Ionic liquid-impregnated activated carbon for biohydrogen purification in an adsorption unit

N Y Yusuf, M S Masdar, W N R W Isahak, D Nordin, T Husaini, E H Majlan, S A M Rejab and C L Chew

1 Department of Chemical and Process Engineering, Faculty of Engineering and Built Environment, 43600, UKM Bangi, Selangor, Malaysia
2 Research Centre for Sustainable Process Technology (CESPRO), Faculty of Engineering & Built Environment, 43600, UKM Bangi, Selangor, Malaysia
3 Fuel Cell Institute, Universiti Kebangsaan Malaysia, 43600, UKM Bangi, Selangor, Malaysia
4 Sime Darby Research Sdn. Bhd., Jalan Pulau Carey, 42960 Pulau Carey, Selangor, Malaysia

E-mail: shahbud@ukm.edu.my

Abstract. Biological methods for hydrogen production (biohydrogen) are known as energy intensive and can be operated at ambient temperature and pressure; however, consecutive productions such as purification and separation processes still remain challenging in the industry. Various techniques are used to purify and separate hydrogen. These techniques include the use of sorbents/solvents, membranes and cryogenic distillation. In this study, carbon dioxide (CO₂) was purified and separated from biohydrogen to produce high purity hydrogen gas. CO₂ capture was studied using the activated carbon (AC) modified with the ionic liquid (IL) choline chloride as adsorbent. The physical and chemical properties of the adsorbents were characterized through XRD, FTIR, SEM-EDX, TGA, and BET analyses. The effects of IL loading, flow rate, temperature, and gas mixture were also investigated based on the absorption and desorption of CO₂. The CO₂ level in the biohydrogen composition was analyzed using a CO₂ gas analyzer. The SEM image indicated that the IL homogeneously covered the AC surface. High IL dispersion inlet enhanced the capability of the adsorbent to capture CO₂ gas. The thermal stability and presence of the functionalized group of ILs on AC were analyzed by TGA and FTIR techniques, respectively. CO₂ adsorption experiments were conducted using a 1 L adsorber unit. Hence, adsorption technologies exhibit potential for biohydrogen purification and mainly affected by adsorbent ability and operating parameters. This research presents an improved biohydrogen technique based on adsorption technology with novel adsorbents. Two different types of commercial CO₂ adsorbents were used in the experiment. Results show that the IL/AC exhibited properties suitable for CO₂ adsorption. The IL/AC sample presented a high CO₂ uptake of 30 wt. % IL when treated at 30 °C for 6 h under a flow rate of 1 L/min. The presence of IL increased the selectivity of CO₂ removal during the adsorption process. This IL/AC can be regenerated for several times without any significant loss in the performance.
1. Introduction

Hydrogen is a renewable energy resource that plays a key role in sustainable economic growth because of its carbon-neutral characteristics. Scholars have focused on improving hydrogen and biological production methods. Biohydrogen production is important because it utilizes renewable energy resources and is operated at ambient temperature and atmospheric pressure [1]. Biohydrogen is used in highly energy efficient fuel cells or internal combustion engines [2]. Biohydrogen production requires purified hydrogen. Therefore, hydrogen should be separated from different gaseous by-products, mainly carbon dioxide (CO$_2$) formed during fermentation.

Studies have investigated the separation of CO$_2$ from other gasses [3, 4]. Gas mixtures, such as CO$_2$ and methane (CH$_4$), are commonly separated through cryogenic distillation, chemical absorption, adsorption, and membrane technology [5-7]. Adsorption is an alternative method for amine scrubbing and membrane technologies, where adsorption capacity is mainly affected by large specific surface area, adsorbent type, and low regenerated heat [8, 9]. Adsorption techniques feature low operational requirements, ease of control, and high efficiency [5].

