



# A BRIEF SURVEY OF BIOMASS HYDROLYSIS AS A VITAL PROCESS IN BIO-REFINERY

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#### ABSTRACT

This report presents a mini-survey of biomass hydrolysis, which tends to unfold the concept of what the process is all about, categories involved, and reporting concerning the processes involved in each category. The categories entail the chemical and biological means where acid catalysts and enzymes are used, respectively, including reports of some works carried out in the literature.

Keywords: Biomass, Bio-refinery, Hydrolysis, Renewable Chemicals, Monosaccharide.

### INTRODUCTION

Lignocellulosic biomass is commonly referred to as a non-starch, fibrous component of plant biomass comprising three key constituents: lignin, hemicellulose, and cellulose (<u>Basu, 2010</u>). These categories of materials can be a source from forestry residues (<u>Sathitsuksanoh et al., 2010</u>) such as sawdust, wood waste, mill scrap; energy-woody crops such as switchgrass, willow, poplar; industrial and municipal solid wastes like paper mill sludges, wasted paper, recycled newspaper; and, residues from agriculture such as rice husk, maize cob, maize stalk, sugarcane bagasse (Ajayi et al., 2020; Ingale et al., 2014; Madu & Agboola, 2018; Oyegoke et al., 2021; Tsunatu et al., 2017).

However, this lignocellulosic biomass can be converted into biofuel-like bioethanol production without competing with the food market. In addition, bioethanol production from lignocellulosic biomass would generate residue in the form of lignin which can further be employed to produce valuable fuel additives, including power generation. The conversion process was designed to reuse all solids by transforming them into a profitable and valuable product (Farone and Cuzens, 1996). Some of these lignocellulosic biomasses include rice husk, wood, maize cob, sorghum bagasse, sugarcane bagasse, and many other wastes (Basu, 2010, Yanuar et al., 2014). The conversion of lignocellulosic biomass into bioethanol fuels includes hydrolysis of cellulose into fermentable sugars and fermentation of the sugars to ethanol (Sun and Cheng, 2002). And the significance of the hydrolysis steps cannot be overemphasized.

However, this report presents a mini-survey of the concept, categories, and trends of some works in the literature on biomass hydrolysis into simple sugars like glucose and fructose in the presence of enzymes or acid catalysts.

### CONCEPT OF BIOMASS HYDROLYSIS

The process majorly involves breaking down biomasses into small unit (otherwise known as reduced or fermentable sugars) in the presence of either an enzyme or catalyst, referred to as hydrolysis. Whitten et al. (2003) and Jan (2007) referred to hydrolysis as a disruptive reaction that entails splitting a compound into two parts, one of which combines with the hydrogen (H) ion of water and the other combines with the hydroxyl (OH) ion of water. The process has been identified as one of the vital components in producing lignocellulosic bioethanol.

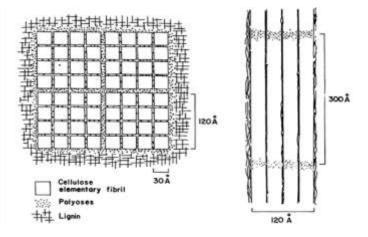


Figure 1. Model of ultrastructural organization of the cell wall components of biomass like wood. Source: Liang-tseng et al. (1987).

In addition, Joseph and Ronald (2010) defined the hydrolysis of biomass to be the conversion of cellulose into sugar which principally degrades the chemical bonds present in cellulose (as shown in Figure 1), involving the hydrolytic cleavage of  $\beta$ -1,4-glycosidic bond, which is catalyzed by the action of a cellulolytic

enzyme, or by  $H^{\top}$  ions of an acid. Figure 2 shows are reaction mechanism of hydrolysis.

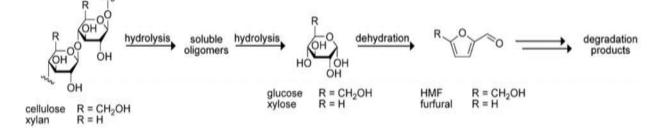


Figure 2. Hydrolysis of cellulose and xylan into monomeric sugars through oligomer and side reaction leading to side products like furfural. Source: Joseph and Ronald (2010).

