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# A microwave-promoted atom-efficient diesterification of aromatic carboxylate with 1,4-dibromobutane in water

Shenglong Ding<sup>a</sup>, Lin Bai<sup>b</sup>, Yong Cong<sup>a</sup>, and Ting Chen<sup>b</sup>

<sup>a</sup>College of Chemistry and Engineering, Lanzhou University, Lanzhou, P. R. China; <sup>b</sup>Institute of Green Chemistry Experiment and Teaching, Lanzhou City University, Lanzhou, 730070, P. R. China

#### ABSTRACT

A rapid and efficient diesterification reaction of aromatic carboxylate with 1,4-dibromobutane was carried out in water at 100 °C in the presence of base and phase transfer catalyst under focused microwave irradiation. This microwave-promoted protocol offers an atom-efficient and environment-friendly approach to synthesize butamethylenediesters in high yield.

#### **GRAPHICAL ABSTRACT**



Ar =  $-C_6H_5$ ,  $-C_6H_4Br$ ,  $-C_6H_4I$ ,  $-C_6H_4NO_2$ ,  $-C_6H_4CH_3$ ,  $-C_{10}H_7$ , *a*-furan, -CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub>, -CH<sub>2</sub>C<sub>10</sub>H<sub>7</sub>, -CH=CHC<sub>6</sub>H<sub>5</sub> ARTICLE HISTORY Received 26 March 2018

#### **KEYWORDS**

1,4-dibromobutane; diesterification; focused microwave irradiation; phase transfer catalysis; water solvent

#### Introduction

From the point of view of green chemistry's interest, attention has been focused on atom-economic reactions, environment-friendly solvents, and methodologies from both academic and chemical industry.<sup>[1]</sup>

Water is unanimously viewed as an excellent green solvent. In particular, its hydrophobic effect and hydrogen-bonding capability would lead to a higher product yield and a higher selectivity.<sup>[2]</sup> However, the less solubility of most organic substrates in water disfavors many reactions. It has been opined that phase transfer catalysts (PTC) under the microwave (MW) irradiation could be used to solve these problems.<sup>[3]</sup>

It has long been shown that MW irradiation has become an attractive technique for accelerating organic synthesis. Microwave offers several advantages like fast heating speed, high thermal efficiency, mild experimental conditions, simple operation, convenient control, environment protection, and low labor.<sup>[4]</sup>

The esterification reaction of carboxylic acid with alkyl halide is one of the most important and commonly employed reactions in organic and bioorganic

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CONTACT Shenglong Ding 🔯 disl@lzu.edu.cn 🗈 Department of Chemistry, Lanzhou University, P.O. Box: 730000, Lanzhou, P. R. China

synthesis.<sup>[5,6]</sup> Efforts have been made to replace the traditional synthesis of esters by using Fischer's method, catalyzed by a concentrated inorganic acid.<sup>[7]</sup> It has been well-known for the formation of carboxylates by the reaction of metal salts of carboxylic acids (RCO<sub>2</sub><sup>-</sup>M<sup>+</sup>, where M may be silver, lead, one of the alkali or alkaline earth metals or tertiary alkyl ammonium) and alkyl halides that the generally applicable synthetic procedure is unfavorable, due principally to poor yields, resulting from the dehydrohalogenation reactions. Although the procedure has been notably improved by alternatively using polar aprotic solvents such as DMF, HMPA, DMSO or phase transfer catalysts (PTC) of the quaternary alkyl ammonium,<sup>[5,6,8–10]</sup> these media offer some disadvantages like toxicity, harmfulness, costs, and hardness in the product isolation. In addition, ionic liquids have successfully been utilized as catalysts or solvents in the reaction of carboxylic acids with alkyl halides in high yield and higher selectivity.<sup>[10–16]</sup>

Methylene diesters of carboxylic acids and their derivatives are substantial products or intermediates in the chemical and pharmaceutical industries.<sup>[17-19]</sup> The most reported methods for synthesis of methylene diesters include the condensation of carboxylic acid with 1, *n*-diols,<sup>[20]</sup> or 1, *n*-dihaloalkanes,<sup>[16,21,22]</sup> acid chloride (acyl chloride) with 1,*n*-diols<sup>[23]</sup> and the transesterification.<sup>[24]</sup> Recently, an alternative procedure for it to carry out the reaction in ionic liquids<sup>[16]</sup> or with the assistance of MW heating was found.<sup>[23,25]</sup> Although methylene diesters are readily available, the main conventional synthetic methods have several drawbacks such as longer reaction time, low yields, the use of harmful solvents, and sensitive expensive catalysts, and a tedious post-treatment.

In our recent work, we used water as the solvent, by coupling with PTC under MW irradiation, in the esterification and etherification reaction of benzyl chloride and p-hydroxy-benzoic acid.<sup>[26]</sup> This successful water-PTC-MW combination encouraged us to develop a simple and novel method for the diesterification from carboxylic acids (or their salts) and dihaloalkane.

