SHORT COMMUNICATION



Phosphotungstic acid encapsulated in USY zeolite as catalysts for the synthesis of cyclohexylbenzene

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Abstract

In this work, a new type of catalyst, USY-HPW, was successfully prepared by encapsulating phosphotungstic acid (HPW) into ultra-stable Y zeolite (USY). The obtained catalyst USY-HPW was characterized by various techniques including N_2 adsorption/desorption isotherms, XRD, SEM, TG-DSC, XPS, NH₃-TPD, FT-IR, Py-IR. The catalytic properties of USY-HPW were evaluated by using a model alkylation reaction of benzene with cyclohexene to form cyclohexylbenzene. The conversion rate of cyclohexene can reach as high as 99.99%. Compared with USY alone, USY-HPW displayed markedly improved selectivity and yield for the target product of cyclohexylbenzene, ca. 5.41% and 8.73%, respectively. Besides, reusability tests indicated the high durability USY-HPW as the yield of cyclohexylbenzene can still reach to 83.50% after eight runs. All these results demonstrate that USY-HPW catalyst has good performances and holds good potential in acid catalyzed organic chemistry.

Graphical abstract



Keywords Ultra-stable Y zeolite (USY) · Phosphotungstic acid · Benzene · Cyclohexene · Cyclohexylbenzene

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Introduction

Cyclohexylbenzene is an important chemical intermediate with a wide range of applications, for example, as electrolyte additive for overcharge protection of lithium ion battery [1, 2] and the raw material in the synthesis of TFT liquid crystal materials [3]. In addition, the oxidation decomposition of cyclohexylbenzene can afford important chemical substances, such as phenol and cyclohexanone [4], which are critical raw materials for the production of bisphenol A,

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phenolic resin, adipic acid [5], as well as ε -caprolactone [6], adipic acid [7] and caprolactam [8]. As a result, cyclohexylbenzene has attracted more and more attention researchers, and the synthesis of cyclohexylbenzene has become a research hotspot.

Basically, synthesis cyclohexylbenzene is mainly divided into three broad categories, including the selective hydrogenation of biphenyl [9], the hydroalkylation of benzene [10–12] and the alkylation reaction of benzene and cyclohexene [13–16], which are summarized in Table 1. Among them, the hydroalkylation of benzene is the most simple and important method. But most of catalysts used in this method are precious metal compounds, and their catalytic conversion efficiencies still remain to be improved. The process of the selective hydrogenation of biphenyl is simple, but it is disappointing that the catalysts for this reaction are immature. In particular, the biphenyl is obtained via high-temperature dehydrocoupling or oxidative coupling of benzene with a low conversion and yield. Therefore, the research on the synthesis of cyclohexylbenzene via the alkylation of benzene and cyclohexene has become particularly urgent.

The traditional synthesis of cyclohexylbenzene utilizing sulfuric acid or hydrofluoric acid as catalyst can be conducted at mild reaction conditions with good yield. On the other hand, the reaction has many shortcomings such as large catalyst consumption, non-reusability, serious equipment corrosion and environmental pollution [17]. With the increasing requirements for environmental protection and resource utilization, the search for pollution-free, easily separated and recyclable catalysts has become a research hotspot [18–31]. Sato has studied the liquid-phase alkylation

of benzene with cyclohexene over SiO₂-grafted AlCl₃ catalyst and found that the catalyst was active but unsatisfactory reusability [13]. Song has developed a novel reusable catalytic system, $Sc(OTf)_2$ with [Bmim][SbF₆] ionic liquids, in the Friedel-Crafts alkylation of aromatic compounds with alkenes [14]. Alexander has found that in apolar reaction media the nitrosonium cation (NO⁺) activated alkenes toward electrophilic substitution of arene substrates to yield the alkylated arene [15]. Karshtedt has described a novel methodology for the hydroarylation of unactivated olefins utilizing a series of Pt complexes activated by Ag(I) salts [16]. However, these catalysts described above were expensive and just displayed moderate catalytic activity. At this time, solid acid catalyst stands out among many catalysts because of its high activity, good selectivity, reutilization and environmental friendliness [32].

