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A simple, one-pot oxidative esterification of aryl aldehydes through dialkyl acetal using hydrogen peroxide

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Abstract A simple and an efficient one-pot procedure has been developed to synthesize various aryl carboxylic esters directly from aryl aldehydes using hydrogen peroxide without any catalyst. The reaction proceeds smoothly at room temperature. A preliminary investigation suggests the formation of dialkyl acetal as an intermediate during the reaction sequence.

Keywords Aryl aldehyde · Oxidative esterification · Hydrogen peroxide · Dialkyl acetal

Introduction

Aryl carboxylic esters are very important and useful synthetic intermediates or structural elements in the manufacture of pharmaceuticals and agrochemicals [1, 2]. Generally, aryl carboxylic esters are prepared by the condensation of aryl carboxylic acids and alcohols, using the Fischer esterification [3–5]. Carboxylic acids are activated by converting them into acyl chlorides or anhydrides which in turn are employed in the synthesis of corresponding esters [6]. One-pot oxidative esterification reaction is receiving increasing attention over classical methods due to its economic

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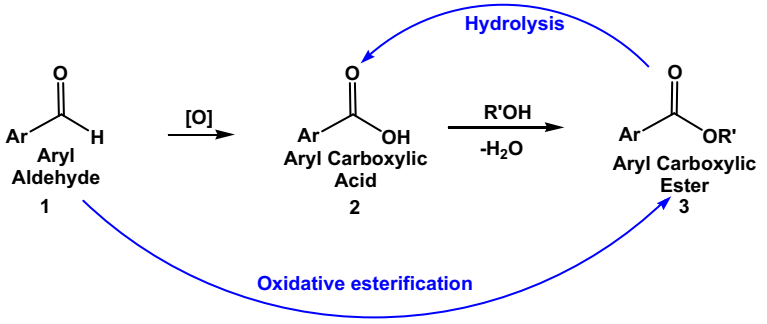
viability [7] (Scheme 1). For oxidative esterification reactions, various metal catalysts such as palladium [8–12], copper [13], manganese [14], iridium [15], tungsten [16], zinc [17], vanadium [18], calcium and magnesium [19], nanoparticle catalysts such as gold–nickel oxide nanoparticles [20], gold nanoparticles [21, 22] and iron oxide nanoparticles [23], N-heterocyclic carbene (NHC) catalysts [24–26], ionic liquids [27, 28], oxoammonium salts [29], and reagents such as iodine [30] and $TsNBr_2$ [31] have been used. Heavy-metal oxidants such as manganese(IV) oxide [32] and expensive reagents such as *tert*-butyl hydroperoxide (TBHP) have also been reported [33]. Catalyst recovery methods and reactivation is critical in these protocols. An ultrasound-assisted condition is also reported for this reaction [34]. Hydrogen peroxide is a versatile green oxidant used to effect various organic transformations [35], and it is also used in oxidative esterification of secondary alcohols into ketones, primary alcohols into carboxylic acids [36, 37], and aldehydes into carboxylic acids [38] in the presence of a phase transfer catalyst (PTC).

The synthesis of aryl carboxylic esters by oxidative esterification under mild conditions with simple reagents would be an attractive approach. We made an earnest attempt at direct oxidative esterification of alcohols and aryl aldehydes using hydrogen peroxide under mild condition (Scheme 2) without any catalyst, and it was found to be successful with appreciable conversion. Hence, we are reporting this hitherto unreported reaction.

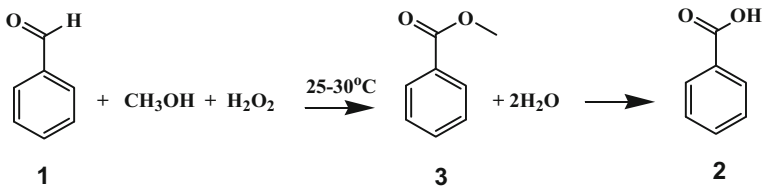
In this newly developed simple and efficient protocol, hydrogen peroxide being a green oxidant generates water as the only by-product. With 26 equi. of alcohol as a reactant/solvent and 8 equi. of H_2O_2 (46 % solution), various aryl aldehydes were converted into their corresponding aryl esters with up to 73 % yields.

