Cordierite honeycomb supported Mo(VI)/ZrO$_2$ for microwave assisted Pinacol-Pinacolone rearrangement

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ZrO$_2$, Mo(VI)/ZrO$_2$, SO$_4^{2-}$/ZrO$_2$ and Pt-SO$_4^{2-}$/ZrO$_2$ supported on honeycomb monoliths have been prepared and characterized for their physico-chemical properties such as surface acidity, crystalinity, functionality and morphology. These materials have been used as solid acid catalysts in the pinacol rearrangement of benzopinacol under microwave irradiation. A few diols have also been subjected to pinacol rearrangement to obtain a good conversion of rearrangement products with high selectivity. Optimization of reaction conditions has also studied to determine the most suitable reaction conditions for the effective synthesis of pinacolone derivatives. Up to 98% conversion of benzopinacol is observed under a set of optimized reaction conditions. A reactivation and reusability study of zirconia based solid acid catalysts has also performed.

Keywords: Pinacol rearrangement, Zirconia, Solid acids, Microwave

Cordierite honeycombs (HC) have played a key task as catalyst carriers in the field of catalysis.[1] Honeycomb monolith supports are uni-body structures and are made up of interconnected repeating cells or channels. Catalysts coated over HCs are largely used in automotive applications like selective reduction of NOx, combustion of volatile organic compounds, ozone abatement in jets etc., which involve gas phase reactions[2]. The advantages of catalysts coated on HCs over powder catalysts are highly effective, simple recovery and complete separation of the catalysts from the reaction mass. Though the application of HCs in gas phase reactions is vastly studied, their applicability in the liquid phase reactions as catalytic carriers is least explored[3].

Solid acids are a group of heterogenous catalysts that have gained a great interest among researchers in the field of catalysis because of various advantages associated with them when compared to homogenous catalysts. A few advantages of heterogenous catalysts involve non-toxicity, non-corrosiveness, ease of handling, less expensive, easy to recover and reuse, etc[4]. Some of the heterogenous materials that have been used as solid acid catalysts involve; metal oxides, mixed metal oxides, zeolites, heteropolyacids, clays, etc[5]. Among these, metal oxides especially zirconia (ZrO$_2$) and its modified forms have been studied to a large extent because of its unique acido-basic and redox properties. Pure zirconia can be modified with anions or cations to modify its properties. It is reported that the acidic properties of pure zirconia can be increased drastically by incorporating either SO$_4^{2-}$ or Mo(VI) or W(VI) or V(V) ions which can even generate 'super acidic' sites on zirconia[6]. Since sulfated zirconia is associated with some disadvantages such as deactivation, sulfate free-zirconia based solid acids such as Mo(VI)/ZrO$_2$, W(VI)/ZrO$_2$, V(V)/ZrO$_2$, etc., are preferred in organic reactions. These sulfate free solid acids have been employed in many industrially important reactions such as transesterification, esterification, alkylation, acylation etc. The efficiency of these solid acids can be further enhanced by coating them on catalyst carriers such as honeycomb monoliths (HC). HCs coated with solid acids are more economical, effective and eco-friendly than their powder forms.

The pinacol-pinacolone rearrangement is a valuable process for preparing aldehydes or ketones through elimination of water from skeletal rearrangement of diols. This is an acid catalyzed reaction and has been well studied for a long time and served as a standard topic in most undergraduate

organic textbooks[7]. Mineral acids such as H_2SO_4, H_3PO_4, HCl, etc., have been utilized as catalysts in this reaction[8]. In order to replace these harmful catalysts, during the past two decades a few efforts have been made to replace them by eco-friendly solid acid catalysts. Zeolites such as ZSM-5, X & Y, SAPOs, AlPOs and HPAs have been examined as catalysts for pinacol rearrangement[9,10]. Though SAPO molecular sieves have weak acidity as compared to zeolites, these were found to be less active in this reaction[9] because of their constrained pore diameter. Zeolites ZSM-5, X & Y have been reported to show better activities, but also gave higher coking rates for pinacol rearrangment[11]. Though $AlPO_4$ molecular sieves and their transition metal modified forms were also reported[8] to be highly efficient catalysts in pinacol rearrangement reaction, but the preparation of these catalysts is a tedious process.

