

Herbicidal ionic liquids with 2-(2,4-dichlorophenoxy)propionate anion

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Please cite as: CHEMIK 2016, 70, 9, 555–563

Introduction

In the last two decades the interest in this fascinating group of compounds that are ionic liquids has been growing continuously [1]. One of the most important properties of ionic liquids is their inflammability and non-volatility, for which they are considered slightly polluting substances [2, 3]. A new possibility of using ionic liquids is to incorporate a herbicidal anion in their structure. This action gives herbicidal ionic liquids (HILs) [4]. Thanks to the activity against unwanted vegetation, they can help to improve both quality and quantity of crops. HILs, due to their unique physicochemical properties, allow the reduction of an effective herbicide dose, as well as the control of its toxicity and soil mobility [4 ÷ 13]. Herbicidal anions that are usually used in synthesis of a herbicidal ionic liquid are groups derived from phenoxyacids such as (4-chloro-2-methylphenoxy)acetic (MCPA), [4 ÷ 7], (2,4-dichlorophenoxy)acetic (2,4-D) [8, 9], 2-(4-chloro-2-methylphenoxy)propionic (MCP) [10, 11] and 4-(4-chloro-2-methylphenoxy)butyric acid (MCPB) [12]. Other substances used include derivatives of benzoic acid, e.g. dicamba [14], nicotinic acid, e.g. clopyralid [15], sulfonyleureas, e.g. Metsulfuron-methyl (MSM) [16] or one of the most popular non-selective herbicides – glyphosate [17]. In the recent years, the 2nd generation of a herbicidal ionic liquid was also described, where a herbicidal anion was successfully combined with a cation exhibiting biological activity as a growth regulator [18] or fungicide [19].

Result discussion

Herbicidal ionic liquids were obtained in a two-step synthesis. The first step involved the quaternisation of 2-(2-dimethylaminoethoxy)-ethanol with respective alkyl bromides, which produced a series of alkyl[2-(2-hydroxyethoxy)ethyl]dimethylammonium bromides.

In the next step they underwent a substitution reaction of a bromide anion to herbicidal anion – 2-(2,4-dichlorophenoxy)propionate (2,4-DP). Potassium bromide was a by-product of a metathesis reaction carried out in methanol and it was separated. In order to remove residues of the by-product, the resulting compounds were additionally purified using anhydrous acetone and dried *in vacuo*. The ionic liquids obtained are presented in Table I with their yield, purity and main physicochemical properties.

The yield of the two-step reaction in all the cases was very high, falling into a range of 86 to 94%. All the organic salts obtained were at a liquid state at ambient conditions, which resulted in their classification as room temperature ionic liquids (RTILs). Purity of the salts synthesised was determined by a two-phase titration as per the standard PN-EN ISO 2871–2:2010.

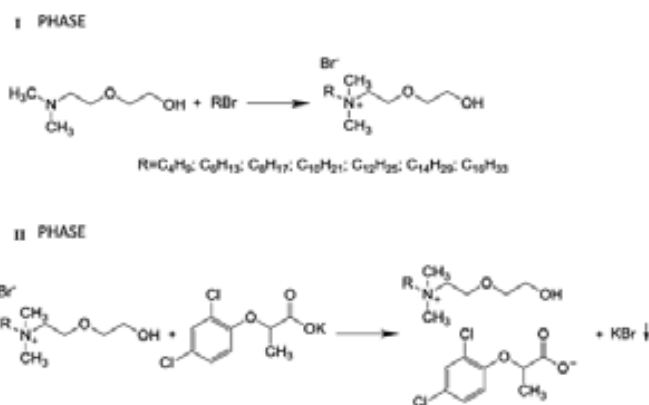


Fig. 1. Reaction of receiving herbicidal ionic liquids

Table I

Synthesized salts

Salt	R	Yield %	Purity %	Density ^a g·cm ⁻³	Viscosity ^a Pa·s	Refraction index ^a
1	C ₄ H ₉	94	98	1.217	6.729	1.516
2	C ₆ H ₁₃	89	98	1.164	3.272	1.506
3	C ₈ H ₁₇	92	99	1.147	2.604	1.501
4	C ₁₀ H ₂₁	93	98	1.138	2.167	1.504
5	C ₁₂ H ₂₅	91	99	1.119	9.268	1.511
6	C ₁₄ H ₂₉	88	98	1.098	2.606	1.501
7	C ₁₆ H ₃₃	86	99	1.089	1.818	1.499