Presently, the conventional methods for hydrolysis are categorized into two based on thermochemical route (otherwise known as acid-catalyzed hydrolysis) and biochemical route (also known as enzyme-catalyzed hydrolysis); both of which require pretreatment steps to utilize lignocellulosic biomass. (Yanuar et al., 2014)

## HYDROLYSIS BY CHEMICAL MEANS

This process is also called acid hydrolysis or thermo-chemical hydrolysis. In some instances, this process is known as saccharification. It is a highly efficient chemical process for converting biomass into simple sugar or monosaccharides in the presence of an acid catalyst. Concentrated acid serves a dual purpose in the hydrolysis of biomass. This process is achieved by breaking or disrupting its network of inter- and intra-chain hydrogen linkages. The strong acids decrystallizes the cellulose and make it accessible for reagents to enable reaction initiation. And by catalysing hydrolysis from glycoside bonds, strong acids separate cellulose and hemicellulose into sugars (Joseph and Ronald, 2010). Figure 3 diagrammatically depicts the phenomena mentioned earlier about the hydrolysis process in bioenergy production (Kang et al., 2018).

The report of Xiang et al. (2003) indicated that the acid-catalyzed hydrolysis is controlled not only by acid concentration and temperature instead also by the physical state of the cellulose. The report further indicated that the cellulose structure stays in the stable crystalline form under low temperature and acid conditions. Therefore, the dominant reaction method is endwise hydrolysis resulting into glucose which becomes the main sugar product. However, when acid concentration and/or temperature are raised to a certain level, the cellulose structure becomes unstable by the breakage of hydrogen bonding, the primary force that holds the cellulose chains. Once the cellulose crystalline structure is broken or disrupted, acid molecules penetrate the inner layers of the cellulose chains pictorially shown in Figure 3 (Xiang et al., 2003).

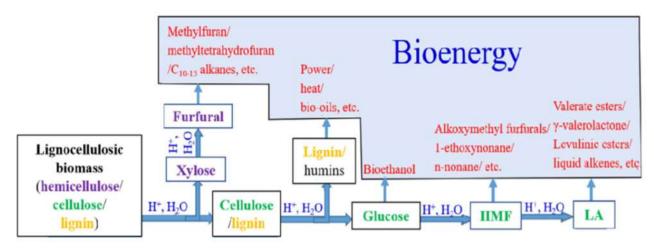


Figure 3. Pictorial representation of acid hydrolysis for bioenergy. Source: Kang et al. (2018).

The state of hydrogen bonding affects kinetic behavior. If the hydrolysis reaction is carried out under a condition that severely disrupts hydrogen bonding (high-temperature condition), glucose and its oligomers are formed. Since oligomers are more stable than monomers against decomposition, adopting such a reaction condition may increase sugar yield. Quick quenching of the reactor effluent is an efficient way to raise the glucose yield, especially in the high-temperature zone. Glucose-lignin recondensation occurs during acid hydrolysis of cellulose and is an essential factor causing loss of sugar (Xiang et al., 2003), as shown in Figure 4.

### DILUTE ACID HYDROLYSIS

Dilute acid hydrolysis is generally carried out on biomass intended to hydrolyse cellulose and hemicellulose into reduced monomeric, furfuran or organic acids. This process is usually performed with diluted acid at elevated temperatures. However, lignin produced during this process, also known as hydrolysis lignin, is generally not suitable for use as a functional material (Hata and Nonaka 2018).

Some of the literature that examined the process included Bardone et al. (2014), which employed dilute sulphuric acid in hydrolyzing the sugarcane bagasse into glucose, recording a yield of 70.9% glucose (at 155 °C, 10 min, and 2 w/v % sulphuric acid). Another author was Gurgel et al. (2012), whose works also investigated the similar transformation of sugarcane bagasse into glucose, resulting in a yield of 69.8% glucose (at 210 °C, 0.14% H<sub>2</sub>SO<sub>4</sub>, 10

min). And many other works in the literature (Karimi et al., 2006; Pedroso et al., 2019).

