Food Hydrocolloids 81 (2018) 449–455

Contents lists available at ScienceDirect

Food Hydrocolloids

journal homepage: www.elsevier.com/locate/foodhyd

Structural and physicochemical properties of heat moisture treated and citric acid modified acha and iburu starches

Buliyaminu Adegbemiro Alimi [a, b, *], Tilahun Seyoum Workneh [b]

[a] Department of Food Science and Technology, Faculty of Agriculture and Agricultural Technology, Federal University of Technology, P. M.B. 65, Minna, Nigeria

[b] Bioresources Engineering, School of Engineering, College of Agriculture, Engineering and Science, University of KwaZulu-Natal, Private Bag X0l, Pietermaritzburg, Scottsville, 3209, South Africa

A R T I C L E I N F O

Article history:
Available online 14 March 2018

Keywords:
Acha
Heat moisture treatment
Citric acid modification
Starch
FTIR
Gelatinization

A B S T R A C T

Starches extracted from *acha* (*Digitaria exilis*) and *iburu* (*Digitalia iburua*) were subjected to heat moisture treatment and citric acid modification. Successful applications of the treatments are expected to promote commercial utilization of the starches from these underutilized crops and improve the economy and livelihood of stakeholders along their value chains. Granules of the starches from the two cereal grains were polyhedral in shape. Generally, modifications led to the clustering of the polyhedral of the starches granules. Indentations were observed on the surfaces of citric acid modified (CAM) starches. This could be due to the weathering effect of acid hydrolysis. Modification methods employed did not affect the characteristic type-A crystal polymorph of the native starches. The starches generally exhibited single prominent peaks at (2Θ) 16 and 24, and a doublet at 17-19. Crystallinity index of the starches ranged from 27.01-27.84% for *acha* and 29.01–30.47% for *iburu*. FTIR analysis and high peak gelatinization temperatures of the starches suggested heterogeneity in the matrices. CAM starches of the two grains remained in liquid form throughout the pasting cycle. *Acha* starches are lighter and whiter in color. All the starch samples exhibited promising functional properties that could place them as good materials for different industrial uses. Results of this study further exposed the potentials of *acha* and *iburu* starches for food and pharmaceutical applications.

1. Introduction

The continued interest in starch research stemmed from its array of functional properties. These properties place it as an indispensable ingredient for innumerable domestic and industrial applications (Alimi, Workneh, & Sibomana, 2016a). Utilization of starch in food system is dictated by its dominant properties. For instance; starch contribution to the textural properties of some foods makes it an important agent of thickening, stabilizing, gelling, bulking and water retention (Singh, Kaur, & McCarthy, 2007). Its lipophilic property is also important in fat based food. However, these properties vary according to the biological source of the starch and are governed by the size, shape, structure and chemical composition of its granules (Zavarese & Dias, 2011). Interestingly, starch in its natural form has some shortcomings which limit its use in industrial food processing. Examples of these shortcomings include its inability to withstand the high temperature, pressure and some strong chemical reagents use in most industrial food and pharmaceutical processes. Hence, starch is modified to correct some or all of these anomalies (Alimi, Workneh, & Oke, 2016b).

Properties of native and modified starches from major cereal and leguminous crops have been studied over time. The new thrust in starch research is the exposition of the potentials of starch from underutilized crops that have significant starch content. Some of these crops are abundantly available in developing countries. Successful isolation, characterization and further improvement of the starches from these crops would promote utilization of the starches and expand sources of starch. This would reduce pressure on established sources. It would also enhance commercial utilization of these crops and ultimately improve the economy and livelihood of farmers and other stakeholders along their value chains (Alimi et al., 2016a).

* Corresponding author.Bioresources Engineering, School of Engineering, College of Agriculture, Engineering and Science, University of KwaZulu-Natal, Private Bag X0l, Pietermaritzburg, Scottsville, 3209, South Africa. Tel.: +234 703 6711250.
E-mail addresses: bun5574@yahoo.com, buli.alimi@futminna.edu.ng (B.A. Alimi), Seyoum@ukzn.ac.za (T.S. Workneh).

https://doi.org/10.1016/j.foodhyd.2018.03.027
0268-005X/

Acha (*Digitaria exilis* Stapf) and *iburu* (*Digitalia iburua* Stapf) belong to this class of underutilized cereal crops. They are mostly grown in West Africa and were described as small seeds with big promises because of their excellent agro-ecological and food values. They perform excellently well on low moisture and fertility soil that may not support other cereal crops. They are known to mature within a short period of 6–8 weeks, have high starch content and higher economic returns compared to other cereals (Arueya & Oyewale, 2015; Jideani & Akingbala, 1993). Philip and Itodo (2006) reported that *acha* and *iburu* have greater performance potentials than some major cereal crops when same quantity of extra inputs are applied during growing period.

Acha and *iburu* grains have good content of sulphur amino acids. Sulphur amino acids are known for their importance in proper heart functioning and nerve transmission (Jideani & Jideani, 2011). They were also believed to have neutraceutical potential because of their use in some culture to manage diabetes (Jideani & Jideani, 2011).