^a at 20°C

The compound structure was confirmed by means of proton and carbon nuclear magnetic resonance. The ¹H NMR showed a characteristic resonance signal from protons of two methyl groups present both at quaternary nitrogen atoms at approx. 3.1 ppm, as well as two methylene groups, which appeared as multiplets at approx. 3.4 and 3.6 ppm. On the other hand, the signals from aromatic ring protons in anions were seen as two doublets at approx. 6.9 ppm (*J* = 8.9 Hz) and 7.4 ppm (*J* = 2.6 Hz) and doublet of doublets at approx. 7.2 ppm (*J*_{1,2} = 8.8 Hz, *J*_{1,3} = 2.6 Hz).

The values of viscosity for the salts synthesised were in a range of 1.82 Pa·s (for the salt 7 with the longest alkyl substituent – hexadecyl) to 9.27 Pa·s (for the salt 5 with a dodecyl substituent). According to the literature [20, 21], viscosity of ionic liquids usually increases as non-polar fragments (e.g. a carbon chain) in a cation get bigger. However, such relationship was not observed for the ionic liquids obtained, where instead the property analysed decreased in the series:

$$9.27 \text{ (5)} > 6.73 \text{ (1)} > 3.27 \text{ (2)} > 2.61 \text{ (6)} > 2.60 \text{ (3)} > 2.17 \text{ (4)} > 1.82 \text{ (7)}$$

Similar results were obtained for other homologous series of herbicidal ionic liquids containing phenoxyacids in the anion

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(MCPA, MCPB). For these compounds, there was also no relation observed between the alkyl or alkoxyethyl substituent and the measured viscosity [5, 12]. This specific behaviour of herbicidal ionic liquids may be related to a different coordination ability of phenoxy-carboxylic anions, as opposed to conventional, structurally simpler anions composing ionic liquids.

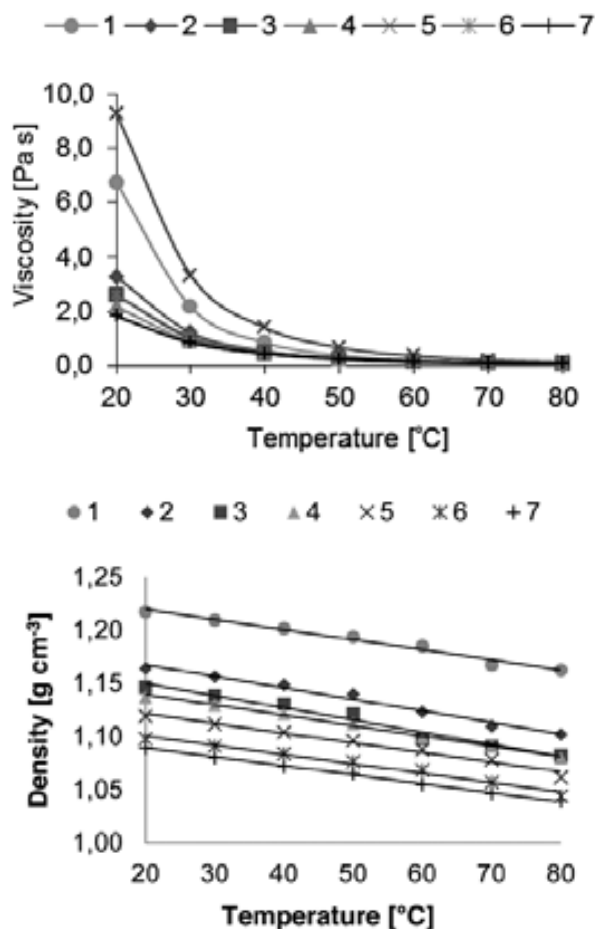


Fig. 2. The effect of the temperature on viscosity and density for obtained ionic liquids

Moreover, similarly to other ionic liquids, viscosity of the compounds 1–7 decreased non-linearly with the increase in temperature (Fig. 2). The highest drop occurred at the increase in temperature from 20 to 40°C. As a result, ionic liquid viscosity was in a range of 0.43 to 1.39 Pa·s. The further increase in temperature, from 40 to 80°C, caused a small decrease in the parameter analysed, which for all the salts was approx. 0.1 Pa·s in the end.

