Effects of steam-treatment on the chemical component and thermal resistance of bagasse particle for natural-bonded composites product

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Abstract. The objective of this study was to evaluate the chemical composition of steam-treated bagasse particle by using autoclave. Fine size particle was obtained from the milling process of steam-treated bagasse. Before testing, the particle was pre-treated for 5, 10 and 15 minutes. The determination of extractives, lignin, α-cellulose, holocellulose and ash-content were conducted in accordance with the standard outline in the Technical Association Pulp and Paper Industry (TAPPI) test methods. Overall results show that the chemical properties have been diminished over time. The chemical properties with the highest percentage of extractive, lignin, ash and holocellulose contents (which was 3.9%, 39.15%, 1.45% and 73% respectively, was extracted at 121ºC for 5 minutes). The lowest percentage of extractive, lignin, ash and holocellulose contents (which was 0.8%, 25.30%, 0.88% and 51% respectively, was extracted at 121ºC for 15 minutes). It is concluded that longer steam treatment time (beyond 15 minutes) may degrade the chemical properties of bagasse. In addition, thermo-gravimetric analysis (TGA) was also carried out to investigate the thermal resistance of bagasse particle. Based on the results, the 10 minutes steamed samples have the highest peak degradation temperature, which was 286.8 ºC, while the 15 minutes steamed samples have the lowest peak degradation, which was 246.4 ºC. This shows that longer steaming period (more than 10 minutes) will decrease the thermal resistance of the bagasse particle.

1. Introduction
In the last decade, biomass sources such as sugarcane bagasse has attracted the interest of government and business entities because of their economic viability [1]. According to Basta et al. [2], the utilization of local agro-based wood products such as sugar-cane bagasse will add economic value and provides a potentially inexpensive alternative to the existing commercial artificial wood-panel mills.

In this research, sugarcane bagasse was used as the base material, to investigate the effects of steam process on its chemical component and thermal resistance. Sugarcane (Saccharum officinarum L.) bagasse is an agriculture waste that produced from sugarcane juice and sugar processing. According to Eggleston [3], the chemical composition of sugarcane is approximately 73 to 76% water, 10 to 16% soluble solids (primarily sucrose), and 11 to 16% (dry) fibers. Cane processing crushes the entire stalk to extract the sucrose, from which refined sugar is produced and the remains that contain the crushed rind and pith fibers
are called bagasse. As bagasse is a waste product, the practice of open burning and landfilling was usually done to remove this type of lignocellulosic biomass, thus may cause an unfavorable impact on the environment. Generally, bagasse contains about 65% fiber, 25% pith cell and 10% water soluble, and its chemical content is 55.2% cellulose, 16.8% hemicellulose and 25.3% lignin [4].

Normally, the sugarcane bagasse is used to produce the energy required during the process by the industries by burning it. This is done to cut the cost and lower the use of materials by using the waste materials. On the opposite, more valuable uses can be produced using this by-product rather than the direct energy generation through combustion [5]. The uses of bagasse have become a hot research topic around the world in the preparation of composites material [6-8]. In this research, bagasse was used to produce binderless board in order to cope with the issues regarding human health and the environment. Kenaf, coconut husk, wood waste and oil palm were among the popular materials used to produce this type of board. Lignin is among the important components that act as a binder between fibers for the production of binderless boards, while hemicellulose is not favourable as it absorbs moisture [9].

