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Carbon nanofiber/graphite-felt composite supported Ru catalysts for hydrogenolysis of sorbitol

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A B S T R A C T

Carbon nanofiber/graphite-felt (CNF/GF) composite was used to support ruthenium (3.0 wt.%) for sorbitol hydrogenolysis. The CNF/GF composite was synthesized by in situ growing a CNF layer on graphite felt using Ni as catalyst and ethane as carbon source, and Ru was deposited by incipient wetness impregnation. The composite and catalyst were characterized by scanning electron microscopy, N_2 physisorption, X-ray diffraction, and high resolution transmission electron microscopy. The monolithic Ru catalyst was then used as stirring blades in an autoclave, in which sorbitol hydrogenolysis was carried out. The results showed that, compared with the powder Ru/CNF catalyst, the structured Ru catalyst had a lower activity but a much higher total selectivity, from 57.0% to 79.1%, to ethylene glycol, propylene glycol and glycerol.

1. Introduction

Fibrous carbon, as a novel carbonaceous material, has demonstrated outstanding catalytic performance in heterogeneous catalysis especially for liquid phase reaction because of its mesoporosity and low diffusion resistance as compared with other conventional supports [1–3]. However, the catalyst with carbon nanofibers (CNFs), which is usually in powder form, has some drawbacks concerning its commercial application. The ultrafine particles cannot be used in fixed bed reactors and would be very difficult to be recovered when used in slurry reactors. Therefore, a number of efforts have been made to incorporate CNFs into a macroscopic structure in the last few years. One approach was to grow CNFs on-site on a macroscopic shaping substrate material, e.g. metal filters [4] or foams [5,6], graphite felts [7], monoliths [8,9], and glass fibers [10], to form structured CNF/substrate composites. The application of these structured composites is supposed to combine the advantages of slurry phase reaction (short diffusion length) and fixed bed operation (without catalyst attrition, agglomeration and separation). Nevertheless, there are few investigations concerning the application of these CNF-based composites in catalysis especially in liquid phase reaction so far.

On the other hand, conversion of biomass to commodity chemicals has received extensive attention owing to the scarcity and high expenses of fossil resources [11,12]. Along this line, sorbitol hydrogenolysis producing ethylene (EG), propylene glycols (PG) and glycerol (GL) is a promising green process to produce important commodity chemicals from biomass instead of petroleum [13,14]. Catalytic hydrogenolysis of sorbitol usually occurs over activated carbon supported Ru catalysts and in slurry or trickled-bed reactors. However, the slurry reactor operation has a big problem to separate the catalyst, while in trickled-bed reactor the selectivity to desired products may be limited owing to the strong resistance to diffusion in the micropores of the catalyst pellets. In our recent work [15], CNFs supported Ru catalyst has been shown to have attracting behaviors in sorbitol hydrogenolysis when compared with activated carbon supported Ru catalyst, but the hydrogenolysis reaction was carried out in a stirred tank reactor with Ru catalyst supported on aggregated CNFs in submicron sizes. Hence, in this work an attempt is made to support Ru on a structured carbon nanofiber/graphite felt (CNF/GF) monolith for sorbitol hydrogenolysis. Moreover, the monolithic catalysts over CNF/GF structure are used as stirrer blades, whose concept was proposed and successfully applied by Moulijn and co-workers [16]. With the catalytic stirring blades, problems of catalyst attrition and agglomeration during reaction and catalyst separation after reaction can be avoided and traditional stirred reactors can be used for convenience [17].

In this work, the structure, morphology and porosity of the composites, the supported Ru catalysts, and their catalytic performance in sorbitol hydrogenolysis have been investigated. The primary results are reported in this paper. To the best of the authors' knowledge, this structured Ru catalyst for sorbitol hydrogenolysis has never been reported hitherto.