At 20°C, the ionic liquids obtained had higher density than water, with values in a range of 1.089 g·cm⁻³ for 7 to 1.217 g·cm⁻³ for 1 (Tab. 1). It was observed that for all the ionic liquids tested the density decreased linearly with the increase in the temperature (Fig. 2). The lines, showing the relation between density and temperature for the compounds 1–7, can be described by the equation $y = ax + b$, where coefficient a was approx. -0.0008, while b varied from 1.106 to 1.239 g·cm⁻³ ($R^2 > 0.97$). Finally, at 80°C, the ionic liquids obtained had the density lower by approx. 0.06 g·cm⁻³, with values in a range of 1.04 g·cm⁻³ for 7 to 1.16 g·cm⁻³ for 1. It was also shown that the increase of an alkyl chain length from C₄ (1) to C₁₆ (7) resulted in the decrease in the density in the entire temperature range analysed, which is consistent with the results obtained for other herbicidal ionic liquids [5, 12]. The relation observed (Fig. 3) is caused by the increase in the free volume of ionic liquid molecule, as the carbon substituent gets longer.

Table 1 presents the results of refractive index measurements for the salts synthesised at 20°C. The values measured were in a range

of 1.499 for the salt containing hexadecyl group (7) to 1.516 for the salt with a butyl substituent (1). It was observed that the increase in sample temperature reduces the value of refractive index. Moreover, just as for density measurements, this decrease can be described with a linear equation. For the lines representing the relation between the refractive index and temperature for compounds 1–7, coefficient a was approx. -0.0003, while b varied from 1.506 to 1.521 g·cm⁻³ ($R^2 > 0.98$). At highest measurement temperature (80°C), the refractive index decreased in the series:

1.498 (1) > 1.492 (5) > 1.488 (2) > 1.486 (4) > 1.484 (3) > 1.482 (6) > 1.477 (7)

The analysis of the data collected failed to find a clear correlation between the length of an alkyl substituent in compounds 1–7 and the refractive index values obtained in the temperature range analysed (Fig. 3).

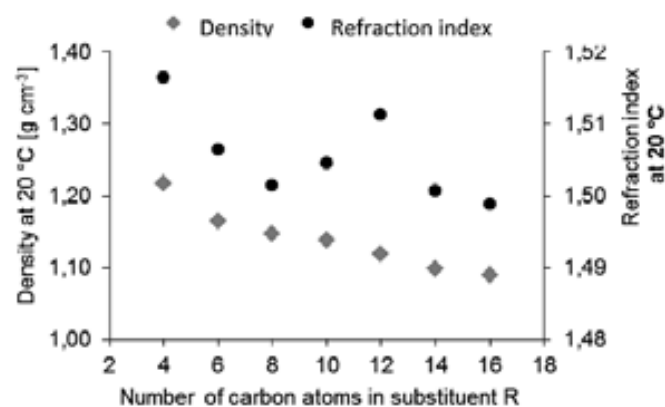


Fig. 3. The effect of number of carbon atoms in the alkyl substituent (R) on density and refractive index for synthesized ionic liquids

For compounds 1–7, the solubility was determined for water and popular organic solvents such as methanol, isopropanol, acetone, acetonitrile, DMSO, ethyl acetate, chloroform, hexane and toluene [22]. The solvents are ranked in a descending order of polarity according to the Snyder scale (Tab. 2).

