

# Novel Benzothiazole Ionic Liquids as Catalysts for the Synthesis of Parabens

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**Abstract**—A simple and green approach to the esterification of *p*-hydroxybenzoic acid and aliphatic alcohols to obtain parabens was developed. First, two novel benzothiazole ionic liquids [HBth]H<sub>2</sub>PW<sub>12</sub>O<sub>40</sub> (IL1) and [HBth]-H<sub>4</sub>PMo<sub>12</sub>O<sub>41</sub> (IL2) were synthesized with benzothiazole and heteropolyacids as starting materials. The synthesized ionic liquids were characterized by FTIR spectroscopy, TGA, PXRD analysis, and SEM. The application of IL1 and IL2 as catalysts for the synthesis of parabens was explored. The results showed that the ILs had a high catalytic activity in the synthesis of parabens, and, at the same time, they could be easily recovered and reused five times without loss of activity.

**Keywords:** green chemistry, parabens, benzothiazole, ionic liquids, heteropoly acids, esterification, catalysis

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## INTRODUCTION

Parabens are internationally recognized broad-spectrum high-efficiency food preservatives and bactericide agents [1]. In the United States, Europe, Japan, Canada, South Korea, Russia, and other countries, parabens are allowed to be used in food and widely in soy sauce, vinegar, and other condiments, preserved products, baked goods, sauces, beverages, yellow wine, fruits, and vegetables [2, 3]. Moreover, parabens are also widely used in medicine and cosmetics [4]. The typical synthetic approach to parabens involves catalysis with conc. H<sub>2</sub>SO<sub>4</sub>. This method has some disadvantages, including long reaction time and using of a concentrated acid, which corrodes and produces corrosive waste water, thereby harming the environment [5]. Therefore, more efficient and environmentally friendly catalysts have been explored in recent years, such as biological enzymes, sulfamic acid [6, 7], chitosan sulfate [8], vitamin C [9], and [Hmim]<sup>+</sup>BF<sub>4</sub><sup>-</sup> [10], as well as ultrasound-assisted synthetic approaches have been tested [11–13].

Ionic liquids (ILs), one of the most important branches of green chemistry, are used as excellent solvents and efficient and reusable catalysts in organic synthesis.

Over the past years, our group has explored efficient, green, and low-cost catalysts for esterification [6–11]. In continuation to our work on catalytic esterification [14–16], in the present work we have developed an efficient and environmentally friendly procedure for the IL-catalyzed synthesis of paraben compounds.

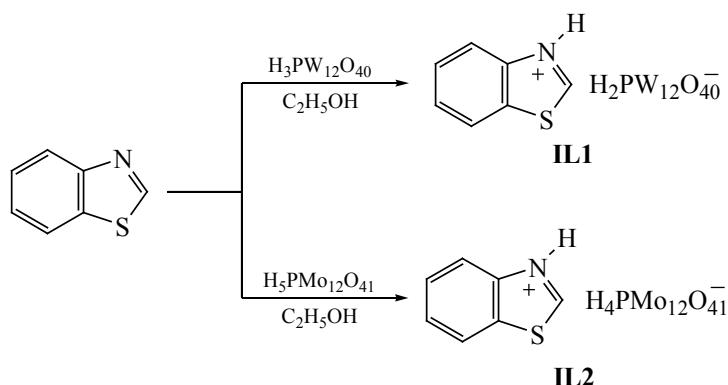
## RESULTS AND DISCUSSION

First, we designed and synthesized two heteropolyacid–benzothiazole ionic liquids (ILs): [HBth]H<sub>2</sub>PW<sub>12</sub>O<sub>40</sub> (**IL1**) and [HBth]H<sub>4</sub>PMoO<sub>41</sub> (**IL2**) (Scheme 1).

The novel ionic liquids **IL1** and **IL2** were prepared by the reaction of benzothiazole with heteropoly acids H<sub>3</sub>PW<sub>12</sub>O<sub>40</sub> and H<sub>5</sub>PMo<sub>12</sub>O<sub>41</sub> and characterized by IR spectroscopy. Figures 1 and 2 present the FTIR spectra of **IL1** and **IL2**, respectively.

As seen in Figs. 1 and 2, the spectra contain NH absorption bands near 3510 cm<sup>-1</sup>, implying that benzothiazole has reacted with HPAs. The band at 1698 cm<sup>-1</sup> was assignable to C=N stretching vibrations. The absorption peaks of the benzene ring were observed at about 3050 and 1430 cm<sup>-1</sup>, and the absorption peak of the C–S bond was at about 1260 cm<sup>-1</sup>. The

Scheme 1.



1,2-disubstituted of benzene ring appears at about  $800\text{ cm}^{-1}$ . All the above information provided evidence for the preparation of benzothiazole ionic liquids **IL1** and **IL2**.

