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Extraction of Pectin from Watermelon Rind: Parametric Sensitivity and Characterization

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Abstract: This work presents the parametric sensitivity of pectin extraction from watermelon rind. The effect of process variables such as citric acid concentration (0.5 - 5 g/100ml), particle size (95 µm - 300 µm) and heating time (25 min - 90 min) on pectin yield was investigated. The extracted pectin was characterized to determine the moisture content, ash content, methoxyl content, equivalent weight, anhydrouronic acid (AUA), degree of esterification (DE). The optimum yield (17.90 %) was obtained at a heating time of 90 min, citric acid concentration (CAC) of 5.0 g/100ml and particle size of 95 µm. The pectin shows an off-white colour. Other properties were appreciably consistent with literature. The result shows that pectin extracted from watermelon rind is suitable for relevant industrial application such as food processing industry and jelling agent in jams production.

1. INTRODUCTION

Pectins are known to be complex polysaccharides (carbohydrate polymer) usually made up of -D-galacturonic acid unitsbeing linked by α-(1→4) linkages (Liew*et al.,* 2014). D-Galacturonic acid is mainly a sugar acid. It is an oxidized form of D-galactose which is usually presentin thepolygalacturonic acid in pectin. It is characterized with the presence of an aldehyde group at C1 and a carboxylic acid group at C6 (Khan*et al.,* 2015).

It is commonly found in the cell walls and middle lamellae of higher plants. It is also a polysaccharides consisting of about 300-1,000 chains of galacturonic acid units (Yeoh*et al.,* 2008). Pectin is an odourless solid powder characterize with off white colour. It is soluble in pure water and stable at room temperature. At moderate concentration it exhibits non-Newtonian pseudoplastic behaviorbut Newtonianbehavior in dilute solution. Pectin hydrate very rapidly when water addition thereby forming gels (Sundar et *al.,* 2012). It can also be used in pharmaceutical industry to encapsulate drugs and to permit the release of the active substance into the blood transmission (Herbstreith and Fox, 2006). As emulsifier, thickener, stabilizer and texturizer in food processing industry, jelling agent in the production of jams and jellies (Liu *et al.,* 2006). Pectin can also be used to stop diarrhea (frequent and excessive bowel disorder) predominantly in children (Sriamornsak, 2003). It has also been proven that pectin lowers blood cholesterol levels which are beneficial for human health (Liu *et al.,* 2006).Pectin can be produced from variety of plants like apple pomace, spent guava, lime peel, soy hull, cocoa husk, citrus peels using acidified extraction method with yields of about 12 and 25 % pectin respectively (Ismail*et al.,* 2012). The yield of pectin and its degree of esterification tends to vary with variations in fruit peels, change in parameters and nature of extraction carried out.

Thedemand for pectin in the global market has been reported to befar above 30,000 tons annually (Yeoh*et al.,* 2008).

Watermelon (citrullus*lanatus*) is a thirst quenching fruit that belongs to the cucurbitaceae family; it is widespread in tropical and subtropical regions of the world. Due to the solubility of watermelon fiber, its aids reduction in cholesterol level, risk of heart disease, a relevant source of fiber, which is important to help the digestive track to function well by preventing constipation, hemorrhoids (swollen anal veins) and diverticular diseases (Sultana and Bari, 2003). Watermelon is one of the under-utilized fruits cultivated in the tropical and subtropical part of the world (Achu*et al.,* 2005). Therefore a significant percentage of the watermelon crop is wasted every year due to the inability to sell the total produce within the peak of growing season. This waste is as a result of the second class melons being left in the field. Approximately 30 % of the watermelon crop goes un-harvested every year. This wasted crop signifies an important potential for derivation of value-added products such as pectin. The main aim of thisstudy is to the optimization of extraction and characterization of pectin from watermelon rind.

2. METHODOLOGY

2.1. Materials

The watermelon rinds were collected from KasuwanGwariMarket in Minna, Niger State. All reagent used are of analytical grade.

2.2. Sample Preparation

Watermelon rinds were washed with warm water to remove some of the sugars, ground in an electric grater and then pressed to remove water. It was then dried in an oven at 90 °Cuntil a constant weight is attained. This dried watermelon rind was then crushed into flour and was used as the raw material for further analysis.

