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Design of quaternary ammonium type-ionic liquids as desiccants for an air-conditioning system

Sho Maekawa, Ryo Matsumoto, Kenta Ito, Toshiki Nokami, Jian-Xin Li, Hiroshi Nakayama, Toshiyuki Ito

HIGHLIGHTS

- The liquid desiccant air-conditioning system allows reducing energy consumption compared to the conventional compressor-type air conditioners.
- Ionic liquid, cholinium dimethylphosphate ([Ch][DMPO4]) displayed the best dehumidification capability and the lowest equilibrium water vapor pressure.
- [Ch][DMPO4] also exhibited a less corrosive effect on metals and is non-toxic.
- The liquid desiccant-type air conditioner is expected to avoid the epidemic of the flu, tuberculosis, and any new types of corona virus diseases.

ABSTRACT

The liquid desiccant air-conditioning system allows reducing energy consumption compared to the conventional compressor-type air conditioners. In order to develop desiccant materials for air conditioners, we have investigated the dehumidification capability of quaternary ammonium Ionic Liquids (ILs) and the equilibrium water vapor pressure of aqueous solutions of these ammonium salts. Among the seven tested types of ILs, 2-hydroxy-N,N,N-trimethylethan-1-aminium dimethylphosphate ([Ch][DMPO4]) displayed the best dehumidification capability and the lowest equilibrium water vapor pressure. Furthermore, the 80% aqueous solution of [Ch][DMPO4] exhibited a less corrosive effect on four types of metals, i.e., steel (hot dip zinc-aluminum alloy plated steel), copper (C1100P), aluminum (A5052), and stainless steel (SUS: SUS304). It should be noted that this [Ch][DMPO4] is not only non-toxic but also exhibits a stable nature; the aqueous solution produced no odor after storing for over 1 year under ambient conditions.
1. Introduction

Global warming issues have caused increasing energy problems in electric power, because the electric power consumption significantly depends on the operating conditions of the air conditioners in the summer season [1]. Our sensible temperature is dependent on the humidity, so that appropriate control of the humidity is very important for us [2]; the estimated energy savings in electricity can reach 13% by increasing the air-conditioning temperature to 28°C from 27°C when the outside temperature is 31°C [2]. However, it is very uncomfortable for us in a room at 28°C under a high-humidity environment. Therefore, control of the humidity is the key to legalizing the energy savings and contributes to our sustainable human life. The development of an efficient air-conditioning system that enables controlling both the humidity and room temperature is now an important task in our society. A significant interest has been paid to developing a liquid desiccant-type air conditioner [3–9]; it is estimated that the liquid desiccant air conditioner has at least a 20% lower electric energy versus the conventional compressor-type air conditioners [4–6]. Furthermore, the liquid desiccant-type air conditioner can be driven with continuous ventilation during the air-conditioning process; this property is expected to avoid the epidemic of the flu, tuberculosis, and any new types of corona virus diseases among people who share the same room during working or living; the liquid desiccant-type air conditioner is a favorable air-conditioner system for hospitals, schools, homes, and supermarkets [3–9]. However, the present liquid desiccant-type air conditioner has a serious weak point that it uses a lithium chloride (LiCl) aqueous solution as the desiccant source [3–9]. The LiCl aqueous solution is very caustic to iron or copper, and it easily crystallizes when different salts are added for enhancing the dehumidification ability; this requires expensive corrosion-resistance pipes and prohibits its wide use. Furthermore, lithium is produced in only limited countries. Therefore, it is strongly required to find alternative desiccant materials which could replace LiCl.

