Supported Ruthenium and Tetrapropylammonium Bromide Catalysts for Oxidative Carboxylation of 1-Decene

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Cyclic carbonate compounds are valuable for a range of applications and can be synthesized by a one-pot reaction involving epoxidation of olefin followed by reaction of the epoxide with CO_2. This study used supported ruthenium catalysts for the epoxidation step (first step), where a combination of tetrapropylammonium bromide and zinc bromide was used for the cycloaddition of carbon dioxide. The supported ruthenium catalyst, prepared by a sol-immobilization method, allowed the effective epoxidation of 1-decene in air (using oxygen as the main oxidant) at 90 °C in the presence of a catalytic quantity of radical initiator. This approach was applied to the one-pot multi-step oxidative carboxylation of 1-decene in the presence of 1 % Ru/support-$Pr_4NBr/ZnBr_2$ catalyst.

Keywords: Ruthenium, Cyclic carbonate, Tetrapropylammonium bromide.

INTRODUCTION

Carbon dioxide has attracted increasing attention as an economical, abundant and non-toxic chemical feedstock for the synthesis of important organic chemicals. Moreover, recycling of CO_2 could help reduce the levels of CO_2 emission. Aresta and Dibenedetto [1] reported that approximately 110 million tons of CO_2 were being used each year for chemical syntheses, with 90 million tons being applied to urea production. The synthesis of salicylic acid (a precursor of aspirin) from CO_2 and phenol has been known since 1890. The application of CO_2 in processes involving organic chemicals, *e.g.* cyclic carbonates [2] and polycarbonates [3] has also been reported.

The value of cyclic carbonates to chemical industry should not be under-estimated; examples include the extensive applications of propylene and ethylene carbonates as electrolytes for lithium-ion batteries, aprotic polar solvents, starting materials for thermoplastics, monomers for polycarbonate synthesis and intermediates in pharmaceutical synthesis. Moreover, application of cyclic carbonate compounds is one of the most successful CO_2 fixation strategies [4]. Since 1962, cyclic carbonates have been directly synthesized from olefins [5]. Although there

have been separate studies exploring the epoxidation and cyclo-addition of CO_2 to an epoxide, although the literature describing integrated approaches is limited [1,6-10]. In those studies, the oxidants used tend to be either *tert*-butyl hydroperoxide (TBHP) or hydrogen peroxide [7,9,11-13]. However, even though its use presents challenges, molecular oxygen is a preferable oxidant because it is 'green' and has superior atom efficiency. The work published by Aresta *et al.* [7,14] used oxygen in the oxidative carboxylation of styrene, in the presence of either homogeneous rhodium complexes or various metal oxides as catalysis. The chosen solvent in their study, dimethylformamide (DMF), was not inert for the epoxidation of alkenes. DMF has been reported to be an oxygen transfer agent, generating significant quantities of by-products, such as N-formyl-N-methyl-formamide [15]; this abandon the environmental advantages conferred by using oxygen as the oxidant. Bai and Jing [4] used a homogeneous metalloporphyrin catalyst to convert different olefins to their corresponding cyclic carbonates and in case of styrene, the yield of cyclic carbonate was 89 %. Kumar *et al.* [16] used isobutyraldehyde as a sacrificial reductant in the direct oxidative carboxylation of alkenes using an immobilized magnetic chitosan catalyst of cobalt(II) acetylacetonate complex and quaternary triphenylphosphonium bromide. The sequential

introduction of reaction gases, oxygen and carbon dioxide to the reaction mixture, resulting in propylene and butylene starting materials with yields of up to 85 %.

A key factor in the integration of two stages is that the reaction conditions and the catalysts for each step must be compatible. It has been reported that the catalyst used in the epoxidation step, which in this instance is $MoO_2(acac)_2$ was deactivated by tetrabutylammonium bromide catalyst in the cycloaddition reaction of CO_2 to epoxides. The mechanism considered responsible for this phenomenon is the degradation of $MoO_2(acac)_2$ and the primary oxidant, TBHP by bromide [11]. By adding tetrabutylammonium bromide after the completion of epoxidation reaction, researchers were able to surmount this drawback.

