**Physicochemical Analysis and Kinetics of Monosaccharide Formation in Sulfite Acid Hydrolysis of Wood Raw Materials**

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**Abstract.** This paper studies the sulfite acid hydrolysis of wood raw materials, looking closely at its physicochemical steps. The process has two phases: a liquid phase (sulfite acid solution) and a solid phase (wood chips). The stages include: capillary impregnation, breakdown of hemicellulose and cellulose, monosaccharide formation, and secondary changes. Experiments used poplar wood, a 5% sulfite acid solution, and temperatures of 150-180 °C. A MacKenna-Shoorl method determined the amount of reducing substances in the hydrolyzate. We also looked at the hydrolysis, how the water to wood ratio affects it, how sugars form, and the good things about using sulfite acid. The liquid parts obtained could be useful in making biopolymers, while the solid parts could be used as alternative fuels or composite material filler.

**Keywords:** Wood raw materials, hydrolysis, sulfite acid, heterogeneous process, hemicellulose, cellulose, monosaccharides, reducing substances, water to solid ratio, hydrolysis kinetics, MacKenna-Shoorl method, biopolymer, alternative fuel

**INTRODUCTION**

Wood raw material hydrolysis has a heterogeneous nature, with two key phases: a liquid phase of sulfite acid and distilled water, and a solid phase of wood raw material, or wood chips. The wood hydrolysis process has these steps. Wood chips are soaked in a low concentration sulfite acid solution [1]. Chemical reactions occur between the sulfite acid solution and wood components, including polysaccharide breakdown. This creates monosaccharides and a byproduct, cellolignin [1, 2]. Monosaccharides then break down, creating other products. At first, capillary forces drive the soaking of wood chips. Solution goes into the wood fibers, pushing air out of the wood capillaries. Some air stays in the capillaries and diffuses out [3]. The sulfite acid solution slowly starts chemical reactions. How good the sulfite acid wash is directly affecting how fast the chemical reaction happens. The faster the wash, the faster the reaction. The reactions can be divided into two stages [4]. The start of temperature rise causes the first stage, which activates and breaks down wood components, starting the easy to hydrolyze hemicellulose breakdown, because it is more amorphous than cellulose. As hemicellulose breaks down into monosaccharides, they move into the reaction liquid, letting sulfite acid go deeper. The liquid then breaks down cellulose, forming most of the monosaccharides [5, 6, 7].

Because hydrolysis takes a while, the monosaccharides change a lot. They break down into products like fural, acetic acid, and formic acid. The products can be used in ways such as the liquid fractions used as food for Xanthomonas campestris and Cupriavidus necator bacteria to create biopolymers [8]. The solid fractions (cellolignin) can be used to provide energy or as filler. Research has shown that poplar wood works best for hydrolysis [9]. So, poplar wood chips (under 2 mm) and a 5% sulfite acid solution made in the lab acted as raw materials. Treating wood raw materials with sulfite acid reduces the poisonous nature of the hydrolyzate to biopolymers [10, 11]. As sulfite acid use makes it simpler to purify of hydrolyzates. Also, sulfite acid is a good option due to being simple to store and use, cheap, and decreases equipment wear because of its low corrosive properties.

**MATERIALS AND METHODS**

A capsule device studied acid hydrolysis in acid. Prepared poplar wood chips went into 30 ml stainless steel capsules. The capsules then went into a heating jacket. Wood raw material hydrolysis occurred at 150–180 °C. A DM90 (0 − 2,5 MPa) manometer measured pressure, and a TPM 210 thermoregulator measured temperature [10]. An analytical scale weighed the poplar wood and placed it into capsules. We then put water and sulfite acid solution into each capsule based on the weighing results [11]. The total mass in each capsule equaled 34.5 g.