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2. Experimental

2.1. CNF/GF composite and Ru catalyst preparation

A CNF/GF composite was synthesized by an approach similar to the one used by Ledoux and Pham-Huu [7]. The graphite felt (GF, Shanghai Qijie Carbon Material Co., Ltd.) was first cut into predefined shapes and sizes and calcined in an oven at 500 °C for 6 h, over which 3 wt.% Ni was deposited by incipient wetness impregnation of an ethanol solution of nickel nitrate. CNFs were then grown on the GF using C_2H_6 as carbon source. Two CNF/GF composites were prepared, one with a CNF/GF ratio of around 0.7, denoting as CNF/GF1, and the other with a CNF/GF ratio of around 1, denoting as CNF/GF2.

All the composite samples were purified with 4 mol/L hydrochloric acid for several times and washed with deionized water until the filtrate was neutral to remove the remaining Ni, which were designated as CNF/GF-HCl. Subsequently the composite CNF/GF2 was treated with 30% solution of hydrogen peroxide or 4 mol/L nitric acid at 60 °C for 2 h and designated as CNF/GF2-H_2O_2 and CNF/GF2-HNO_3, respectively. The as-synthesized composite CNF/GF2, designated as CNF/GF2-UNT, was also used for comparison. Ru (3.0 wt.%) was supported on CNF/GF composites by incipient wetness impregnation with $RuCl_3$ aqueous (obtained from Heraeus Corp.) as precursor. The structured catalysts were aged at room temperature for 18 h, dried in air at 120 °C for 12 h and reduced in a stream of H_2/Ar (90/270 mL/min) for 5 h before activity test.

2.2. CNF/GF and catalyst characterization

The textural properties of the composites and catalysts were measured by N_2 physisorption on an ASAP 2010C (Micromeretics, USA). Scanning electron microscopy (SEM, JOEL JSM3360LV, Japan) and transmission electron microscopy (TEM, JOEL JSM2010, Japan) were employed to characterize the morphologies and structures of the CNF/GF supports and the supported Ru catalysts. The anchorage stability of CNFs on graphite fiber was measured by ultrasound treatment in ethanol solution [9].

2.3. Catalyst tests

The Ru structured catalysts were tailored to rectangle shapes of 2 cm × 1 cm and then fixed onto the stirrer as stirring blades, as shown in Fig. 1. With the catalytic blades, sorbitol hydrogenolysis was carried out in a 500 mL autoclave (Parr 4575A, USA), to which 20 wt.% sorbitol aqueous solution (360 g) and 10.0 g CaO (as basic promoter) were charged. Hydrogenolysis was carried out at 220 °C and a hydrogen pressure of 8.0 MPa for 6 h with a stirring rate of 600 rpm. The samplings were analyzed by an HPLC (HP 1100, Agilent, USA) using a Platisil ODS C18 column and an RID detector at 25 °C. On the basis of the carbon atom balance, the selectivity to specific polyol was expressed as the ratio of the sorbitol converted into this product to the total sorbitol converted. The detailed procedures are referred to Ref. [15].

3. Results and discussion

3.1. Characterization of carbon nanofiber/graphite-felt composite

Typical SEM images of the as-synthesized composite are shown in Fig. 2, which illustrate the multiscale structure of the composite. As seen from the low magnification images (Fig. 2a and b), the GF consists of uniform graphite microfiber with a diameter of about 25 μm while the graphite microfiber in composite is covered with a CNF thin layer whose thickness depends on the grown CNF amount. In the composite, there are a large number of voids of a few tens of micrometers to a few hundred micrometers. The image of a single graphite microfiber (Fig. 2c) and the one with much higher magnification (Fig. 2d) present the morphology and structure of the CNF layer on the microfibers. Entangling CNFs with uniform diameter of about 60 nm are grown on the microfiber surface. The results are very similar to the work reported by Tribolet and Kiwi-Minsker [4] and Ledoux and Pham-Huu [7]. They have synthesized CNFs on sintered metal filter with a diameter of about 70 nm and on carbon felt with a CNF diameter of 30 nm. However, the morphology and structure of the CNF/GF are different from the CNFs grown on solid carbon foam [5] and Ni foam [6], which had CNF diameters ranging from a few tens of micrometers to a few hundred micrometers and did not completely covered the surfaces of the foams. When used as structured monolith catalyst, the CNF/GF composite with high porosity and hierarchical pores would allow faster mass transfer and produce narrower residence time distribution as compared with conventional activated carbon granule supported catalyst, which has micropores mostly smaller than 2 nm.

