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A Survey on Native Cassava Starch and Fruit Harvesting Maturity

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Abstract - *Accurate identification of fruit harvest timing is quite a complicated issue. We know several methods of determining harvest maturity in climacteric fruits; these include measurement of: firmness, total solids contents, respiration rate, and ethylene emission measurement as well starch tests. On the other hand non-climacteric fruits (no collective maturity phase) are identified as suitable for harvesting by evaluating their color. The first three aforementioned basic methods can be combined into a single tool, the so-called Streiff index. All the characteristics identified herein, i.e.: starch assay, total solids (including sugars, tannins, vitamins, organic acids, etc.), as well as firmness, corresponding to protopectin-pectin conversion, produce caloric response.*

Starch is one of the most important sources of reserve of carbohydrate in plants and the main source in the human diet due to its abundance in the nature. There no other food ingredient that can be compared with starch in terms of sheer versatility of application in the food industry. Unprocessed native starches are structurally too weak and functionally too restricted for application in today's advanced food and industrial technologies. The results showed the steps of thermal decomposition, changes in temperatures and in gelatinization enthalpy and small changes in crystallinity of the granules.

Key Words: Fruits, Starch; cassava; hydrogen peroxide, thermal analysis, harvesting maturity

1. INTRODUCTION

The timing of fruit picking (harvest maturity) significantly impacts the duration of storage and quality parameters at the time of commercial operations. To make sure fruit maintain good quality and stability in storage, they should be collected at the most accurately defined time (Błaszczyk, 2006). This is also one of those factors which can be controlled with precision (Tomala, 2002). In order to determine harvest maturity in apples it is necessary to use assessment methods which enable precise evaluation of their condition. Generally these methods are not readily available for fruit farmers since they involve use of costly equipment (e.g. the procedure of measuring ethylene concentration in apple core). Others require a few measurements to be performed simultaneously, e.g. concentrations of ethylene, total solids and fruit firmness (Łysiak, 1998). In recent years there have been attempts to use ultrasound waves in evaluating fruit ripeness and in accessing color changes during the process of ripening (Mizrach *et al.* 2000). Yet, the methods are still not recognized as fully legitimate and reliable in this type of measurements.

Starch granules are made of glucose polymers, named amylose and amylopectin and they are found inside the vegetable cells from where they are extracted and treated for the industrial applications by food, textile, pulp and paper industry. These glucose polymers that make up the starch come in two molecular forms, linear and branched. The first is referred to as amylose and the latter as amylopectin. Amylose, mostly a linear chain, typically consists up to 3,000 anhydroglucose units (AGU) interconnected primarily by α-1.4 glycosidic linkages and is reported that it contains a few branched networks. The selection of the starch for industrial uses is made considering its availability and also its physicochemical characteristics that vary depending on the source.

Cassava (*Manihot esculenta,* Crantz) is an important vegetable crop in tropical regions where on a food energy production basis, it is ranked fourth after rice, wheat and corn as source of complex carbohydrates.4, 12 the typical composition of cassava root is moisture (70%), starch (24%), fiber (2%), protein (1%) and other substances including lipids and minerals (3%). The cassava starch has special technological properties, which allow its utilization in many industrial applications. Among these properties, there are the absence of the typical "cereal flavor" of corn and other cereal starches, its ability of higher swelling degree during cooking, and its lower pasting temperature, if compared again with cereal starches. Its low protein and lipid contents must also be valued contributing to its neutral fl avor and white color. 6, 16, 17 Starches may be oxidized by different chemicals as sodium hypochlorite, 9 bromine, 14 potassium and ammonia persulphate, 8 potassium permanganate17 and hydrogen peroxide. 7

The oxidation process aims to introduce carbonyl and carboxyl groups which increases clarity and reduces retro

gradation of cooked starch pastes providing lower viscosity and low temperature stability. 3, 6 When starch is heated in the presence of enough water, its crystalline organization decomposes to form amorphous regions. This molecular disordering is called gelatinization and is frequently observed as endothermic phenomenon using differential scanning calorimetric (DSC).11 Thermogravimetry can be helpful to show the behavior of starch granules when heating leads to also used to study the starch granules structural changes.1 There are few papers about modified starches considering their thermo analytical behavior due to the fact that starch studies are often developed by private companies. They produce highly valued and specialized modified starches for several industrial applications, especially for the food sector.

