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Effect of Particle Size on the Modification of a Two-Stage Dilute Acid Hydrolysis Process of Biomass

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Abstract

In this work the influence of milling on clean depithed sugar cane bagasse (as a pretreatment step) on two stage acid hydrolysis was evaluated. Two different depithed bagasse samples were studied, very fine powder that passed mesh No 325 and coarse bagasse without milling (3 cm fiber bundle length). The effect of bagasse particle size on parameters was studied as well as hydrolysis of the two particle sizes were carried out. In the present study, the bagasse material was analyzed by scanning and transmission electron microscopy (SEM and TEM) which confirmed that the particle size after the milling process was decreased to the nanometer scale form (20-50nm) that catalyzed the hydrolysis process. Hydrolysis time, temperature and liquid to solid ratio were kept constant at 30 min, 100 ^{o}C and 10:1 respectively, while concentrations of sulfuric acid were varied. In the first stage the concentration of acid ranged between 2% to 10% (wt/wt) and in the second stage it ranged between 10% to 30% (wt/wt). The results revealed that on using fine powder bagasse, in the first acid hydrolysis stage, the total dissolved sugars were 2 to 1.3 times more than that on using coarse bagasse, based on acid concentrations. Concerning concentration of xylose in the hydrolyzed solution, increasing acid concentration from 2% to 10%, on using coarse bagasse, xylose concentration was increased from 4 to 9.9 mg/L while on using fine powder bagasse it increased from 7.5 to 12.9 mg /ml. In case of second stage, on using fine powder bagasse the total dissolved sugars were 1.7 times more than on using coarse bagasse. Concerning concentration of xylose in the hydrolyzed solution, on increasing the acid concentration from 10% to 30%, the increase was from 0.7 to 1.8 mg / ml on using coarse bagasse, while on using fine powder bagasse the increase was from 1 to 2.3 mg /ml. The higher efficiency of fine powder bagasse may be attributed to that; the mechanical action can promote degradation and damage of lignocellulose internal structure and destroy the crystalline structure of cellulose, in addition generation of higher surface area of the biomass particles. Such actions increase accessibility and mass transfer of acid which lead to higher hydrolysis of hemicellulose and cellulose.

Keywords: biomass, hydrolysis, dilute acid hydrolysis, sulfuric acid, mechanical refining and milling.

1. Introduction

Last decade, the conversion of biomass to soluble sugar has a great attention due to largely demand of biofuel and also to decrease the pollution arising from biomass burning. Biomass such as bagasse, corn Stover, rice straw and wheat straw harvested every year with large quantities, burning those biomasses increase global warming and the environmental pollution. They caused many diseases to the human and all living organisms. They are considered as renewable source of energy due to abundant as a raw material for biomass conversion to biofuel. Wyman [1] stated that the conversion of lignocellulosic biomass to biofuels improve energy security and reducing greenhouse emissions. Bagasse, corn Stover, rice straw and wheat straw biomass contain nearly the same components with small differences in chemical composition. Lignocellulose structural material composed mainly of cellulose, hemicellulose, lignin, pectin with small percent of extractives and ash [2]. Chemical composition of biomass differs from plant to plant with small changes in the component percentage. Celluloses and hemicelluloses are structural carbohydrates as they form the bulk of the plant cell's

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supporting structure [3]. Cellulose is the primary component in the plant body builder, accounting for around 40% of the total biomass component.

It is made up primarily of crystalline glucose units that are linked together with beta 1, 4 linkages to produce a strong polymer structure [4]. The beta 1, 4 bond between glucose units strengthens cellulose, makes it more difficult to hydrolyze, and necessitates more energy to completely hydrolyze the cellulose component to soluble glucose units. While, in lignocellulosic materials, about 50–90 percent of the cellulose is bonded laterally by hydrogen bonds and forms a crystalline structure [5]. Hemicellulose is mostly composed of xylose, with minor amounts of arabinose, glucose, and galactose accounting for around 30% of the biomass component, with the primary component being xylose units that are attached to one another. Xylose is the weakest component in the biomass structure, and it quickly hydrolyzes to soluble sugar in the first stage of acid hydrolysis [6].