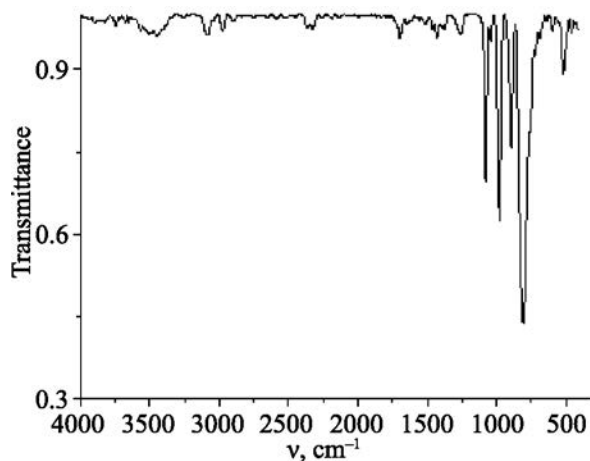
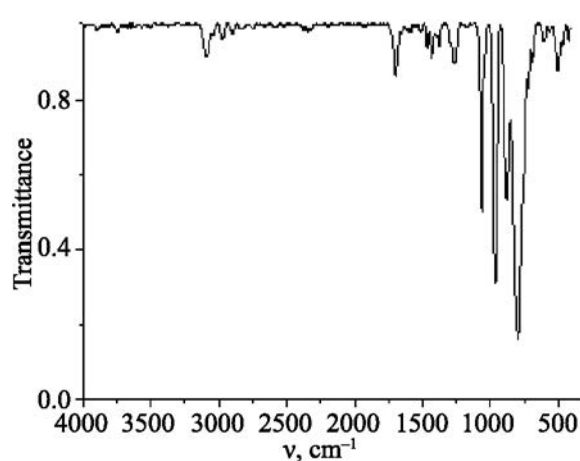
The thermal stability of **IL1** and **IL2** were evaluated by TGA analysis. Figures 3 and 4 show the TGA curves of **IL1** and **IL2**, respectively.

As seen from Figs. 3 and 4, the TGA curves showed three weight loss peaks. The first was observed at  $100\text{--}120^\circ\text{C}$  and was assignable to a loss of  $\text{H}_2\text{O}$ . The second peak at  $310\text{--}330^\circ\text{C}$  for **IL1** and at  $280\text{--}300^\circ\text{C}$  for **IL2** was associated with the degradation of the benzothiazole ring. The third peak at  $400\text{--}450^\circ\text{C}$  for **IL1** and at  $540\text{--}560^\circ\text{C}$  for **IL2** were assigned to the degradation of the Keggin core in the HPAs. Comparing the TGA curves of the two ionic liquids we can see that the second and third weight losses in **IL2** occurred at higher temperatures than in **IL1**. The possible reason is that the P–Mo–O entity in the Keggin frame of **IL2** is thermally more stable than in **IL1**.

We also obtained powder XRD patterns of **IL1** and **IL2** (see Figs. 5 and 6, respectively).

The XRD powder patterns show characteristic peaks of **IL1** and **IL2** ( $2\theta\ 6^\circ\text{--}9^\circ$ ) and benzothiazole ( $2\theta\ 25^\circ\text{--}33^\circ$ ). From the intensity of the characteristic peak we can conclude that crystallinity of **IL2** was higher compared to **IL1** [17].

The activity of the synthesized ionic liquids **IL1** and **IL2** as esterification catalysts was studied by the example of the synthesis of parabens. The synthesis of methyl paraben catalyzed by **IL1** was selected as a model reaction to optimize the reaction conditions. The effects of the catalyst amount and the reaction temperature and time were assessed. Methanol forms an azeotropic mixture with water, and, therefore, refluxing was chosen as the optimal temperature to achieve dehydration and improve the yield of the ester. The resulting data are shown in Table 1. As seen from Table 1, the yields of methyl paraben increased at **IL1** concentrations higher than 10 mol % (Table 1, entries 4–6). However, from the

Fig. 1. FTIR spectrum of **IL1**.Fig. 2. FTIR spectrum of **IL2**.

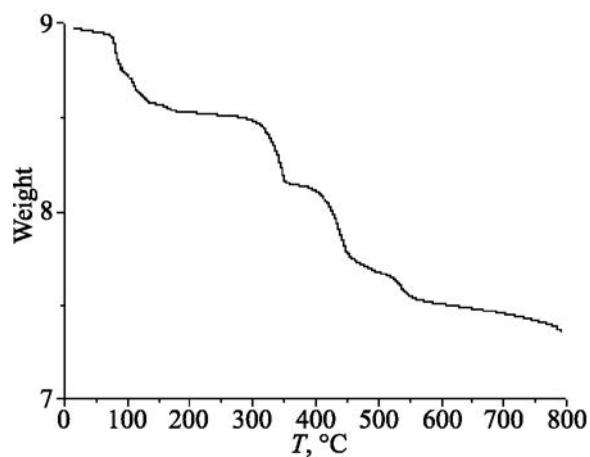


Fig. 3. TGA curve of IL1.

