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Oxidative esterification of primary alcohols at room temperature under aqueous medium

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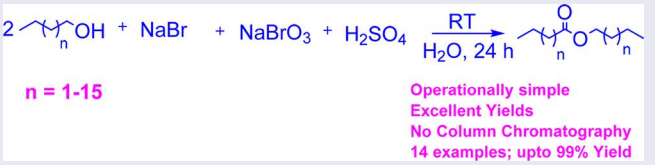
ABSTRACT

Oxidative esterification of aliphatic primary alcohols with bromide and bromate couple in aqueous acidic medium at room temperature is reported with a wide range of substrate scope for both aliphatic and cyclic alcohols and obtained excellent yields of products.

GRAPHICAL ABSTRACT



ARTICLE HISTORY
Received 1 January 2018

KEYWORDS
Aliphatic esters; aqueous medium; bromide–bromate couple; oxidative esterification

Introduction

The selective oxidation of alcohol is one of the most important transformations in organic synthesis. Moreover, importance and versatility of the corresponding carbonyl products are useful in chemical industries. Among all carbonyl compounds, esters are one of the most abundant classes of chemicals, widely utilized in fine chemicals, pharmaceuticals, natural products, agrochemicals, and food additives.[1] Plants and vegetable oils and their constituent fatty acids have been used as precursors in the chemical industry for their transformation into high-valued functional chemicals and biofuels.[2] Typically, aliphatic monoesters found in jojoba oil can be easily synthesized from the fatty acids and alcohols derived from triacylglycerol oils.[3] Particularly, long-chain fatty esters are used in the preparation of waxes for industrial applications[4a] and as alternate feedstock in the production of biodiesel.[4b,c] Traditional methods of esters are prepared by the reaction of carboxylic acids or acid derivatives (acyl chlorides and anhydrides) with alcohols.[5] Considering the fact that alcohols are less corrosive and more accessible than acids or aldehydes, the direct aerobic oxidation of alcohols to esters is an important alternative, which has been accomplished with several homogeneous[6–8] and heterogeneous[9] catalytic systems. Even though heterogeneous catalytic systems are recyclable, the catalyst preparation involves multistep and drastic conditions are required along with bases or

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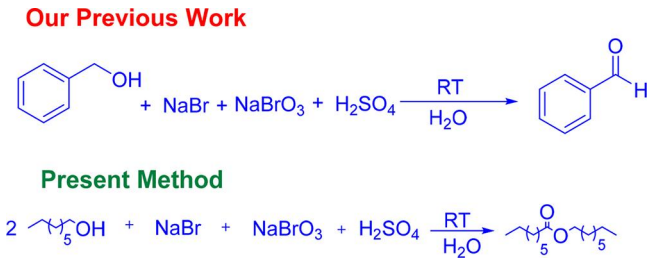


Scheme 1. Oxidative esterification of alcohols.

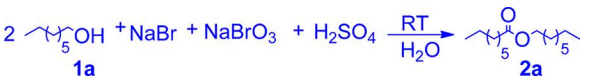
additives.[9g] An existing literature, for the synthesis of esters using precious metals like Pd, Co, Ru, Rh, Ce, Au, and Pt are generally used in the synthesis of esters from alcohols,[6] and use of these metals involves high cost and issues related to environment problems. Some metal-free conditions have also been reported which include organic polymer resin,[7a] L-proline catalyzed,[7b] $NaBrO_3$ and catalytic amount of HBr,[7c] IPy_2BF_4 and I_2 system[4d] and Br_2 and HBr[7e] etc. in these reported methods organic solvents and moderately high temperature are essential to produce esters. An attractive approach is the direct transformation of alcohols to esters under ambient and solvent-free conditions without the use of the corresponding acid or acid derivative (Scheme 1).

Results and discussion

In continuation of our efforts on the development of sustainable and environment-friendly process for the oxidations[10] and oxidative halogenations,[11,12] we report herein a selective oxidation of aliphatic primary alcohols to corresponding esters using a bromide–bromate couple under aqueous medium at room temperature.

