DE GRUYTER
Green Process Synth 2014; 3: 411–418

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Greener route to 4-vinyl-1-cyclohexane 1,2-epoxide synthesis using batch and continuous reactors

Abstract: Polystyrene 2-(aminomethyl)pyridine (Ps.AMP) supported molybdenum (Mo)(VI) complex (Ps.AMP.Mo) was prepared, characterized and assessed as a catalyst for batch and continuous epoxidation of 4-vinyl-1-cyclohexene (4-VCH) using *tert*-butyl hydroperoxide (TBHP) as an oxidant. The effect of various parameters such as reaction temperature, feed molar ratio (FMR) of 4-VCH to TBHP and catalyst loading on the conversion of TBHP to 4-vinyl-1-cyclohexane 1,2-epoxide (4-VCH 1,2-epoxide) was studied to optimize reaction conditions in a batch reactor. The long-term stability of Ps.AMP.Mo was evaluated by recycling a sample of the catalyst several times in batch experiments. A detailed evaluation of Mo leaching from the polymer supported catalyst was investigated by isolating any residue from reaction supernatant solutions and then using these residues as potential catalysts in epoxidation reactions. The efficiency of Ps.AMP.Mo catalyst for continuous epoxidation studies was assessed using a FlowSyn continuous flow reactor by studying the effect of reaction temperature, feed flow rate and FMR of 4-VCH to TBHP on the conversion of TBHP and the yield of 4-VCH 1,2-epoxide. The experimental results confirmed very high selectivity and efficiency of Ps.AMP.Mo catalyst for batch and continuous epoxidation.

Keywords: 4-vinyl-1-cyclohexene; alkene epoxidation; continuous flow reactor; heterogeneous catalysis; polymer supported Mo(VI) catalyst.

DOI 10.1515/gps-2014-0058
Received July 28, 2014; accepted September 17, 2014

Abbreviations

FMR	feed molar ratio
GC	gas chromatography
Mo	molybdenum
Ps.AMP	polystyrene 2-(aminomethyl) pyridine
Ps.AMP.Mo	polystyrene 2-(aminomethyl) pyridine supported Mo(VI) complex
TBHP	*tert*-butyl hydroperoxide
t	time (min)
4-VCH	4-vinyl-1-cyclohexene
4-VCH 1,2-epoxide	4-vinyl-1-cyclohexane 1,2-epoxide

1 Introduction

Epoxides are key raw materials or intermediates in organic synthesis, particularly for the functionalization of substrates and production of a wide variety of chemicals such as pharmaceuticals to plastics and paints to adhesives [1–3]. The conventional methods of epoxides production within industries employ either peracetic acid [4] or chlorohydrin [5] as oxidation reagents in liquid phase batch reactions. However, such processes are not environmentally benign as the former produces an equivalent amount of acid waste, whilst the latter yields chlorinated by-products and calcium chloride waste. In recent years, more and more attention has been focused on developing greener and more efficient epoxidation processes, employing environmentally benign oxidants such as *tert*-butyl hydroperoxide (TBHP), since it is atom efficient, environmentally benign and safer to handle [6, 7]. A notable industrial implementation of alkene epoxidation with TBHP was the Halcon process described by Kollar (1967) [8]. This process uses homogenous molybdenum (Mo)(VI) to catalyze liquid phase epoxidation of propylene for the production of propylene oxide. However, homogenous catalyzed epoxidation processes are not economically viable for industrial applications due to major

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requirements in terms of work-up, product isolation and purification procedures.

Consequently, researchers have focused on developing stable heterogeneous catalysts for epoxidation by immobilization of catalytically active metal species on organic or inorganic materials [9–11]. Polymers have gained attention as suitable support for transition metal catalysts as they are inert, nontoxic, insoluble and often recyclable [12]. A number of chelating functional Mo-containing copolymers have been prepared and used as catalysts for the epoxidation of alkenes in the presence of TBHP as an oxidant [13–15]. Despite numerous published works on polymer supported Mo(VI) catalyzed alkene epoxidation with TBHP, there appears to have been no significant effort to move the chemistry on from small-scale laboratory batch reaction to medium or large scale production technology in continuous flow process.

In this work, an efficient and selective polystyrene 2-(aminomethyl)pyridine (Ps.AMP) supported Mo(VI) complex (Ps.AMP.Mo) was used as a catalyst for batch and continuous epoxidation of 4-vinyl-1-cyclohexene (4-VCH) using environmentally benign TBHP as an oxidant. An extensive assessment of the catalytic activity, stability and reusability of Ps.AMP.Mo catalyst was conducted in a classical batch reactor. Experiments were carried out to study the effect of catalyst loading, feed molar ratio (FMR) of 4-VCH to TBHP and reaction temperature on the yield of 4-vinyl-1-cyclohexane 1,2-epoxide (4-VCH 1,2-epoxide) to optimize the reaction conditions in a batch reactor. A detailed evaluation of Mo leaching from the polymer supported catalyst was investigated by isolating any residue from reaction supernatant solutions and then using these residues as potential catalysts in epoxidation reactions. Furthermore, the efficiency of Ps.AMP.Mo catalyst for continuous epoxidation studies was assessed using a FlowSyn continuous flow reactor by studying the effect of reaction temperature, FMR of 4-VCH to TBHP and feed flow rate on the conversion of the oxidant and the yield of 4-VCH 1,2-epoxide. The continuous flow epoxidation using the FlowSyn reactor has shown considerable time savings, high reproducibility and selectivity in addition to remarkable improvements in catalyst stability compared to reactions carried out in a batch reactor.