Further, organic synthesis can be made much better by using a suitable method of heating. One of the methods is microwave assisted organic synthesis which is an emerging heating technology. Microwave-assisted organic transformations accelerate reactions from hours to minutes and minutes to seconds. Microwave-assisted organic transformations have many advantages over conventional heating methods. It provides quick results by drastically reducing reaction times, uniform heating occurs throughout the material, high efficiency of heating, increases product conversion with high purity of the product by reducing undesirable side reactions and environmental heat is avoided[12,13].

Keeping in view the importance of zirconia based solid acids in organic synthesis, the role of HC as catalyst carriers in liquid phase fine chemical synthesis and advantages of microwave heating method, the work presented in this article is aimed to evaluate the catalytic activity of HCs coated with zirconia and its modified forms (ZrO_2, Mo(VI)/ZrO_2, SO_4^{2-}/ZrO_2 and Pt-SO_4^{2-}/ZrO_2) in Pinacol-Pinacolone rearrangement of diols. The catalytic material were prepared and characterized for their physico-chemical properties by using techniques such as NH_3-TPD, powder XRD, FTIR, SEM and TEM. A systematic study on the effect of various factors such as nature of the catalyst, reaction temperature, reaction time, reactivation towards the conversion of pinacol and selectivity of the products was conducted.

Materials and Methods

Honeycomb Monoliths with dimensions: height = 1.20 cm, diameter = 2.00 cm and hole = 0.2 cm were procured from Shreya ceramics India Ltd. Zirconyl nitrate, zirconium hydroxide, ammonium molybdtate, chloro platinic acid, diols, methanol and H_2SO_4 were supplied either by M/S SD Fine Chemicals India Ltd., or Merck India Ltd.

Preparation of catalysts in honeycomb (HC) coated forms

Zirconia and its modified forms [Mo(VI)/ZrO_2, SO_4^{2-}/ZrO_2 and Pt-SO_4^{2-}/ZrO_2] were coated on HCs by using 'dip and dry' method[3].

Typically, a HC was coated with zirconia by using a dilute solution of zirconyl nitrate (2.0 g) dissolved in 50 mL of deionized water. The resulting solution was coated on HC by dipping and drying in a furnace preheated at 400 °C for 10–12 times till ~0.1 g of zirconia was coated on the HC.

Similarly, Mo(VI)/ZrO_2 consisting of 5% Mo(VI) was coated on a HC by using dilute solution consisting of zirconyl nitrate (4.8 g) and ammonium molybdate (0.1 g). SO_4^{2-}/ZrO_2 was prepared by using zirconium hydroxide (3.0 g) and 3 M sulfuric acid (1.5) by following impregnation method[14].

Pt-SO_4^{2-}/ZrO_2 was coated on a HC by following two steps: In the first step, SO_4^{2-}/ZrO_2 was prepared by using zirconium hydroxide (3.0 g) and 3 M sulfuric acid (1.5) by following impregnation method. The obtained SO_4^{2-}/ZrO_2 was treated with 1% chloro platinic acid (1.0 mL) to obtain Pt-SO_4^{2-}/ZrO_2. In order to coat Pt-SO_4^{2-}/ZrO_2 on a bare HC, slurry consisting of Pt-SO_4^{2-}/ZrO_2 was prepared and coated on the HC till approximately 0.1 g of the catalyst is coated.

Preparation of catalysts in powder forms

The catalysts ZrO_2, Mo(VI)/ZrO_2 and Pt-SO_4^{2-}/ZrO_2 were also prepared in their powder forms by impregnation method.

