

# CO<sub>2</sub> Capture Using Silica and Molecular Sieve Impregnated with [hmim][Tf<sub>2</sub>N]

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**Abstract**—Solid supported ionic liquid can provide high surface area for better CO<sub>2</sub> adsorption as well as reduces the amount of ionic liquid used for CO<sub>2</sub> absorption. In the present work, SiO<sub>2</sub> and Molecular Sieve were impregnated with 10wt% of [hmim][Tf<sub>2</sub>N], and tested for CO<sub>2</sub> adsorption at 10-40 bar. From the screening process, 10%[hmim][Tf<sub>2</sub>N]-Si was observed to give higher CO<sub>2</sub> adsorption compared to [hmim][Tf<sub>2</sub>N]-MolSieve, and 10%[hmim][Tf<sub>2</sub>N]-Si was selected for further investigation. The experiment was further investigated by measuring CO<sub>2</sub> adsorption at 25, 40, and 60 °C. using 10%[hmim][Tf<sub>2</sub>N]-Si. The highest CO<sub>2</sub> captured showed by 10%[hmim][Tf<sub>2</sub>N]-Si was at 298 K. The CO<sub>2</sub> adsorption was also modeled using Langmuir and Freundlich isotherms, and the adsorption was best fitted using Freundlich isotherm at 25 °C with 0.9813 R<sup>2</sup> value.

**Index Terms**—[hmim][Tf<sub>2</sub>N], solid support ionic liquid, CO<sub>2</sub> adsorption, adsorption isotherm.

## I. INTRODUCTION

Fossil fuels are hydrocarbons formed from dead plants and animals, and primarily consist of fuel, coal and natural gas. Fossil fuels are an important non-renewable energy sources such as to generate electricity and for transportation use. Although fossil fuels are known as the largest energy sources, but they are also the largest contributor of CO<sub>2</sub> emissions. The burning of fossil fuels produces energy together with CO<sub>2</sub>, and this CO<sub>2</sub> is released to the environment which then causes global warming issues. Organic amine especially monoethanolamine (MEA) is the most used solvent as it provides 70-90% of CO<sub>2</sub> removal [1] and produces a relatively pure CO<sub>2</sub> stream.

However, CO<sub>2</sub> capture technologies should not be limited only to amine-based technologies. New technologies and research are always desired to find potential solvents in order to produce an efficient CO<sub>2</sub> separation process. Currently, there is trend developing on using ionic liquids as an alternative solvent to capture CO<sub>2</sub>. Ionic liquids are organic salts with melting points below ambient or reaction temperature, composed of organic cation and organic or inorganic anion [2]. The advantages of ionic liquids are chemical and thermal stability, negligible vapor pressure, and ionic liquids can be tuned and designed for a particular application, by choice of cations and anions, and also

substitution of functional groups [3].

Based on the published research, ionic liquids such as imidazolium and pyridinium based have shown promising results in CO<sub>2</sub> capture, and anions are proved to play primary role in CO<sub>2</sub> absorption using ionic liquids [3]-[5]. However, the issue of ionic liquids is their high viscosity which can limit the mass transfer of solutes in the ionic liquid [6]. Besides, non-supported ionic liquid requires a large amount of ionic liquid used for either reaction or separation and it is costly since ionic liquid are an expensive solvent [6]. Therefore, the alternative of this problem is to use supported ionic liquid on solid material. The porous solid support can provide high surface area that is necessary to accommodate intimate contact between CO<sub>2</sub> and ionic liquid, and also to increase the mass transfer of solute (gas) into solvent (ionic liquid).

As to our knowledge, solid supported ionic liquid has been studied in desulfurization [7] and also as a catalyst, for example in the Friedel-Crafts reactions, however, for gas separation, ionic liquids are usually supported onto membrane to increase the permeability of gas through the membrane [8]. Therefore, in this research, ionic liquid was supported onto silica dioxide as to provide more available data for porous solid supported ionic liquids in the gas separation application.