2 Materials and methods

2.1 Materials

All chemicals used for this study were purchased from Sigma-Aldrich Co. Ltd. (Gillingham, Dorset, UK) and the purity of each chemical was verified by gas chromatography (GC). The alkene involved in this study was 4-VCH (≥99.5) and the oxidant employed was TBHP solution in water (70% w/w). The water content of TBHP was removed by Dean-Stark apparatus from the toluene solution following a modified procedure that was previously reported by Sharpless and Verhoeven [16].

2.2 Preparation of Ps.AMP.Mo catalyst

The synthesis of Ps.AMP.Mo was carried out by suspension polymerization of known mixtures of divinylbenzene [12% (w/w)], vinylbenzyl chloride [25% (w/w)] and styrene [63% (w/w)] in the presence of 2-ethylhexanol as a porogen. This was followed by amination of the formed resin by refluxing with an excess of 2-(aminomethyl)pyridine in ethanol to form Ps.AMP resin. The Mo was loaded onto the Ps.AMP resin by reaction with an excess of molybdenyl acetylacetonate under reflux in toluene for 4 days. The excess molybdenyl acetylacetonate was removed by exhaustive extraction with acetone in a Soxhlet, replacing the earlier blue-colored solvent with fresh batches until the refluxing solvent remained colorless. The final product (Ps.AMP.Mo) was recovered by filtration and dried under vacuum at 40°C. The crosslinked resin beads are commonly used as catalyst support due to their high porosity, large surface area and robust spherical particles with uniform size distribution [17]. The properties of the prepared Ps.AMP.Mo catalyst are given elsewhere [18].

2.3 Batch and continuous epoxidation studies

Batch epoxidation of 4-VCH with TBHP as an oxidant in the presence of Ps.AMP.Mo catalyst was carried out in a jacketed four necked glass reactor of 0.25 l capacity. A FlowSyn reactor (supplied by Uniqsis Ltd., Shepreth, Cambridgeshire, UK) was used to conduct continuous epoxidation experiments. It was equipped with two independent HPLC pumps, a control interface and SquirrelView software with data logger (supplied by Grant Instruments Ltd., Shepreth, Cambridgeshire, UK). The catalytic fixed bed was made of a stainless steel column of length 130 mm (internal diameter 7 mm and outer diameter 10 mm). All of the reaction parameters including reaction temperature, feed flow rate and pressure limits were set using the control interface of the FlowSyn reactor. The stainless steel fixed bed reactor was packed with the Ps.AMP.Mo catalyst (1.5±0.02 g) and enclosed in an electronically controlled column heater. The SquirrelView software and data logger precisely monitor and record the temperature profile of the mobile phase in the column.

Before starting a continuous epoxidation experiment, 4-VCH was fed continuously by an HPLC pump for a desired period until the column reactor and tubings were completely saturated. The heating to the column was set to the required value and was allowed to reach the desired temperature. Once the column was saturated with alkene and the desired temperature achieved, the continuous epoxidation experiment was started. The reactants (4-VCH and TBHP) were continuously fed at a desired flow rate by two HPLC pumps to the packed column via a mixing chamber. As soon as the reactants entered the column, they reacted in the presence of Ps.AMP.Mo catalyst to produce epoxide. The fluid leaves the outlet port of the packed column and is collected by a fraction collector at regular time intervals. The samples from both batch and continuous alkene epoxidation studies were taken at specific time intervals and were analyzed by Shimadzu GC-2014 gas chromatograph.

3 Results and discussion

3.1 Batch epoxidation results

Batch epoxidation experiments were conducted under different reaction conditions to study the effect of various parameters such as reaction temperature, FMR of 4-VCH to TBHP and catalyst loading on the yield of 4-VCH 1,2-epoxide. Reusability and supernatant studies were carried out to evaluate the long term stability of the polymer supported catalyst for epoxidation reactions. Samples were collected for each batch experiment at regular time intervals and analyzed by GC. The analytical error was found to be within ±3% for all experiments conducted in a batch reactor.

