



Ionic liquid as a new binder for activated carbon based consolidated composite adsorbents

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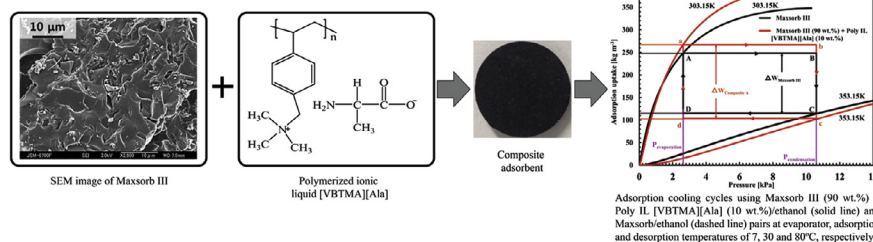
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HIGHLIGHTS

- Consolidated activated carbon composite is synthesized with polymerized ionic liquid (PIL) as binder.
- Synthesized composite has been characterized for adsorption heat pump applications.
- Composite with PIL as binder exhibits high porous properties.
- Composite with PIL as binder shows high thermal conductivity and volumetric uptake of ethanol.

GRAPHICAL ABSTRACT



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ABSTRACT

Consolidated composite adsorbents have gained much attention as next generation adsorbents in adsorption heat pump (AHP) applications due to some of their salient features such as improved uptake to volume ratio and high thermal conductivity. Synthetic polymers, which are generally used as binders for the composite adsorbents impose negative impact on adsorption capacity resulting from poor affinity for the refrigerant and pore blockage. To address these issues, a polymerized ionic liquid (IL) was explored as a potential binder in making consolidated activated carbon composite. Polymerized IL [VBTMA][Ala] (vinylbenzyltrimethyl ammonium alanate) was synthesized and characterized. The composite adsorbent was prepared with a mass ratio of 90% Maxsorb III and 10% of Poly IL [VBTMA][Ala]. It is observed that surface area and pore volume of new composite were increased to more than 11% and 18%, respectively, compared to polyvinyl alcohol (PVA) as a binder. Sorption tests for ethanol uptake were performed using thermogravimetric technique at 303.15 K, 323.15 K and 343.15 K with various evaporator pressures. For a typical operating condition of AHP system, composite using polymerized IL as binder showed 22% higher net ethanol uptake than the net uptake of parent material Maxsorb III whereas a remarkably high 85% increase in thermal conductivity was observed. Thus, polymerized IL could be considered as strong candidate for making consolidated composite adsorbents in AHP applications.

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1. Introduction

Ionic liquids (ILs) are composed entirely of ions and have been used as promising solvents/materials as they possess many unique

and attractive physicochemical properties, including negligible vapor pressure, excellent chemical and thermal stability, high ionic conductivity, a large liquid range and noncorrosive nature [1]. The physicochemical properties of ILs, including their viscosity, hydrophobicity, density, and solubility can be tuned by simply selecting different combinations of cations and anions as well as attached substituents, leading to the use of the terms “designer” and “task-specific ILs” [2]. Such unique properties allow ILs to be employed in a wide range of applications including extraction [3–5], organic and inorganic synthesis [6], biomass dissolution [7], enzymatic reactions [8], energy application [9], capture and separation of CO₂ [10–16]. Additionally ILs have been used as absorbents in thermally-driven vapor “adsorption” heat pump systems [17–22]. Majority of these studies utilize monomeric form of ILs, which are liquid at normal operating conditions. However, polymeric form of ionic liquids (PILs) have been recognized as excellent sorbents as they exhibit not only the interesting characteristics of ILs but also the general properties of macromolecular structure [23]. Polymerized form of ILs have been predominantly used for CO₂ capture and separation applications. Some representative examples are; Tang et al. [24] observed enhanced CO₂ sorption capacity and fast sorption/desorption rates of the formed PILs compared to room temperature ILs. Shahrom et al. [25] studied amino acid-based polymerized ionic liquids (AAPILs) to enhance the CO₂ adsorption. The results showed that AAPILs are capable of capturing CO₂ more than other ionic liquids. Similar results can be found in several other studies [26,27].

No significant study have been reported on the use of polymerized ILs for thermally-driven “adsorption” heat pump (AHP) systems. Unlike vapor “adsorption” systems reported earlier, AHP uses solid adsorbent for adsorbing refrigerant vapor. Highly porous activated carbons (ACs) are promising adsorbents for these AHP systems. For an effective adsorption process, adsorbents are required to possess huge surface area with high adsorption uptake of refrigerant vapor [28–34]. Therefore, the adsorbents are either in pellet or powder form [28,29]. The punitive results are poor thermal interaction [35] with the heat transferring media leading to the poor thermal efficiency and low volumetric capacity. Consequently, attempts have been made to formulate consolidated composite adsorbents with suitable binders added to parent adsorbents. Numerous binders have been investigated for thermal conductivity enhancement such as polyvinyl alcohol (PVA) [35,36], polyvinylpyrrolidone (PVP) [37], expanded graphite [38,39], polytetrafluoroethylene (PTFE) [40] to synthesize composite adsorbents. Nonetheless, such binders have poor affinity for refrigerant vapor [35,36] and pore blockage thereby reducing the uptake potential of such composite adsorbents. Hence, it is imperative to explore improved binders that not only exhibit high thermal conductivity but also are efficient adsorbents.