## II. EXPERIMENTAL SECTION

### A. Materials

Ionic liquid used in the present work was 1-hexyl-3-methylimidazolium bis(trifluoromethylsulfonyl) imide ([hmim][Tf<sub>2</sub>N]). [hmim][Tf<sub>2</sub>N] was purchased from Merck and used without further purification. SiO<sub>2</sub> (0.065-0.200 mm) was purchased from Acros Organic, molecular sieve 13X (8-12 mesh) was purchased from Sigma Aldrich, and CO<sub>2</sub> with 98% purity was purchased from Mox-Linde Gases Sdn. Bhd.

### B. Preparation of Solid Supported [hmim][Tf<sub>2</sub>N]

[hmim][Tf<sub>2</sub>N] was dried overnight in a vacuum oven at 70 °C, and SiO<sub>2</sub> was calcined at 500 °C for 3 hours. In a round bottom flask, appropriate amount of [hmim][Tf<sub>2</sub>N] was mixed with methanol and stirred for 5 minutes, and then, an appropriate amount of SiO<sub>2</sub> was added. Using the reflux condenser, the mixture was stirred for 24 hours at room temperature. After that, the methanol was removed using rotary evaporator resulting in physical immobilization of [hmim][Tf<sub>2</sub>N] into the SiO<sub>2</sub>. The solid supported ionic liquid was denoted as 10%[hmim][Tf<sub>2</sub>N]-Si. Then, the prepared 10%[hmim][Tf<sub>2</sub>N]-Si was dried in vacuum oven for 2 days at 80 °C to remove remaining solvent and moisture. The

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composition of ionic liquid impregnated into SiO<sub>2</sub> was 10% (by weight). Molecular sieve supported with [hmim][Tf<sub>2</sub>N] was also prepared using the same procedure and denoted as 10%[hmim][Tf<sub>2</sub>N]-MolSieve. The prepared samples were then characterized using Thermogravimetric Analyzer (TGA) and Surface Area and Porous Analyzer.

### C. CO<sub>2</sub> Adsorption Measurement

CO<sub>2</sub> adsorption for pure [hmim][Tf<sub>2</sub>N], pure SiO<sub>2</sub>, pure molecular sieve, and solid supported [hmim][Tf<sub>2</sub>N] was measured using a gravimetric method. The amount of CO<sub>2</sub> adsorbed/absorbed is determined by the pressure difference between the pressure when the gas is first introduced and after the equilibrium is reached. The amount of CO<sub>2</sub> captured in the solubility experiment can be determined using equation below [9]:

$$n_{CO_2} = \frac{P_{ini}V_{tot}}{Z_1(P_{ini}T_{ini})RT_{ini}} - \frac{P_{eq}(V_{tot}-V_s)}{Z_2(P_{eq}T_{eq})RT_{eq}} \quad (1)$$

where

$p_{ini}$ : Initial pressure introduced to the system

$p_{eq}$ : Final pressure after the system reaches equilibrium

$V_{tot}$ : Total volume of the adsorption/absorption cell

$V_{sample}$ : Volume of sample used

$T_{ini}$ : Initial temperature at initial pressure

$T_{eq}$ : Equilibrium temperature at equilibrium pressure

$Z_{ini}(p_{ini}, T_{ini})$ : Compressibility factor as a function of  $p_{ini}$  and  $T_{ini}$

$Z_{eq}(p_{eq}, T_{eq})$ : Compressibility factor as a function of  $p_{eq}$  and  $T_{eq}$

$R$ : Gas constant

### D. Adsorption Isotherms

Generally, adsorption isotherm describes the interaction between adsorbate and adsorbent, and thus critical for the optimization of adsorption mechanism pathways, expression of the surface properties and capacities of adsorbents, and effective design of the adsorption systems [10], [11]. The equilibrium isotherm is also used for the theoretical evaluation and interpretation of thermodynamic parameters such as heats of adsorption (Allen *et al.*, 2004). In the present work, adsorption isotherm was carried out using Langmuir and Freundlich isotherms, and the best fitted model to describe the adsorption process was determined by the correlation coefficients ( $R^2$ ) value.