Finally, these catalysts such as ZrO_2, Mo(VI)/ZrO_2, SO_4^{2-}/ZrO_2 and Pt-SO_4^{2-}/ZrO_2 (both HC and powder forms) were calcined at 550 °C for 5 h in a muffle furnace. For easy identification the catalytic material ZrO_2, Mo(VI)/ZrO_2, SO_4^{2-}/ZrO_2 and Pt-SO_4^{2-}/ZrO_2 are abbreviated as Z, MZ, SZ and P-SZ, respectively.
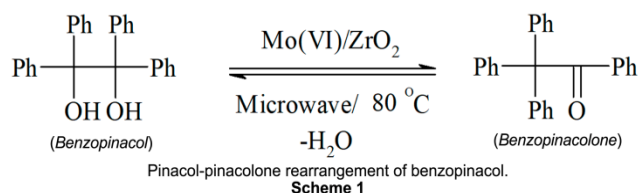
Catalyst characterization

The total surface acidity was analysed by NH_3-TPD technique over Mayura-TPD instrument. In this technique, 0.1 g of the catalyst was degassed under N_2 stream for 2 h at 100 °C, ammonia (99.99%) gas was injected into the stream until saturation was reached and cooled to 50 °C. The system was maintained at 50 °C for 30 min and then the catalyst

surface was flushed with N$_2$ for 2 h at 50 °C. The amorphous or crystalline nature of the catalysts was determined by recording their Powder XRD (PXRD) patterns on a X-ray diffractometer (Philips X'pert) using CuKα (λ = 1.5418 Å). The FTIR spectra of the catalyst samples were recorded using Schimdzu spectrometer by KBr pellet technique with the wave number range 4000–500 cm^{-1} for the functionalized samples. The catalyst was heated at the rate of 5 °C min^{-1} in N$_2$ to 700 °C. SEM and TEM images were recorded on JEOL-2010 and Philips CM-200, respectively.

Catalytic activity determination in pinacol rearrangement

The catalytic activity of the HC as well as powder form of catalysts was determined in the microwave assisted liquid-phase reaction of benzopinacol (Scheme 1). The Mechanism of acid catalyzed pinacol rearrangement of benzopinacol[8] is shown in Scheme 2. The mechanism involves the transfer of proton (H$^+$) from the acid catalyst to -OH group of the benzopinacol followed by the dehydration to form carbenium ion intermediate. The next step involved the migration of a phenyl group to the cation site followed by the formation of benzopinacolone with regeneration of the acid site of the catalyst.

Pinacol rearrangement of benzopinacol was carried out in a specially designed glass reactor suitable for honeycomb form of catalytic material (Photo 1). 1 mmol of benzopinacol and methanol (solvent) was taken in the reactor and heated at 80 °C for a definite period of time in a microwave reactor (Labmatrix) fitted with a water cooled condenser and magnetic stirrer. The reaction was monitored by TLC analysis for the completion of the reactant. After the reaction, the hot reaction mixture was filtered to separate the catalytic material (HC form or powder form). The reaction products were quantitatively analyzed by GC and qualitatively by GC-MS (Varian). The rearrangement reactions were also carried out with different diols to synthesize their respective pinacolone derivatives.

Pinacol rearrangement of benzopinacol was also carried out using conventional heating method. The reactions were carried out on a with hot plate magnetic stirrer.

Reusability study of catalysts

After the completion of the reaction, the catalysts (both HC and powder forms) were filtered through Whatman filter paper, washed with methanol and dried for 1 h at 120 °C. The catalysts were calcined for 1 h at 550 °C. Thus regenerated catalysts were again subjected to pinacol rearrangement reaction. Such reusability studies were taken-up for 6 consecutive reactions.

Pinacol-pinacolone rearrangement of benzopinacol.
Scheme 1

Mechanism of pinacol rearrangement of benzopinacol over an acid site (H+) of the catalyst.
Scheme 2
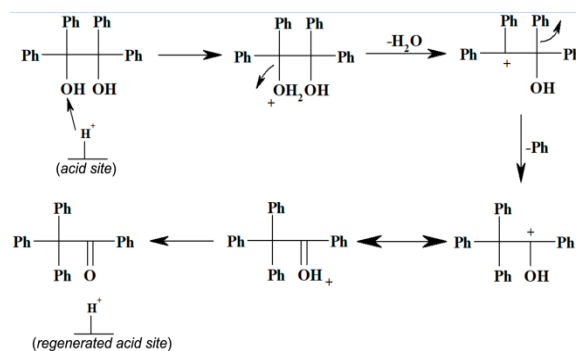
Specially designed glass reactor for microwave assisted honeycomb catalytic reactions.
Photo 1