Experimental

Laboratory-grade solvents and reagents were used as received. Boiling points and melting points were determined on an Elchem Microprocessor-based DT apparatus and are uncorrected. For thin-layer chromatography (TLC), Merck silica gel 60 F254 pre-coated plates were used, visualized by exposure to UV light (254 nm). ^{1}H NMR and ^{13}C NMR were determined on a Bruker 400-MHz spectrometer using



Scheme 1 Synthesis of Aryl carboxylic esters



Scheme 2 Synthesis of Aryl carboxylic esters using one-pot oxidative esterification

DMSO-d_6 and $CDCl_3$ solvents. Chemical shifts (δ) are reported in ppm and coupling constants (J) are in Hertz (Hz). GC-MS data were obtained at 70 eV using a Thermo-fisher Trace Ultra GC and PolarisQ MS using HP-5 (column 30 m length × 0.32 mm inner diameter × 0.25 μm film thickness).

General procedure for the synthesis of methyl benzoate

A mixture of benzaldehyde (5 g, 47.2 mmol) and methanol (50 mL, 26 equi.) was stirred at 30 °C and 25 mL of 46 % H_2O_2 (8 equi.) was added dropwise over 10 min. The reaction mixture was stirred at 30 °C for 3 h and the progress of the reaction was monitored through TLC. After the completion of the reaction, 250 mL of water was added to the reaction mixture and the obtained products were extracted using ethyl acetate (2 × 100 mL). The combined ethyl acetate layer was treated with 500 mL of 20 % solution of sodium bisulfite solution, followed by 100 mL of saturated solution of sodium bicarbonate and then washing with 100 mL of saturated sodium sulfate solution. Finally, ethyl acetate was distilled off and the product was collected as a residue. Colourless liquid; bp 196–198 °C; ^{1}H NMR (400 Hz, $CDCl_3$) δ 3.90 (s, 3H, CH_3), 7.43 (m, 2H, ArH), 7.53 (m, 1H, ArH), 8.02 (m, 2H, ArH); ^{13}C NMR (100 Hz, $CDCl_3$) δ 52.12, 115.43, 128.40, 129.62, 130.20, 132.97, 167.20; MS (EI): *m/z* (%) = 136 (29) [M^+], 105 (100), 92 (29), 77 (73), 51 (28).

Caution: The mixture of hydrogen peroxide and organic solvents forms organic peroxides which can ignite easily and burn rapidly and intensely. Proper care must be taken while handling this mixture. It is essential to treat the entire reaction mass with excess sodium bisulfite to ensure complete destruction of organic peroxides.

Ethyl benzoate (Product of Entry 2, Table 3)

Colourless liquid; bp 210–211 °C; ^{1}H NMR (400 Hz, $CDCl_3$) δ 1.40 (t, J = 7.2 Hz, 3H, CH_3), 4.39 (q, J = 7.2 Hz, 2H, –CH_2), 7.44 (m, 2H, ArH), 7.54 (m, 1H, ArH), 8.04 (m, 2H, ArH); ^{13}C NMR (100 Hz, $CDCl_3$) δ 14.37, 61.02, 128.37, 129.59, 130.56, 132.87, 166.74; MS (EI): *m/z* (%) = 150 (59) [M^+], 122 (23), 105 (100), 77 (29), 51 (10).

Isopropyl benzoate (Product of Entry 3, Table 3)

Colurless liquid; bp 264–266 °C; ^{1}H NMR (400 Hz, CDCl3) δ 1.28 (d, J = 6.0 Hz, 6H, CH_3), 5.16 (m, 1H, CH), 7.34 (m, 2H, ArH), 7.45 (m, 1H, ArH), 7.95 (m, 2H,

ArH); ^{13}C NMR (100 Hz, $CDCl_3$) δ 22.05, 68.47, 128.37, 129.61, 131.02, 132.80, 166.27; MS (EI): *m/z* (%) = 164 (16) [M^+], 123 (34), 105 (100), 77 (29), 51 (9).