2.3 Pectin Extraction

Pectin was extracted from watermelon rind with different citric acid concentrations (CAC = 0.5, 1.0, 2.5, 3.0 and 5 g %), particle size ((300, 120, 100, and 95 µm) and heating times (HT = 25, 35, 50, 70, and 90 min) at 90°C (solute/solvent 1:50) using water. To isolate pectin, hot acid extract was pressed in a cheese cloth bag and the concentrated "juice" was cooled to 27 °C. The watermelon pectin was precipitated by alcohol-juice treatment 2:1 (v/v). The mixture of solvent and precipitate was stirred for ten minutes and then left for one hour in order to allow pectin flotation. The floating pectin was filtered through cheesecloth, rinsed with alcohol and then pressed. The pressed pectin was dried to constant weight at 55 °C and finally cooled in adesiccator. The yield was calculated on a dry weight basis (initial weight of sample). The hard pectin cake was ground into powdered form (Rasheed, 2008).

Plate 1: watermelon rind sample

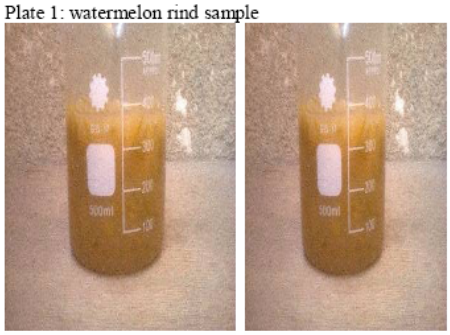

Plate 2: Mixture of watermelon rind slurry after heating

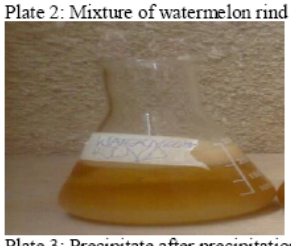

Plate 3: Precipitate after precipitation

Plate 4: Sample of extracted pectin

2.4 Characterization of Extracted Pectin

The dried pectin obtained from watermelon rind was subjected to the following quantitative test in order to be characterized.

2.4.1 Yield of Pectin

The percentage yield of the pectin was determined as the dry pectin weight divided by the dried weight of the pressed peel.

$$\text{Yield (\%)} = \frac{W_1 - W_2}{W_1} \times 100\ \% \qquad (1)$$

W_1= Sample before extraction
W_2= Sample after extraction

2.4.2 Determination of moisture content

Two (2) g of pectin sample was transferred into a dried empty petri dish placed into the oven set at 120 °C for 30 min thereafter the petri dish was removed, cooled in a dessicator and weighed.

$$\textit{Moisture content} = \frac{\textit{Weight of wet sample} - \textit{weight of dry sample}}{\textit{weight of dry sample}} \times 100 \qquad (2)$$

2.4.3 Determination of ash content

Two grams of pectin sample was accurately weighed in to a weighed crucible. The crucible was transferred to a furnace set at 120 °C to burn off all the organic matter. The carbon charred and then burnt off as carbon dioxide, leaving a dark ash; this process lasted for 2 h. The crucible was taken out of the furnace and placed in a desiccator to cool. The crucible after cooling was reweighed again. The ash content was calculated using:

Ash Content (%) =(Weight of Ash / Weight of pectin sample) × 100 (3.0)

2.4.4 Determination of Equivalent Weight

About 0.5 g of pectin sample was weighed into a 250 mL conical flask and moistened with 5 mL ethanol, 1 g of sodium chloride and 20 mL of distilled water and few drops of phenol red indicator were added. The mixture was stirred to ensure pectin was fully dissolved. After the above process, the mixture was titrated with 0.1 M sodium hydroxide to give pink colour at the end point. The equivalent weight of pectin can be expressed as

Equivalent weight(mg/mol)=

$$\frac{\text{Weight of pectin sample (g)}}{\textit{Volume of alkaline (ml)} \times \textit{Molarity of alkaline}} \times 100 \qquad (4)$$

2.4.5 Determination of Methoxyl Content (ME)

The determination of methoxyl content was carried out by adding 25 mL of 0.25 M sodium hydroxide to the neutral solution (equivalent weight) and mixed thoroughly and allowed to cool for 30 minute at room temperature. 25 mL of 0.25 M hydrochloric acid was added and titrated with 0.1 M sodium hydroxide to the same end point as before.

$$ME\ (\%) = \frac{mL\ of\ alkali \times N\ of\ alkali \times 31 \times 100}{Weight\ of\ pectin\ sample} \quad (5.0)$$

Anhydro-Uronic-Acid (AUA)

$$\%\ AUA = \frac{176 \times 0.1x \times 100}{W\ (g) \times 1000} + \frac{176 \times 0.1y \times 100}{W\ (g) \times 1000} \quad (6.0)$$

Where x = mL of sodium hydroxide used in determination of equivalent weight content.
y = mL of sodium hydroxide used in determination of methoxyl content.
W = Weight of sample

Determination of Degree of Esterification (DE)

$$DE\ (\%) = \frac{176 \times Meo\ \% \times 100}{31 \times AUA\ \%} \quad (7.0)$$

Where Meo% = methoxyl content,
AUA% = anhydrouronic acid content.