Usually, the first step of pretreatment is required to break down the lignocellulosic materials due to the association between three major components of the plant cell wall, which are cellulose, hemicellulose and lignin [10]. In this investigation, a steam-treatment technique for lignocellulosic materials (LCMs) was proposed due to its low costs, high efficiency and eco-friendly features [11]. This serve as an alternative technique to the common technique of using chemicals as a treatment method in which a higher cost is needed. Another interesting method was done by Nonaka et al. [12] where a high pressing temperature of 200 – 280℃ was applied to manufactured binderless particleboard from sugarcane bagasse. Steam explosion is one of the pretreatment methods for lignocellulosic materials that was widely used [13]. It both can be done with or without the presence of a catalyst (alkali or acid) [14]. Furthermore, it was also known that the use of steam explosion had positive effects on the properties of boards made from natural particles [15-17]. Materials with lower fiber density, improved strength properties, higher thermal stability and lower moisture absorption may be achieved by steam explosion, depending on the chemistry and distribution of the lignin component in the steam-treated fibers [18]. This treatment method of using steam at high temperature or liquid water was primarily operated by instigating the steam process within the biomass, thus systematically converts it into a structure that can be smoothly digested by enzymes [19]. Better properties of the resultant composites were achieved by treating sugarcane bagasse [20].

The ionization of water in such conditions will make the hemicellulose releases acetic acid, thus auto-catalyzes it. Hemicellulose of LCMs will undergo hydrolysis in which will ensure the increase of accessibility to cellulose and also circumvent the amassing of inhibitory by-products [21, 22]. The product obtained from the pre-treatment can be separated and filtered into two fractions, which are solid cellulose-enriched fraction and liquid soluble hemicellulose sugar. The presence of hemicellulose sugar in LCMs is detrimental to the production of particleboard as it absorbs moisture which leads to degradation of the fiber-matrix interfacial region, creating poor stress transfer efficiencies, thus as it will lower the mechanical properties and also dimension stability [23]. Therefore, the objective of this study was to investigate the effect of steam as a treatment technique on the chemical component and thermal resistance of bagasse particles for natural-bonded composites product.

2. Materials and methods

2.1 Preparation of raw materials for the steam treatment process

The sugarcane (Saccharum Officinarum) bagasse was used in this study. The bagasse was obtained from several vendors and places in Shah alam, Selangor, Malaysia. The bagasse was collected and air-dried for five days prior to the preparation of particles. After air dried, the bagasse was cut into fine small particles by using crusher and steamed at a constant temperature of 121℃ by using an autoclave machine. The time taken to steam the bagasse particles was set at different interval of 5, 10 and 15 minutes.
2.2 Chemical analysis

The major chemical compositions of the steam-treated bagasse particle was tested and determined. These included the extractive, lignin, α-cellulose, holocellulose and ash content. The chemical constituent of the bagasse was determined in accordance with the standard outlined in Technical Association of the Pulp and Paper Industry (TAPPI) test methods. The extractive content was determined and carried out in reference to the T2 04 cm-88 and T2 64 cm-88 methods [24]. The alcohol-toluene procedure followed the T204 cm-97 for 6 hours [25]. The lignin content of the sample was tested in reference following the T222 cm-88 method [25]. The α-cellulose and holocellulose was determined following the T203 cm-74 [24]. The ash content of the steam-treated bagasse was determined by following the procedure outlined in reference to the T211 cm-93 method [24].

2.2.1 Extractive content. The extractive contents were determined in reference to TAPPI T 204 om-88. The extracts were filled in a 250 ml flasks and was evaporated to dryness on a rotor vapor. The flask were then dried in an oven at 105°C for 1 hour, then was cooled in a desiccator and was weighed to the nearest 0.1 mg. The alcohol-toluene, the ethanol ‘washing solution’ and the extractives were determined together. The content of the hot water extractives was determined by using the same method individually.