Methyl 2-chlorobenzoate (Product of Entry 4, Table 3)

Colurless liquid; bp 232–234 °C; ^{1}H NMR (400 Hz, $CDCl_3$) δ 3.90 (s, 3H, CH_3), 7.27 (m, 1H, ArH), 7.39 (m, 2H, ArH), 7.78 (m, 1H, ArH); ^{13}C NMR (100 Hz, $CDCl_3$) δ 52.40, 126.56, 130.09, 131.05, 131.38, 132.54, 133.67, 166.15; MS (EI): *m/z* (%) = 170 (37) [M^+], 172 (13), 139 (100), 111 (29), 75 (23).

Methyl 3-chlorobenzoate (Product of Entry 5, Table 3)

Colurless liquid; bp 230–232 °C; ^{1}H NMR (400 Hz, $CDCl_3$) δ 3.80 (s, 3H, CH_3), 7.23 (m, 1H, ArH), 7.39 (m, 1H, ArH), 7.79 (m, 1H, ArH), 7.89 (s, 1H, ArH); ^{13}C NMR (100 Hz, $CDCl_3$) δ 52.34, 127.68, 129.66, 131.88, 132.91, 134.51, 165.81; MS (EI): *m/z* (%) = 170 (59) [M^+], 172 (20), 139 (100), 111 (31), 75 (21).

Methyl 4-chlorobenzoate (Product of Entry 6, Table 3)

Colurless liquid; bp 245–247 °C; ^{1}H NMR (400 Hz, $CDCl_3$) δ 3.88 (s, 3H, CH3), 7.37 (d, J = 8.4 Hz, 2H, ArH), 7.93 (d, J = 8.4 Hz, 2H, ArH); ^{13}C NMR (100 Hz, $CDCl_3$) δ 52.20, 128.67, 130.95, 139.33, 166.14; MS (EI): *m/z* (%) = 170 (48) [M^+], 172 (17), 139 (100), 111 (28), 75 (20).

Methyl 3-hydroxybenzoate (Product of Entry 9, Table 3)

Pale brown solid; bp 70–71 °C; ^{1}H NMR (400 Hz, DMSO-d_6) δ 3.81 (s, 3H, CH_3), 7.03 (m, 1H, ArH), 7.29 (m, 1H, ArH), 7.37 (m, 2H, ArH), 9.83 (s, 1H, OH); ^{13}C NMR (100 Hz, DMSO-d_6) δ 52.00, 115.55, 119.77, 120.26, 129.77, 130.81,157.46, 166.22; MS (EI): *m/z* (%) = 152 (61) [M^+], 121 (100), 93 (39), 65 (30), 63 (12).

(Dimethoxymethyl) benzene (4)

Colurless liquid; bp 86–88 °C/18 mmHg; ^{1}H NMR (400 Hz, $CDCl_3$) δ 3.34 (s, 6H, CH_3), 5.40 (s, 1H, CH), 7.47 (m, 3H, ArH), 7.52 (m, 2H, ArH); ^{13}C NMR (100 Hz, $CDCl_3$) δ 52.70, 103.21, 125.70, 126.71, 128.21, 128.88, 129.01, 129. 82; MS (EI): *m/z* (%) = 152 (1) [M^+], 121 (100), 91 (25), 77 (14).

Result and discussion

The conversion of benzaldehyde **1** into the methyl benzoate **3** was considered as a representative reaction and hence initially the reaction was attempted with 13 equi. of methanol and 8 equi. of H_2O_2 (46 % solution) at RT which led to the formation of 48 % methyl benzoate **3** with 49 % conversion of benzaldehyde after 3 h (Entry 1, Table 1). We attempted to derive the optimal conditions for the synthesis of

methyl benzoate by varying the reactant quantity, reaction duration and temperature. Accordingly, when the reaction was allowed to continue for 24 h, it led to the formation of methyl benzoate **3** (with 21 % yield) and benzoic acid **2** (with 70 % yield) with conversion of 91 % benzaldehyde. When the reaction was carried out at 65 °C, the complete conversion of benzaldehyde was observed with the formation of 38 % methyl benzoate and 62 % benzoic acid (Entries 2 and 3, Table 1). Reaction with 26 equi. of methanol at RT substantially favoured the formation of methyl benzoate (the desired product) up to a maximum of 71 % with a total conversion of 73 % (Table 1, Entry 4).