Langmuir isotherm is originally used to describe gas adsorption onto activated carbon. This isotherm is the simplest model of physical adsorption which assumes monolayer adsorption where once a molecule occupies a site, no further adsorption can take place [12], no interaction between adsorbed adsorbate even on the adjacent site [13], and the enthalpy of adsorption is the same for each molecule (all sites possess equal affinity for the adsorbate) [14]. This model can be used to estimate the maximum adsorption capacity of adsorbate on the adsorbent surface. This model is expressed as:

$$q_e = \frac{q_m K_L P_e}{1 + K_L P_e} \quad (2)$$

where

$q_e$ : Amount of CO<sub>2</sub> adsorbed per unit weight of adsorbent at equilibrium (mmol/g)

$q_m$ : Maximum adsorption capacity (mmol/g)

$P_e$ : Equilibrium pressure (bar)

$K_L$ : Langmuir constant related to the free energy of adsorption (g/mmol).

In Freundlich isotherm, the adsorption is not restricted to the formation of monolayer. This model can be applied to multilayer adsorption, with non-uniform distribution of adsorption heat and affinities over the heterogeneous surface [15]. Freundlich isotherm is defined as:

$$q_e = K_F \cdot P_e^{1/n} \quad (3)$$

where

$K_F$  (mmol<sup>1-(1/n)</sup> · g<sup>1/n</sup> · g<sup>-1</sup>) and  $n$  (dimensionless): Freundlich constant related to the adsorption capacity and intensity

$q_e$ : Amount of CO<sub>2</sub> adsorbed per unit weight of adsorbent at equilibrium (mmol/g)

$P_e$ : Equilibrium pressure (bar)

The adsorption is classified as favorable adsorption if the Freundlich constant,  $n$ , has the range of 1 to 10 [16].

## III. RESULTS AND DISCUSSION

This part covers the screening process of CO<sub>2</sub> adsorption using solid supported ionic liquid at different pressure and temperature, characterization data using Surface Area and Porous Analyzer, and adsorption isotherm study using Langmuir and Freundlich isotherm models.

### A. Textural Properties Data of SiO<sub>2</sub> and Molecular Sieve before and after the Impregnation

Surface Area and Porous Analyzer was used to determine the textural properties data (surface area, pore size, and pore volume) of SiO<sub>2</sub> and molecular sieve before and after the impregnation with [hmim][Tf<sub>2</sub>N]. Table I shows the textural properties data for molecular sieve and 10%[hmim][Tf<sub>2</sub>N]-MolSieve, and Table II shows the textural properties data for SiO<sub>2</sub> and 10%[hmim][Tf<sub>2</sub>N]-Si. From Table I, surface area, pore size, and pore volume for molecular sieve were decreased after impregnated with [hmim][Tf<sub>2</sub>N]. This shows that the surface and pores were covered and filled with [hmim][Tf<sub>2</sub>N].

TABLE I: TEXTURAL PROPERTIES DATA FOR MOLECULAR SIEVE AND 10%[HMIM][Tf<sub>2</sub>N]-MOLSIEVE

Sample	Pure MolSieve	10%[hmim][Tf <sub>2</sub> N]-MolSieve
Surface area (m <sup>2</sup> /g)	690.08	580.40
Pore size (Å)	10.01	9.34
Pore volume (cm <sup>3</sup> /g)	0.1857	0.1435

TABLE II: TEXTURAL PROPERTIES DATA FOR SiO<sub>2</sub> AND 10%[HMIM][Tf<sub>2</sub>N]-SI

Sample	Pure SiO <sub>2</sub>	10%[hmim][Tf <sub>2</sub> N]-Si
Surface area (m <sup>2</sup> /g)	524.05	398.69
Pore size (Å)	48.95	51.53
Pore volume (cm <sup>3</sup> /g)	0.6724	0.5338

In Table II, after the impregnation with ionic liquid, the surface area and pore volume of SiO<sub>2</sub> were also decreased, however, the pore size of SiO<sub>2</sub> was increased from 48.95 Å to

51.53 Å. This is due to the filling of smaller micropores with ionic liquid during the impregnated process [17]. The same trend was also observed by Gray and co-workers (2008) where in their work, the pore size of polymer beads was increased after the impregnation with tertiary amine [17].