We initiated our studies with 1-octanol (**1a**) as a substrate and subjected it to oxidative esterification with catalytic amount (25 mol% w.r.t. **1a**) of commercially available NaBr and $NaBrO_3$ (2:1) dissolved in 10.0 mL of H_2O followed by the slow addition of 2.0 mL of aqueous H_2SO_4 (0.5 eq. H_2SO_4) to the reaction mixture, which generates hypobromous acid (BrOH) in situ. The BrOH oxidized 1-octanol to octyloctanoate (**2a**) at room

Table 1. Optimization of condition for **2a**[a].



Entry	Bromine source	Acid/oxidant	Yield (%)[b]
1[c]	NaBr + $NaBrO_3$	H_2SO_4	25
2[c]	NaBr + $NaBrO_3$	H_2SO_4/H_2O_2	27
3	NaBr + $NaBrO_3$	H_2SO_4	98
4	NaBr + $NaBrO_3$	HCl	90
5	NaBr + $NaBrO_3$	HNO_3	91
6[c]	HBr	H_2O_2	n.d.
7	HBr	H_2O_2	n.d
8[d]	NaBr + $NaBrO_3$	H_2SO_4	98

[a]**1a** (*n*-octanol) 1.0 g, NaBr 0.523 g (0.66 eq.) $NaBrO_3$ 0.383 g (0.33 eq.) and sulfuric acid 0.2 mL (0.5 eq.) 10 mL of water at room temperature.
[b]Crude yield of the product confirmed by NMR.
[c]Catalytic amount of bromine source was used.
[d]The reaction out 50.0 g scale.

temperature and obtained 25% of **2a** after 24 h (Table 1, entry 1). Further, to improve the yield of the reaction, 2.0 eq. of H_2O_2 was used; no improvement was observed (Table 1, entry 2). This indicates that the amount of ester formation was equivalent to the amount of reagent used. Then stoichiometric amount of bromide and bromate couple (2:1) was used to achieve complete conversion of the substrate; under these conditions, 98% of **2a** formation was observed (Table 1, entry 3). The reaction was performed. Using mineral acids such as HCl and HNO_3, the yield of **2a** was declined to 90 and 91%, respectively (entries 4 and 5). Further, we screened with catalytic and stoichiometric amounts of HBr and H_2O_2 system, the yield of **2a** was not observed (Table 1, entries 6 and 7). Finally, the optimized conditions were fixed for the preparation of **2a** with stoichiometric ratio of bromide and bromate couple (2:1) and 0.5 eq. of H_2SO_4 with respect to alcohol (entry 3) for further substrate scope. To check the feasibility for the method, we performed the reaction at 50.0 g scale under optimized conditions and obtained the corresponding ester in 98% yield (entry 8). This reaction suggests the scope for upscaling the products without any difficulties for commercial/industrial production.

Under these optimized conditions on hand (Table 1, entry 3), the generality of the oxidative esterification of alcohols was examined (Table 2). Different primary aliphatic alcohols (butanol, pentanol, hexanol, heptanol, nonan-1-ol, decan-1-ol, undecan-1-ol, dodecan-1-ol, tetradecan-1-ol, and hexadecan-1-ol) were subjected to the optimized conditions and corresponding esters **2b–2k** were obtained in excellent yields (quantitative yields in many cases). It may be noted that the yields mentioned in Table 2 are crude yields after extraction and removal of the solvent but found to be pure by NMR analysis without separation by column chromatography.

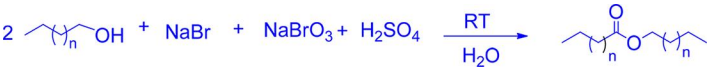
Then, the substrate scope was extended to cyclic primary alcohols (Table 3). The reactions of acyclic alcohols such as cyclohexyl methanol (**1 l**), cyclohexyl ethanol (**1 m**), and cyclohexyl propanol (**1n**) were also provided the corresponding esters (**2l–2n**) in excellent yields (90–97%).