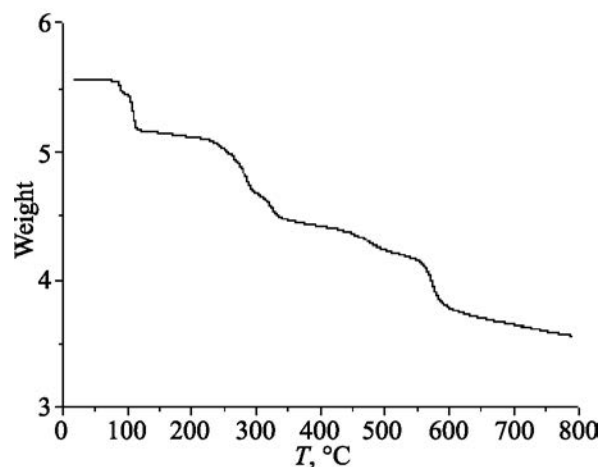


Fig. 4. TGA curve of IL2.

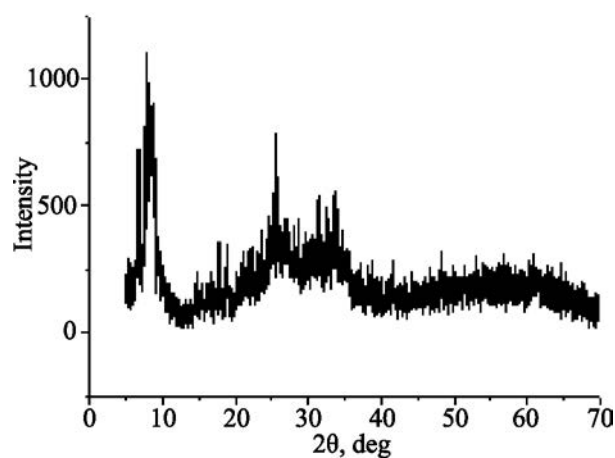


Fig. 5. Powder XRD pattern of IL1.

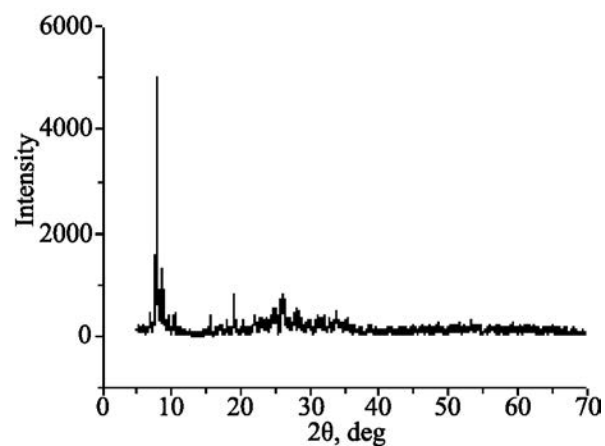
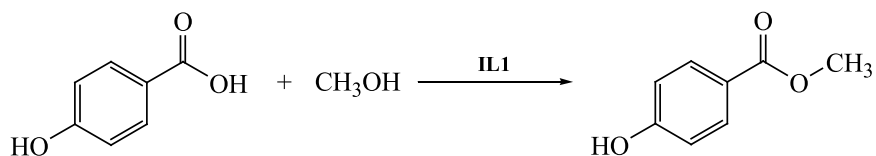


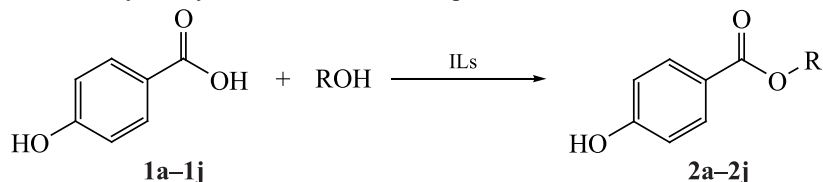
Fig. 6. Powder XRD pattern of IL2.

Table 1. Optimization of the reaction conditions in the model reaction<sup>a</sup>

Entry no.	IL1, mol %	Time, h	Yield of methyl paraben, % <sup>b</sup>
1	0	8	31.1
2	1	8	35.6
3	5	6	65.5
4	10	4	93.2
5	15	6	94.0
6	20	6	94.1

<sup>a</sup> *p*-Hydroxybenzoic acid 0.1 mol, methanol 0.4 mol, reflux.

<sup>b</sup> The yields refer to the pure product after recrystallization.

**Table 2.** Synthesis of parabens catalyzed by benzothiazole ionic liquids<sup>a</sup>

Entry no.	R	Product no.	Temperature, °C	Time, h	Yield, % <sup>b</sup>		mp, °C
					IL1	IL2	
1	CH <sub>3</sub>	<b>2a</b>	65	4	93.2	91.6	126–128 (126–127 [21])
2	C <sub>2</sub> H <sub>5</sub>	<b>2b</b>	78	4	89.5	89.6	116–118 (116–118 [22])
3	<i>i</i> -C <sub>3</sub> H <sub>7</sub>	<b>2c</b>	83	4	88.9	88.3	85–87 (84–86 [23])
4	C <sub>4</sub> H <sub>9</sub>	<b>2d</b>	118	5	88.8	87.3	66–67 (68–69 [24])
5	<i>i</i> -C <sub>4</sub> H <sub>9</sub>	<b>2e</b>	108	5	87.6	86.4	74–77 (76–78)[25]
6	<i>i</i> -C <sub>5</sub> H <sub>11</sub>	<b>2f</b>	133	6	85.1	84.9	58–60 (58–59 [26])
7	<i>i</i> -C <sub>8</sub> H <sub>17</sub>	<b>2g</b>	183	6	80.1	79.5	38–42 (novel compound)
8	C <sub>10</sub> H <sub>21</sub>	<b>2h</b>	233	7	79.8	79.2	31–33 (30–34 [27])
9	C <sub>12</sub> H <sub>25</sub>	<b>2i</b>	105	8	77.9	77.6	39–41 (36–38 [15])
10	C <sub>16</sub> H <sub>33</sub>	<b>2j</b>	105	8	76.9	76.8	61–63 (64–65 [28])