3.1.1 Effect of reaction temperature

Alkene epoxidation with alkyl hydroperoxides require a thorough screening of the reaction temperature in order to achieve high conversion of the oxidant and high product selectivity. Hence, experiments were conducted to study the effect of different reaction temperatures on the yield of epoxide. Reaction temperatures used for this study were 60°C, 70°C, 80°C and 90°C. As expected, higher reaction temperatures gave a higher yield of 4-VCH 1,2-epoxide at a fixed reaction time. It can be seen from Figure 1 that the reaction reached equilibrium within the first 5 min for experiments carried out at 80°C due to the distinct exothermic effect observed during the first 5 min of the reaction, i.e. the temperature of the reaction mixture overshoots the desired temperature, which was controlled immediately to maintain the reaction mixture at 80°C. This effect, however, was not significant when the experiments were conducted at 60°C and 70°C. The experiments were replicated twice for all three temperatures, i.e. 60°C, 70°C and 80°C and the same behavior was observed in all cases. When the epoxidation reaction was conducted at 90°C, it was difficult to collect samples because of the uncontrollable exothermic nature of this reaction and difficulty in maintaining a constant reaction temperature. As a result, the experiment conducted at 90°C was not reported. By contrast, the trends for the yield of 4-VCH 1,2-epoxide for experiments conducted at 60°C and 70°C were quite similar. A significantly higher yield of 4-VCH 1,2-epoxide was obtained at 80°C as compared to the experiments conducted at 60°C and 70°C. The yield of 4-VCH 1,2-epoxide after 5 min at 60°C, 70°C and 80°C was ~18%, ~28% and ~94%, respectively. By contrast, the yield of 4-VCH 1,2-epoxide at 260 min was ~95% for all three temperature ranges. Ambroziak et al. [14] observed similar exothermic effects while studying the effect of reaction temperature on cyclohexene epoxidation with TBHP catalyzed by polymer-supported Mo(VI) complex. It is evident from Figure 1 that ~95% yield of 4-VCH 1,2-epoxide was achieved at 10 min for reactions at 80°C. Since it was not possible to conduct the epoxidation experiment beyond 80°C in a controlled manner, 80°C was considered to be the optimum reaction temperature for this process.

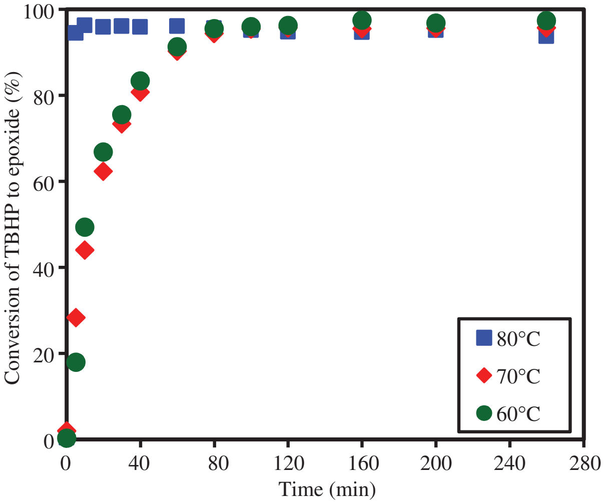

Figure 1 Effect of reaction temperature on the conversion of *tert*-butyl hydroperoxide (TBHP) to 4-vinyl-1-cyclohexane 1,2-epoxide (4-VCH 1,2-epoxide) for epoxidation of 4-VCH with TBHP catalyzed by polystyrene 2-(aminomethyl)pyridine supported Mo(VI) complex (Ps.AMP.Mo) catalyst using a batch reactor at feed molar ratio (FMR) of 4-VCH to TBHP: 5:1; catalyst loading: 0.3 mol% Mo; stirrer speed: 400 rpm.

3.1.2 Effect of FMR

The efficiency in the use of oxidant and its conversion to the desired product is a major criterion for assessing the proficiency of alkene epoxidation with alkyl hydroperoxide. Batch experiments were carried out at 2.5:1, 5:1 and 10:1 molar ratio of 4-VCH to TBHP to study the effect of different FMRs of 4-VCH to TBHP on the yield of 4-VCH 1,2-epoxide. No significant effect in the rate of formation of epoxides was found when the FMR of 4-VCH to TBHP was increased beyond 2.5:1, as shown in Figure 2. For instance, the yield of 4-VCH 1,2-epoxide obtained at 260 min for 2.5:1, 5:1 and 10:1 FMR of 4-VCH to TBHP was ~92%, ~96% and ~98%, respectively. However, the reaction reached equilibrium at 80 min for an FMR of 4-VCH to

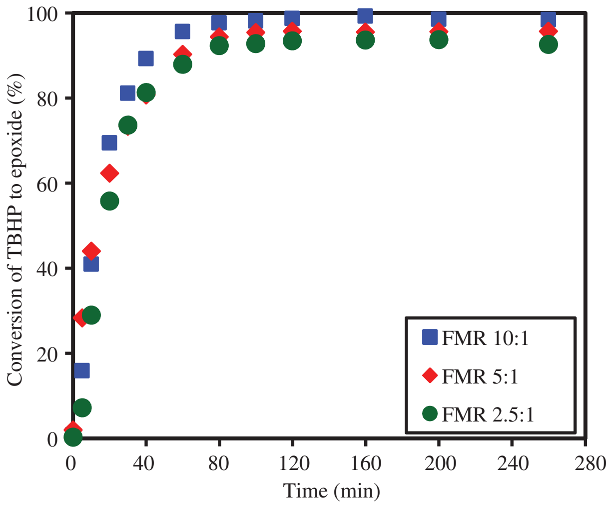

Figure 2 Effect of feed molar ratio (FMR) on the conversion of *tert*-butyl hydroperoxide (TBHP) to 4-vinyl-1-cyclohexane 1,2-epoxide (4-VCH 1,2-epoxide) for epoxidation of 4-VCH with TBHP catalyzed by polystyrene 2-(aminomethyl)pyridine supported Mo(VI) complex (Ps.AMP.Mo) catalyst using a batch reactor at reaction temperature: 70°C; catalyst loading: 0.3 mol% Mo; stirrer speed: 400 rpm.