### B. Percentage of [hmim][Tf<sub>2</sub>N] Impregnated inside SiO<sub>2</sub> and Molecular Sieve

The amount of [hmim][Tf<sub>2</sub>N] impregnated in solid support was determined from the weight loss (%) data obtained from the thermal analysis using Thermogravimetric Analyzer. Table III shows the percentage of [hmim][Tf<sub>2</sub>N] that was successfully impregnated inside the solid support. The composition of [hmim][Tf<sub>2</sub>N] introduced into the solid support was 10% (by weight). For 10% [hmim][Tf<sub>2</sub>N]-Si, the percentage of [hmim][Tf<sub>2</sub>N] that was successfully impregnated was 11.75%, and the amount was 1.75% excess from the original amount of ionic liquid introduced. This excess in the weight loss might be contributed from the adsorbed moisture within the SiO<sub>2</sub> pores [17].

For 10% [hmim][Tf<sub>2</sub>N]-MolSieve, the amount of ionic liquid that was successfully impregnated was only 5.86% out of 10% ionic liquid added. This might be due to the smaller pore size of molecular sieve which blocked the ionic liquid from 'entering' the pores. Besides, the pore volume of molecular sieve was also smaller than SiO<sub>2</sub>. This smaller pore volume might also be one of the reason for less uptake of ionic liquid (5.86%) than the amount added (10%) into the molecular sieve, and resulting in 4.14% excess amount of ionic liquid. From this result, it might also be explained that the solid support with micropore pore size is not suitable to be used for [hmim][Tf<sub>2</sub>N].

TABLE III: PERCENTAGE OF IONIC LIQUID SUCCESSFULLY IMPREGNATED INSIDE THE SOLID SUPPORT

Sample	Weight loss (%) <sup>a</sup>
10% [hmim][Tf <sub>2</sub> N]-Si	11.75
10% [hmim][Tf <sub>2</sub> N]-MolSieve	5.86

<sup>a</sup>Weight loss (%) represents the percentage of [hmim][Tf<sub>2</sub>N] inside the solid support

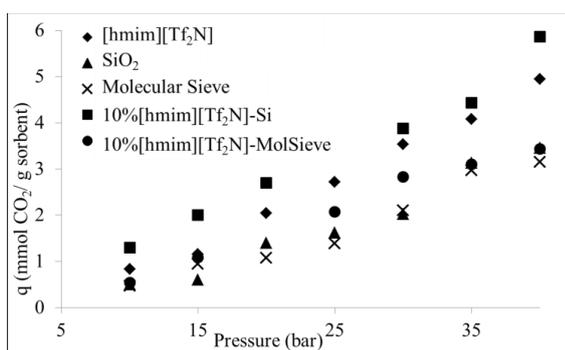


Fig. 1. Gas adsorption capacity for pure SiO<sub>2</sub>, pure molecular sieve, pure [hmim][Tf<sub>2</sub>N], 10% [hmim][Tf<sub>2</sub>N]-Si, and 10% [hmim][Tf<sub>2</sub>N]-MolSieve (at 25 °C).

### C. Screening Process of CO<sub>2</sub> Captured Using SiO<sub>2</sub> and Molecular Sieve as Solid Supports

In the screening process, the CO<sub>2</sub> adsorption measurement was performed using SiO<sub>2</sub>, molecular sieve, [hmim][Tf<sub>2</sub>N], [hmim][Tf<sub>2</sub>N]-Si, and [hmim][Tf<sub>2</sub>N]-MolSieve. The purpose of this screening process was to select which solid support can provide higher adsorption after the impregnation with

ionic liquid. For this part, the CO<sub>2</sub> adsorption was measured at 10–40 bar at 25 °C. Fig. 1 below shows the graph of CO<sub>2</sub> adsorption capacity,  $q$  (mmol CO<sub>2</sub>/g sorbent) as a function of pressure at 25 °C.

From Fig. 1, it shows that both pure SiO<sub>2</sub> and molecular sieve gave lower adsorption than pure [hmim][Tf<sub>2</sub>N]. After the impregnation with ionic liquid, the adsorption capacity for 10% [hmim][Tf<sub>2</sub>N]-MolSieve was still lower than pure [hmim][Tf<sub>2</sub>N], however for 10% [hmim][Tf<sub>2</sub>N]-Si, the adsorption capacity was higher than pure [hmim][Tf<sub>2</sub>N]. This lower adsorption capacity by 10% [hmim][Tf<sub>2</sub>N]-MolSieve might be explained using two reasons; first is due to the blockage of pores by ionic liquid which limits the amount of CO<sub>2</sub> adsorbed, and the second is due to less percentage ionic liquid impregnated inside the molecular sieve pores compared to SiO<sub>2</sub>. However, the second reason did not affect the screening process decision since the selection of solid support for further investigation was made based on solid support that can give higher CO<sub>2</sub> adsorption capacity than pure ionic liquid ([hmim][Tf<sub>2</sub>N]).