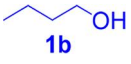
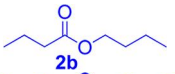
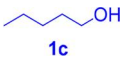
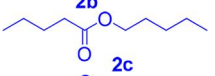
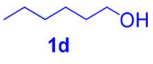
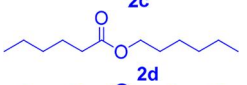
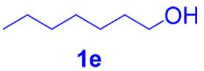
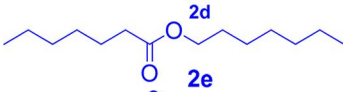
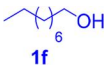
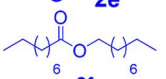
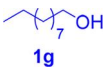
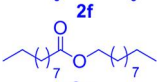
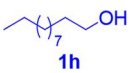
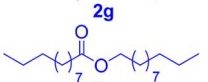
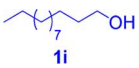
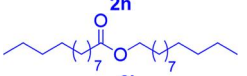
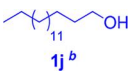
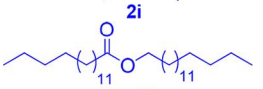
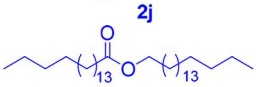
Under the optimized conditions, the oxidative esterification of aryl alkyl alcohols such as 3-phenylpropan-1-ol (**3a**) was subjected, the mixtures of corresponding aldehyde **3b**, acid **3c**, and ester **3d** were observed in 43, 30, and 18% yields (GC–MS), respectively (9% unreacted **3a** remains). Further, we performed the reaction of two different alcohols (**1a** and **1d**) under the standard conditions to get a single cross-coupled ester, but two different homocoupled esters were observed as major products **2a** and **2d** in 47 and 45% yields (by GC–MS) (Scheme 2). 2-Octanol was also tested under the same conditions and the mixture of 2-octanol and 2-octanone was observed.

The reaction progress on esterification of 1-octanol was monitored with time, the conversion of 1-octanol and formation of octyloctanoate are shown in Figure 1. A sharp linear decrease in the concentration of 1-octanol was observed up to 10 h with the simultaneous formation of the corresponding ester. With time, the concentration of alcohol is decreased and becomes slow and tends to complete after 24 h. This study indicates that the reaction is faster in the beginning and requires 24 h for the complete conversion.

Further to understand reaction mechanism, some control experiments were performed (Scheme 4). It was observed that in the presence of TEMPO as well as BHT no ester formation was observed under the optimized conditions as confirmed by GC–MS. These reactions suggest that the reaction may proceed through the radical pathway. To know

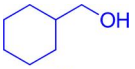
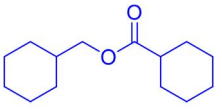
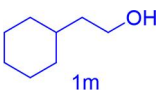
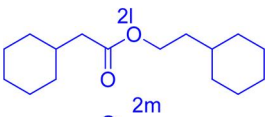
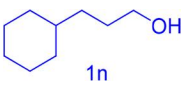
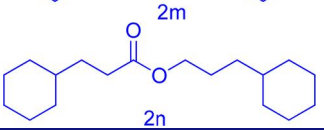
Table 2. Substrate scope of aliphatic acyclic alcohols[a].



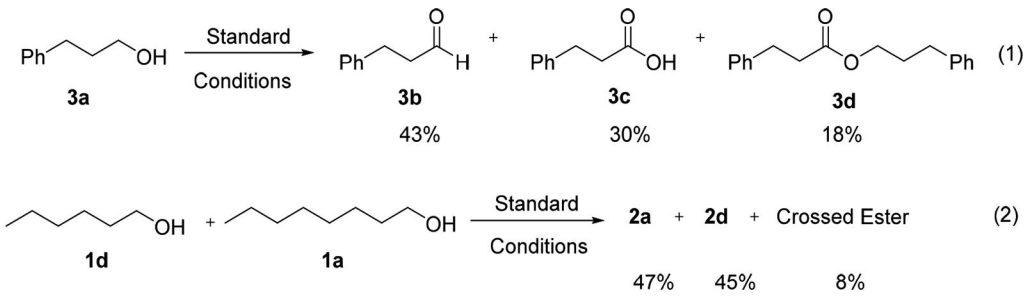
Reactant	Product	Yield (%)
 1b	 2b	92
 1c	 2c	90
 1d	 2d	96
 1e	 2e	97
 1f	 2f	98
 1g	 2g	99
 1h	 2h	99
 1i	 2i	99
 1j[b]	 2j	99
 1k[c]	 2k	99

[a]Reagents and conditions: alcohol 1.0 g, NaBr (0.66 eq.) $NaBrO_3$ (0.33 eq.) and sulfuric acid (0.5 eq.) w.r.t. alcohol and 10 mL of water, crude yield of the product confirmed by NMR.
[b]Dichloroethane (5 mL) as solvent.
[c]Dichloromethane (5 mL) as solvent.

