

Green Chemistry on Solid Support for Clean Synthesis

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Abstract—R. Bruce Merrifield's Peptide Synthesis in 1963 using cross linked polystyrene support opened an exciting area of research and since then many new reagent bound, substrate bound and catalyst bound methods have been developed. Present paper discusses some work on the polystyrene-divinylbenzene supported transition metal complex catalysts in general and Ruthenium-schiff base complex in particular and its application in different reactions viz. Hydrogenation of styrene to ethylbenzene and oxidation of benzyl alcohol to benzaldehyde. The polymer bound complex was found to be more effective than homogeneous counterpart. It was also found recyclable and reusable. Untapped potential of such catalysts is also discussed.

Index Terms—Green chemistry, hydrogenation, hazardous reactions, oxidation, polymer support, polymer supported catalysts

I. INTRODUCTION

Catalysis is extremely important in Industrial Chemistry due to enhancement of rate of reaction without getting involved in the reaction. Homogeneous Catalysts are known for high activity and selectivity but have problem in loss of precious heavy metal, corrosion and plating out on the reactor wall. As there is no recovery of the catalyst, reusing it is out of consideration. Heterogeneous catalysts are not soluble like homogeneous and hence can be recovered and even reused but lacks the activity and selectivity of homogeneous catalysts. If a homogeneous catalyst is anchored on to a solid support, its activity and selectivity can be retained and it can be rendered suitable for recovery and reuse. As catalysts are extremely important in green chemistry to decrease/eliminate pollution at source by enhancing favorable reaction and supported catalysts are attractive due to combination of advantages of heterogeneous and homogeneous catalysts; this green chemistry on solid support is of current interest.

Solid supports used in supported catalysts are Inorganic oxides like clay, silica etc and organic polymers. Organic Polymers have advantages due to ease of handling and easy availability. Poly [styrene-divinylbenzene] is a popular polymer support because of having a wide range of physical properties where the porosity, surface area and solution characteristics may be altered by varying the degree of cross-linking. Transition metals useful in catalysis are becoming expensive and less available. The present article reviews some work done on synthesis, characterisation and evaluation of catalytic activity of

Poly[styrene-divinylbenzene] bound Ru and Pd complex catalysts and discusses future potential of Polymer bound catalysts.

II. PROCEDURE

A. Synthesis of Polymer Bound Catalysts Ru and Pd Complexes.

Synthesis of such catalysts is simple and visual observation at each stage can confirm successful synthesis. First the polymer was functionalised. Poly[styrene-divinylbenzene] of different cross-linking viz. 2,5,8,14 and 15% cross-linking were chloromethylated with HCl, paraformaldehyde, acetic anhydride and 1,2-dichloroethane using AlCl_3 as a catalyst. The chlorine content was confirmed by elemental analysis. Functionalised polymer was converted to polymeric ligand which was then treated with metal. In order to anchor the Schiff base, chloromethylated Poly[styrene-divinylbenzene] was treated with ethylene-diamine using THF as a solvent. The reaction was kept for 48 hrs at room temperature. The beads were washed with deionised water, methanol and dry dioxane, dried at 60 C and refluxed in a solution of salicylaldehyde in benzene for 24 hrs. The resin was Soxhlet extracted with ethanol and dried at 80 C. The Schiff base loaded Polymer was kept in ethanolic solution of RuCl_3 at room temperature for 7 days. The catalyst was washed with ethanol and dried in vacuum at 70 C. Elemental analysis was performed to confirm nitrogen and metal loading was determined by spectroscopic method. Methods to anchor ligands Edta, 1-2-diaminopropane, glycine on to polymer and chelation with Ru and Pd is described earlier [1]-[13].

B. Characterisation of Polymer Bound Ru and Pd Complex Catalysts

Surface area and Pore volume of catalysts were determined by BET method. Chemical composition was determined by elemental analyser for C and H and Duma's method for N. Swelling studies of prepared catalysts was performed using different polar and non-polar solvents. Spectral characterisation was carried out using FTIR spectrophotometer, UV visible spectrophotometer, Electron spin magnetic resonance (ESR) spectrometer and electron spectroscopy for Chemical analysis (ESCA). Morphology of the catalyst was studied using Scanning electron microscope (see Fig. 1) [1]. Thermal analysis was carried out using TGA.