Adsorption using adsorbents, such as activated carbon (AC), has gained increasing research attention. AC can be used to separates gas mixtures, such as CO$_2$ and CH$_4$ [5, 10]. In a previous study, AC derived from carbonized coconut shell char was obtained through CO$_2$ activation at a carbonization temperature of 600 °C for 2 h [11]. Results showed that the effects of activation temperature and time and flow rates of CO$_2$ affect the surface area, total and micropore volumes, and yield of AC. The adsorption capacity or uptake of AC is highly influenced by preparation conditions.

Chemical modifications are mainly used to minimize the production cost, increase the removal efficiency of CO$_2$, and enhance the selectivity of the adsorbent. Chemicals containing nitrogen groups are suitable for modifying the CO$_2$ adsorbent because of the Lewis acid–base interaction [12]. In the study of Abbott et al. in 2001, a range of quaternary ammonium salts were heated with ZnCl$_2$ and the freezing points of the resulting liquids were measured; the lowest melting point (23 °C - 25 °C) was obtained when choline chloride was used as ammonium salt [13]. This IL was used as CO$_2$ adsorbent in physical or chemical adsorption. In another study, room temperature ionic liquid (RTIL) was used as substitute for amines because of its properties, such as low vapor pressure, wide liquid range, thermal resistance, and tenability [14].

In this study, an adsorbent material was prepared using AC impregnated with IL to increase the CO$_2$ adsorption efficiency. These materials were impregnated with IL for gas separation and purification; IL has become a well-known topic in recent separation and purification research because of its unique properties [15]. The developed adsorbent was characterized in terms of surface morphology and element composition (scanning electron microscopy, SEM-EDX), functional group content (Fourier transform infrared spectroscopy, FTIR), crystallinity (X-ray diffraction, XRD), stability and degradation (thermogravimetric analyzer, TGA), and surface properties (Brunauer–Emmett–Teller, BET). The effects of several parameters, such as flow rate, pressure, temperature, and adsorbent types, on CO$_2$ adsorption were also investigated. The CO$_2$ adsorption capacity was compared between the fabricated and fresh (commercial) AC.

2. Materials and methods

2.1. Production of ionic liquid (IL)

The chemicals used were of analytical reagent (AR) grade. Choline chloride (C$_5$H$_{11}$CINO) was purchased from Sigma Aldrich Company (USA). Zinc chloride (ZnCl$_2$) was obtained from Friedemann Schmidt Chemical Company (WA). The IL (choline chloride, 2ZnCl$_5^-$) was prepared using the process shown in Equation 1:

\[
\text{Choline chloride} + 2\text{ZnCl} \rightarrow \text{choline chloride. 2ZnCl}_5^- \quad (1)
\]

IL was prepared following the standard procedures [16]. IL solution with a ratio of weight percentage of 1:2 was produced by stirring the mixture at 60 °C for 3 h.
2.2. Impregnated AC
AC derived from coconut shells was provided by Afigen Sdn Bhd. (Malaysia). Fresh materials were cleaned and dried at 100 °C for 12 h. The materials were then sieved to obtain > 3 mm particle sizes for adsorption in the column unit. The adsorbents were modified using fresh AC and IL through impregnation methods; this method is commonly known as wet impregnation. Fresh AC and its surface properties were modified by using 30 wt. % IL to determine the effects of IL loading on the physical properties and adsorption performance of AC. The modified adsorbents were wetted in solid–liquid ratio (7:3) for 5 min, heated at 60 °C for 7 h, and dried.

2.3. Characterization of adsorbents
Fresh and impregnated AC was characterized through several techniques including SEM-EDX, FTIR, XRD, TGA, and BET surface area analyses.

2.3.1. Surface morphology and element composition. SEM type ZEISS Supra VP55 was used to examine the pore structure, topography, and composition of the samples. The SEM instrument was equipped with a secondary electron detector to visualize the crystal shape, surface morphology, and structure of particles or agglomerates. Product surface characteristics were also investigated through SEM with detectors for secondary and backscatter electrons. The backscatter detector is designed to analyze the differences in molecular density. The backscatter detector allows the visualization of differences in compositional density in a granule or tablet.