Zeolites have good thermal and hydrothermal stabilities because of their special porous structure and adjustable acidity. Those make zeolites have good catalytic ability and high selectivity participating in Friedel–Crafts alkylation reactions [33]. USY zeolites have a large specific surface area and three-dimensional channels, containing a 1.30 nm super-cages which are connected by a twelve-membered ring channel with a pore diameter of 0.74 nm. The unique shape framework and acidic sites of the zeolites provide catalytic activity for the main catalytic processes inside the crystal [34].

Heteropoly acids (HPAs) can be easily dissolved in polar solvents and exhibit excellent catalytic performance in many reactions owing to its unique structures, strong acidity and oxidative properties. Phosphotungstic acid $H_3PW_{12}O_{40}$.

Table 1 List of synthesis methods of cyclohexylbenzene

Production methods	Synthetic process	Catalyst	Operation Condition	Results Conversion Selectivity
Hydrogenation of biphenyl [9]	← → → → → → → → → → → → → → → → → → → →	Skeletal Ni	343 K, 1.5 MPa	100% 95%–99%
Hydroalkylation of benzene and H_2 [10–12]	metal site H ₂ Acid site	Pd/HY	423 K, 2 MPa, H ₂ /C ₆ H ₆ 1	42% 77%
	~	Pt/Ni/Re- 13X	443 K, 3.5 MPa	20% 75%
		Mo(CO)/Y	363 K, 0.1 MPa	Conversion20%
Alkylation of cyclohexene and benzene [13–16]		SiO ₂ -AlCl ₃	253 K, 0.1 MPa	83.8% 97.2%
	×	Sc(OTf) ₃ [Bmim] [SbF ₆]	293 K, Mol=20:1	99% 90%
		NO ⁺	303 K Mol=9:1	78% 86%
		$AgBF_4$	353 K	Yield:65%

xH₂O (HPW) is one of the most widely used HPAs. But on the other hand, the small specific surface area, low thermal stability and difficulty in recovery restrict the use of HPW in zeolite catalysts. In order to exert its catalytic ability, HPW is usually incorporated in zeolites by impregnation or in situ synthesis methods which offer applications as Brønsted acid catalyst [35–39]. For example, HPW clusters with diameters 1 nm can be introduced inside the USY cages of 1.3 nm and 0.74 nm of channels, allowing for a significant increase in the surface area of the HPW with a possible increase in its thermal stability, in addition to creating Brønsted acid sites within the USY support. So the preparation of catalysts by encapsulating Zeolite with heteropoly acids is the main direction for the current research of heteropoly acid catalyst [40, 41].

In this article, we report a composite catalyst, USY-HPW, which was prepared by the encapsulation of HPW inside USY for catalyzing benzene alkylation with cyclohexene. USY-HPW displayed high catalytic efficiency as well as selectivity for the production of cyclohexylbenzene. The reusability tests showed that the USY-HPW still have high activity after running eight times. These results suggest that highly efficient USY-HPW catalyst can provide significant advances in the development of environmentally benign processes in the chemical industry.

Experimental

Chemical reagent and materials

Dibasic sodium phosphate ($Na_2HPO_4 \cdot 12H_2O$) and sodium tungstate dihydrate ($Na_2WO_4 \cdot 2H_2O$) were purchased from Tianjin Kemiou Chemical Reagent Co. Ltd., China. Hydrochloric acid (HCl) was purchased from Luoyang Chemical Reagent Co., Inc., China. USY zeolite was supplied by Tianjin Yuanli Chemical Co. Ltd., China. Benzene, cyclohexene, cyclohexylbenzene and decane were obtained from Shanghai Macklin Biochemical Technology Co. Ltd., China. Ethanol was purchased from Tianjin Sailboat Chemical Reagent Technology Co. Ltd., China.