This research study is an effort to explore the utility of polymerized IL as a binder for the synthesis of composite adsorbents wherein the parent adsorbent is activated carbon. Polymerized amino acid based IL named as Poly IL [VBTMA][Ala] (vinylbenzyltrimethyl ammonium alanate) has been synthesized and introduced as binder for Maxsorb III (commercially available activated carbon). The key expected benefits of using polymerized IL for this study are: i) superior thermal conductivity than parent adsorbent (Maxsorb III); ii) ensures the composite in solid phase only during AHP operations, which is highly desirable; iii) polymerized ILs are known to have superior adsorption uptake than monomers whereas the traditional binders such as PVA, PVP, and PTFE do not possess any adsorption capability. In order to ascertain these properties, a detailed chemical characterization of the synthesized polymerized IL and thermo-physical studies together with adsorption investigation of the composite adsorbent has been undertaken.

2. Materials

Vinylbenzyltrimethyl ammonium chloride [VBTMA] [Cl] and anion exchange resin (Amberlyst A26 OH form) were procured from Sigma-Aldrich Chemical Co. (St. Louis, MO) and were used as received. The amino acid alanine, and solvents ethanol, methanol, and acetonitrile were obtained from Merck, Malaysia. 2,2-azobisisobutyronitrile (AIBN) as radical initiator was purchased from R & M, Malaysia. Activated carbon powder, namely Maxsorb III provided by Kansai Coke & Chemicals Co. Ltd., Japan. It possesses surface area and pore volume as 3293 m² g⁻¹ and 1.71 cm³ g⁻¹ respectively [41–44].

3. Experimental

3.1. Synthesis procedure of polymerized amino acid based ionic liquid

Poly IL [VBTMA][Ala] was synthesized and stored as described elsewhere [25]. Briefly, specified (10 g) amount of [VBTMA] [Cl] was mixed with ethanol (200 mL) and stirred few minutes to fully dissolve it. The solution of ethanol and [VBTMA] [Cl] was then passed through the resin [Amberlyst A26 (OH⁻ form)] column slowly. Basically, in this AER method mobile anions (Cl⁻) from [VBTMA] [Cl] + ethanol solution were exchanged with anions that were electrostatically bound to the functional groups (OH⁻) contained within the resin polymer. The resulting vinylbenzyltrimethyl ammonium hydride [VBTMA] [OH] solution was collected and then neutralized the solution with equimolar amount of alanine at 300 rpm for 24 h. Ethanol and water were removed by rotary evaporator at water bath temperature 323.15 K with rotation speed 100 rpm under reduced pressure. After removing those solvents, methanol and acetonitrile with a ratio of 3:7 were added to the mixture to precipitate out the unreacted alanine, which was separated by filtration. Added solvents were removed again by rotary evaporator to produce the desired amino acid (alanine) IL (AAIL) monomer.

2,2-azobisisobutyronitrile (AIBN) was used for the polymerization of monomer amino acid (alanine) IL (AAIL). The specified ratio (1:10) of monomer form of IL and AIBN were used for conducting polymerization. Few millilitres of methanol were added and stirred for 24 h at 338.15 K under reflux and inert atmosphere. The resulting polymerized amino (alanine) IL (Poly IL [VBTMA][Ala]) was dried in vacuum oven at 353.15 K and further dried using an air oven at 373.15 K [25].

3.2. Characterization of Poly IL [VBTMA][Ala]

Degradation temperature (T_d) of the synthesized Poly IL [VBTMA][Ala] was measured by PerkinElmer STA 6000 thermogravimetric analyzer (TGA) with heating rate of 40 K/min and flow rate of nitrogen 20 ml/min. Differential scanning calorimetry (DSC) of type Mettler Toledo model DSC 1 was employed to measure the glass transition temperature (T_g) of Poly IL [VBTMA][Ala]. DSC experiment was conducted with heating rate of 20 K/min and flow rate of nitrogen 50 ml/min in temperature range from 123.15 K to 403.15 K. ¹H-NMR spectra of synthesized monomer and polymer form of [VBTMA][Ala] was recorded on a Bruker Avance DRX-400 spectrometer operating at 400 MHz with deuterated methanol-d₄ as a solvent. The chemical shifts (δ) are reported in ppm. Multiplicities are abbreviated as follows: s for singlet, d for doublet, t for triplet, q for quartet, m for multiplet and br for broad. [VBTMA][Ala]: ¹H-NMR (Methanol-d₄, 400 MHz, ppm) 1.30 (d, 3H), 3.14 (s, 9H), 3.33 (q, 1H), 4.55 (s, 2H), 5.40 (d, 1H), 5.91 (d, 1H), 6.85 (q, 1H), 7.57 (d, 2H), 7.63 (d, 2H); Poly IL [VBTMA][Ala]: ¹H-NMR (Methanol-d₄, 400 MHz, ppm) 1.37 (d, 3H), 1.62 (br, 2H), 2.17

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