The size range of the sample is ~0.1 μm to few millimeters. The dried adsorbent was coated with a conducting material (e.g., Au) to reflect electrons (sputter coating) prior to the SEM analysis. SEM analysis was performed to determine the porosity and morphology of the adsorbent materials, EDX analysis was used to determine elements inside the adsorbent. This method can be used to observe the specimen in high vacuum or low vacuum, wet conditions, and at cryogenic or high temperatures.

2.3.2. Functional group and crystallinity. IL and the adsorbent AC (fresh and impregnated) were characterized by FTIR spectrometry and recorded with infrared spectrometer instrument-type Buck Scientific M-500 with KBr as pellet at a frequency range of 4000 cm\(^{-1}\) to 600 cm\(^{-1}\). The infrared spectra of the adsorbent samples were recorded using FTIR spectrometer (Perkin Elmer, UK). Attenuated total reflection (ATR) method was used for sample preparation. This FTIR analysis generated absorption or transmission spectra as a function of wave number, ranging from 4000 cm\(^{-1}\) to 500 cm\(^{-1}\). The sample was weighed (1−5 mg) at ambient temperature by using Spectrum One with SPECAC Golden Gate ATR (Perkin Elmer).

XRD analysis was performed for fingerprint characterization of the crystallinity and structures of the samples. This method was used to identify the shape and type of crystals produced when using AC. XRD was conducted to identify and characterize the crystal structure and size. This method analyzes and determines the distribution of crystal orientation in the thin film crystal sample.

Crystallinity analysis was performed in 1 g of the sample through XRD using Bruker AXS D8 Advance with X-ray radiation source of Cu Ka (40 kV, 40 mA) to record the 2ρ diffraction angle from 108 to 808 at a specific wavelength (λ = 0.154 nm). Based on Bragg’s law, the XRD powder (~10 mg) pattern showed the intensity of the diffracted beams as a function of 2-theta (2θ) at temperatures ranging from 5 °C to 80 °C and 25 °C. This analysis was conducted using XRD Bruker AXS D8 Advance with a working voltage of 40 kV and current at 40 Ma, respectively.

2.3.3. Stability/degradation and surface properties. A thermal gravimetric or thermogravimetric analyzer (TGA) was used to measure the amount of weight changes in a material, either as a function of increasing temperature or time, in an atmosphere of nitrogen (N\(_2\)) or air. A sample pan that is supported by a precision balance is used. The pan is placed in a furnace and heated or cooled during the experiment, and the sample mass is monitored. The balance obtains weight changes up to 1 μg. In this experiment, 5 – 10 mg of the sample is used for characterization by using N\(_2\) gas carrier. TA-
60WS thermal analysis system from Shimadzu Corporation is used for temperatures up to 1100 °C. The percentage of weight loss over temperature was recorded.

The surface properties were prepared and characterized using Micrometrics ASAP 2020 Version 4.02 in N₂ gas at 77 K. In this analysis, the principle of physical adsorption was used to obtain adsorption and desorption isotherms in the sample. The surface area is calculated by BET (Brunauer–Emmett–Teller) method. Based on BET results, the surface properties of AC adsorbent are shown. The BET surface area, micropore volume and pore size were also calculated from the isotherms. Prior to the analyses, the samples were degassed at 350 °C for 4 h for N₂ adsorption to remove all the volatile compounds and obtain the adsorption isotherm of each sample.

2.4. Adsorption–desorption study for fresh and impregnated AC
The CO₂ separation from gas mixtures of CO₂/H₂ gases was performed in a lab scale experimental single column unit of pressure swing adsorption (PSA) by feeding mixture gases of 50/50 mol % CO₂/H₂ to a single adsorption column unit as shown in figure 1. The column dimensions with a 4.8 cm internal diameter, ID, and 33 cm height were used. The column was connected to 3 mm (1/8 in) stainless steel tubing. The flow rate of feed gases was monitored by gas flow controllers (flow meter). The concentration of product stream was detected by CO₂ analyzer and read by HOBO ware software system in the computer. This procedure of experiment was repeated using the different adsorbent as propose with different parameters.