This study explored the possibility of integrating the epoxidation of alkenes and cycloaddition of CO_2 to epoxides steps of the reaction. To investigate the epoxidation aspect of the process, highly active supported ruthenium catalysts were used with 1-decene and for the cycloaddition of CO_2 to 1,2-epoxydecane, a Pr_4NBr and $ZnBr_2$ catalytic system was used under solvent free conditions. As reported [10], an epoxide selectivity was significantly reduced in the presence of tetrabutylammonium bromide and no effect of supported gold catalysts was observed on the cycloaddition of CO_2 with 1,2-epoxydecane. Therefore, a one-pot, two-step approach for the oxidative carboxylation of 1-decene, is carried out. To our best of knowledge, this is the first study using supported ruthenium catalyst for epoxidation of alkene under these green conditions.

EXPERIMENTAL

The chemicals employed in the present work were acquired from commercial suppliers and used without additional purification. Loading of metal catalyst on the support was stated in terms of metal percentage by weight of support. Both the wet-impregnation method and the sol-immobilization method were applied to catalyst preparation in the present work.

Sol immobilization method: The sol-immobilization method as described by Jones *et al.* [17] was used to prepare the supported Ru catalysts (2 g). In brief, an appropriate quantity of $RuCl_3 \cdot xH_2O$ (Sigma-Aldrich) was added to deionized water (800 mL) with continuous stirring. To protect and stabilize the Ru nanoparticles, a freshly prepared 1 wt.% solution of poly(vinyl alcohol) (*m.w.* = 10 000, 80% hydrolyzed) was added (PVA/Ru (by wt) = 0.65). After a further 15 min of stirring, a dark brown sol was generated by the addition of a freshly prepared solution of sodium borohydride (0.2 M; molar ratio $NaBH_4$/Ru = 5). The sol was stirred for a further 30 min with dropwise addition of H_2SO_4 to adjust the acidity to pH = 2. The TiO_2 support (~1.98 g) was then added and the mixture stirred for 2 h prior to wash thoroughly with deionized water (2 L) and dried at 110 °C for 16 h.

Wet-impregnation method: In the wet impregnation method, catalyst (1 g) was prepared by dissolving an appropriate quantity of $RuCl_3 \cdot xH_2O$ in deionized water and added an appropriate amount of TiO_2 support and allowed water to evaporate with continuous stirring at 80 °C. The obtained paste was dried for 16 h at 110 °C and grounded prior to calcination for 3 h in static air at 300 °C (heating rate = 20 °C/min).

Catalyst testing: For a typical epoxidation reaction, catalyst (0.1 g) was added to 1-decene (53 mmol, 10 mL) in a 50 mL round-bottomed glass flask equipped with a reflux condenser. A radical initiator *tert*-butyl hydroperoxide (0.01 mL) was added and the reaction mixture was heated to 90 °C on a hot-plate with magnetic stirring. At the end of selected reaction time, a mixture was cooled to room temperature and filtered prior to analysis using gas chromatography.

Following the above epoxidation step, a reaction mixture was transferred to a stainless-steel autoclave (50 mL) with a Teflon inner lining for the one-pot synthesis of cyclic carbonates. After addition of co-catalysts tetrapropyl ammonium bromide (Pr_4NBr, 0.4 g) and $ZnBr_2$ (0.16 g), a reaction vessel was sealed and the cycloaddition reaction completed under 10-20 bar CO_2 for 4 h. After cooling the reactor in an ice-bath and depressurising, the catalyst was obtained by filtration. The reaction products and unreacted 1-decene were examined using a Varian star 3400 CX GP system with a CP wax 52 capillary column (25 m, 0.35 mm ID, 0.2 micron) coupled with an FID detector.

