American Journal of Food Technology

ISSN 1557-4571

Academic Journals Inc.

www.academicjournals.com

American Journal of Food Technology 7 (7): 386-397, 2012
ISSN 1557-4571 / DOI: 10.3923/ajft.2012.386.397

The Yield Improvement of Resistant Starches from Africa Locust (*Parkia biglobosa*): The Influence of Heat-moisture, Autoclaving-cooling and Cross-linking Treatments

[1,2]Abdoulaye Sankhon, [1]Wei-Rong Yao, [1]Heya Wang, [1]He Qian and [2]Moustapha Sangare
[1]School of Food Science and Technology, Jiangnan University, Lihu Road 1800, P.O. Box 214122 Wuxi, Jiangsu Province, P.R. China
[2]Departement Chimie Faculte des Sciences de la Nature Universite Julius Nyerere de Kankan, Guinea

Corresponding Author: Abdoulaye Sankhon, School of Food Science and Technology, Jiangnan University, Lihu Road 1800, P.O. Box 214122 Wuxi, Jiangsu Province, P.R. China Tel: +86 510 85328726 Fax: +86 510 85329081

ABSTRACT

Native paprika starch was treated by heat-moisture treatment (HMT), autoclaving-cooling (Aut-cd) with and without anhydride acetic acid/adipic acid (Aut-acid) and sodium trimetaphosphate/sodium (STMP/STPP) tripolyphosphate cross-linking to produce Resistant Starch (RS). The resistant starch yield, crystalline structure, as well as other physicochemical properties of the modified starch was investigated. The yield of resistant starch was 36.33 and 50.16%, by HMT and Aut-col, respectively, whereas 48.56 and 50.16%, respectively were obtained by Aut-acid and STMP/STPP. The scanning electron microscopy images clearly illustrated that the granular structure of native parkia starch was disrupted and a continuous network with irregular shape was formed after HMT and autoclaving-cooling cycles. The subsequent chemical cross-linking appeared to make the network structure more compact and dense. X-ray diffraction patterns showed that B- and V types coexisted in all the modified parkia starches and the DSC results also showed that the formation of amylose double helices could contribute to the changes in the X-ray diffraction patterns. The RS-rich powder presented crystalline due to the process of heat moisture and autoclaving-cooling cycles with cross-linking treatment induced starch retrogradation. The procedure proposed might be used for production of a RS-rich powder from parkia starch with high resistant starch level and functional properties.

Key words: Native parkia starch, heat-moisture, autoclave-cooling, cross-linking, resistant starch

INTRODUCTION

The Resistant Starch (RS) represents one of the most important functional ingredients that have recently emerged from food science and nutrition. It has been generally defined as the fraction of dietary starch that is not digested in the small intestine of healthy individuals (Koksel *et al*., 2008; Lee and Shin, 2006; Sajilata *et al*., 2006). Non-viscous resistant starch; shares some of the physiological properties of non-starch polysaccharides and have similar metabolic effects which may be of importance in the dietary control of certain disease conditions (Bobboi *et al*., 2004). Resistant starch (RS) is desirable in the human diet principally because of its prebiotic effects and associated health benefits for the colon. The high levels of butyric acid in the

proximate colon where most fermentation of RS occurs may explain the prevalence of cancer and ulcerative colitis in the distal colon (Topping and Clifton, 2001). Starches rich in RS also contain elevated levels of slowly digestible starch and reduced levels of rapidly digestible starch (Vonk *et al*., 2000). Foods with a low glycemic index improve satiety, may help prevent obesity and Type- II diabetes (Roberts, 2000; Warren *et al*., 2003). The starch blocking stability of speckled kidney beans (*Phaseolus vulgaris*) alpha-amylase inhibitor (a-AII) for application as a nutraceutical additive against diabetes and obesity was assessed reported by Jiang *et al*. (2009). The reported satiety caused by RS may be explained by its low glycemic index. It is concluded that resistant starch diet modifies carbohydrate and lipid metabolism and this may be of clinical significance especially in the control of hyperglycemic and hyperlipidaemic condition (Bobboi *et al*., 2003). RS has been divided into four categories which are type-1, -2, -3 and -4 for physically inaccessible starch, raw crystalline starch, retrograded starch and chemically modified starch, respectively (Khondkar *et al*., 2009). Among the four types of RS, the type-3 is of great interest due to its thermal stability during food processing (Kim and Kwak, 2004). It was previously reported that RS type-3 could be produced by physical treatments including autoclaving (Aparicio-Saguilan *et al*., 2005; Onyango *et al*., 2006) and extrusion (Unlu and Faller, 1998). On the other hand, RS type-4 could be produced by chemical reactions, in particular, cross-linking where sodium trimetaphosphate/sodium tripolyphosphate (STMP/STPP), or a mixture of acetic anhydride and adipic acid have been used as cross-linking agents (Sajilata *et al*., 2006).

