which help in the formation of pore [18]. In this study, the production of AC was varied with different concentrations of IL and temperature including tested with CO<sub>2</sub> adsorption to prove low concentration of IL can be one of the activating agents to replace conventional activating agents including offers as a better adsorbent for CO<sub>2</sub> capture application.

## 2. Experimental

### 2.1 Materials and Reagents

The chemicals and solvent used in the synthesis of  $[C_4Py][Tf_2N]$  IL are pyridine (109728, Merck,  $\geq 99.5\%$ ), 1-bromobutane (801602, Merck,  $\geq 98\%$ ), ethyl acetate (109623, Merck, 99.5%), lithium bis (trifluoromethanesulfonyl)imide,  $LiTf_2N$  (544094, Sigma Aldrich,  $\geq 95\%$ ) and ethanol (64-17-5, HmbG, 95%). All chemicals were used without any drying and further purification. Rubber seed shell (RSS) collected from a local rubber plantation, Institut Petanian Titi Gantong, Perak was used as a precursor material in this study.

### 2.2 Synthesis of Ionic Liquid

For the preparation of 1-butylpyridinium bis (trifluoromethylsulfonyl) imide,  $[C_4Py][Tf_2N]$  IL, two steps procedure was used as reported by Enayati and Faghihian [9] and Yunus *et al.*, [36].

The first step for the preparation of 1-butylpyridinium bromide,  $[C_4Py][Br]$  was quaternization reaction. Under nitrogen atmosphere, a mixture of pyridine (0.5 mol) and 1-bromobutane (0.55 mol) in ethyl acetate was refluxed and stirred  $50^\circ C$  for 72 h until a white precipitate was formed. The white precipitate was washed with ethyl acetate for several times and then dried in a rotary evaporator at  $70^\circ C$  and finally vacuum oven for 24 h to remove an excess of solvent.

The second step for the preparation of  $[C_4Py][Tf_2N]$  was anion exchange reaction. In this step, an equimolar mixture of  $[C_4Py][Br]$  and  $LiTf_2N$  in deionized water was stirred at room temperature for 24 h. The aqueous phase was removed from the product and then the product was washed several times with deionized water until it is free from halogenated ion. The product was then dried in a rotary evaporator at  $70^\circ C$  for several hours and finally vacuum oven at  $50^\circ C$  for 72 h to ensure complete removal of water content in the product.

### 2.3 Preparation and Carbonization of RSS

Firstly, the fresh RSS was washed repeatedly with distilled water to remove all dust and foreign materials before drying at  $110^\circ C$  overnight. Then, these dried RSS were crushed, grinded and sieved to a particle size of 1 mm. Subsequently, the sieved RSS were kept into an airtight container until further use for the experimental work. For activation, samples were impregnated with different concentrations of IL which are 1% and 10% (1g and 10g) in 100 mL of ethanol. Ethanol was used to provide homogeneity during the impregnation process. About 10g of RSS was used for the preparation of each sample. The mixture was mixed in a Schott bottle with constant stirring at room temperature for 24 hours to ensure the IL is fully adsorbed into RSS. Then, the mixture was filtered and dried at  $110^\circ C$  overnight in an oven, before carbonization process. The impregnated RSS was carbonized in a tubular furnace at  $500^\circ C$  and  $800^\circ C$  under a steady flow of nitrogen gas for 2 hours. Afterward, the produced AC was cooled at room temperature before washed a few times with warm distilled water until pH between 6.5-7 is achieved. Later, the AC was dried at  $110^\circ C$  overnight in an oven and was kept into an airtight container for characterization.

## 2.4 Sample Characterization

### 2.4.1 Ionic liquid characterization

The purity and chemical structure of  $[C_4Py][Tf_2N]$  IL was verified by using Nuclear Magnetic Resonance, NMR (Bruker Avance 500 MHz spectrometer). A sample size of  $\leq 10$ mg was used and dissolved in  $\sim 0.6$ cm<sup>3</sup> of deuterated methanol solvent (MeOD). The <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded at room temperature in terms of chemical shifts and multiplicities. The chemical shift denoted as  $\delta$  and reported in parts per million (ppm) while spin multiplicities are abbreviated as s (singlet); d (doublet), t (triplet) and m (multiplet). The surface functional groups of  $[C_4Py][Tf_2N]$  IL were analyzed by using Fourier Transform Infrared Spectroscopy, FTIR (Perkin Elmer Spectrum One). The spectra were recorded between 4000 to 400 cm<sup>-1</sup> resolution in the transmission mode.