Table 3. Substrate scope of aliphatic cyclic alcohols[a].

Reactant	Product	Yield (%)
 1l	 2l	90
 1m	 2m	92
 1n	 2n	97

[a]Reagents and conditions: alcohol 1.0 g, NaBr (0.66 eq.) $NaBrO_3$ (0.33 eq.) and sulfuric acid (0.5 eq.) w.r.t. alcohol and 10 mL of water, crude yield of the product confirmed by NMR.



Scheme 2. Additional experiments.

the reaction intermediate, we performed the reaction of **1a** with 1-octanal (**5**) and 1-octanoic acid (**6**); in the former case, quantitative yield of **2a** was observed by GC–MS.

In the latter case also, the quantitative yield was observed with respect **1a**, but the acid **6** was remains unreacted. In both of these reactions, the reagent (bromide, bromate, and acid) was reduced to half from the optimized conditions. As one mole of BrOH required to convert **1a** to **5**, but as these reactions (Scheme 3, **condition A**) started with corresponding 1-octanal (**5**) and 1-octanoic acid, only 50% of the reagent required to convert the octanal to acylbromide as explained under Scheme 4. The acyl bromide reaction with another molecule of **1a** provides the desired ester. These reactions suggest that the reaction proceeds through aldehyde intermediate but not the acid. The reaction of **1a** was performed in dark under the optimized conditions, but no product formation was observed (Scheme 3).

Based on the control experiments and literature reports for such transformation, a probable reaction mechanism has been proposed (Scheme 4). The mechanism may proceed through several steps. Initially, bromide–bromate couple in aqueous acidic medium generates reactive species BrOH. The BrOH species may exist in equilibrium as bromine radical and hydroxyl radical [Eq. (2)]. This bromine radical abstracts a proton from the alcohol and generates corresponding alkyl radical [Eq. (3)], its subsequent reaction with

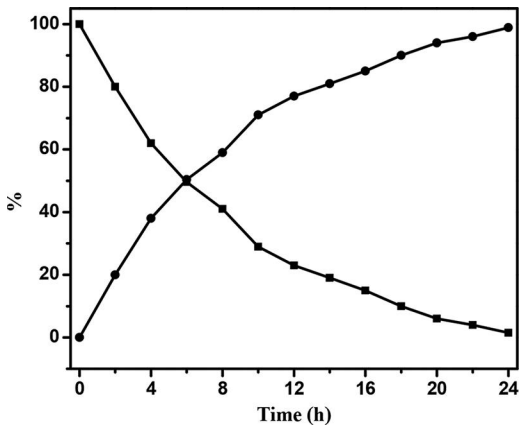
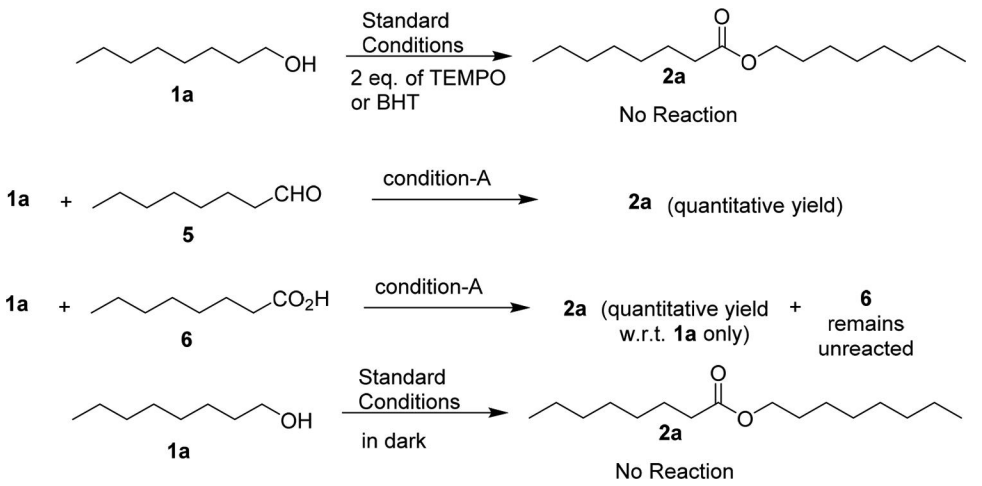
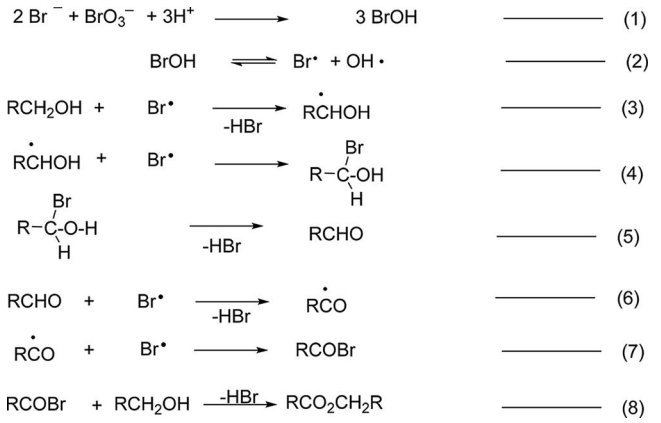


