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Glucose and 5-hydroxymethylfurfural production from cellulosic waste by sequential alkaline and acid hydrolysis

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ABSTRACT

Glucose and 5-hydroxymethylfurfural production from waste paper towel was evaluated by sequential alkaline and acid hydrolysis. Effects of alkali/acid doses, treatment time and temperature on hydrolysis performance were investigated by using Box-Behnken and Box-Wilson statistical experiment design methods. Operation parameters for alkaline hydrolysis were percent NaOH, treatment time and temperature and for acid hydrolysis percent acid and treatment time. Total paper conversion of the sequential process was 72% indicating significant waste reduction beside glucose and 5-hydroxymethylfurfural formation. The maximum yields for glucose and 5-hydroxymethylfurfural were 61.23% and 19.70% in acid and alkaline hydrolysis, respectively. Main products in the effluent of alkaline hydrolysis were glucose and 5-hydroxymethylfurfural. Highest glucose concentration (25.2 gL^{-1}) in alkaline hydrolysis was obtained at 16% NaOH, 119 °C, and 74 min. The 5-hydroxymethylfurfural concentration and paper conversion at this condition was 7.22 gL^{-1} and 28.47%, respectively. When the solid residue of alkaline hydrolysis was treated with 5% H_2SO_4 at 135 °C for 120 min then 30.7 gL^{-1} glucose and 0.87 gL^{-1} 5-hydroxymethylfurfural was obtained with 44.35% paper conversion.

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1. Introduction

Fossil fuels are intensively used to meet todays increasing world energy demand [1,2]. However, the depletion risk of those reserves in the near future has forced many countries to search for sustainable and more environmental friendly options [3,4]. In this context biofuels and bio-oils are considered as potential alternatives [1,5–7], which can be produced by biological or thermochemical processes from carbohydrate containing waste materials [8]. During the biological route microorganisms can convert carbohydrates like glucose into biogas and liquid biofuels by fermentation. The thermochemical route on the other hand is accomplished by pyrolysis, gasification or hydrothermal processes [9]. Glucose can easily be metabolized by various types of microorganisms for biofuel production and its dehydrated form 5-hydroxymethylfurfural is a platform chemical [10] that can be used to produce alkane-bio oil which has almost the same chemical composition as petroleum [7]. However, glucose is usually not readily available in nature and therefore has to be produced by proper technologies from starch or cellulose [11]. Waste tissue paper is rich in cellulose (>90%) [12] and can be evaluated to produce glucose by chemical [13–17] and enzymatic [8,18,19] hydrolysis. Theoretically 1.1 g of glucose can be obtained by acid hydrolysis of 1 g cellulose [20,21]. Tissue paper is widely used in the community for personal hygiene and hand drying purposes. With 4.1% annual growth rate the world tissue paper consumption is expected to reach 32 million tonnes in 2015 [22]. By considering this amount, 90% cellulose content of tissue paper equals to 31.68 million tonnes of glucose reserve. Waste tissue paper can be recycled, composted or incinerated [23]. But usually its life-span ends up in the landfill or sewage resulting in a huge glucose squander. It was reported that about 270.000 trees in the form of tissue paper are flushed down the drain or end up as garbage all over the world every day [24]. The energy content of waste tissue paper is (15.2 MJ/kg) however, this energy content can be increased two to eight fold when it is converted to biofuels like bio-ethanol (26.8 MJ/kg) or bio-hydrogen (120 MJ/kg) [25].

Acid and alkaline hydrolysis of cellulose results in the formation of glucose, carboxylic acids and 5-HMF [6]. Therefore those

Abbreviations: ACH, Acid hydrolysis; ALH, Alkaline hydrolysis; BBSED, Box-Behnken statistical experiment design; BWSED, Box-Wilson statistical experiment design; COD, Chemical oxygen demand; 5-HMF, 5-hydroxymethyl furfural; PC, Paper conversion; WPT, Waste paper towel.

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processes could be used to treat waste tissue paper for the production of glucose and 5-HMF.

Acid hydrolysis of cellulose is a known process [26,27] and sulphuric acid is mostly preferred due to its less corrosive and cheap application compared to other acids such as HCl or HNO_3 [28]. Acid hydrolysis is accomplished using concentrated and diluted acids. Concentrated acid hydrolysis is usually applied at room temperature with acid concentrations above %50 [29]. In this process, the acid promotes the swelling, solubilisation and decrystallyzation of cellulose [15] resulting in the formation of glucose and oligosaccharides after the breakage of the β-glycosidic bonds [17]. During dilute acid hydrolysis first long chain cellulose is converted into small chain high Degree Polymerization (DP) cellulose. Next high DP is converted into low DP oligomers and then the oligomers to glucose. 5-HMF is an intermediate compound that is produced during acid hydrolysis and can be converted to bio-oil [7] after the recovery from the hydrolysate [30]. Theoretically 0.77 g 5-HMF is produced from 1 g cellulose with the dehydration of glucose [31].

