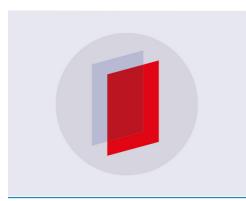
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# **Production of Glucose from Waste Bark Acacia Mangium Using Delifnification and Chemical Hydrolysis Process**

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**Abstract.** The bark of Acacia mangium is lignocellulosic biomass from waste pulp which can be recovered by means of hydrolyse to produce simple sugar called glucose. To produce glucose, there are two stages of the process performed is process using solvent delignification NaOH and KOH. The parameters analyzed were the biomass with a solvent ratio (1:6, 1:8, 1:10, 1:12). The second stage cellulose is then hydrolyzed to see the influence of sulfuric acid concentration of the resulting glucose content (0.1; 0.25; 0.40; 0.55% (v/v)). Lignin removal efficiency using NaOH and KOH solvent that is almost equal to 62.39%. Decrease hemicellulose using KOH solvent (36%) are more efficient than solvent only 29.3% NaOH. Efficiency increased levels of cellulose using NaOH or KOH solvent almost equal approximately 85.5%, but with KOH the lower alkaline properties provide a more stable rise. Solvent delignification process with NaOH provide large enough glucose content at a ratio of 1: 6 and 1: 8 with sulfuric acid concentration in the range of 0.1 to 0.4 and 0.5% v/v. Increased glucose content in the solvent of NaOH at a ratio of 1:6 reached 69.88 ppm for sulfuric acid concentration of 0.55% v/v, whereas the content of glucose degradation products of cellulose with delignification process using KOH solvent is in the range of 3.25 ppm to 77.67 ppm. The highest value is produced by the hydrolysis of cellulose to the sulfuric acid concentration of 0.4% v/v, and the KOH solvent ratio is 1:10. The higher the ratio, the lower the KOH solvent content of glucose produced.

#### 1. Introduction

Indonesia is an agricultural country which has advantages in terms of waste biomass. Biomass is one of the alternative energy sources has the potential to replace fossil energy are dwindling because its existence is very abundant in Indonesia. In addition to forest biomass, biomass can also be obtained from industrial waste. One example of industries that produce waste biomass, i.e., wood pulp which is used as raw material and produce bark as industrial waste. Waste bark of Acacia mangium is lignocellulosic biomass that can be reused as raw material for alternative energy, such as glucose to be used as bio-ethanol or gas products through a thermal process that can be converted into biofuels and others. Carbohydrate polymers contained in the biomass can be converted into simple sugars through a

process of hydrolysis. Hydrolysis is the process of solving the polysaccharides in this case cellulose and hemicellulose with an acid solution into its constituent sugar monomers. Because the acacia bark waste has a high enough lignocellulose compounds such as lignin, hemicellulose and cellulose are hydrolyzed need to do so before delignification process.

### 2. The Process of delignification

Delignification is a process of changing the chemical structure of biomass berlignoselulosa by removing lignin by selectively from hemicellulose and cellulose, causing the decomposition of chemical bonds both covalent bonds, hydrogen bonds or bond van der Waals with components of other chemicals in the ingredients berlignoselulosa (cellulose and hemicellulose) and cultivated other components remain intact. Because lignin is degrading cellulose which has a high crystalline structure that is more resistant to hydrolysis than the hemicellulose. Delignification process can be done in the heat (thermal), chemical and biological. Chemical delignification typically using solvents of acids, bases, acid-base combinations [1], Alkaline-glycerol, alkaline-alcohols and organic solvents [2]. If lignin is separated thereby, substrate cellulose and hemicellulose are left will be more accessible to degrading enzymes including enzyme hydrolysis. The resulting solid cellulose can then be used for production into glucose by acid hydrolysis and enzyme process [3].