### 2.4.2 Activated carbon characterization

The surface area and porosity of AC samples were evaluated by using Micromeritics ASAP 2020 (Micromeritics Instruments). Before analysis, each AC sample was degassed at 200°C for 16 hours to eliminate all the moisture and contaminant from the surface. The specific surface area of the AC samples was calculated by using Brunauer-Emmett-Teller (BET) equation from nitrogen adsorption isotherm. The total pore volume was determined evaluated at P/P<sub>o</sub> of 0.99. Besides, the surface morphology was analyzed by using Field Emission Scanning Electron Microscopy, FESEM (ZEISS Microscopy).

## 2.5 CO<sub>2</sub> Adsorption Study

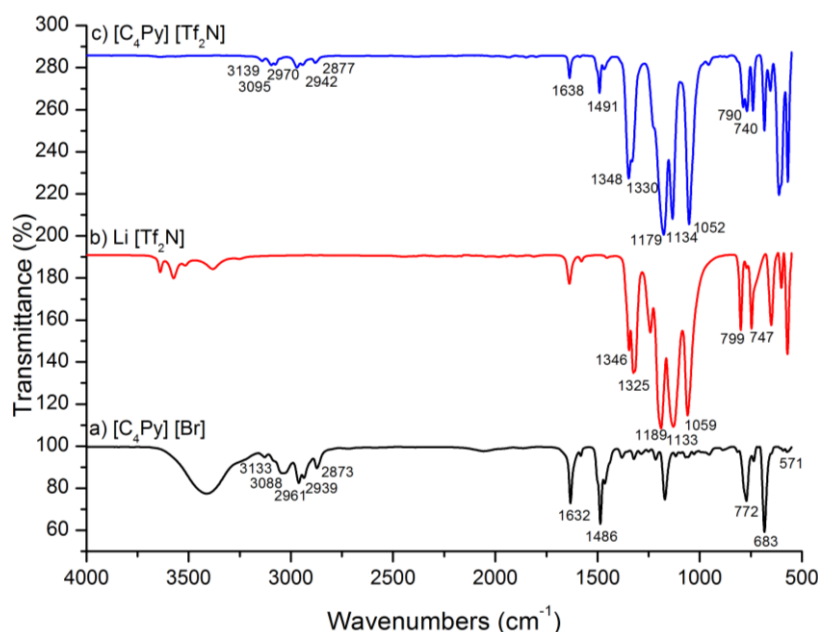
CO<sub>2</sub> adsorption study of the AC samples was studied using High Pressure Volumetric Analyzer II, HPVA (Micromeritics Instruments) static volumetric analyzer at 1 bar and 25°C. Each AC sample was degassed at 200°C for 16 hours before adsorption measurements. The volumetric method was described by Rashidi *et al.*, [30].