Ionic liquids (ILs) have gained strong interests due to their unique physical and chemical properties and their applied fields have recently remarkably expanded [10–15]. Lee et al. reported the vapor pressure of several imidazolium ILs [16]. Since then several groups have investigated the relationship between the structure of the ILs and their moisture absorbance capability [16–36]. Luebke and co-workers reported that the origin of the water absorbance property of 1-ethyl-3-methylimidazolium acetate ([C2mim][OAc]) might be due to the strong interaction of the oxygen atom of the acetate anion with a water molecule based on the results of molecular simulation study [31]. Mu et al. indicated that water molecules in the air were trapped by the hydrogen-bonding between the acetate anion and the hydrogen atoms that were located at the 4- and 5-protons of the [C2mim] cation [35]. Kanakubo et al. suggested that the dehumidification capability of the ILs was dependent on not the cationic part but the anionic part of the IL [36]. Since many types of ILs have a moisture absorbent property, ILs are proposed as a desiccant source for the liquid desiccant type air-conditioning system [37–46]. Luo et al. attempted to use of several ILs as desiccants for an air conditioner [37,38]. Zegenhagen [41], Verela [42], and Giannetti [43,44] also demonstrated the use of ILs as desiccant materials for an air-conditioning system. We recently reported that tributyl(methyl)phosphonium dimethylphosphate ([P4441][DMPO4]) displayed an excellent dehumidification capability; the [P4441][DMPO4] aqueous solution (77% (w/w)) could be used as a desiccant for a liquid desiccant air conditioner [46]. The list of previously investigated ILs for their dehumidification property of vapor pressure versus water is shown in Fig. 1.

However, we further recognized the weak points of our developed [P4441][DMPO4] as follows. The first is that an aqueous solution of [P4441][DMPO4] exhibited a slightly corrosive behavior against copper metal (Table S1, entry 5), though the neat [P4441][DMPO4] showed no corrosive effect towards many metals [46]. The corrosive effect of an aqueous solution of [P4441][DMPO4] was not serious; it was significantly weaker than those of the LiCl aqueous solution and imidazolium type ILs. The second is the cost of manufacturing [P4441][DMPO4]; it is slightly more expensive versus the imidazolium or ammonium ILs. In order to solve these difficulties, we decided to again investigate the design of ILs as an ecological liquid desiccant-type air conditioner material.

Feyecon reported the possibility of choline carboxylate ILs, 2-hydroxy-N,N,N-trimethyl-1-aminium alkylcarboxylates ([Ch][RGO2]), in particularly, choline lactate ([Ch][Lac]) acted as an efficient desiccant [39]. Schaber [19] and Schifflet et al. [25] reported the results of the investigation of the physical properties of the [Ch] type ILs. Aparicio et al. investigated the water capture mechanism of [Ch][Lac] by a DFT simulation study [40]. We were fascinated by the choline type ILs [47] as a desiccant reagent because the choline type ILs were well known as safe and economical ILs [39,47]. Since choline is a quaternary ammonium cation, we decided to investigate the dehumidification property focusing on the quaternary ammonium type ILs as listed in Fig. 1. Although it was reported that [Ch][Lac] exhibited a good desiccant property [39], unfortunately, we found that the aqueous solution of [Ch][Lac] gradually decomposed at over 80°C and produced a bad smell during the experiments. We also revealed that the acetate ILs produced an odor by partial decomposition, though their dehumidification capabilities were excellent [46]. Zhao [20] and Zheng [27] reported the high dehumidification property of 1,3-dialkyylimidazolium dimethylphosphate. As already mentioned, we established that the [DMPO4] type ILs generally afforded a high dehumidification ability [46]. We now report the results of the systematic evaluation of several types of quaternary ammonium salts including choline as a desiccant for a liquid desiccant air-conditioner.
2. Experimental

2.1. Method of evaluation of desiccant capability of ILs

(1) We evaluated the dehumidification capability of the ILs by the same method reported in our previous article [46]; a sealed Ziplock® freezer bag (L) that includes the IL sample (ca. 1.0 g) in a lab dish (32 × 15 mm) and the humidity sensor (TR-74U) was placed in an oven (at 30 °C). The humidity change inside of the bag was recorded and the dehumidification rate of the sample liquids was determined by the time it reached a 50% humidity between the maximum humidity and that of the lowest one after reaching the equilibrium state. For the details, see our previous report [46]. Although this method was not applicable to measure the accurate dehumidification capability, it is useful for the evaluation of the dehumidification ability of the ILs. The Ziplock® freezer bags were purchased from Asahi Kasei Home Products, Ltd.

