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POV-based molecular catalysts for highly efficient esterification of alcohols with aldehydes as acylating agents

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ABSTRACT

The selective oxidative esterification of aldehydes with alcohols to the corresponding esters has been one of the hot spots in scientific research and industrial synthesis. However, the application of precious metal catalytic systems is limited by their complicated synthetic steps and high cost. Thus a highly efficient, green, recyclable selective synthesis method of esters catalyzed by polyoxovanadate (POV)-based molecular catalysts has been developed in this paper. The results show that supramolecular interaction between POV and 1,3-dibenzylimidazolium bromide (Act_2Im) can efficiently convert alcohols and aldehydes to the corresponding esters in high yield under much milder conditions. Mechanistic insight is also provided based on the control experiments, single crystal X-ray diffraction and cyclic voltammetry studies.

Esterification represents a very common and one of the most essential reaction in the synthesis of pharmaceuticals, polymers, solvents and fragrances [1–3]. In comparison, conventional esterification reactions involve the use of protonic acid (H_2SO_4, HCl, H_3PO_4, *etc.*) as catalyst to catalyze the coupling of alcohols and carboxylic acids or activated derivatives [1], which is very corrosive to equipment and troublesome to post-treat accompanied by high energy consumption, many by-products and environmental problems. Amberlyst-15, as a solid acid catalyst in place of corrosive strong acids exhibits excellent performance in oxidative esterification of aldehydes with accompanied by the use of large amount of additives [4]. With the advancement of science and technology, selective esterification systems can be achieved efficiently in the presence of a series of transition metal-based catalysts under mild conditions. Esterification catalyzed by rare noble metal (Ru [5–8], Rh [9], Ir [10], Os [11], Pd [12–16]) catalysts ligated with organic ligands generally associated with the shortcomings of being expensive, complicated/commercially unavailable organic ligands, and difficult to recycle (Scheme 1a). In addition, coin metal (Au [17–22]) nanomaterial-catalyzed esterification also suffers from plenty of drawbacks such as being expensive and large deletion of active sites due to uneven loading while copper-based nanomaterials often suffer from low universality of substrates, difficulties in the study of catalytic sites and catalytic mechanisms [23,24]. Meanwhile, organo-catalyst such as L-proline was developed for the esterification of alcohols due to the inherent non-metal feature, mild reaction conditions and broad functional group tolerance. However, a large number of unsalvageable expensive halogenated reagents (NBS) and catalysts were used, thus generating in copious waste and resulting in poor atom-economy [25]. Therefore, it is still promising to develop new environmentally friendly and atomically economical green catalysts for the synthesis of esters.

Polyoxometalates [26–33], as a class of multinuclear pre-transition metal-oxygen clusters with unparalleled structural diversity and outstanding physicochemical properties have been widely used in the field of catalytic oxidation [34–47], especially for the catalyzed esterification due to their metal ions in the highest valence states being able to act as electron storage containers. Yet, Keggin-type and transition metal-substituted single-lacunary polyoxometalate derivatives are by far the predom-

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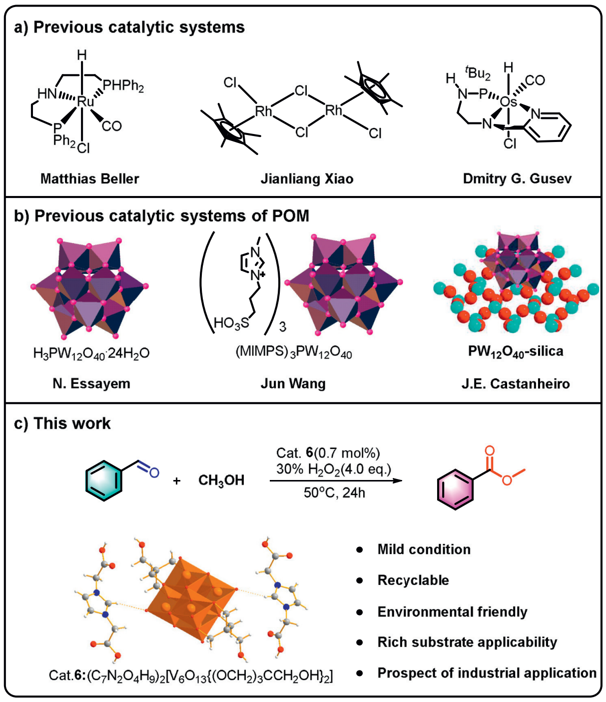