C. Evaluation of Catalytic activity of Polymer bound Ru and Pd Complex Catalysts

Catalytic activity of prepared polymer bound Ru and Pd complexes was evaluated by studying kinetics for reactions like hydrogenation of styrene, 1-hexene, cyclohexene, Nitrobenzene and Oxidation of Benzyl alcohol, cyclohexane

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and Toluene. Kinetics of these reactions was studied at atmospheric pressure by measuring gas uptake using a glass manometric apparatus. Kinetics of hydrogenation of styrene and oxidation of benzyl alcohol by catalyst 14PRu(III)salen are discussed. Effect of various parameters on the rate of reactions was studied. Life cycle of catalysts was studied under repeated catalytic cycles (see Table I- III) [2].

III. RESULTS AND DISCUSSION

A. Catalyst Characterization

Catalysts are easy to prepare as shown in II A and visual change is observed on every step of synthesis. Successful functionalization of the polymer was confirmed by elemental analyses at different stages of preparation of the catalyst. A decrease in the surface area was observed after loading the metal ions, on to the polymer which might be due to the blocking of pores of the support as well as due to the steric hinderance. The nature of the solvent is an important factor that can be varied in order to control the activity and selectivity of polymer-supported catalyst. It is therefore essential to study the swelling behavior of the catalyst in different solvents. A decrease in swelling was observed as the nature of the solvents was changed from polar to non-polar. Maximum swelling was observed in water, which might be due to hydrogen bonding of water molecules with amino groups. However, methanol was chosen as a suitable solvent for catalytic reactions because of better swelling of catalyst and miscibility with the substrate. Clear change in the morphology of the polymer support after introduction of the ligand and metal ions was observed in SEM (see Fig.1). TG analysis showed that thermal stability of the polymer support and the catalyst is upto 350 °C .the probable structure of the catalyst was also proposed on the basis of spectroscopic studies [2].

B. Catalytic Reactions

Suitability of a polymer bound catalysts for different reactions are studied. Results show suitability of one such catalyst for multiple reactions say for ex: 14PRu(III)Salen for hydrogenation of styrene to ethylbenzene and oxidation of benzyl alcohol to benzaldehyde [2]. The kinetics of hydrogenation of styrene and oxidation of benzylalcohol for polymer-anchored catalyst and corresponding homogeneous complex [Ru(III)salen C1] was investigated. The catalytic reaction was carried out in a kinetic regime at atmospheric pressure in the temperature range of 25 – 45 °C. The stirring of the reaction mixture was maintained at an optimized rate (600 rpm) throughout the experiment to minimize diffusion. The influence of various parameters on the rate of reactions was studied.

C. Kinetics of Hydrogenation of Styrene

The effect of substrate concentration on the rate of hydrogenation was determined in the range of 4.36×10^{-3} mol dm⁻³ of Ru (III) (see Table 1). It was observed that the rate of hydrogenation increases linearly with respect to substrate concentration. The order of reaction calculated from the linear plot of log (initial rate) vs.log [styrene] was found to be

unity. A linear plot of 1/rate vs. 1/[styrene] indicates that the rate of hydrogenation of styrene, R, is related to the concentration of styrene, [S], by the relationship:

$$1/R = a \times 1/[S] + b$$

where a and b are the slope and intercept of the plot.

The effect of [catalyst] on the rate of hydrogenation of styrene was studied in the range of 1.48×10^{-6} to 5.94×10^{-6} mol dm⁻³ of Ru(III) at 35°C, 1 atm pressure and constant [styrene] of 8.73×10^{-3} mol dm⁻³ (see Table 1). A linear increase in the rate of reaction was found with increase in [catalyst] indicating absence of any dimerization of metal complex in the range studied. The order of reaction calculated from the plot of log (initial rate) vs. log [catalyst] was found to be unity indicating the availability of all the probable active sites for the catalytic reaction. Thus, better dispersion on the surface could be achieved.

The kinetics of hydrogenation of styrene was studied in the range of 30 - 45°C at a fixed catalyst concentration of 2.97×10^{-6} mol dm⁻³ of Ru (III) at 1 atm pressure and [styrene] of 8.73×10^{-3} mol dm⁻³ (see Table 1). An increase in the rate with temperature was found. Energy of activation calculated from Arrhenius plot was found to be 104.7 kJ mol⁻¹ and corresponding entropy of activation was found to be -148.4 eu. The rate of hydrogenation of styrene was found to decrease as the nature the solvent was changed from polar to non-polar. Higher rate of reaction in polar solvents might be due to better swelling of polymer support and therefore availability of catalytic sites.