RESULTS AND DISCUSSION

Epoxidation of 1-decene using 1 % Ru/TiO₂ catalyst: If two reactions are to be combined in a one-pot process, the compatibility of the two reactions must first be examined. Hence, in step 1 (epoxidation of 1-decene) and step 2 (cycloaddition of 1,2-epoxydecane to CO_2), were first examined individually in order to examine the roles of various catalysts. The aim of the initial work was to investigate the effect of using supported ruthenium catalyst for the epoxidation of 1-decene using oxygen from air as primary oxidant at atmospheric pressure as a first step in this one-pot reaction. Unlike the oxidation of internal alkenes, the oxidation of linear terminal alkenes necessitates the use of a radical initiator [10]. In the present work, TBHP was selected in this role.

1 % Ru/TiO₂ has been selected to study epoxidation of 1-decene under solvent-free conditions. The reaction was conducted for 24 h at 90 °C in air with catalytic amount of TBHP. First, the blank reaction was tested in the presence of TBHP only, which showed a low activity in the epoxidation reaction (2 % conversion) and only 10 % selectivity to 1,2- epoxydecane (Fig. 1). TiO_2 also showed a low conversion of 1-decene (4 %) and only 16 % epoxide selectivity. Significant increase in epoxide yield has been achieved in the presence of 1 % Ru/ TiO_2, which prepared using sol-immobilization method, where the conversion of 1-decene increase from 4 to 16 % and the epoxide selectivity increase from 16 to 37 % (Fig. 1).

It is well known that high selectivity to epoxide in epoxidation reaction remains a challenge, therefore number of other byproducts were detected and quantified. A number of products formed during the reaction was reported earlier [10,18]. As significant quantities of allylic products were formed during the reaction of 24 h, it would be desired to analyze product profile during the reaction course, as some products may be formed during oxidation of others. In order to understand the detailed reaction profile of 1-decene epoxidation over 1 % Ru/ TiO_2 catalyst, time online studies have been carried out for 96 h.
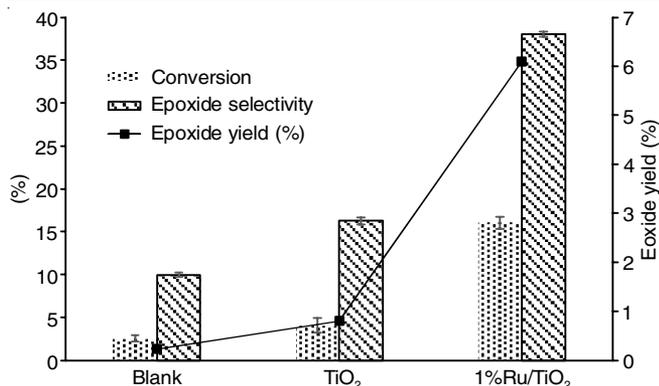
Fig. 1.   Effect of TiO$_2$ and 1 % Ru/TiO$_2$ on 1-decene epoxidation. Reaction conditions: catalyst (0.1 g), 1-decene (53 mmol, 10 mL), TBHP (0.064 mmol, 0.01 mL), 90 °C, atmospheric pressure air, reaction time 24 h, rate of stirring 900 rpm. Error bars indicate range of data based on three repeat experiments

As indicated in Fig. 2, conversion of 1-decene progressively increased from 1 to 35 % as the reaction time was increased from 4 to 96 h with no induction period. Hence, an increase of 1-decene conversion is expected with longer reaction runs. Moreover, while a low selectivity for the epoxide was observed at the beginning of the reaction (with allylic compounds, *e.g.* 1-decen-3-ol, 1-decen-3-one, 2-decen-1-ol and 2-decenal) forming the major products, the epoxide selectivity increased with increasing reaction time up to 48 h. For reaction times greater than 48 h, however, the epoxide selectivity steadily decreased to 18 % at 96 h. This is generally attributed to the ring opening reaction of the epoxide with water produced as a byproduct of condensation reactions or from the decomposition of hydroperoxy intermediate to allylic ketone and water as described [18]. Hydrolysis of epoxide to diol follows readily once water has formed. Similar increases in alkene conversion and epoxide selectivity with increasing reaction times were reported for the terminal alkenes 1-decene, 1-hexene and 1-octene [18,19].