2.2.2 Alpha-cellulose and holocellulose content. The specimen was placed inside a 300 mL beaker and 75 mL of 17.5% NaOH reagent was added into the beaker. The pulp was stirred with the apparatus until it was completely dispersed. After the dispersion of the pulp, the stirrer was rinsed with 25 mL of 17.5% NaOH reagent, to complete a 100 mL reagent added to the pulp. 30 minutes after the first addition of the NaOH reagent, 100 ml of distilled water was added to the pulp suspension and was stirred thoroughly with a rod. The beaker was then left for another 30 minutes, to further complete the total extraction time to around 60 minutes. After the extraction, the pulp was stirred and filtered through a filter funnel for further testing. The alpha-cellulose of the filtrate was determined pipetting 25 mL of the filtrate and 10 mL of 0.5N potassium dichromate solution into a 250 mL flask. While slowly swirling the flask, 50 mL of concentrated H₂SO₄ was carefully added. The solution was remained hot for a period of 15 minutes, before adding 50 mL of water. Two to four drops of Ferroin indicator was added and the solution was also titrated with 0.1N ferrous ammonium sulfate solution to a purple color. Finally, a blank titration was also done by substituting the pulp filtrate with 12.5 mL of 17.5% NaOH and 12.5 mL of water.

The holocellulose raw material was suspended in 150 ml of water at 75°C and the solution is stirred mechanically. 10 drops of glacial acetic acid and 1.5 g sodium chlorite was added into the reaction under vigorous stirring. After 1 hour the same amount of reagents was added and this process is repeated until the total reaction time was 4 hours. The solution is then cooled, filtered, and washed with distilled water until free of acid, then with acetone and left to dry. The holocellulose is then estimated gravimetrically.

2.2.3 Lignin content. The determination of the lignin content was done according to the standard set in TAPPI T 222 cm-02. The specimen was placed inside a beaker and was then added with 15 mL of cold (10 - 15°C) 72% sulfuric acid. The acid was gradually added in small increments while being stirred and macerated with a glass rod. To assure a complete dispersion of the material, the beaker will be covered with a watch glass and was kept in a water bath at 20±5°C for 2 hours. 300 to 400 mL of water was then added into a flask and the material from the beaker was transferred into the flask. 3% of concentrated sulfuric acid was then added to a total volume of 575 ml for the specimen to be rinsed and diluted. The solution was boiled for 4 hours by using a reflux condenser. After the boiling, the flask was kept in an inclined position to help the insoluble material (lignin) to settle. The settled insoluble material (lignin) was then transferred into a filter by using some hot water and also a glass rod. The lignin on the filter was washed with hot water and oven dried before it was weighed.

2.2.4 Ash content. The ash content was referred to T211 cm-93 method (Table 1). The following procedure is as follows; 2 g of cellulosic particles was weighed accurately in a crucible and heated over a Bunsen flame. After the particles had turn into a black residue, the sample was then placed inside a furnace
and heated at 575ºC for three hours. When the burning is completed and no black particles are present, the crucible was taken out of the furnace and cooled in a desiccator. The sample was then weighed for further analysis. For the cold-water soluble evaluation, 300 ml of distilled water was mixed with 2 g of particles. The mixture was stirred and left at ambient temperature for 24 hours. The residue was then filtered by using a filter paper and dried in an oven at 105ºC until its constant weight for the determination of cold water soluble. The hot water-soluble evaluation was done as follows; 100 ml of distilled water was mixed with 2 g of sample and left to digest for 3 hours. The aforementioned sample was filtered in a crucible and washed with hot distilled water. The extracted sample was dried in an oven at 105ºC until it had achieved a constant weight for hot water-soluble determination.

### Table 1. Standard for chemical analysis

| Property                  | Standard     |
|---------------------------|--------------|
| Alcohol-toluene solubility| T204 cm-97   |
| Hot-water solubility      | T2 64 cm-88  |
| Klasson lignin            | T222 cm-88   |
| Holocellulose             | Wise method  |
| Alpha-cellulose           | T203 cm-74   |
| Ash Content               | T211 cm-93   |

#### 2.3 Physical Resistance

**2.3.1 Thermo-gravimetric analysis (TGA)**

In each run, 10 mg of particle bagasse was used and heated from room temperature to 900ºC with a dwell time of 10 min. Nitrogen was used as the carrier gas for this experiment and the heating rate was fixed at 20ºC/min [26]. The rise in temperature in accordance to the weight loss of each particle sample with time was continuously recorded. The raw data obtained was used in plotting the graphs and determining the thermo-gravimetric (TGA) thermo grams. The degradation of the particle was obtained and observed from the thermo-gravimetric (TGA) graph.