![Figure 1. Schematic of the adsorption–desorption system.](image)

In the adsorption–desorption study, fresh and impregnated AC was observed using the same experimental setup throughout the project. For the desorption process, air (3 L/min) was used as carrier gas and supplied through the adsorber unit to eliminate CO₂ from the adsorbent materials. Different flow rates (0.1 L/min, 0.5 L/min, and 1.0 L/min) were used to investigate the performance of fresh and impregnated AC as the adsorbent by passing gas streams containing the CO₂/H₂ mixture through the adsorption column unit. The capacity of adsorption CO₂ was observed using HOBO ware system in a computer connected to the CO₂ analyzer.

3. Results and discussion

3.1. Scanning electron microscopy (SEM-EDX)
The surface micrograph of adsorbents was studied using SEM. This instrument has a common tungsten electron gun with an operational volt ranging 0.1 to 3.0 kV. SEM results show the microscope photos of the compound at different magnifications. Thus, the surface morphology of the chemically modified adsorbents is shown in figure 2.
Figure 2. Surface morphologies of the adsorbents a) SEM photograph of fresh ACs: i) Mag: 3.00 K X (2 µ) and ii) Mag: 1.00 K X (10 µ) and b) impregnated ACs: i) Mag: 3.00 K X (2 µ) and ii) Mag: 1.00 K X (10 µ).

As shown in figure 2, the AC show bigger pores in materials that are expected to have a better CO\textsubscript{2} adsorption compared to other types of adsorbent materials. However, for impregnated AC, the surface layer has been fully covered by IL, making surfaces smoother. Based on the adsorption image, the pores in the sample IL was used to improve the adsorption performance and capacity over time. This result is supported by the adsorption performance unit that improved the AC surface and adsorption properties.

EDX was used to confirm the presence of elements on the surface of fresh and impregnated AC. Aside from the presence of pore in the mass percentage of IL loading, the existence of elements O and C was also observed in the physical morphology of AC. As shown in table 1, O and C elements are more abundant in impregnated AC compared with fresh AC because IL causes the increase in element percentage.

The physical adsorption is caused by van der Waals and electrostatic forces between adsorbate molecules and atoms at the adsorbent surface. A large specific surface area is important for a large adsorption capacity; however, a large internal surface area in a limited volume produces a large number of small-sized pores between adsorption surfaces [17].

The increase in physical affinity causes the increase in adsorption capacity. Numerous works about the separation of CO\textsubscript{2} show a high adsorption capacity at atmospheric pressure. Moreover, the advantages of CO\textsubscript{2} separation, such as hydrophobicity, affordability, and low energy requirement, are also discussed.

| Table 1. Elements of fresh and impregnated AC. |
|------------------|-----------------|-----------|-------|
| Materials        | Elements | Wt (%) | Σ     |
| Fresh AC         | -O       | 69.0    | 2.4   |
|                   | -C       | 25.5    | 2.4   |
|                   | -K       | 5.5     | 0.9   |
| Impregnated AC   | -O       | 72.7    | 0.2   |
|                   | -C       | 27.3    | 0.2   |
3.2. Fourier transform infrared (FTIR) and spectroscopy and x-ray diffraction (XRD)

In Fourier transform infrared (FTIR) spectroscopy, infrared radiation is passed through a sample. The resulting spectrum represents the molecular absorption, creating a molecular fingerprint of the sample. FTIR was used to provide information about the identification of API and impurities and quantification of components in mixtures, e.g., the determination of amorphous material content in a crystalline sample (percent crystallinity). This method was also used to examine changes in the solid state because of interactions with excipients, i.e., compatibility studies in order to choose the most stable excipients for the final product. The region from 1500 to 400 wave numbers is referred to as the fingerprint region. Absorption bands in this region are generally caused by the intramolecular phenomenon and highly specific to each material.

