Characterization of Saccharide Sugar in Corn Seed (Zea Mays Saccharata) By Using Gas Chromatography Mass Spectrometry Method

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Abstract

This study characterizes the saccharide sugar contained in whole sweet corn seeds by extracting using Ultrasound Assisted Solvent Extraction for 60 minutes with 24 kHz frequency using ethanol solvent with concentrations of 70%, 75%, 80%, and 85%. The concentrated extract was then characterized by using Gas Chromatography Mass Spectrometry (GCMS) method. Sample test conducted were tested to odor, color and shelf life of extract and determination of water content, ash content, and characterization of saccharide sugar from GCMS test. The odor and color test results show that the extract had a distinctive smell of sweet corn, yellow, and had the ability to store good extract in the condition of the room. The water content of each sample ranged from 3% - 9% with the lowest water content was in 85% concentration sample which was 3.62%, while the ash content ranged from 1.5% with the highest ash content which was in 85% concentration sample which was 1.59%. The results of characterization were identified by 3 compounds having the highest percentage of 2-Furaldehyde, Hexamethyl Cyclotriloc xlane, and 1,2,4-Trimethyl Benzene with the percentage of 26.94%, 9.95% and 13.82% respectively. 2-Furaldehyde or Furfural serves as a tongue nerve stimulator and has great potential to be developed as an important non-petroleum-based chemical raw material. The GCMS results provide information that the obtained 2-Furaldehyde has a molecular formula C_5H_4O_2 or C_4H_3OCHO which is a monosaccharide group which is aldose with the number of C atoms classified as pentose. The retention time was 3.062 minutes with a mass peak of 300 m/z with molecular weight was 96 g/mol.

Keywords:
Furfural; GCMS; Sweet Corn; Saccharide

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INTRODUCTION

Sweet corn, or often called Zea Mays Saccharata is a corn that is often consumed as roasted corn or vegetables contain high levels of sugar (Simon & Balabbo, 2015). Since the current low-calorie sugar trends are starting to get the public's attention, sugar from cereals and grains such as sweet corn is a study to be developed because besides being an important food (Gan et al., 2014) also sweet corn contains vitamin C, vitamin E and some other minerals (Kwabih, 2004; Haddadi, 2016). Sweet corn provides a relatively high profit when it is cultivated properly. In addition to the seeds, other parts of sweet corn plants have economic value such as stems and young leaves for livestock feed, old stems and leaves (after harvest) for compost green manure, stems and dried leaves as fuel substitute for firewood, young corn for vegetables, cakes, bakwan (veggies pan cakes) and various other processed foods. The age of sweet corn production is shorter, so it can benefit
Experimental Procedure

Preparation of raw materials

The raw material in the form of corn kernels (Zea Mays Saccharata) weighed as 150 gr was then crushed by using crusher to a small size of about 1-2 mm to facilitate the extraction process.

Extraction

Isolation of saccharide sugar was carried out by extraction of grain of corn that has been smoothed and weighed as 10 grams then put into glass beaker and ethanol (C₂H₅OH) 70% was added. Determination of solvent content was based on previous research on soybeans, inositol content (sugar derivatives) in optimum soybeans at 80% ethanol solvent (McDonald, 2012). The sample is then fed into ultrasonic extraction (sonicator) by Ultrasonic-Assisted Solvent Extraction method. Ultrasonic wave assisted extraction is performed with a frequency of 20 KHz. Temperature and time of extraction are arranged as fixed variable, under operating conditions with temperature 33°C for 60 minutes. Extraction results were filtered using Whatman filter paper number 311844, then the solvent was evaporated using a Rotary Vacuum Evaporator at a pressure of 24 KPa and a temperature of 50°C and thick concentrated was obtained. Experiments were performed with different concentration of C₂H₅OH solvent 70%, 75%, 80%, and 85% respectively.

Water content analysis

Sweet corn (Zea Mays Saccharata) residue after the filtration process is weighed into the cup by first weighing the plate in empty condition after being put on a constant weight. Grains and sweet corn drops are fed into the oven to gravimetricize the sample. Grains and samples that have been sterilized for ± 4 hours cooled into the desiccator for 10 minutes are then weighed to a constant weight.