### **CONCENTRATED ACID HYDROLYSIS**

Hydrolysis of lignocelluloses using concentrated acids achieves near-theoretical sugar yields and fewer degradation products than the more commonly employed dilute acid hydrolysis process (Shiraki et al. 2020). This concentrated acid hydrolysis is a process that has been reported to have been in use since the early twentieth century. However, it is presently discouraged due to the large amount of acid consumed in transforming biomass or materials into simple sugars, which makes the process less economically favourable. However, the invention of effective acid recovery solutions for the concentrated acid hydrolysis process has reactivated the interest in this process, which was initially regarded as being economically less viable due to the large amounts of the fund invested in the purchase of acid required in the process. (Størker et al., 2012).

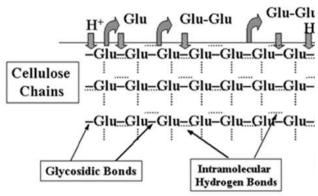


Figure 4. The illustration of crystalline cellulose structure and acid hydrolysis mechanism. Source: Xiang et al. (2003).

The good sugar yields and low content of fermentation inhibitors indicate that two-stage concentrated acid hydrolysis may be a good alternative for the single-step approach in processing the biomass, especially maize cob, softwood, sugarcane bagasse, and many others (Størker et al., 2012). Moreover, concentrated sulfuric acid hydrolysis has been reported by Shiraki et al. (2020) to be the most effective process to recover the maximum yield of monomeric sugars from woody biomass since concentrated sulphuric acid can completely inflate and hydrolyse cellulose. And during this process, the authors further report that the sulfuric acid lignin is quantitatively produced as a by-product, which is challenging to use because of self-condensation between the lignin molecules under acidic conditions (Shiraki et al., 2020).

Moreover, the literature review indicates that hydrolysis with concentrated sulphuric acid has key benefits: low cost, low temperature, high sugar yield, and quick reaction. Recovery of the wasted sulphuric acid in this process has been a significant challenge despite the vast benefit it tends to offer. The literature survey indicated that this challenge had been an age-long problem seeking a solution (Kobayashi et al., 1977; Goldstein et al., 1989). It was also reported that some works attempt recovering waste sulfuric acid with the use of ion-exchange resin films in order to recycle the sulphuric acid, which has been proven to be so challenging due to the homogeneity of the acid catalysts with the substrate in the early twenty century (Nanguneri and Hester 1990).

The lignin obtained as a by-product, called sulfuric acid lignin (SAL), has no use (Yoshihara et al., 1984; Klason, 1923). This was because the lignin loses its thermo-plasticity and solubility in organic solvents during this process, making it challenging to utilize this lignin as a material (Shiraki et al., 2020). SAL was prepared with high yield and high purity and is generally employed in determining the lignin content of a woody biomass (Yoshihara et al., 1984; Klason, 1923). For the practical application of concentrated sulfuric acid hydrolysis, it is essential to reduce production costs, which can be achieved by utilizing the high-value-added lignin (Shiraki et al., 2020).

The hydrolysis (or saccharification) of biomasses (like sawdust, rice husk, wood, and many others) with concentrated sulfuric acid has been extensively investigated to obtain monosaccharides that could be transformed into the production of bioethanol and other chemicals by fermentation and also to address some challenges recorded in literature or faced in practice currently like loss of lignin value at this process.

In recent times, Shiraki et al. (2020) succeeded in identifying technology that would result in a high-yield production of sugars and thermoplastic lignin by the concentrated sulfuric acid treatment of softwood with t-butyl alcohol (TBA) as an additive. The authors' study was found to be the first report to demonstrate

this competency of TBA to upgrade lignin after investigating the contribution of different parameters such as sulfuric acid concentration, reaction time, and TBA amount, to understand ways of controlling the functionality of the lignin and maximize sugar yields alongside. With findings reported by Shiraki et al. (2020), it was ascertained that the economic efficiency of the concentrated acid hydrolysis process is considerably improved by upgrading the lignin (Shiraki et al., 2020). This discovery, therefore, renders the process vital in the realization of sugar platform biorefineries.