### 3. Results and discussion

#### 3.1 Chemical component

The results obtained in this study shows a significant difference between the chemical component of raw bagasse and steamed treatment bagasse. It was shown that there are changes between the chemicals properties when performing steam treatment on the bagasse. The extractive, lignin, holocellulose and ash content of the steamed bagasse shows a decreasing trend as the steam time interval increases, compared to the α-cellulose which shows an increase trend in its percentage. This shows that a longer steaming period of the bagasse may diminishes a majority of its chemical properties.

Table 2 shows the alcohol toluene extraction percentage decreased from 7.0% to 1.0%, while by hot water extraction it decreased from 26.0% to 16.0%. The decreasing amount of extractive collected was expected as the extractive will be auto-hydrolyzed by the steam thus lessening the amount extractive within the bagasse [27]. The amount of extractive obtained in this study was surprisingly higher than the ones obtain from the previous researches (Table 3). The highest values of the hot water and organic solvent extractive obtained by Guilherme et al. [28] is 12.5% and 1.6%.

Other than the extractive, the percentage of lignin content of the bagasse also shows a significant decrease as the steaming period increases. The values of lignin from 43.0% decreases to 28.0%. It was found that the decreasing amount of lignin may occur due to the steam that modify the chemical structure of the lignin, thus lessen the amount of lignin collected over time. Although, the
The lowest lignin content obtained in this study was 28.0%, the amount was still higher than the ones obtained by Nonaka et al. [12] which is 25.1%. This shows that treating the bagasse with steam is an effective way to procure large amounts of lignin.

Table 2. Chemical compositions of different steam treatment times *Saccharum Officinarum L.*

| Time interval of steam treatment (min.) | Alcohol toluene extractive (%) | Hot water extractive (%) | Chemical composition |
|----------------------------------------|--------------------------------|--------------------------|----------------------|
|                                        |                                |                          | Lignin | α-cellulose | Holocellulose | Ash |
| 0                                      | 7.0                            | 26.0                     | 43.0    | 43.0        | 71.0          | 1.7 |
| 5                                      | 2.4                            | 27.6                     | 35.0    | 44.0        | 70.0          | 1.4 |
| 10                                     | 3.6                            | 16.0                     | 30.0    | 45.3        | 60.0          | 1.0 |
| 15                                     | 1.0                            | 29.0                     | 28.0    | 47.0        | 57.0          | 0.9 |

Note: The data presented were average values of at least 3 samples

The α-cellulose and holocellulose obtained in this study were of a different trend from one another, as one has a decreasing trend and the other an increasing trend. The increment of α-cellulose was due to the hydrolyzation of cellulose, thus increasing the accessibility of procuring the α-cellulose. This notion was supported as previously, Phinichka and Kaenthong [29] had obtained high amount of alpha cellulose when steaming sugarcane bagasse. The decreasing of holocellulose was due to the increasing severity of steaming that affected the degradation of hemicellulose, therefore reducing in lower holocellulose yield [30]. The steam treatments on the holocellulose and α-cellulose of the bagasse actually gave similar results to those presented by Panyakaew and Fotios [31], which is 79.5% and 57.0%, albeit a little bit higher than the one obtained in the study.

The average ash content collected from the experiment was 1.7%, 1.4%, 1.0% and 0.9% respectively. The results obtained shows that the increasing steam period had affected the ash content within the bagasse. It was concluded that the decrement of ash content in the study was due to some of the ash components within the sample had already been solubilised, resulting in lower ash content yield [30]. In comparison to the previous study, Pippo et al. [32] found that the ash content obtained is much higher, which is 4.0%.