According to Yulia [7], hemicellulose is the weakest component in the biomass composition to be hydrolyzed. Moreover, Lignin acts as a cement between the cellulose and hemicellulose chains, causing the biomass structure to become too inflexible to withstand environmental changes [8].It makes up around a quarter of the overall biomass content. Lignin is an extremely complicated structure that is made up mostly of phenylpropane units connected in a three-dimensional structure [9].

To convert biomass to soluble sugar, a variety of techniques are utilized, including two-stage dilute acid hydrolysis, two-stage concentrated acid hydrolysis, liquid hot water, and enzymatic hydrolysis [10]. The concentrated acid hydrolysis process uses a substantial amount of acid and necessitates the use of resistance equipment for acid recovery. While biomass hydrolysis with low to moderate dissolved sugar yield requires heat more than 200 ^{o}C in the liquid hot water hydrolysis process. [11]. The main objective of the biofuel hydrolysis process is to find a simple and easy way to convert biomass to a soluble sugar with the maximum increase in dissolved sugar yield. The two stage dilute acid hydrolysis process is the most widely used process for biomass hydrolysis and also consider the oldest process for converting biomass to a soluble sugar [12].
Due to the continual rise in biomass discharge, numerous improvements to the dilute acid hydrolysis process were proposed to maximize the dissolved sugar yield while reducing acid consumption [13]. The process entails contacting the biomass with dilute acid at a predetermined time and concentration. The main benefit of the two-stage dilute hydrolysis process is that there is no acid recovery required due to the low acid concentration. As a result, the residual acid can be neutralized with sodium hydroxide, sodium oxide, or sodium carbonate and the gypsum can be separated as a solid product after neutralization [14].

The two-stage dilute acid hydrolysis process has two stages: the first is to contact the biomass with dilute acid hydrolysis to dissolve the hemicellulose in the biomass to obtain soluble xylose sugar with small amounts of glucose, arabinose, and galactose dissolved sugar, and the second is to affect cellulose hydrolysis to obtain mainly dissolved glucose sugar [15]. Prehydrolysis is a restricted hydrolysis caused by dilute acids. The hemicellulose fraction is hydrolyzed, leaving the cellulose and lignin portions essentially unaltered [16].

The temperature employed throughout the process, time of the procedure, acid type, and acid concentration and liquid to solid ratio are all important elements in the two-stage dilute acid hydrolysis process. Low concentrations of acid used are useful in many ways; such as the recovered acid with low concentration can be easily concentrated to the same needed concentration. Low acid concentrations are advantageous in many ways, including the fact that recovered acid with a low concentration can be quickly concentrated to the required concentration. Acid loss will also be reduced because the amount of acid utilized will be reduced to zero.

Finally, because the acid content has been reduced to less than half, corrosion to the process equipment will be reduced. The aim of this work is to study effect of mechanical milling or refining of depithed bagasse on the efficiency of dilute acid hydrolysis to produce biofuel. Two stages dilute sulfuric acid hydrolysis will be carried out on using two different particle sizes of depithed bagasse. Because this point was not addressed in the literature for depithed bagasse, more research will be conducted, and the suggested mechanism will be explored.

2. Experimental

Bagasse (solid waste) is sourced from the Qena Paper Industry Company, which manufactures white paper from residual bagasse from the Qus Sugar Company in Upper Egypt. Bagasse was depithed multiple times using a depither and centrifuge, and then washed multiple times to remove dirt, unusual, and heavy solid contaminants. The depithed bagasse sample was left in a semi-closed dry area for 5 days to eliminate moisture until the bagasse moisture switched to air moisture. The moisture content of the sample was evaluated by drying it in a drier for 8 hours at 105 °C. The moisture content is determined by the weight loss before and after drying the sample. Moisture was measured several times, with an average moisture content of 6.0 to 6.5 % of the total biomass weight. The primary average value of ash samples up to 1.2 % was obtained

by taking a known weight of biomass sample and burning it in a furnace at 900 ºC for about 20 minutes.