Effect of preparation method: One of the important factors, which can affect the activity of a catalyst is the preparation
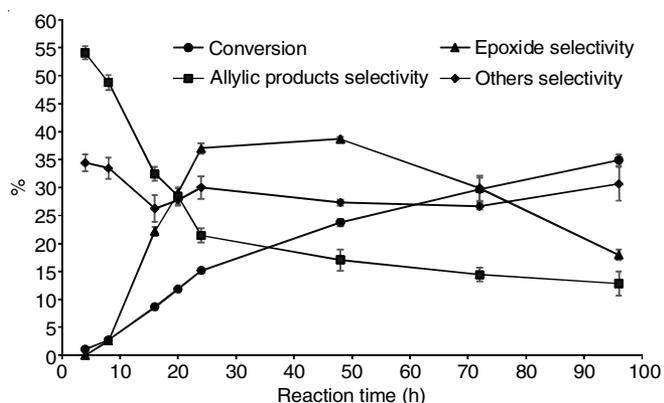

Fig. 2.   Effect of reaction time on conversion and selectivity. Reaction conditions: 1 % Ru/TiO$_2$ (0.1 g), 1-decene (53 mmol, 10 mL), TBHP (0.064 mmol, 0.01 mL), 90 °C, atmospheric pressure air, rate of stirring 900 rpm. Allylic products = Σ (1-decen-3-one, 1-decen-3-ol, 2-decenal, 2-decen-1-ol). Others = Σ (C$_7$ + C$_8$ + C$_9$ acids, C$_8$ + C$_9$ aldehyde, C$_7$ + C$_8$ alcohols, 3-nonen-1-ol, 3-nonanone, cyclo-dodecane, 2-decenoic acid). Error bars indicate range of data based on three repeat experiments

method [20]. Fig. 3 displays that 1 % Ru/TiO$_2$ catalyst prepared using two different preparation methods and so-immobilization was more active for the epoxidation of 1-decene compared to wet impregnation method. Conversion and epoxide selectivity are 15 % and 37 %, respectively when using so-immobilization as preparation method for 1 % Ru/TiO$_2$, whereas a conversion and epoxide selectivity were 10 % and 29 % with wet-impregnation method.
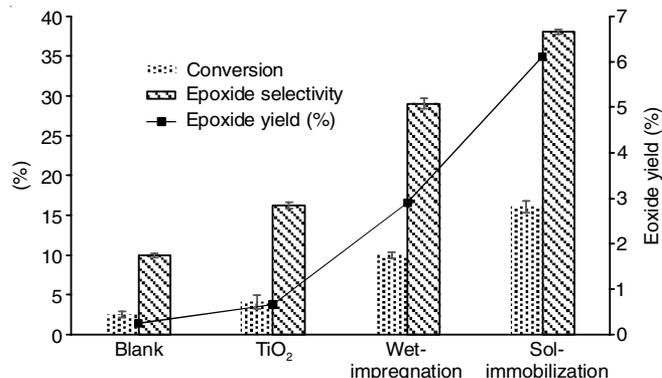

Fig. 3.   Effect of the catalyst preparation method on 1-decene oxidation. Reaction conditions: 1 % Ru/TiO$_2$ (0.1 g), 1-decene (53 mmol, 10 mL), TBHP (0.064 mmol, 0.01 mL), 90 °C, atmospheric pressure air, rate of stirring 900 rpm. Allylic products = Σ (1-decen-3-one, 1-decen-3-ol, 2-decenal, 2-decen-1-ol). Others = Σ (C$_7$ + C$_8$ + C$_9$ acids, C$_8$ + C$_9$ aldehyde, C$_7$ + C$_8$ alcohols, 3-nonen-1-ol, 3-nonanone, cyclododecane, 2-decenoic acid). Error bars indicate range of data based on three repeat experiments

