CO₂气氛下 MCF 负载氧化钒催化剂上乙苯脱氢反应

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摘要: 以介孔氧化硅泡沫 MCF 为载体合成了一系列负载型氧化钒催化剂 (V 含量为 2%~10%)。采用 N₂ 吸附、X 射线衍射和 H₂ 程序升温还原对 V/MCF 催化剂的结构和织构性质进行了表征, 并评价了催化剂在 CO₂ 气氛下的乙苯脱氢性能。V/MCF 催化剂具有较高的乙苯脱氢活性, 其中 V 含量为 6% 的催化剂具有最高的反应活性。V/MCF 催化剂的乙苯脱氢活性显著高于 V/MCM-41, 这是由于前者具有较高的可还原性以及较好的扩散性能。CO₂ 气氛下的乙苯转化率明显高于 N₂ 气氛下的, 这归因于 CO₂ 与乙苯发生氧化脱氢, 并通过逆水煤气变换反应在线除去脱氢反应生成的氢。

关键词: 氧化钒; 介孔氧化硅泡沫; 负载型催化剂; 乙苯脱氢; 苯乙烯; 二氧化碳

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Ethylbenzene Dehydrogenation in the Presence of CO₂ over MCF-Supported Vanadium Oxide Catalysts

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Abstract: A series of vanadia catalysts supported on mesocellular silica foam (MCF) with a V content ranging from 2% to 10% were studied with respect to their performance in the dehydrogenation of ethylbenzene (EB) to styrene in the presence of CO₂. The structural and textural characterization of these catalysts was done using N₂ adsorption, X-ray diffraction, and temperature-programmed reduction. These catalysts were found to be effective for the dehydrogenation reaction and the 6%V/MCF catalyst showed the highest activity. The MCF-supported vanadia catalysts exhibited far higher activity than their conventional V/MCM-41 counterparts, which can be attributed to the higher reducibility and better diffusion of reactants and products in the former catalysts. A higher conversion was obtained during EB dehydrogenation with CO₂ than with N₂. This is due to the oxidative dehydrogenation of EB with the help of oxygen that originates from CO₂ as well as the coupling of EB simple dehydrogenation with the reverse water-gas shift reaction.

Key words: vanadia; mesocellular silica foam; supported catalyst; ethylbenzene dehydrogenation; styrene; carbon dioxide

Styrene is one of the most important monomers in the petrochemical industry and is extensively used for the manufacture of polymers, copolymers, and reinforced plastics. Commercially, styrene is mainly produced by the catalytic dehydrogenation of ethylbenzene (EB) in the presence of a large amount of superheated steam at a high tempera-

ture (600–650 °C) with K-promoted iron oxide as a catalyst [1]. However, it is well known that this traditional process suffers from several disadvantages such as high energy consumption and thermodynamic equilibrium limitations. Alternatively, the use of CO₂, which is a mild oxidant and a greenhouse gas for the selective dehydrogenation of EB to

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styrene, has attracted much recent attention [2–10]. This new route is believed to be an energy-saving and environmentally benign process. It is estimated that the energy required for producing 1 ton of styrene in the new process using CO_2 is 7.9×10^8 J, which is only 13% of that needed in the current commercial process using steam [11]. Moreover, it provides a higher equilibrium yield of styrene [11]. Contrary to the process that uses oxygen as a strong oxidant, the dehydrogenation of EB with CO_2 (a soft oxidant) produces styrene with much higher selectivity. Because the industrial Fe-K based catalyst is not effective for EB dehydrogenation with CO_2 , a range of catalysts based on supported or mixed oxides of Fe, V, Cr, Ce, Zr, etc., have been investigated for this process [2–10]. Among them, vanadia and chromia exhibited high catalytic activity for the dehydrogenation of EB with CO_2 .

A vanadia catalyst supported on a mesoporous silica such as MCM-41 and SBA-15 had good performance for the dehydrogenation of EB with CO_2 [9,10,12,13]. Unlike MCM-41 and SBA-15, which have one-dimensional mesopore structures, mesocellular silica foam (MCF) is a new class of three-dimensional hydrothermally robust material with ultra-large mesopores (up to 50 nm) [14]. MCF materials have advantages over their more ordered MCM-41 and SBA-15 counterparts in terms of better reactant and product diffusion, which allows them to overcome internal mass transfer limitations [15,16]. However, catalytic applications of metals incorporated into MCF-type materials are scarce. In this study, we report on the catalytic behavior of MCF-supported vanadia catalysts for the dehydrogenation of EB with CO_2 . For comparison, a vanadium oxide catalyst supported on MCM-41 was also studied.