However, in my research, heat-moisture treatment, autoclaving-cooling and cross-linking method were applied to obtain RS and improve the yield of resistant starches .Therefore, in this study, parkia starch was subjected to heat-moisture treatment, autoclaving-cooling treatment repeated and subsequent cross-linking reactions with acetic acid/adipic acid and sodium trimetaphosphate/sodium tripolyphosphate . Then, the contents of RS produced were examined and the physicochemical properties were also characterized.

MATERIAL AND METHODS

Africa locust bean (Parkia biglobosa) seeds were purchased from Madinah local market (Conakry, Guinea) in March 2011. The sample was shipped down to Wuxi, China through TNT® 84 mailing company (No. GD923580841WW).

Porcine (pancreatic α-amylase, amyloglucosidase) were purchased from Sigma-Aldrich (shanghai) and were used for analyzing the content of RS. The other chemicals sodium trimetaphosphate/sodium tripolyphosphate and anhydride acetic acid/adipic acid were purchased from Sinopharm Chemical Reagent Co. Ltd. and all other reagents used were of analytical grade.

Preparation of starch powder: The isolation of starch from Parkia biglobosa seed was performed according to the method of Sira and Amaiz (2004) with slight modification. Visible dirt and contaminants were removed from the dark-colored Parkia seed (1 kg) which was then steeped in a solution of sodium hypochlorite (35 g) and potassium hydroxide (50 g) in water (2 L) at room temperature (28°C) for 3 h. The pH of the steep solution was elevated to 9 and the mixture was maintained at 100°C in a thermostat water bath for 3 h. Then, the solution was drained and the seeds were immersed in water and left overnight at ambient temperature. Finally, the seeds were thoroughly washed, manually dehulled and the cotyledon was washed repeatedly until the wash