(2) The equilibrium water vapor pressure was measured using the apparatus shown in Fig. 2. The dehumidification capacity of a 30% (w/w) aqueous solution of LiCl was also determined; 9.63 kPa water vapor pressure (25 °C); 20.0 kPa water vapor pressure (52 °C) and 161 kPa water vapor pressure (50 °C) (Figure S1). The results are in good agreement with the ones reported in Ref. [48].

(3) Synthesis of 2-hydroxy-N,N,N-trimethyl-1-ammonium dimethyl phosphate ([Ch][DMPO4]) (CAS 118878-98-6): 2-(N,N-Dimethylamino)ethanol (213.0 g, 2.39 mol) was mixed with tri-methyl phosphate (337.0 g, 2.41 mmol) at 0 °C, and the mixture was then stirred at 55 °C for 24 h. After allowing the mixture to cool to room temperature (rt), the mixture was washed three times with ether and dried under reduced pressure at 1.0 torr rt for 3 h. To the resulting salt (534.2 g) was added activated charcoal (2.0 g) dissolved in deionized water (1.0 L) and the mixture was stirred at 55 °C for 5 h, then the charcoal removed by filtration through a glass sintered filter with a Celite® pad, the filtrate was evaporated to dryness, and further dried by lyophilization to afford [Ch][DMPO4] (500.0 g, 2.18 mol) in 91% yield; Mp 57 °C; 1H NMR (600 MHz, ppm, CDCl3) δ 3.37 (9H, s), 3.59 (3H, s), 3.60 (3H, s), 3.59–3.61 (4H, m), 4.03–4.05 (4H, m), 4.89 (2H, s, OH); 13C NMR (150 MHz, ppm, CDCl3) δ 52.46, 52.50, 54.4, 56.3, 68.2; ESI-MS m/z (M+) calcd for CH3NO+ 104.1076, found 104.1064, m/z (X−) calcd for C3H10O4P− 125.0003; found 125.0001.

2.2. Determination of water vapor pressure

Fig. 2. Schematic view of our apparatus for the equilibrium water vapor pressure measurement.

system, i.e., the dimethylphosphate ([DMPO4]) salts of choline ([Ch]), 2-hydroxy-N-(2-hydroxyethyl)-N,N-dimethylethan-1-aminium ([BHEDMA]), 2-hydroxy-N,N-bis(2-hydroxyethyl)-N-methylethan-1-aminium ([THEMA]), N,N-diethyl-N-methylethanaminium ([N2221]), and N1,N1,N2,N2-hexamethylethane-1,2-diaminium ([HMEDA]). To obtain the information for discussing the origin of the dehumidification property of IL aqueous solutions, we also investigated the moisture absorbent property of choline ([Ch]) salts such as choline methylsulfate ([Ch][MeSO4]) and choline bis(trifluoromethylsulfonyl)amide ([Ch][NTf2]) (Fig. 1).

2. Results and discussion

The dehumidification capabilities of seven types of quaternary ammonium salts, which are listed in Fig. 1, are listed in Table 1. It is

| Entry | Salts | Dehumidification capability, %/mol | Dehumidification rate, %/ (min−1·mol−1) |
|-------|-------|-----------------------------------|-----------------------------------------|
| 1     | [Ch][DMPO4] | 8939 | 687 |
| 2     | [Ch][MeSO4] | 5381 | 739 |
| 3     | [Ch][NTf2] | 4510 | 7.6 |
| 4     | [BHEDMA] | 2649 | 306 |
| 5     | [THEMA] | 3347 | 318 |
| 6     | [HMEDA] | 4150 | 638 |
| 7     | [N2221] | 2484 | 281 |
| 8     | 30% LiCl aq. solution | 725 | 191 |

a The results using 1.0 g of the salts.

b 5.0 g of 30% (w/w) LiCl aq. solution was used.
important to prepare the ILs by a simple method, because fairly high amounts of the ILs are needed for the air-conditioning system. Although [Ch][OAc] is a natural compound and is expected to exhibit a high dehumidification capability, and since the acetate ILs are generally unstable, we choose dimethylphosphate ([DMPO4]), methylsulfate ([MeSO4]), and bis(trifluoromethylsulfonyl)amide ([NTf2]) as the anionic parts for the present study. It was noted that no significant odor was detected from the aqueous solutions of these ILs.