Delignification of wood and agricultural materials chemically with an alkali solution of glycerol or glycerol in an aqueous or aqueous media, in an autoclave or atmospheric pressure, for pulp production. According to the reaction temperature increases from 438 to 498 Kwill increase the loss of lignin from 55.5 into 88.0%, but lower revenues remaining cellulose from 91.8 to 82.3%, while using 10% sodium hydroxide catalyst for 8 hours of reaction time for Ailanthus wood chips in the process of non- catalytic lignin loss from 47.9 to 76.8%, while 96.2 to 93.3% for cellulose, in a range of temperature and reaction time, are the same.

Process of delignification chips wood Ailanthus altissima and Spruce orientalis using solvents, which are similar to those performed by Ayhan Demirba 1998 ie using glycerol and alkaline-glycerol as solvent with or without a catalyst at a temperature varying ie (438, 458, 478, and 498 K) at atmospheric pressure and the results were compared with other organic solvents, release of lignin percentage rose from 15.2 to 19.0% when the concentration of NaOH catalyst is increased, while the temperature of the process does not significantly affect delignification products.

An important parameter in the process of delignification of wood or biomass using an organic solvent is the ability of a solvent to develop the structure of the wood. When a polar solvent is absorbed by the wood, bound polar solvent hydrogen bonding. Timber development push opens the wooden structure and freedom so that the lignin fibers apart.

Delignification process with acid solvents. These processes are often operated without the addition of a catalyst. In such a case, acetic acid is released from wood during the pulping provide the necessary acidity, and the reaction temperature in the range 185-200°C. Acid catalyst can be used to lower the temperature and pressure of the reactor. Organic acids, such as acetic, oxalic acid and salicylic acid, as well as accelerate the delignification, but the process is not specifically explain the advantages of using a catalyst. Some salt of alkaline earth metals such as calcium and magnesium chloride is an effective catalyst for delignification wood and lignocellulosic materials with a high content of methanol and ethanol. The resulting yield ranged between 54 to 57% for softwood.

The process of alkaline organic solvents. Methanol, ethanol, etc. Used as a solvent with a base catalyst such as NaOH,  $Na_2CO_3$ ,  $NH_3$ , and amines. The surplus compared with the delignification alkalimethanol single stage is due to the fact that the first stage removes most of the hemicellulose, which is in the process one stage consumes most of the charge of sodium hydroxide.

Oxygen-alkali delignification can reduce the use of sulfur and eliminate the use of chlorine. Delignification with aqueous-organic solvent effectively separated fractions lignocellulose. Hemicellulose dissolved in the aqueous phase, lignin is soluble in the organic phase, and cellulose remains in solid form. Advantages organosolv process is high-yield products, solvents, and lignin easy to be recovered, it can be processed into glucose [4].

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Lignin in the organic phase will undergo condensation reactions, and polymerization makes its solubility decreases. At high temperatures the organic solvent reduces the surface tension of alcohol, thus encouraging penetration into the chip alkali and lignin breakdown products of wood diffused into alcohol so that the distribution will be uniform reagent in the timber. At the same time, alcohol also degrades lignin before condensed at a high temperature.

Acacia mangium trees have brown wood color pale to dark brown, plain or striped dark and light patterns, smooth to a slightly rough texture, the wood somewhat hard to hard. Mangium acacia wood used as raw material for the manufacture of pulp, paper, particle board, crates, and others. Paper fibers used to make packaging materials of paper, cardboard and as a staple for taking the paper industry. Acacia trees could improve the ecological balance so that it can improve soil structure, especially for vulnerable areas such as hills and mountains. Benefits of acacia wood taken from the acacia tree are very broad. Acacia wood is widely used for making various types of furniture. But the extraction of natural resources such as timber must also consider the environmental balance.