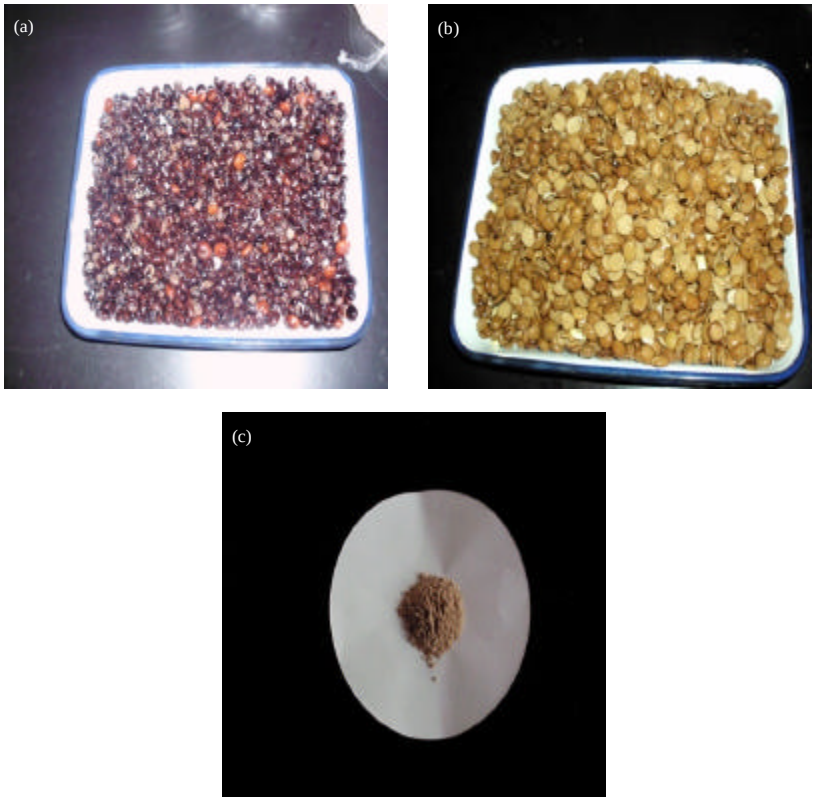

Fig. 1(a-c): *Parkia biglobosa* seeds (a) Raw, (b) Treated with NaOCl (35 g)+KOH (50 g)+H_2O (2 L) at room temperature (28°C) for 3 h, The pH of the steep solution was elevated to 9 and the mixture was maintained at 100°C in a thermostat water bath for 3 h, (c) The resultant starch was dried at 60°C in a hot air oven, then grounded to powder using a mortar and pestle and stored in cellophane

pH was neutral. The cotyledon was blended with water for 24 h using a domestic blender. The homogenate was filtered through muslin cloth and the filtrate was allowed to settle overnight. The supernatant was decanted and the sediment was centrifuged at 4500 rpm for 10 min using a ZOPR-52D refrigerated centrifuge (Hitachi Koki Co Ltd., Tokyo, Japan). The sedimented starch was re-suspended in water and the process was repeated six times. The resultant starch was dried at 60°C in a hot air oven, then grounded into powder using a mortar and pestle and stored in cellophane and wrapped before usage Fig. 1.

Heat-moisture treatment (HMT): HMT was performed according to the method of (Sair, 1967) with slight modification for the preparation of heat-moisture treated parkia starch. Starch powder was weighed into glass jars and the moisture level was increased to 16% by adding the appropriate amount of distilled water. The mixture was stirred and the glass jars sealed before equilibration at room temperature (28°C) for 24 h. Then, the jars were placed into air oven for 24 h at 105°C. After cooling at ambient temperature, the jars were opened and the samples were dried at 30°C for 48 h in air oven. Subsequently starch was ground to powder using a mortar and pestle pass through a 100 μm sieve.

Autoclaving-cooling (Aut-col) treatment: Aut-col treatment was performed according to the method of (Sievert and Pomeranz, 1989). Starch dispersion (starch: water 1:4) was autoclaved at 110°C for 120 min and cooled to room temperature (28°C). The gelatinized starch was stored at 5°C for one day which termed as one cycle. Then, three additional cycles were carried out, followed by freeze-drying. After the resistant starch digestibility was analyzed for the treated samples.

Cross-linking with anhydride acetic acid/adipic acid (Aut-acid): Aut-acid parkia starch was prepared according to the method of Kim *et al.* (2010) with little modifications. The lyophilized starch sample (10 g) which was treated with repeated autoclaving-cooling as mentioned above, was suspended in 20 mL distilled water with 0.42 g of NaCl (1.2%, solid basis, sb). After mixing for 15 min, the solution was blended with 0.18 g of H_2O_2 (0.44%, sb) for 15 min at 30°C and the pH was adjusted to 8.2 with 1 M NaOH. Then, the cross-linking agent was prepared by mixing 2.26 g of anhydride acetic acid (5.66%, sb) with 52 mg of adipic acid (0.13%, sb) in a boiling water for 15 min and was slowly added, meanwhile 1 M NaOH was used to keep pH stable at 8.2. After all the cross-linking agent was added within 90 min, the reaction was further continued for 1 h. pH of the reaction mixture was adjusted to 6.0 with 1 M HCl and the final solution was centrifuged (4500 g) for 15 min to remove the supernatant. The obtained modified starch was washed with distilled water (250 mL), then freeze-dried.

Cross-linking with sodium trimetaphosphate/sodium tripolyphosphate (STMP/STPP): STMP/STPP cross-linked parkia starch was prepared according to the method of (Woo and Seib 2002) with little modifications. Ten grams of lyophilized starch sample obtained after repeated autoclaving-cooling was suspended in 20 mL distilled water and sodium sulfate (4 g, 10.0%, sb), STMP (4.752 g, 11.88%, sb), STPP (48 mg, 0.12%, sb) were added. pH of the solution was adjusted to 11.5 by adding 1 M NaOH (about 50 mL) and then, the solution was stirred continuously and warmed to 45°C, for 3 h. After pH was adjusted to 6.5 with 1 M, HCl, the modified starch was collected by centrifugation (4500 g, 15 min) and washed with distilled water (250 mL), then, freeze-dried.