The objective of this work was to evaluate by thermo analytical techniques cassava natural and oxidized starches aiming to understand their behavior when treated with hydrogen peroxide in the presence and absence of ferrous sulphate. The thermogravimetry (TG), differential thermal analysis (DTA) and differential scanning calorimetric (DSC), as well as optical microscopy and X-ray powder diffractometry, were used to describe selected properties of the cassava starches.

2. MATERIAL AND METHODS

The value of total soluble solids, identified by means of ATAGO refract meter (PN-90/A-75101:2002) To achieve this the homogenized raw material was subjected to drying in a laboratory dryer, at the temperature of 105 °C, and then refined in a ball grinder and pelleted by means of Laormann apparatus. Calorific value was determined by incinerating the sample in oxygen atmosphere, in bomb calorimeter placed in water. Statistical analyses of the obtained results were conducted by means of Stat graphics 4.1 Plus. Mean values were compared using Student's t-test and Duncan's test at the level of significance p = 0.05 for n = 16.

The material selected for the study comprised three species of fruit, most popular in the Central European market; These were represented by 'Jonagold' apples, 'Elkat' strawberries and 'Bonaparte' tomatoes. Typical harvesting times, as well In accordance with the design, samples were collected three times during the process of fruit maturation, starting at the early stage after the green fruit has been formed and ending at the time of the potential harvest maturity. Each time analytical tests were carried out in 16 fruits of each variety, in accordance with Student's t-test identifying the significant number of repetitions in one group of fruit: The analytical tests conducted at each stage were designed to identify the level of fruit maturity based the following factors: - contents of water, ash and volatile substances measured with the use of TGA 701 thermogravimetric analyzer LECO, in compliance with standards: (PN-90/A-75101:2003); (PN-EN 1135:2002) ; (PN-C-04708-3: 1997). - Calorific value of dry matter with the use of AC 500 bomb calorimeter LECO, according to standard: PN-EN 14918:2010(U).

Hydrogen peroxide and other chemicals used in this study were analytical reagent grade (Merck). Cassava starch was extracted in laboratory according to the literature: 4 Cassava roots were washed, peeled, milled, sieved and the mash was washed. The solid was retained on the sieve (200 meshes) and the suspension kept for two hours decanting; then the starch was filtered, washed and dried in an oven at 35ºC. The obtained cassava starch was maintained in a desiccators over anhydrous calcium chloride until constant mass (sample "a"). Solutions of hydrogen peroxide (perhydrol at 30%) were prepared at 1, 2 and 3% (v/v) and standardized by iodometric method. In each ten grams (10.0g) of the obtained cassava starch was added at 50.0mL of H2O2 at 1, 2 or 3% (samples "b", "c" and "d", respectively), and stirred for 15 minutes (magnetic stirrer); after this time each suspension was filtered, washed, dried at room temperature and kept in a desiccators over anhydrous calcium chloride until constant mass. Solutions of hydrogen peroxide (perhydrol at 30%) were prepared at 1, 2 and 3% (v/v) and standardized by iodometric method. In each ten grams (10.0g) of the obtained cassava starch was added at 50mL of standard H2O2 at 1,2 or 3% and 0.01% of FeSO4 (samples "e", "f" and "g", respectively). Each suspension was stirred by 15 minutes.

After this time each one was filtered, washed, dried at room temperature and kept in desiccators over anhydrous calcium chloride until constant mass. The samples treated with H2O2 and FeSO4 showed rapid darkening what was not noticed for the starch treated only with H2O2. Thermal Analysis TG and DTA curves were registered using a simultaneous TG 60 system (Shimadzu) under an air fl ow at 100mL min-1 and at a heating rate of 10°C min-1. Alumina crucibles were used for the TG and DTA experiments. Mass and baseline calibrations were realized according to manufacturer and an empty alumina crucible was used as reference.