## 3. Result and Discussion

### 3.1 Characterization of $[C_4Py][Tf_2N]$ Ionic Liquid

#### 3.1.1 FTIR spectra

The FTIR spectra of pure  $[C_4Py][Br]$ , Li  $[Tf_2N]$  and  $[C_4Py][Tf_2N]$  are shown in Figure 1. As reported by Enayati and Faghihian [9] and Zhang *et al.*, [38] for  $[C_4Py][Br]$  IL as in Figure 1(a), the butylpyridinium, C<sub>4</sub>Py cation characteristic can be observed at: 3133 cm<sup>-1</sup>, 3041 cm<sup>-1</sup> (aromatic C-H), 2961 cm<sup>-1</sup>, 2939 cm<sup>-1</sup>, 2873 cm<sup>-1</sup> (aliphatic C-H), 1632 cm<sup>-1</sup> (aromatic C-C), 1486 cm<sup>-1</sup>, 772 cm<sup>-1</sup> (-CH<sub>3</sub>), 683 cm<sup>-1</sup> (-CH<sub>2</sub>), and for Br anion at : 571 cm<sup>-1</sup> (C-Br). Besides, as demonstrated by Ramesh and Lu [27], there are some important peaks in FTIR spectra of pure Li  $[Tf_2N]$  and these can be seen in Figure 1(b). The bis (trifluoromethylsulfonyl) imide, Tf<sub>2</sub>N anion can be observed at: 1346 cm<sup>-1</sup>, 1325 cm<sup>-1</sup>, 1131 cm<sup>-1</sup> (C-SO<sub>2</sub>-N), 1189 cm<sup>-1</sup> (-CF<sub>3</sub>), 1059 cm<sup>-1</sup> (S=O), 799 cm<sup>-1</sup> (combination of C-S and S-N), 747 cm<sup>-1</sup> (S-N). As a result, the desired  $[C_4Py][Tf_2N]$  IL shows an evidence for the appearance of the cation and anion characteristics from the previous precursor salt,  $[C_4Py][Br]$  and Li  $[Tf_2N]$  using anion exchange reaction. The band can be observed and summarized as per Table 1. Therefore, the above result proved that the desired ionic liquid which is  $[C_4Py][Tf_2N]$  IL was successfully synthesized.



**Fig. 1.** FTIR spectra for (a) [C<sub>4</sub>Py][Br]; (b) Li [Tf<sub>2</sub>N]; (c) [C<sub>4</sub>Py][Tf<sub>2</sub>N]

**Table 1**

Summary FTIR spectra bands of [C<sub>4</sub>Py][Tf<sub>2</sub>N]

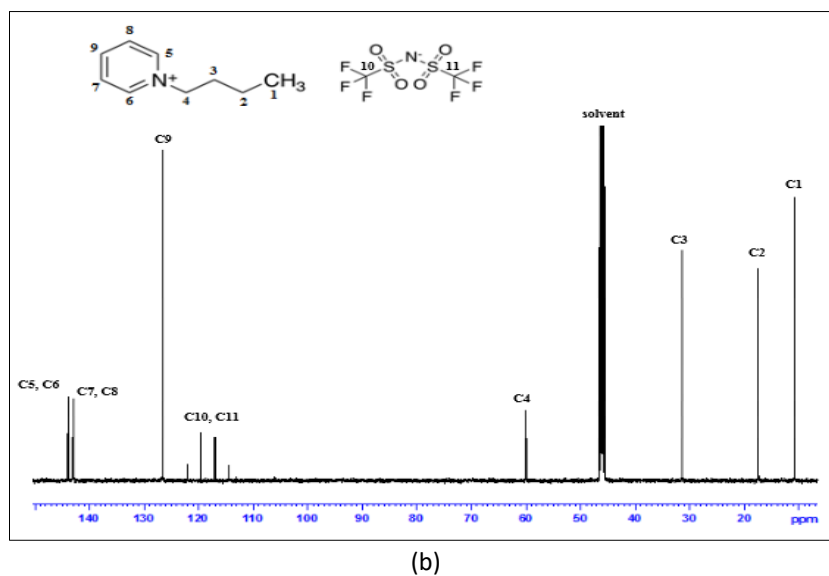
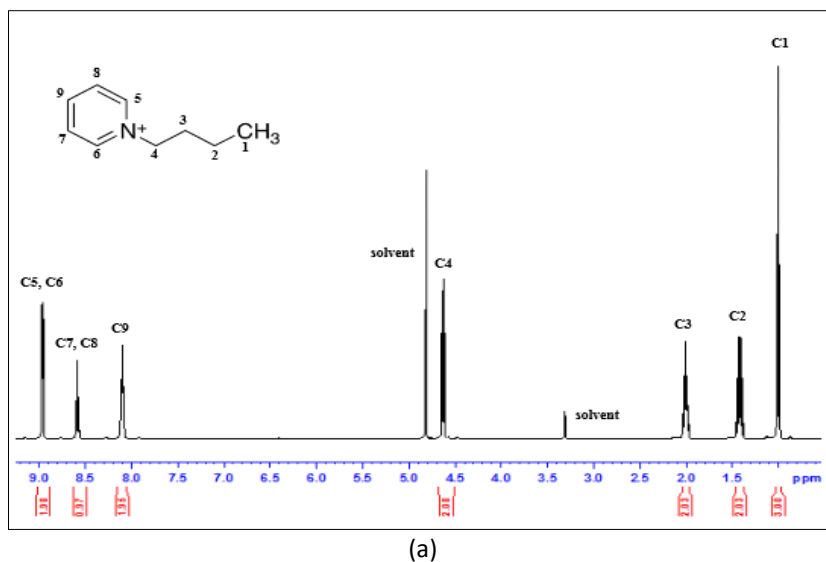
| Band Assigned              | Wavelength (cm <sup>-1</sup> ) |
|----------------------------|--------------------------------|
| Aromatic C-H               | 3139, 3095                     |
| Aliphatic C-H              | 2970, 2942, 2877               |
| Aromatic C-C               | 1638                           |
| -CH <sub>3</sub>           | 1491                           |
| C-SO <sub>2</sub> -N       | 1348, 1330, 1134               |
| -CF <sub>3</sub>           | 1179                           |
| S=O                        | 1052                           |
| Combination of C-S and S-N | 790                            |
| S-N                        | 740                            |