#### HYDROLYSIS BY BIOLOGICAL MEANS / ENZYMATIC HYDROLYSIS

Given the reaction conditions investigated, fermentation inhibitor levels in softwood hydrolyzates did not increase to levels affecting anaerobic fermentation by Saccharomyces cerevisiae ATCC 96581. Concentrated acid decrystallization and hydrolysis may also be a viable alternative for saccharification of xylan-rich lignocellulosic biomass like hardwoods. However, some furfural production must be expected, possibly necessitating a detoxification treatment before fermentation (Størker et al., 2012)

This process is also known as enzymatic hydrolysis. Some other schools of thought referred to it as a biochemical routed hydrolysis method (Isah et al., 2019; Monday Osagie, 2017; Nnaemeka et al., 2021). This process involves the breakdown of cellulosic and hemicellulosic polymers into fermentable sugars like glucose and xylose in the presence of an enzyme. The enzymatic hydrolysis is sometimes preferred over other methods due to its mild operating conditions and less formation of inhibitors (Kang et al., 2018).

Fan et al. (1987) define enzymatic hydrolysis of cellulose as a process that accomplishes degradation of cellulose (insoluble reactant) to glucose in the presence of a soluble catalyst (enzymes) which proceeds at a shallow rate. Therefore, its pretreatment before hydrolysis is vital in facilitating the rate of biomass hydrolysis. However, the author further indicated that the reaction rate is influenced by the mode of enzyme action and structural features of cellulose, including the mode of interaction between cellulose and enzyme.

The literature survey indicated there are scanty works on the investigation of kinetic modeling of this process and the impact of mass transfer on it. The understanding would aid in better improving the mode of interaction of the cellulose with the enzyme. It is, therefore, necessary for further works to consider a study on this aspect.

### SUMMARY

Among available methods of hydrolysis, literature survey shown that concentrated acid hydrolysis is one of the future methods which is built on thermochemical route, with many of its advantages as regards to its milder operating condition using a lower operating temperature and pressure; the higher sugar recovery and conversion rate when compared with dilute acid hydrolysis. Higher efficiency of sugar recovery for both glucose and xylose, which is one of the advantages of this process, can reach over 90% of theoretical yield at lower operating temperature and pressure. Hence the diminishing sugar yield owing to inhibitors' formation can be minimized.

More so, concentrated acid hydrolysis has a higher reaction rate, which unswervingly indicates a shorter reaction time when compared with enzymatic hydrolysis. The major limitation in using enzymatic hydrolysis is finding the suitable pretreatment method and less expensive method of enzyme preparation to make readily available the lignocellulosic substrate. Because of the exceptionally complex and intense obstinacy of lignocellulosic use of corrosive resistance reactors due to higher toxicity and Biochem corrosivity or specialized non-metallic constructions. The vital https://doi.org/10.1007/BF02936475 need for the acid recovery process is to make the process economically achievable. Concentrated acid hydrolysis remains Gurgel L.V.A., Marabezi K., Zanbom M.D., and Curvelo A.A. da valuable and, in much use today, as this method has been proven to have a low overall cost for the production of bioethanol (Groenestijn et al., 2006). The concentrated acid hydrolysis profitable implementation, as reported by Arkenol, Inc., a company in USA. This company has established a technology that has been proven to be technically viable for commercial application with a component with ion exchange resins designed to separate the remaining acid-sugar mixture into the acid catalyst and sugar components without diluting the sugar.

A clean stream of mixed sugar (both hexoses and pentoses) for fermentation, was this process claimed to be capable of producing since the separated sulfuric acid was recycled and reconcentrated to the level demanded by decrystallization and hydrolysis steps; and the slight quantity of acid left in the sugar solution was neutralized with lime to make hydrate gypsum, CaSO4·2H2O, an inexplicable precipitate which could be separated from the sugar solution and used as an agricultural soil conditioner. Masada Resource Group has also established large-scale cellulosic ethanol production projects in North America (Taherzadeh & Karimi, 2007) using the approach mentioned earlier to recover their acid catalyst from the end-products.

Due to the requirement of the large amounts of acid, concentrated acid hydrolysis was earlier reported as an economically not feasible process, but the improved development of effective acid recovery technologies and the high adaptability of this process towards different substrates has renewed its interest (Janga et al., 2012, Moe et al., 2012; Yanuar et al., 2014).

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A BRIEF SURVEY...



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