



Effect of citric acid concentration and hydrolysis time on physicochemical properties of sweet potato starches



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ABSTRACT

Physicochemical properties of citric acid treated sweet potato starches were investigated in the present study. Sweet potato starch was hydrolyzed using citric acid with different concentrations (1 and 5%) and time periods (1 and 11 h) at 45 °C and was denoted as citric acid treated starch (CTS1 to CTS4) based on their experimental conditions. The recovery yield of acid treated starches was above 85%. The CTS4 sample displayed the highest amylose (around 31%) and water holding capacity its melting temperature was 47.66 °C. The digestibility rate was slightly increased for 78.58% for the CTS3 and CTS4. The gel strength of acid modified starches ranged from 0.27 kg to 1.11 kg. RVA results of acid thinned starches confirmed a low viscosity profile. CTS3 starch illustrated lower enthalpy compared to all other modified starches. All starch samples exhibited a shear-thinning behavior. SEM analysis revealed that the extent of visible degradation was increased at higher hydrolysis time and acid concentration. The CTS3 satisfied the criteria required for starch to act as a fat mimetic. Overall results conveyed that the citric acid treatment of sweet potato starch with 5% acid concentration and 11 h period was an ideal condition for the preparation of a fat replacer.

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1. Introduction

Today's dietary concern is the consumption of huge quantity of fat and sugar. With the mounting incidence of diabetes and obesity, low calorie foods have acquired the huge esteem. In general, the best suitable approach in terms of fat reduction diets involves either the use of low-fat foods or fat substitutes or modifications such as trimming of fat from foods [1,2]. Fat Replacers consist of mixtures of lipid-originated fat substitutes, protein- or carbohydrate-originated fat mimetic, or their combinations [3]. Carbohydrate-based Replacers incorporate water into a gel type structure, resulting in a lubricant or flow properties similar to those of fats in food systems [2]. Even though a variety of fat replacers have been developed, there are unfortunately no ideal fat replacers which completely function like conventional fat [4]. Native starch can sometimes be used to replace fat [1]; however starch modified by acid or enzymatic hydrolysis, oxidation,

dextrinization, cross linking, or mono-substitution is more commonly used to achieve desired functional and sensory properties [1]. Generally, acid hydrolysis occurs more rapidly in amorphous regions than in crystalline region and the residue after prolonged acid hydrolysis consists of acid-resistant crystalline parts of amylopectin [5]. Thys et al. [6] investigated the functional properties of acid-thinned pinhao starch and it showed low syneresis, high solubility, thermo reversibility and melting point similar to fat. They concluded that the acid treatment was efficient in producing a potential fat substitute from pinhao starch. Amaya-Llano et al. [7] produced acid hydrolyzed jicama starch and used as a fat substitute in yoghurt. The addition of hydrolyzed jicama starch (2.03 g/100 g) as a fat substitute in the preparation of stirred yoghurt had good functional and sensorial properties. Ma et al. [8] reported that enzymatic hydrolyzed corn starch could be used as fat replacers. The hydrolyzed starch with fine particles was used to produce low fat mayonnaise and the result indicated that the 60% fat-reduced mayonnaise with fat replacers had similar sensory quality as compared with the high fat one.

The following are the criteria for a starch based fat mimetic – (a) Starch should contain an amylose content of ~20% [9]. (b) Starch ought to require a granule size of 2 μm or in similar size to liquid micelle to act as fat mimetic [10]. (c) According to FDA [11] a starch-based fat mimetic is supposed to be partially or completely digestible. (d) Starch must possess a DE (dextrose equivalent) of

Abbreviations: NS, native sweet potato starch; CTS, citric acid treated starch; HT, hydrolysis time; AC, acid concentration; DE, dextrose equivalent; WHC, water holding capacity; PV, peak viscosity; BD, break down; TV, trough viscosity; SB, setback; FV, final viscosity; Pt, pasting time; PT, pasting temperature; To, onset temperature; Tp, peak temperature; Tc, final temperatures; ΔH, gelatinization enthalpy.

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≤ 5.0 [12]. (e) Starch gel with a melting point close to that of the fats (37–45 °C) could be used as a fat substitute [13]. (f) Starch must possess high water-holding capacity [9] and better emulsifying properties [4]. (g) Starch should display shear thinning characteristic [14].

Sweet potato (*Ipomoea batatas* (L.)) belongs to the Convolvulaceae family [15]. Sweet potato is considered as the world's most important and under-exploited crop [16]. Industrial application of sweet potato flour and starch is insignificantly causing a negative growth in its production. The intended use of sweet potato starch for industrial purpose depends on advanced processing technologies to prepare sweet potato starch with desirable functional properties, and on thorough indulgent of the effect of processing conditions on their properties. Basing on this background the research was aimed to study the influence of hydrolysis time and acid concentration on the sweet potato starch properties and evaluate the distinctive properties of citric acid treated sweet potato starch as fat mimetic.