**RESULTS AND DISCUSSION**

Before starting, the support cylinder had to heat to 10 °C higher than the working hydrolysis temperature. After the initial heat, the thermoregulator was set to working temperature, and the hydrolyzer went into the support heating cylinder. Hydrolysis occurred over time. The first capsule was removed from the cylinder with a special holder and cooled fast in cold water. The other capsules were removed during that same time to find the process. The capsule contents were filtered by hand through a cloth filter to isolate the solid material from the liquid. We measured the number of reducing components as well as the number of monosaccharides. Sulfite acid was created with equipment that uses clean sulfur. The sulfur was put into the equipment, lit, and closed with a dense stopper with an oxygen meter. This meter controlled the sulfur burning and steam levels. The burning sulfur makes sulfite (SO₂) gas that is cooled in a cooling chamber. The cooled gas moves to the column, in which the gas travels downward in a circular motion. In this process, the gas goes into the water. The equipment sorbs about 40% газ. The resulting sulfite acid concentration measures 8%. We watered this solution to 5 mass% for the studies.

A MacKenna-Shoorl method determined the amount of reducing components. This method measures volume and modifies the Bertran method. Volume measures use a Fehling solution with a definite copper amount. The amount of copper returned through sugars while boiling figures out from the residue of unused copper (II), using a iodometric method. Process of measuring: Put 1.0 ml hydrolyzate solution and 1.0 ml 2N hydrochloric acid in a heat resistant flask. Heat the flask in a water bath at 80 °C for 10 minutes to hydrolyze the saccharides within the hydrolyzate. Afterward, put 8 ml of distilled water, 10 ml of Fehling I solution, and 10 ml of Fehling II solution. Boil the mixture for two minutes on a hot plate, then immediately cool it to 25 °C in cold water. Put 2 g potassium iodide and 15 ml of 20% sulfuric acid to the mixture and titrate using 0.1N sodium thiosulfate solution, using 1% starch solution as an indicator, stirring constantly. Keep titrating until the blue tint runs out. Under those exact same conditions, do different control analysis (empty experiment) without hydrolyzate. By the difference in sodium thiosulfate solution amount used for working and empty analysis, the empirical table shows the content of reducing substance in the hydrolyzate sample. Put the separated dry residue into an AND MS-70 moisture thermogravimetric analyzer to measure the quantity of dry material in the hydrolyzing mixture. The water to solid ratio, that is, the ratio of raw plant material mass to acidic water solution mass, is key during the hydrolysis.

The amount of sugar from the raw materials and its concentration in the hydrolyzate, as well as the amount of acid used, depend on this. Empirically, the best ratio has been found to be 1:5.8. To model mathematically, the hydrolysis has chemical reaction speed rates to hydrolyze hemicellulose, cellulose, and monosaccharides. So, studies attempted to find the relevant speed rates for further math modeling. The initial conditions used poplar wood concentration values. In order to discover the chemical reaction speed rate for easy to hydrolyze polysaccharides (mainly hemicellulose) in poplyar wood experimentations were done in the settings. Fine wood raw material with 2 mm divisions were used. Conducted hydrolysis with 4 mess sulfite acid. Temperatures: 150, 160 °C, 170 °C and 180 °C; Duration of hydrolysis: 40 minutes at 160 °C, 30 minutes at 170 and 180 °C. Figure 1 shows the change in hemicellulose during hydrolysis. The wood hemicellulose drops over time. The graph has three sections: Soak section as you set up for hydrolysis. Straight section defining hemicellulose going down during the main hydrolysis. Tensity decreases here because the rate of hydrolysis goes down due to hemicellulose reduction. It is from here that you can find the reaction for modeling.

**FIGURE 1.** Kinetics of hemicellulose content change versus hydrolysis time in the sample

The chemical reaction rate constant for hemicellulose hydrolysis is expressed using:

(1)

To find the cellulose hydrolysis rate constant, treated cellulose mass was prepared as follows: Pine wood raw material was first dried in a vacuum oven at 105 °C. It was next treated with 2% sodium hydroxide solution for   
45 minutes at 80 °C using a magnetic stirrer. The resulting cellulose fiber was filtered, washed several times in distilled water until neutral, and dried at 60 °C for 4 hours. Then, the fibers were bleached in a mixture of glacial acetic acid, 1.7% sodium chlorite, and distilled water at 90 °C for 12 hours under constant stirring; at this step, lignin was removed. After bleaching, the cellulose underwent hydrolysis, and the chemical reaction rate constant was found. Hydrolysis was done at 150, 160, 170, and 180 °C in 5% sulfurous acid solution by taking capsules out every 10 minutes. The bleached fibers were washed again and dried at 80 °C to a constant weight. The first-order kinetic equation for cellulose hydrolysis is expressed in the following integral form: Formula 2.