Preparation of catalysts

An aqueous solution of a certain molar ratio of dibasic sodium phosphate and sodium tungstate dihydrate was added to the USY zeolites, and the reaction mixture was stirred at room temperature for 12 h, then the pH was adjusted to 2–3 with hydrochloric acid. After stirring at 80 °C for 24 h, the sample was filtered and washed with hot deionized water several times until neutral in order to remove the phosphotungstic acid anions on the external surface. Finally, the

sample was dried at 90 $^{\circ}$ C for 12 h and calcined at 400 $^{\circ}$ C for 3 h to afford the USY-HPW catalyst.

Characterization of catalysts

BET surface area and pore structure of USY and USY-HPW were characterized by measuring N2 adsorption/desorption isotherms with Quantachrome NOVA4200 distribution analyzer. SEM images were obtained using a Zeiss Auriga FIB-SEM. TG-DSC test was used STA 449 F3 Jupiter synchronous thermal analyzer to study the structural weight loss of substances and the decomposition of USY-HPW. The crystal structures of the catalyst were recorded on X-ray Power Diffraction (XRD) using Rigaku ULTIM III X-ray diffractometer. High resolution X-ray photoelectron spectroscopy (XPS) was carried out on a VG ESCALAB 250 to measure the composition of elements in the catalyst. NH₃-TPD experiments were characterized by auto-catalytic adsorption system Quantachrome Autosorb IQ 2.02 to perform the acidity of the catalyst. The infrared spectroscopies of the catalysts were performed on Fourier transform infrared spectrometer (FT-IR) using Nicolet Nexus 470. Lewis acid or Brønsted acid sites presented on the surface of the catalyst adsorbed with pyridine were carried out using Nicolet IR 200 spectrometer.

Evaluation of catalysts

The activity of the catalyst was evaluated by the alkylation reaction of benzene and cyclohexene in a 100-mL autoclave reactor at desired temperature. The catalyst, a certain amount of benzene and cyclohexene were placed into the autoclave which was pressurized to 3 MPa and heated to 150 °C. The reactor was naturally cooled to room temperature after the reaction was completed. Then, the catalyst was filtered and separated to afford a reaction mixture which was analyzed with GC7900 gas chromatograph. Conversion rates of cyclohexene and selectivity of cyclohexylbenzene were calculated with an FID detector using n-decane as an internal standard.

Results and discussion

Characterization

Figure 1 displays the XRD patterns of zeolite samples USY (A) and USY-HPW (B). The sample USY-HPW depicts the similar typical characteristic crystalline diffraction peaks with USY, which confirming that the framework structure of the zeolite is preserved after encapsulation. However, the peaks intensity in XRD also indicates the crystallinity of the zeolite. Obviously, the peaks intensity



Fig. 1 XRD patterns of USY (a) and USY-HPW (b)

of USY-HPW decrease compared with that of USY. The reason is that the relative strong acid used in the synthesis of USY-HPW makes the aluminum species dissolved which leading to the collapse of structure and decrease in relative crystallinity.

SEM images of USY and USY-HPW are shown in Fig. 2. USY-HPW maintained the basic shape of the USY. In detail, as presented in Fig. 2a and 2b, the surfaces of USY are intact and smooth, no obvious pores appears. From Fig. 2c and 2d, it can be significantly rough. The amount of small particles adsorbed on the surface of the USY increase slightly after the treatment. Therefore, the encapsulation has a significant etching effect on the zeolite, which affects the pore and textural property of USY-HPW.

The N₂ adsorption/desorption isotherms of USY and USY-HPW are shown in Fig. 3a. The isotherm slope of the USY-HPW is larger than that of the zeolite USY, indicating the formation of mesopores after encapsulating. And the range of the hysteresis loop is slightly expanded, which means that the pore size of catalyst becomes larger after encapsulating in phosphotungstic acid. The pore sizes distribution of the catalysts exhibited in Fig. 3b are around 3.82 and 3.89 nm for USY and USY-HPW. Apparently, the total pore volume of USY-HPW is larger than showed that of USY (Fig. 3b and Table 2). In addition, the characterization shows that the BET surface area and total pore volume have greatly improved after encapsulating phosphotungstic acid, which can facilitates the alkylation reaction of benzene and cyclohexene. After reaction, the isotherm slope and range of the hysteresis loop of catalyst become smaller, which indicates the pore size same situation. The reason for the decrease in BET surface area and total pore volume (Table 2) is that catalyst sintered at high temperature or some organic compounds deposited on the surface and block the pores of USY-HPW, which make the activity loss and the catalytic performance of USY-HPW decline.

