The effect of heating on the effectiveness of coal activated charcoal on glucose syrup purification

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Abstract. Glucose syrup is a sweetener that is widely used in the candy, confectionery, biscuit, ice cream and etc. The glucose syrup can be produced from tapioca with hydrolysis resulting in crude syrup that needs further purification. The purpose of this study was to understand the effect of heating on the effectiveness of coal activated charcoal in the process of purifying of glucose syrup. This research compared the heating and non-heating process in the activation of the charcoal to refine the glucose syrup. The parameters observed were reducing sugar, dextrose equivalent, sweetness level, total solids, clarity, and ash content. The results of this study showed that heating coal activated charcoal resulted in better quality syrup with reducing sugar value of 147.28 g / L, DE value of 43.81%, total solids content of 33.82%, sweetness level of 30°Brix, clarity level of 69.856T, and ash content of 0.18%.

1. Introduction
Glucose syrup is a sweetener that is widely used in food industries. Glucose syrup has the advantage of being unable to crystallize during heating at high temperatures and has a higher solubility when compared to sucrose [1]. Glucose syrup can be obtained from the starch hydrolysis process using an enzyme, acid catalyst or a combination of both. Syrup used in this study was crude glucose syrup from enzymatic hydrolysis following several process namely liquefaction and saccharification. The results of starch hydrolysis from the process of liquefaction and saccharification contain a mixture of impurities that reduce purity and colour stability. Impurities can be produced from the basic ingredients of starch used, or arise in the hydrolysis process [2]. Therefore, the purification process is necessary.

One method of purification is adsorption. Typical adsorbents such as activated carbon, bentonite, casein, and ion-exchange resins have been studied for removal of polyphenols and brown colour compounds from fruit syrups such as date syrup [3]. Out of all mentioned adsorbents, the activated carbon is used more than the others due to its higher adsorption capacity. In general, activated charcoal can be activated in 2 ways, namely chemical activation with alkali or acids like metal hydroxides, carbonate salts, chlorides, sulphates, phosphates from alkaline earth metals and in particular ZnCl₂, CaCl₂, inorganic acids such as H₂SO₄ and H₃PO₄ and physical activation which is the process of breaking carbon chains from organic compounds with the help of heat [4]. In this study, the chemical using was minimized thus heat treatment was preferred. However, the effect of temperature on the effectiveness of various types of activated charcoal in clarifying glucose syrup was not yet widely known. Therefore, in this study, we want to know the effectiveness of heating in the activation the coal charcoal on the quality of the purified glucose syrup resulted.
2. Methods

2.1. Materials
The materials used in this study were crude glucose syrup from the enzymatic process, α-amylase, CaCl₂, HCl, activated charcoal, zeolite, coarse sand, fine sand, and 3,5 dinitrosalicylic acid (DNS).

2.2. Purification of glucose syrup
1 liter of glucose syrup from enzymatic hydrolysis was prepared. 1% of coal granulated activated charcoal was added to glucose syrup. The syrup then divided into two parts in which one part heated at 70 - 80°C for 30 minutes and another one was without heating treatment. After that, glucose syrup placed in a tube filter with 20 cm of zeolite, 20 cm of coarse sand, and 40 cm of fine sand. The filtering product was then analyzed for the parameters tested.

2.3. Observation parameter

2.3.1. Reducing sugar (g/L). We measured reducing sugar using the method described by Apriyantono [5]. In an alkaline atmosphere, reducing sugars would reduce the DNS to form a compound that the absorbance can be measured at a wavelength of 550 nm [6]. The standard curve was made using a standard glucose solution containing 0, 25, 50, 75, 100, 125, and 150 ppm of glucose. Each 1 ml pipette and put into a test tube with the addition of DNS solution and mixed it vigorously. Then placed it in boiling water for 5 minutes then cooled to room temperature. Absorbance was measured at 550 nm. Determination of reducing sugar in the sample was done with the same procedure. The data obtained were plotted in the standard curve equation.

