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Furfural production from xylose and bamboo powder over chabazite-type zeolite prepared by interzeolite conversion method

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ABSTRACT

Furfural was directly obtained from bamboo powder in water–toluene biphasic solvents at 443 K over a chabazite-type (CHA) zeolite prepared with an interzeolite conversion method from a faujasite-type zeolite. The CHA zeolite was effective for the hydrolysis of hemicellulose fraction in ball-milled bamboo powder to xylose and selective dehydration of xylose to furfural.

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1. Introduction

For the establishment of sustainable chemicals production from biomass, the development of environmentally benign system using heterogeneous catalysts is indispensable. Furfural is commercially produced from bagasse and corncob with sulfuric acid treatment, in which hemicellulose fraction is hydrolyzed into five- and six-carbon sugars, followed by the dehydration of five-carbon sugars (xylose) to furfural by mineral acid. The development of furfural production system using solid acid catalysts in aqueous media is preferable because of the elimination of neutralization process and easy separation of products. There are several reports for the production of furfural from hemicellulose into furfural using solid acid catalysts in high-temperature liquid water and real biomass [1–8]. SAPO-44, which is a chabazite-type (CHA) silicoaluminophosphate, is reported to be the most effective and stable for furfural production from hemicellulose (82% furfural yield) and bagasse in water–toluene biphasic solvent [4]; however, it requires a large quantity of a limited phosphorus resource. Thus, the development of hot-water-stable aluminosilicate zeolite is desirable. Chabazite-type aluminosilicate zeolite has been synthesized using an expensive N, N, N-trimethyl-1-adamantammonium cation as a structure-directing agent. Recently, Sano et al. succeeded to prepare acid-tolerated CHA-type zeolites by an interzeolite conversion method, in which a faujasite-type (FAU) zeolite as silica and alumina source, inexpensive benzyltrimethyl-ammonium hydroxide (BTMAOH), and small amounts of the seed crystal were hydrothermally treated in aqueous sodium hydroxide solution. His group demonstrated that a CHA-type zeolite membrane fabricated on a porous α-alumina tube was highly stable towards several common mineral acids such as sulfuric acid [9,10].

For the sustainable production of valuable chemicals, the utilization of non-edible and wasted biomass is indispensable. The bamboo, which contains 20–25% of hemicellulose, is highly propagative biomass and harmful bamboo invasion damages vegetation and houses. Several parts of bamboo forest are logged; however, the treatment of large amounts of bamboo chips is an acute problem in Asia. From the view point of biomass utilization, Sun and co-workers reported that partial decomposition of bamboo chips to xylo-oligosaccharides is possible in liquid water at 423 K [11]; however, there is no report for chemicals production from bamboo with solid acid catalysts in water soenlrg far.

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In this paper, we report the furfural production from bamboo powder over a CHA-type aluminosilicate zeolite prepared by an interzeolite conversion method. The CHA-type zeolite was effective for the hydrolysis of hemicellulose fraction of bamboo powder to xylose and selective dehydration of xylose to furfural in water.

2. Experimental

2.1. Raw material

A bamboo sample harvested in Akita, Japan was used in this study. The sugar composition of the bamboo sample was determined by the NREL method. The amount for each fraction of the bamboo sample was determined three times, individually. The bamboo powder was obtained by a cutter-mill (denoted as CM) with a size of less than 500 μm. A CM sample was pulverized with a ball-mill (denoted as BM) for 48 h [12,13].

2.2. Zeolite catalysts

In this study, three types of zeolites were examined. A CHA-type zeolite catalyst was prepared by an interzeolite conversion method [9, 10]. A FAU-type zeolite (HSZ-360HUA from TOSOH, Si/Al = 7, FAU-7) was dealuminated to Si/Al = 20 (FAU-20) by the treatment with 0.5 mol dm^{-3} of sulfuric acid solution at room temperature for 18 h. The mixture of FAU-20 (25 g), BTMAOH (35 g of 40 wt% aqueous solution), aqueous sodium hydroxide (2.5 g), ion-exchanged water (40 ml), and small amounts of seed of a CHA-type zeolite (0.5 g) were prepared to a gel sample. The hydrothermal treatment of the gel sample was carried out at 397 K for 7 days by using Teflon-lined autoclave, followed by drying at 373 K and calcination at 823 K to obtain a CHA-type zeolite (CHA-20). A mordenite-type zeolite with Si/Al = 10 (MOR-10, specific surface area 400 $m^2 g^{-1}$ and total pore volume 0.25 $cm^3 g^{-1}$) was obtained from Catalysis Society of Japan. The CHA- and MOR-type zeolites were protonated with aqueous ammonium nitrate solution followed by calcination at 823 K before the reaction.

The amount of acid sites of CHA-20 and MOR-10 was determined by an ammonia TPD method.