Reusability: To examine the reusability, above reaction was performed using the 1 % Ru/TiO$_2$ catalysts (prepared by sol-immobilization) in an excess quantity. At the end of the reaction, a catalyst was filtered, washed with acetone and dried in an oven at 110 ºC for 16 h, after which the required quantity of catalyst for a normal reaction was taken for reuse. The activity data for fresh and reused catalysts are presented in Table-1. It can be seen that the reaction with fresh catalyst achieved a 15 % conversion of 1-decene and a 37 % epoxide selectivity, whereas reused catalyst (when dried without prior washing) displayed diminished activity and did not allow effective reuse. This can be attributed to catalyst deactivation due to the presence of adsorbed reaction products. Hence, when used catalyst was washed with acetone prior to reuse, increased activity relative to the unwashed catalyst is indicated by a 10 % conversion and 24 % epoxide selectivity. Nevertheless, the washed and reused catalyst displayed lower activity than the fresh catalyst, probably due to the blocking of active sites by carbon.

Although the main difficulty with heterogeneous catalysts, especially in the liquid phase, is the leaching of active components into the solution. However, in present case, ICP analysis indicated no leaching of ruthenium.

Cycloaddition of carbon dioxide to 1,2-epoxydecane: According to He *et al.* [21], synthesis of cyclic carbonates *via* cycloaddition of carbon dioxide to an epoxide generally involves the use of quaternary ammonium salts as homogeneous catalysts. A synergistic effect of Lewis acids (*e.g.* ZnBr$_2$) and Lewis bases upon the catalytic cyclic carbonates synthesis from epoxides and CO$_2$ has previously been reported [22], in

TABLE-1
CATALYST REUSABILITY STUDY FOR EPOXIDATION OF 1-DECENE: 1 % Ru/TiO$_2$

Washing conditions	Conversion (%)	Epoxide selectivity (%)
Fresh catalyst	15	37
Reused without washing, dried static air at 110 °C for 16 h	7	17
Reused and washed with acetone (200 mL), dried static air at 110 °C for 16 h	10	24

Reaction conditions: 1 % Ru/TiO$_2$ (0.1 g), 1-decene (53 mmol, 10 mL), TBHP (0.064 mmol, 0.01 mL), 90 °C, atmospheric pressure air, reaction time 24 h, rate of stirring 900 rpm

which the Lewis acid activated the epoxide and the quaternary ammonium salt facilitated the epoxide ring opening. When various molar ratios of ammonium salt (Lewis base) to Lewis acid were examined, the optimum molar ratio was shown to be 2:1. In the present study, quaternary ammonium salt tetra-propylammonium bromide was used at appropriate reaction temperature for the epoxidation step (90 ºC). An increase in the conversion of 1,2-epoxydecane to the corresponding cyclic carbonates with increasing reaction time is evident in Table-2, with 65 % conversion at 1 h and close to 100 % conversion within 3 h. Significantly, a selectivity maintained similar values (95-98 %) throughout the reaction.

TABLE-2
EFFECT OF THE REACTION TIME

Reaction time (h)	Conversion (%)	Cyclic carbonate selectivity (%)	Cyclic carbonate yield (%)[a]
1	65	95	44
2	85	95	61
3	97	98	74

Reaction conditions: Pr$_4$NBr (0.75 mmol, 0.2 g), ZnBr$_2$ (0.355 mmol, 0.08 g), 1,2-epoxydecane (26.88 mmol, 5 mL), 90 °C, 20 bar CO$_2$ rate of stirring 900 rpm. (a) Yield obtained by using GC analysis.