2.3.2. Dextrose equivalent (%). Dextrose equivalents (DE) measured by using a formula:

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DE = \frac{\text{reducing sugar formed (w/v)}}{\text{initial substrate (w/v)}} \times 100\%
\]

2.3.3. Solid content (%). The solid content was measured using the oven method [7]. 2-5 grams of sample was dried using an oven at 100-105°C for 4-6 hours until the weight of the samples was constant then the weight of the sample after heated was divided with the initial weight and multiplied by 100%.

2.3.4. Sweetness level (°Brix). The sweetness of the glucose syrup was tested using a "hand-refractometer" device. To measure the sweetness, the sample was placed on the surface of the hand-refractometer and then the number displayed on the device was the sweetness level.

2.3.5. Clarity level (T). The clarity level was measured using a spectrophotometer. This method principally measured the absorbance (A) and further transferred it into Transmittance (T) in which the absorbance of the sample at the maximum wavelength considered the most visually clear. Where the higher the absorbance, the more turbid or unclear a sample. The transmittance then calculated using formula \(A = 2\log T\) to obtain the level of clarity.

2.3.6. Ash content (%). The method used in this study was a furnace method. The porcelain cup was heated 3 hours in a furnace, then weighed, and placed 2 g of the sample inside the cup. Then it was heated at 500-600°C for 3 hours. The cup containing the ashes removed from the furnace, and then weighed. The weight of the ash obtained was then divided by the weight of the initial material and multiplied by 100%.
3. Results and discussion

3.1. Reducing sugar
Reducing sugars are sugars which have free aldehyde or ketone group in the sugar component that can be oxidized to an alkaline atmosphere which can reduce metals acids (aldonic acid, ketonic acid or sugar components will be oxidized to uronic acid) [8].

The value of reducing sugar indicates the number of simple sugars contained in food. Simple sugars formed in glucose syrup production show the effectiveness of the enzymes used for saccharification. The value of reducing sugar was tested by the DNS method by using a spectrophotometer. The reducing sugars of the sample are presented in figure 1.

![Figure 1](image)

**Figure 1.** The correlation between heating and non-heating to reducing sugars (g/L)

Figure 1 shows the difference between heating treatment and non-heating. There was an increase in both samples compared to control, however the glucose syrup with heating had a higher reducing sugar value than the sample with the non-heating process. This is because the heating process helps activate the charcoal. Heating helps the pores of activated charcoal to open and made it easier to absorb other compounds contained in glucose syrup. The increase of temperature may contribute to the better diffusion of adsorbate in the porous microstructure of activated carbon, as both temperature-dependent dispersion forces and electrostatic interactions affect the adsorption process [3].

3.2. Dextrose equivalent (%)
The glucose content in syrup is expressed by dextrose equivalent (DE), which is commercially reducing sugar levels which are calculated as dextrose (glucose) based on the basis of dry matter [9]. The higher the DE solution, the higher the glucose level and the lower the dextrin level. DE does not state the actual glucose content of the product but is related to the reducing sugar content of all types of sugar contained in the product. Glucose syrup with a low DE value has high viscosity and low sweetness.
The value of DE (Figure 2) on heating is influenced by an increase in the value of reducing sugars. Heating which is an activation step in activated charcoal causes the value of reducing sugar to increase because the components of starch that have not been decomposed into glucose are absorbed by activated charcoal while simple components such as glucose are not absorbed by activated charcoal due to the more polar nature. In addition, the increase in DE value was also influenced by filtration which causes a decrease in the value of dry solids, because only small particles pass through filtration. Although in figure 2 there is a clear difference between both the DE with heating and non-heating process met the Standards-based on SNI 01-2978-1992 which is a minimum DE of 30% [10].