1 Experimental

1.1 Catalyst preparation

The mesoporous MCF support was prepared as described previously [14] using a the Pluronic P123 triblock copolymer ($\text{EO}_{20}\text{PO}_{70}\text{EO}_{20}$) surfactant with 1,3,5-trimethylbenzene (TMB) as the organic swelling agent. A solution of P123: TMB:1.6 mol/L HCl:TEOS = 2:1.5:75:4.25 (mass ratio) was prepared at 40 °C. After 24 h at 40 °C, the milky reaction mixture was transferred to an autoclave and crystallized at 100 °C for 48 h. The solid product was filtered, washed, dried at 90 °C, and eventually calcined at 600 °C for 5 h in air to remove the template.

The MCF-supported vanadia catalysts were prepared by the impregnation of MCF with a certain amount of NH_4VO_3 dissolved in an aqueous solution of oxalic acid. After impregnation, the catalysts were dried and calcined in air at

550 °C for 4 h. The obtained catalysts are denoted $n\text{V}/\text{MCF}$, where n represents the V content in the catalysts. For comparison, a 6%V/MCM-41 catalyst with a V content of 6% was prepared in the same way using mesoporous MCM-41 as a support.

1.2 Catalyst characterization

Small-angle X-ray scattering (SAXS) experiments were performed on a Bruker NanoSTAR U SAXS system equipped with a high-resolution pinhole chamber using $\text{Cu } K_\alpha$ radiation and a 106 cm sample-to-detector distance. The wide-angle X-ray diffraction (XRD) patterns were collected on a Bruker D8 Advance X-ray diffractometer using $\text{Cu } K_\alpha$ radiation at 40 kV and 20 mA. The BET specific surface areas and pore volumes of the catalysts were determined by the adsorption/desorption of nitrogen at liquid nitrogen temperature with a Micromeritics ASAP 2010 instrument. The pore sizes of the catalysts were obtained from the peak positions of the distribution curves determined from the adsorption branches of the isotherms. Temperature-programmed reduction (H_2 -TPR) profiles were obtained on a Micromeritics AutoChem II apparatus loaded with 100 mg of catalyst. H_2 -TPR experiments were carried out in 10% H_2 -90% Ar flowing at 30 ml/min, with a ramping rate of 10 °C/min from 200 to 750 °C.

1.3 Catalytic tests

The oxidative dehydrogenation of EB in the presence of CO_2 was carried out in a flow-type fixed-bed microreactor at 550 °C under atmospheric pressure. To supply the reactant, a gas mixture of N_2 and CO_2 (6:1 molar ratio, unless otherwise stated) at a flow rate of 60 ml/min was passed through a glass evaporator filled with liquid EB and maintained at 15 °C. The catalyst load was 80 mg, and the molar ratio of CO_2 to EB was kept at a constant 20 unless otherwise stated. Before the reaction, the catalyst was pretreated at 550 °C in N_2 for 2 h. The hydrocarbon products were analyzed using an on-line gas chromatograph (GC) equipped with a flame ionization detector and a 2 m long stainless steel column packed with 15% DNP. CO and CO_2 were analyzed on-line by another GC equipped with a thermal conductivity detector and a 6 m long stainless steel column packed with Porapak Q.

2 Results and discussion

2.1 Characterization of catalysts

2.1.1 XRD results

The MCF-supported vanadia samples were analyzed using SAXS. X-ray scattering is observed if uniformly sized particles are present as opposed to X-ray diffraction, which is associated with periodic structures [14]. The SAXS data of the V/MCF catalysts with V loadings ranging from 2% to 10% are shown in Fig. 1. The scattering pattern of the MCF support is well resolved and shows one intense primary peak and two higher-order peaks with exponentially decreasing intensities. The occurrence of higher-order peaks is indicative of the narrow size distribution of the spherical cells. A decrease in the scattering intensity with an increase in vanadium loading is observed, which may be caused by a partial structural collapse of the MCF pores [14]. However, the mesocellular arrangement of the MCF framework was still well retained in the MCF-supported samples at a V content of up to 6%. As the vanadium content increased to 8% and 10% the scattering peaks became obscure, implying that the MCF structure had collapsed. This was also clearly seen by the substantial decrease in surface area and pore volume for the 8%V/MCF and 10%V/MCF catalysts as compared with the parent MCF (Table 1).