Scheme 1. Catalytic systems for esterification reaction. (a) Previous catalytic systems. (b) Previous catalytic systems of POM. (c) This work: esterification reaction catalyzed by POV-based molecular catalysts.

inant polyoxometalate-based esterification catalysts accompanied by disadvantages such as high temperature, poor substrate applicability and atomic diseconomy (Scheme 1b) [48–56]. The combination of vanadium catalysts and H_2O_2 for the oxidative esterification of aldehydes has been developed for many years. However, the unrecyclability of catalysts and the use of large quantities of strong acid reagents [57], complex catalyst synthesis steps and poor substrate universality [58] limited its application. Meanwhile, imidazole ionic liquids are rapidly gaining in popularity in the field of organic catalysis with the advantages of high boiling point, non-volatile and excellent stability [59] in recent years. Forming *N*-heterocyclic carbenes (NHCs) under basic conditions is a crucial factor to induce aldehydes to undergo polarity reversal and thus the intermediates were capable of nucleophilic substitution reactions [60–68]. However, difficulty to recover and reuse in catalytic systems restrict their practical applications. Thus, a novel catalytic system combines polyoxometalates with imidazole ionic liquids which is expected to improve their respective catalytic performance and enhance the recyclability of the catalyst was developed. Herein, six POV-based molecular crystals $\{(C_8H_{15}N_2)_2[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]$ (**1**, CCDC: 2131494), $(C_{10}H_{19}N_2)_2[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]$ (**2**, CCDC: 2118473), $(C_{11}H_{13}N_2)_2[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]$ (**3**, CCDC: 2,118,470), $(C_{11}H_{21}N_2)_2[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]\cdot 2DMF$ (**4**, CCDC: 2130958), $(C_{17}H_{17}N_2)_2[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]\cdot 2ether\cdot 4/3DMF$ (**5**, CCDC: 2118471), $(C_7H_9O_4N_2)_2[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]\cdot 4DMF$ (**6**, CCDC: 2118474)$\}$ have been successfully synthesized in this work, which can efficiently catalyze the esterification of aldehydes and alcohols using 30% H_2O_2 as the sole oxidant with advantages of mild condition, retrievability, rich substrate applicability, environmental friendly, synthetically cheap, noble-metal free and prospects for industrial applications (Scheme 1c). These six catalysts were characterized by single crystal X-ray diffraction, ^1H NMR and FT-IR, respectively. Interactions between imidazole ionic and POVs were also investigated by single crystal X-ray diffraction while proposed mechanism for the POV-Based Ionic Liquid-catalyzed esterification of aldehydes with alcohols was investigated by single crystal X-ray diffraction, control experiments, EPR and cyclic voltammetry studies.

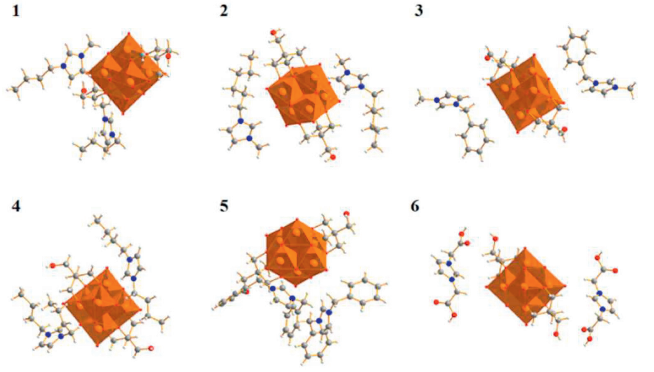


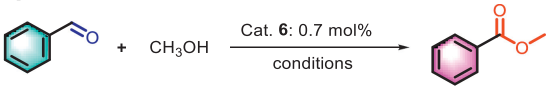
Fig. 1. Crystal structure of compounds **1–6**.