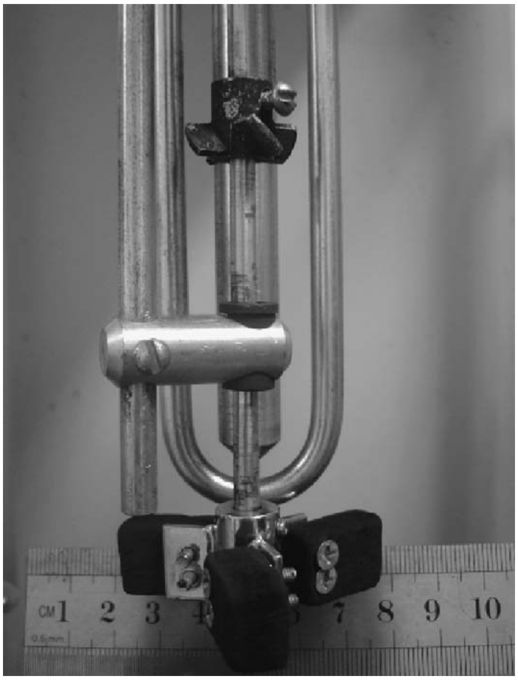

Fig. 1. Stirrer with structured Ru catalysts as blades (blade size: 20 mm × 10 mm × 8 mm).

The anchorage stability of the CNFs on the GF surface, which is intimately related to the abrasion resistance of catalytic material, was measured by ultrasound test. Fig. 3 shows the weight losses of CNF/GF composites during ultrasound treatment. The weight losses of all samples occurred during the first 60 min and almost no further loss was found thereafter. The biggest weight loss for the composites was <2%, indicating that the CNFs were hardly brushed off from the graphite fibers. This result is better than Jarrah et al's work [9], where the weight loss reached 10%. As a matter of fact, the reaction medium remained clear after reaction, also demonstrating the strong abrasion resistance and mechanical strength of the composite. This may be because of the penetration of anchoring CNFs into the graphite microfiber and the formation of numerous micro- and nanobridges between the nanofibers [18].

The N_2 physisorption isotherms of the CNF/GF composites with different CNF amounts and different post-surface modifications (not shown for conciseness) are characteristic of mesoporous structure with the hysteresis loops. They all had a broad mesopore peak around 20–100 nm corresponding to the interstices among