Depithed dry bagasse samples containing 6.5 % moisture were refined in a TOSHIPA food processor, screened to various particle sizes, and the sample that passed screen mesh No 325 was maintained as fine powdered bagasse. The untreated samples with no refining, also known as coarse or untreated bagasse with a 3 cm length, and the refined bagasse or very fine powder bagasse passing through mesh No 325 are used (as seen in Fig.1). The bagasse was dried in air moisture, which aids in the refining process

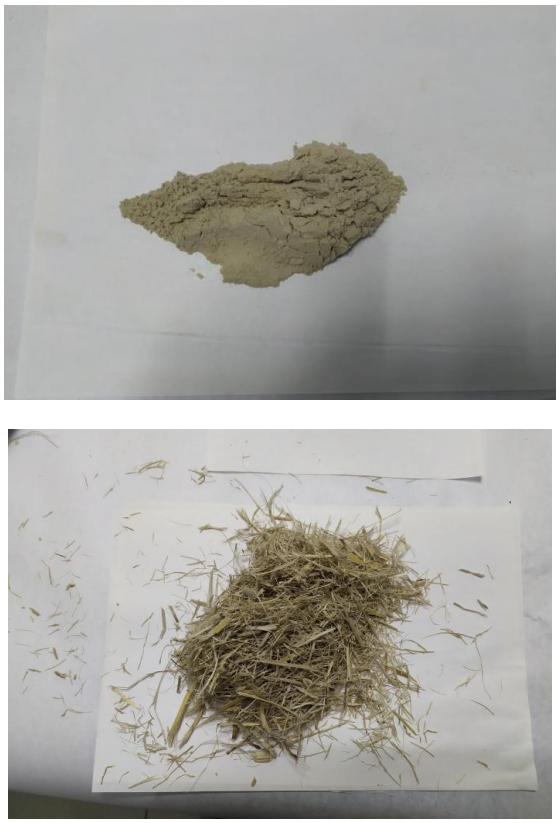

Fig. 1 Two bagasse samples: with milling, fine powder bagasse passed from mesh No 325 (left) and coarse bagasse fibre bundles without milling (right).

Mechanical milling (refining) was performed as a pre-treatment stage in this study. During the dilute sulfuric acid hydrolysis process, fine powder bagasse that passed screen 325 mesh number demonstrated to be more efficient than coarse bagasse without milling.

Mechanical milling may alter the physio-chemical characteristics of biomasses, increasing the chemical reactivity of lignocellulose and making it more accessible to the acid hydrolysis process, according to prior research [17-19]. Mechanical milling of biomass can proceed via three mechanisms, according to Yuan et al, 2016, [20], depending on the severity of milling power:

- Cutting fibres, resulting in a shorter fibre length.
- Shearing is a technique that fibrillates fibres on their outside surfaces.
- Compression, which causes internal fibrillation of fibres.

Following the National Renewable Energy Laboratory (NREL) procedure for determining the bagasse component, 0.3 g of biomass sample is reacted with 3 ml of concentrated sulfuric acid 72% (wt/wt) for about one hour, mixing the component every 5.0 minutes, diluting the concentrated acid concentration to 4.0 %, and boiling the slurry to 121ºC for one hour. The biomass component output was 37.0 to 39.2 percent cellulose, 24.2 to 25.6 % lignin composition, and 21.8 to 23 percent hemicellulose.

Soluble sugars were evaluated utilizing a Shodex SUGAR SC-LG column and a Shimadzu LC-10 with a system controller SCL-10A vp, Pump LC-10 AD vp, equipment refractive index detector RID-10A, Oven CTO-10A vp, and Degaser DGU-10A. At 80 °C, the flow rate is 0.7 ml/min, and the injection volume is 10 µL. Mobile phase 100% deionized water. All chemical reagents utilized in the paper company's laboratory were acquired from Alpha Chemical Laboratory. All chemicals were used are obtained with no further purification, and all chemicals were prepared weight by weight, as well as dry weight loss for all samples.

2.1 Characterization

The surface morphology of the fine powder bagasse was examined by JEOL-JSM-5500 LV scanning electron microscope and JEOL-JEM-1010 Transmission Electron Microscope (TEM).

3. Results and Discussion

3.1 Surface Characterization

3.1.1 SEM Study

Fig. 2 illustrated the surface morphology of fine powder bagasse after milling process; SEM images showed the morphology changes which indicated particle size distribution corresponding to milling process. The morphology of the resulting materials showed the reduction on fiber length after the milling may be responsible for catalyzing in the hydrolysis biomass.