#### **Results and discussion**

The one-pot esterification of carboxylic acid with alkyl halide in water was carried out with two equivalents potassium salt of carboxylic acid in a focused MW synthesis system, by which the reaction temperature, pressure, MW power, and reaction time were easily controlled with a better reproducibility.

As a starting point, the diesterification was initially done using potassium benzoate and 1,4-dibromobutane for optimizing its reaction conditions. A variety of bases were applied. The MW irradiation temperature, time, the kind of base, and its amount were investigated. Results are summarized in Table 1.

Table 1 indicates that the effect of bases on the reaction was significant (Table 1, Entries 1–6). Desired diesterification products were not obtained in the absence of base. By comparison,  $K_2CO_3$ ,  $K_3PO_4$ , and  $Na_2CO_3$  were more effective. Owing to its economic and environment-friendly advantages,  $K_2CO_3$  was selected as the base. It was found that the amount of  $K_2CO_3$  was still important (Table 1, Entries 3, 7–10). 1 mmol of  $K_2CO_3$  in 1 mL water worked most efficiently. The strength and concentration of alkali also play a very important role. Weak alkalis were beneficial in proceeding

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Entry	Base (mmol)	Time (min)	Temp (°C)	Yield (%) <sup>b</sup>		
1	None	30	100	Trace		
2	KOH (1 mmol)	30	100	57		
3	$K_2CO_3$ (1mmol)	30	100	92		
4	$K_3PO_4$ (1 mmol)	30	100	89		
5	KF (1 mmol)	30	100	85		
6	Na <sub>2</sub> CO <sub>3</sub> (1mmol)	30	100	90		
7	K <sub>2</sub> CO <sub>3</sub> (0.5 mmol)	30	100	84		
8	K <sub>2</sub> CO <sub>3</sub> (0.75 mmol)	30	100	88		
9	K <sub>2</sub> CO <sub>3</sub> (1.25 mmol)	30	100	90		
10	K <sub>2</sub> CO <sub>3</sub> (1.5 mmol)	30	100	87		
11	$K_2CO_3$ (1 mmol)	30	80	86		
12	$K_2CO_3$ (1 mmol)	30	90	91		
13	$K_2CO_3$ (1 mmol)	30	110	86		
14	$K_2CO_3$ (1 mmol)	30	120	82		
15	$K_2CO_3$ (1 mmol)	25	100	64		
16	$K_2CO_3$ (1 mmol)	28	100	81		
17	$K_2CO_3$ (1 mmol)	32	100	83		
18	$K_2CO_3$ (1 mmol)	35	100	67		

Table 1. Optimization of reaction conditions of the diesterification of potassium benzoate with 1,4-dibromobutane<sup>a</sup>.

<sup>a</sup>Reaction conditions: potassium benzoate (5 mmol),1,4-dibromobutane (2.5 mmol), TBTA (1 mmol), water (1 mL). MW power: 10 W.

<sup>b</sup>Average isolated yield from twice reactions.

forward reactions, but the strong alkali such as KOH led to a dramatic decrease in the yield of diesterification. The products were hydrolyzed.

The effects of temperature and time were then studied on the yield of diesterification. The results are summarized in Table 1 (Entries 3, 11–18). The highest yield of 92% can be obtained at  $100^{\circ}$ C after 30 min MW irradiation.

Instead of MW, comparative reactions were carried out in a 50 mL three-necked flask with a reflux condenser and a thermometer in an oil bath. Other reaction conditions were kept as identical as those indicated in Entry 3 (Table 1). The mixture was refluxed at boiling temperature and held for 5 h under vigorous stirring. An isolated yield of butane-1,4-diyl dibenzoate of 90% was obtained. Obviously, the MW irradiation dramatically shortened the reaction time from 5 h to 30 min.

As it stands, the solubility of organic substrates in water could be enhanced with the aid of a PTC. Quaternary ammonium salts such as tetrabutylammonium chloride (TBAC), tetrabutylammonium bromide (TBAB), trimethylbenzylammonium chloride (TMBAC), hexadecyltrimethylammonium bromide (HDTMA), and polyethylene glycol (PEG-400) were applied herein as PTCs in the diesterification of potassium benzoate with 1,4-dibromomethane. The effect of PTCs on the reaction is listed in Table 2. The reaction proceeded very slowly or not at all in the absence of PTC.<sup>[26]</sup>

Table 2 indicates that quaternary ammonium salts are much better than PEGs. It may be attributed to the easy hydrolyzation of PEGs under alkaline conditions. Among the quaternary ammonium salts, TBAB gave the higher yield. Aromatic carboxylates can easily be combined with quaternary ammonium salts. Otherwise, bromide anion of TBAB is a better leaving group, compared with chloride anion of TBAC.

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Table 2. Optimization of PTC of the diesterification of potassium benzoate with 1,4-dibromobutane<sup>a</sup>.