### 3.1.2 NMR spectroscopy

The synthesized IL, 1-butylpyridinium bis (trifluoromethylsulfonyl) imide, [C<sub>4</sub>Py][Tf<sub>2</sub>N] was characterized by the proton and carbon NMR (<sup>1</sup>H NMR and <sup>13</sup>C NMR) to evaluate purity and chemical structure of the samples in deuterated methanol (MeOD) solvent. Based on the structure, the total number of Hydrogen and Carbon atoms is 14 and 11 respectively. Both <sup>1</sup>H NMR and <sup>13</sup>C NMR spectrum of [C<sub>4</sub>Py][Tf<sub>2</sub>N] ionic liquid is shown in Figure 2, further demonstrated its structure. In the <sup>1</sup>H NMR spectrum (Figure 2(a)), the chemical shift peaks at 8.593 ppm, 8.585 ppm and 8.099 ppm are related to aromatic pyridine ring (assigned as C5 to C9). The chemical shift peaks of C5 and C6 is the same because of their share with the same environment based on the structure. The same concept applied for C7 and C8. The peak at 4.630 ppm is assigned for N-CH<sub>2</sub> bond (C4) while the peaks at 1.414 ppm and 2.008 ppm were assigned for CH<sub>2</sub> bond (named C2 and C3). Lastly, the chemical shift peaks at 0.998 ppm is referred to CH<sub>3</sub> bond and is assigned as C1. In the <sup>13</sup>C NMR spectrum (Figure 2(b)), the chemical shift peaks at 143.95 ppm, 142.99 ppm and 126.62 ppm are related to aromatic pyridine ring (C5-C9). For the peaks at 119.57 ppm is referred to carbon in bis (trifluoromethylsulfonyl) imide [Tf<sub>2</sub>N] (assigned as C10 and C11). The peak at 60.06 ppm is assigned to N-CH<sub>2</sub>. The peak at 31.47 ppm and 17.48 ppm is related to CH<sub>2</sub> (C2 and C3) and at 10.80 ppm is attributed to CH<sub>3</sub> (C1). All the NMR

data of [C<sub>4</sub>Py][TF<sub>2</sub>N] IL presented shows close similarity between chemical shift reported by Yunus *et al.*, [36]. The summary for NMR spectroscopy (<sup>1</sup>H NMR and <sup>13</sup>C NMR) for [C<sub>4</sub>Py][Tf<sub>2</sub>N] IL are as follows

- <sup>1</sup>H NMR (500MHz, MeOD): δ 0.998 [t, 3H, CH<sub>3</sub>], δ 1.414 [m, 2H, CH<sub>2</sub>], δ 2.008 [m, 2H, CH<sub>2</sub>], δ 4.630 [t, 2H, N-CH<sub>2</sub>], δ 8.099 [t, 2H, Py], δ 8.585 [t, 1H, Py], δ 8.953 [d, 2H, Py]. Yield:83%
- <sup>13</sup>C NMR (125MHz, MeOD): δ 143.95, 142.99, 126.62, 119.57, 60.06, 31.47, 17.48, 10.80. Yield: 87%