TBHP of 10:1, and this is considered as the optimum FMR for this study.

3.1.3 Effect of catalyst loading

An increase in catalyst loading increases the number of active sites per unit volume of reactor, leading to an increase in the yield of 4-VCH 1,2-epoxide. The effect of catalyst loading was investigated by conducting batch experiments using 0.15 mol% Mo, 0.3 mol% Mo and 0.6 mol% Mo loading. In this research, catalyst loading was defined based on the active Mo component instead of total mass of Ps.AMP.Mo catalyst, due to the slight differences in Mo content obtained from different batches of the prepared Ps.AMP.Mo catalyst. However, all batch and continuous studies in this work were conducted from the same batch of the prepared catalyst. The rate of epoxide formation for experiments carried out with 0.6 mol% Mo recorded a significantly high yield of 4-VCH 1,2-epoxide compared to 0.15 mol% Mo and 0.3 mol% Mo as shown in Figure 3. By contrast, despite the equilibrium being reached within the first 20 min for catalyst loading of 0.6 mol% Mo and within 100 min for 0.3 mol% Mo catalyst loading, the yield of 4-VCH 1,2-epoxide at 260 min for catalyst loading of 0.3 mol% Mo and 0.6 mol% Mo was found to be similar, i.e., ~95%. It should be noted that 92% yield of epoxide was achieved at 20 min for catalyst loading 0.6 mol% Mo and this is regarded as the preferred catalyst loading for epoxidation of 4-VCH with TBHP using the Ps.AMP.Mo catalyst.

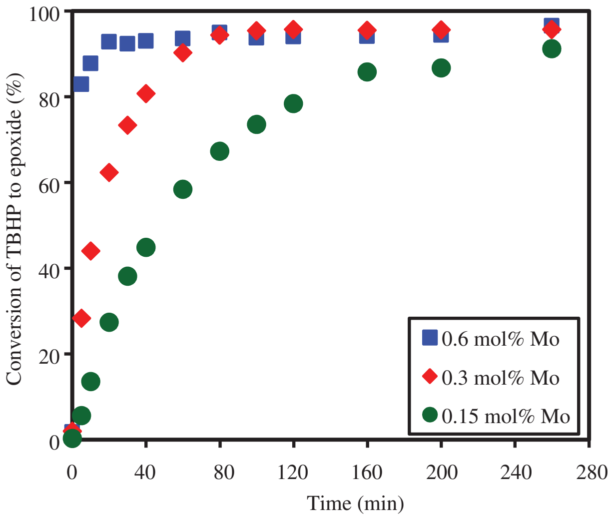

Figure 3 Effect of catalyst loading on the conversion of *tert*-butyl hydroperoxide (TBHP) to 4-vinyl-1-cyclohexane 1,2-epoxide (4-VCH 1,2-epoxide) for epoxidation of 4-VCH with TBHP catalyzed by polystyrene 2-(aminomethyl)pyridine supported Mo(VI) complex (Ps.AMP.Mo) catalyst using a batch reactor at a reaction temperature: 70°C; feed molar ratio (FMR) of 4-VCH to TBHP: 5:1; stirrer speed: 400 rpm.

3.1.4 Catalyst reusability and supernatant studies

The reusability potentials of Ps.AMP.Mo catalyst were evaluated by recycling the catalyst several times for batch experiments. In this study, fresh Ps.AMP.Mo catalyst was used in epoxidation experiments and plotted as Run 1 (see Figure 4A). At the end of the experiment, i.e. Run 1, the catalyst particles were filtered from the reaction mixture, washed carefully with 1,2-dichloroethane and stored in a vacuum oven at 40°C, while the filtrate obtained was vacuum distilled to recover the residue of the catalyst particles. The obtained residue from Run 1 was used as a potential catalyst for epoxidation known as supernatant studies and plotted as 'After Run 1' (see Figure 4B), to evaluate the extent of Mo leaching from the polymer supported catalyst. The stored Ps.AMP.Mo catalyst particles were reused in the subsequent experiment and plotted as Run 2 and the residue obtained from Run 2 was employed as the catalyst for epoxidation reaction and plotted as 'After Run 2'. This procedure was repeated for the successive catalyst reusability and supernatant studies experiments.