All the ionic liquids synthesised were well soluble in the majority of solvents: acetone, isopropanol, acetonitrile, methanol, ethyl acetate, chloroform and toluene. However, the solubility in water and in DMSO depended on the length of an alkyl chain in a cation. It was found that the longer the alkyl chain in the cation is, the highest the hydrophobicity of the synthesised compounds is. As a result, the ionic liquids with tetradecyl (6) and hexadecyl (7) substituents were insoluble, while the salt with the dodecyl cation (5) showed limited solubility in water. The similar behaviour was observed for the compounds in DMSO, where good solubility was found only for 1 (with the butyl substituent), 2 (with the hexyl substituent) and 3 (the octyl substituent). Moreover, none of the compounds obtained was soluble in non-polar hexane, which is consistent with reports on other herbicidal ionic liquids [12, 16, 17].

Greenhouse experiments on weeds: the lamb's quarter (*Chenopodium album* L.) and cornflower (*Centaurea cyanus* L.) were conducted to determine the herbicidal activity of the compounds synthesised. They were applied together with a commercial formulation at a reduced dose of 400 g active ingredient per hectare (the recommended dose for the weeds tested is 600 g a.i./ha). The herbicidal ionic liquids tested maintained the biological activity of the anion (Tab. 3). A higher fresh weight reduction was obtained for the cornflower. The results at a level of 78–94% prove the high efficacy of the salt, however the values obtained were usually slightly lower than the commercial herbicide containing 2,4-D in

a form of dimethylammonium salt. **6** showed the highest activity against the cornflower, while compound **5** proved to be the most effective against the lamb's quarter. Moreover, compounds **3–7** showed higher herbicidal activity against the lamb's quarter than the reference formulation.

Table 2

The solubility of prepared ionic liquids at 25°C

Salt	Water	Methanol	DMSO	Acetonitrile	Acetone	Isopropanol	Ethyl acetate	Chloroform	Toluene	Hexane
	9.0a	6.6	6.5	6.2	5.1	4.3	4.3	4.1	2.3	0.0
1	+	+	+	+	+	+	+	+	+	-
2	+	+	+	+	+	+	+	+	+	-
3	+	+	+	+	+	+	+	+	+	-
4	+	+	+/-	+	+	+	+	+	+	-
5	+/-	+	+/-	+	+	+	+	+	+	-
6	-	+	+/-	+	+	+	+	+	+	-
7	-	+	+/-	+	+	+	+	+	+	-

^a polarity in Snyder's scale; + soluble (> 10.0% w/v); +/- limited solubility (3.3÷10.0% w/v); - solubility (<3.3% w/v)

Table 3

Fresh weight reduction of cornflower and common lambsquarters

Object	Fresh mass reduction [%]	
	cornflower	lamb's quarter
1	82	62
2	78	58
3	92	71
4	90	73
5	93	79
6	94	75
7	91	74
DMA 2,4-D ^a	94	65

^a reference formulation – 2,4-D dimethylammonium salt

Figure 4 presents the relation between the efficacy of the salt tested and the number of carbon atoms in alkyl chain (R). It shows that the herbicidal activity increases with an alkyl substituent length in the compounds tested. A clear increase in efficacy was observed for compound **3** – with the octyl or longer substituent (**4–7**). Maximum fresh mass reduction of weeds corresponds to the compounds with chains having 12–14 carbon atoms. Making the substituent longer results in a slight decrease in the activity of the salts tested.

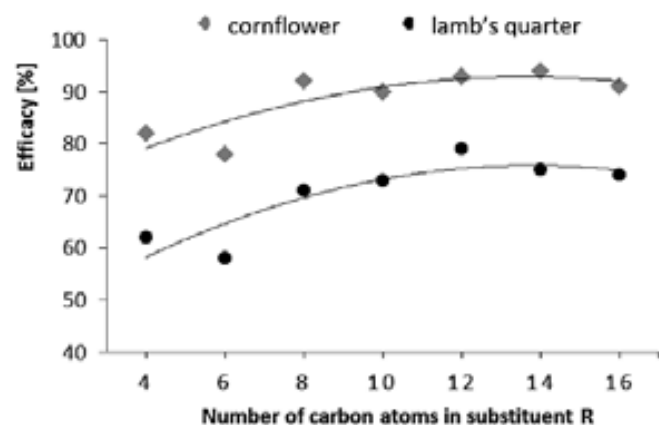


Fig. 4. The dependence of efficacy on number of carbon atoms in the alkyl substituent (R) for obtained ionic liquids

