High Selective Isomerization of Glucose to Fructose Catalyzed by Amidoximed Polyacrylonitrile

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INTRODUCTION

Over the past few years, there has been considerable interest and investment in research into the catalytic conversion of biomass feedstocks into renewable fuels and chemicals. Glucose, a platform compound for biomass, can be obtained from the hydrolysis of cellulose. The conversion of glucose to fructose expands the range of biomass utilization for the reasons that fructose is not only used widely as a sweetening agent in food and beverages but also an important starting material in those catalytic systems, which made the catalysts irreversible and influence quality of products. Fixed amine is another potential catalyst for isomerization of glucose to fructose. In a previous study, we synthesized an amphoteric polyacrylonitrile (PAO) with an amidoxime functional group to catalyze the isomerization of glucose and achieved 32% yield of fructose for 2 h at 100 °C. Delidovich et al. improved the selectivity of fructose greatly to 93% with 33 wt % yield of fructose after reacting 4 h at pH 8.5 and 80 °C. Herein we prepared an amphoteric polymer compound

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ABSTRACT: The isomerization of glucose to fructose provides an important way to expand the utilization of biomass. Herein, an amidoximated polyacrylonitrile (PAO) with an amidoxime functional group was prepared and used as an active heterogeneous catalyst for the isomerization of glucose to fructose. The PAO was characterized by SEM, XPS, and FTIR. The yield of fructose reached 48.9% with a selectivity of 98.6% for a 5 h reaction in aqueous solution at an initial pH of 6.5 and 85 °C. The pH caused a great influence on the conversion of glucose and selectivity of fructose while a little effect on the yield of fructose in the range of pH 5–10. The activation energy of isomerization reaction was evaluated as 79.7 kJ mol⁻¹. The catalysis mechanism was proposed, and the synergistic effect of oxime and amino groups played an important role in the isomerization of glucose. PAO maintained good catalytic activity after four cycles.

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containing an amidoxime group as a heterogeneous catalyst for the isomerization of glucose to fructose. This system showed high catalytic activity for the isomerization reaction under mild conditions. The amidoxime (−C(═NOH)NH₂) structural unit, where a carbon atom connects both an oxime group and an amino group, shows amphoteric nature in aqueous solution, which is conducive to the proton transfer in aqueous solution, and the oxime group also has nucleophilicity.

**RESULTS AND DISCUSSION**

Characterization of the Catalyst. The amidoximated polyacrylonitrile was successfully prepared. The morphology, chemical functional groups, and element content were characterized by scanning electron microscopy (SEM), Fourier transform infrared spectra (FT-IR), and X-ray photoelectron spectra (XPS). The SEM images presented the morphology of PAN and PAO solid particles, as shown in Figure 1. From Figure 1, it can be observed that PAN particles were dispersed (Figure 1a), and the surface of PAN was smooth and dense (Figure 1b). After ammoximation, the surface of PAO become rough and wrinkled (Figure 1d). The diameter of round particles PAO remained at about 20–50 µm.

The FT-IR spectra of PAN and PAO are illustrated in Figure 2. The peak at 2243 cm⁻¹ was vibration absorption of −C═N on PAN, and it disappeared after the modification reaction. The peak at 1730 cm⁻¹ corresponds to carbonyl stretch, which maybe from remaining DMF, a solvent commonly used in the production of PAN. The appearances of new peaks at 3305 cm⁻¹ (−NH₂), 3172 cm⁻¹ (−OH), 1639 cm⁻¹ (−C═N−), 1386 cm⁻¹ (C−N), and 916 cm⁻¹ (N−O) were consistent with the transformation of nitrile groups to amidoxime groups. The new peaks at 700–500 cm⁻¹ of recycled PAO were due to the ring vibration of monosaccharide, which came from the absorption of carbohydrates on the PAO surface.

The elements C atomic %, N atomic %, and O atomic % on the surface of PAN, PAO, and recycled PAO were analyzed by XPS, as listed in Table S1 and shown in Figure S2. From Table S1 and Figure S2a, it can be seen the O atomic % of PAO obviously increased from 3.14 to 18.8% owing to the change of the cyano group into the amidoxime group. However, C atomic % and O atomic % of PAO has slightly increased after recycling because of the absorption of carbohydrates in the solution. In Figure S2b–d, the new peaks in PAN at 533.9, 401.9, and 283.7 eV were related to −C═N−O. Therefore, we believed the amidoximed polyacrylonitrile was successfully prepared. From the above analysis, it can be estimated to contain 0.0116 mol of amidoxime unit per gram of the PAO catalyst.