The process of re-planting acacia trees is necessary to maintain the environmental balance. Thus, the acacia tree replanting is necessary to maintain the natural decay even though the development of an acacia tree can be done quickly. Acacia wood calorific value of about 4800-4900 kcal/kg so that the wood can also be used as firewood and charcoal. The leaves can be used as animal feed. Branches and dried leaves are falling can be used for fuel. The composition and components from acacia wood consist of lignin 24.89%, 43.85% cellulose, pentosan 17.87% 0.99% silica and 0.25% ash. Research on the production of glucose from acacia bark waste has not been conducted, but the manufacture of activated carbon from acacia wood using a solvent  $H_3PO_4$  and  $ZnCl_2$  reach 1038.77 ± 4:48 m2 / g and 957.47 ± 7:39 m2 / g. Acacia mangium can also be used as raw material for the manufacture of liquid smoke [6].

#### 2.1. Hydrolysis Process

Hydrolysis is a continuation of the pretreatment delignification process that will transform cellulose into glucose, These changes include the process of solving the polysaccharides in lignocellulosic biomass, namely cellulose and hemicellulose into their constituent sugar monomers. Complete hydrolysis occurs when cellulose produces glucose, while hemicellulose produces several monomer pentose sugars (C5) and hexoses (C6) [7]. Hydrolysis can be done chemically (acid) or enzymatically. Enzymatic hydrolysis has several advantages over acid hydrolysis, among other things does not happen sugar hydrolysis degradation, lower process conditions (low temperature), potentially high yields and the cost of maintenance of equipment are relatively low because of no corrosive materials. Some of the disadvantages of the enzymatic hydrolysis, among others require more time, and the enzyme is inhibited by the product. On the other hand the price of enzymes currently more expensive than sulfuric acid, however, development continues to be done to lower the cost and improve the efficiency of hydrolysis and fermentation.

The conversion of cellulose to glucose, this phase continues to be developed either by acid hydrolysis for cellulose fibers are very rigid and difficult to be broken down into glucose, The acid used is acetic acid, phosphoric acid, hydrochloric acid, and sulfuric acid. The rate of hydrolysis process will be increased by a high acid concentration. In addition, to increase the rate of hydrolysis process, a high acid concentration will also lead ions bound controls such as SiO, phosphate, and salts such as Ca, Mg, Na, and K in starch.

Biomass fibers consisting of components soluble fiber and insoluble. Examples of fibers which are insoluble in water are cellulose, hemicellulose, and lignin, whereas that of water-soluble pectin, gum, some hemicellulose, glucans, and mucilase. Cellulose is a long polymer  $(C_6H_{10}O_5)n$  formed by more 100-2000 D-glucose molecules with a bond of 1,4- $\beta$ -glycosidic. Abundant cellulose in plant cell walls. Cellulose is never found in a pure state in nature, but it is always associated with other polysaccharides such as lignin, pectin, hemicellulose, and xylan. Cellulose fibers form a component of plant cell walls. Most cellulose associated with lignin so often referred to as lignocellulose. Cellulose has the chemical formula ( $C_6H_{10}O_5$ ) and a molecular weight of 162,

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In general, cellulose insulation is done through an extraction process using an alkaline solution. Cellulose is the major component of wood that can be hydrolyzed by the enzyme into glucose which can then be processed further as fermented which will produce alcohol. To hydrolyze cellulose in the lignocellulose will be much more difficult compared to hydrolyze cellulose-free because the lignocellulose is a material very tightly so that in normal conditions would be inert and can not be penetrated or intruded by water especially enzymes.

Therefore, it required an initial process (*pretreatment*) To prepare the material to be disacarification by enzymes and fermented by microorganisms which have been free from lignin and hemicellulose. Without pretreatment, sugar resulting from the hydrolysis of less than  $\pm$  20% while with pretreatment results will increase to  $\pm$  90%. To increase the surface area of the lignocellulosic do the downsizing (size reduction) as a first step of processing the material. In many processes used lignocellulosic materials measuring  $\leq$  3 mm. This pretreatment success is determined by the amount of lignin and hemicellulose are missing from biomass materials.