Figure 4A shows that a high rate of epoxide formation was observed in Run 1 as compared to the subsequent runs. This is due to the availability of sufficient active sites in the fresh catalyst that are available for adsorption by the reacting species as compared to subsequent runs. The yields of 4-VCH 1,2-epoxide for Run 2 and Run

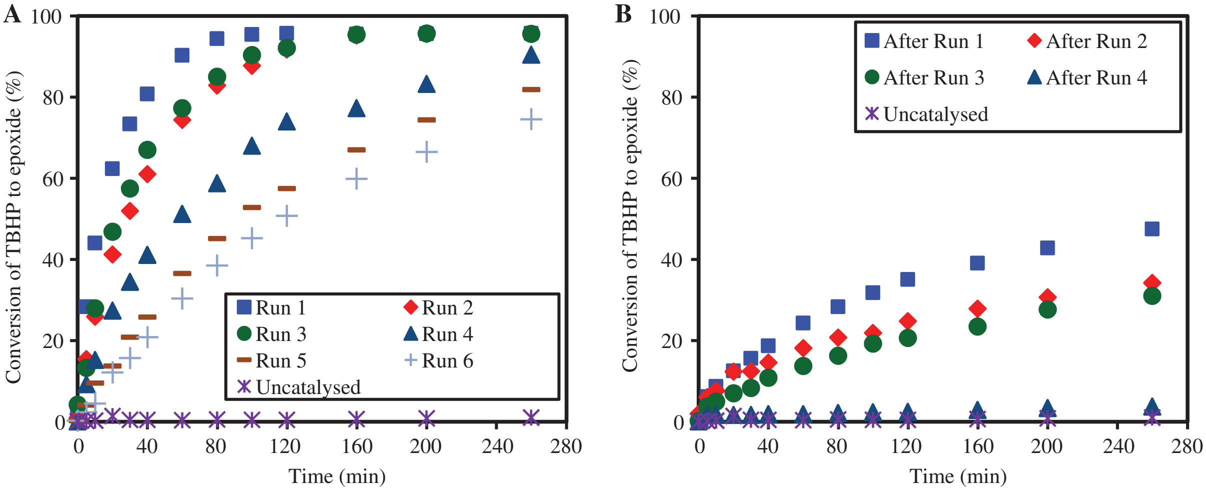

Figure 4 (A) Catalyst reusability studies of polystyrene 2-(aminomethyl)pyridine supported Mo(VI) complex (Ps.AMP.Mo) catalyst for 4-vinyl-1-cyclohexane (4-VCH) epoxidation with *tert*-butyl hydroperoxide (TBHP) using a batch reactor at reaction temperature: 70°C; catalyst loading: 0.3 mol%; feed molar ratio (FMR) of 4-VCH to TBHP: 5:1; stirrer speed: 400 rpm; (B) supernatant studies of 4-VCH epoxidation with TBHP catalyzed by residues isolated from supernatant solutions of Ps.AMP.Mo catalyst using a batch reactor at reaction temperature: 70°C; FMR of 4-VCH to TBHP: 5:1; stirrer speed: 400 rpm.

3 were similar, as both reached equilibrium at 160 min. However, a gradual decrease in the rate of epoxidation was observed in the subsequent experimental runs (Runs 4–6). The yields of 4-VCH 1,2-epoxide for Runs 4, 5 and 6 at 260 min were ~90%, ~81% and ~74%, respectively. Supernatant studies of Ps.AMP.Mo catalyst revealed that some catalytically active Mo species were present in the residues isolated from Run 1 to Run 3, but became negligible 'After Run 4' (Figure 4B). The leaching of Mo could either be soluble leached complexes or as traces of Mo containing microgel released as a result of mechanical attrition of the beads or both.

3.2 Continuous epoxidation results

Continuous epoxidation was carried out in a FlowSyn continuous flow reactor to study the effect of reaction temperature, FMR of 4-VCH to TBHP and feed flow rate on the conversion of TBHP and the yield of 4-VCH 1,2-epoxide at steady state, i.e., at 2 h. Samples were collected in a fraction collector for each experiment conducted in a continuous reactor at regular time periods and analyzed by GC. The analytical error was within ±3% for all the experiments conducted in a continuous flow reactor. By adapting the epoxidation reaction under continuous flow, maximum conversion of TBHP could be achieved within a short reaction time, due to higher specific surface and heat transfer rate [19], since a small volume of the reaction mixture is in contact with a relatively large volume of Ps.AMP.Mo catalyst.

3.2.1 Effect of reaction temperature

The effect of reaction temperature on the conversion of TBHP and the yield of 4-VCH 1,2-epoxide was investigated at 60°C, 70°C and 80°C. Experiments were carried out using 1:1 stoichiometric ratio of 4-VCH to TBHP for all the temperature ranges, while the feed flow rate was maintained at 0.1 ml/min. As shown in Figure 5A, an increase in reaction temperature resulted in higher conversion of TBHP and higher yield of 4-VCH 1,2-epoxide. For instance, ~50% conversion of TBHP was achieved at steady state for reaction conducted at 80°C, whilst for the same period of time reactions carried out at 60°C and 70°C achieved ~43% and ~30% conversion of TBHP, respectively. Hence, it can be concluded that 80°C is the optimum reaction temperature for both batch and continuous epoxidation of 4-VCH with TBHP in the presence of Ps.AMP.Mo catalyst.