Comparison of Catalytic Activity of Various Functional Groups. Some small molecules, such as organic amines and oxime compounds, were employed to compare the effect of the chemical functional group on the isomerization of glucose to fructose, and the results are listed in Table 1. From Table 1, we can observe that organic amines including ethylenediamine, diethylamine, triethylamine, and tetramethylammonium hydroxide (TMAOH) displayed weak catalytic activity; the yield and selectivity of fructose were less than 14 % and 70%, respectively. Oximes including acetoxime, diacetyl-oxime, and diacetylmonoxime showed lower selectivity of fructose (less than 55%). In our previous study, we found that although the catalytic activity of the single functional group was poor, the synergistic effect of phenol groups and tertiary amine group showed good catalytic efficiency for the isomerization of glucose to fructose. Along the thinking, we further synthesized a small-molecular compound containing...
amidoxime, that is, amidoximed acetonitrile (AO) and found that the synergism of the oxime group and amino group was indeed beneficial to the isomerization of glucose. A yield of 38.4 and 88.3% selectivity of fructose was achieved by using AO as the catalyst for reaction 2 h at 90 °C (entry 10, Table 1). This shows that not only the functional groups but also the synergistic effect of different functional groups on the isomerization of glucose will be different.

**Catalytic Activity.** The effect of both temperature and reaction time on the isomerization reaction catalyzed by PAO was investigated in the temperature range of 80–95 °C, and the results are shown in Figure 3. PAN has showed little catalyst activity for isomerization of glucose (Table S2). Temperature caused a significant influence on the isomerization of glucose. Increasing the temperature will speed up the reaction rate. Furthermore, the higher the temperature was, the earlier the maximum yield of fructose appeared. In this study, the maximum yield of fructose was 48.9% for a 5 h reaction at an initial pH of 6.5 and 85 °C, and the selectivity of fructose still remain at a relatively high level, up to 98.6%. Since the reaction active energy of the isomerization of glucose into fructose is relatively large under neutral aqueous solution, most of the reactions were carried out at above 100 °C.7–14 Glucose and fructose were all unstable and could be degraded in aqueous solution at high temperature. Therefore, it was relatively difficult to achieve a high selectivity of fructose at high temperature. In this work, in order to obtain the high selectivity, we employed PAO to catalyze the isomerization of glucose to fructose under relatively mild conditions.

It has been reported in many literatures that isomerization reaction of glucose to fructose is a first order with respect to the concentration of glucose.20,23,24 In this work, based the experimental data, we found that the plots of ln(Gf) versus t were all straight lines for initial reaction 1.5 h (Figure S3), which indicated that the reaction was first order with respect to the concentration of glucose in this catalytic system. Consequently, the kinetic constants k of the first-order reaction was calculated and listed in Table S3 for different temperatures. The activation energy Ea of conversion of glucose was evaluated as 79.7 kJ·mol−1 by the Arrhenius plots of ln k versus 1/T, as shown in Figure 4. The Ea of PAO is less than those of Sn-β (93 kJ·mol−1),10 AlCl3 (110 kJ·mol−1),23 and NaOH (121 kJ·mol−1).20 Thus, PAO could be considered as an effective catalyst for isomerization of glucose to fructose.

**Influence of Initial Solution pH.** The pH of solution is one of the important influencing factors in the isomerization of glucose to fructose.27 The effect of initial pH0 on the isomerization reaction was investigated in the range of pH 3.0–11.0, as shown in Figure 5. From Figure 5, we found that the initial pH0 could cause remarkable influence on the isomerization reaction. The conversion of glucose increased with an increasing value of initial pH. The yield of fructose increased remarkably when pH of solution increased from 3 to 5, and the yield of fructose changed little in the range of pH 5–10. The yield reduced when pH > 10, which may be due to the increase of byproducts caused by the degradation of monosaccharides in alkaline environment.28 The selectivity of fructose was very high under acidic conditions, and the selectivity of fructose almost reached 100% at pH 3 and decreased gradually with the increase of pH and decreased to 83% at pH 9.

**Influence of Neutral Salt.** Neutral salt was employed to understand the behavior of the isomerization reaction, and the results are shown in Figure 6. The addition of the neutral salt decreased both the conversion of glucose and yield of fructose. After adding the neutral salt, it was observed that the pH of the solution increased slightly (Figure S4), which implied that the concentration of OH− in the solution increased. The ζ
potential on the surface of PAO also increased in diluted NaCl solution (Table S4, entry 3 and 4). The inorganic salt can promote protonation of amine by electrostatic interaction of the anion with the protonated amino group. In the amine-based catalytic systems reported, the OH\(^-\) generated in situ by protonation of amine was usually considered to be the activity species in the process of isomerization of glucose into fructose. Therefore, the catalytic activity of the amine-based catalyst could be improved by adding neutral salts. In this work, it was found that although the addition of salt increased the pH and produced more OH\(^-\), the catalytic activity of PAO reduced. This indicated that the amidoxime group rather than OH\(^-\) played an essential role in the process of isomerization of glucose to fructose in this catalytic system.