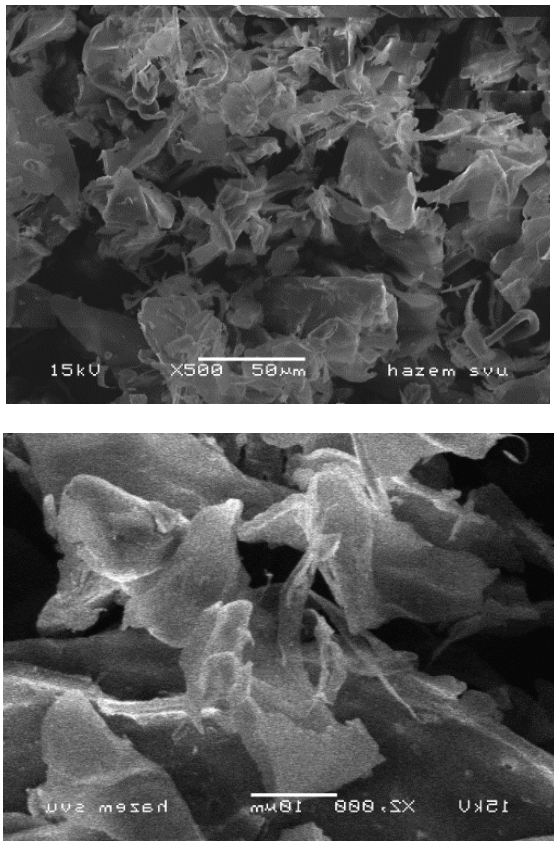

Fig. 2 SEM of fine powder bagasse after milling

3.1.2 TEM study

Transmission electron microscopy (TEM) has been used as a suitable method to determine the effect of milling on the bagasse structure as it increases the surface area via decreasing the particle size from 3 cm fibre bundle length to 20-50 nm as clear in Fig. 3. Large pores with distinct size and shape are observed, after milling, the particles are seen spread at the surface which catalyse the hydrolysis process. Meanwhile, it reduces bagasse particle size and thus increases surface area that promotes lignocellulose hydrolysis and decreases cellulose crystallinity.

Hydrolysis time, temperature, acid concentration, liquid to solid ratio, and particle size are all important parameters in the dilute acid hydrolysis of biomass. The relationship between variations in acid concentrations and two forms of biomass, namely extremely fine powder bagasse that passed through mesh no 325 and coarse bagasse with 3 cm fiber bundles length, was investigated in this research. To assure the major effect of acid concentration on particle size change, all other parameters such as time, temperature, and liquid to solid ratio are kept constant.

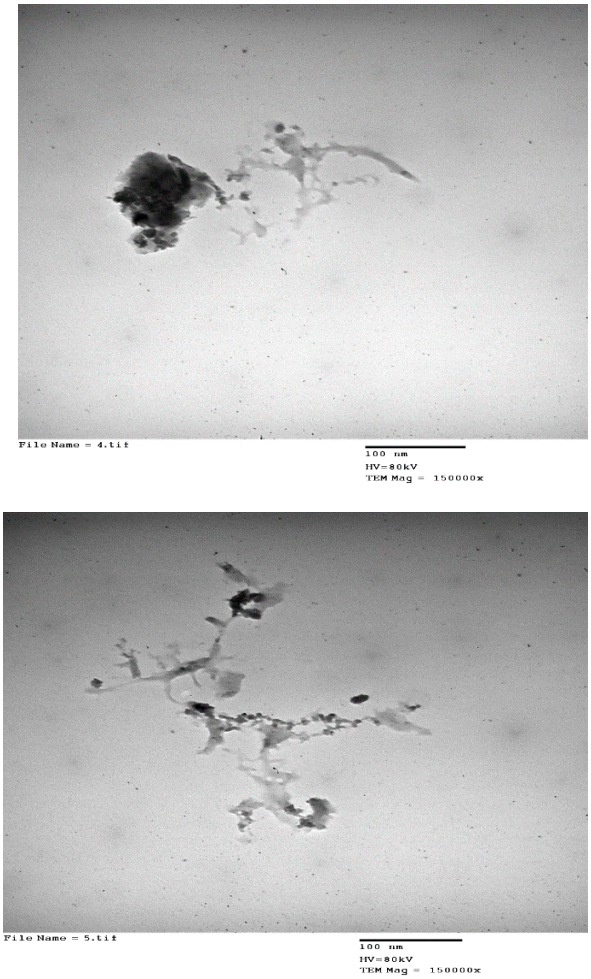

Fig. 3 TEM images of fine powdered bagasse.