The results of the dehumidification tests are shown in Table 1. The dehumidification capabilities of [N2221][DMPO4] (entry 7) and a 30% aqueous LiCl solution (entry 8) were also investigated as the controls in the present study, because our evaluation system is not applicable to obtain the accurate dehumidification capability of a desiccant but useful for the evaluation of its ability [46]. As shown in Table 1, all the tested ILs exhibited a better dehumidification property than the 30% LiCl aq. solution, in particular, [Ch][DMPO4] showed the best dehumidification capability (entry 1), though [Ch][MeSO4] showed a faster dehumidification rate (entry 2) than [Ch][DMPO4] (entry 1). The dehumidification capability of [Ch][DMPO4] attained 89.39% mol/C01 (entry 1) which was 10 times higher than that of the 30% (w/w) LiCl aqueous solution and the rate was 3.4-fold faster than the LiCl aqueous solution (entry 8).

Although we anticipated that the hydroxyethyl group in the ammonium cation may be related to the dehumidification capability, the results were different from our expectation. The [Ch][DMPO4] afforded a superior dehumidification capability compared to the bis- and tris-hydroxyethyl-substituted ILs (entries 4 and 5). However, the dehumidification capability of the tris(hydroxyethyl)-substituted salt ([THEMA][DMPO4]) was higher than that of the bis(hydroxyethyl)-substituted salt ([BHEDMA][DMPO4]) (entries 4 and 5). In addition, the bis(trimethylammonium) salt ([HMEDA][DMPO4]) produced a higher dehumidification capability than [N2221][DMPO4] (entries 6 and 7).

In a liquid desiccant type air conditioner, the desiccant materials should be used as aqueous solutions to regenerate the dry state by the moisture change between the outside air. In order to smooth the moisture change, a low equilibrium water vapor pressure is favorable. Hence, we next measured the equilibrium water vapor pressure of these IL aqueous solutions (30, 50, and 80 wt%) using the apparatus illustrated in Fig. 2 and the results were shown in Figs. 3 and 4.

The lowest vapor pressure IL was again [Ch][DMPO4] (Fig. 3a). The vapor pressures of the aqueous solutions of [Ch][DMPO4] (Fig. 3a) were much lower than those of the bis- and tris-hydroxyethyl-substituted ammonium salts (Fig. 3b and c). Comparing these properties of [BHEDMA][DMPO4] (Fig. 3b) and [THEMA][DMPO4] (Fig. 3c), the bis-hydroxyethyl-substituted IL showed a lower vapor pressure than [THEMA][DMPO4]. On the other hand, no significant difference was observed in the water vapor pressure between [N2221][DMPO4] (Fig. 4a) and [HMEDA][DMPO4] (Fig. 4b). It was thus found that the origins that

![Fig. 3. Vapor pressure of hydroxyethyl group-substituted quaternary ammonium ILs. (a) The equilibrium water vapor pressure of [Ch][DMPO4] aq. solution. (b) The equilibrium water vapor pressure of [BHEDMA][DMPO4] aq. solution. (c) The equilibrium water vapor pressure of [THEMA][DMPO4] aq. solution.](image)

![Fig. 4. Vapor pressure of two types of quaternary ammonium ILs. (a) The equilibrium water vapor pressure of [N2221][DMPO4] aq. solution. (b) The equilibrium water vapor pressure of [HMEDA][DMPO4] aq. solution.](image)
determine the dehumidification capability and the equilibrium water vapor pressure should be different.

