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# Hydrothermal conversion of glucose into organic acids with bentonite as a solid-base catalyst

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## ABSTRACT

In this study, a new process of using bentonite as a solid base catalyst in the hydrothermal conversion of glucose into lactic acid was proposed. Results showed that bentonite can act as a solid-base catalyst in conversion of glucose into lactic acid, and the yield of lactic acid increased from 5% in the absence bentonite to 11% in the presence of bentonite. Also, it was found that the presence of bentonite can greatly improve the production of acetic acid, and a considerably high acetic acid yield of 27% could be obtained in the two-step reaction. Since the bentonite is quite cheap and abundant in nature, the reported results should be helpful to facilitate studies for developing a new and green process for the conversion of carbohydrate biomass into value-added chemicals.

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## 1. Introduction

Dependence on fossil fuels as the main energy sources has led to serious energy crisis and has affected environmental sustainability. Since biomass is one of the most important renewable resources, utilization of energy and chemicals from renewable biomass resources would reduce atmospheric $CO_2$ increasing caused by fossil fuel combustion and our dependence on petroleum-derived feedstock. The conversion of biomass into chemicals has received increasing attention for sustainable energy and environment [1–3]. Among the proposed processes of biomass conversion, hydrothermal reactions using high temperature water (HTW) as an environmentally friendly reaction medium have received more attention, because HTW has unique features compared to ambient liquid water; for example, the ion product (Kw) at 250–300 °C is approximately three orders of magnitude greater than that of ambient liquid water, which can help to promote the reactions significantly [4,5]. Extensive researches have demonstrated the high efficiency of hydrothermal conversion of biomass into chemicals [6–8]. Our group has also performed a series of studies on the hydrothermal conversion of biomass into chemicals, particularly for organic acids such as lactic acid, acetic acid and formic acid [9–11].

Lactic acid is regarded as an important platform chemical and its application involves in foods, pharmaceutical and cosmetics [12,13]. Recently, lactic acid is getting attention in manufacture of biodegradable plastics such as polylactic acid (PLA), whose market growth is expected in a sustainable society with a low impact on the environment. Acetic acid and formic acid, which are currently mainly produced from fossil fuel, are also essential feedstock [14,15].

In traditional process of conversion of biomass into lactic acid, strong alkali catalysts such as NaOH, KOH are generally used, which leads to a serious corrosion of the reactors and environmental problem. Thus, the development of solid base catalysts to solve these problems is strongly desired. For the production of acetic acid and formic acid, $H_2O_2$, $O_2$ or air is generally used as an oxidant and thus lead to high energy costs due to compressing gas or even potential insecurity hazards.

Bentonite is a wet clay mineral, mainly containing montmorillonite with layered crystal structure. Its high cation exchange capacity due to isomorphous substitution of Mg for Al in the central alumina plane can be expected to have Bronsted-base sites or show catalytic activity in the conversion of carbohydrate into lactic

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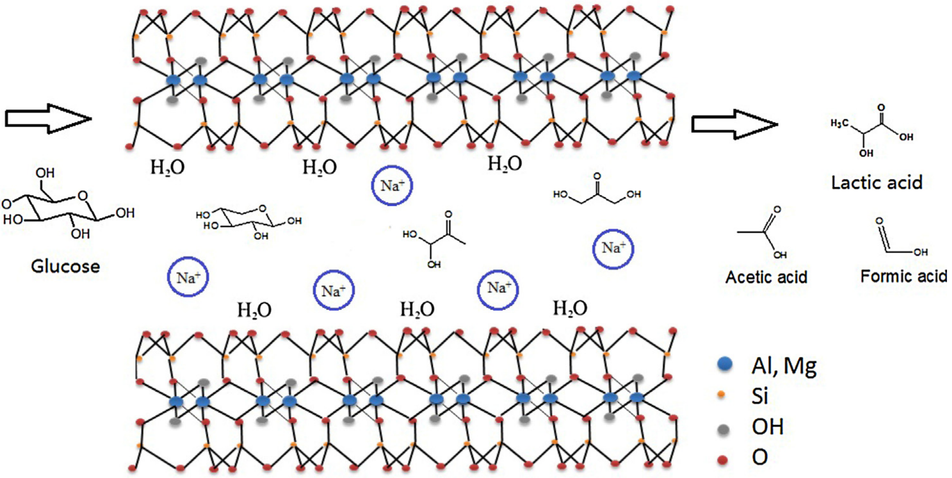

**Fig. 1.** Schematic of hydrothermal conversion of glucose with sodium bentonite.

acid. That is, bentonite may be feasible to use as the solid base catalyst for the production of lactic acid from carbohydrate in water as described in Fig. 1.