3.2.2 Effect of feed flow rate

The effect of feed flow rate was investigated at feed flow rates of 0.1 ml/min, 0.13 ml/min and 0.16 ml/min. Continuous epoxidation experiments were carried out at 80°C (i.e. the optimum reaction temperature) and at stoichiometric FMR of 4-VCH to TBHP of 1:1. Experiments carried out at a flow rate of 0.16 ml/min, which corresponds to feed residence time of ~3 min in the reactor, gave ~36% conversion of TBHP and ~29% yield of 4-VCH 1,2-epoxide at steady state as shown in Figure 5B. The residence time

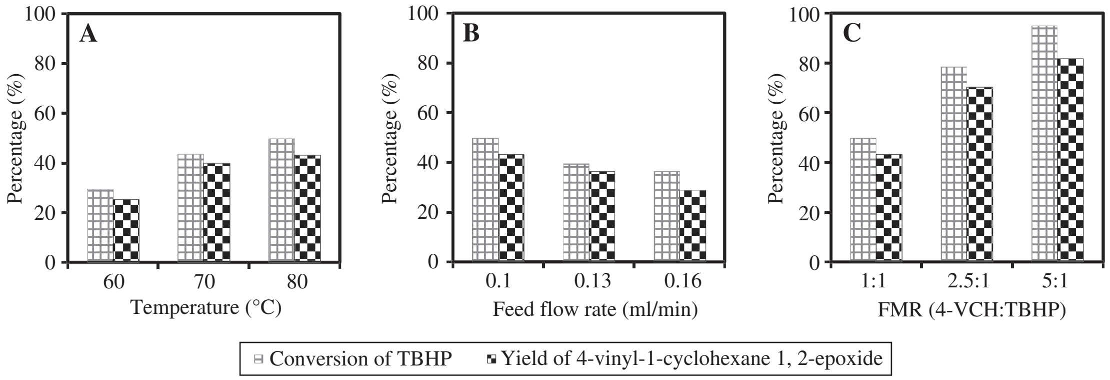

Figure 5 Conversion of *tert*-butyl hydroperoxide (TBHP) and the yield of 4-vinyl-1-cyclohexane 1,2-epoxide (4-VCH 1,2-epoxide) at steady state for continuous epoxidation of 4-VCH with TBHP using a FlowSyn continuous flow reactor in the presence of polystyrene 2-(aminomethyl)pyridine supported Mo(VI) complex (Ps.AMP.Mo) catalyst (~1.5 g): (A) effect of reaction temperature at feed flow rate: 0.1 ml/min; feed molar ratio (FMR) of 4-VCH to TBHP: 1:1; (B) effect of feed flow rate at reaction temperature: 80°C; FMR of 4-VCH to TBHP: 1:1. (C) effect of FMR of 4-VCH to TBHP at reaction temperature: 80°C; feed flow rate: 0.1 ml/min.

of the feed was increased to ~4 min by reducing the flow rate to 0.13 ml/min yielding ~40% conversion of TBHP and ~36% yield of 4-VCH 1,2-epoxide. However, TBHP conversion and yield of 4-VCH 1,2-epoxide increased to ~50% and ~43%, respectively, when the flow rate was further reduced to 0.1 ml/min (residence time of ~5 min). It can be concluded that an increase in feed flow rate caused reduction in feed residence time in the reaction zone (packed column) that consequently led to decrease in both the conversion of TBHP and the yield of 4-VCH 1,2-epoxide. Hence, feed flow rate of 0.1 ml/min can be considered as an optimum for continuous synthesis of 4-VCH 1,2-epoxide using Ps.AMP.Mo catalyst in a FlowSyn reactor.

3.2.3 Effect of FMR

In most of the catalyzed epoxidation processes, an excess amount of alkene is employed in order to suppress side reactions and enhance the conversion of the oxidant [20]. The effect of FMR was studied at 1:1, 2.5:1 and 5:1 molar ratios of 4-VCH to TBHP and at temperature and flow rate of 80°C and 0.1 ml/min, respectively. Figure 5C illustrates that the experiment conducted at 5:1 molar ratio resulted in ~82% yield of epoxide and 94% conversion of TBHP as compared to the experiments carried out at 2.5:1 and 1:1 molar ratios, which gave 70% and 43% yield of epoxide, respectively. By contrast, the residence time required for ~80% yield for continuous flow reaction conducted at 70°C is ~5 min, whereas it takes >40 min (Figure 2) to arrive at a similar yield of epoxide in a batch reactor. Based on the results obtained from this study, a continuous flow reactor for epoxidation of 4-VCH can be considered to be more efficient than the batch reactor and 5:1 FMR of alkene to TBHP can be regarded as the optimum for continuous epoxidation of 4-VCH with TBHP in the presence of Ps.AMP.Mo catalyst.

3.2.4 Reusability studies of Ps.AMP.Mo catalyst

The reusability studies of Ps.AMP.Mo catalyst were conducted for continuous epoxidation of 4-VCH with TBHP in a fixed bed reactor using the same catalyst packing for four consecutive runs and each experiment lasted for 6 h. Therefore, the catalyst packing was used continuously for a period of 24 h. The reusability studies confirmed that Ps.AMP.Mo catalyst had negligible loss in catalytic activity when used for 24 h. TBHP conversion and the yield of 4-VCH 1,2-epoxide at steady state for each 6 h experiment was found to be in the range of 95±4% and 82±4%, respectively. It could be concluded that the problem of attrition of catalyst particles and Mo leaching observed using the batch reactor was eliminated in the continuous reactor. Hence, Ps.AMP.Mo catalyst could be reused several times in a continuous reactor for epoxidation of 4-VCH with TBHP.