3. RESULTS AND DISCUSSION

3.1 Parametric sensitivity

3.1.1 Effect of particle size on pectin extraction yield

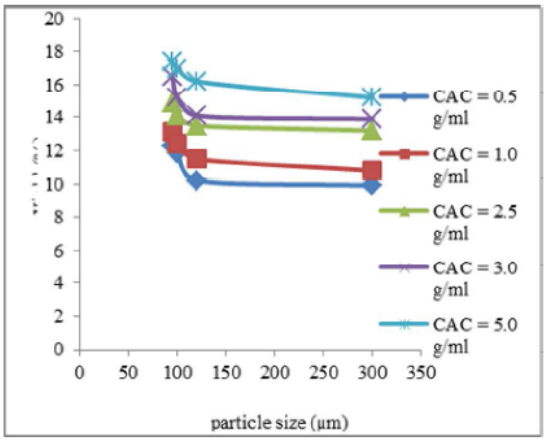

Figure 1: Effect of the size of the particle on pectin extraction yield with different CAC

From Figure 1 it was observed that percentage yield of pectin for 95 µm was 12.30 % with citric acid concentration (CAC) of 0.5 g/ml. This value is higher when compared with percentage yield of pectin of 9.90 % obtained from 300 µm at the same CAC. From the figure it was clearly shown that the same observations were made at various CAC. This result agrees well withthe work of Canteri-Schemin*et al* (2005) who stated that small particle size has high tendency to produce higher yield of pectin. It was also observed that maximum yield of pectin was 17.4 % at thiscondition.

3.1.2 Effect of heating time on pectin extraction with different particle sizes

The effect of extraction time was investigated between 20 and 90 minutes.

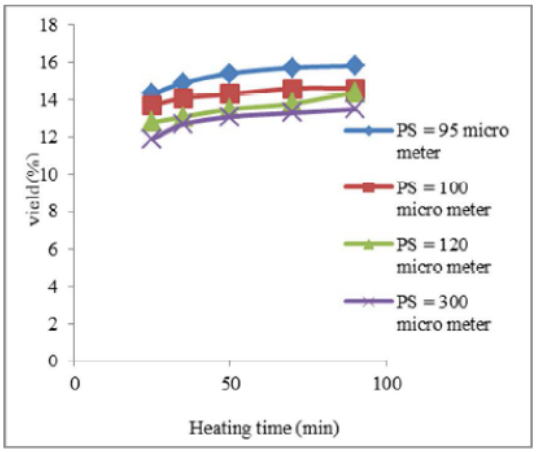

Figure 2: Effect of heating time on pectin extraction with different particle sizes

From Figure 2 it was observed that pectin extraction yield increased with increase in time. Theextraction yield increased from 14.30 % to 15.80 % as the timeincreases from 25 min to 70 min for all particle size with a particle size of 95 µm exhibiting the highest yield. It was also observed that there is no appreciable increase in pectin extraction yield (15.70 % to 15.80 %) from 70 min to 90 min for all particle size. This result agrees with the report of Rasheed (2008) which stated that pectinyield increased from 12.5 to 15.5 % as the temperature increased from 90 min to 110 min. Therefore optimum pectin extraction yield could be obtained within 70 min to 90 min.

3.1.3 Effect of citric acid concentration on pectin extraction at different heating time

Figure 3 shows the effect of increasing citric acid concentration with different heating time on pectin extraction yield.

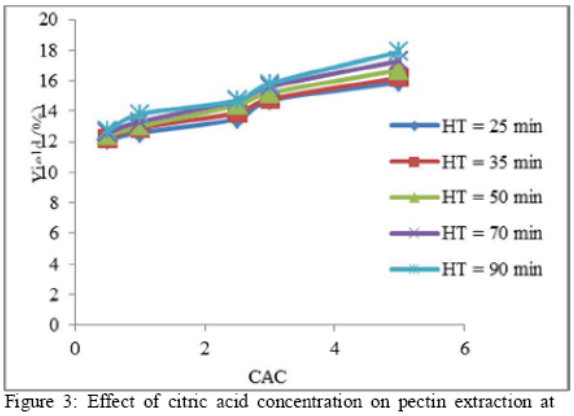

Figure 3: Effect of citric acid concentration on pectin extraction at different heating time

It is clearly shown that as the citric acid concentration increases from 0.5 g/ml to 5.0 g/ ml, the pectin

extraction yield increases from 12.10 to 15.90 % at 25 min heating time and the same observations were made at other heating time. It is also observed that optimum pectin extraction yield of 17.90 % was obtained with citric acid concentration of 5.0 g/ml, particle size of 95µm and heating time of 90 min.This value is slightly higher than the 15.19 % reported by Canteri-Schemin*et al* (2005). Therefore to obtain high percentage yield of pectin, adequate citric acid concentration, particle size and heating time are required.