2.3. Catalytic reaction

Hydrolysis-dehydration reactions were carried out in a batch reaction mode using an autoclave (100 cm^3). Reaction procedure was as follows; after the addition of reactant (1.0 g of bamboo powder or 0.1 g of xylose), 0.2 g of catalyst, 10 cm^3 of water, and 40 cm^3 of toluene into the reactor, the reactor was purged with nitrogen gas. Then, the reactor was heated to reaction temperature and kept for required reaction time. After the reaction, the products in the aqueous and organic phases were recovered from the reactor, respectively. Toluene was added for the extraction of furfural from the aqueous phase for the suppression of decomposition of furfural [14,15]. Chemicals in the aqueous and organic phases were analyzed by high performance liquid chromatography (HPLC, Shimadzu, column; Rezex RPM-Monosacharide Pd^{2+} (8%)) and gas chromatography (column; DB-BAX) with a flame ionization detector (GC-FID) (Shimadzu, GC-14B), respectively.

2.4. Evaluation of catalytic activities

The xylose conversion and furfural yield were calculated based on five-carbon sugars content in the reactant.

Xylose conversion (%)

$$= (1-(\text{mol of unreacted xylose in aqueous phase/mol of reactant xylose}))\times 100.$$

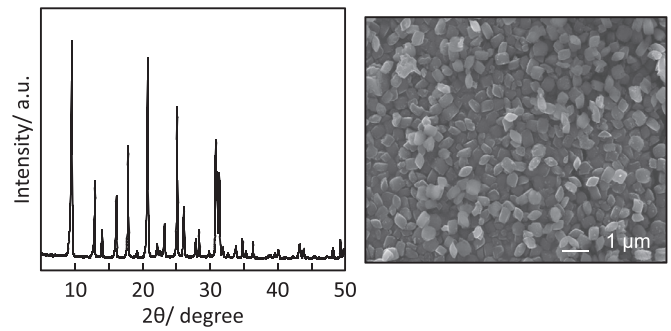

Fig. 1. XRD pattern and SEM image of CHA-20.

Furfural yield from bamboo powder (%)

$$=(\text{mol of furfural in organic and aqueous phases/mol of five-carbon sugar in bamboo})\times 100.$$

Furfural yield from xylose (%)

$$= (\text{mol of furfural in organic and aqueous phases/mol of xylose})\times 100.$$

3. Results and discussion

3.1. Bamboo and zeolite catalyst

The amount for each fraction of the bamboo sample was determined three times, individually. The average composition values were 71.0% of holocellulose, 43.8% of α-cellulose, 29.7% of lignin, 2.3% of ashes, and 1.1% of wax (extractives). The sugar composition of the bamboo sample was 44.6% of glucose, 0.6% of mannose, 21.9% of xylose, and 1.5% of arabinose. The bamboo sample contained 23.4% of five-carbon sugars.

The specific surface area and total pore volume values for the FAU samples were 518 $m^2 g^{-1}$, 0.50 $cm^3 g^{-1}$ (FAU-7) and 621 $m^2 g^{-1}$, 0.56 $cm^3 g^{-1}$ (FAU-20), respectively. The synthesized CHA-type zeolite showed highly crystalline with a particle size of 0.5–1.5 μm (Fig. 1). The specific surface area, external surface area, and total pore volume of the CHA-20 sample, was 548 $m^2 g^{-1}$, 3.5 $m^2 g^{-1}$ and 0.33 $cm^3 g^{-1}$, respectively.

3.2. Effect of zeolite catalyst

Furfural could be directly obtained from bamboo powder (CM) in 2 h (5%) and 10 h (35%) at 443 K in water–toluene biphasic solvent (Fig. 2). Sánchez et al. reported that furfural was obtained from corncob in water at 453 K [16]. The formation of furfural without zeolite in our results shows that the hydrolysis of hemicellulose in bamboo powder to xylose proceeded in high-temperature liquid water at 443 K. Fig. 2 also shows that zeolite catalysts were effective for furfural production from bamboo powder and their catalytic behavior depended on types of zeolite. CHA-20 exhibited the highest activity for the conversion from CM to furfural for 2 h (21%) and 10 h (55%). FAU-20, which had the similar Si/Al ratio to that of CHA-20, was not effective for furfural production from bamboo powder. FAU-7, which had larger amounts of acid sites than those of FAU-20, showed similar furfural yields as shown by FAU-20. MOR-10 showed a low activity (10%) at 2 h; however, it increased to 48% in 10 h.

Figs. 3 and 4 show the conversion and furfural yield from xylose, which is a five-carbon sugar formed by the hydrolysis of hemicellulose, over zeolites at 443 K in water–toluene biphasic solvents. The conversion of xylose and furfural yield after 6 h in water at 443 K without zeolite catalysts was 92% and 57%, respectively.

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