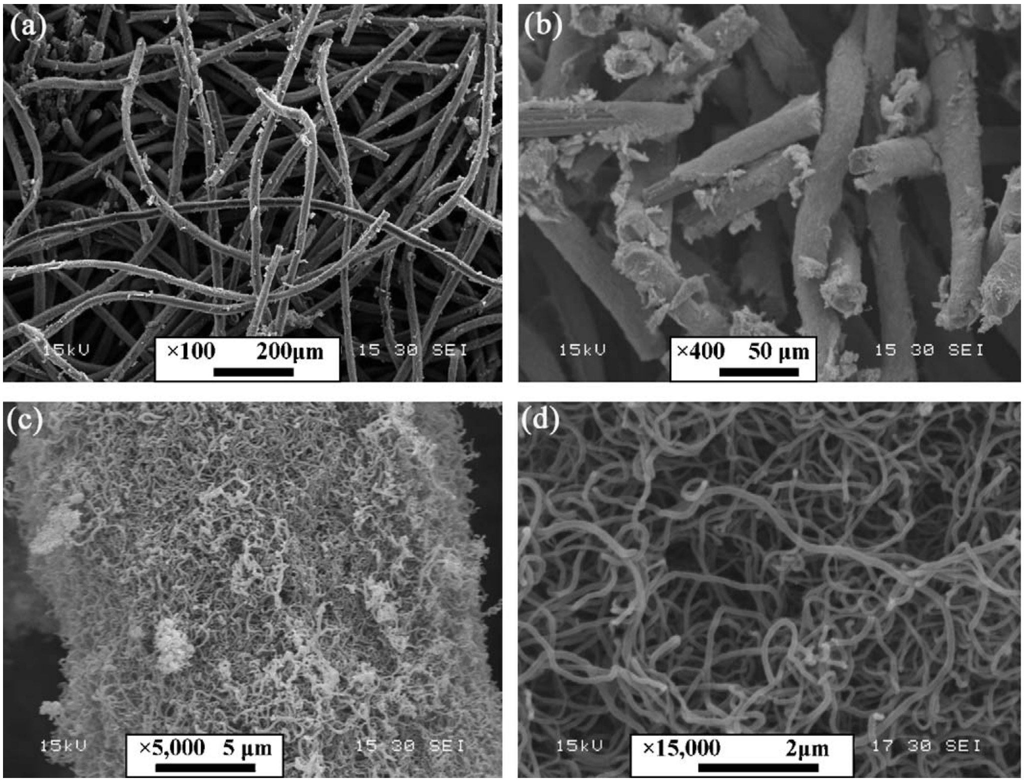

Fig. 2. SEM images of as-synthesized composite CNF/GF2-UNT.

the interlaced nanofibers and a rather sharp peak around 3–4 nm associated with the surface roughness of CNFs [20], resembling the typical curve for nanoscopic CNFs [19]. The mesoporous structure of CNF/GF composite is more favorable for sorbitol hydrogenolysis where compounds with large molecules are involved and hydrogen participates in a serial of intermediate reactions.

More detailed textural parameters of the CNF/GF composites and the corresponding structured Ru catalysts are listed in Table 1. All the composites possessed relatively high specific surface areas (45.87–72.52 m^2/g) and pore volumes (0.068–0.126 cm^3/g), among which only $<5\%$ of the pore volume was ascribed to micropore volume (<2 nm), further indicating the mesoporosity of the CNF/GF composites. Concerning the rather low surface area of GF (<1 m^2/g), the CNFs anchored on the GF actually have a specific surface area more than 100 m^2/g, large enough to be used as a catalyst support. The specific surface area of CNF/GF composite is comparable to the reported CNF/structured substrate, larger than those of CNF/metallic filter (27.2 m^2/g) [4] and CNF/cordierite monolith (about 30 m^2/g) [8], but less than those of CNFs/carbon foam (146 m^2/g) [5] and CNF/carbon felt (about 100 m^2/g) [7]. This has been caused by the difference in the active metal, carbon source and operating condition for CNF growth.

Moreover, the surface areas of the CNF/GF composites treated with HNO_3 and H_2O_2 were increased by 12% and 18%, respectively. This surface area increase could be ascribed to the additional oxygen containing groups and defects on the CNF surface, which were introduced by the oxidative treatment with HNO_3 and H_2O_2 and served as the adsorption sites during N_2 physisorption [21].

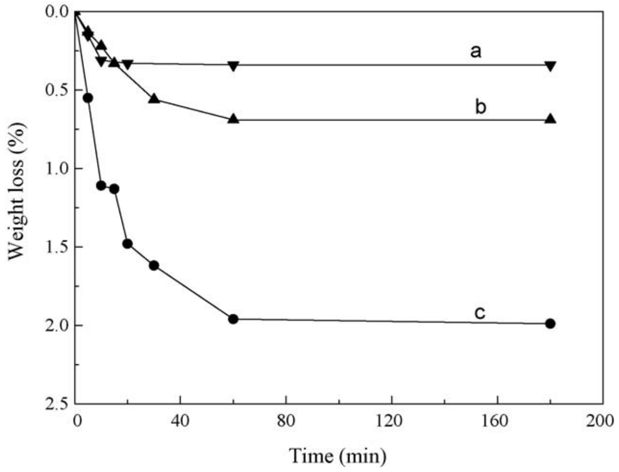

Fig. 3. Weight loss of composites versus time during ultrasound test: (a) CNF/GF2-UNT; (b) CNF/GF2-HCl; (c) CNF/GF1-HCl.