Synthesis of POV-based molecular catalysts: In order to investigate the effect of different structure cations on the reaction, $C_8N_2H_{15}Br$ (abbreviated as (MeBuIm)Br), $C_{10}N_2H_{19}Br$ (abbreviated as (MeHeIm)Br), $C_{11}N_2H_{13}Br$ (abbreviated as (MeBnIm)Br), $C_{11}N_2H_{21}Br$ (abbreviated as $(Bu_2Im)Br$), $C_{17}N_2H_{17}Br$ (abbreviated as $(Bn_2Im)Br$) and $C_7N_2O_4H_9Br$ (abbreviated as $(Act_2Im)Br$) were synthesised by mixing imidazole/1-methylimidazole with excess haloalkane at 60 °C for 24 h (Figs. S1-S12 in Supporting information), we subsequently conducted the synthesis of compounds **1–6** by electrostatic assembly between imidazolium-based ILs and $(C_{16}H_{36}N)_2[V_6O_{13}\{(OCH_2)_3CCH_2OH\}_2]$\{abbreviated as $(TBA)_2V_6$\} in excellent yields and characterized by single-crystal X-ray diffraction (Tables S1-S3 in Supporting information and Fig. 1), NMR (Figs. S13-S18 in Supporting information), FT-IR (Figs. S19-S24 in Supporting information) and ESI-MS (Fig. S25 in Supporting information).

The selective aerobic oxidation of benzaldehyde and methanol to form methyl benzoate was chosen as a template reaction. Initially, compounds **1–6** were conducted to catalyze the template reaction (Table 1). The yields of methyl benzoate are 57%/54%/60%

Table 1

Optimization of reaction conditions.[a]



Entry	Catalyst	H_2O_2 (equiv.)	Yields (%)[b]
1	$(TBA)_2V_6$	4.0	70
2	(MeBuIM)Br	4.0	43
3	(MeHeIM)Br	4.0	47
4	(MeBnIM)Br	4.0	51
5	$(Bu_2IM)Br$	4.0	50
6	$(Bn_2IM)Br$	4.0	55
7	$(Act_2IM)Br$	4.0	62
8	Cat. **1**	4.0	57
9	Cat. **2**	4.0	54
10	Cat. **3**	4.0	60
11	Cat. **4**	4.0	64
12	Cat. **5**	4.0	82
13	Cat. **6**	4.0	99
14	Cat. **6**	1.0	52
15	Cat. **6**	2.0	73
16	Cat. **6**	3.0	90

[a] Reaction conditions: Cat. **6** (0.7 mol%), benzaldehyde (1 mmol), CH_3OH (2.0 mL), 30% H_2O_2 (4.0 equiv.) at 50 °C.

[b] The yield of isolated products.

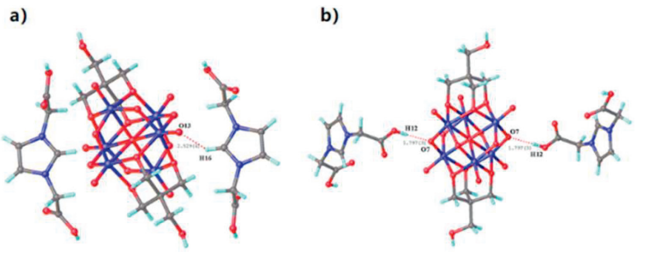


Fig. 2. Different hydrogen bonds of cat. **6**.

with catalysts **1**, **2** and **3**, respectively, which might be ascribed to the same substituent at position 1 and different substituents at position 3 on the imidazole ring have little effect on the reaction (Table 1, entries 8–10). Methyl benzoate can be generated in higher yield when compounds **4–6** were employed as catalysts due to the better electronic and potential for asymmetric induction of this structure in carrying out the induction of aldehydes (Table 1, entries 11–13). Benzaldehyde and methanol produced methyl benzoate in a high yield of 82% catalysed by cat. **5** due to the kinetic stabilization effect from steric bulk (Table 1, entry 12) [69–71]. Notably, the highest products yield of up to 99% was achieved with cat.**6** while $(TBA)_2V_6$ and $(Act_2Im)Br$ exhibits much lower catalytic activity (Table 1, entries 1 and 7), respectively, that indicates synergistically catalytic effect from both POV and Act_2Im. Moreover, compared to catalysts **1–5**, the catalytic activity is also much lower when (MeBuIM)Br, (MeHeIM)Br, (MeBnIM)Br. $(Bu_2IM)Br$ and $(Bn_2IM)Br$ are used as catalysts, respectively (Table 1, entries 2–6). It might be ascribed to supramolecular interactions caused by hydrogen bond (O13···H16) between POV and Act_2Im (Fig. 2) which facilitate the catalysis process. Also, the change in absorbance can be attributed to supramolecular interactions between the POV and Act_2Im in solution (Fig. S26 in Supporting information). Methyl benzoate can also be generated but in much lower yield when the amount of hydrogen peroxide is reduced indicating that the optimal amount of hydrogen peroxide is 4.0 equiv. (Table 1, entries 14–16).