Up to 0.5 mg of adsorbent sample was used for all tests. The FTIR spectra of the IL and AC are shown in figure 3. IL (figure 3 a) has the most complicated and clear spectrum. A broad adsorption peak was also observed at 3300 cm\(^{-1}\) to 3700 cm\(^{-1}\) attributed to O–H and N–H (IL case) stretch functional group; this result indicates the presence of bonded hydroxide in all the sample measurements. During infrared radiation, absorbed IR radiation excites molecules into a higher vibrational state. The wavelength of light absorbed by a particular molecule is a function of the energy difference between the at-rest and excited vibrational states. The wavelengths that are absorbed by the sample are characterized by their molecular structure.

Figure 3. Functional group of IL and AC: a) Comparison between IL and IL injected with CO\(_2\) gas and b) comparison of fresh and impregnated AC.

The O–H stretching absorption of the hydroxyl group is sensitive to hydrogen bonding. In the gas phase and in a diluted CCl\(_4\) solution (0.01 M), small- to moderate-sized alcohols exhibit a sharp absorption at the 3620 cm\(^{-1}\) to 3670 cm\(^{-1}\) region. In a more concentrated solution or in a pure liquid, a hydrogen bond between the hydroxyl groups occurs, and this bond lowers and broadens the stretching frequencies of the participating O–H bonds.
In the IL region (figure 3 a), N–H bend (primary amines only) comes from 1622 cm$^{-1}$. The bands at 1474 cm$^{-1}$ show a C–H bend or scissoring stretch. The bands at 1500 cm$^{-1}$ to 1600 cm$^{-1}$ of impregnated AC corresponded to the C=O functional group. The IL sample also shows two important adsorption peaks from 1004 cm$^{-1}$ to 1100 cm$^{-1}$ that refers to the C–O stretching functional group. However, fresh and impregnated AC shows C–N stretching (aliphatic amines) from 1250 cm$^{-1}$ to 1020 cm$^{-1}$. The C–N stretching vibration of aliphatic amines is observed as medium or weak bands in the region 1250 cm$^{-1}$ to 1020 cm$^{-1}$. In aromatic amines, the band is usually strong and at the region from 1335 cm$^{-1}$ to 1250 cm$^{-1}$.

For impregnated AC (figure 3 b), during the carbonization and activation process, the adsorption peaks of the functional groups disappeared. This phenomenon is caused by the raw material spectrum that vaporized as volatile materials when the sample was heated [18]. The activation process was successfully proven. To confirm this phenomenon, the fresh AC spectrum was used as the reference sample. As a result, the trend of the impregnated AC was almost similar with the fresh AC. Thus, the prepared material of impregnated AC was successfully converted without any carbon loss.

XRD analysis is based on the constructive interference of monochromatic X-rays and a crystalline sample. X-rays are generated by a cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample. The interaction of the incident rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg’s Law ($nλ = 2d \sin θ$). This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample.

Figure 4 shows the comparison between fresh and impregnated AC. The impregnated AC was prepared by using ZnCl$_2$, which is affordable, and ionic liquid-type choline chloride (ammonia) with a ratio of 30% (w/w). As shown in figure 4, fresh and impregnated AC was in amorphous phase. AC is a non-crystalline material and a type of amorphous carbon that is composed of graphite carbon microcrystals and non-graphite carbon [19].

Results also showed that the intensity peaks increased with increasing the activation temperature but decreases with increasing the impregnation ratio of the materials. The low intensity peaks range from 20° to 40°, which correspond to the graphite lattice. The fresh and impregnated AC samples were observed to determine the characteristic x-ray diffraction pattern by providing a unique “fingerprint” of the crystals in the sample. When properly interpreted, by comparing with the standard reference patterns and measurements, this fingerprint allows the identification of crystalline. Thus, further FTIR analysis is needed for quantification of components in mixtures, e.g., the determination of the amorphous material in a crystalline API, as the absorbance is proportional to the content of the given component. Moreover, the percent crystallinity of a sample is determined using FTIR.