%Water Content = \( \frac{(B - A)}{B} \times 100\% \)  

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Where,
A: Sample Weight after gravimetry  
B: Sample Weight after extraction (dregs)

Analysis of Ash content

Analysis of ash content is the continuation of the process of water content. The cup and the
Table 1. Determination of Water Content of Each Sample

| Sample | Empty dish weight + Filter Paper (gram) | Dregs weight + Empty dish + Filter Paper (gram) | Sample Weight (Dregs) (gram) | Weight after Oven (gram) | % Water content |
|--------|----------------------------------------|-----------------------------------------------|----------------------------|------------------------|-----------------|
| 70%    | 53.9207                                | 63.6116                                       | 9.6909                     | 54.8627                | 9.72%           |
| 75%    | 54.0947                                | 62.8716                                       | 8.7769                     | 54.6865                | 6.74%           |
| 80%    | 41.5055                                | 49.6723                                       | 8.1668                     | 41.8737                | 4.51%           |
| 85%    | 52.8644                                | 59.7846                                       | 6.9202                     | 53.1149                | 3.62%           |

Table 2. Determination of Ash Content of Each Sample

| Sample | Empty dish weight + Filter Paper (gram) | Dry sample weight (after gravimetry) (gram) | Weight after furnace (gram) | Ash weight (gram) | Ash content % |
|--------|----------------------------------------|---------------------------------------------|----------------------------|-------------------|---------------|
| 70%    | 53.9207                                | 0.942                                       | 54.8483                    | 0.0144            | 1.529%        |
| 75%    | 54.0947                                | 0.5918                                      | 54.6772                    | 0.0093            | 1.571%        |
| 80%    | 41.5055                                | 0.3682                                      | 41.8679                    | 0.0058            | 1.575%        |
| 85%    | 52.8644                                | 0.2505                                      | 53.1109                    | 0.004             | 1.597%        |

sample are put into the furnace and then the temperature is set at 550°C for ± 5 hours or until the sample turns to ash. The graft and the ash sample have been weighed for the weight of the ash produced.

\[
\% \text{ Ash Content} = \frac{B}{A} \times 100\% \tag{2}
\]

Where,
A: Gravimetry weight sample weight
B: Ash Weight

Analysis of content

Extracts obtained from various variations of the solvent after concentrating with the rotavaporator until all solvents evaporated under 60°C operating conditions for ± 1 hour per sample or ensuring the solvent had evaporated. The result of the solution that has been concentrated in the pipette as much as 5 ml into the petri dish to be measured the level of content of each sample by first know the weight of empty petridish. Determine the residual weight and weight of the yield (Y).

\[
Y = \frac{\text{Volume of Total Sample}}{\text{Volume of analyzed Sample}} \times \text{Residue Weight} \tag{3}
\]

Characterization

The characterization of saccharide sugar was analyzed by GCMS method (gas chromatography mass spectrometry).

RESULTS AND DISCUSSION

The results obtained are characterization of saccharide sugar content contained in sweet corn seeds through GCMS method and color, odor, shell extract as well as determination of moisture content, ash content, and other content.

Water Content

Water content analysis was done by using the oven as a drying medium (gravimetry). The sample was fed into the oven, so all the water in the sample evaporates shown the constant sample weight. Weight loss indicates that the amount of water contained in the sample. In the result of water content analysis on the smallest sample at 85% concentration is 0.0361 gr. For percentage of water content can be calculated and shown with the following Table 1.

The water content obtained as shown in Table 1, i.e. in the samples at each concentration of 70%, 75%, 80%, and 85% have the percentage of water content are 9.72%, 6.74%, 4.51%, and 3.62% respectively.

Ash Content

The process of spreading is done by dry ashing process. It uses high heat in the presence of oxygen. The sample was detected in a furnace without flame until white ash and constant weight
Table 3. Yield Analysis

| Sample | Volume of Total Sample (mL) | Petridish weight + Residue (gram) | Empty petridish weight (gram) | Residue Weight (gram) | Yield level (gram) |
|--------|-----------------------------|----------------------------------|------------------------------|----------------------|-------------------|
| 70%    | 76.0                        | 31.7578                          | 31.7187                      | 0.0391               | 0.5943            |
| 75%    | 80.0                        | 31.7589                          | 31.7114                      | 0.0475               | 0.7600            |
| 80%    | 82.0                        | 35.0279                          | 34.9881                      | 0.0498               | 0.6527            |
| 85%    | 77.0                        | 35.1905                          | 35.1455                      | 0.0450               | 0.6930            |

Figure 1. Yield Level Analysis

Yield Levels

Yield is the ratio of the quantities of products produced from sweet corn extraction or the percentage of products obtained from comparing the initial weight to the final weight. So it can be known to lose weight on the processing. The higher the resulting Yield value signifies the increasing number of products produced. The Yield content was analyzed after the concentration process using rotary vacuum evaporator. Each sample was piped 5 ml for each concentration variation of 70%, 75%, 80%, and 85%. The results of Yield content analysis were shown in Table 3.