The number of studies on alkaline hydrolysis of cellulose are less than acid hydrolysis in the literature. Cellulose hydrolysis under alkaline conditions proceeds by the peeling, stopping and scission reactions [32]. At alkaline conditions with temperatures below 170 °C, chain dissolution occurs from the reducing ends of the amorphous parts of cellulose known as peeling [33]. This dissolution continues as long as reducing ends exist within the chain, however it is stopped whit the formation of stabile acid terminal units [33]. Differently from peeling, at alkaline conditions with temperatures higher than 170 °C the glycosidic bonds are cleaved by scission resulting in a decrease of the cellulose weight and its degree of polymerization [33] and the peeling proceeds from accessible newly formed reducing end groups. Depending on acid or alkaline cellulose hydrolysis, the hydrolysate might contain a number of intermediate dissolved organic molecules such as fructose, pyruvaldehyde, glyceraldehyde, 1,2,3-benzentriol, humins [6] and the soluble chemical oxygen demand (COD) can be used to get an idea of the total concentration of those dissolved organics compounds [34–39]. 5-HMF is a product in acid hydrolysis however, it can also be formed during alkaline hydrolysis [7,40]. The pathway and end products of cellulose hydrolysis was reported to be dependent on the pH of the media [6,41]. At acidic conditions the main products are 5-HMF and levulinic acid. However, at alkaline conditions carboxylic acids such as acetic and lactic acid are the end products [6]. The carboxylic acids mainly formed during alkaline hydrolysis reduce the pH resulting a shift from alkaline to acidic conditions and leading the formation of 5-HMF from glucose [7].

The crystallinity is limiting the conversion of cellulose in both acidic and alkaline hydrolysis [42,43]. Alkaline hydrolysis is successfully applied to cellulose as a pretreatment prior enzymatic hydrolysis which enables to increase the number of accessible reducing sugars ends and to impact the crystallinity for more convenient microbial degradation [8,44–46]. With a similar approach alkaline hydrolysis of WPT prior acid hydrolysis might enhance the hydrolysis efficiency which in our knowledge has not been reported before.

In light of the information given above WPT was chosen as feedstock to produce glucose and 5-HMF by sequential alkaline and acid hydrolysis. Box-Behnken and Box-Wilson statistical experiment design methods were used to determine most convenient hydrolysis conditions resulting maximum glucose and 5-HMF formation.

2. Materials and methods

2.1. Experimental setup and procedure

WPT subjected to sequential alkaline and acid hydrolysis was obtained from the toilets of Pamukkale University Engineering Faculty. The WPT was first cut into small pieces (<0.5 cm) and then dried in an oven at 50 °C for two days prior the experiments. Hydrolysis experiments were performed in 250 mL serum bottles (Isolab-Germany Boro 3.3) which were placed in an autoclave operated at adjusted temperature and time (HIRAYAMA HG-50). The initial dry weight of the WPT was 3 g and dry weight to liquid ratio was kept constant as 1/20 (w/v) during all experiments. Acid hydrolysis experiments were done after alkaline hydrolysis. Acidic and alkaline conditions were adjusted using analytical grade chemicals of NaOH and H_2SO_4 from Merck Co. Initial and final dry weights of solid fractions were recorded for each experiment. All experiments were done in duplicate. Operation conditions for acid and alkaline hydrolysis were determined using statistical experiment designs as explained in Section 2.3. Paper conversion in the text defines the paper mass reduction percentage. Glucose and 5-HMF yields were calculated as following [31,47]:

$$\text{Glucose Yield } (\%) = \frac{\text{Glucose concentration in the hydrolysate } (gL^{-1}) \times \text{Hydrolysate volume (L)}}{\text{Glucose equivalent in raw paper towel (g)}} \times 100 \quad (1)$$

$$5-\text{HMF Yield } (\%) = \frac{5-\text{HMF concentration in the hydrolysate } (gL^{-1}) \times \text{Hydrolysate volume (L)}}{5-\text{HMF equivalent in raw paper towel (g)}} \times 100 \quad (2)$$

2.2. Analytical methods

After each experiment the solid fraction was separated from the liquid by vacuum filtration (12 μm pore size and 110 mm diameter filter) using a Buchner funnel. The filtrate was subjected to glucose, 5-HMF and COD analysis and the solid fraction was dried at 50 °C after washing with distilled water until the pH was 7. Glucose was determined according to the phenol-sulphuric acid method [48]. 5-

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