In order to get more deep insight of the USY-HPW catalyst, the XPS analysis is carried out (Fig. 4). The survey spectrum (Fig. 4a and 4b) indicates the presence of phosphorus and tungsten after encapsulating phosphotungstic acid into USY. As shown in Fig. 4c, the peaks at 33.35 eV and 35.5 eV correspond to W4f_{7/2} and W4f_{5/2}, respectively. The



Fig. 2 SEM images of USY(**a** and **b**) and USY-HPW(**c** and **d**)



Fig. 3 N₂ adsorption/desorption isotherm (a) and pore volume distribution (b) of USY and USY-HPW

Table 2 Textural properties of different catalysts

Catalysts	$S(m^2/g)^a$	V(cm ³ /g) ^b	D(nm) ^c
USY	304.17	0.20	3.82
USY-HPW	768.30	0.37	3.89
After Reaction	237.77	0.17	3.83

^aBET surface area

^bTotal pore volume calculated by single point method at P/Po=0.99 ^cBJH desorption average pore diameter

difference of binding energy between W4f_{7/2} and W4f_{5/2} is 2.15 eV. In the high-resolution P2p spectra (Fig. 4d), the peaks at 131.37 eV and 132.15 eV are related to P2p3/2 and P2p1/2, respectively. Combined with the XPS analysis, it can be confirmed that Keggin-type heteropoly acid (HPW) formed between the phosphorus and tungsten encapsulated in USY during the preparation of the USY-HPW catalyst.

According to the TG-DSC characterization analysis of HPW-USY catalyst (Fig. 5), the TG curve corresponds to the change of sample mass at different temperatures and the mass decreases with increasing temperature. It is observed that its weight loss is mainly divided into three processes. It is physically adsorbed water below 100 °C and bound water of the catalyst between 100 °C and 260 °C. The mass loss is 12.88% in the above temperature range, which shows a slightly bulging exothermic peak on the DSC curve. There is an endothermic peak at 260 °C. At the same time, the sintering reaction on the surface of the sample starts to proceed, and the weight loss rate is very fast. At this stage, the mass loss is 39.71%. When it is above 350 °C, there is a 39.71%mass loss. It may be the structural water of phosphotungstic acid. After 460 °C, the phosphotungstic acid begins to decompose, so the DSC curve shows a large exothermic process and reach a peak at 670 °C. At that time, the structure change of USY-HPW and the framework collapse occur, which may cause a decrease in catalyst activity.

The NH₃-TPD tests of the USY and USY-HPW are shown in Fig. 6, the two kinds of acid sites are corresponding to the weak acid peak around 160 °C and the strong intensity acid site appears at a high temperature around 450 °C, respectively. The result indicates that the acid center has been changed and relatively reduced after phosphotungstic acid being encapsulated in the USY, which may be the ascribed to interaction between the HPW and the USY. USY-HPW not only maintains the large specific surface area and the regular pore structure of USY, but also changes its acid amount and strength, which will facilitate the reaction between benzene and cyclohexene.

The FT-IR was also tested in order to get more details of the USY-HPW catalysts (Fig. 7), there are two significant differences between USY-HPW and USY in the area around 3500 cm^{-1} and 1050 cm^{-1} . Skeleton vibration spectrum of zeolite appears in the range of $200-1300 \text{ cm}^{-1}$, obviously at two spectral bands 800 cm^{-1} and 1020 cm^{-1} . The distinct decrease in absorption indicates the change of the skeleton in the USY-HPW catalyst. The broad stretching intense characteristic peak appearing around 3500 cm^{-1} is slightly wider in USY-HPW than in USY, which may be owing to a combination of vibrations related to hydroxyl groups in the pore structure. An absorption band at $555-560 \text{ cm}^{-1}$ is the signal of vibrations of the structural double ring in the USY zeolite.