Table 1
Textural properties of the CNF/GF composites and the corresponding supported Ru catalysts.

Sample	Specific surface area (m^2/g)[a]	Pore volume (cm^3/g)[b]	Micro pore volume (cm^3/g)[c]
CNF/GF2-UNT	48	0.09	0
Ru/CNF/GF2-UNT	26	0.07	0.003
CNF/CNF/GF1-HCl	46	0.07	0.003
Ru/CNF/GF1-HCl	28	0.04	0.004
CNF/GF2-HCl	62	0.07	0.004
Ru/CNF/GF2-HCl	42	0.09	0.003
CNF/GF2-HNO_3	54	0.12	0.002
Ru/CNF/GF2-HNO_3	n.a.	n.a.	n.a.
CNF/GF2-H_2O_2	73	0.12	0.004
Ru/CNF/GF2-H_2O_2	52	0.13	0.002

All original felts of the composites were calcinated at 500 °C for 6 h in air.

[a] The specific surface area of the pores which are calculated with the Brunauer–Emmett–Teller equation.

[b] The volume of pores which are smaller than 300 nm and determined from the N_2 desorption isotherms using the Barrett–Joyner–Halenda equation.

[c] The volume of pores which are smaller than 2 nm. n.a. not available.

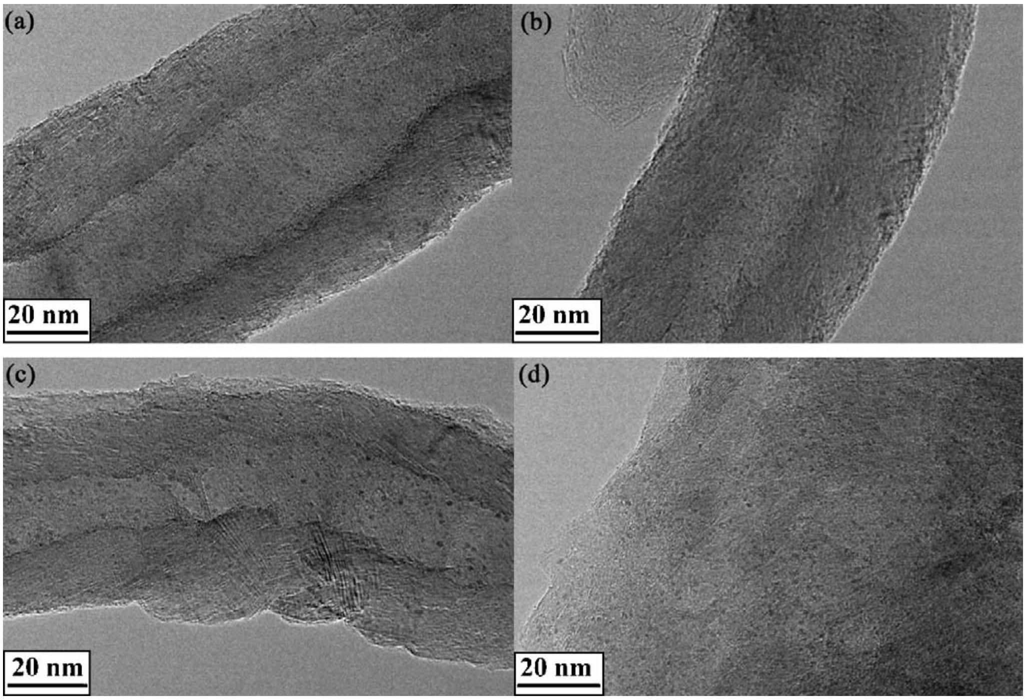

Fig. 4. HRTEM images of Ru catalysts: (a) Ru on as-synthesized CNF/GF2-UNT; (b) Ru on CNF/GF2-HCl; (c) Ru on CNF/GF2-H_2O_2 which was purified and modified with 30% H_2O_2; (d) Ru on powder CNF.