The formation of benzoic acid **2** was attributed to the hydrolysis of the produced ester **3**. Initially, we thought that the conversion of **1** into **3** was through benzoic acid **2** as the intermediate, and hence we employed benzoic acid directly under the same reaction conditions as mentioned in Entry 4 of Table 1, which failed to produce methyl benzoate (Scheme 3). This conclusively revealed that the reaction did not proceed through benzoic acid as the intermediate.

When we increased the methanol to 39 and 52 equi (Entries 5 and 6, Table 1) a by-product of (dimethoxymethyl) benzene **4** (dialkyl acetal) was obtained with yields of 13 and 20 %, respectively, with concomitant decrease of the methyl benzoate yield. This could possibly indicate that the reaction proceeds through an intermediate of dialkyl acetal type (acetal intermediate) (Scheme 4). In order to capture the dialkyl acetal intermediate **4**, the reaction was carried out under dilute H_2O_2 concentration (Table 2).

As per Entry 1, Table 2 substantial formation of acetal intermediate **4** (67 %) was observed after 2 h along with 8 % of ester **3** when just 1.6 equi. of H_2O_2 solution (46 % solution) was used. However, on continuing the reaction to 3 h, the dialkyl acetal intermediate **4** was decreased to 59 % (desired ester was increased to 18 %) (Entry 2, Table 2), which could be due to a portion of the acetal intermediate **4**

Table 1 Synthesis of methyl benzoate

Entry	MeOH equi.	Time (h)	Conversion[a] (%)	Yield[a] (%)	
				2	**3**
1	13	3	49	0	48
2	13	24	91	70	21
3[b]	13	3	100	62	38
4	26	3	73	1	71
5[c]	39	3	73	0	60
6[d]	52	3	76	0	56

Hydrogen peroxide (46 % solution, 8 equi.) was added to the solution of benzaldehyde in MeOH for 10 min and the mixture was stirred at 30 °C for the specified duration

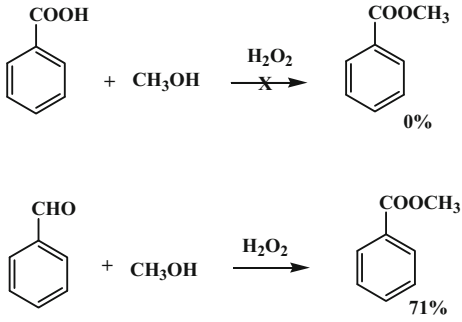
[a] GC results

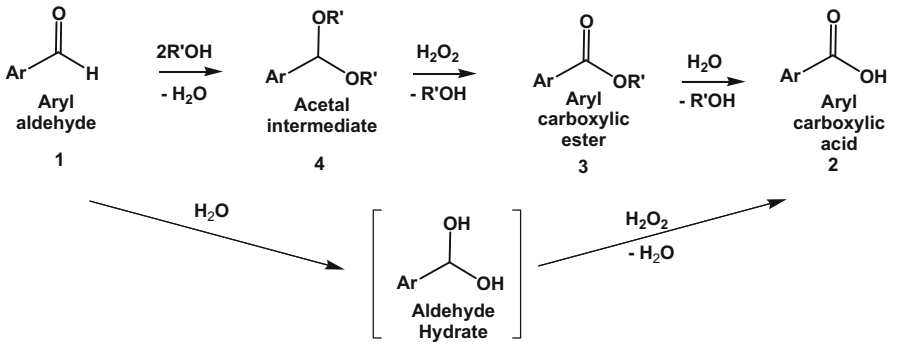
[b] Reaction temperature is 65 °C

[c] (Dimethoxymethyl) benzene was detected as by product (13 %)