In addition, SiO<sub>2</sub> was also able to adsorb small amount of CO<sub>2</sub>, therefore, it can also be explained that the amount of CO<sub>2</sub> adsorbed by 10% [hmim][Tf<sub>2</sub>N]-Si might be contributed from SiO<sub>2</sub> itself. Thus, it can be explained that both adsorption and absorption are involved in the CO<sub>2</sub> capture process.

Fig. 1 also shows when the pressure was increased from 10 to 40 bar, the adsorption capacity was also increased for all samples. For example, at 25 °C, the adsorption capacity for 10% [hmim][Tf<sub>2</sub>N]-Si increased from 1.303 mmol/g to 5.860 mmol/g when the pressure was increased from 10 to 40 bar. When the pressure increases, more gas molecules are 'forced' into the sorbent resulting in higher adsorption capacity.

From the screening process, SiO<sub>2</sub> was selected for further investigation to study the CO<sub>2</sub> adsorption at different temperatures.

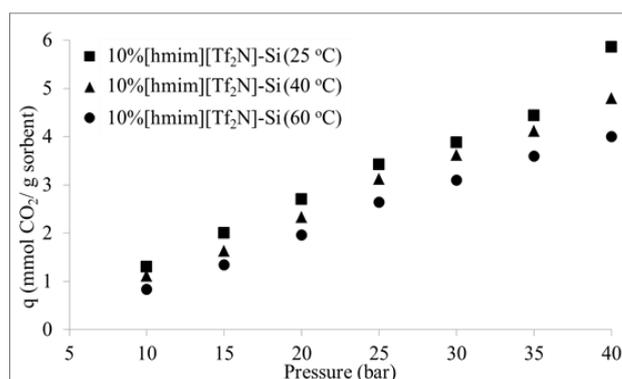


Fig 2. Graph of adsorption capacity,  $q$ , as a function of pressure.

### D. CO<sub>2</sub> Adsorption Using 10% [hmim][Tf<sub>2</sub>N]-Si at Different Temperature

The CO<sub>2</sub> adsorption measurement for 10% [hmim][Tf<sub>2</sub>N]-Si was further investigated using three different temperatures. Fig. 2 shows the adsorption capacity ( $q$ ) as a function of pressure at 25, 40 and 60 °C. From Fig. 2, it can be seen that the adsorption capacity decreased as the temperature increased from 25 to 60 °C. For example, at 40 bar, the adsorption capacity for [hmim][Tf<sub>2</sub>N]-Si was 5.860 mmol/g at 25 °C, while at 60 °C, the adsorption capacity was 3.998 mmol/g. Increase in temperature leads to increase in

the kinetic energy of the gas molecules, thus resulting in less adsorbed molecules on the surface of the sorbents. The adsorption capacity was the highest for [hmim][Tf<sub>2</sub>N]-Si at 25 °C.

### E. Adsorption Isotherm

There two adsorption isotherms used in this study were Langmuir and Freundlich isotherms. The relative correlation coefficient,  $R^2$  was calculated to determine whether the experimental data fit into the isotherm models. These isotherm models were applied to solid supported [hmim][Tf<sub>2</sub>N] at 25, 40, and 60 °C. Fig. 3 and Table IV show the Langmuir isotherm graph and Langmuir parameters.

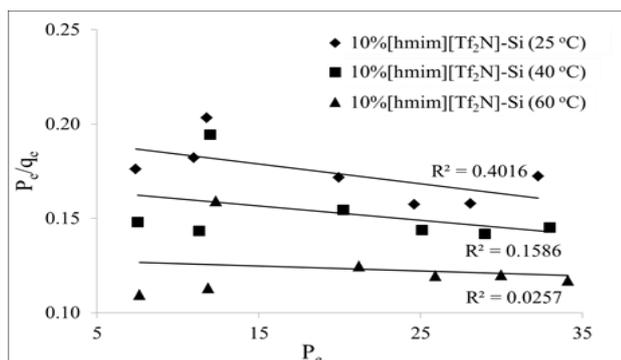


Fig. 3. Langmuir adsorption isotherm plot.