3.2 Effect of hydrolysis time

Time is an important factor in the biomass hydrolysis process; the optimum time is required to obtain the highest dissolved sugar yield from the biomass while also avoiding the production of unfavourable materials such as furfural and hydroxy methyl furfural (HMF), which are produced from xylose and glucose degradation, respectively. Thus, as time increases in the first stage, xylose degradation increases to form furfural, while glucose degradation increases to form (HMF) in the second stage. As a result, the time for the first stage was kept constant throughout the experiments at 30 minutes to avoid xylose degradation to furfural, while the time for the second stage was kept constant at one hour because cellulose requires more power to affect hydrolysis and to avoid glucose hydrolysis to (HMF). [21].

3.3 Effect of hydrolysis temperature

According to our earlier investigations, sugar degradation is linked to the high temperature utilized in the hydrolysis process, and as the temperature rises, more soluble sugar is degraded to hazardous compounds, with soluble xylose degrading to furfural

and glucose degrading to (HMF). To avoid soluble sugar breakdown into hazardous components, the temperature was kept constant throughout the trials at 100 ºC.

3.4 Effect of liquid to solid ratio

The soluble sugar grew as the liquid to solid ratio increased, but the power required for acid recovery increased as well, necessitating the use of the optimal liquid to solid ratio to balance the optimum soluble sugar with acid recovery. To avoid any effect on the soluble sugar concentration, the liquid to solid ratio was kept constant throughout the trials at 10:1. [21].

4. Dilute acid hydrolysis process

4.1 1st stage dilute acid hydrolysis process

In this stage the fine powder bagasse and coarse bagasse reacted with different concentrations of dilute sulfuric acid (2, 4, 6, 8, and 10%) for 30 minutes in water bath with no pressure, liquid to solid ratio 10:1 and bagasse moisture 6.5%. The produced soluble monosaccharide is detected by high performance liquid chromatography (HPLC) such as xylose, arabinose, galactose, glucose.

Table 1: 1st stage dilute acid hydrolysis process: concentration of dissolved sugars at different acid Concentrations on using powder and coarse bagasse

Sample NO	Bagasse type	Acid concentration	Dry weight mg	Dissolved dry weight mg	Dissolved dry weight percentage	Xylose mg/ml	Arabinose mg/ml	Galactose mg/ml	Glucose mg/ml	Total dissolved sugars mg/ml	Acetic acid Ml
1	Fine powder	2%	935	229	24.5 %	7.8	1.7	1.5	0.9	11.9	0.003
2	coarse	2%	935	164	17.5 %	4.0	1	0.4	0.3	5.7	0.002
3	Fine powder	4%	935	270	28.8 %	9.7	2.6	2.3	1.3	15.9	0.03
4	coarse	4%	935	236	25.2 %	7.9	1.9	1.6	1.1	12.5	0.01
5	Fine powder	6%	935	312	33.4 %	12.3	2.8	2.6	1.7	19.4	0.12
6	coarse	6%	935	246	26.3 %	8.1	2.2	2.3	1.3	13.9	0.13
7	Fine powder	8%	935	320	34.2 %	12.5	2.9	2.9	1.9	20.2	0.18
8	coarse	8%	935	265	28.3 %	9.6	2.5	2.2	1.2	15.5	0.19
9	Fine powder	10%	935	330	35.3 %	12.9	3.1	3.0	2.3	21.3	0.21
10	coarse	10%	935	278	29.7 %	9.9	2.8	2.4	1.5	16.6	0.22