3.3. Thermogravimetric analysis (TGA) and physic sorption properties (BET analysis)

TGA is used to show if a compound contains components such as solvents or other loosely bound molecules that might evaporate upon heating. The presence of solvents or hydrates is usually not
desirable because this might cause destabilization of the compound. Furthermore, this method is used to distinguish between solvates and “pure” polymorphic forms. The weight changes of an adsorbent, as a function of time, in N₂ atmosphere was measured.

**Table 2.** Fresh and impregnated AC decomposition before and after adsorption–desorption

| Material in adsorption unit | Percentage reduction (%) |
|----------------------------|--------------------------|
|                           | 1st                      | 2nd                     | 3rd                     |
| Fresh AC Before           | 0.77                     | 2.73                    | 20.27                   |
| After                      | 1.01                     | 4.38                    | 34.32                   |
| Impregnated AC Before     | 1.19                     | 6.27                    | 43.61                   |
| After                      | 1.98                     | 8.39                    | 40.34                   |

**Figure 5.** Comparison of weight loss study by TGA method.

The IL (quaternary ammonium salts) starts to decompose at 350 °C, and the high thermal stability of ILs is shown in figure 5. ZnCl₂ starts to vaporize and form ZnO at 480 °C. The decomposition of fresh and impregnation AC before and after adsorption is shown in table 2. As shown in figure 5, weight loss for impregnated AC is significant during adsorption compared with before adsorption. This phenomenon is caused by IL surrounding the surface of AC that have higher CO₂ levels compared with fresh AC. These IL show a greater tendency to dissolve CO₂ than other gases, like methane, ethane, oxygen, nitrogen, argon, and carbon monoxide. Therefore, the selective adsorption of CO₂ from flue gas is possible [20, 21].

Biomass product, such as coconut shells, is composed of hemicellulose, cellulose, and lignin. The pyrolysis of coconut shells is attributed to the pyrolysis of these polymers. Nunn et al. (1985) reported that the decomposition of cellulose occurs at 200 °C to 400 °C, whereas lignin decomposes at temperature ranges of 150 °C to 750 °C [22]. Furthermore, the rate of decomposition was low. At temperatures higher than 400 °C, the aromatization process is the most important reaction at low mass and loss rate [23].

**Table 3.** Comparison of fresh and impregnated AC through BET analysis (N₂).

| Material   | BET surface area (m²/g) | Micro-pore area (m²/g) | Pore volume (cm³/g) | Micropore volume (cm³/g) | Pore size (Å) |
|------------|-------------------------|------------------------|---------------------|--------------------------|---------------|
| Fresh AC   | 863.04                  | 689.79                 | 0.41                | 0.32                     | 19.14         |
| Impregnated AC | 117.49                 | 93.23                  | 0.06                | 0.04                     | 19.14         |
BET results showed that the surface area decreased and the pore characteristics changed after chemical modifications. When the pressure is lower, the reaction between AC and CO\textsubscript{2} was slow. Thus, the BET surface area and total and micropore volumes were relatively small. With increasing pressure, the BET surface area and total and micropore volumes were increased because of the higher reaction rate between carbon and CO\textsubscript{2} and higher releasing rate of volatile matter.

![Figure 6. Schematic of the mechanism of pore filling with IL loading (AC: below and IL loading: upper): a) 0 wt. %, b) 10 wt. %, c) 20 wt. %, and d) 30 wt. %.
](image)

Table 3 shows the pore volume and micropore area that instantly decreased because of the IL that covered the surface of the adsorbent materials. IL has a greater tendency to cover the entire surface without interfering the area of pore sizes. Based on BET results, as shown in figure 6, 30 wt. % covered mostly the pore, and the capability of impregnated AC to adsorb CO\textsubscript{2} could be affected.