Figure 1. Reaction profile.



Scheme 3. Control experiments, **Condition A: 1a** (*n*-octanol) 1.0 g, NaBr 0.262 g (0.33 eq.) $NaBrO_3$ 0.192 g (0.165 eq.) and sulfuric acid 0.1 mL (0.25 eq.) 10 mL of water at room temperature.



Scheme 4. Plausible mechanism.

bromine radical and elimination of HBr generates stable aldehyde [Eqs. (4) and (5)]. Further, through the subsequent reaction of aldehyde with bromine radical and another molecule of alcohol yields the desired ester with loss of HBr.

Conclusion

In conclusion, we have developed oxidative esterification of aliphatic primary alcohols using environment-friendly reagents under aqueous acidic medium at room temperature. All the primary alcohols provided quantitative yields of homocoupled esters. The products showed pure by NMR without separation column chromatography, but simple extraction and removal of the organic solvent. Compared to the reported methods, present system has advantages of metal-free conditions, elimination of multistep catalyst preparation (under heterogeneous medium), and the use of commercially available reagents.

Experimental section

General methods

All commercially available chemicals and reagents were used without any further purification unless otherwise indicated. ^{1}H and ^{13}C NMR spectra were recorded at 500, 600, and 125, 150 MHz, respectively. The spectra were recorded in $CDCl_3$ as a solvent. Multiplicity was indicated as follows: s (singlet); d (doublet); t (triplet); m (multiplet); dd (doublet of doublets), etc. Coupling constants (*J*) were given in Hz. Chemical shifts are reported in δ relative to TMS as an internal standard. The peaks around δ values of 7.26 (^{1}H NMR), 77.0 (^{13}C NMR) correspond to $CDCl_3$.

General procedure for octyl octanoate (2a)

A total of 1.0 g of 1-octanol (7.69 mmol) was taken in a 50-mL round-bottomed flask, to it NaBr 0.523 g (0.66 eq.), $NaBrO_3$ 0.383 g (0.33 eq.), and 10 mL of H_2O [comprises the bromide and bromate in 2:1 molar ratio] were added[6f]. The reaction mixture was stirred vigorously to dissolve the contents completely. To the above reaction mixture, the aqueous H_2SO_4 solution (0.5 eq.) was added slowly under stirring over a period of 2.5 h at room temperature (prepared by adding 0.21 mL of 98% H_2SO_4 to 1 mL of water). The reaction mixture was allowed to stir for up to 24 h. After the completion of reaction, the product was extracted with CH_2Cl_2 (3 × 15 mL), the organic layer was dried with Na_2SO_4 and removal of the solvent obtained octyloctanoate in 98% yield (0.953 g) as colorless liquid. The product was confirmed by GC–MS as well as by NMR.

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