Kinetics of styrene hydrogenation was also studied using [Ru (III)salen C1] complex obtained in solution. A lower uptake of H₂ was obtained as compared to heterogenized catalyst even though a 10³ time's higher concentration of metal ions was taken for conveniently measuring the rate of reaction. Energy of activation was found to be 72.8 kJ mol⁻¹.

D. Lifecycle Study of the Catalyst

Recycling efficiency of catalyst was tested at 35°C by injecting a known amount of substrate (i.e 20 micro litre) at every 3300 sec. intervals. The rate of reaction was measured as a function of time for both used and fresh catalyst upto 6 hours. It was found that the maximum rate of reaction was maintained for 3 – 5 hours for fresh catalyst after which it decreased slowly, this might be due to leaching of metal ions from the surface of the polymer.

Rate equation

The reaction mechanism for catalytic hydrogenation in homogeneous medium is studied widely. The formation of hydrodo species, and preferential attachment of substrate to this intermediate complex has been well established by experimental studies and thermodynamic properties. On the basis of evidence from literature, the mechanism and rate equation are proposed[2].

E. Kinetics of Oxidation of Benzylalcohol

Kinetics of oxidation of benzyl alcohol for polymer-anchored catalyst was investigated and influence of various parameters on the rate of oxidation was studied.

The effect of substrate concentration on the rate of reaction was determined in the range of 4.83×10^{-3} to 19.32×10^{-3} mol dm^{-3} at 35°C and 1 atm pressure at constant catalyst concentration of 2.97×10^{-6} mol dm^{-3} of Ru(III) (see Table 2). The rate of oxidation reaction was found to increase linearly with respect to substrate concentration. The order of reaction was found to be unity.

The effect of [catalyst] on the rate of oxidation of benzyl alcohol was studied in the range of 1.48×10^{-6} to 5.94×10^{-6} mol dm^{-3} of Ru (III) at 35°C and 1 atm pressure and at constant [benzyl alcohol] of 9.66×10^{-6} mol dm^{-3} . A linear increase in the rate of reaction was observed with increase in catalyst concentration indicating absence of any dimerization of metal complex in the range studied. The order of reaction calculated from the plot of log (initial rate) vs. log [catalyst] was also found to be unity.

TABLE I: SUMMARY OF THE KINETICS OF HYDROGENATION OF STYRENE BY POLYMER BOUND CATALYST AT 1 ATM PRESSURE USING 20 ML SOLVENT[2].

| [Ru(III)] (mol dm^{-3}) 10^6 | [Styrene] (mol dm^{-3}) 10^3 | Temperature ($^\circ\text{C}$) | Solvent | Rate of Reaction ($0.0017 \text{ dm}^3 \text{ sec}^{-1}$) |
|---|---|-------------------------------------|----------|--|
| 2.97 | 4.36 | 35 | Methanol | 0.07 |
| | 8.73 | | | 0.22 |
| | 13.09 | | | 0.38 |
| | 17.46 | | | 0.54 |
| 1.48 | 9.66 | 35 | | 0.09 |
| 2.97 | | | | 0.22 |
| 4.45 | | | | 0.36 |
| 5.94 | | | | 0.50 |
| 2.97 | 8.73 | 30 | | 0.09 |
| | | 35 | | 0.22 |
| | | 40 | | 0.40 |
| | | 45 | | 0.55 |
| 2.97 | 8.73 | 35 | Methanol | 0.22 |
| | | | Ethanol | 0.19 |
| | | | THF | 0.10 |
| | | | Benzene | 0.07 |

The kinetics of oxidation of benzyl alcohol was studied in the range of $25 - 40^\circ\text{C}$ at a fixed catalyst concentration of 2.97×10^{-6} mol dm^{-3} of Ru(III) at 1atm pressure and [benzyl alcohol] of 9.66×10^{-6} mol dm^{-3} (see Table 2). An increase in the rate with temperature was found. Energy of activation calculated from Arrhenius plot was found to be 47.9 kJ mol^{-1} and corresponding entropy of activation was found to be -28.4 eu . The rate of oxidation of benzyl alcohol was found to decrease as the nature of the solvent was changed from polar to non polar (see Table 2). Higher rate of reaction in polar solvents might be due to better swelling of the polymer support and therefore availability of catalytic sites.