Methods hydrolysis with a weak acid will remove lignin, but weak hydrolysis of cellulose will result in a strong acid at the time of high temperature. With a background process, benefits and weaknesses of the studies that have been conducted by researchers above, this study tries see how the relationship between the delignification process using NaOH and KOH solvent to changing levels of biomass waste lignocellulose acacia bark (LKKA) and the influence of sulfuric acid concentration on the hydrolysis of the glucose produced.

## 3. Experiments

Waste bark of acacia (LKKA) derived from Pulp and Paper Mill Headland Enim Palembang South Sumatra Indonesia, NaOH and KOH as a solvent (Merck) and to the hydrolysis process used  $H_2SO_4$  (Merck). LKKA cut into small pieces to a size of 1 cm x 1 cm and then the chips dried at a temperature of 75°C and in crushing into powder and sieved and can be the size of about 35 mesh.

#### 3.1. The process of delignification

- a) Bark powder samples *Acacia mangium* 30 grams put in a 500-ml Erlenmeyer flask
- b) NaOH solution with a concentration of 5% (w / v) was added to the solid and liquid ratio of 1: 6, 1: 8, 1:10, and 1:12
- c) The mixture was homogenized at a temperature of 85oC for 60 minutes with a stirring speed of 500 rpm.
- d) Samples were put into the autoclave for 30 minutes at a temperature of 121°C and atmospheric pressure.
- e) Samples were filtered and washed to a neutral pH by using distilled water.
- f) The residue was dried in an oven at 100 °C for 60 minutes.
- g) Step a) s / df) was repeated using KOH solution with a concentration of 5% (w / v) with solid and liquid ratio of 1: 6, 1: 8, 1:10, and 1:12
- h) The residue was further analyzed by Chesson-Data.

#### 3.2. Hydrolysis Process Chemicals

- a) Residue delignification 5 grams inserted into erlenmeyer flask and added with 50 mL  $H_2SO_4$  solution with varying concentrations of 0.1%;0.25%;0.4%;and 0.55% (% v/v).
- b) Samples were homogenized at 100°C for 60 minutes with a stirring speed of 500 rpm.
- c) Hydrolysis was filtered by using a vacuum pump to separate the residue and the hydrolyzate.
- d) The hydrolyzate is cooled and then analyzed the content of glucose using a spectrophotometer.

The variables of the research are the use of types of bases in the pretreatment stage, the ratio of solid and liquid during the delignification process, and the concentration of sulfuric acid hydrolysis process chemically.

#### 4. Result and Discussion

The study was conducted by two (2) phases namely biomass delignification process acacia bark waste (LKKA) and hydrolysis to produce glucose.

Processed acacia bark waste delignification carried out using alkaline solvents are strong bases NaOH

and KOH alkaline weak. Deliginifikasi process acacia bark waste (LKKA) using NaOH and KOH solvent is intended to break the bonds of lignin compounds in the waste bark of acacia (LKKA), for crosslinking of the aromatic structure of lignin can slow the process of hydrolysis to produce glucose. It also can reduce levels of hemicellulose and cellulose fiber content of the biomass increase. According [8] NaOH solution can attack and damage the structure of lignin in the crystalline and amorphous and partially separating the hemicellulose. OH- ions of NaOH will break the bonds of the basic structure of lignin while Na + ions will bind with lignin to form sodium phenolics. These salts are soluble phenolics. Lignin dissolved marked with black in a solution called black liquor (black liquor). Bases alkali KOH has a reaction or higher than the base strength solution of NaOH and NH<sub>4</sub>OH (NH<sub>4</sub>OH pKb> NaOH pKb> pKb KOH). PKb value influences the strength of a base, the smaller the pKb value greater base strength of the solution. The stronger the alkaline nature of the solution the more powerful the reaction and the more lignin and hemicellulose biodegradable. PKb value influences the strength of a base, the smaller the pKb value greater base strength of the solution. The stronger the alkaline nature of the solution the more powerful the reaction and the more lignin and hemicellulose biodegradable. PKb value influences the strength of a base, the smaller the pKb value greater base strength of the solution. The stronger the alkaline nature of the solution the more powerful the reaction and the more lignin and hemicellulose biodegradable.