Results and Discussion

Catalyst characterization

Surface acidity and acid site distribution of the catalysts

NH_3-TPD method was used to determine the surface acidity as well as acid site distribution of the catalysts. The total surface acidity (TSA) values of the catalysts calcined at 550 °C (Table 1) were found to be in the following order:

P-SZ ≥ SZ > MZ > Z

Pure zirconia was found to be least acidic when compared to its modified forms. When pure zirconia was modified with either Mo(VI) or Pt-SO_4^{2-} ions, the surface acidity was found to increase drastically which can be attributed to the formation of electron deficient states upon modification. It is also reported that upon incorporation of Mo(VI) ions on zirconia, Mo-O-Zr linkages are formed which are responsible for the higher acidity of MZ catalytic material[6].

Further, when the acid site distribution of the catalysts was observed (Fig. 1), pure zirconia consisted of 'weak' and 'moderate' acid sites, but modified forms such as MZ or P-SZ consisted of 'moderate', 'strong' and 'very strong' *(super acid)* acid sites. However, P-SZ consists of more 'super' acid sites when compared to MZ. This indicates that the incorporation of Mo(VI) or Pt-SO_4^{2-} ions on zirconia can generate strong as well as super acidic sites. The acid values are in accordance with the values reported in the literature[6].

Crystallinity of the catalysts

The crystalline nature of the catalysts was analyzed by powder XRD technique. The powder XRD patterns of Z, MZ and P-SZ are given in Fig. 2. All these solid acids were found to be crystalline in nature. The diffraction pattern of pure zirconia consisted of reflections pertaining to a mixture of monoclinic (2θ = 24.5, 28.2, 33.1, 47.3, 56.2) and tetragonal phases (2θ = 30.2, 35.3, 49.8, 60.0). However, diffraction patterns of MZ and P-SZ consisted of reflections related to only tetragonal phase. The PXRD pattern of SZ catalyst is not shown in the figure, as the patterns of SZ and P-SZ were found to be similar. The presence of tetragonal phase in either MZ or P-SZ indicates that the incorporation of Mo(VI) or Pt-SO_4^{2-} ions on zirconia can result in phase transition from monoclinic to catalytically active meta stable tetragonal phase[15]. Further, it is reported that the tetragonal phase of zirconia is more catalytically active.

Morphology of the catalysts

The morphology of the honeycomb form of solid acid catalysts (Z, MZ, P-SZ) were determined by analyzing their SEM images (Fig. 3). From these images, it is observed that the active catalytic material is firmly coated on the bare honeycombs and the coated catalytic materials are in the form of flakes.
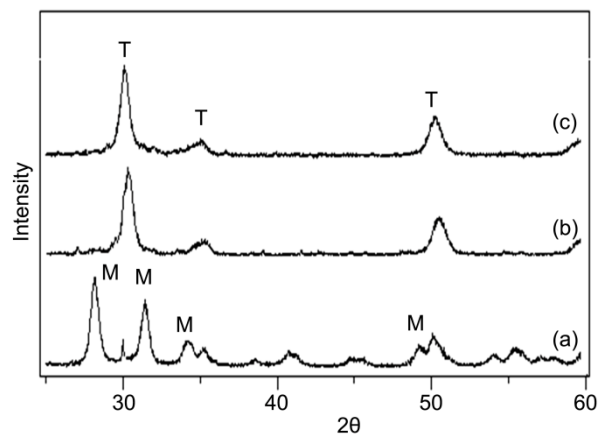
Table 1 — Acid site distribution and total surface acidity of catalysts used for the present work

Catalyst	Acid site distribution (mmols/ g)				Total surface acidity (mmol/g)
	Weak	Moderate	Strong	Very Strong	
ZrO_2	0.03	0.38	-	-	0.41
MZ	-	0.09	0.90	0.05	1.04
Pt-SZ	-	0.06	0.98	0.22	1.26
SZ	-	0.08	0.96	0.20	1.24

Fig. 1 — NH_3-TPD profiles of (a) Z, (b) MZ and (c) P-SZ.