With the above optimized conditions in hand, we examined the selective oxidation of various aldehydes and methanol/ethanol required to determine the generalizability of the current conditions (Fig. 3). Some readily available benzaldehyde derivatives bearing electron-rich groups gave lower yields (compounds **8–13**) and electron-deficient groups gave excellent yields (compounds **14–16**) of corresponding product. The large dipole moment, high polarity and high intermolecular attraction of the nitro compound resulted in an extremely low yield of 37% (compounds **17**). It is very pleasing that both 2-naphthaldehyde and 1,4-phthalaldehyde were able to produce the corresponding esters/diesters in yields of 85% and 84%, respectively (compounds **18** and **19**). We further investigated the aerobic oxidative esterification of the less reactive ethanol with aldehyde derivatives to produce the corresponding esters (compounds **21–30**), ethanol can also undergo facile oxidative esterification to produce the corresponding products smoothly.

Surprisingly, conversion of benzaldehyde, 4-fluorobenzaldehyde, 4-chlorobenzaldehyde and 2-naphthaldehyde to the corresponding

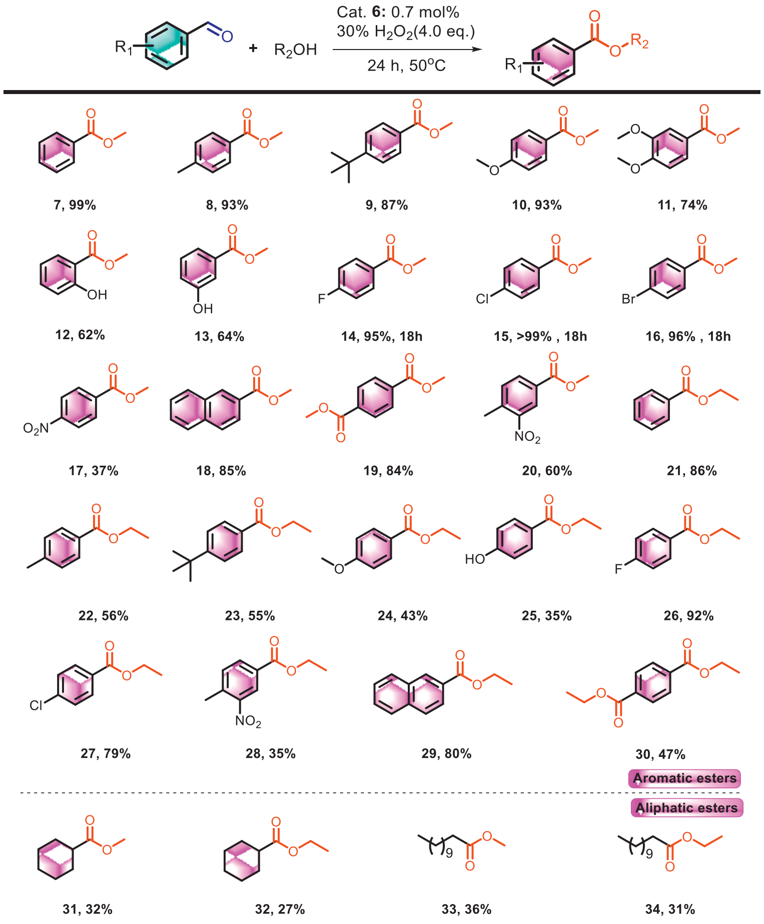


Fig. 3. Esterification of aldehydes and alcohols catalyzed by cat. **6**. Reaction conditions: Cat. **6** (0.7 mol%), benzaldehyde (1 mmol), CH_3OH (2.0 mL), 30% H_2O_2 (4.0 equiv.) at 50 °C. The yield of isolated products.