After the last chemical process followed by a thermal process, the heating process LKKA has been dissolved in a solvent base in an autoclave at a temperature of 121 ° C and atmospheric pressure. The temperature is expected in the dissolved lignin, hemicellulose cellulose down while riding. According to when the temperature is raised from temperature autoclave  $438^{\circ}$ K to  $498^{\circ}$ K. Loss of lignin increased from 55.5 to 88.0%, but lower the cellulose content of 91.8 to 82.3%.

The delignification carried out by means of chemical, physical and biological substrates will make cellulose and hemicellulose are left will be more accessible and inclusive degradative enzyme hydrolysis so that the glucose produced more. In addition one of the factors that influence the delignification process is the sample size because the sample size can decide the long polymer chains into shorter polymer chains to facilitate the separation of lignin from the cellulose.

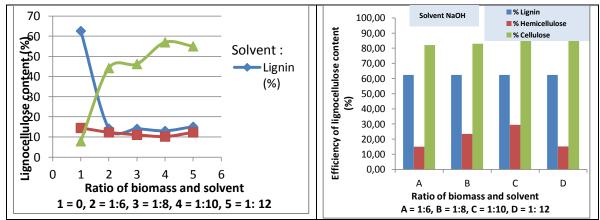
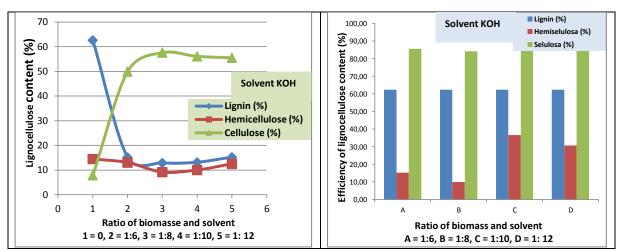


Figure 1. (a, b) Biomass Ratio and Solvent Effect of NaOH on Content Changes Efficiency Lignocellulose

Delignification process using NaOH and KOH solvent make lignin levels dropped quickly, while the hemicellulose content down for biomass and solvent ratio of 1: 6 and 1: 8, after stagnating at the moment is increased and vice versa cellulose ratio rose quite sharply. Lignin removal efficiency using solvents NaOH and KOH (Figure 31b and 32b) is almost the same, namely to 62.39%. Decrease hemicellulose using KOH solvent (36%) are more efficient than solvent only 29.3% NaOH. Efficiency increased levels of cellulose using NaOH or KOH solvent almost equal approximately 85.5%, but with KOH larger atomic mass gives rise to a more stable.



**Figure 2. (a, b)** Biomass Ratio and Solvent Effect of KOH on The content of Lignocellulose efficiency in delignification process LKKA

Decreased lignin solvent greatest when using NaOH occurs at a 1:10 ratio (S: L) 13.03%, while for the KOH solvent at a ratio of 1: 8 (S: L) lignin originally 62.61% without pretreatment degraded changed be 12.91%, it seems that KOH at smaller concentrations was able to lower the lignin content compared with NaOH, KOH because of OH- ions break the bonds of the basic structure of lignin while K + ions will bind to form a potassium phenolic lignin [10].

Salt is soluble phenolics, lignin dissolved marked with black on the so-called black liquor solution (*black liquor*). In addition, the decrease in the levels of lignin and hemicellulose in KOH is better because OH<sup>-</sup> ions in KOH are more easily released and prove that bases with larger atomic masses are stronger and easier to reduce lignin. KOH is more reactive than NaOH because of the periodic table is getting down his increasingly strong bases as the distance between atoms in the metal atom with the OH farther away, it causes the OH ions more easily released and easy to react with other compounds. The addition of NaOH and KOH solution (strong bases) on acacia bark can be spread energy through ionic conduction which causes heating.