The present commercial liquid desiccant conditioner uses a 30% LiCl aqueous solution as the desiccant and the equilibrium water vapor pressure of the 30% LiCl aqueous solution exhibited 15.1 hPa at 30 °C (Figure S1). Based on the results shown in Figs. 3 and 4, we estimated the equilibrium water vapor pressure at 30 °C for the five ILs (Fig. 5). To our delight, [Ch][DMPO₄] gave very satisfactory results about this property, and the vapor pressure was assumed to be less than 15 hPa over a 40 wt% concentration. However, unfortunately, the other four types of quaternary ammonium salts afforded no satisfactory vapor pressure even at an 80 wt% concentration (Fig. 5).

Brennecke and Ficke proposed that an IL, which consisted of a small sized non-protic cation showed a higher moisture absorbance property than an IL which has a protic substituent in the cationic part due to the decreased interaction of the water molecule with the IL pair, because introduction of the hydroxyethyl moiety in the cationic part strengthened the cation-anion interaction [49]. Since the vapor pressure of the [Ch] [DMPO₄] aq. solution was lower than those of [BHEDMA][DMPO₄] and [THEMA][DMPO₄], the results supported Brennecke’s proposal [49].

On the other hand, from the standpoint of the dehumidification property of the neat ILs, except for the case of [Ch][DMPO₄], [HMEDA] [DMPO₄] showed the highest dehumidification capability. It was reported that the 1,1’-(1,6-hexanediyl)-bis(imidazolium)dichloride-silica hybrid material showed a significant water vapor absorption [50]. These results seem to suggest that the polycationic quaternary ammonium salts could become good desiccant materials, though such a compound may not suitable as a desiccant source for an air-conditioner system.

The cost of piping materials could be reduced when less or non-corrosive liquids are employed. Therefore, the corrosive effect of the desiccant materials on metals was evaluated. We examined the caustic effect of the [Ch][DMPO₄] aqueous solution against four types of metal plates that were commonly used as the pipes of air-conditioners as follows: the metal plates were dipped in 5.0 mL of a 30% LiCl or 80% [Ch] [DMPO₄] aqueous solution as shown Fig. 6a-1 and b-1, respectively. After gentle mixing, the test liquids avoided contacting the stirrer bar to the metal plate at 80 °C for 24 h, the metal plate was removed from the bottles and carefully washed with deionized water. The metal plate was then dried under reduced pressure and its weight was measured. As shown in Fig. 6 and Table 2, the corrosive effect of the 80% [Ch]
[DMPO4] aqueous solution on steel (hot dip zinc-aluminum alloy plated steel), Al, and stainless steel (SUS) was very weak; no color change of the metal surface or the IL solutions was observed (Fig. 6b-2). However, a small weight loss was observed for the Cu plate, though no color change of the liquid was observed (Fig. 6b-2). On the contrary, the 30% LiCl aqueous solutions gradually colored during mixing and a significant amount of precipitates were produced for all the metal samples (Fig. 6a) and significant weight losses of the three metal plates (steel, Cu, and stainless steel) were observed (entry 1 in Table 2). Among the tested ILs, [Ch][DMPO4] afforded the lowest corrosion to the metal plates. The results of the corrosive tests of the other ILs were shown in Table S1 in the supplementary information. Although the 80% [Ch][DMPO4] aqueous solution showed a weak corrosive effect against copper (entry 2), the level was slightly weaker than that of the 77% aqueous solution of [P4441][DMPO4]. However, [Ch][DMPO4] is more economical than [P4441][DMPO4]. We recently reported that [P4441][DMPO4] exhibited a favorable property as a liquid desiccant material [48]. The dehumidification capability of [Ch][DMPO4] was slightly inferior to that of [P4441][DMPO4]. However, [Ch][DMPO4] is more economical than [P4441][DMPO4] and it would be prepared at less than 1/3 the cost compared to the latter IL. Therefore, we believe that [Ch][DMPO4] might be the best economical IL as a desiccant for use in a liquid desiccant type air conditioner to date. It is expected that the IL-mediated liquid desiccant air conditioner allows a significant savings in the consumption of electricity compared to the conventional compressor-type air conditioner. Furthermore, since the system can be driven with continuous ventilation, we strongly expect that the liquid desiccant air conditioner using the IL