We report here the base catalytic activity of bentonite in conversion of glucose into lactic acid, as well as the formation of acetic and formic acids. To the best of our knowledge, there has been no report on the use of bentonite as solid base catalysts in the lactic acid, as well as acetic and formic acids production from saccharides.

## 2. Experimental

### 2.1. Experimental materials

Glucose (AR, Sinopharm Chemical Reagent Co., Ltd) was used as reagent and lactic acid (1.0 N, Alfa Aesar) was used for the qualitative analysis of liquid products. Bentonite (WSG-156) was supplied by Shanghai Wanzhao Fine Chemical Co., Ltd. Deionized water (LD-UPW, 18.2 MΩ) was used throughout the study.

### 2.2. Experimental procedure

#### 2.2.1. One step reaction

All experiments were conducted in a series of batch SUS 316 tubing reactors (9.5 mm outer diameter, 1 mm wall thickness, and 120 mm length) with inner volumes of 5.7 mL. Detailed information of this reactor has been described in our previous research [12]. A brief description of the experimental procedure is as follows. Desired amount of glucose, bentonite and deionized water were first added into the reactor. Then, the reactor was sealed and put into a salt bath which had been heated to the desired temperature (250–350 °C). The reactor was horizontally shaken continuously during the experiment. After desired reaction time, the reactor was taken out from the salt bath and immersed into water bath instantly to quench the reaction. After cooling to room temperature (25 °C), the liquid sample was filtered through a 0.22 μm filter and preserved for further analysis.

#### 2.2.2. Two-step reaction

A two-step reaction divided the reaction into two steps, which included a first step of either put the reactor at room temperature for 12 h or in salt bath (275 °C) for 20 s and a second step of reaction in salt bath (275 °C) for 30 to 90 s. Between the interval of the first and second step reactions, the reactor was exposed in air for 3 min. After the second step reaction, the reactor was taken out from the salt bath and immersed into water bath to terminate the reaction after the second step reaction.

### 2.3. Products analysis

The filtered liquid samples were diluted ten times by weight with deionized water and analyzed by HPLC. HPLC analysis was performed with an Agilent 1200 system, which equipped with two KC-811 columns (SHODEX), a tunable ultraviolet/visible (UV/vis) absorbance detector adjusted to 210 nm and a differential refractometer detector. The solvent was 2 mM $HClO_4$ with a flow rate of 1.0 mL min$^{-1}$. The solid samples were collected and identified by a Shimadzu 6100 X-ray diffractometer equipped with Cu K$\alpha$ radiation ($\lambda = 1.5406$ Å), employing a scanning rate of 0.2° s$^{-1}$ and with $2\theta$ ranging from 10 to 70°. The total organic carbon concentration (TOC) in the liquid sample was also measured with a TOC analyzer (Shimadzu TOC 5000A).

### 2.4. Characterization of catalyst

The amount of Brønsted-base sites in bentonite was characterized by a conventional $CO_2$ adsorption method, which used an automatic chemical adsorption instrument (AutoChem II 2920) and was performed as follows. 70 mg of bentonite was loaded into a quartz tube and hold at 500 °C for 40 min under He atmosphere. Then, $CO_2$ gas was provided instead of He after the bentonite was cooled to 35 °C. After the absorption of $CO_2$ on the bentonite was saturated, temperature of bentonite was raised to 1000 °C at a rate of 10 °C/min to desorb the $CO_2$. A mass spectrometer was used to analyze the desorbed products to obtain the $CO_2$ temperature programmed desorption (TPD) profile.

### 2.5. Definition

In this research, the yields of organic acids were defined as:

$$\text{Yield, mol\%} = \frac{\text{C in organic acids, mol}}{\text{C in intital glucose, mol}} \times 100\%$$

The carbon balance was defined as:

$$\text{Carbon balance, mol\%} = \frac{\text{total organic carbon (TOC) after reaction, mol}}{\text{C in intital glucose, mol}} \times 100\%$$

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