3.2. Characterization of structured Ru catalysts

Ru (3 wt.%) was supported on the CNF/GF composites by incipient impregnation and the catalysts were characterized by N_2 physisorption. All the isotherms and PSD curves of the structured catalysts (not given for conciseness) remained similar to those of the corresponding supports. The textural parameters of the structured catalysts are also listed in Table 1. The structured catalysts had apparently smaller surface areas than did the supports, owing to the occupation of the adsorptive sites on the CNF surfaces by highly dispersed Ru nanoparticles.

The CNFs on the GFs were scraped off and dispersed in an ethanol solution and measured by TEM, and the results are presented in Fig. 4. From the representative high resolution TEM images of the Ru catalysts supported on untreated (CNF/GF2-UNT, Fig. 4a), purified (CNF/GF2-HCl, Fig. 4b) and oxidatively modified (CNF/GF2-H_2O_2, Fig. 4c) composites, one can see that Ru particles were highly dispersed on the surface of the CNFs with a narrow size distribution centered around 1–2 nm. The result was in line with our previous work [15], in which Ru was also highly dispersed on the CNF surface in powder catalyst, the HRTEM image of which is also presented for comparison (Fig. 4d). The high dispersion of Ru nanoparticles on the CNFs could be associated to the strong metal–support interaction between Ru nanoparticles and the graphite edges of the CNFs, as has been previously observed in studying of CNF supported Pd catalyst [22].

In typical XRD patterns of the Ru catalysts (not given for conciseness), the crystallinity of the CNF/GF composites was easily seen, while no legible diffraction peaks associating with Ru were observed. The absence of Ru diffraction peaks may be caused by the high dispersion of Ru nanoparticles (<2 nm, which has broadened the diffraction peaks) and the relative low content of Ru in the catalysts. This phenomenon is in good line with the TEM results.

It is interesting to note that the oxidative modification of CNF/GF composite seems to have increased the average diameter of the supported Ru particles (about 2 nm), compared to the Ru particles, about 1 nm, on the as-synthesized and purified composites. The larger Ru particles on oxidized CNF were also examined by Plomp et al. [23]. Nevertheless, Tribolet and Kiwi-Minsker [22] found that for Pd/CNF, modification with H_2O_2 decreased the Pd particle sizes on the CNFs. This difference may have been caused by the different catalyst preparation methods.

3.3. Catalytic performance for sorbitol hydrogenolysis

The monolith catalysts were fixed on the stirrer and their performances in sorbitol hydrogenolysis were evaluated. The results are listed in Table 2. Also listed are the reaction results of blank experiment with CNF/GF2-UNT as stirrer and our previous result with powder Ru/CNF catalyst in slurry reactor for comparison.

The blank experiment with CNF/GF showed that the composite itself had no activity, though some of the Ni for CNF growth, which is usually active for hydrogenation, remained after purification in the composite. This is because the Ni particles were embedded in the end of CNF according to the CNF growth mechanism [24] and could not be accessed by the reactant molecules. Another surprise was that Ru on the as-synthesized CNF/GF (Ru/CNF/GF2-UNT) showed the best conversion though with the lowest selectivity.

As shown in Table 2, the sorbitol conversions and product selectivities of the monolith catalysts depended greatly on the

Table 2
Sorbitol conversion and product selectivity on different catalysts.

Catalyst	Sorbitol conversion (%)	Product selectivity (%)			
		PG	EG	GL	Sum
CNF/GF-UNT (blank)	0	0	0	0	0
Ru/CNF/GF-UNT	52.1	28.9	21.0	16.2	66.1
Ru/CNF/GF1-HCl	22.0	26.8	25.6	15.0	67.5
Ru/CNF/GF2-HCl	35.8	40.8	23.6	14.7	79.1
Ru/CNF/GF-HNO_3	34.9	38.2	22.8	13.9	74.9
Ru/CNF/GF-H_2O_2	41.3	39.9	24.9	14.3	79.1
Ru/CNF-HCl	68.3	29.2	19.4	8.4	57.0

supports. Compared with the powder Ru/CNF catalyst, all the structured Ru catalysts presented significant higher selectivities to EG, PG and GL. The best total selectivity of 79.1% was superior to that of the comparative powder catalyst of 57.0%. This result, especially the selectivity to ethylene glycol (13.4%), is quite promising and is better than the only result with structured catalyst reported by Cao et al. [25]. They supported nickel and rhenium on cordierite monolith and obtained a total selectivity of 72.5% at a sorbitol conversion of 44.8%.