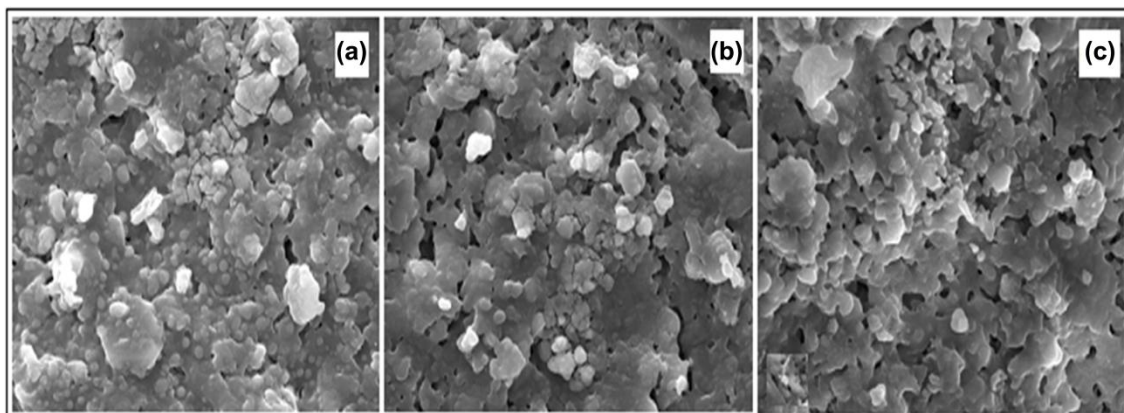Fig. 2 — PXRD patterns of (a) Z, (b) MZ and (c) Pt-SZ. *[M = Monoclinic; T = Tetragonal].*

Fig. 3 — SEM pictures of (a) Z, (b) MZ and (c) P-SZ. *[Magnification = 2000x ; Resolution = 10 μm].*

The firm coating of the catalytic material also indicates that the method used for coating is suitable.

Functionality of the catalysts

The functionality of the solid acid catalysts was analyzed by using FTIR technique. The FTIR spectra of Z, MZ and P-SZ are shown in Fig. 4. For all the catalysts the spectra consisted of peaks at 3400 cm^{-1}, 1646 cm^{-1} which are due to the presence of stretching and bending modes of -OH groups of water molecules, respectively. The presence of a band at 1361 cm^{-1} indicates the presence of M-OH bending mode. A peak at 1118 cm^{-1} in Fig. 4 indicates the presence of Zr-O-Zr stretching mode[16] which is getting diminished in spectra for MZ and P-SZ as shown in Fig. 4b and 4c. The FTIR spectrum of SZ catalyst was found to be almost similar to P-SZ.

Catalytic activity studies

Microwave assisted pinacol-pinacolone rearrangement reactions were carried out over Z, MZ, SZ and P-SZ (both powder as well as HC forms). The reactions were carried out in a specially designed reactor which can support honeycomb monoliths (Photo 1). For comparison, the reactions were also carried out by using conventional heating method. Initial studies were carried out by using 0.5 M benzopinacol solution in methanol (solvent). The values pertaining to the conversion (%) of benzopinacol over these catalysts are given in Table 2. A direct correlation between the acidity and the conversion of benzopinacol was observed. Pure zirconia being least acidic was less active in the pinacol rearrangement where as modified forms such as MZ, SZ and P-SZ were found to be highly active. Even though SZ and P-SZ were most active in this
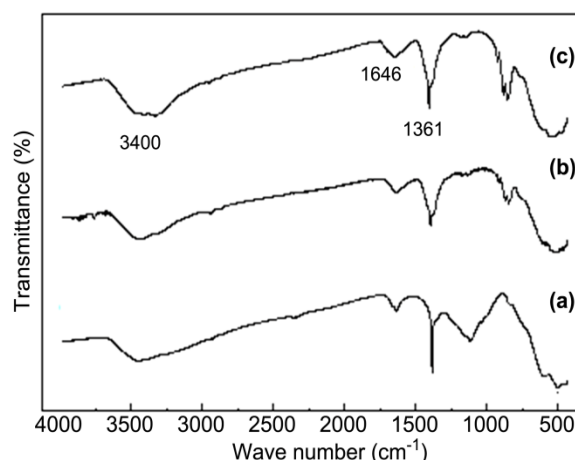

Fig. 4 — FTIR spectra of (a) Z, (b) MZ and (c) P-SZ.