Wide-angle XRD patterns of the V/MCF samples with various V content are shown in Fig. 2. No diffraction peaks

Fig. 1. Small-angle X-ray scattering patterns of MCF-supported vanadia catalysts. (1) MCF; (2) 2%V/MCF; (3) 4%V/MCF; (4) 6%V/MCF; (5) 8%V/MCF; (6) 10%V/MCF.

corresponding to vanadium oxides were detected in the V/MCF catalysts with a V content of up to 6% suggesting that the vanadium species in these samples are highly dispersed on the surface. As the V content was increased to 8%, weak diffraction peaks of crystalline V₂O₅ appeared indicating the formation of bulk vanadia at a high V content. Increasing the V content further on the MCF support results in an increase in the peak intensity. In contrast, the 6%V/MCM-41 catalyst shows weak diffraction peaks due to the crystalline V₂O₅, indicating that the aggregation of V₂O₅ crystallites in the V/MCF catalysts begins at a V content higher than that contained in the V/MCM-41 catalysts.

Fig. 2. Wide-angle XRD patterns of the mesoporous silica supported vanadia catalysts. (1) 6%V/MCM-41; (2) 2%V/MCF; (3) 4%V/MCF; (4) 6%V/MCF; (5) 8%V/MCF; (6) 10%V/MCF.

2.1.2 N₂ adsorption

The textural properties of the mesoporous silica supported vanadia catalysts are summarized in Table 1. As the V loading increases, the surface area and pore volume of the V/MCF samples reduce gradually. A high surface area and pore volume are observed in the MCF-supported samples with a V loading of up to 6%, which is indicative of the preservation of the MCF framework. This is consistent with the SAXS results (Fig. 1). The higher pore diameter ob-

Table 1 Physico-chemical characteristics of the mesoporous silica supported vanadia catalysts

Sample	$A_{\text{BET}}/(\text{m}^2/\text{g})$	$V_p/(\text{cm}^3/\text{g})$	D/nm	H ₂ -TPR results	
				$T_M/^\circ\text{C}$	H ₂ uptake (mmol/g)
MCF	612	1.67	22.0	—	—
2%V/MCF	531	1.57	22.0	510	0.111
4%V/MCF	455	1.45	22.0	505	0.275
6%V/MCF	420	1.34	22.1	442	0.495
8%V/MCF	225	0.89	29.3	496	0.707
10%V/MCF	199	0.78	31.3	505	0.991
MCM-41	949	0.94	3.0	—	—
6%V/MCM-41	531	0.60	2.8	513	0.530

D —Pore diameter at the maximum pore size distribution; T_M —Temperature of maximum hydrogen consumption.

served for the 8%V/MCF and 10%V/MCF samples compared to the parent MCF is related to the collapse of the MCF structure in the two former samples.

2.1.3 H₂-TPR results

To investigate the reducibility of the vanadium species dispersed on the support, H₂-TPR profiles of the V/MCF samples with various V content obtained between 200–750 °C are compared in Fig. 3, and the H₂-TPR data is summarized in Table 1. All the V/MCF samples only have one sharp reduction peak at 442–510 °C, which is assigned to the reduction of V⁵⁺ [17]. The peak temperature decreases with an increase in the V content from 2% to 6% suggesting that larger VO_x domains may be favorable for the reduction of vanadium species. Increasing the V content on the MCF support leads to a higher peak temperature indicating that the formation of bulk V₂O₅ will retard the reduction. The 6%V/MCF sample has the highest reducibility. In contrast, a close comparison of the profiles for the 6%V/MCF and 6%V/MCM-41 samples reveals that at the same V content level, the reduction peak temperature is much lower for the MCF catalyst than for its MCM-41 counterpart. This implies that the 6%V/MCF catalyst exhibits a higher reducibility than 6%V/MCM-41.