	Br(CH <sub>2</sub> ) <sub>4</sub> Br + 2 C	о <sub>5</sub> Н <sub>5</sub> — О-К+ –	$\begin{array}{c} \begin{array}{c} \text{K}_2\text{CO}_3, \text{PTC} \\ \end{array} \\ \hline \text{H}_2\text{O}, \text{MW} \end{array} \\ \begin{array}{c} \text{O} \\ \text{C}_6\text{H}_5 \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \text{O} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \end{array} \\ \end{array} \\ \end{array} \\ \begin{array}{c} \text{O} \\ \end{array} $ \\ \begin{array}{c} \text{O} \\ \end{array} \\ \end{array} \\ \end{array} \\ \end{array} \\ \end{array}  \\ \begin{array}{c} \text{O} \\ \end{array} \\		
РТС	TBAC	TBAB	ТМВАС	HDTMA	PEG-400
Yield ( %) <sup>b</sup>	90	92	25	None	31

<sup>a</sup>Reaction conditions: potassium benzoate (5 mmol), 1,4-dibromobutane (2.5 mmol),  $K_2CO_3$  (1 mmol), PTC (1 mmol), water (1 mL). MW irradiation at 100 °C for 30 min on 10 W.

<sup>b</sup>Average isolated yield from twice reaction.





<sup>a</sup>Reaction conditions: potassium benzoate (5 mmol), 1,4-dibromobutane (2.5 mmol),  $K_2CO_3$  (1 mmol), TBAB (1 mmol), water (1 mL). MW irradiation at 100 °C for 30 min on 10 W.

<sup>b</sup>Numbers in parentheses are the average isolated yields from twice reactions.

Various aromatic carboxylates, including heteroaromatic acids, reacted well with 1,4dibromobutane under the reaction conditions optimized above (Tables 1 and 2) in high yield.

A wide array of aromatic carboxylates could be efficiently diesterifisized in high yield, no matter which substitute bears on the aromatic ring, either an electron-withdrawing group or an electron-donating group. It is worth pointing out that (2E)-butane-1,4-diyl bis (3-phenylacrylate) (**3k**) is a trans-isomer (Table 3), which was identified by <sup>1</sup>H NMR and melting point. In all cases examined, the diesterification reactions were clean,

essentially forming pure diesters in a short reaction time. The products were simply isolated by filtration.

#### **Experimental**

#### **General methods**

All commercially available reagents were used without further purification. Column chromatography was performed on silica gel (200–400 mesh). Melting points were determined on a WRS-1A digital instrument and uncorrected. <sup>1</sup>H (400 MHz) and <sup>13</sup>C (100 MHz) NMR spectra were recorded on a JNM-ECS400M spectrometer in CDCl<sub>3</sub> with TMS as an internal standard at room temperature, unless otherwise indicated. EI–MS data were measured with a TRACE DSQ GC–MS spectrometer. Elemental analysis (% C, H, N) was performed by Vario EL analyzer. Microwave reactions were conducted in a CEM Focused MW Synthesis System (CEM Corp., Matthews, NC). All substrates and reagents were obtained from commercial sources and 1, 4-dibromobutane was used as received.

#### General experimental procedures

In a 10-mL glass tube, (potassium) aromatic carboxylate (5.0 mmol), 1,4-dibromobutane (2.5 mmol),  $K_2CO_3$  (1.0 mmol), TBAB (1.0 mmol), 1 mL H<sub>2</sub>O, and a magnetic stir bar were placed. The vessel was sealed with a septum and placed into the MW cavity. Microwave irradiation of 10 W was used, with the temperature was ramped from room temperature to 100 °C. Once 100 °C was reached, the reaction mixture was held at this temperature for 30 min. After cooling the mixture to room temperature, the reaction vessel was opened and the contents poured into a beaker with 50 mL water. The product was washed fully and filtered under vacuum. The product was recrystallized from 95% ethanol or purified by column chromatography on silica gel using petroleum-ethyl acetate (3:1 v/v) as the eluent, giving analytically pure products.

#### Butane-1,4-diyl dibenzoate (3a)

White crystal, mp (°C) 82–83. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta = 1.90-2.00$  (m, 4H), 4.40 (m, 4H), 7.25–7.50 (m, 4H), 7.52–7.57 (m, 2H), 8.03–8.06 (m, 4H). <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta = 25.5$ , 64.4, 128.3, 129.5, 130.2, 132.8, 166.5. MS (*m/z*): Calculated for C<sub>18</sub>H<sub>18</sub>O<sub>4</sub>: 298.12, Found: 298. Anal. Calcd. For C<sub>18</sub>H<sub>18</sub>O<sub>4</sub>: C, 72.47; H, 6.08; Found: C, 72.46; H, 6.05;

#### Conclusions

In this work, we present a simple, highly efficient protocol for the synthesis of butamethylenediesters in water. The diesterification reactions proceeded under focused MW irradiation in combination with PTCs, which constitutes a novel and powerful approach. Its valuable features are atom-efficiency, short reaction time, broad substrate 6 🕢 S. DING ET AL.

cope, and environment protection. From a synthetic perspective, these results explore new possibilities of applying this entry to preparing diesters in water.

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