The level of yield shown in Table 3 shows that in the 4 samples obtained from the extraction results in the form of isolates it was obtained showed a significant difference. From the table above shows that the content of each yield concentration 70%, 75%, 80%, and 85% is 0.5943 gr, 0.7600 gr, 0.6527 gr, and 0.6930 gr.

The highest yield content shown in Figure 1 that the highest content of yield present in the ethanol solvent variation was 0.7600 gram at 75% ethanol content. This shows that corn samples of every 5 ml of inositol solution at 75% ethanol solvent concentration have a yield content of 76%.

Characterization of Saccharide Sugar

Characterization of saccharide sugar using GCMS method by analyzing the recording of GCMS tool. GCMS recordings that detected some of the compounds contained in sweet corn seed extract identified 29 compounds with the largest percentage of area in sweet corn seed extract as many as three compounds that can be seen in Table 4 on the analysis of the largest chemical composition of compounds making sweet corn kernels.

The results of the characterization in Table 4 related to the analysis of the largest chemical composition of sweet corn kernels compound, identified 3 compounds that have the largest percentage of 2-Furaldehyde, Hexamethyl Cyclotriloxane, and 1,2,4-Trimethyl Benzene with a percentage of 28.68% each, 10.60%, and 14.72% with molecular weights of 96 g/mol, 222 g/mol, and 120 g/mol respectively.
Table 4. Analysis of Highest Chemical Composition of Sweet Corn Seed Compounds

| Peak# | Ret.Time (min) | Area     | Area % | Molecular Weight (gr/mol) | Conc. Mark Name Ret. Index |
|-------|----------------|----------|--------|---------------------------|---------------------------|
| 1     | 3.062          | 1206424  | 26.94  | 96                        | 2-Furaldehyde              |
| 3     | 3.267          | 445720   | 9.95   | 222                       | Hexamethyl-Cyclotrisiloxane|
| 8     | 4.936          | 619111   | 13.82  | 120                       | 1,2,4-Trimethyl-Benzene    |

The chromatogram shown in Figure 2 shows that peak 1 of 2-Furaldehyde is in an area of 1206424 with an abundance of 26.94%, peak 2 i.e. Hexamethyl Cyclotrisiloxane is in the area of 445720 with an abundance of 9.95% and 1,2,4-Trimethyl Benzene at peak 3 is in the area of 619111 with an abundance of 13.82%. 2-Furaldehyde includes heterocyclic aldehyde group sugars, whereas Hexamethyl Cyclotrisiloxane and 1,2,4-Trimethyl Benzene belong to the class of organosilicon and aromatic hydrocarbons.

2-Furaldehyde or Furfural with a structure in Figure 3, serves as a tongue nerve stimulator and has great potential to be developed as an important non-petroleum-based chemical raw material as it is a compound of food crops and it is possible if processed into biofuels that can reduce CO$_2$ emissions (Rios et al., 2015).

The spectral pattern of Figure 4 with the 2-Furaldehyde peak was at the peak of 40 m/z, 49 m/z, 67 m/z, 83 m/z, 96 m/z (bottom), and 106 m/z with an average retention time of 3.050 minute until 3,067 minutes. The GCMS results provide information that the obtained 2-Furaldehyde has a molecular formula C$_5$H$_4$O$_2$ or C$_4$H$_3$OCHO which is a monosaccharide group that is aldose with the number of C atoms it has classified as pentose. The retention time was 3.062 min at a mass peak of 300 m/z and the base peak at 96 m/z with molecular weight was 96 g/mol.
CONCLUSION

Sweet corn extract extracted using Ultrasound Assisted Solvent Extraction produces an extract that has a good shelf life and smells typical of sweet corn and yellow extract solution. The percentage of sweet corn seed extract at 85% concentration variation has water content 3.62% and ash content 1.59%, while the highest yield content at 75% is 76%. The result of characterization using GCMS method obtained by compound 2-Furaldehyde identified as a group of saccharide sugar, ie group of monosaccharide that is aldosa with the number of atom C which belongs is classified as pentose with molecular weight was 96 gr/mol.

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