Fig. 3. H₂-TPR profiles of the mesoporous silica supported vanadium oxide catalysts. (1) 2%V/MCF; (2) 4%V/MCF; (3) 6%V/MCF; (4) 6%V/MCM-41; (5) 8%V/MCF; (6) 10%V/MCF.

2.2 Catalytic performance

The dehydrogenation of EB was performed at 550 °C using CO₂ as a mild oxidant over the MCF-supported vanadium oxide catalyst. Styrene is the major product in this reaction whereas benzene and toluene are minor by-products. For all catalysts in this study, the selectivity for styrene was more than 98%. Figure 4 is a plot of the dehydrogenation of EB on the V/MCF catalysts with varying

Fig. 4. EB conversion as a function of reaction time on the supported vanadia catalysts. (1) 2%V/MCF; (2) 4%V/MCF; (3) 6%V/MCF; (4) 8%V/MCF; (5) 10%V/MCF; (6) 6%V/MCM-41.

vanadium content as a function of reaction time. This figure shows that the EB conversion declines gradually with time on stream, and this is indicative of catalyst deactivation, which is often encountered in most catalytic systems during the dehydrogenation of EB and is primarily due to coke formation [9,18,19]. The catalysts are black after reacting for 6 h, which is indicative of coke formation. It is also clear from Fig. 4 that the catalytic activity of the V/MCF catalysts is strongly dependent on the V loading. EB conversion initially increases with the increase of V content from 2% to 6%. A further increase in the V content leads to a decrease in EB conversion. The highest activity for EB dehydrogenation was achieved on the catalyst with a vanadium content of 6%, which demonstrates the excellent performance of 6%V/MCF for the oxidative transformation of EB in the presence of CO₂. The 6%V/MCF catalyst gives a 70.7% conversion of EB with 98.0% selectivity for styrene after 0.5 h on stream and a 56.0% conversion of EB with 98.5% selectivity for styrene after 6 h on stream. We observed similar catalytic behavior in our previous study for V catalysts supported on MCM-41 [9]. The redox behavior of vanadium oxide plays a key role in EB dehydrogenation with CO₂ over the vanadium oxide-based catalysts, and the facile redox cycle between fully oxidized and reduced vanadium species yields a more effective catalyst [18,20,21]. Therefore, the increase in conversion with V loading can be attributed to an increased number of active V⁵⁺ sites as shown by the higher H₂ consumption as well as the higher reducibility, which was evident because of the lower temperature of the reduction peak. The decrease in conversion with a further increase in vanadium loading is due to the loss of active V⁵⁺ sites. These sites are accessible to the reactants on the catalyst surface and result from the enhanced formation of bulk V₂O₅ crystallites (Fig. 2) and the

lower reducibility as evidenced by the higher temperature of the reduction peak. The 2%V/MCF catalyst has a far lower activity than the 8%V/MCF and 10%V/MCF catalysts, which can be attributed to the far less active V⁵⁺ sites present on the former catalyst, and these catalysts have a similar reduction peak temperature.

We compared the dehydrogenation of EB on the vanadia, which was dispersed on the two different mesoporous siliceous supports (MCF and MCM-41) at the same V loading of 6%. Figure 4 shows that the catalytic activity of the 6%V/MCM-41 catalyst is obviously lower than that of 6%V/MCF. The 6%V/MCM-41 catalyst only gives a 57.1% EB conversion after reaction for 0.5 h and a 33.0% EB conversion after reaction for 6 h. This finding suggests that, with a similar V content, MCF is superior to MCM-41 as a support. From the H₂-TPR results, the V-containing mesoporous MCF is more active than the V/MCM-41 catalyst because the former catalyst exhibits enhanced reducibility, as indicated by the lower reduction peak temperature. MCF has a three-dimensional framework structure with ultra-large mesopores (ca. 22 nm) whereas MCM-41 has a one-dimensional hexagonal structure with relatively small mesopores (ca. 3 nm). Therefore, the better diffusion of reactants and products in the V/MCF catalyst compared with the V/MCM-41 catalyst could be another reason for its superior performance. Liu et al. [22] found faster molecular transport in the V/MCF material compared to V/MCM-41 by in situ Raman spectroscopy, and the former catalyst showed higher activity for the oxidative dehydrogenation of propane to propylene under O₂.