**Fig. 2.** (a) <sup>1</sup>H NMR (b) <sup>13</sup>C NMR of [C<sub>4</sub>Py][Tf<sub>2</sub>N] IL

### 3.2 Characterization of Synthesized Activated Carbon

#### 3.2.1 BET surface area

Table 2 shows the summary of the preparation conditions and synthesized AC produced from RSS impregnated with IL. During carbonization, all the moisture, volatile matter and non-carbon are removed to develop porosity and surface area to enhance their adsorption capacity [7]. Besides, the concentration and carbonization temperature play an important role in the production of AC. From the table, the S<sub>BET</sub> of raw RSS is 19.6575 m<sup>2</sup>/g and V<sub>T</sub> is 0.0020 cm<sup>3</sup>/g. After carbonization at 500°C,



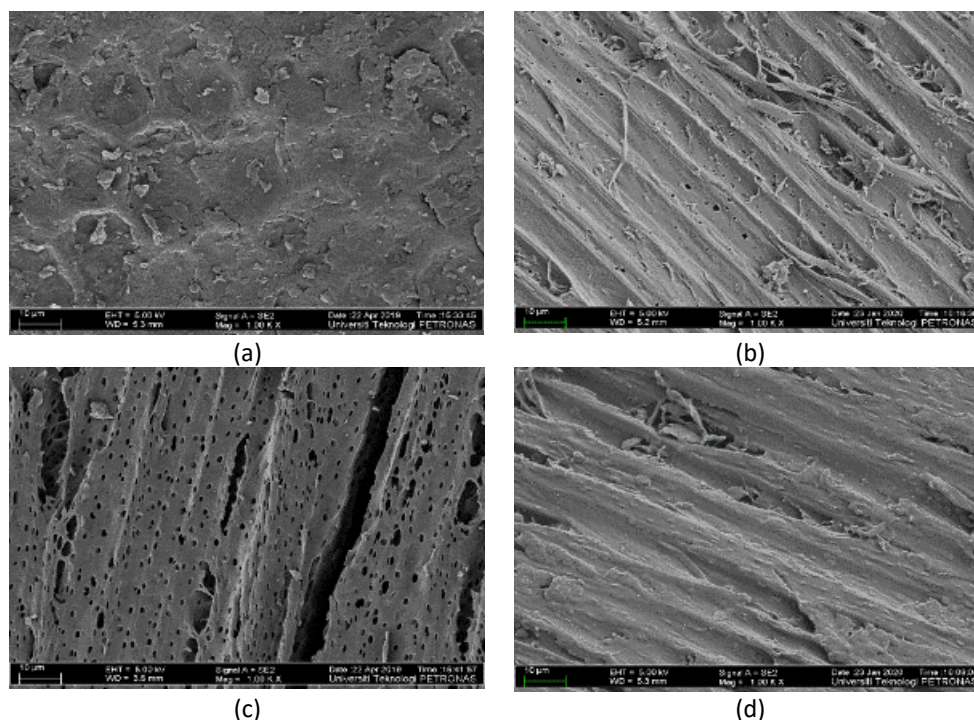
sample AC1 at 1% IL concentration shows an increase of  $S_{BET}$  and total pore volume,  $V_T$  which are  $107.5465 \text{ m}^2/\text{g}$  and  $0.0451 \text{ cm}^3/\text{g}$  compared to sample AC10 at 10% IL concentration where  $S_{BET}$  and  $V_T$  are decreased drastically to  $0.0113 \text{ m}^2/\text{g}$  and  $0 \text{ cm}^3/\text{g}$ , respectively. A similar trend was observed as reported by Azry *et al.*, [6], as the impregnation ratio (IR) increases, the surface area decreases. From their study uses sawdust and phosphoric acid,  $\text{H}_3\text{PO}_4$  as their precursor and activating agent, the  $S_{BET}$  decreases from  $1135.624 \text{ m}^2/\text{g}$  to  $399.013 \text{ m}^2/\text{g}$  at IR of 1:1 to 1:3 and temperature of  $500^\circ\text{C}$  for 30 minutes. This is due to the maximum amount of acid covering all the surface thus inhibit contact with the sample surface and lowering the activation process. In this study, AC10 shows this phenomenon as it  $S_{BET}$  and  $V_T$  are decreased due to the excessive IL filled up the pores on the surface and completely blocked the pore, thus slow down the activation process to form pores [5]. It can conclude, IL functions as an activating agent to create new pores at low concentrations. Additionally, after increase temperature at  $800^\circ\text{C}$ , the sample AC2 shows the highest  $S_{BET}$  and  $V_T$  which are  $392.8927 \text{ m}^2/\text{g}$  and  $0.2059 \text{ cm}^3/\text{g}$  respectively. This is because all the volatile matter had been released and develop more pores at a high temperature. Sample AC2 can be considered as a better adsorbent for  $\text{CO}_2$  adsorption.