Table 2 — Catalytic activity of Z, MZ, P-SZ and SZ (both powder asnd HC coated forms) pinacol-pinacolone rearrangement of benzopinacol

Catalyst	Conversion of benzopinacol (%)	
	Microwave heating	Conventional heating
Z	34 (16)	08 (05)
MZ	95 (42)	18 (10)
P-SZ	98 (46)	19 (10)
SZ	96 (43)	18 (09)

[Reaction conditions: Reaction temperature = 80 °C , reaction time = 10 min, catalyst weight = 0.02 g, reaction mixture = 0.5 M pinacol solution in methanol].

Note: Numbers in the parenthesis in refer to the values pertaining to powder form of the catalysts.

reaction, formation of by-products other than benzopinacolone was observed which makes SZ or P-SZ less selective than either Z or MZ. i.e., Z and MZ were 100% selective towards the formation of benzopinacolone. GC-MS analysis of the reaction mixture revealed that one of the by-product was

benzophenone. Formation of by-products can be attributed to the presence of 'very strong' acidic sites (super acidic) on P-SZ. Even though pure zirconia is 100% selective, conversion of benzopinacol is least when compared its modified forms. This information indicates that MZ is a suitable catalytic material among other materials used for the present study. Suitability of MZ catalyst for pinacol rearrangement of benzopinacol can also be attributed to the presence of 'moderate' and 'strong acidic' sites on it which may be desirable for the formation of benzopinacolone. Further optimization studies were conducted by using MZ as the catalyst for the above mention rearrangement reaction.

Effect of nature of catalyst (powder form v/s honeycomb coated form)

The rearrangement reactions were carried out over both powder forms as well as honeycomb coated forms of Z, MZ, SZ and P-SZ (Table 2). The numbers in the parentheses of Table 2 (Column 2 and 3) are the values of conversion of benzopinacol over powder form of the catalysts. Even though same amount of the catalyst (0.02 g) was used in both powder form as well as HC coated form, an almost 2 fold increases in the conversion of benzopinacol was observed when the catalysts were used in HC coated forms. This shows the effectiveness of a catalyst when coated on a honeycomb in the form of a thin layer. To conclude, HC coated form of the catalyst is more effective and efficient than the powder forms.

Effect of solvent

The pinacol rearrangement reactions were carried by using different solvents such as methanol, *n*-butanol and acetone. The results are presented in Table 3. It was observed that the polarity of the solvents play an important role in this reaction. Higher conversion of benzopinacol was observed when polar solvent such as methanol was used. However, the conversion of benzopinacol was low when acetone was used as a solvent. But, negligible conversion was seen when *n*-butanol which in highly

Table 3 — Effect of solvent on the yield of benzopinacolone

Solvent	Conversion of benzopinacol (%)
Methanol	95
n-butanol	05
Acetone	33

[Reaction conditions: Reaction temperature = 80 °C, reaction time = 10 min, catalyst weight = 0.02 g of MZ on HC, reaction mixture = 0.5 M pinacol solution in solvent]

polar solvent was used in the reaction. This study indicates that, a solvent with suitable polarity is desirable to obtain highest possible conversion of benzopinacol to the product. Hence, methanol was selected as a solvent for further studies.

Effect of heating method (microwave heating v/s conventional heating)

The pinacol rearrangement reactions were carried out by using two different methods such as conventional heating and microwave hating and the results are given in Table 2. Both types of reactions were carried out over HC coated with MZ catalytic material at 80 °C. In case of conventional heating, the reaction proceeded very slowly and conversion of benzopinacol was 18% in 10 min and reached 65% for 110 min where as in case of microwave heating method, the conversion was 95% in just 10 min. In conventional heating the heat is first imparted to the reactants by transferring through the wall of the vessel making it a very slow as well as an inefficient method with regards to transportation of energy into the reacting system. However, microwave assisted reactions are superior with higher reaction rates, reduced reaction time in managing and initiating the reaction equilibrium when compared to conventional heating method[17].