4 Conclusions

Ps.AMP.Mo was successfully prepared and characterized. The performance of this catalyst was assessed for the epoxidation of 4-VCH in batch and continuous reactors using

TBHP as an oxidant. Ps.AMP.Mo catalyst has been proven to be long lived, stable and suitable for the epoxidation of 4-VCH. In comparison with reactions conducted in a batch reactor, the continuous flow reaction in a FlowSyn reactor shows substantial benefits that include fast heat and mass transfer, short setup and reaction times, flexibility of scaling-up reactions, complete non attended operation and safer and more environmentally friendly operating procedures.

Acknowledgments: The financial support from Engineering and Physical Sciences Research Council (EPSRC) (grant no. EP/H027653/1) and The Royal Society Brian Mercer Feasibility award is gratefully acknowledged. M. L. Mohammed is thankful to London South Bank University, UK and Tertiary Education Trust Fund (TETFund), Nigeria for the PhD scholarships.

References

[1] Sienel G, Rieth R, Rowbottom KT. In *Epoxides, Ullmann's Encyclopedia of Industrial Chemistry*, Wiley-VCH: Weinheim, 2000.
[2] Bauer K, Garbe D, Surburg H. *Common Fragrance and Flavor Materials: Preparation, Properties and Uses*, 4th ed., Wiley-VCH: Weinheim, 2001, pp. 143–145.
[3] Yudin, KA. *Aziridines and Epoxides in Organic Synthesis*, Wiley-VCH: Weinheim, 2006, pp. 185–186.
[4] Garcia-Bosch A, Company A, Fontrodona X, Ribas X, Costas M. *Org. Lett.* 2008, 10, 2095–2098.
[5] Bezzo F, Bertucco A, Forlin A, Barolo M. *Sep. Purif. Technol.* 1999, 16, 251–260.
[6] Liu Y, Tsunoyama H, Akita T, Tsukuda T. *Chem. Commun*. 2010, 46, 550–552.
[7] Singh B, Rana BS, Sivakumar LN, Bahuguna GM, Sinha AK. *J. Porous Mater*. 2013, 20, 397–405.
[8] Kollar J. Epoxidation process, Patent US 3351635 A, 1967.
[9] Arnold U, Habicht W, Döring M. *Adv. Synth. Catal*. 2006, 348, 142–150.
[10] Nath GR, Rajesh K. *Asian J. Chem*. 2012, 24, 4548–4550.
[11] Angelescu E, Pavel OD, Ionescu R, Bîrjega R, Badea M, Zavoianu R. *J. Mol. Catal. A: Chem*. 2012, 352, 21–30.
[12] Gupta KC, Sutar AK. *Polym. Adv. Technol*. 2008, 19, 186–200.
[13] Mbeleck R, Ambroziak K, Saha B, Sherrington DC. *React. Funct. Polym*. 2007, 67, 1448–1457.
[14] Ambroziak K, Mbeleck R, He Y, Saha B, Sherrington DC. *Ind. Eng. Chem. Res*. 2009, 48, 3293–3302.
[15] Ambroziak K, Mbeleck R, Saha B, Sherrington D. *Int. J. Chem. React. Eng*. 2010, 8, A125.
[16] Sharpless KB, Verhoeven TR. *Aldrichimica Acta* 1979, 12, 63–82.
[17] Sherrington DC. *Chem. Commun*. 1998, 2275–2286.
[18] Mohammed ML, Patel D, Mbeleck R, Niyogi D, Sherrington DC, Saha B. *Appl. Catal., A* 2013, 466, 142–152.
[19] Melero JA, Iglesias J, Sainz–Pardo J, de Frutos P, Blázquez S. *Chem. Eng. J*. 2008, 139, 631–641.
[20] Jähnisch K, Hessel V, Löwe H, Baerns M. *Angew. Chem., Int. Ed*. 2004, 43, 406–446.

Bionotes

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Misbahu L. Mohammed is currently working towards a PhD in Chemical Engineering within the School of Engineering at London South Bank University (LSBU), UK under the supervision of Professor Basu Saha. Before joining LSBU, Mohammed received his MSc with Distinction in Advanced Process Engineering from Loughborough University, UK and has been working as a Lecturer in the Department of Pure and Applied Chemistry, Usmanu Danfodiyo University, Sokoto, Nigeria. He is presently on study leave pursuing a PhD with scholarships awarded to him by the School of Engineering, LSBU and Tertiary Education Trust Fund (TETFund), Nigeria. His PhD research focuses on a novel heterogeneous catalytic process for the synthesis of commercially important epoxide building blocks. He won the first prize at the Institution of Chemical Engineers (IChemE), UK research event (2012) at Air Products, Basingstoke, UK.