[d] (Dimethoxymethyl) benzene was detected as by product (20 %)

Scheme 3 Selective formation of methyl benzoate from benzaldehyde





Scheme 4 Proposed course of oxidative esterification reaction and by products formation

being converted into the ester **3**. A further 1.6 equi. addition of H_2O_2 to the above reaction mixture led to a significance decrease of acetal intermediate **4** to 34 % (along with a significant increase of ester **3** to 38 %) in 1 h (Entry 3, Table 2). The reaction eventually showed 23 % acetal intermediate **4** with 56 % product **3** after 24 h (Entry 4, Table 2).

Table 2 Synthesis of methyl benzoate with dilute peroxide concentration

Entry	H_2O_2 equi.	Time (h)	Conversion[a] (%)	Yield[a] (%)	
				4	**3**
1	1.6	2	75	67	8
2[b]	1.6	3	78	59	18
3[b]	3.2	4	73	34	38
4	3.2	24	79	23	56

H_2O_2 was added to the solution of benzaldehyde (5 g) in 100 mL MeOH for 10 min and the mixture was stirred at 30 °C for specified duration

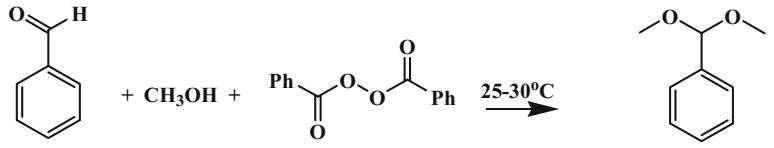
[a] GC results

[b] Entries 2 and 3 are the same reactions distinguished by different amounts of H_2O_2 addition

In a separate attempt, (dimethoxymethyl) benzene **4** was prepared by the reaction of benzaldehyde **1** with 1 equi. of benzoyl peroxide in methanol solvent (Scheme 5). Formation of 90 % (dimethoxymethyl) benzene **4** was observed which was isolated and then treated with methanol and H_2O_2 under the conditions mentioned in Entry 4, Table 1, in which the formation of 65 % methyl benzoate was observed. This confirms conclusively that the oxidative esterification reaction depicted in this report proceeds through acetal intermediate **4** mentioned in Scheme 4.

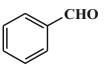
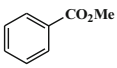
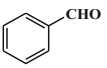
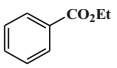
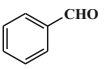
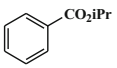
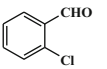
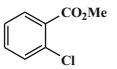
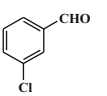
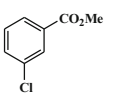
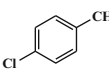
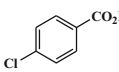
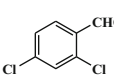
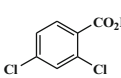
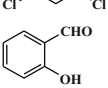
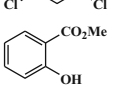
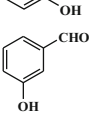
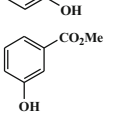
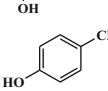
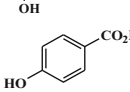
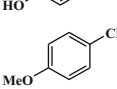
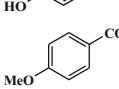
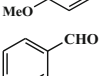
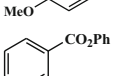
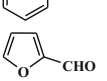
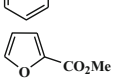
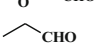
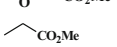
Screening of various aldehydes were carried out (Table 3) under the optimized conditions mentioned in Entry 4 of Table 1. During the screening experiments, the holding time of the reaction was increased to 24 h if the reaction did not show appreciable conversion after 3 h. As mentioned earlier, benzaldehyde afforded 71 % methyl benzoate with 73 % conversion (Entry 1, Table 3 and Entry 4, Table 1). With ethanol, benzaldehyde afforded 58 % ethyl benzoate with 59 % conversion (Entry 2, Table 3). When isopropyl alcohol was employed instead of methanol in the same reaction, benzaldehyde afforded 24 % isopropyl benzoate and 21 % benzoic acid with 45 % conversion; the reaction was found to be slow and it required 24 h for this conversion (Entry 3, Table 3). This observation can be attributed to the steric factor possessed by the isopropyl group. Formation of benzoic acid is due to direct oxidation of benzaldehyde with H_2O_2. This was confirmed by doing separate blank reactions between benzaldehyde (1 g) and H_2O_2 (5 mL) under the same conditions, which afforded 24 % benzoic acid with 25 % conversion of benzaldehyde after 3 h.