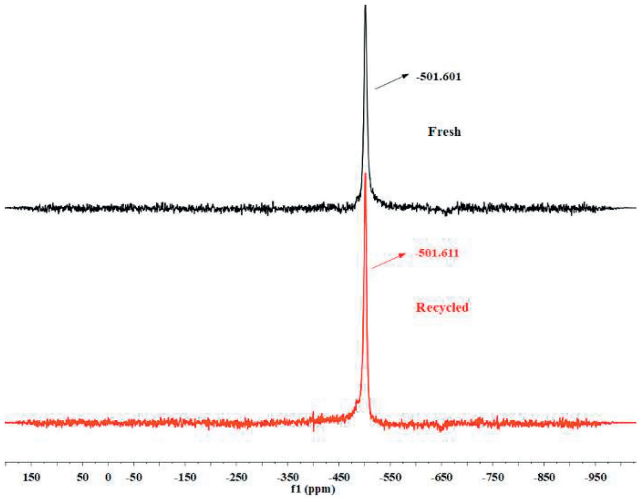


Fig. 4. The ^{51}V NMR spectra of cat. **6** before and after reaction.

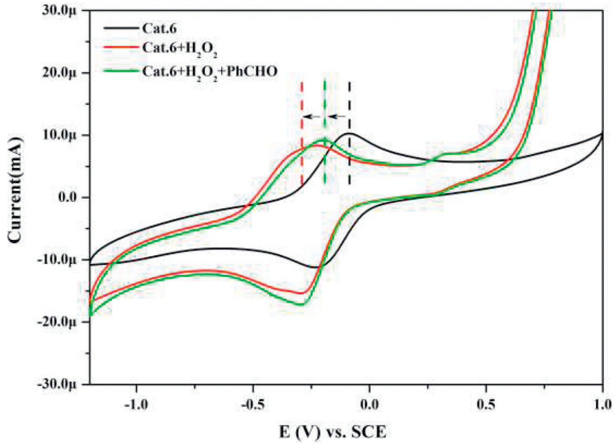


Fig. 5. CVs comparison of the cat. **6** (black curve), cat. **6** + H_2O_2 (green curve), cat. **6** + H_2O_2 + PhCHO (green curve). Cyclic voltammetry was carried out under CH_3OH solution with 0.1 mol/L $TBAPF_6$, 1×10^{-4} mol/L related analyst, respectively. Scan rate: 100 mV/s.

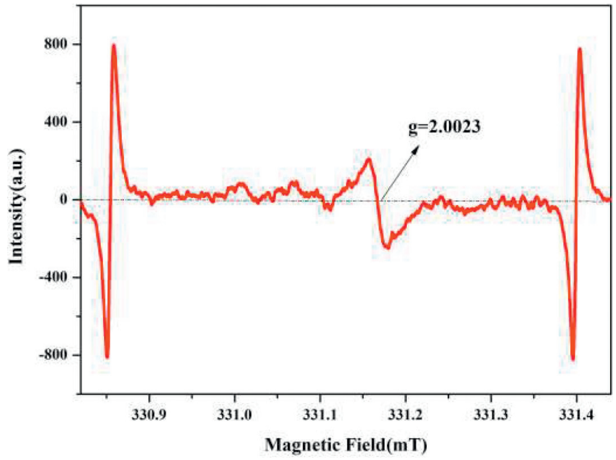


Fig. 6. Normalized EPR spectra of DMPO spin adducts.

esters in yields of up to 86%/92%/79%/80%, respectively. Linear aliphatic aldehyde and cyclohexanecarboxaldehyde can also undergo mild oxidation to give the corresponding products in good yields (compounds **31–34**). To further demonstrate the industrial practicality of this catalytic system, a 10 mmol reaction of benzaldehyde was conducted to give 1.348 g of methyl benzoate in 99% isolated yield, all these results indicate that the catalytic system is unparalleled in the selective oxidation of aldehydes to afford the corresponding esters even has the prospect of industrial application.

After the reaction, the reaction solution is directly spun off, filtered, and the filtrate is rinsed with a large amount of ether and deionised water and then dried in a vacuum drying oven. In order to verify whether the catalyst can be recycled and reused after the reaction, we first characterized the anionic skeleton of the catalyst by FT-IR (Fig. S29 in Supporting information), the strong and narrow IR absorption band with a maximum at 941 cm^{-1} is undoubtedly belonging to the v(V=O) vibrations, and the absorption band at 813.08, 790.96, 709.34 cm^{-1} belonging to the v(V-O-V) vibrations, a narrow and weak absorption peak at 1024 cm^{-1} comes from the C–O vibration on the anion backbone implies that Tris-OH was successfully modified to the POV, the result shows the IR spectra of the catalyst before and after the reaction were basically identical. The cations of recovered catalyst were characterized by ^1H NMR (Fig. S30 in Supporting information) and ^{51}V NMR (Fig. 4), proving that the cations also did not change before and after reaction. Furthermore, the single-crystal X-ray diffraction experiments also indicates that the structure of the catalyst remains totally unchanged from its original state. In addition, catalytic performance of recycled catalyst was also conducted for aerobic oxidation of benzaldehyde and methanol, methyl benzoate was obtained in 98% yield.

