

## Effect of extraction on furfural production by solid acid-catalyzed xylose dehydration in water

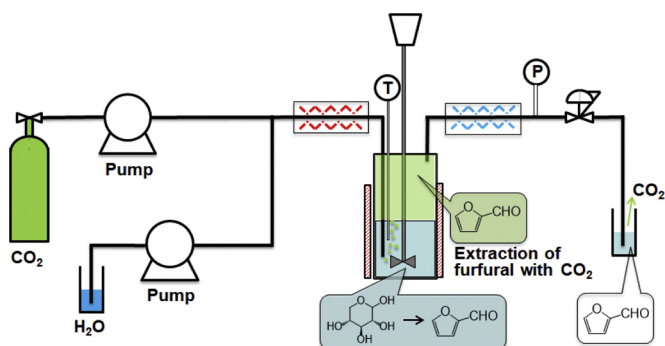


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### GRAPHICAL ABSTRACT



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### ABSTRACT

Production of furfural by means of xylose degradation was investigated at 423 K with zeolites or an ion-exchange resin as a solid acid catalyst in water, water/toluene, or water/CO<sub>2</sub>. All the catalysts gave higher xylose conversions than did hydrothermal conditions without a catalyst. When the ion-exchange resin (Amberlyst 70) was used as the catalyst, furfural extraction with toluene effectively increased the yield relative to that of the reaction in water alone. The furfural yield obtained with the water/CO<sub>2</sub> batch system was lower than that with the biphasic water/toluene system because the solubility of furfural in supercritical CO<sub>2</sub> was lower than that in toluene. The furfural yield was increased by using a semibatch reactor system with continuous CO<sub>2</sub> flow. The maximum furfural yield (52.3%) was achieved with Amberlyst 70 and a CO<sub>2</sub> flow rate of 3.77 g min<sup>-1</sup> at 423 K.

### 1. Introduction

The conversion of inedible biomass to useful chemicals has attracted considerable attention as a method to contribute to the goal of establishing a sustainable society [1–3]. Cellulose, a polysaccharide that is composed of glucose monomers and is the main component of inedible biomass, can be used for bioethanol production via saccharification and

fermentation and can also be converted into the platform chemical hydroxymethylfurfural by means of hydrolysis and dehydration reactions [4–6]. However, biomass must be pretreated before the conversion of cellulose. During pretreatment, the hemicellulose that is also present in biomass is easily hydrolyzed to C5 sugars such as xylose [7,8], so the conversion of xylose to value-added chemicals is important for making efficient use of the products of biomass degradation. Of the

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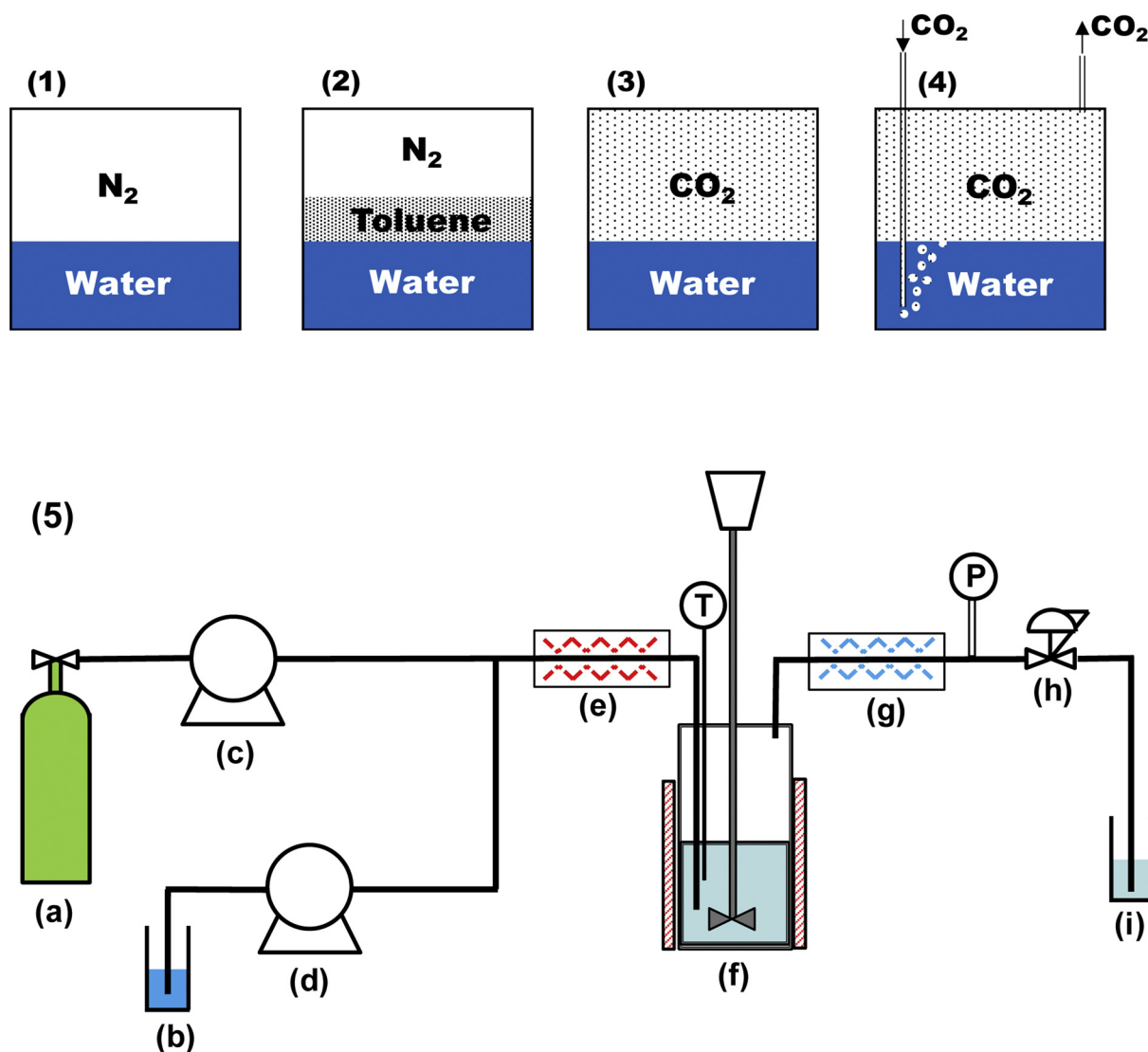