Once the reaction conditions were optimized, the oxidative esterification was extended to mono-substituted chloro benzaldehydes under the optimized condition. The 2-chloro, 3-chloro and 4-chloro benzaldehydes found to afford 66, 69 and 71 % of corresponding methyl benzoates, respectively (Entries 4, 5 and 6, Table 3). However, these reactions required 24 h for the conversion (71, 73 and 76 %, respectively) that was realized by benzaldehyde in 3 h.The reaction with 2, 4-dichlorobenzaldehyde was very slow and exhibited only 20 % conversion with 10 % formation of corresponding methyl benzoate even after 24 h (Entry 7, Table 3). In general, chloro-substituted benzaldehydes exhibited sluggish reaction rates when compared to benzaldehyde. When hydroxybenzaldehydes were engaged, 3-hydroxybenzaldehyde showed 73 % formation of methyl-3-hydroxybenzoate with 73 % conversion (Entry 9, Table 3). With 2-hydroxybenzaldehyde (Salicylaldehyde), the reaction was slow, as 16 % methyl salicylate was observed even after 24 h with 16 % conversion (Entry 8, Table 3). When 4-hydroxybenzaldehyde was



Scheme 5 Preparation of (dimethoxymethyl) benzene

Table 3 Synthesis of various aldehydes using oxidative esterification with hydrogen peroxide

Entry	Aldehyde	Ester	Conversion[b] (%)	Yield (%)[b]		
				4	**2**	**3**
1			73	1	1	71[c]
2			59	–	1	58
3			45[a]	–	21	24
4			71[a]	3	–	66
5			73[a]	4	–	69
6			76[a]	4	–	71
7			20[a]	10	–	10
8			16[a]	–	–	16
9			73	–	–	73
10			70[a]	–	–	10[d]
11			53[a]	–	15	11[e]
12			0	–	–	–
13			41	–	–	38
14			99	99	–	–

The mixture of aldehyde (5 g) and alcohol (26 equi.) was stirred at 30 °C and 8 equi. of 46 % H_2O_2 was added dropwise over 10 min. Reaction was stirred at 30 °C for 3 h

[a] Monitoring after 24 h

[b] GC results

[c] Yield is 69 % based of wt% assay using acetophenone as internal standard

[d] 46 % hydroquinone was observed as a by-product

[e] 26 % mequinol was observed as a by-product

engaged, only 10 % methyl-4-hydroxybenzoate was observed with 70 % conversion after 24 h. The major by-product was identified as hydroquinone which was formed to the extent of 46 %. A similar phenomenon was observed with 4-methoxybenzaldehyde which afforded 11 % methyl-3-methoxybenzoate along with 26 % mequinol (4-methoxyphenol) (Entries 10 and 11, Table 3).

When benzaldehyde was treated with phenol, no reaction was observed (Entry 12, Table 3). When a heterocyclic aldehyde such as furfuraldehdye was reacted, 38 % methyl 2-furoate was formed with 41 % conversion. However, product isolation was found to be difficult (Entry 13, Table 3). When an aliphatic aldehyde such as propionaldehyde was used, formation of propionaldehyde dimethyl acetal was observed (Entry 14, Table 3).

Conclusion

A simple and efficient generic protocol has been developed for the oxidative esterification of aryl aldehydes at room temperature with yields up to a maximum 73 %, and the reaction proceeds through dialkyl acetal as intermediate. This synthetic protocol without any catalyst could be of significant value in organic synthesis.

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