Kinetics of oxidation of benzyl alcohol using unbound complex [Ru(III)salen Cl] is reported by us earlier and a probable reaction mechanism has already been suggested. The formation of oxo complex and the transfer of oxygen via this route might be responsible for the reaction. The energy of activation was calculated using Arrhenius plots and was found to be $103.4 \text{ kJ mol}^{-1}$ showing a lower catalytic activity as compared to heterogenized catalyst.

Reaction mechanism for reactions was proposed [2].

F. Discussion

Kinetics of above reactions using corresponding unbound complexes show polymer bound catalyst is more active than its homogeneous counterpart. Catalysts were found to be recyclable and reusable [2]. Thus catalysts are suitable for multiple reactions and are compatible with gaseous reactants. Such polymer bound catalysts can be easily tailored as per requirement of a reaction.

Such versatility of catalyst for reactions using H_2 or O_2 gas can be further explored for hazardous reactions like

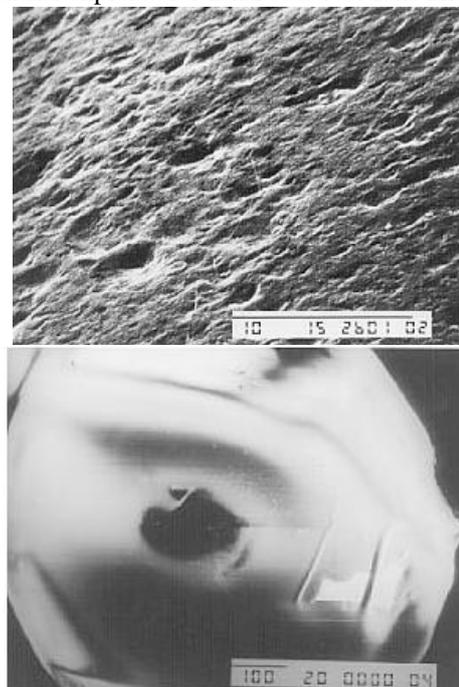


Fig. 1. Scanning electron micrograph of (a) polymer support ; (b) Polymer bound catalyst [1].

TABLE II: SUMMARY OF THE KINETICS OF OXIDATION OF BENZYL ALCOHOL BY POLYMER BOUND CATALYST AT 1 ATM PRESSURE USING 20 ML SOLVENT[2].

| [Ru(III)] (mol dm^{-3}) 10^6 | [Benzyl alcohol] (mol dm^{-3}) 10^3 | Temperature ($^\circ\text{C}$) | Solvent | Rate of Reaction ($0.0017 \text{ dm}^3 \text{ sec}^{-1}$) |
|---|--|-------------------------------------|----------|--|
| 2.97 | 4.83 | 35 | Methanol | 0.31 |
| | 9.66 | | | 0.49 |
| | 14.49 | | | 0.65 |
| | 19.32 | | | 0.79 |
| 1.48 | 9.66 | 35 | | 0.32 |
| 2.97 | | | | 0.49 |
| 4.45 | | | | 0.61 |
| 5.94 | | | | 0.70 |
| 2.97 | 9.66 | 25 | | 0.30 |
| | | 30 | | 0.40 |
| | | 35 | | 0.49 |
| | | 40 | | 0.56 |
| 2.97 | 9.66 | 35 | Methanol | 0.49 |
| | | | Ethanol | 0.45 |
| | | | THF | 0.30 |
| | | | Benzene | 0.13 |

Halogenations, say bromination using Elemental bromine or chlorination using chlorine gas. Thus fine-tuning of the catalyst-support interface along with a detailed understanding of catalytic reaction mechanisms, permits the generation of reusable and recyclable polymer-supported catalysts and also facilitates the design and realization of

supported catalysts that are significantly more active and selective than their non-supported counterparts. These superior supported catalysts are accessible through the optimization of four basic variables in their design: (i) polymer backbone rigidity, (ii) the nature of the linker, (iii) catalyst site density, and (iv) the nature of the catalyst attachment [14], [15].