Effect of reaction temperature

The microwave-assisted rearrangement reactions were conducted at various temperatures ranging from 60 °C to 120 °C over HC coated with MZ catalytic material and the results are shown in Fig. 5. From the
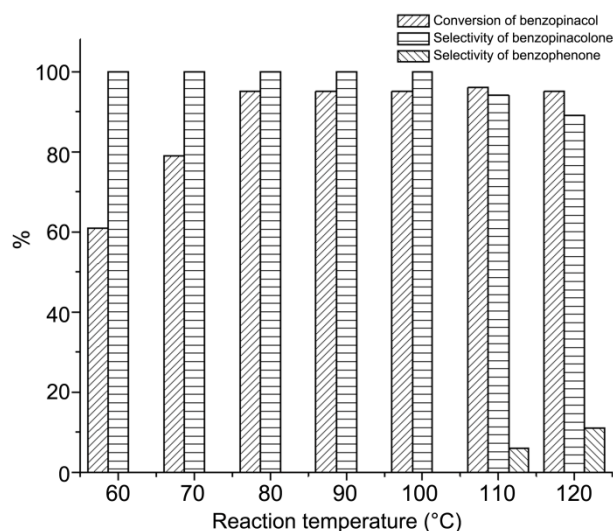

Fig. 5 — Effect of reaction temperature on the conversion of benzopinacol.

figure it is clear that conversion of benzopinacol increased when the reaction temperature was increased up to 80 °C, beyond which conversion (%) of benzopinacol had no effect i.e., not much increase in the conversion (%) was observed. Therefore, for further studies the reaction temperature of 80 °C was chosen as the optimized temperature. Interestingly, as the reaction temperature was increased beyond 100 °C, benzophenone was also found to form as the by-product. Benzophenone may be formed by the oxidation of benzopinacol.

Pinacol rearrangement of diols over HC coated with Mo(VI)/ZrO$_2$ (microwave synthesis)

Different diols were subjected to pinacol rearrangement over MZ catalytic material under microwave-irradiation under optimized reaction conditions and the results are presented in Supplementary Data, Table S1.

For comparison, the rearrangement reactions were also carried out using conventional heating method under optimized conditions with various diols. It was observed that the rearrangement reactions could be efficiently carried out over HM coated with MZ in good yields with a superior selectivity (100%) in a very short reaction time in 'minutes' where as it took

'hours' in conventional heating method. The effect of heating method with respect to the conversion of various diols is given in Table 4. Up to 95% conversion of diols to their respective products was obtained in just 10 min whereas approximately same conversion was achieved in 3 h when the reactions were carried out by conventional heating method. Therefore, microwave assisted synthesis could be inferred as a promising and an efficient technique for the synthesis of fine chemicals in terms of shorter reaction time with high purity product yield compared to conventional heating method.

In general, the tertiary diols (Entries 1, 2, 3, 5, 7, 8), the rearrangement reactions underwent smoothly and hence excellent conversion of diols (up to 95%) was observed. On the other hand cyclic diols (Entries 4, 6) like flurenone and anthrone pinacol underwent rearrangement to give ring enlargement products named as spiroketones (flurenyl and anthryl pinacolones, respectively).

Effect of reactivation and reusability of catalysts (both powder forms and HC forms)

The catalysts (MZ, SZ, P-SZ) after the reactivation were reused for 6 reaction cycles and the related data is presented in Fig. 6. From the figure it

Table 4 — Effect of heating method on the conversion of diols over MZ catalyst

Entry (Refer to Table S1)	Conversion of diols (%)	
	Microwave heating	Conventional heating
1	95 (10)	89 (180)
2	93 (10)	85 (180)
3	80 (10)	71 (180)
4	92 (10)	88 (180)
5	78 (10)	69 (180)
6	92 (10)	82 (180)
7	92 (10)	81 (180)
8	89 (10)	81 (180)