<sup>a</sup> Reaction conditions: *p*-hydroxybenzoic acid 0.1 mol, alcohol 0.4 mol, IL 10 mol %.

<sup>b</sup> The yields refer to the pure products after recrystallization.

perspective of economic benefits, we chose as optimal reaction conditions 10% mol of **IL1** under reflux for 4 h (Table 1, entry 4).

Under the optimal reaction conditions we synthesized ten paraben compounds **2a–2j** by the reaction of *p*-methylbenzoic acid and different alcohols. The results are shown in Table 2.

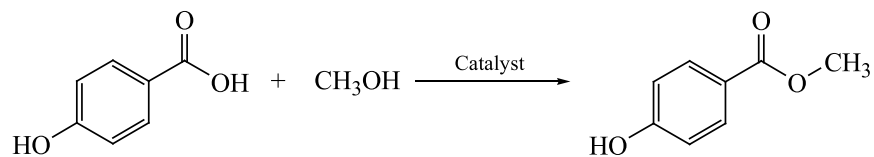
As seen from Table 2, ionic liquid **IL1** and **IL2** exhibited a good catalytic effect in the synthesis of parabens. This results can be explained not only by a high acidity of **IL1** and **IL2** [18], but also by their ability, due to the Keggin core, to easily adsorb reactants in the solid bulk and thus make the whole reaction system a “pseudoliquid phase”. In addition, ILs worked as phase-transfer catalysts, thereby increasing the yield of esterification [19, 20].

To substantiate the important role of ionic liquids **IL1** and **IL2** in the studied esterification, we employed other catalysts (conc. H<sub>2</sub>SO<sub>4</sub>, chitosan sulfate, ascorbic acid, and sulfamic acid) to synthesize methyl paraben and compared their activity with the activity of the ionic liquids. The results were shown in Table 3. As seen from

Table 3, **IL1** and **IL2** proved to be the most efficient catalysts.

The reusability of the catalysts was assessed in the model synthesis of methyl paraben under the optimal reaction conditions (*p*-hydroxybenzoic acid 0.1 mol, methanol 0.4 mol, **IL1** or **IL2** 10 mol %, reflux 4 h). The ILs were recovered by filtration and used in the next reaction after drying. The results after 5 catalytic cycles are shown in Table 4. As seen from the table, the yield of methyl-paraben only slightly decreased with increasing recycle number of ILs. It therefore was concluded that the two synthesized ILs catalysts have high recyclability.

The structure of the catalysts in the course of the reusability experiments was monitored by SEM (Figs. 7 and 8). As seen from the figures, the surface of fresh ionic liquids (Figs. 7a and 8a) was uneven, so they had higher catalytic activities. After three catalytic cycles, the surface changed only slightly (Figs. 7d and 8d), but they still preserved their activity. However, after the fourth run (Figs. 7e and 8e), the surface underwent stronger changes, which slightly decreased the catalytic activity.

**Table 3.** Comparison of catalyst activities in the synthesis of methyl paraben<sup>a</sup>

Entry no.	Catalyst, mol %	Time, h	Yield of methyl paraben, % <sup>b</sup>
1	<b>IL1</b> (10)	4	93.2
2	<b>IL2</b> (10)	4	91.6
3	Sulfuric acid (50)	4	80.4
4	Chitosan sulfate (40)	8	80.8
5	Ascorbic acid(40)	6	82.3
6	Sulfamic acid(30)	6	82.9

<sup>a</sup> Reaction conditions: *p*-hydroxybenzoic acid 0.1 mol, methanol 0.4 mol, reflux.

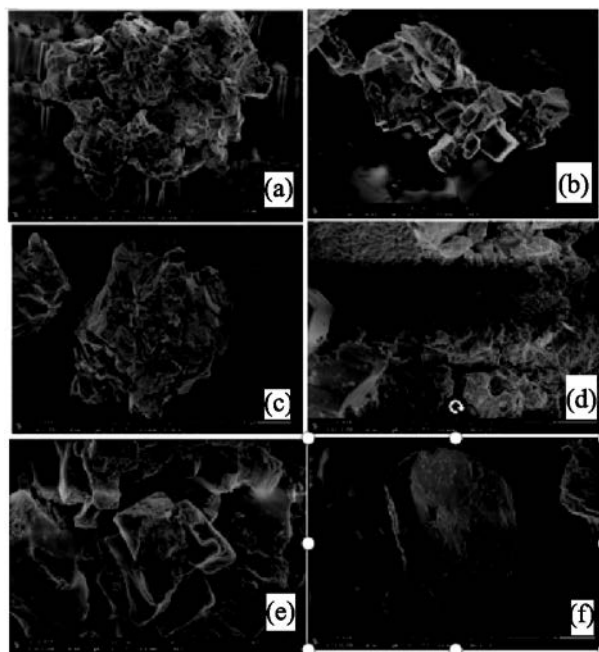
<sup>b</sup> The yields refer to the pure product after recrystallization.

