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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 Cyclotriloxane, and 1,2,4-Trimethyl Benzene with the percentage of 26,94%, 9,95% and 13,82% respectively. 2-Furaldehyde includes heterocyclic aldehyde group sugars, whereas Hexamethyl Cyclotriloxane and 1,2,4-Trimethyl Benzene belong to the class of organosilicon and aromatic hydrocarbons. 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.

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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

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from the time side (Ayunda, 2014; Szymanek et al., 2015).

Carbohydrates in corn kernels are about 188-221 grams per kilogram (Barátová *et al.,* 2016; Coşkun *et al.,* 2006) or 70% starch of total weight (Auliah, 2012) and contain reducing sugars (glucose and fructose), sucrose, polysaccharides and starch as saccharides (Fitch, 2012). The sugar content in sweet corn endosperm is 5-6% (Simon & Balabbo, 2015). Research Szymanek et al. in 2015 reported sweet corn sugar content is 32%. Ordinary corn contains only 2% sugar or half of sugar content in sweet corn or about 25-30% sugar content from ordinary corn (Ugur & Maden, 2015). Sucrose stored in this sweet corn can reach 29.9% higher than other types of maize (Lertrat & Pulam, 2007). Corn bran consists of 75% hemicellulose, 25% cellulose, and 0.1% lignin. If it is compared, the maize fiber content without the epidermis is very low than the whole seeds. The sugar content of sweet corn is what will determine the quality of sweet corn (Surtinah *et al.,* 2016).

The content of sugar (saccharides) in high sweet corn seeds can then be used as a sweetener. As a sweetener, it is classified as a nutritional or non-nutritive sweetener. Nutritional sweeteners contain carbohydrates and as energy providers are classified into monosaccharides or disaccharide groups containing 4 kcal/g calories or sugar alcohol (polyol) (Fitch, 2012).

MATERIALS AND METHOD

Apparatus

The apparatus used were analytical scale, ultrasonic extraction tool, sample bottle, beaker glass (Pyrex), glass funnel (Pyrex), measuring cup (Pyrex), spatula, petridish cup, vacuum filter, rotary vacuum evaporator, sample bottle, and dropper drops, glassware (Pyrex), desiccator, GCMS (Shimadzu GC 2010 Plus), suction ball, volume pipette (Pyrex), thermometer, glass funnel (Pyrex), and electric oven (Karl Fischer).

Material

Some of the ingredients used are sweet corn type obtained from Antang Makassar market, South Sulawesi and the solvent used are C_2H_5OH from Baker Analyzed with concentration 99,9%, and filter paper *Whattman* number 311844.

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 (C_2H_5OH) 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 33^0C for 60 minutes. Extraction results were filtered using Whattman filter paper number 311844, then the solvent was evaporated using a Rotary Vacum Evaporator at a pressure of 24 KPa and a temperature of 50^0C and thick concentrated was obtained. Experiments were performed with different concentration of C_2H_5OH 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.

$$\%\text{Water Content} = \frac{(B - A)}{B} \times 100\% \qquad (1)$$

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^{0}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.

$$\% \text{ Ash Content} = \frac{B}{A} \text{ x } 100\% \qquad (2)$$

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^{0}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).

$$Y = \frac{\text{Volume of Total Sample}}{\text{Volumen of analyzed Sample}} \text{ x Residue Weight} \qquad (3)$$

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	0.0398	0.6527
85%	77.0	35.1905	35.1455	0.0450	0.6930