3. 3. Solid content
Total solid is the number of solids contained in a material. Total solids show solids in a substance that is dissolved or not dissolved in water. Total solids were analyzed using the oven method [7]. Solids obtained in the syrup with heating treatment are higher than on that non-heating process. Syrup yielded with heating treatment tends to be closer to or higher than the substrate. Whereas without heating it is lower than the substrate. The high total solids in the heating treatment are due to the high content of simple components such as glucose in the syrup with heating treatment compared to non-heating. Activation of activated charcoal causes better adsorption ability so that other components in the syrup are absorbed. These simpler components were easier to pass through the filtration media. Whereas syrup with non-heating treatment has a lower total solid because there are still many other components and compounds that are more complex, it was difficult to pass through the filtration medium.
3. 4. *Sweetness level*

The sweetness level shows the value of dissolved solids in glucose syrup. Total dissolved solids in glucose syrup show the value of simple water-soluble sugars found in syrup. The level of sweetness in the sample with a heating treatment was higher than sample with non-heating treatment (figure 4).

Figure 4 shows that the syrup resulted from the process with a heating treatment had a higher level of sweetness than syrup with non-heating treatment. The sweetness level which is the total dissolved solids increases with the heating treatment because the components are soluble in water while the non-water or non-soluble components have been adsorbed by activated charcoal, while without heating there is no change because the activated charcoal is not activated so that the absorption of the component is not lower water-soluble. The active adsorption power of charcoal is due to the presence of a very large number of micropores, causing capillary symptoms which result in adsorption power [4].

**Figure 3.** The correlation between heating and non-heating to total solid content (%)

**Figure 4.** The correlation between heating and non-heating to the sweetness level (°Brix)
3.5. Clarity level (T)
Purification of glucose syrup aimed to obtain syrup with a clearer appearance and free from ion compounds. The clarity level in this study was measured using a spectrophotometer at a maximum wavelength of 370 nm. The clearer the glucose syrup the lower the absorbance and the higher the transmittance (T). The results of this study indicate that the sample with heating treatment has a higher clarity value (69.856 T) compared to the sample with non-heating treatment (54.909 T).

![Graph showing Clarity level (T) for Control, Heating, and Non-heating treatments](image)

**Figure 05.** The correlation between heating and non-heating to clarity level (T)

Activation treatment on activated charcoal by heating can increase the absorption of activated charcoal so that the pores of the activated charcoal are more open and the ability to absorb the components causing higher turbidity. A higher level of clarity in the heating treatment was caused by activation carried out on activated charcoal. While the treatment without heating the ability to absorb is still low but there is an increase in the clarity of the substrate because even though the ability to absorb is lower but the use of activated charcoal without heating can absorb the components that cause turbidity but in lower amounts. This happens because, at low-temperature activation, some complex compounds in charcoal have not decomposed/evaporated like sulfur and nitrogen compounds and other compounds in charcoal [4].

3.6. Ash content (%)
Ash content is the number of minerals contained in food. Analysis of ash content can be done by wet dry. Every food product has a different standard of ash content. The ash content in both sample with heating and non-heating process was lower than control. However, albeit the ash content in the sample with heating treatment was higher than in the sample with the non-heating process, the difference was not significant.
The correlation between heating and non-heating to ash content

The results of the observation (figure 6) show that the control has a higher ash content. This is caused by the control treatment that has not been through the purification process with coal activated charcoal. Purification causes a decrease in ash levels because not all minerals can pass through the filtration medium. Besides that, filtering through zeolite is an ion exchanger so that the minerals in glucose syrup will be absorbed by the zeolite. Natural zeolite has a skeletal structure, containing empty space occupied by cations and free water molecules that allow ion exchange or chemisorptions [11].

4. Conclusion

It is concluded that the treatment heating of coal activated charcoal produces glucose syrup with better quality than non-heating with reducing sugar values of 147.28 g/L, DE of 43.81%, total solids of 33.82%, sweetness level of 30°Brix, a clarity level of 69.856T, and an ash content of 0.18%.

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