**Table 4.** Reusability of **IL1** and **IL2** in the synthesis of methyl paraben<sup>a</sup>

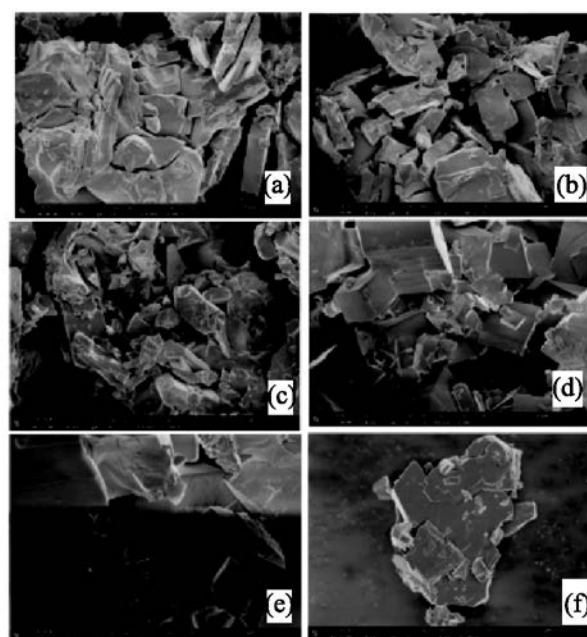
Number of cycle		Fresh	1	2	3	4	5
Yield, % <sup>b</sup>	<b>IL1</b>	93.2	89.9	88.6	87.4	83.6	80.3
	<b>IL2</b>	91.6	89.4	88.0	87.4	86.7	85.7

<sup>a</sup> Reaction conditions: *p*-hydroxybenzoic acid 0.1 mol, methanol 0.4 mol, **IL** 10 mol %, reflux 4 h.

<sup>b</sup> The yields refer to the pure product after recrystallization.



**Fig. 7.** SEM images of **IL1** of (a) before use and after (b) 1, (c) 2, (d) 3, (e) 4, and (f) 5 catalytic cycles.



**Fig. 8.** SEM images of **IL2** of (a) before use and after (b) 1, (c) 2, (d) 3, (e) 4, and (f) 5 catalytic cycles.



## EXPERIMENTAL

**Materials and methods.** All chemicals were of analytical grade and used without further purification. The melting points were determined in an open glass capillary and are uncorrected. The IR spectra (400–4000  $\text{cm}^{-1}$ ) were recorded on a Bruker Vector-22 FTIR spectrometer for samples prepared as KBr pellets. The powder XRD patterns were obtained on a Bruker D8 Quest diffractometer. The TGA analysis was performed on a TA Discovery instrument in flowing  $\text{N}_2$  with a heating rate of 10 min. The SEM images were obtained on a FEI Philips Verios 460 microscope. The C, H, O analyses were performed with a Fisons EA 1108 (CHNSO) elemental analyzer.

**Synthesis of ionic liquids IL1 and IL2 (general procedure).** A solution of heteropolyacid ( $\text{H}_3\text{PW}_{12}\text{O}_{40}$  or  $\text{H}_3\text{PMo}_{12}\text{O}_{41}$ ) (1 mmol) in ethanol (20 mL) was cooled to 0–5°C, and then a solution of benzothiazole (1 mmol) in ethanol (5 mL) was added dropwise, the mixture was stirred at room temperature until completion of the reaction (by TLC), filtered. The solid was washed with ethyl acetate and dried to obtain the target product.

**[HBth] $\text{H}_2\text{PW}_{12}\text{O}_{40}$  (IL1).** Yellow powder, yield 70.4%, mp > 250°C. IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3516  $\nu(\text{N-H})$ , 3086  $\nu(\text{C-H})$ , 1698  $\nu(\text{C=N})$ , 1430  $\nu(\text{aromatic C-H})$ , 1216  $\nu(\text{C-S})$ , 795  $\gamma(1,2\text{-Ph-H})$ .

**[HBth] $\text{H}_4\text{PMoO}_{41}$  (IL2).** White powder, yield 71.2%, mp > 250°C. IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3502  $\nu(\text{N-H})$ , 3065  $\nu(\text{C-H})$ , 1697  $\nu(\text{C=N})$ , 1431  $\nu(\text{aromatic C-H})$ , 1262  $\nu(\text{C-S})$ , 809  $\gamma(1,2\text{-Ph-H})$ .

**Synthesis of paraben compounds 2a–2h (general procedure).** A mixture of *p*-hydroxybenzoic acid (10 mmol), alcohol **1a–1g**, and **IL1** or **IL2** was refluxed until completion of the reaction (by TLC). The alcohol was evaporated, and the residue was extracted with ethyl acetate (3 × 10 mL). The organic phases were combined, dried, and concentrated. The precipitate was recrystallized from methanol to obtain the target product.