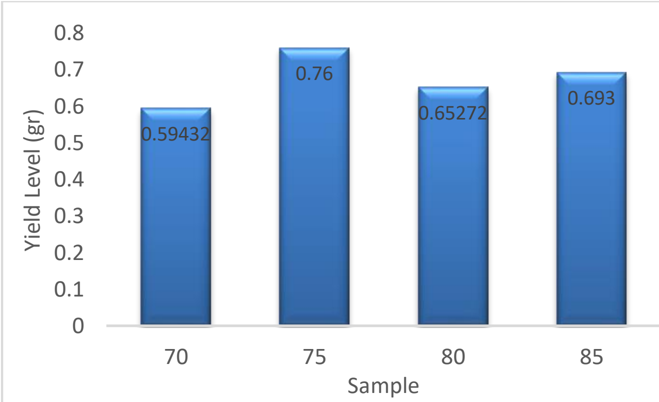

Figure 1. Yield Level Analysis

was achieved. The oxidation of the components on the furnace is carried out at a temperature of 550^{0}C. The residue of the furnace on the furnace obtained the highest ash content at 85% concentration is 0.00159 gr of ash with the percentage of ash content as follows in Table 2.The ash content shown in Table 2 shows that the percentage of ash content obtained in a sample with a concentration of 85% indicating that in each 0.2505 gram samples obtained ash as 1.59%.

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*

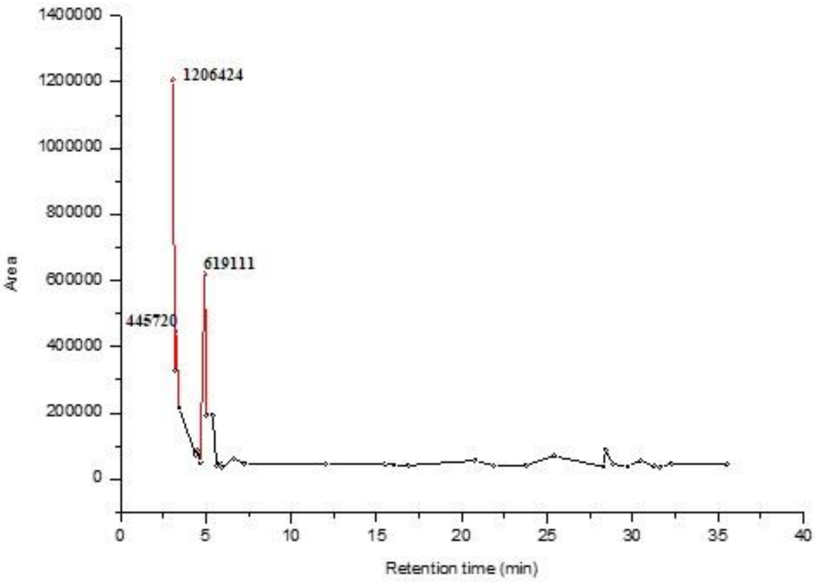

Figure 2. Chromatogram of Sweet Corn Seeds Extract

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 Cyclotriloxane 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 anabundance of 13.82%. 2-Furaldehyde includes heterocyclic aldehyde group sugars, whereas Hexamethyl Cyclotriloxane and 1,2,4-Trimethyl Benzene belong to the class of organosilicon and aromatic hydrocarbons.

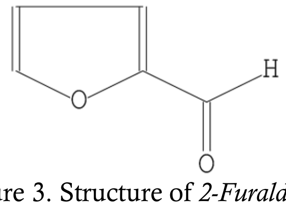

Figure 3. Structure of *2-Furaldehyde*

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 $C_5H_4O_2$ or C_4H_3OCHO 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.

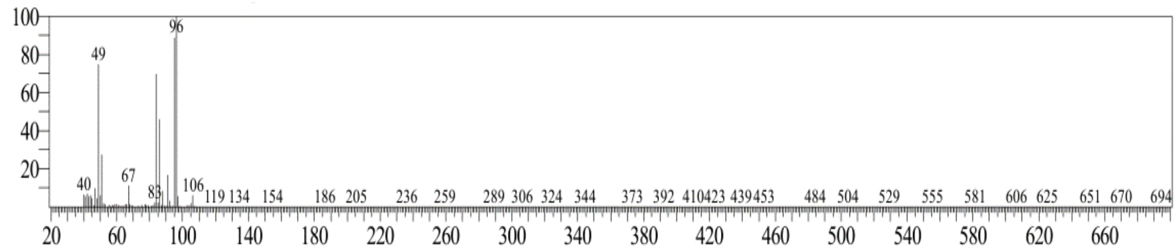

Figure 4. Spectrogram of Mass Fractionation

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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