2.1 MICROSCOPY

Microscopy analysis was carried out using a stereoscopic microscope (Olympus SZX9), with polarizing filter and camera (Cybernetics' Cool Snap Pro Color). The photographs were identified and scaled using Image Pro Plus.

2.2 X RAY DIFFRACTION

X-ray powder patterns were obtained by using a model D-5000 X-Ray diffract meter (Siemens), with Cu Kα radiation and a setting of 40kV and 20mA. All the experiments, including the extraction of starch, thermal methods and X ray diffractometry were made in triplicate.

3. RESULTS AND DISCUSSION

Statistically significant changes were observed in the value of total solids in Bonaparte variety of tomatoes. During the maturation process the value of this parameter nominally increased to over 1%. According to Toor et al. (2006), the contents of total solids in tomatoes depended mainly on cultivars. During the ripening process the amount of soluble substances increases nearly two-fold. During growth of tomato fruit there is also an increased proportion of sugars (Dominguez *et al* 2012).

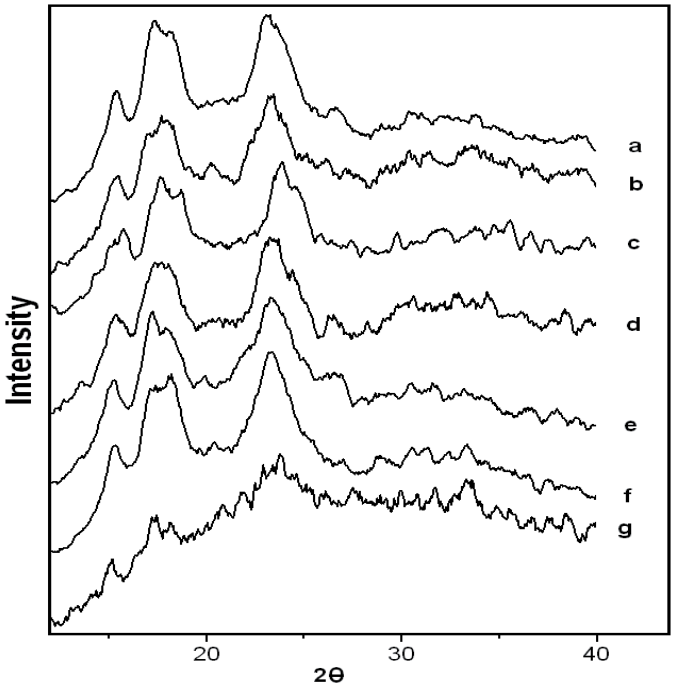
Correlation tables were generated for the consecutive samples, to present the assessed analytical parameters. In course of fruit and vegetable ripening, the quantity of monosaccharides in cell sap increases; these dissolve in water contributing to increased total soluble solids (Dominguez *et al.,* 2012). Statistically significant changes were observed in the value of total solids in Jonagold apple variety. During the maturation process the value of this parameter increased by nearly 3%, which is linked with the formation of simple sugars such as fructose and glucose in the fruit. Similarly, the contents of volatile substances, fragrances and essential oils showed a tendency for growth during fruit maturation. Similarly, the contents of volatile substances, fragrances and essential oils showed a tendency for growth during fruit maturation, to finally reach the threshold value of 1%. On the other hand no statistically significant differences were identified in the contents of water and ash in the fruit. During the final stage these parameters reached the values of 94.5% and 0.45% respectively. Musse *et al.* (2008) reported that in the case of tomatoes changes in water content during the process of ripening was not statistically significant. These authors noticed that during vegetation, after eighteen days, before harvest, the content of moisture was at the level of 95.6% and two days before potential harvest it decreased to 95.0%.