Rene Mbeleck

Rene Mbeleck graduated in Chemistry from the University of Paris-Sud XI in 2003. He received his PhD in 2009 working under the supervision of Professor David C. Sherrington FRS on alkene epoxidations catalyzed by molybdenum (VI) supported on polymers. In 2009, he joined the group of Professor Dr Olof Ramström in Stockholm (Sweden) at the Royal Institute of Technology on a Marie Curie Postdoctoral Fellowship. There, he worked on the design and synthesis of new water soluble Hoveyda-Grubbs catalysts for cross-metathesis reactions. From 2010–2011, he worked as an Engineering and Physical Sciences Research Council (EPSRC) Research Associate with Professor Basu Saha at LSBU on the development of a continuous clean alkene epoxidation process technology for production of important commercially epoxide building blocks. His research interests include organic and polymer synthesis, organometallic chemistry and development of novel supported catalysts for developing new clean synthetic methods.

Dipesh Patel

Dipesh Patel received his Bachelor in Chemical Engineering from Mumbai University, India in 2003. After graduation, he worked for a pharmaceutical company (2003–2004). In 2004, he came to the UK to pursue an MSc in Advanced Process Engineering in the Department of Chemical Engineering at Loughborough University, UK (2004–2005). This was followed by a PhD in Chemical Engineering at Loughborough University (2006–2011) under the supervision of Professor Basu Saha. In 2011, he joined the Centre for Green Process Engineering (CGPE), LSBU as a postdoctoral Research Assistant (PDRA) and became a Research Fellow in 2013. His research work includes working on the production of value added products using a batch and multifunctional reactors. He has published research findings in high impact academic journals and presented research papers at international conferences and national research events. He was awarded the second prize at a national research event organized by the IChemE in 2011. He is an Associate Member of the IChemE (AMIChemE) and an Associate Fellow of The Higher Education Academy (AFHEA). In November 2014, he took up a Lectureship position in Chemical Engineering at the University of Hull, UK.

David C. Sherrington

David C. Sherrington was a Sir W H Tate Scholar at the University of Liverpool, where he graduated with BSc Hons (1966) and PhD Degrees (1969). In 1972 he was appointed to a lectureship in the Department of Pure and Applied Chemistry, University of Strathclyde, and was promoted to Senior Lecturer in 1983 and to Reader in 1987. In the period (1984–1987) he was seconded as Polymer Science Area Head to Unilever Research, Port Sunlight, returning to the University of Strathclyde in 1987 as Professor of Polymer Chemistry and, in due course, Head of the Organic Chemistry Section. He was elected Fellow of the Royal Society of Chemistry in 1981, Fellow of the Royal Society of Edinburgh in 1990 and was awarded his DSc from the University of Liverpool in 1992. In 1993, his work on supported reagents and catalysts was recognized by the award of the Beilby Medal by the Royal Society of Chemistry, and his contribution to polymer chemistry by the award of the RSC Macro Group Silver Medal in 2002. His research philosophy of integrating organic synthetic methodology with polymer synthesis has led to a number of important scientific contributions, particularly relating to the synthesis, characterization and application of porous polymer particulates, but also in other important fields including catalysis, metal speciation, surfactants, molecular imprinting and branched polymers. He was elected Fellow of the Royal Society in 2007. He was International Editor of the journal *Reactive Polymers* 1987–1996 and was the editor and co-author of four books. He and his group had published ~350 refereed papers and reviews. In addition to this he was Visiting Professor at universities in Pisa, Tanta, Nankai, Bombay, Oulu, Gyeongsang and Bordeaux, as well as at the Tokyo Institute of Technology. He passed away on the 4th of October 2014.

Basudeb Saha

Basudeb Saha is Professor of Chemical and Process Engineering and Director of Centre for Green Process Engineering at LSBU. Previously (1997–2010), he worked at Loughborough University as a Research Associate, Lecturer, Senior Lecturer and a Reader in Chemical Engineering. His current research covers greening of alkene/terpene epoxidation, biodiesel production from used cooking oil, graphene nanocomposites from supercritical carbon dioxide, CO_2 conversion to value added chemicals and fuels and continuous synthesis of value added products using multifunctional reactors such as the FlowSyn continuous flow reactor, reactive distillation column and chromatographic reactor. He has a proven track record of delivery on research contracts in terms of the scientific and training aspects, has established collaborative research programs with several leading international research groups, and his services are in demand for lecture, peer review, examination and teaching activities, both in the UK and overseas. He is recognized as a leading expert in his field and has been appointed as a Visiting Professor in the University of Barcelona, Spain, Saga University, Japan and University of Burgos, Spain. He has published over 150 research papers (including heavily cited research articles), book chapters and patents. He has received many invitations to visit laboratories overseas and to give invited/keynote lectures at national and international conferences. He was an Associate Editor of *The Canadian Journal of Chemical Engineering* (2009–2012) and is currently an International Advisory Board Member of *The Canadian Journal of Chemical Engineering* (2013–present). He is an Editor of *Green Processing and Synthesis* journal (2012–present) and an Editorial Board Member of *Process Safety and Environmental Protection* journal (2014–present). He is a Fellow (FIChemE) of the IChemE and The Higher Education Academy (FHEA).