Summary and conclusions

An efficient method of obtaining new ionic liquids with herbicidal activity was developed and presented in this paper. The discussed ionic liquids contain the alkyl[2-(2-hydroxyethoxy)ethyl]dimethylammonium cation and the 2-(2,4-dichlorophenoxy)propionate anion. Nuclear magnetic resonance spectra allowed the identification of characteristic signals coming from the cation and anion, which confirms the structure of the compounds obtained. All the compounds synthesised were solid under ambient conditions. The effect of the alkyl substituent on the basic physicochemical parameters of the organic salts obtained was observed. As the number of carbon atoms in the chain increased, the solubility in water and DMSO of the ionic liquids synthesised decreased. Moreover, it was also observed that increasing the length of the alkyl substituent in the cation of the ionic liquids by 12 carbon atoms results in a density decrease by approx. 0.12 g·cm⁻³. For the ionic liquids synthesised, the values of viscosity, density and refractive index decreased with the increase in temperature. Furthermore, the last two parameters changed in a linear manner. The analysis of herbicidal activity led to the conclusion that the biological activity of the 2,4-DP anion was maintained. Moreover, an analysis was performed for efficacy as a function of the number of carbon atoms in alkyl substituent (R) for the ionic liquids obtained. The herbicidal activity increased greatly for ionic liquids containing the octyl or longer substituent. The highest values of fresh mass reduction for the lamb's quarter were observed for the ionic liquids containing a dodecyl group, whereas for the cornflower the highest values were obtained using the salt with the tetradecyl substituent.

Experimental part

Materials

2-(dimethylaminoethoxy)ethanol (98%, Sigma-Aldrich), 1-bromobutane (99%, Sigma-Aldrich), 1-bromohexane (99%, Sigma-Aldrich), 1-bromooctane (99%, Sigma-Aldrich), 1-bromododecane (98%, Sigma-Aldrich), 1-bromododecane (97%, Sigma-Aldrich), 1-bromotetradecane (97%, Sigma-Aldrich), 1-bromohexadecane (97%, Sigma-Aldrich), 2-(2,4-dichlorophenoxy)propionic acid (98%, Alfa Aesar), potassium hydroxide (85%, POCH).

Synthesis

Synthesis of alkyl[2-(2-hydroxyethoxy)ethyl]dimethylammonium bromides:

0.02 mole of 2-(2-dimethylaminoethoxy)ethanol was dissolved in 50 cm³ acetonitrile, followed by dropping in 0.02 mole of respective alkyl bromides. The system was stirred at 50°C under a reflux condenser for 24 hours, after which the solvent was evaporated, and then the raw product was carefully dried.

Obtaining herbicidal ionic liquids:

0.01 mole of alkyl[2-(2-hydroxyethoxy)ethyl]dimethylammonium was dissolved in 20 cm³ of methanol, followed by the addition of 0.01 mole of potassium 2-(2,4-dichlorophenoxy)propionate obtained prior in the reaction of 2,4-DP acid neutralization with potassium hydroxide in water. The reaction of an anion substitution was conducted for 3 hours under a reflux condenser at 45°C. The final reaction mixture was cooled, precipitated the inorganic salt (potassium bromide) filtered off, and the solvent was evaporated using a rotational vacuum evaporator. The product obtained was dissolved in acetone, after which the precipitate consisting of unreacted substrates and residues of the by-product were filtered off, and the solvent was evaporated. The final product was dried *in vacuo* at 50°C for 24 hours.

butyl[2-(2-hydroxyethoxy)ethyl]dimethylammonium 2-(2,4-dichlorophenoxy)propionate (1):

¹H NMR (CD₃OD) δ [ppm] = 0.87 (t, J = 6.95 Hz, 3H), 1.29 (s, 2H), 1.62 (d, J = 6.76 Hz, 3H), 1.65 (m, 2H), 3.22 (s, 6H), 3.37 (m, 2H), 3.60 (m, 4H), 3.70 (m, 2H), 3.89 (s, 2H), 4.43 (q, J = 6.6 Hz, 1H), 6.7 (d, J = 8.91 Hz, 1H), 7.13 (dd, J_{1,2} = 8.8 Hz, J_{1,3} = 2.6 Hz, 1H), 7.38 (d, J = 2.6 Hz, 1H); ¹³C NMR (CD₃OD) δ [ppm] = 178.8; 154.7; 130.7; 128.5; 126.1; 124.6; 116.3; 77.8; 73.7; 66.8; 65.6; 64.5; 61.9; 52.0; 33.0; 30.6; 27.6; 23.7; 23.6; 19.5; 14.7.