3.1 THERMAL ANALYSIS

The TG curve of the obtained and untreated cassava starch, the treated cassava starches with H2O2 (1, 2 or 3%) b-d and the treated cassava starches with H2O2 (1, 2 or 3%) each with FeSO4 0.01 %. These curves showed mass losses in three steps and thermal events corresponding to these losses. A great similarity is observed in the DTA profiles. The TG of the obtained cassava starch showed mass losses in three steps and thermal events corresponding to these losses. The first mass loss was between 30 – 105oC (8.0 %) corresponding to the endothermic peak at 70oC, which is attributed to the dehydration that occurs in a single step. Once dehydrated, the compound is stable up to 250oC and above this temperature the thermal decomposition occurs in two consecutive and/or overlapping steps between 250 and 513oC. The first mass loss (76%) of the anhydrous compound was observed between 250 – 428ºC corresponding to the exothermic at 355ºC with oxidative process. The last mass loss (15.9%) was between 428 – 513ºC corresponding to the sharp exothermic peak, ascribed to the oxidation of the organic matter.

The TG of the obtained cassava starches treated with standard H2O2 solutions at 1, 2 or 3% are shown in the b–d, respectively. It can be observed mass losses in three main steps and four thermal events corresponding to these losses. The first mass loss (7.8%), occurs between 30–103oC, 30 – 102oC (7.0%) and 30 – 100oC (7.0%), which are attributed to the dehydration that occurs in a single step corresponding to the endothermic peaks at 71, 70 and 70ºC, respectively. Once dehydrated, the compounds are stable up to 260, 261 and 261oC, respectively, and above this temperature the thermal decomposition occurs in two consecutive and/ or overlapping steps between 260 – 545oC, 261 - 554ºC and 261 – 547ºC. The first mass loss (74.8%) of the anhydrous compound observed between 260 – 395ºC corresponding to the endothermic peak at 311ºC and exothermic at 357ºC, which are attributed to the thermal decomposition that occurs initially without oxidative process followed by oxidative process. The last mass loss (17.2%), between 395 – 545ºC corresponding to the exothermic is ascribed to the slow oxidation of the organic matter.

The TG of cassava starch treated with standard H2O2 solutions at 1, 2 or 3% and 0.01% of FeSO4, respectively. It can be observed mass losses in three main steps and four thermal events corresponding to these losses. The first mass loss (8.8%) occurs between 30 – 93oC, 30 – 110oC (11.0%) and 30 – 102oC (7.3%), corresponding to the endothermic peaks at 73oC, 64ºC and 73ºC respectively, which are attributed to the dehydration that occurs in a single step. Once dehydrated, the compounds are stable up to 252, 264 and 259oC, respectively, and above this temperature the thermal decomposition occurs in two consecutive and/or overlapping steps between 252–528oC, 264 - 544ºC and 259 – 530ºC. The first mass loss (75.8%) of the anhydrous compound observed between 252 – 405ºCcorresponding to the endothermic peak at 314 and exothermic at 362ºC, which are attributed to the thermal decomposition that occurs initially without oxidative process followed by oxidative process. The last mass loss (15.3%), between 456 – 528ºC corresponding to the exothermic at 504ºC is ascribed to the slow oxidation of the organic matter.



CONCLUSION

All the TG curves showed similarity with mass loss in three steps. Significant difference is observed in DTA profiles, especially in starches after the treatment with H2O2 and H2O2 with FeSO4. The DSC curves allowed us to determine the onset temperature, peak temperature and gelatinization enthalpy. All the values of peak temperature (Tp) and gelatinization enthalpy (Hgel) of the treated starches were lower than those untreated. Authors created new method and new tool useful for determination of harvest date or maturity stage It seems important to create a table of calorific values for fruits in their harvest maturity time for each of their species and varieties The major quantity of final product of the treated starches with H2O2 and FeSO4 was attributed to incorporation of the iron at the starch. Maintained all the experimental conditions (starch extraction, treatment of each sample and equipments calibrations and parameters) the results are reproductive. X-ray diffraction confirmed the characteristic of native pattern and the decrease of intensity of main peaks suggesting that the crystallinity has little influence of the treatment.

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