![Figure 7. Results of BET analysis for adsorption: isotherm tabular report quantity adsorbed (cm\textsuperscript{3}/g STP) with absolute pressure (mmHg) for N\textsubscript{2} gas.
](image)

As shown in figure 7, 30 wt. % of IL covered all the pores of AC, resulting to a lower adsorption compared. Based on BET results about the physical characteristics of the surface adsorbents, IL covered the surface micropore when the gas carrier flow thorough the measurements. As a result, fresh AC showed higher adsorption results compared with impregnated AC that detect the surface layer of the adsorbent.
4. Adsorption and desorption of CO₂

4.1. Adsorption of fresh and impregnated AC

Active catalysts for this reaction are typically prepared using an impregnation method, and the identification of improvements in methodology preparation produces more active catalysts that retain stability is important. Li et al. (2013) studied the mixture of IL and water that was used to absorb CO₂, and results showed that the aqueous solution of IL was also good at absorbing CO₂ [24].

However, to reduce the cost and viscosity with high capturing effects, a nonionic surfactant was added to the aqueous solution of this IL. The gas mixture of CO₂/H₂ was used throughout the study. The adsorption of CO₂ was conducted in the adsorber single column unit. AC were impregnated by IL at a loading rate of 0, and 30 wt. % of these AC were studied for the adsorption and separation of CO₂ from the flue gas. The adsorption capacity of CO₂ with several flow rates is shown in table 4.

The capacity of loading gas with a flow rate of 0.1 L/min shows stable increments and acts as optimum parameters for different flow rates of gas concentrations. The IL loading rate of the fresh AC was 0 %, whereas the loading rate of impregnated AC was 30 %. Temperature used was 30 °C, and Co is indicated as the initial concentration of CO₂ before the adsorbent (mol/mol), whereas C is shown as the concentration of CO₂ after the adsorbent (mol/mol). figure 8 show that the AC supported by IL increases CO₂ adsorption over time. The flow rate of 1.0 L/min adsorbs faster. The adsorption capacity of impregnated AC in the adsorber unit clearly showed better results that adsorbs nearly 90 % of CO₂ in a for 6 h experiment. However, the cycling process should be performed to compare the performance of the adsorbents between the impregnated AC in the future.

| Percentage of CO₂ gas loading (%) | Adsorption of fresh AC with different flow rates (L/min) | Adsorption of impregnated AC with different flow rates (L/min) |
|----------------------------------|--------------------------------------------------------|-------------------------------------------------------------|
| 10                               | 5.47, 5.19, 5.50                                      | 5.46, 5.39, 5.59                                           |
| 30                               | 30.96, 30.41, 32.38                                   | 30.42, 32.37, 34.50                                       |

**Figure 8.** Comparison of CO₂ adsorption behavior of fresh AC on different flow rates of gas: i) 1.0 L/min impregnated AC, ii) 1.0 L/min fresh AC, iii) 0.5 L/min impregnated AC, iv) 0.5 L/min fresh AC, v) 0.1 L/min impregnated AC, and vi) 0.1 L/min fresh AC.
4.2. Desorption of fresh and impregnated AC

Different desorption mechanisms are compared using air with a flow rate of 3.0 L/min as the carrier that has been injected inside the chamber. As shown in figure 9, the impregnated AC was desorbed faster. Desorption of the impregnated AC at a flow rate of 1.0 L/min occurred within a minute. Thus, IL affects the CO$_2$ adsorption and desorption performance. Moreover, the amino functional IL adsorbs higher amount of CO$_2$ than other conventional methods [25](Yang & Wang, 2015).

![Figure 9. CO$_2$ desorption behavior of fresh AC at different flow rates of gas: i) 1.0 L/min impregnated AC, ii) 1.0 L/min fresh AC, iii) 0.5 L/min impregnated AC, iv) 0.5 L/min fresh AC, v) 0.1 L/min impregnated AC, and vi) 0.1 L/min fresh AC.](image)

Impregnated AC (covered with IL) show improved adsorption and desorption of CO$_2$. For a short time period, impregnated AC show a great tendency to capture CO$_2$; this IL is best described with the cycling performance and affects different loading rates to elucidate the adsorbent performance in future study.