However, the major attraction of these grains to researchers is their high starch content of about 75.5% (Arueya & Oyewale, 2015). Hence, studies are being carried out continuously to characterize their starches. These are with the view of exposing their potentials for domestic and industrial applications, and enhancing their values. Primary work on the properties of native *acha* and *iburu* starches was conducted by Jideani and Akingbala (1993). They were further investigated by Jideani, Takeda, and Hizukuri (1996). The studies revealed that the starches have properties typical of non-waxy cereal starches. However, native starches from these grains have characteristic shortcomings that are general to starches in their natural form. These shortcomings limit their industrial applications (Emeje et a. 2012). Hence, studies are being conducted to improve on the properties of their native starches (Emeje at al. 2012; Olu-Owolabi, Olayinka, Adegbemile, & Adebowale, 2014; Arueya & Oyewale, 2015). However, reports on heat moisture treatment and citric acid modification of the starches from these grains are scarce in literature.

There are different starch modification methods. The choice to be employed is dependent on the intending industrial end-use of starch. Heat moisture treatment, a type of hydrothermal modification, is a preferred method of starch modification for food and pharmaceutical applications. This is mainly because of the safety of heat moisture treated starch to health of consumers (Alimi, Workneh, & Oyeyinka, 2017). Heat moisture treatment also imparts favorable changes to the properties of starch. The enhanced properties are essential for some specific industrial applications (Alimi et al., 2016a). On the other hand, citric acid modification is a mild acid treatment of starch. It is relatively safe for food and pharmaceutical uses; since organic acid is the major treatment agent as compared to mineral acids. Citric acid modified starches find important applications in products where clarity is of essence. Examples include cheese and candies. Therefore, successful modification of the starches from *acha* and *iburu*, using these two modification methods, could expand their potential utilization for food and non-food uses. Hence, the objectives of this study were to investigate the effects of heat-moisture and citric acid modifications on some physicochemical properties of *acha* and *iburu* starches.

2. Materials and methods

Good quality *acha* and *iburu* were purchased from a local market in Kano, Nigeria. Native starch was extracted according to the alkaline (using aqueous sodium metabisulphite) steeping method previously reported by Arueya and Oyewale (2015). Toluene emulsification was used to separate protein from starch. Starch obtained was dried in a forced convective air oven at 45 °C for 24 h, packed in an airtight Ziplok bag and stored at 4 °C until further analyzes.

2.1. Heat-moisture treatment

Heat moisture treatment was carried out on native starches of *acha* and *iburu* as described by Alimi et al. (2016b). Moisture content of the starches was determined and raised to 25% through dispersion in distilled water. The slurries were heated in a convective hot air circulation oven at 110 °C for 16 h. The heat moisture treated starches obtained were left to cool, packed in Ziploc bags and stored at 4 °C until further analyzes.

2.2. Citric acid modification

Protocol described by Falade and Ayetigbo (2015) were used to prepare citric acid modified starches. Native starches (300 g) were dispersed in distilled water (400 mL) to obtain starch suspensions. The suspensions were brought to alkaline pH (about 9) through addition of 10 mL of 1 M NaOH and held for 30 min. The suspensions were intermittently stirred during the holding period. Citric acid (15% of weight of dry starch) and sulphuric acid (1% by weight of dry starch) were carefully added to distilled water to make 100 mL acidic solution which was then added to alkaline suspensions. The mixtures were left for 5 h at prevailing laboratory condition. The mixtures were then washed with 600 mL distilled water, filtered to remove excess liquid and dried in a convective hot air oven at 50 °C for 24 h. They were then milled, packed and stored as indicated above.

2.3. Scanning electron micrograph

Thin layer of starch samples was mounted on stubs with the aid of double-sided adhesive tape and sputter-coated with gold using EIKO IB-3 ion coater (EIKO Engineering, Hitachinaka, Japan). Morphology of starch granules was thereafter captured with a scanning electron microscope (EVO LS 15, ZEISS International, Oberkochen, Germany). Sizes of the granules were measured using an image analysis software (AnalySIS, Soft Imaging System, Berlin, Germany).

2.4. X-ray diffraction

An X-ray diffractometer (D8 Advance, BRUKER AXS, Germany) coupled with a sample changer and image plate detector was used to capture the diffraction patterns of the starches. The scanning was registered at Bragg angle (2Θ) 3° to 40° at scan step 0.035° and step time 0.5 s. Multi peak fittings to get integrated areas of crystalline peaks (*Ac*) and amorphous peaks (*Aa*) were done using EVA software (BRUKER, Germany). Crystallinity index (*Xc*) was calculated as below

$$Xc(\%) = \frac{100Ac}{(Ac + Aa)} \quad (1)$$

2.5. Infrared spectra

Infrared (IR) spectra of native and modified starches were obtained with the use of Fourier Transform Infrared (FTIR) spectrometer (Spectrum 100 series, Perkin Elmer, Beaconsfield, UK). The wavelength range was 4000 to 380 cm^{-1} at resolution of 4 cm^{-1}.