**Methyl paraben (2a).** White solid, mp 126–128°C (126–127°C [21]). IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3292  $\nu(\text{O-H})$ , 3037  $\nu(\text{Ph-H})$ , 2958  $\nu(\text{C-H})$ , 1679  $\nu(\text{C=O})$ , 1593, 1514, 1438  $\nu(\text{C-H}_{\text{arom}})$ , 1278  $\nu(\text{C-O-C})$ , 850  $\gamma(1,4\text{-Ph-H})$ .

**Ethyl paraben (2b).** White solid, mp 116–118°C (116–118°C [22]). IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3212

$\nu(\text{O-H})$ , 2977  $\nu(\text{C-H})$ , 1680  $\nu(\text{C=O})$ , 1605, 1445  $\nu(\text{C-H}_{\text{arom}})$ , 1295  $\nu(\text{C-O-C})$ , 844  $\gamma(1,4\text{-Ph-H})$ .

**Isopropyl paraben (2c).** White solid, mp 85–87°C (84–86°C [23]). IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3400  $\nu(\text{O-H})$ , 2987  $\nu(\text{C-H})$ , 1690  $\nu(\text{C=O})$ , 1587, 1502, 1446  $\nu(\text{C-H}_{\text{arom}})$ , 1285  $\nu(\text{C-O-C})$ , 853  $\gamma(1,4\text{-Ph-H})$ .

**Butyl paraben (2d).** White solid, mp 66–67°C (68–69°C [24]). IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3381  $\nu(\text{O-H})$ , 2949, 2975  $\nu(\text{C-H})$ , 1681  $\nu(\text{C=O})$ , 1587, 1502, 1454  $\nu(\text{C-H}_{\text{arom}})$ , 1285  $\nu(\text{C-O-C})$ , 844  $\gamma(1,4\text{-Ph-H})$ .

**Isobutyl paraben (2e).** White solid, mp 74–77°C (76–78°C [25]). IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3241  $\nu(\text{O-H})$ , 2959, 2884  $\nu(\text{C-H})$ , 1680  $\nu(\text{C=O})$ , 1596, 1511, 1454  $\nu(\text{C-H}_{\text{arom}})$ , 1285  $\nu(\text{C-O-C})$ , 853  $\gamma(1,4\text{-Ph-H})$ .

**Isopentyl paraben (2f).** White solid, mp 58–60°C (58–59°C [26]). IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3260  $\nu(\text{O-H})$ , 2950  $\nu(\text{C-H})$ , 1680  $\nu(\text{C=O})$ , 1596, 1511, 1454  $\nu(\text{C-H}_{\text{arom}})$ , 1285  $\nu(\text{C-O-C})$ , 853  $\gamma(1,4\text{-Ph-H})$ .

**Isooctyl paraben (2g).** White solid, mp 38–42°C. IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3485  $\nu(\text{O-H})$ , 2958  $\nu(\text{C-H})$ , 1718  $\nu(\text{C=O})$ , 1633, 1554, 1489  $\nu(\text{C-H}_{\text{arom}})$ , 1239  $\nu(\text{C-O-C})$ , 834  $\gamma(1,4\text{-Ph-H})$ . Found, %: C 71.95; H 8.90; O 19.21.  $\text{C}_{15}\text{H}_{22}\text{O}_3$ . Calculated, %: C 71.97; H 8.86; O 19.17.

**Decyl paraben (2h).** White solid, mp 31–33°C (30–34°C [27]). IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3485  $\nu(\text{O-H})$ , 2940  $\nu(\text{C-H})$ , 1718  $\nu(\text{C=O})$ , 1587, 1445  $\nu(\text{C-H}_{\text{arom}})$ , 1267  $\nu(\text{C-O-C})$ , 853  $\gamma(1,4\text{-Ph-H})$ .

**Synthesis of dodecyl paraben (2i) and hexadecyl paraben 2j (general procedure).** *p*-Hydroxybenzoic acid (0.1 mol), toluene (10 mL) and **IL1** or **IL2** were added in succession to alcohol dodecyl or hexadecyl (0.11 mol). The mixture was refluxed until completion of the reaction (by TLC). The solvent was evaporated, and the residue was extracted with ethyl acetate (3 × 10 mL). The organic phases were combined, dried, and concentrated. The precipitate was recrystallized from methanol to obtain the target product.

**Dodecyl paraben (2i).** White solid, mp 39–41°C (36–38°C [15]). IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3390  $\nu(\text{O-H})$ , 2945, 2911  $\nu(\text{C-H})$ , 1688  $\nu(\text{C=O})$ , 1595, 1511, 1465  $\nu(\text{C-H}_{\text{arom}})$ , 1275  $\nu(\text{C-O-C})$ , 852  $\gamma(1,4\text{-Ph-H})$ .