(2)

The results for the change in cellulose content versus hydrolysis time are in Figure 2.

**FIGURE 2.** Monosaccharide levels in solution during hydrolysis

The chemical reaction rate constant for the re-hydrolysis of the hydrolyzate can be shown as:

(3)

For ease, Table 1 shows the chemical reaction rate constants for hemicellulose, cellulose, and monosaccharides at 150, 160, 170, and 180°C over different time periods, after they were determined.

**TABLE 1.** Calculation of the chemical reaction rate constant

|  |  |  |  |
| --- | --- | --- | --- |
| **Processing time** | **Component name** | | |
| **Hemicelluloses** | **Celluloses** | **Monosaccharides** |
| **k, с-1** | **k, с-1** | **k, с-1** |
| 150 °C | 0,00025 | 0,00014 | 0,00011 |
| 160 °C | 0,0004 | 0,00023 | 0,00014 |
| 170 °C | 0,0007 | 0,00038 | 0,00016 |
| 180 °C | 0,0013 | 0,00053 | 0,00019 |

Based on the data, the reaction rate constant for hemicellulose formation at 150 °C is 0.00025 s⁻¹, and it increases notably as the temperature rises. For instance, there are increases of +60% from 150 °C to 160 °C, +75% from 160 °C to 170 °C, and +86% from 170 °C to 180 °C, which is typical for an exothermic reaction. Hemicellulose is a component that degrades easily, with this process starting in the initial hydrolysis stage.

Looking at cellulose formation, the approximate rate constant at 150 °C is 0.00014 s⁻¹, with a lower rate increase of +64% from 150 °C to 160 °C, +65% from 160 °C to 170 °C, and +39% from 170 °C to 180 °C. Cellulose is more stable and, therefore, more resistant to temperature. However, the reaction rate increases considerably between 170 °C and 180 °C.

For monosaccharide formation, the rate constant at 150 °C is 0.00011 s⁻¹, rising slightly with temperature, such as +27% from 150 °C to 160 °C, +14% from 160 °C to 170 °C, and +19% from 170 °C to 180 °C. This component both forms and decomposes into products like furfural, acetic acid, and formats, which slows the overall reaction rate increase. In short, 150 °C is a slow initial stage for the hydrolysis process, where components begin to react but with low efficiency.

The ranges for optimum temperatures are: hemicellulose at 160-170 °C, as it is a quickly degrading component; cellulose at 170-180 °C, which has a more stable structure but becomes active at higher temperatures; and monosaccharides, which have more of a balance between formation and decomposition, with formation increasing at higher temperatures but with a higher risk of decomposition.

**CONCLUSION**

A study was done on the sulfite acid hydrolysis of wood raw material, detailing its physical and chemical properties and its kinetic parameters related to temperature. The results showed that the hydrolysis process has a heterophase nature, including capillary impregnation, component decomposition, and the transfer of created products into the solution. The analysis used raw material consisting of a fraction of poplar wood up to 2 mm and a 5% solution of sulfite acid. As temperature increased, so did the reaction rate constants for hemicellulose, cellulose, and monosaccharides. Hydrolysis begins slowly at 150 °C, but reactions become notably active between 150-180 °C. Hemicellulose, the fastest degrading component, showed a maximum k = 0.0013 s⁻¹ at 180 °C. Cellulose and monosaccharides reacted more slowly but with a stable rate. Amounts of reducing substances in the hydrolyzate were found using the MacKenna-Shoorl method. The study included an examination of the created products. The key rate constants needed for mathematical modeling of hydrolysis kinetics were determined based on this data. Results point to wood raw materials, especially poplar wood, as an efficient and economically suitable material for sulfite acid hydrolysis. The liquid fractions created are hopeful for use in biotechnology and biopolymer production, and solid residues could be a source of energy or fillers for composite materials.

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