**Table 2**  
 Surface Area and Porosity of Raw RSS and Synthesized AC

| Sample Name | Temperature ( $^\circ\text{C}$ ) | Concentration (%) | Specific Surface Area, $S_{bet} \text{ (m}^2/\text{g)}$ | Total Pore Volume, $V_t \text{ (cm}^3/\text{g)}$ |
|-------------|----------------------------------|-------------------|---|--|
| Raw RSS     | -                                | -                 | 19.6575   | 0.0020   |
| AC1         | 500                              | 1                 | 107.5465  | 0.0451   |
| AC2         | 800                              | 1                 | 392.8927  | 0.2059   |
| AC10        | 500                              | 10                | 0.0113  | 0  |

### 3.2.2 FESEM analysis

In order to verify the surface area and porosity in Table 2, FESEM analysis was conducted to observe the surface morphology of raw RSS and synthesized AC (AC1, AC2 and AC10). Besides, the efficiency of adsorption capacity can be determined from the surface morphology. As shown in Figure 3(a), the morphology of raw RSS is irregularly shaped, rough and very low pores on the surface of a precursor. After carbonization, sample AC1 had a very few pores at temperature  $500^\circ\text{C}$  as shown in Figure 3(b). This shows the development of pores still an early stage as the activation rate is low at this temperature. As the activation temperature increased at  $800^\circ\text{C}$ , sample AC2 shows more pores rapidly developed as in Figure 3(c). The formation of pores in AC2 would help for  $\text{CO}_2$  diffuse into the pores thus increase the  $\text{CO}_2$  adsorption capacity. This agrees well as sample AC2 has the highest  $S_{BET}$  and  $V_T$ . Besides, the high concentration of IL caused a coating on the surface of AC10 and completely blocked the formation of pore including preventing from  $\text{CO}_2$  adsorption process to occur. The surface morphology of AC10 is shown as Figure 3(d).



**Fig. 3.** FESEM Analysis at magnification of 1000X (a) Raw RSS (b) AC1 at 1% IL concentration at 500°C (c) AC2 at 1% IL concentration at 800°C (d) AC10 at 10% IL concentration at 500°C

### 3.3 CO<sub>2</sub> Adsorption Analysis

The CO<sub>2</sub> adsorption capacity test for each sample was conducted at 25°C and 1 bar and shown in Figure 4. The highest CO<sub>2</sub> adsorption capacity was observed in sample AC2 at 50.783 cm<sup>3</sup>/g, followed by AC1 at 33.927 cm<sup>3</sup>/g and AC10 at 10.022 cm<sup>3</sup>/g. AC2 contributes to the highest CO<sub>2</sub> adsorption capacity because it has a high surface area compared to AC1 and AC10. A high surface area allows CO<sub>2</sub> to diffuse into the pores of AC [28]. Additionally, the performance of CO<sub>2</sub> adsorption capacity of sample AC2 in this study also is compared with other published literature. From these, it can be seen that sample AC2 has higher adsorption capacity compared to Norit<sup>®</sup> SX2 (commercial AC) at 42.112 cm<sup>3</sup>/g and palm kernel shell (PKS) AC at 47.712 cm<sup>3</sup>/g. The highest CO<sub>2</sub> adsorption capacity is bamboo AC at 67.200 cm<sup>3</sup>/g followed by rice husk AC at 69.440 cm<sup>3</sup>/g. From Figure 4, it can be concluded that the CO<sub>2</sub> adsorption capacity of sample AC2 is comparable with other biomass materials.