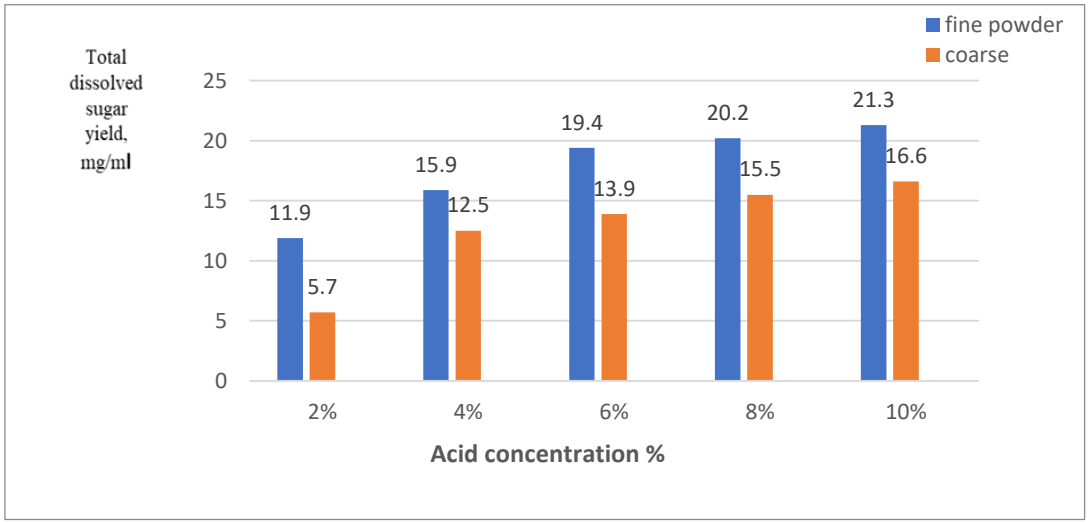

Fig. 4 1st stage process, dissolved sugar yield (mg/ml) at different acid concentrations for powder and coarse bagasse

Overall, the results in Table 1 showed that when acid concentrations increased, total dissolved sugar and all dissolved sugars (xylose, arabinose, galactose, and glucose) increased as well. Fine powdered bagasse yields more dissolved sugars in the hydrolyzed solution than coarse bagasse, according to the findings. It was discovered that fine powdered bagasse produces 2 to 1.3 times more total dissolved sugars than coarse bagasse Fig. 4. In terms of xylose concentration, when sulfuric acid concentrations were increased from 2% to 10%, xylose concentration in the hydrolyzed solution grew from 4 to 9.9 mg/ml when

coarse bagasse was used, while it increased from 7.5 to 12.9 mg/ml when fine powder bagasse was used Fig 5.

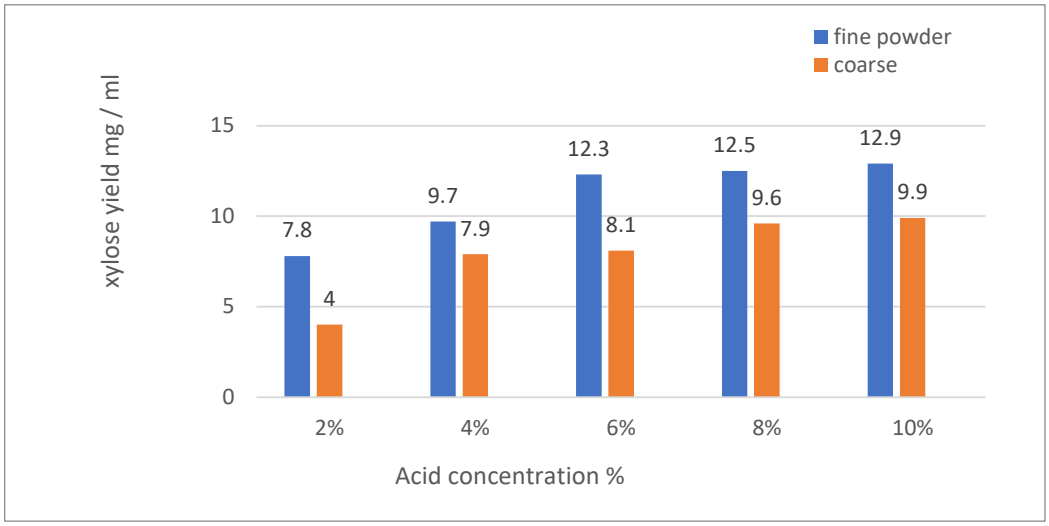

Fig.5, 1st stage dissolved xylose yield mg/ml of two bagasse type with different acid concentrations.

4.2 2nd stage dilute acid hydrolysis process.