We describe the possible ways in which the addition of neutral salt affects the catalytic activity of PAO, as illustrated in Scheme S1. Though the addition of salt promoted the protonation of the amino group, oxime negative ion species and amine positive ion species were attracted by the cation and anion of salts in solution, respectively. In this way, the nucleophilicity of the oxime group in amidoxime became weaker, which slowed down the rate of deprotonation of glucose to an enediol intermediate that is generally considered as the rate control step of the isomerization of glucose to fructose.

**Catalyst Reuse and Activity Recovery.** After the catalyst PAO was filtered from reaction solution and washed with water, the reusability of PAO was evaluated in terms by performing three consecutive reaction cycles, and the result is shown in Figure 7a. In Figure 7a, the reacted PAO was recycled by filtration and cleaned with water. Unexpectedly, there was an obvious decrease of conversion of glucose and yield of fructose from run 1 to run 2. However, from run 2 to run 3, both conversion of glucose and yield of fructose remained almost constant. The peaks at 700–500 cm\(^{-1}\) in Figure 2 were closed to the ring vibration of sugar, and the C atomic % and O atomic % in Table S1 increased slightly, which may be due to a small amount of sugar physically adsorbed onto the PAO surface because of the high adsorption of the PAO surface. The types and numbers of other peaks in Figures 2 and S2 of recycled PAO were consistent to of the fresh PAO.
This suggested that the PAO itself has not undergone change in the chemical structure, indicating that it was a stable catalyst.

Interestingly, an alkaline washing process can make the catalyst PAO recover its catalytic activity well. The recycle PAO was filtrated from reaction solution and stirred in diluted NaOH solution (pH 10–10.5) for 10 min at 90 °C. The alkaline washing PAO recovered its catalytic activity to a great extent. The experimental results are shown in Figure 7b. After a run of four times, the yield of fructose still remained 41.5% and the selectivity was 97.6%. In order to understand this behavior, the PAO after alkaline washing was characterized by FT-IR, and the FTIR spectra are illustrated in Figure S5. There was no significant change in the FTIR spectra of the three samples, which indicated that no significant chemical changes occurred in PAO after alkaline washing. However, the N−O peak of Recycling PAO is 921 cm$^{-1}$, which is slightly larger than 916 cm$^{-1}$ of the other two samples. This may be due to the fact that negative ions of oxime-group residues in the amidoxime group obtained the proton after reaction and formed the oxime-group neutral species, and its peak position moved to the high wave number. We performed the ζ potential measurement for the PAO aqueous solution by using 0.15% xanthan gum as the suspending agent, and the results are listed in Table S5 in Supporting Information. It was observed that the ζ potential of recycled PAO by water washing was +1.02 mV (Table S4, entry 5), the positive charge of the solution came from the protonated amino group in PAO, and the ζ potential of recycled PAO after alkaline washing was −3.28 mV (Table S4, entry 6). The negative charge of the solution came from the oxime anion which has better catalytic activity than the neutral oxime form.

**Possible Catalytic Mechanism.** It is well known that there are two types of reaction mechanisms for the catalytic isomerization of glucose to fructose.32 One is the intramolecular hydrogen shift mechanism promoted by the Lewis acid catalysts from the C-2 to C-1 position.25,33 The other is the Lobry de Bruyn–Alberda van Ekenstein (LdB–AvE) mechanism,34 in which the base catalyst extract protons at C-2 of glucose to form enediol intermediates, followed by the keto−enol tautomerization reaction to produce fructose.23,35

Based on the above experimental phenomena, we suggested the possible catalytic mechanism of PAO prefer to the LdB–AvE mechanism which involved following processes: ring-opening of glucose, abstraction of protons and formation of enediols, supply of protons and rearrangement and ring-closure of fructose, as shown in Scheme 1. It was reported that only 0.002% chain glucose exists in aqueous solution at 30 °C.36 Cyclic glucose would undergo a ring opening step at the beginning of the isomerization of glucose to fructose.25,37 In this work, the ring-opening of cyclic glucose may be induced by groups, oxime anion and amino cation on PAO. In this catalytic system, the oxime anions on PAO easily capture the proton from glucose to form the enediol intermediate. Subsequently, the ene-diol intermediate would gain a proton from the protonated amine group and rearrange to generate the acyclic fructose. After the rotation of C1−C2 bond, the acyclic fructose was induced to form cyclic fructose by the proton transfer between the oxime anion and amino cation on PAO and glucose.