This heating process resulting in the content of hemicellulose which binds cellulose can be detached, and lignin in the cell wall that prevents the cellulose began to fall. Then the liquid ratio of 1:10 to 1:12 cellulose content tends to decrease for the second solvent. Impairment of cellulose is thought to occur because of the structure of cellulose are regularly open and freely cellulose molecules dispersed in a solution of NaOH and KOH. With the structure of cellulose dispersed freely into the solution of NaOH and KOH, cellulose expected to participate dissolved by NaOH and KOH when the screening process. Hemicellulose has an amorphous structure so that the use of NaOH and KOH can eliminate lignin simultaneously extracting hemicellulose. In this delignification process using NaOH and KOH solution because the solution can damage the structure of lignin in the crystalline and amorphous and separate hemicellulose portion has an amorphous structure, so the use of strong bases can eliminate lignin simultaneously extracting hemicellulose[8]. Based on the pictures 3.1a and 3.2a, the influence of the ratio solid and liquid give effect to reduced levels of hemicellulose from 14.49% to 11.1% change in the ratio of 1:10 NaOH (solid: liquid). And for the hemicellulose content of 14.49% KOH turn out to be 9.18%.

Decreased levels of hemicellulose after treatment delignification indicated that delignification also have an impact on the depolymerization of hemicellulose, it is because hemicellulose is a carbohydrate compound constituent fuel cells with lignocellulose but with a degree of depolymerization that is lower compared to cellulose, consequently hemicellulose are particularly vulnerable to high temperatures, acids, and bases compared with cellulose [9].

Solid and liquid at a ratio of 1: 6, 1:10, and 1:12 there was an increase compared with the hemicellulose content at a ratio of 1: 8 for KOH and 1: 6, 1: 8, 1:12 hemicellulose increased compared to a 1:10 ratio for NaOH. This occurs due to the addition of NaOH and KOH in each of the samples

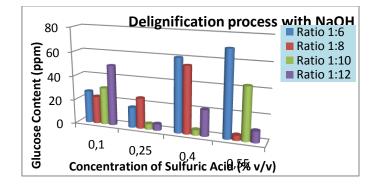
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resulted in the level of solubility of carbohydrates slower than the oxidation or reaction with other compounds so that the carbohydrate content in the media decreases. When the solubility of carbohydrate slows down, the oxidation reaction occurs to form more complex compounds and eventually accumulate to a hemicellulose content rises.

Decreased levels of lignin are also affected by pH, lignin will be dissolved at a high pH that is in the black liquor as lignin phenolic hydroxyl groups are ionized to form salts and polar. Such treatment will break down lignin into smaller particles.

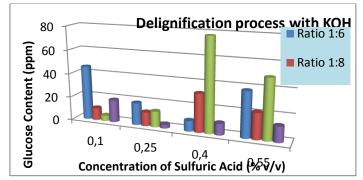
Cellulose is expected to result in the presence of the delignification process, the cellulose obtained results will increase. Because with the previous delignification process could make less lignin and cellulose easily hydrolyzed. Cellulose is a component that dominates carbohydrates derived from plants close to 50% because cellulose is the main component of the structural elements and the most important part of the cell walls of plants. In figure 3.1 and 3.2. seen that the influence of the ratio of solid and liquid, subsequent to pretreatment (delignification), cellulose content tends to increase subsequent to the pretreatment. In the solid and liquid KOH ratio of 1: 8 produces the highest cellulose content of 57.62%, whereas for the NaOH at a 1:10 ratio produces the highest cellulose content of 56, 98%. It is clear that the process of solving the structure of lignin and hemicellulose is able to result in an increased amount of free cellulose contained in the biomass.