[Reaction conditions: Reaction temperature = 80 °C, catalyst weight = 0.02 g of MZ on HC, reaction mixture = 0.5 M pinacol solution in methanol]
Note: Numbers in the parenthesis refer to the reaction time (minutes)
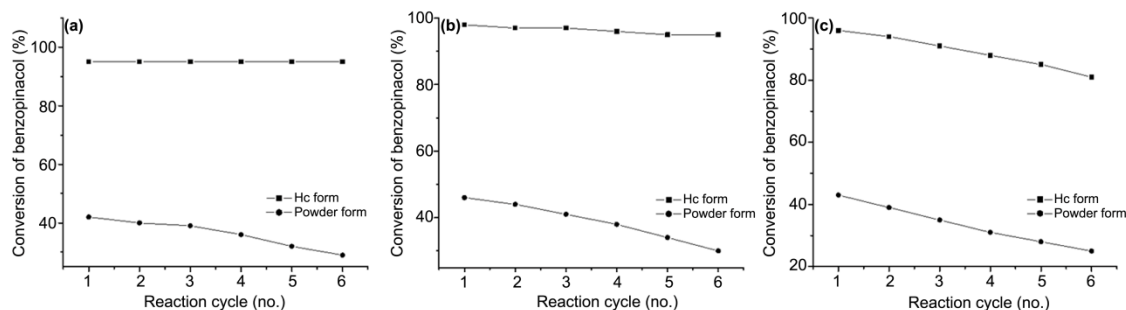

Fig. 6 — Effect of reusability of HC and powder forms of (a) MZ, (b) P-SZ and (c) SZ on the conversion of benzopinacol.

is clear that the HC form of the catalyst are more reusable than their powder forms. Low reusability of powder form of catalysts can be due to loss of the catalyst during separation process which is avoided in case of HC form of catalysts. This particular property makes the HC form of catalysts more economical as compared to the powders. Further, in case of SZ and P-SZ catalysts (either HC form or powder form), a decrease in the conversion of benzopinacol was observed when reused, which can be attributed to deactivation of active sites by strong adsorption of reactants or reaction intermediates on the super acidic sites of SZ or P-SZ (Fig. 6b). It was also observed that the extent of deactivation over SZ catalyst was found to be higher than on P-SZ catalyst (Fig. 6c). i.e., SZ get rapid deactivation upon reuse. This difference in the extent of deactivation can be attributed to the presence of Pt on P-SZ. It is reported that the deactivation of SZ can be prevented by the addition of Pt on SZ.[15] However, in case of MZ coated on HC (Fig. 6a), no such deactivation or loss of activity was observed which makes HC coated with MZ a facile and efficient catalytic material for Pinacol rearrangement.

Conclusions

Pinacol rearrangement of diols was effectively carried out on zirconia based solid acid catalysts, Mo(VI)/ZrO_2 was found to be an efficient catalyst for this reaction especially when coated on a honey comb carrier. Up to 98% conversion of pinacol (diol) into pinacolone with 100% selectivity was achieved under optimized reaction conditions. Methanol was found to be the most suitable solvent for the rearrangement reactions. A correlation between the total surface acidity and the catalytic activity of the catalysts was observed. Modified forms of zirconia i.e., Mo(VI)/ZrO_2 and Pt-SO_4^{2-}/ZrO_2 which possessed higher acidity were more active than pure zirconia. Further, the selectivity of the reaction products was influenced by the nature of acid sites on the catalyst. Mo(VI)/ZrO_2 which consisted of moderate and strong acid sites was more selective (100%) towards the formation of benzopinacolone where as Pt-SO_4^{2-}/ZrO_2 was less selective as it produced benzophenone as the by-product. When the catalytic activity of honeycomb coated and powder form of the catalysts was compared, honeycomb form of catalysts was found to be efficient, economical and reusable catalysts. Microwave assisted pinacol rearrangement of diols was a quicker and efficient technique when compared to conventional heating method. In general, a microwave-assisted reactions over honeycomb coated with zirconia based catalysts is an important and intelligent step towards the synthesis of industrially important fine chemicals.

Supplementary Data

Supplementary data associated with this article are available in the electronic form at: http://nopr.niscair.res.in/jinfo/ijca/IJCA_59A(02)181-188_SupplData.pdf.

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