**CONCLUSIONS**

In this work, a polymer PAO with an amidoxime functional group was successfully prepared and characterized by SEM, FTIR, and XPS. The PAO was used as the heterogeneous catalyst and exhibited excellent catalytic activity for the isomerization of glucose to fructose under mild reaction conditions. The maximum 48.9% yield of fructose with 98.6% selectivity of fructose was achieved for the 5 h reaction at an initial of pH 6.5 and 85 °C. The activation energy $E_a$ for the isomerization of glucose was evaluated as 79.7 kJ·mol$^{-1}$. Although conversion of glucose and selectivity of fructose was seriously influenced by the initial pH of solution, the yield of fructose could remain relatively stable at pH 5–10 in this catalytic system. The catalyst can be reused through stirring in diluted NaOH solution for ion exchange with water and explained a good reusability at least for four consecutive reaction cycles.
**EXPERIMENTAL SECTION**

**Materials.** Monohydrate glucose, NH₂OH, and ethane diamine were purchased from the Kelong Reagent Company. Polyacrylonitrile (PAN, Mw = 50,000, 98%) was purchased from the Wonyang Reagent Company. Fructose was purchased from the Adamas Company. CH₃ONa and acetoxime was bought from Aladdin. All chemicals were used as received.

**Instruments.** FTIR spectra were collected by a Bruker Fourier infrared spectrometer. ¹H and ¹³C NMR spectra were collected by a Bruker AM-400M NMR spectrometer. SEM images of PAN and PAO were taken by field emission scanning electron microscopy (JSM-7500F, JEOL, Japan). Carbon, nitrogen, oxygen atomic % of PAO, PAN, and recycled PAO surface were determined on XPS (AXIS Ultra DLD, Kratos, England). The concentrations of glucose and fructose were detected by high-performance liquid chromatography (HPLC, LC-10T, Surwit, China), with a RI detector (RI-201B, Shodex, Japan) and a sugar-D chromatographic column (4.6 ID × 250 mm, Nacalai Tesque, Japan). ζ potential was measured by Zetasizer Nano ZS90.

**Synthesis of AO.** The synthesis of AO is presented in Supporting Information.

**Synthesis of PAO Particle.** A 200 mL aqueous solution containing NH₂OH-HCl of 3 g was adjusted to pH 7 by adding NaOH solution. The solution was poured into a 250 mL flask, and 1.5 g of PAN was added. The mixture was stirred and heated to 70 °C in a water bath and then kept at 70 °C for 4 h. Subsequently, the white solid was filtered and washed with water three times. The white powder PAO was obtained by evaporating by vacuum overnight.

**Experimental Methods.** A typical reaction was carried out as follows: 0.1 g monohydrate glucose, 0.1 g PAO, and 10 mL water were put into a reaction tube. The mixture was put in the water bath and stirred at different temperatures. After a certain reaction time, the reaction tube was cooled down in an ice bath. The reaction solution was filtrated through a 0.22 μm millipore filter. The solid was collected, and the concentrations of glucose and products in reaction solution were determined by HPLC analysis. Concentrations of glucose and products in reaction solution were determined by HPLC analysis. The mobile phase was a mixture of acetonitrile/H₂O = 75/25 at a flow rate of 0.6 mL/min. The temperatures of the column and detector were 35 and 40 °C, respectively. The pH of solution was measured with a pH meter and adjusted with hydrochloric acid or NaOH solutions. The conversion of glucose (Con %) and the yield (Yield %) and selectivity (Sel %) of fructose are calculated based on following formulas:

\[
\text{Con} \% = \left(1 - \frac{\text{concentration of glucose}}{\text{initial concentration of glucose}}\right) \times 100% \\
\text{Yield} \% = \frac{\text{concentration of fructose}}{\text{initial concentration of glucose}} \times 100\%
\]

\[
\text{Sel} \% = \frac{\text{Yield} \%}{\text{Con} \%} \times 100\%
\]

**ASSOCIATED CONTENT**

**Supporting Information**

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.1c02577.

Synthesis of amidoxime acetonitrile, XPS results of PAO, catalyst activity of PAN for isomerization of glucose to fructose, kinetic behaviors, influence of the neutral salt, FT-IR spectra of recycled PAO after washing with NaOH, and ζ potential results (PDF)

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**Notes**

The authors declare no competing financial interest.

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