In the second stage of dilute acid hydrolysis process the remaining solid from the 1st stage is dried to 4% moisture content and reacted with different concentrations of sulfuric acid (10, 20, 30 %) for both fine powder (passed from mesh No 325) and coarse one (fiber bundles of 3 cm), liquid to solid ratio and temperature are kept constant at 10:1 and 100 °C respectively.

Table 2: 2nd stage dilute acid hydrolysis process: concentrations of dissolved sugars with different acid Concentrations on using powder and coarse bagasse

Sample NO	Bagasse type	Acid concentration	Dry weigh t mg	Dissolved dry weight mg	Dissolved dry weight percentage	Xylose mg/ml	Arabinose mg/ml	Galactose mg/ml	Glucose mg/ml	Total dissolved sugars mg/ml	Acetic acid ml
1	Fine powder	10%	960	135	14.1 %	1.0	0.1	0.1	1.7	2.9	0.001
2	coarse	10%	960	120	12.5 %	0.7	0.1	0.1	0.8	1.7	0.001
3	Fine powder	20%	960	176	18.5 %	2.0	0.7	0.8	3	6.5	0.003
4	coarse	20%	960	145	15.1 %	1.2	0.3	0.2	2.1	3.8	0.001
5	Fine powder	30%	960	189	19.7 %	2.1	0.8	0.9	4.7	8.5	0.02
6	coarse	30%	960	165	17.2 %	1.8	0.6	0.7	2.7	5.8	0.03

Table 2 (2nd stage acid hydrolysis) follows the same trend as Table 1 (1st stage acid hydrolysis), in that as acid concentrations grew, so did the amount of total dissolved sugars and all sugars (xylose, arabinose, galactose, and glucose). It's also worth noting that powder bagasse is more efficient than coarse bagasse during acid hydrolysis. Fig. 6 demonstrates that when fine powder bagasse is used, total dissolved sugars are 1.7 times more than when coarse bagasse is used.

On raising the acid from 10% to 30%, the quantity of glucose (mg/ml) increased from 0.8 to 2.7 mg/ml when using coarse bagasse, whereas the amount of xylose increased from 1.7 to 4.7 mg/l when using powder bagasse Fig.7.

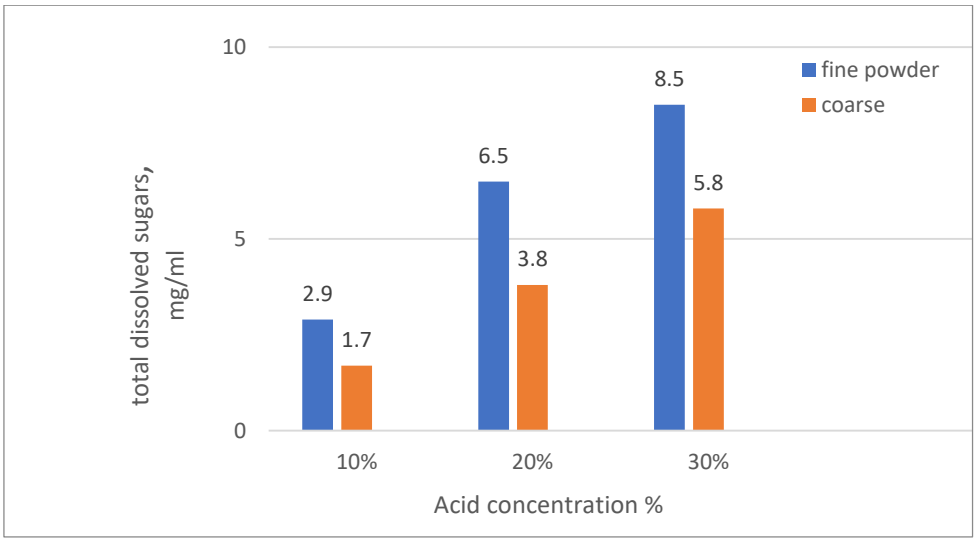

Fig.6. 2nd stage dilute acid hydrolysis process, total dissolved sugars (mg/ml) with different acid concentrations.