5. Conclusion

This study observed the effects of incorporating IL in AC on CO$_2$ adsorption. All AC materials (fresh and impregnated) that were characterized by using different analysis methods, such as SEM (morphology of surface), EDX (element composition), TGA (weight loss percentage), physisorption analyzer BET model (surface properties), FTIR (new chemical bond/functional group), and XRD (crystallinity of the adsorbent), were investigated and characterized. IL is for a promising solvent for separations and purifications, specializing in carbon capture process. IL exhibit improved CO$_2$ adsorption property by using an adsorber in real-time unit. Moreover, the porosity and surface area and IL activation play an important role in the adsorption of flue gas. As a result of adsorption in the actual adsorber unit, the impregnated AC reacts faster and adsorbs larger amounts of CO$_2$ than those of fresh AC in a short period of time at a high flow rate. To elucidate the adsorption process, further studies should focus on the performance of the adsorber unit. The cycling of adsorbent materials needs to be observed to obtain the optimal performance of the impregnated AC with focus on thermodynamic parameters.

References

[1] Debabrata D, Khanna N and Dasgupta C N 2014 American J. of Biomass and Bioenergy 3 408.
[2] White C M, Steeper R R and Lutz A E 2006 Int. J. Hydrogen Energy 31 1292.
[3] Choi S, Drese J H and John C W 2009 Chem. Sus. Chem. 2 796.
[4] Li J R, Sculley J and Zhou H C 2011 Chem. Rev. 112 869.
[5] McEwen J, Hayman J D and Yazaydin A O 2013 Chem. Phys. 412 72.
[6] Thu K, Kim Y D, Ismil A B, Saha B B and Ng K C 2014 Appl. Therm. Eng. 72 200.
[7] Su F S, Lu C Y, Chung A J and Liao C H 2014 *Appl. Energy* **113** 706.
[8] Chen S J, Fu Y, Huang Y X, Tao Z C and Zhu M 2016 *App. Energy* **179** 329.
[9] Yan T, Li T X, Wang R Z and Jia R 2015 *Appl. Therm. Eng.* **77** 20.
[10] Yang H, Gong M C and Chen Y Q 2011 *J. Nat. Gas. Chem.* **20** 460.
[11] Shenghui G, Jinhui P, Wei L, Kunbin Y, Libo Z, Shimin Z and Hongying X 2009 *Applied Surface Science* **255** 8443.
[12] Yin C Y, Aroua M K and Daud W M A W 2007 *Separation Purification Technology* **52** 403.
[13] Abbott A P, Glen C, David L D, Helen L M, Raymond K R and Vasuki T 2001 *Chem. Commun* 2010–11.
[14] Wang Q, Xia W and Guo W 2013 *Chemistry- An Asian Journal* **8** 1879.
[15] James J and Davis H 2002 *American Chemical Society* 247.
[16] Duan Z, Gu Y and Deng Y 2006 *Catal. Commun.* **7** 651.
[17] Motoyuki S 1990 *Adsorption Engineering* 25 Kodansha LTD Tokyo Japan.
[18] Yacob A 2008 *The Malaysian Journal of Analytical Sciences* **12** 264.
[19] Jiang J 2010 *Chemical Industry Press*.
[20] Jacquemin J, Gomes M F C, Husson P and Majer V 2006 *J. Chem. Thermodynamics* **38** 490.
[21] Anderson J L, Dixon J K and Brennecke J F 2007 *Accounts of Chemical Research* **40** 1208.
[22] Nunn R T, Howard J B, Longwell P J and Peters A W 1985 *Lignin Ind. Eng. Chem. Proc. Des. Dev.* **24** 84.
[23] Fisher T, Hajaligol M, Waymack B and Kellog D 2002 *J. Anal. Appl. Pyrolysis* **62** 331.
[24] Li T, Kong Q and Chen C 2013 *J. Chem. Ind. Eng* 600.
[25] Yang N and Wang R 2015 *J. Serb. Chem. Soc.* **80** 265.