**Hexadecyl paraben (2j).** White solid, mp 61–63°C (64–65°C [28]). IR spectrum (KBr),  $\nu$ ,  $\text{cm}^{-1}$ : 3391  $\nu(\text{O-H})$ , 2921  $\nu(\text{C-H})$ , 1690  $\nu(\text{C=O})$ , 1596, 1511, 1465  $\nu(\text{C-H}_{\text{arom}})$ , 1261  $\nu(\text{C-O-C})$ , 862  $\gamma(1,4\text{-Ph-H})$ .

## CONCLUSIONS

In summary, we have developed a practical, useful and green method for synthesis of parabens using the ionic liquids [HBth]H<sub>2</sub>PW<sub>12</sub>O<sub>40</sub> and [HBth]H<sub>2</sub>PMo<sub>12</sub>O<sub>40</sub> as catalysts. The catalysts could be reused 5 times without significant loss of activity. Ionic liquids showed the best activities compared with the reported catalysts. In future experiments, we plan to test ionic liquids in other esterifications, in view of their high efficiency, reusability, economy, and environmental friendliness. Additionally, studies to extend the substrate scope are in progress at our laboratory.

## ACKNOWLEDGMENTS

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## CONFLICT OF INTEREST

The authors declare no conflict of interest.

## REFERENCES

- Darbre, P.D., Aljarrah, A., Miller, W.R., Coldham N.G., Sauer, M.J., and Pope, G.S., *J. Appl. Toxicol.*, 2004, vol. 24, p. 5.  
<https://doi.org/10.1002/jat.958>
- Soni, M.G., Burdock, G.A., Taylor, S.L., and Greenberg, N.A., *Food Chem. Toxicol.*, 2001, vol. 39, p. 513.  
[https://doi.org/10.1016/s0278-6915\(00\)00162-9](https://doi.org/10.1016/s0278-6915(00)00162-9)
- Sonia, M.G., Taylor, S.L., Greenberg, N.A., and Burdock, G.A., *Food Chem. Toxicol.*, 2002, vol. 40, p. 1335.  
[https://doi.org/10.1016/s0278-6915\(02\)00107-2](https://doi.org/10.1016/s0278-6915(02)00107-2)
- Pugazhendhi, D., Pope, G.S., and Darbre, P.D., *J. Appl. Toxicol.*, 2005, vol. 25, p. 301.  
<https://doi.org/10.1002/jat.1066>
- You, Z.-X., Li, C.-H., and Guo, H.-Y., *Chin. J. Synth. Chem.*, 2019, vol. 27, p. 154.  
<https://doi.org/10.15952/j.cnki.cjsc.1005-1511.2019.02.18098>
- Liu, Y.-T., Wang, J.-Y., Yin, D.-W., Liu, B., and Fu, Q., *J. Shaanxi Univ. Sci. Techn.*, 2014, vol. 32, p. 62.  
<https://doi.org/10.3969/j.issn.1000-5811.2014.03.013>
- Lv, B., Zhang, X.-L., Liu, Y.-T., and Yin, D.-W., *China Condiment*, 2016, vol. 41, p. 49.  
<https://doi.org/10.3969/j.issn.1000-9973.2016.01.011>
- Yin, D.-W., Miao, D.-L., and Liu, Y.-T., *Spec. Petrochem.*, 2010, vol. 27, p. 49.  
<https://doi.org/10.3969/j.issn.1003-9384.2010.06.013>
- Liu, Y.-T., Liu, B.-B., and Yin, D.-W., *China Condiment*, 2014, vol. 39, p. 95.  
<https://doi.org/10.3969/j.issn.1000-9973.2014.07.023>
- Liu, Y.-T., Zhang, X.-L., Yin, D.-W., and Yang, X.-G., *China Food Addit.*, 2014, vol. 7, p. 63.  
<https://doi.org/10.3969/j.issn.1006-2513.2014.07.001>
- Liu, Y.-T., Zhou, Y., Yin, D.-W., and Yan, H.-X., *Chin. Chem. World*, 2010, vol. 51, p. 505.  
<https://doi.org/10.19500/j.cnki.0367-6358.2010.08.018>
- Khan, N.R. and Rathod, V.K., *Process Biochem.*, 2018, vol. 75, p. 89.  
<https://doi.org/10.1016/j.procbio.2018.08.019>
- Raimondi, M.V., Listro, R., Cusimano, M.G., La Franca, M., Faddetta, T., Gallo, G., Schillaci, D., Collina, S., Leonchiks, A., and Barone, G., *Bioorg. Med. Chem.*, 2019, vol. 27, p. 721.  
<https://doi.org/10.1016/j.bmc.2019.01.010>
- Yin, D.-W., Liu, Y.-T., and Liang, G.-T., *China Condiment*, 2013, vol. 38, p. 46.  
<https://doi.org/10.3969/j.issn.1000-9973.2013.06.011>
- Liu, Y.-T., Wang, Y.-W., Zhang, X.-L., and Yin, D.-W., *China Condiment*, 2015, vol. 40, p. 93.  
<https://doi.org/10.3969/j.issn.1000-9973.2015.01.023>
- Liu, Y.-T., Liu, K., Yin, D.-W., Dang, Y., and Yang, L., *China Condiment*, 2019, vol. 44, p. 40.  
<https://doi.org/10.3969/j.issn.1000-9973.2019.04.009>
- Zhang, X.-J., Chen, M.-C., Feng, J.-C., Niu, Y.-H., Li, Q.-M., and Jia, D.-M., *Polym. Mater. Sci. Eng.*, 2004, vol. 20, p. 64.  
<https://doi.org/10.3321/j.issn:1000-7555.2004.03.017>
- Fang, C.-J., Li, Y., Zhao, W.-F., Wu, W.-B., Li, H., He, C., and Yang, S., *RSC Adv.*, 2018, vol. 8, p. 16585.  
<https://doi.org/10.1039/C8RA02278J>
- Leng, H., Wang, J., Zhu, D.-R., Shen, L., Zhao, P.-P., and Zhang, M.-J., *Chem. Eng. J.*, 2011, vol. 173, p. 620.  
<https://doi.org/10.1016/j.cej.2011.08.013>
- Zhou, Y., Guo, Z., Hou, W., Wang, Q., and Wang, J., *Catal. Sci. Technol.*, 2015, vol. 46, p. 4324.  
<https://doi.org/10.1002/chin.201546272>
- Chen, W.-Y., Guan, L.-T., and Xu, L.-Z., *J. Agric.*, 2013, vol. 3, p. 34.  
<https://doi.org/10.3969/j.issn.1007-7774.2013.10.009>
- Shu, H., Tang, J., Zhou, Z.-Y., and Wu, S.-Z., *Food Sci. Technol.*, 2013, vol. 38, p. 274.