At last, the Py-IR (infrared spectra of pyridine adsorbed) test of USY-HPW catalysts was conducted. The absorption spectrum of coordination bond with pyridine at 1450 cm⁻¹ corresponds to the Lewis acid center and the absorption peak of pyridine ion at 1540 cm⁻¹ can be used as a measure of the Brønsted acid site. In general, Lewis acid and Brønsted acid has a large amount of acid site at 100 °C. It can be seen that the amount of acid decreases rapidly with the increase in temperature in the Py-IR spectra patterns of USY-HPW



Fig. 4 XPS analysis: (a and b) XPS survey spectra of (a) USY and (b) USY-HPW; XPS spectra of (c) W4f, (d) P2p of USY-HPW



Fig. 5 TG-DSC curves of the sample USY-HPW

catalysts (Fig. 8). The lower temperature corresponds to the weak acid site, but the strong acid site corresponding to 400 $^{\circ}$ C is not detected, which means that there are no strong acid centers in the catalyst. Therefore, the weak acid site of the catalyst is the active center of the reaction. It can be deduced that the acid strength is weakened by the



Fig. 6 NH_3 -TPD of USY (a) and USY-HPW(b)

interaction between phosphotungstic acid and USY zeolite, which is mutually confirmed with the results of NH_3 -TPD characterization (Fig. 6).



Fig. 7 FT-IR patterns of USY (a) and USY-HPW (b)



Fig. 8 Py-IR spectra patterns of USY-HPW

Catalytic performance

The catalytic performance of these catalysts has been examined in the synthesis of cyclohexylbenzene from benzene and cyclohexene, and the reaction results are shown in Table 3. It can be found that USY-HPW catalyst is more efficient than USY. The detected conversion rates of cyclohexene can reach up to 99.99% by using USY-HPW. Compared with USY catalyst, the selectivity and yield of cyclohexylbenzene corresponding to USY-HPW catalysts are increased by 5.41% and 8.73%, respectively. Therefore, USY-HPW catalyst presents higher conversion, selectivity and yield than USY catalyst. Durability is an important aspect of any industrial process, and it is also an aspect of investigating the performance of catalysts. Thus, reusability of the catalyst

Table 3	Reactivity	of USY	and USY-HPW	catalysts
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Catalysts	Conversion (%)	Selectivity (%)	Yield (%)	
USY	Y 96.32 90.27		86.95	
USY-HPW	99.99	95.68	95.68	

Reaction conditions: benzene (0.144 mol), cyclohexene (0.012 mol), catalyst (0.3 g), reaction pressure filled with N₂ (3 MPa), reaction temperature (150 °C) and reaction time (3 h)



Fig. 9 Reusability of USY-HPW

was tested by carrying out repeated runs of the reaction at the same conditions. After each reaction, the catalyst is separated by centrifugation, then dried and calcined for the next reaction. It's worth mentioning that the yield of cyclohexylbenzene is up to 83.50% after the running of eighth times (Fig. 9). These results demonstrate that phosphotungstic acid encapsulated in the USY zeolite (USY-HPW) can improve the reactivity and durability in synthesizing cyclohexylbenzene from benzene and cyclohexene.

Conclusions

A new USY-HPW catalyst was successfully prepared through encapsulating the phosphotungstic acid in USY zeolite. The performance of USY-HPW catalyst in facilitating the alkylation reaction of benzene and cyclohexene to synthesize cyclohexylbenzene was studied. It was found that USY-HPW catalyst had Brønsted acid sites and showed good catalytic activity. Especially, the conversion rate of cyclohexene reached 99.99%, and the selectivity of cyclohexylbenzene was 95.68%. Moreover, USY-HPW exhibited excellent durability as the yield of cyclohexylbenzene can still reach 83.50% during the eighth use of catalyst. All in all, the HPW-USY catalyst presents higher activity and stability than USY zeolite in the synthesis of cyclohexylbenzene from the reaction between benzene and cyclohexene.

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