3.2 Characterization of watermelonpectin

Table 3.1 shows the properties of watermelon pectin. The colour of pectin is very important in the characterization of the extracted pectin, it also have effect on the appearance of the extracted pectin. The colour of extracted watermelon pectin was observed to be off white.

Moisture content is the amount of water present in a sample that is responsible for microbial activity. The moisture content of the pectin sample was determined to be 16 %. This result is quite high compared to 11.13 % reported by Ismail*et al* (2012) for dragon fruit and but far lower than 66.60, 95.25 and 80.95 % for lemon,orange and grape respectively reported by Aina*et al (*2012). This might be due to difference in type of fruit.

Table 1:Characterization of watermelon pectin

Parameters	Values
Colour	Off white
Moisture Content (%)	16.0
Ash Content (%)	11.5
MethoxylationDegree (DM) (%)	51.48
Equivalent Weight (mg/mol)	609.76
Anhydrouronic acid (AUA) (%)	59.488
Degree of Esterification (DE) (%)	51.48

The moisture content of pectin should be as low as possible for safe storage and to inhibit the growth of micro-organisms that can affect the pectin quality due to the production of pectinase enzymes (Muhamadzadeh*et al.,* 2010).

Ash content is the quantity of inorganic minerals (in the form of salt and oxides) present in a given sample. The ash content was recorded as 11.5 %. This value show quantitative agreement with 11.5 % reported by Ismail*et al* (2012) for dragon fruit but slightly lower than 12.6 % for cocoa pod husk and appreciably lower than 20.8 % reported for pineapple skin by Mohamed and Hassan (1995). The low value obtained in this study indicates that low quantity of inorganic minerals (in the form of salt and oxides) are present in the watermelon rind. The difference observed when compared to other literature could be attributed to difference in fruit type.

Equivalent weight is expressed as the percentage by weight of pectin sample to the volume of alkaline and the molarity. The equivalent weight which was obtained to be 609.76 mg/mol was used in the calculation of % AUA and % DE. This value was slightly lower than 636.65 mg/mol reported by Ismail*et al* (2012).

Degree of methoxylation is an important factor for pectin assessment. It is an important factor in controlling the setting time of pectin, their combining power with metallic ions and the ability of the pectin to form gels. This determines whether the pectin should be termed high methoxyl i.e. higher than 50 % or termed low methoxyl i.e. lower than 50. DM of watermelon rind pectin is 5.394%. The findings clearly show thatpectin from watermelon rind can be categorized as high methoxyl pectin (HM). According to Woo*et al.* (2010) low methoxyl pectin (LM; DM< 50%) requires the presence of calcium ions to form gel. LM is usually employed in low sugar food applications, such as production of confectionery products, low calorie jams and jellies. It can also be applied in bakery jams and jellies for glazing, retorting, baking, and sterilizing or pasteurizing.

Degree of esterification (DE) determines the gelling nature of pectin and as well as the ratio of the esterified galacturonic acid groups to the total galacturonic acid groups. If the DE is more than 50 % then the pectin is high ester pectin and below 50 % it is low ester pectin. The DE was obtained to be 51.48 %. This value was quite lower than 46.96 % reported by Ismail*et al* (2012) for apple. Therefore, watermelon pectin produced in this study can be categorized as high ester pectin because it has a % DE that is higher than 50 % (Ismail*et al.,* 2012). Highester pectins are excellent stabilizers of milk drinks, this occur via coating of the milk casein particles thereby inhibiting particle agglomeration to form sediment(http://www.cargillfoods.com/ap/en/products/hydrocolloids/pectins/functionality/index.jsp).

The content of AUA measures of pectin purity. Percentage above 65 % is considered to be the typical minimum level for pectin used for various applications (IPPA, 2001). However, the AUA content obtained for watermelon pectin was < 65 %. Pectin with galacturonic acid contents higher than 65 % are classified as having high purity. The result shows slight deviation from this standard and this can be attributed to presence of unwanted materials

4. CONCLUSION

Pectin was successfully extracted from watermelon rind. The highest yield (17.90 %) of pectin from extraction was obtained ata particle size of 95 µm, 5 g/100ml of citric acid and a time of 90 min. The results of characterization shows that the colour of extracted

pectin is off-white, moisture content, ash content, methoxyl content, equivalent weight, anhydrouronicacid and degree of esterification shows that pectin from watermelon rind is suitable for relevant industrial application such as in food processing industry and jelling agent in the production of jams.

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