Carbon monoxide was detected in the effluent gases suggesting that carbon dioxide can behave as a soft oxidant for the oxidative dehydrogenation of EB to styrene. To understand the role of CO₂ in the reaction, we measured the amount of CO in the reaction product with reaction time on the 6%V/MCF catalyst and the results are shown in Fig. 5. The amount of CO that forms in the reaction is ca. 80% that of styrene. Carbon monoxide can be formed through the

Fig. 5. Formation rate of styrene (1) and CO (2) during EB dehydrogenation with CO₂ at 550 °C over the 6%V/MCF catalyst.

direct reaction of CO₂ and EB (i.e., direct oxidative dehydrogenation of EB with CO₂) [7,9,20,23,24] as well as by the reverse water-gas shift reaction. The data in Fig. 5 reveals that both a one-step pathway (i.e., direct oxidative dehydrogenation of EB with CO₂) and a two-step pathway (i.e., reaction coupling of simple dehydrogenation of ethylbenzene with the reverse water-gas shift) are at work during the dehydrogenation of EB with CO₂ [2,9,11,25], as shown below:

To further investigate the influence of CO₂ partial pressure on the catalytic behavior of the 6%V/MCF catalyst, the change in EB conversion after being on stream for 0.5 and 6 h versus CO₂ partial pressure is shown in Fig. 6. The partial pressure of CO₂ changed after adjusting the molar ratio of CO₂ and N₂, while the EB partial pressure, total flow rate and other reaction conditions were kept unchanged. In the absence of CO₂, the 6%V/MCF catalyst gives 63.2% and 45.0% conversion after reaction for 0.5 and 6 h, respectively. Upon introducing a small amount of CO₂ into the reaction, the EB conversion on the 6%V/MCF catalyst increases obviously suggesting that the dehydrogenation reaction was promoted by carbon dioxide. The conversion increases by raising the partial pressure of CO₂ to 14 kPa. A further increase in the CO₂ partial pressure results in a slight decrease in EB conversion. A similar phenomenon was observed over alkali-promoted Fe/active carbon and MCM-41-supported vanadia catalysts [9,26]. There are two causes for the promoting effect of CO₂ on EB dehydrogenation. One is the oxidative dehydrogenation of EB by the oxygen species that originates from CO₂ [20,23,24,27] and the other is the coupling of EB simple dehydrogenation with the reverse water-gas shift reaction, which can shift the dehydrogena-

Fig. 6. Effect of partial pressure of CO₂ on EB conversion over the 6%V/MCF catalyst during the on stream reaction for 0.5 h (1) and 6 h (2).

tion reaction toward the product side [28,29]. A separate reverse water-gas shift reaction was performed at 550 °C by feeding a mixed gas of CO₂ and H₂ over the 6%V/MCF catalyst, and the result is given in Fig. 7. It is clear that the catalyst is active in the reverse water-gas shift reaction.

Fig. 7. Result of the reverse water-gas shift reaction on the 6%V/MCF catalyst. Reaction conditions: 80 mg catalyst, 550 °C, N₂:CO₂:H₂ molar ratio = 25:4:1, total flow rate 60 ml/min.

3 Conclusions

A series of mesoporous V-containing mesocellular silica foam catalysts (V/MCF) were prepared by the wet impregnation method. The mesocellular arrangement of the MCF framework is retained upon vanadium incorporation at least up to a V loading of 6% and, therefore, high surface areas were obtained for the final catalysts. The V/MCF catalysts have high activity for the dehydrogenation of EB to styrene with CO₂. The 6%V/MCF catalyst shows the highest activity. An EB conversion of 70.7% with a styrene selectivity of 98.0% was achieved using this catalyst at 550 °C and after reaction for 0.5 h. EB conversion on the MCF-supported vanadia catalysts is much higher than that on their conventional V/MCM-41 counterparts, which can be attributed to the higher reducibility and favorable conditions for internal mass transfer in the former catalysts. Higher conversions can be obtained for EB dehydrogenation with CO₂ than with N₂, which is caused by the oxidative dehydrogenation of EB by oxygen species that originate from CO₂ as well as the coupling of EB simple dehydrogenation with the reverse water-gas shift reaction.

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