An influence of CO$_2$ pressure upon cyclic carbonates synthesis in the presence of Pr$_4$NBr and ZnBr$_2$ catalysts is shown in Table-3. As the pressure of CO$_2$ increased from 10 to 20 bar, the cyclic carbonates selectivity increased from 91 to 98 % with a concurrent and significant increase in the conversion of epoxide from 73 to 97 %.

TABLE-3
EFFECT OF CO$_2$ PRESSURE ON CYCLIC CARBONATE SYNTHESIS

CO$_2$ pressure (bar)	Conversion (%)	Cyclic carbonate selectivity (%)	Cyclic carbonate yield (%)[a]
10	73	91	66
15	90	94	84
20	97	98	95

Reaction conditions: Pr$_4$NBr (0.75 mmol, 0.2 g), ZnBr$_2$ (0.355 mmol, 0.08 g), 1,2-epoxydecane (26.88 mmol, 5 mL), 90 °C, reaction time 3 h, rate of stirring 900 rpm. [a]Yield obtained by using GC analysis.

Oxidative carboxylation of 1-decene using 1 % Ru/TiO$_2$-Pr$_4$NBr/ZnBr$_2$: Previous work has indicated that the one-pot simultaneous (single-step) oxidation and carboxylation is less successful than the two-step reaction (epoxidation followed by one-pot carboxylation) [10]. The multistep approach was therefore used for the oxidative carboxylation of 1-decene in the presence of 1 % Ru/TiO$_2$-Pr$_4$NBr/ZnBr$_2$. Upon completion of epoxidation reaction, the products were transferred to the batch reactor, followed by addition of CO$_2$, Pr$_4$NBr and ZnBr$_2$. As shown in Table-4, an epoxide formed with 37 % selectivity was thus converted to cyclic carbonate with 24 % selectivity. No remarkable effect of increasing reaction time upon cyclic carbonates selectivity was observed; normally cyclic carbonates were generated whenever the 1,2-epoxydecane yield is high. This suggests that an efficiency of one-pot cyclic carbonates synthesis is primarily determined by the performance of supported ruthenium catalyst. Hence, catalysts with greater activity and selectivity towards 1-decene epoxidation reaction would be more effective for the one-pot multistep reaction (sequential oxidation and carboxylation).

Conclusion

A low cost synthetic protocol for olefins makes their oxidative carboxylation reactions significantly economical. The epoxidation of 1-decene reaction was conducted in the presence of supported ruthenium catalyst and Pr$_4$NBr/ZnBr$_2$ catalyze the cycloaddition reaction. 1 %Ru/TiO$_2$ prepared using sol-immobilisztion method showed a higher activity for the epoxidation of 1-decene. The one-pot multistep become a highly efficient preparation method of cyclic carbonates (sequential oxidation and carboxylation).

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

TABLE-4
DIRECT SYNTHESIS OF CYCLIC CARBONATE STARTING FROM 1-DECENE USING 1 % Ru/SUPPORT-Pr$_4$NBr/ZnBr$_2$

Reaction starting with 1-decene	Temp. (°C)	1-Decene conversion (%)	Selectivity (%)	
			Epoxide	Cyclic carbonate
One pot reaction (cycloaddition step 4 h)	90	15	4 (37)[a]	24
One pot reaction (cycloaddition step 6 h)	90	16	3	24
One pot reaction (cycloaddition 8 h)	90	16	3	25

Reaction conditions: 1 % Ru/SiO$_2$ (0.1 g), Pr$_4$NBr (1.5 mmol, 0.4 g), ZnBr$_2$ (0.71 mmol, 0.16 g), 1-decene (53 mmol, 10 mL), TBHP (0.064 mmol, 0.01 mL), 24 h reaction time for epoxidation, 20 bar CO$_2$ for the one-pot multi step, rate of stirring 900 rpm. [a]Selectivity for the epoxide in the epoxidation reaction.

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