2. Materials and methods

2.1. Materials

The sweet potato was purchased from a local supermarket (Palzhamuthir sollai), Salem, Tamil Nadu, India. Glucose Oxidase–Peroxidase (GOD–POD) kit was obtained from Beacon Diagnostics, Navasari, India. Amyloglucosidase from *Aspergillus niger* (≥ 300 U/mL) (E.C-3.2.1.3), citric acid and all other chemicals and reagents were analytical grade and purchased from Sigma-Aldrich, Steinheim, Germany.

2.2. Starch isolation and preparation of acid-thinned sweet potato starch

Starch was isolated from sweet potato by the method of Wickramasinghe et al. [17]. An edible portion of sweet potato was cut into small pieces and homogenized with distilled water. The slurry was then passed through the double-layered cheesecloth and the filtrate was allowed to settle for a minimum of 3 h at room temperature. The precipitated starch was washed three times with distilled water, dried at room temperature (20–25 °C) for 48 h and then the dried starch was kept in an oven at 50 °C for three hours and ground into fine powder and named as Native sweet potato starch (NS). Citric acid treated starch (CTS) was prepared by the method of Zambrano and Camargo [18]. Starch slurry was prepared by dispersing NS (40 g dry basis) in 1% or 5% citric acid solution kept in a water bath at 45 °C for 1 h or 11 h respectively with constant stirring. After each assay of hydrolysis, the pH was adjusted to 5.5 ± 0.2 by slowly adding aqueous sodium hydroxide (5 g/100 ml). The starch was washed three times with two fold volume of deionised water prior to filtration and dried in a convection oven at 45 °C for 48 h. The dried starch was made into powder and packed in airtight containers for further use and recovery yield was calculated by the following formula.

$$\text{Recovery yield (\%)} = \frac{\text{dry weight of starch after hydrolysis}}{\text{dry weight of starch before hydrolysis}} \times 100$$

2.3. Physicochemical properties of native and modified starches

2.3.1. Analysis of ash, protein, fat and total fiber

Estimation of ash, protein, fat and total fiber in the isolated starch material was carried out following the AOAC protocol [19].

2.3.2. Dextrose equivalent (DE)

The reducing sugar value was measured using the dinitrosalicylic acid method of Miller [20] to determine its dextrose equivalent (DE). Different concentrations of dextrose standard solution were taken in test tubes and dinitrosalicylic acid reagent was added in each of the test tubes. The test tubes were heated in a boiling water bath for 5 min. The Rochelle salt solution was added to each of the test tubes while the contents were still warm. The test tubes were cooled and the absorbance at 560 nm was noted and percentage of reducing sugar was determined. DE was calculated by the formula given by Miller [20]:

$$\text{DE} = \frac{\text{g reducing sugar}}{\text{g dry weight of starch}} \times 100$$

2.3.3. Apparent amylose

Apparent amylose content determination was carried out using a colorimetric iodine affinity procedure [21] briefly a mixture of 0.1 g of the starch sample, 1 ml of ethanol and 9 ml 1 N sodium hydroxide was boiled for 10 min in a boiling water bath and allowed to cool. To a portion (5 ml) of the mixture, 1 ml of 1 N acetic acid and 2 ml of iodine solution were added and Absorbance (A) was read using a Spectrophotometer at 620 nm. The apparent amylose content was calculated as follows:

$$\text{Apparent amylose content (\%)} = 3.06 \times A \times 20;$$

where A = absorbance value

2.3.4. Moisture and dry matter

Moisture content and dry matter were determined by the method of Adebayo, Lateef, and Elizabeth [22]. Two milligrams (2 mg) of starch sample was measured into a previously weighed crucible. The crucible plus sample was then transferred into the oven set at 100 °C, for 24 h. At the end of 24 h, the crucible plus sample was removed from the oven and transfer to desiccator cooled for ten minutes and weighed. The Moisture content and dry matter were determined and expressed in percentage.

2.3.5. Melting point, clear point and thermo-reversibility of the starch gel

Starch gel was prepared by the method described by the National Starch and Chemical Corporation [12] with modification. Sweet potato starch suspension (5%) was prepared with 0.02% (w/v) sodium metabisulfite in a beaker at 80 °C for 10 min and then autoclaved at 121 °C for 15 min. Subsequently beaker was cooled, hermetically sealed and stored at 4 °C for 24 h. The obtained gel was melted in a water bath at 80 °C under agitation. The change in consistency was visually observed, and melting point was considered as the temperature at which the liquid phase was formed and mixing of the gel was possible. The clear point was considered as the temperature at which the sol appeared optically clear. For the gel thermo-reversibility, the gel was melted in a water bath with constant stirring and allowed to cool down to room temperature, followed by refrigeration at 4 °C for 18 h and the gel formation was observed [7].

2.3.6. In vitro digestibility of starch samples

In vitro digestibility of starch was analyzed according to the method of Noda et al. [23] with some modifications. A mixture consisting of 4% (w/v) starch suspension in tubes was placed in a water bath at 100 °C for 10 min to obtain the starch suspension. A 0.5 ml of starch suspension and 0.25 ml of 100 mM acetate buffer (pH 5.0) and 0.25 ml of glucoamylase solution was incubated at 40 °C for 2 h

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