TABLE IV: PARAMETERS FOR LANGMUIR ISOTHERM

Temperature (°C)	Langmuir Isotherm		
	$R^2$	$q_m$	$K_L$
25	0.4016	-1000	-0.00514
40	0.1586	-1250	-0.00476
60	0.0257	-3333	-0.00233

For Langmuir model, the parameters  $K_L$  and  $q_m$  were calculated from the intercept and slope of the plotted isotherm. The essential characteristics of Langmuir equation is expressed as a dimensionless separation factor ( $R_L$ ) [15]:

$$R_L = \frac{1}{1 + K_L P_0} \quad (4)$$

where  $K_L$  (g/mmol) is Langmuir constant and  $P_0$  is initial pressure. The value of  $R_L$  between 0 and 1 indicates favourable adsorption. From the calculation, the negative value of  $R_L$  and  $q_m$  indicates unfavourable adsorption.

Fig. 4 and Table V show the Freundlich isotherm graph and Freundlich parameters. For Freundlich model, the parameter  $K_F$  was calculated from the graph intercept and  $n$  was calculated from the graph slope.

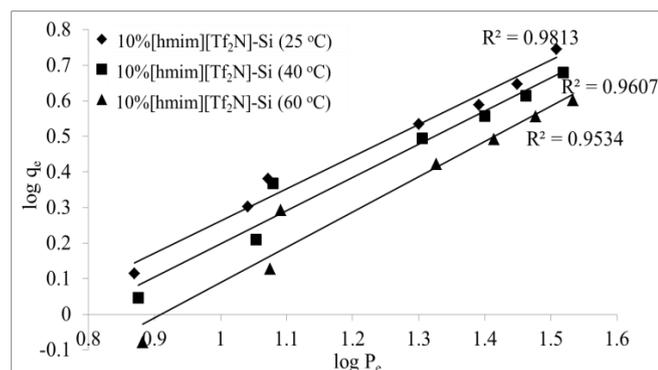


Fig. 4. Freundlich adsorption isotherm plot.

From Table IV and Table V, the  $R^2$  values for Freundlich model (0.9813, 0.9607, and 0.9534) were higher than Langmuir model (0.4016, 0.1586, and 0.0257) at all three temperatures. This indicated that, the adsorption experimental data was best fit to Freundlich isotherm. The  $n$  values were 1.1091, 1.0740 and 1.0080 for 25, 40, and 60 °C, respectively. This ' $n$ ' parameter can be considered as an indication for the adsorption favourability where  $n$  parameter lies between 1 and 10 indicates favourable adsorption [16]. From the  $R^2$  and  $n$  values, the adsorption was favoured at 25 °C. The greater  $K_F$  value at 25 °C (0.2298) than 40 °C (0.1851) and 60 °C (0.1251) also suggests that the greater adsorption tendency at 25 °C.

TABLE V: PARAMETERS FOR FREUNDLICH ISOTHERM

Temperature (°C)	Freundlich Isotherm		
	$R^2$	$K_F$	$n$
25	0.9813	0.2298	1.1091
40	0.9607	0.1851	1.0740
60	0.9534	0.1251	1.0080

## IV. CONCLUSION

Silica support [hmim][Tf<sub>2</sub>N] was successfully synthesized and characterized. The CO<sub>2</sub> adsorption capacity was the highest using [hmim][Tf<sub>2</sub>N]-Si due to high surface area provided by solid support that is necessary to accommodate intimate contact between CO<sub>2</sub> and ionic liquid and thus increase the CO<sub>2</sub> solubility in ionic liquid. The adsorption capacity increased as the pressure increased, while the adsorption capacity decreased as the temperature increased. The adsorption model was best fit to Freundlich isotherm with the highest  $R^2$  value of 0.9813 at 25 °C.

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ionic liquid for CO<sub>2</sub> capture.

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