Fig. 1. Reaction media used for conversion of xylose to furfural: (1) water, (2) water/toluene/ $N_2$ , (3) water/ $CO_2$  in a batch system, and (4) water/ $CO_2$  in a semibatch system. (5) Schematic illustration of the semibatch system: (a)  $CO_2$  cylinder, (b) water, (c, d) syringe pumps, (e) preheater, (f) reactor, (g) cooler, (h) back pressure valve, and (i) ice-cooled water.

chemicals obtained from xylose, furfural is the most promising because of its wide range of applications [9,10] and because it can be converted to other useful products such as furfural alcohol and tetrahydrofuran [11]. The dehydration of xylose to furfural was summarized in the reference [12].

Currently, furfural is commercially produced by dehydration of xylose with aqueous sulfuric acid [9], but catalytic degradation of the furfural product by the aqueous acid decreases the yield of this process. One solution to this problem is to use a water–organic solvent biphasic system for xylose dehydration; the furfural produced in the water phase is immediately extracted into the organic solvent, where acid is not present [9–11]. In addition, because mineral acids are highly toxic and corrosive, heterogeneous acid catalysts have been explored in attempts to improve the environmental friendliness of the xylose dehydration process. Many types of solid acids, including zeolites [13–15] and ion-exchange resins [16], have been used. Not only solid acid catalysts but also acidic ionic liquid with organic solvents was used for the xylose dehydration to furfural [17–20]. In addition, the use of supercritical  $CO_2$  as an extractant instead of an organic solvent has also been investigated because supercritical  $CO_2$  can dissolve organic compounds and is nonflammable and nontoxic [21–24]. In the purification process, the separation of furfural from  $CO_2$  is much simpler than that from

organic solvents. Also, the addition of  $CO_2$  in water enhance the acidity of water by forming carbonic acid, resulting in accelerating the dehydration reactions [25–28]. Morais et al., reported that furfural yield from xylose was 70% at 453 K in water/tetrahydrofuran with 5 MPa of  $CO_2$  [27,28]. Supercritical  $CO_2$  extraction method has been used for sulfuric acid-catalyzed xylose dehydration [21,23], hydrothermal conversion of xylose to furfural at 503 K without acid catalysts (furfural yield 68%) [24], and xylose dehydration catalyzed by sulfated titania and sulfated zirconia at 453 K [22]. Sangarunlert et al. reported that more than 80% yield of furfural was obtained by xylose dehydration using 7 wt% sulfuric acid at 453 K with supercritical  $CO_2$  extraction [23]; however, the strong acid was required for the reaction. Kim et al. reported that solid acid catalysts such as sulfated titania and sulfated zirconia provided about 60% yield of furfural from xylose at 453 K with supercritical  $CO_2$  extraction [22]. However,  $CO_2$  extraction has not been used in combination with zeolites or ion-exchange resins. Therefore, in this study, we explored xylose conversion to furfural with catalysis by an ion-exchange resin and two types of zeolites in batch and semibatch processes with  $CO_2$  extraction to enhance the furfural yield. Our final goal is the cascade utilization of lignocellulosic biomass; thus, cellulose and lignin should be kept as solid after the conversion of hemicellulose to use them efficiently. The reaction temperature (423 K)

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