Elemental analysis CHN for C₁₉H₃₁Cl₂NO₅ (M_{mol} = 424.36 g mol⁻¹): calculated values (%): C = 53.78; H = 7.36; N = 3.30; measured values: C = 53.45; H = 7.11; N = 3.00.

hexadecyl[2-(2-hydroxyethoxy)ethyl]dimethylammonium 2-(2,4-dichlorophenoxy)propionate (7):

¹H NMR (CD₃OD) δ [ppm] = 0.89 (t, J = 6.92 Hz, 3H), 1.31 (s, 26H), 1.60 (d, J = 6.86 Hz, 3H), 1.76 (m, 2H), 3.14 (s, 6H), 3.35 (m, 2H), 3.58 (m, 4H), 3.68 (m, 2H), 3.89 (s, 2H), 4.46 (q, J = 6.86 Hz, 1H), 6.89 (d, J = 8.97 Hz, 1H), 7.16 (dd, J_{1,2} = 8.86 Hz, J_{1,3} = 2.57 Hz, 1H), 7.36 (d, J = 2.58 Hz, 1H); ¹³C NMR (CD₃OD) δ [ppm] = 178.9; 154.7; 130.6; 128.7; 126.1; 124.6; 116.4; 77.9; 73.8; 66.6; 65.8; 64.4; 61.7; 52.1; 33.0; 30.6; 27.3; 23.9; 23.6; 19.6; 14.5.

Elemental analysis CHN for C₃₁H₅₅Cl₂NO₅ (M_{mol} = 592.68 g mol⁻¹): calculated values (%): C = 62.82; H = 9.35; N = 2.36; measured values: C = 62.52; H = 9.18; N = 2.01.

Product analysis:

Density. Density measurements of the ionic liquids synthesised were conducted using oscillation-type automatic densimeter DDM291 I (Rudolph Research Analytical) with temperature adjustment using the Peltier module (0–80°C). The accuracy of temperature stabilisation is 0.02°C, whereas the measurement accuracy according to the instrument specification is at a level of 0.00002 g cm⁻³. Each measurement was performed three times and the final result was the arithmetic average of the values obtained.

Viscosity. Dynamic viscosity of the ionic liquids obtained was measured using rotary cone-plate viscometer Rheotec RC30-CPS (C50-2, for which the range of measured viscosity is 0.005 to 5000 Pa·s), with an ability to adjust temperature using the Peltier module (0–120°C). Ionic liquid samples of approx., 1.5 cm³ volume were tested for viscosity at a temperature range of 20 to 80°C. The measurement error was determined to be below 10⁻⁴ Pa·s.

Refractive index. The refractive index of the synthesis compounds was measured using automatic refractometer J357 (Rudolph Research Analytical) with an electronic temperature control in a range of 15 to 100°C. The accuracy of temperature stabilisation is 0.02°C, whereas the measurement accuracy according to the instrument specification is at a level of 0.00005. The measurements for the compounds analysed were performed at a temperature range of 20 to 80°C. Each measurement was performed three times and the final result was the arithmetic average of the values obtained.

Herbicidal activity – greenhouse experiment. The research was conducted at the Institute of Plant Protection – National Research Institute in Poznań. The efficacy of the herbicidal ionic liquid was determined for the selected test plants: the lamb's quarter (*Chenopodium album* L.) and cornflower (*Centaurea cyanus* L.). The experiment was conducted according to the methods previously described in [5].

The studies funded from statutory 03/32/DSMK/0612 awarded under the grant of the Ministry of Science and Higher Education

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