23. Xie, Z.-B., Jiang, G.-F., Liang, X.-Z., and Wang, L., *China Food Addit.*, 2005, vol. 31, p. 6.  
<https://doi.org/10.3969/j.issn.1006-2513.2005.05.002>
24. Wang, S.-P., Wang, Y.-F., Wang, H.-N., Zhao, G.-X., and Li, J.-J., *China Condiment*, 2017, vol. 42, p. 80.  
<https://doi.org/10.3969/j.issn.1000-9973.2017.09.019>
25. Li, Y.-F., Yang, X.-P., He, X.-G., Xie, S.-G., and Li, Z., *Farm Mach.*, 2013, vol. 9, p. 70.
26. Liu, X.-L. and Zeng, Y.-C., *China Surfactant Deterg. Cosmet.*, 2010, vol. 40, p. 38.
27. Tang, Z., Jiang, Q.-T., Peng, L.-F., Xu, X.-H., Li, J., Qiu, R.-H., and Au, Ch.-T., *Green Chem.*, 2017, vol. 22, p. 5396.  
<https://doi.org/10.1039/c7gc02174g>
28. Liang, X.-W., Huang, C.-H., Ou, S.-Y., and Yan, R.-A., *Food Ferment. Ind.*, 2016, vol. 42, p. 53.  
<https://doi.org/10.13995/j.cnki.11-1802/ts.201611009>
29. Mamedbeili, E.G. (Mamedov), Kyazimova, T.G., Nagiev, Z.M., Abdiev, O.B., and Aliev, K.A., *Russ. J. Org. Chem.*, 2009, vol. 45, p. 74.  
<https://doi.org/10.1134/s107042800901010>
30. Kyazimova, T.G., Mamedov, E.G., Babaev, R.S., and Mamedova, I.M., *Russ. J. Org. Chem.*, 2008, vol. 81, p. 438.  
<https://doi.org/10.1134/S107042720803018X>
31. Mamedbeili, E.G., Kyazimova, T.G., Gasanov, Kh.I., Efendieva, K.M., and Rzabekova, N.N., *Russ. J. Org. Chem.*, 2010, vol. 43, p. 322.  
<https://doi.org/10.1134/S1070428010030036>
32. Sakhautdinov, I.M., Batyrshin, I.R., Sergeeva, N.A., Galin, F.Z., and Yunusov, M.S., *Russ. J. Org. Chem.*, 2012, vol. 48, p. 788.  
<https://doi.org/10.1134/S1070428012060073>
33. Dashko, L.V., Dmitriev, D.V., Pestov, S.M., and Flid, V.R., *Russ. J. Org. Chem.*, 2015, vol. 50, p. 1732.  
<https://doi.org/10.1134/s1070428014120021>
34. Kuznetsov, V.A., Pervova, M.G., and Pestov, A.V., *Russ. J. Org. Chem.*, 2013, vol. 49, p. 1859.  
<https://doi.org/10.1134/S1070428013120300>
35. Klushin, V.A., Galkin, K.I., Kashparova, V.P., Keivodaeva, E.A., Kravchenko, O.A., Smirnova, N.V., Chernyshev, V.M., and Ananikov, V.P., *Russ. J. Org. Chem.*, 2016, vol. 52, p. 767.  
<https://doi.org/10.1134/S1070428016060014>
36. Yarosh, N.O., Zhilitskaya, L.V., Shagun, L.G., Dorofeev, I.A., Larina, L.I., and Voronkov, M.G., *Russ. J. Org. Chem.*, 2013, vol. 49, p. 475.  
<https://doi.org/10.1134/S1070428013030287>