We investigated the temperature dependence of the viscosity of the aqueous solutions of the five ILs (Fig. 7). Three ILs, [Ch][DMPO4], [BHEDMA][DMPO4], and [N2221][DMPO4], showed similar viscosities, on the contrary, the tris(hydroxyethyl)-substituted salt [THEMA][DMPO4] showed a high viscosity and the dicationic IL [HMEDA][DMPO4] exhibited a very high viscosity due to the increased cation-anion interaction of the ILs. The viscosity of the 80% aqueous solution of [Ch][DMPO4] was ca. 1.3-fold higher than that of the 77% aqueous solution of [P4441][DMPO4] [46] (Figure S2). The results indicated that an additive liquid may be needed to reduce the viscosity when this IL aqueous solution would be employed as a desiccant liquid for air conditioners.

4. Conclusion

We investigated seven types of quaternary ammonium ILs as desiccants for the liquid desiccant air conditioner. [Ch][DMPO4] displayed the best dehumidification capacity with the highest dehumidification rate among the tested ILs. Furthermore, the aqueous solution of [Ch][DMPO4] afforded a very favorable equilibrium water vapor pressure. It should also be noted that no significant toxicity of this [Ch][DMPO4] was observed and stability of this IL was very high. The less-corrosive property of the [Ch][DMPO4] solution is another attractive future of this IL. Furthermore, the aqueous solution of [Ch][DMPO4] produced no odor even when stored for over 1 year under ambient conditions. Even if the liquid produces a very slight odor, it might become a serious problem because moisture in the outside air is removed by contacting with the liquid desiccant layer and moves into a room. It is well known that our nose is a very sensitive analyzer that is superior to the usual analytical instruments in the lab. In fact, blenders use only their nose in the blending of perfumes. Therefore, we determined the smell of ILs by our olfactory response.

We recently reported that [P4441][DMPO4] exhibited a favorable property as a liquid desiccant material [48]. The dehumidification capability of [Ch][DMPO4] was slightly inferior to that of [P4441][DMPO4]. However, [Ch][DMPO4] is more economical than [P4441][DMPO4] and it would be prepared at less than 1/3 the cost compared to the latter IL. Therefore, we believe that [Ch][DMPO4] might be the best economical IL as a desiccant for use in a liquid desiccant type air conditioner to date. It is expected that the IL-mediated liquid desiccant air conditioner allows a significant savings in the consumption of electricity compared to the conventional compressor-type air conditioner. Furthermore, since the system can be driven with continuous ventilation, we strongly expect that the liquid desiccant air conditioner using the IL

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**Table 2**

Corrosion test of [Ch][DMPO4] 80 wt% aqueous solution vs metal plates.

| Entry | Liquids                                      | Steel [a] | Cu(C1100P) [b] | Al (AS5052) [c] | Stainless steel (SUS304) [d] |
|-------|----------------------------------------------|-----------|-----------------|-----------------|------------------------------|
| 1     | LiCl 30 wt% aq. solution                     | -0.1270%  | -0.1673%        | 0.0000%         | -0.0000%                    |
| 2     | [Ch][DMPO4] 80 wt% aq. solution              | 0.0000%   | 0.04280%        | 0.0000%         | -0.03242%                   |

[a] Steel (hot dip zinc-aluminum alloy plated steel).
[b] Cu (tough-pitch copper: C1100P).
[c] Al (AS5052).
[d] Stainless steel (SUS: SUS304).
[e] Initial weight: 2.1991 g.
[f] Initial weight: 2.5694 g.
[g] Initial weight: 2.1679 g.
[h] Initial weight: 2.1983 g.
[i] Initial weight: 2.5658 g.
[j] Initial weight: 0.7700 g.
[k] Initial weight: 0.7739 g.
[l] Initial weight: 2.1664 g.

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**Fig. 7.** Temperature dependence of viscosity of 80 wt% aqueous solutions of the five types of ILs.
aqueous solution might contribute to avoiding an outbreak of the flu, tuberculosis, and any new type of corona virus disease in the near future.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jge.2020.09.016.

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