TABLE III: LIFE CYCLE STUDY OF THE CATALYSTS AT 35 °C AND 1 ATM PRESSURE USING 20 ML METHANOL; [RU]= 2.97×10^{-3} MOL DM-3 ; [STYRENE] OF 8.73×10^{-3} MOL DM-3 FOR EVERY INJECTION.

| Time (sec) | Rate of reaction ($0.0017\text{dm}^3\text{sec}^{-1}$) | |
|------------|--|--|
| 3300 | 0.22 (0.22) | |
| 6000 | 0.22 (0.22) | |
| 9300 | 0.22 (0.20) | |
| 12600 | 0.21 (0.18) | |
| 15900 | 0.18 (0.18) | |
| 18600 | 0.15 (0.11) | |
| | | |

Values for the used catalysts are indicated in parenthesis.

IV. FUTURE TRENDS

As recyclability of such catalysts is seen, further research needs to be aimed at development of synthesis specific catalyst packs that can be used in several different synthetic routes including hazardous reactions to eliminate pollutants at source. This may be used either in Batch system or in continuous reactor system. P. Hodge has reported that the use of appropriate combinations of polymer-supported reagents, catalysts and/or scavengers is a powerful approach, both for the synthesis of single organic compounds and for parallel syntheses. A further stage of development is the use of such reactants in flow systems. So far, it has been shown that a variety of flow formats afford excellent chemical yields and, where relevant, excellent enantiomeric excesses. The supports have a longer lifetime than in batch system [16]. Advantages of flow systems typically include the following: there is little or no reaction workup, the support suffers no physical damage in use, automation is relatively easy, and extension to continuous production, even on a large scale, is a possibility [17]. Oe and Uozumi have shown that Kharasch reaction which involves the addition of halogenated compounds to an alkene, in general catalyzed by a ruthenium complex in the presence of a radical initiator such as AIBN (azoisobutyronitrile) [18] can be carried out in water, without the addition of AIBN, with the help of a polymer-supported ruthenium catalyst, thus affording an atom-economic and 'green' functional group transformation [19]. The catalytic system consisted of a ruthenium(III) (pentamethylcyclopentadienyl)dichloride attached to a PS-PEG backbone via a polymer-supported phosphine. A variety of different styrene derivatives were investigated with good results, affording the addition products in up to 98% yield. Aliphatic alkenes could also be used under these conditions, albeit with a somewhat lower yield. Recycling of the catalyst did not affect the catalytic activity to any extent. Ring opening of epoxides using a

supported ruthenium catalyst is demonstrated by Kim and Lee [20]. A ruthenium(III) complex was anchored to a polymer-bound bis(2-picoyl)amine ligand and applied in the reaction of both aromatic and aliphatic epoxides with either methanol or water with essentially complete conversion. Styrene gave a rapid reaction (1 h), while other epoxides required somewhat longer reaction times (2.5–35 h) and 1,2-Epoxy-hexane needed 200 h for complete conversion. The polymer-bound catalyst could be recycled up to ten times with no loss of catalytic activity. Thus platform created by research shows easy synthesis and versatility as well as tailoring ideas and recyclability. A catalyst pack insertion for a particular hazardous batch reaction has been suggested and worked out by Author and some intellectual property is being filed. It may be interesting to develop a Versatile Catalyst Pack like a cartridge in domestic RO machine that can be inserted in reactor for number of cycles and recharged as and when required by easy methods. Such development may eliminate many pollution related problems. Clean synthetic methodologies can be developed on solid support.

V. CONCLUSION

Polymer supported transition metal complex catalysts can be easily synthesized. Thermal stability of 14% cross linked polymer supported catalysts were found to be higher. Polymer supported Ru(III) and Pd(II) complexes were found to be effective catalysts for hydrogenation of 1-hexene to n-hexane, styrene to ethyl benzene and oxidation of benzyl alcohol to benzaldehyde. An enhanced rate of reaction was observed in the case of polymer bound complexes as compared to unbound complexes. Stability of the catalysts under repeated catalytic cycles was studied and most of the catalysts were found to be stable upto 4 cycles. The study shows that better catalytic activity could be achieved by proper selection of polymer support, chelating ligand, swelling agent and the active component to synthesise heterogenized homogeneous catalysts for a given reaction with several advantages over their homogeneous counterpart. Therefore, one-pack catalyst cartridges that can be inserted in a reactor and recharged after life span may be developed from this platform.

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