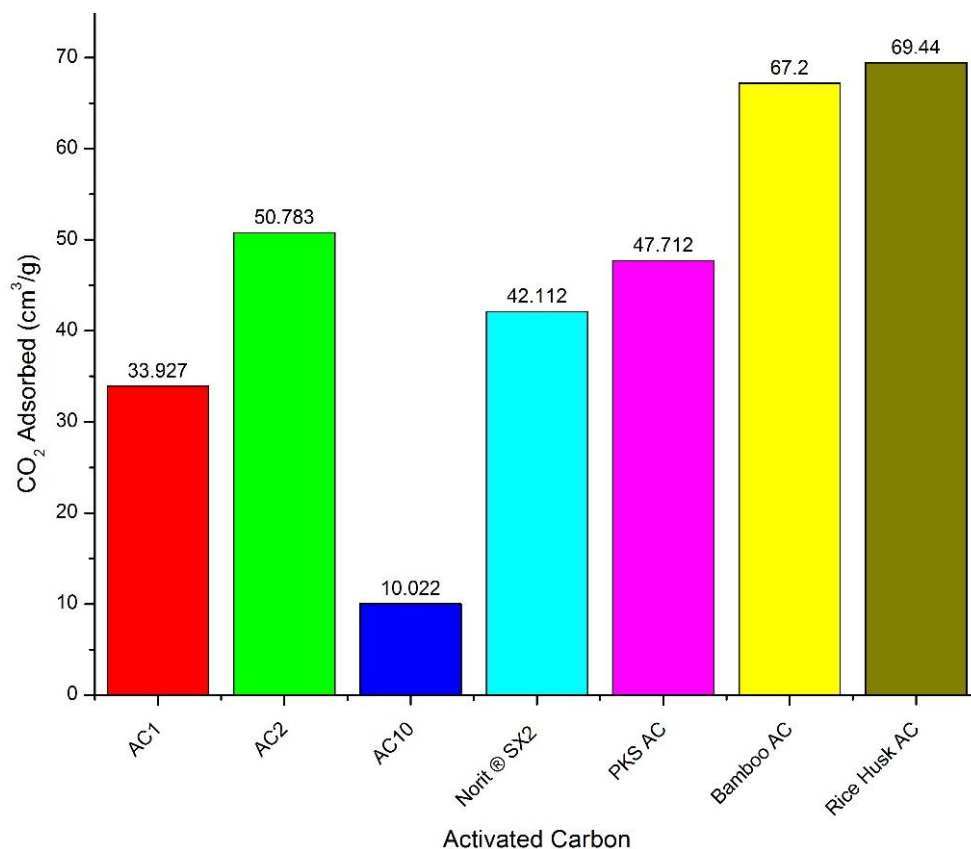


Fig. 4. CO<sub>2</sub> Adsorption Capacity at 25°C and 1 bar [19, 29, 35]

#### 4. Conclusion

As a conclusion, the pyridinium based IL, 1-butylpyridinium bis (trifluoromethylsulfonyl) imide, [C<sub>4</sub>Py][Tf<sub>2</sub>N] was successfully synthesized and can be used as an activating agent in the production of activated carbon due to it can promote porous carbonaceous structure during carbonization and also can enhance the performance of CO<sub>2</sub> adsorption. The sample AC2 contributes to the highest surface area and total pore volume at a low concentration of IL. Moreover, its performance is tested with a static volumetric instrument and shows the highest CO<sub>2</sub> adsorption capacity. Therefore, it can conclude that a low concentration of IL could minimize the use of IL in which offers low-cost and green technology towards CO<sub>2</sub> capture application.

#### Acknowledgements

This work was supported by Yayasan Universiti Teknologi PETRONAS (YUTP-FRG 015LCO-068). The authors also gratefully acknowledge Universiti Teknologi PETRONAS for the technical and facilities support. Support from the Ministry of Education Malaysia through the HICOE award to Centre for Biofuel and Biochemical Research (CBBR) is also acknowledged.

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