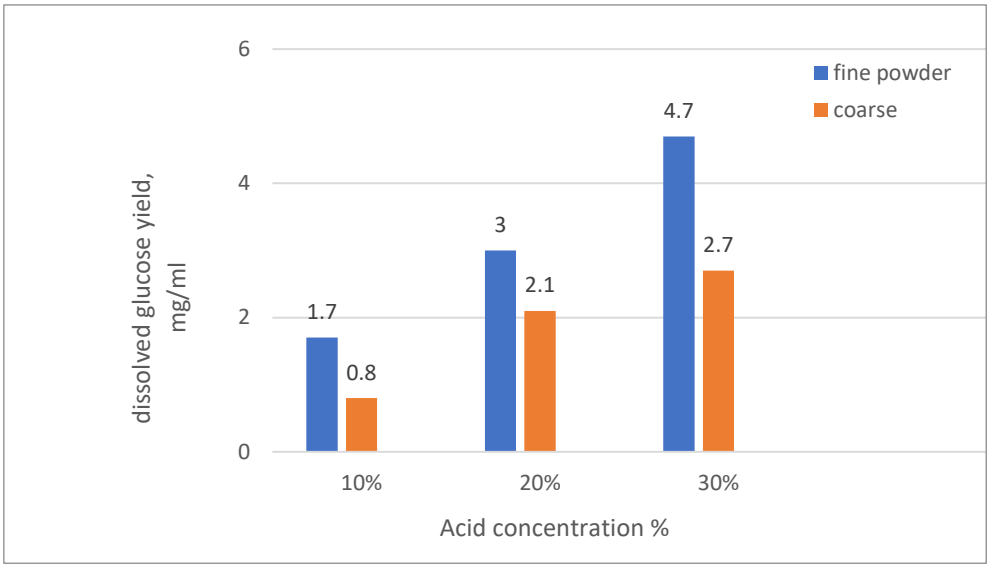

Fig.7 2nd stage dilute acid hydrolysis process, dissolved glucose yield (mg/ml) with different acid concentrations.

4.3 Hydrolysis mechanism for bagasse powder

At low mechanical energy, the first two mechanisms are dominant. Bagasse compact fibre bundles are split and fractured during these stages, resulting in small homogeneous fine particles. Milling and refining are therefore responsible for the generation of fine powder bagasse with a mesh of 325. This powder bagasse has a large surface area while also being potentially permeable. As a result of the higher acid penetration and improved mass transfer during the hydrolysis process, the acid hydrolysis activity increased. The last mechanism (compression) happened when increased milling energy was used, as in this study to generate fine powder bagasse (passes mesh No 325).

Mechanical milling may degrade lignocellulose and modify the structure and physico-chemical characteristics of cellulose at this stage. According to Tang 2005 [19], the crystalline structure of cellulose can be dramatically altered by mechanical activity. It causes dislocation and crystal deficiency. As a result, mechanical refining boosts lignocellulose chemical reactivity by breaking the lignin seal and altering the crystalline structure of cellulose; such actions are required to make cellulose more susceptible to glucose breakdown by dilute acid hydrolysis process. The total dissolved sugars were 1.4 times higher in this study when fine powder bagasse was used instead of coarse bagasse.

Furthermore, the hydrolyzed solution contained 60.3, 17.5, 14.7, and 14.1 mg/ml of xylose, glucose, arabinose, and galactose, compared to 43.2, 11.0, 11.4, and 9.9 mg/ml when coarse bagasse was used.

Conclusion

Bagasse milling was shown to be a very effective pretreatment step before the dilute acid hydrolysis stage. The following were the optimal circumstances for utilizing very fine bagasse powder (passing mesh No. 325): sulfuric acid concentrations ranging from 2% to 10%, while temperature, time, and liquid to solid ratio were all held constant at 100 °C, 30 minutes and 10:1 accordingly.

In the first stage, the amount of dissolved total sugars was about 2 times more than when coarse bagasse was used and 1.7 times higher in the second stage. Xylose, glucose, arabinose, and galactose concentrations were 60.3 mg/ml, 17.5 mg/ml, 14.7 mg/ml, and 14.1 mg/ml, respectively, compared to 43.2 mg/ml, 11.0 mg/ml, 11.4 mg/ml, and 9.9 mg/ml when coarse bagasse was used.

Mechanical power is responsible for the improved efficiency of fine powder bagasse. The particle size after the milling process was decreased to the nanometer scale form (20-50 nm) that catalyzed the hydrolysis process. Meanwhile, it reduces bagasse particle size and thus increases surface area which promotes lignocellulose hydrolysis and decreases cellulose crystallinity.

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