Table 3. Chemical compositions of bagasse obtained by previous researchers.

| Components                  | Percentage (%) | References             |
|-----------------------------|----------------|------------------------|
| Organic solvent extractive  | 1.6 ± 0.2      | Guilherme et al. [28]  |
| Hot water extractive        | 12.5 ± 0.5     | Guilherme et al. [28]  |
| Lignin                      | 20.9           | Nonaka et al. [12]     |
| α-cellulose                 | 57.0           | Panyakaew and Fotios [31]|
| Holocellulose               | N/A            | Barbosa et al. [1]     |
| Ash                         | 4              | Pippo et al. [32]      |

3.2 Thermal properties

The effect of steaming on the thermal behaviour of sugarcane bagasse was also studied by using the thermogravimetric analysis from room temperature to 900°C at a rate of 20°C/min under nitrogen flow. Figure 1 shows the thermogravimetric analysis (TGA) curves of the samples at a different set of steaming period; control, 5 minutes, 10 minutes and 15 minutes. The first stage of weight loss occurs between a temperature of 200 – 300°C. This can be attributed to the lost of moisture within the sample, while the second stages of weight loss occurs around an average of 300°C [33]. Table 4 shows the effect of steaming treatment on the percentage of weight loss of sample and the degradation temperature peak of fibres.
Table 4. Effect of steaming treatment on the raw bagasse samples

| Raw bagasse at different steaming period | Weight Loss (%) | 300°C | 400°C | 500°C | 600°C | Peak degradation temperature (°C) |
|----------------------------------------|-----------------|-------|-------|-------|-------|-------------------------------|
| Control                                |                 | 3.7   | 35.7  | 39.4  | 40.9  | 280.6                         |
| 5 minutes                               |                 | 3.2   | 34.9  | 38.3  | 39.4  | 283.9                         |
| 10 minutes                              |                 | 2.2   | 26.6  | 29.2  | 30.4  | 286.8                         |
| 15 minutes                              |                 | 9.6   | 44.1  | 47.9  | 49.5  | 246.4                         |

The temperature of 100°C – 200°C was not taken as a variable for weight loss percentage, as there is an increase in weight in percentage of the sample due to chemical reactions (reaction with gaseous substances in the purge gas such as O₂, CO₂ with the formation of non-volatile or hardly volatile compounds) and physical transitions (adsorption of gaseous substances on samples) as shown in Figure 1.

Figure 1. TGA curves of steam-treated bagasse particles with different steam time rates

The weight loss of the controlled sample, which had not undergone any treatment had started to decompose at the temperature of 280.6°C, while the other samples had an increase in decomposition time, especially the 5 minutes and the 10 minutes steamed samples. There is a huge disparity in the value obtained for the 15 minutes sample compared to the others, as it has the lowest peak degradation temperature and the highest weight loss percentage. This shows that, as the steaming period increases at a certain point, in which is above the 10 minutes mark, the higher the chance the material will be degraded, thus decreasing the thermal characteristics of the sample.

4. Conclusion

In this study, the chemical component and thermal resistance of steam-treated bagasse was investigated. From the study, it is concluded that longer steaming period may have an unfavourable effect on the chemical component of the samples. This can supported by the fact that, the steamed samples at 121°C for 15 minutes contains the lowest chemical component of extractive, lignin, ash-content and holocellulose which is at
0.8%, 25.30%, 0.88% and 51%. Based on the study, it was evident to say that, the steam-treatment may have a positive effect on the bagasse particles. This was due to the reduction of extractives and ash content of the bagasse particles that may have a detrimental effect on the production of natural bonded board. The steam treated samples at 121ºC for 10 minutes was found to be the optimum condition. It is also to be concluded that, a longer steaming period may affect the thermal resistance of the sample, as it can be seen in the study that, the 15 minutes steam-treated sample has the highest percentage of weight loss, which is 49.5% and a lowest peak degradation temperature of 246.4ºC. The use of steam-pretreated method is quite inexpensive compared to the others that can enhance the particles properties, thus can further exploited in other applications with a lower cost of experimentation.

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