A comparison of the catalytic activity between Ru/CNF/GF1-HCl and Ru/CNF/GF2-HCl showed that the composite with more CNFs amount presented both higher conversion and higher selectivity. The composite with more CNF amount was supposed to have much higher surface area (referring to Table 1) and more Ru particles (owing to the fixed 3.0 wt.% Ru loading) when the same size of structured catalyst was used as stirring blades, and hence the better activity. But too much CNF would lower the packing porosity of the composite and thus increase the mass transfer resistance. Therefore appropriate CNF amount should be grown on the GF as support of structured Ru catalyst for sorbitol hydrogenolysis.

A further analysis of the activities of Ru catalysts on oxidatively modified composites reveals that the surface chemistry of the composites has apparently affected both the conversion and the selectivity. Though the Ru particles on the H_2O_2 modified composite (Ru/CNF/GF2-H_2O_2) were larger than those on other composites, e.g. Ru/CNF/GF2-HCl, they presented higher conversions and yields. Similar results were observed by Tribolet and Kiwi-Minsker [22], who found that in selective hydrogenation of cinnamaldehyde, the larger particles on the CNFs with oxidized surface were more selective to cinnamyl alcohol. On the other hand, Ru/CNF/GF2-HNO_3 presented similar conversion but lower selectivity, as compared to Ru/CNF/GF2-HCl. Although the H_2O_2 and HNO_3 treatments are both usual oxidative modification methods to introduce oxygen containing groups onto carbon surfaces, the activity of Ru catalysts over the CNF/GFs treated with H_2O_2 and HNO_3 varied obviously. Since the Ru dispersions on these two supports obtained from CO chemisorption were very close, this phenomenon could be attributed to the interaction between the CNF/GF support and the Ru particles which disturbs the electronic state of the Ru particles and therefore affects the catalyst activity. Oxidative modification introduced a plenty of oxygen containing groups such as carboxyl and carbonyl onto the CNF surface, and the types and distributions of surface groups differed when different oxidants are used [21]. Thus the surface with different chemistry would not only affect the interaction between Ru particles and CNF but also provide different affinity to the reactants, intermediates and products. Hence the activities and selectivities differed. Janowska et al. [26] have recently reported that, for a Pd catalyst supported on aligned carbon nanotube (CNT) for cinnamaldehyde hydrogenation, the surface chemistry of CNT affected significantly the activity and selectivity. The effect of oxidative modification of the Ru catalyst support on its performance in sorbitol hydrogenolysis, as seen from the results listed in Table 2, is very complicated and requires further investigations.

Because of the high porosity of the structured support, the creeping flow dominating in the monolith catalyst will improve greatly the mass transfer. But it is usually difficult to eliminate the influence of mass transfer for this kind of monolith catalyst that is used as blades. Installing the monolith catalyst blades on the right position of the stirrer to increase the pressure drop across the monolith catalyst, or just decreasing the thickness of the monolith catalyst, will allow more fluid to creep through it and therefore increase the rate of mass transfer. This could be realized with a new stirrer design that can hold thin monolith catalyst and with fluid dynamic experiments and simulation to optimize the configuration of the stirrer and the reactor.

4. Conclusions

CNF/GF composites have been synthesized as a structured catalyst support with multiscale structure, large surface area (45–75 m^2/g), mesoporosity and good mechanical strength. Ru nanoparticles were homogeneously dispersed on the support with a narrow size distribution centered around 1–2 nm. This structured Ru catalyst presented a slightly lower activity but significantly higher selectivity to the desired products. However, the ratio of CNF to GF and the method for surface modification were able to change its catalytic activity remarkably.

Acknowledgements

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