4.2. Effect of H<sub>2</sub>SO<sub>4</sub> concentration on Glucose Production, delignification with NaOH



**Figure 3.** Effect of Glucose Concentration on Production H<sub>2</sub>SO<sub>4</sub> In the process of hydrolysis, delignification with NaOH Solvent

Solvent delignification process with NaOH provide large enough glucose content at a ratio of 1: 6 and 1: 8 with sulfuric acid concentration in the range of 0.1 to 0.4 and 0.5% v/v. Increased glucose content in the solvent of NaOH at a ratio of 1: 6 reached 69.88 ppm for sulfuric acid concentration of 0.55% v/v. NaOH to solvent ratio of 1: 8 glucose produced greater when greater concentration of sulfuric acid, but then declined rapidly when the concentration of sulfates acid plus. The greater the ratio of solvent NaOH the less glucose is produced. However, when compared with KOH NaOH needs less solvent, which means that absorption by NaOH lignin and cellulose by NaOH liberation is more effective than KOH solvent so that the higher the rate of hydrolysis.



**Figure 4.** Effect of Glucose Concentration on Production H<sub>2</sub>SO<sub>4</sub> In the process of hydrolysis, delignification with KOH Solvent

Contents degradation of cellulose to glucose results delignification process using KOH solvent is in the range of 3.25 ppm to 77.67 ppm. The highest value is produced by the hydrolysis of cellulose to the sulfuric acid concentration of 0.4% v / v, and the KOH solvent ratio is 1:10. The higher the ratio, the lower the KOH solvent content of glucose produced. Clearly visible on the graph that the concentration of sulfuric acid plays a major role in the chain hydrolyze cellulose to glucose with a concentration range from 0.1 up to 0.4% v / v. The addition of sulfuric acid at low concentrations will improve the quality of sugar because the H + ions in strong acid glycosides can break bonds contained in the cellulose. Increasing the amount of glucose produced due to the addition of sulfuric acid as a catalyst concentration will lead to collisions between the water molecules and the molecules of starch more quickly and more. As a result of the collision, the activation energy of the reaction will decrease the faster the reaction rate. While the lowest value was found by the solid and liquid ratio of 1: 8 with 0.55% acid concentration is 4.4215 ppm. Glucose lowering due to the number of OH- ions from the water less and less. Increased concentration of acid in the hydrolysis process results in glucose and other sugars to be more degraded form hydroxy methyl furfural (HMF) and furfural that eventually they form formic acid [7].

#### 5. Conclusion

From the research that has been conducted concluded that:

The process of delignification waste acacia bark can be made using solvents NaOH and KOH, wherein KOH is more reactive than NaOH as the atomic mass of KOH greater than NaOH it causes more easily ion OH released and easy to react with other compounds and easy to break down the structure lignin and hemicellulose. Besides the solid and liquid solvents affect the delignification process where the greater the ratio, the greater the efficiency of reduction of lignin and hemicellulose, as well as higher levels of cellulose, but the maximum ratio is 1: 8 to 1:10 for the KOH and NaOH.

The content of the degradation of cellulose to glucose results delignification process using KOH solvent is in the range of 3.25 ppm to 77.67 ppm. The highest value is produced by the hydrolysis of cellulose to the sulfuric acid concentration of 0.4% v / v, and the KOH solvent ratio is 1:10. The higher the ratio the lower the KOH solvent content of glucose produced, but is clearly visible on the graph that the concentration of sulfuric acid plays a major role in the chain hydrolyze cellulose to glucose with a concentration range from 0.1 up to 0.4% v / v. Solid and liquid ratio of 1:10 to KOH with a concentration of  $0.4\% \text{ H}_2\text{SO}_4$  and 1: 6 for NaOH with H<sub>2</sub>SO<sub>4</sub> concentration of 0.55% (v / v) glucose to produce the best product in the hydrolysis process chemical waste acacia bark.

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