In order to understand the mutual influence among the studied substrates, the successive CV investigation are carried out with the groups of cat. **6**, cat. **6** + H_2O_2 and $V_6 + H_2O_2$ + benzaldehyde, respectively. As shown in Fig. 5, the cyclic voltammetry of cat. **6** exhibits one well-defined oxidation wave at −84 mV which is attributed to the $V^{V/IV}$ couple. The oxidation wave of V_6 shift to negative direction from −84 mV to −205 mV with H_2O_2 addition indicating an easier oxidation process due to confirmed interaction between cat. **6** and H_2O_2. Furthermore, more negatively shift happened to the oxidation wave of cat. **6** from −205 mV to −275 mV when reaction substrate benzaldehyde added into cat. **6** + H_2O_2 solution suggesting strong activation upon benzaldehyde with V_6 catalytic system. Otherwise, the necessity of cat. **6** to the studied reaction activity is verified through the CVs of H_2O_2 + benzaldehyde on reverse cyclovoltammograms that no detectable redox waves are observed (Fig. S28 in Supporting information). Furthermore, the oxidative esterification of aldehydes can proceed *via* simple radical-based oxidation of hemiacetal. In order to confirm whether it generates free radical active species during the catalytic process, EPR spin trapping experiments investigation was used to explore the mechanism of catalytic reaction (Fig. 6), the results show an EPR signal with a g-value of 2.0023 which is caused by a free electron in the anion.

Based on previous reports in the literature [45,72] and studies by CV, EPR, single crystal X-ray diffraction and control experiments (Fig. S31 in Supporting information), a tentative mechanism for the selective oxidation of benzaldehyde and methanol is proposed (Fig. 7). Initially, *N*-heterocyclic carbene species (**I**) was generated in the presence of POV which were used as soft base to absorbed proton by pre-forming hydrogen bond (O13···H16). *N*–Heterocyclic carbene attacks benzaldehyde to form a typical Breslow intermediate (**II**) which will further generate complex (**III**) containing a α-substituted hydroxyl group. Subsequently, complex (**III**) transforms to a ketonic intermediate (**IV**), the desired esters and 6 are released *via* a nucleophilic substitution reactions between IV and methanol. In addition, POV are capable of forming HOO-POV (**V**) intermediates from hydrogen peroxide with strong oxidizing

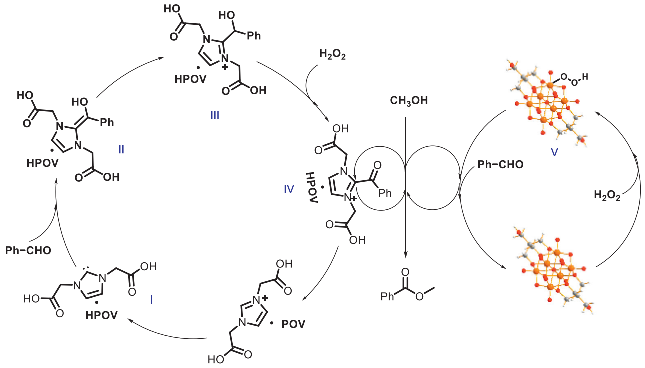


Fig. 7. Proposed mechanism for the **6**-catalyzed esterification of aldehydes with alcohols.

properties and were able to oxidize benzaldehyde and methanol to the corresponding esters.

In conclusion, we have demonstrated highly efficient selective oxidative esterification of aldehydes with alcohols catalyst by POV-based molecular catalysts which is nontoxic, green and recyclable. Supramolecular interactions between POV and Act_2Im facilitate catalysis and enabling the conversion of a wide range of aldehydes to the corresponding esters. Furthermore, such POV-based ionic liquid catalysts can be used for large scale preparation of methyl benzoate and recycled with minimal loss of the activity. The generality of this methodology gives it the potential to be used on an industrial scale

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2023.109061.

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