Resistant starch: Procedure has been proposed which is derived from several RS analysis systems (Champ *et al.*, 1997). Its principle is that in-vitro RS is defined as that starch which is not hydrolyzed by incubation with α-amylase. Amyloglucosidase is added to avoid inhibition by by-products of amylase digestion. Hydrolysis products are extracted with 80% ethanol and discarded. The RS is then solubilized with 2 N potassium hydroxide and hydrolyzed with amyloglucosidase. The procedure is relatively simple with no particular training required and is summarized in Fig. 2. Ground in a mincer. The sample must be weighed to contain 50 mg starch.

Scanning electron microscopy (SEM): Examination by SEM was carried out. Samples were gold coated and scanned by using an Electro scan Quanta 200 environmental scanning microscope (Fei Company, Netherlands).

X-ray powder starch diffraction: X-ray Powder Diffraction, a Shimadzu Lab XRD-6000 was used to examine the crystalline property of starch samples (native starches and modified starches).The scanning region of the two (θ) angles was from 2 to 40° which covers all the significant diffraction peaks of starch crystallites.

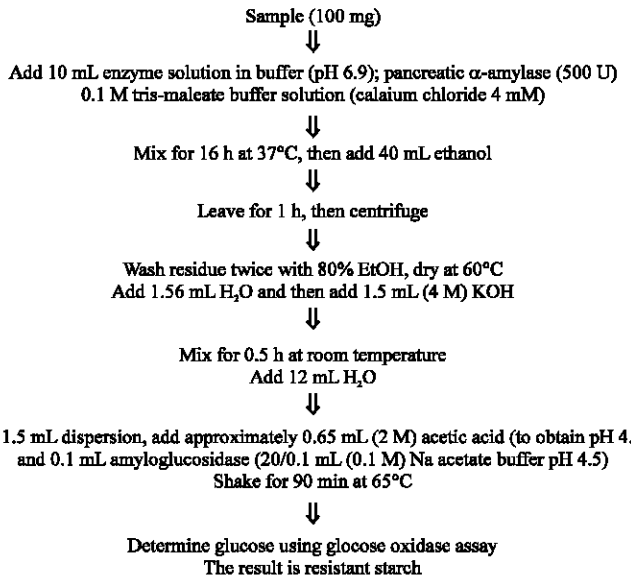

Fig. 2: Method for the determination of resistant starch

Differential scanning calorimetric (DSC): Calorimetric measurements (gelatinization temperature and enthalpy) of the processed parkia starch product were analyzed with the Pyris-1 differential scanning calorimeter (DSC) (PE, USA). Two point six milligrams of sample were weighed directly into DSC aluminum pans and deionizer water, after sealing, the pans were left to equilibrate (24 h) at room temperature (28°C) and then heated from 40 to 120°C at 10°C min^{-1}, An empty pan was used as reference.

Statistical analysis: The test results were processed by using one-way variance analysis (ANOVA). Differences at $p = 0.05$ were considered to be significant. The employed software was (SAS) (version 8.1).

RESULTS AND DISCUSSION

Effect of HMT and Aut-col with cross-linking treatments (Aut-acid and STMP/STPP) on the formation of RS: The yield of RS differed from the type of treatments. As shown in Fig. 3, the resistant starch level of native parkia starch was only 28.96%. Greater than that resistant starch was found in the crackers incorporated with 15 and 20% Unpeeled Pumpkin Pulp Flour (UPPF), with the 20% UPPF crackers having high resistant starch content reported by Aziah *et al.* (2011).

However, it was distinctly increased after the physical and chemical treatments, indicating the lower digestibility of the modified starch samples. After Heat-moisture treatment, autoclaving cooling treatment, autoclaving cooling acetic acid/ acid adipic treatment and autoclaving cooling sodium trimetaphosphate/sodium tripolyphosphate treatment, RS content was significantly increased to 36.33, 43.06, 48.56 and 50.16%, respectively. And when autoclaved at 110°C the starch was completely gelatinized. Amylose was leached from the granules into solution as a random coil polymer, whereas the crystalline regions of clusters of branched amylopectin chains had disappeared. Upon cooling, starch molecules recrystallized and can form tightly packed structures which might be hard to digest by enzymes. Cycling to autoclaving temperature is

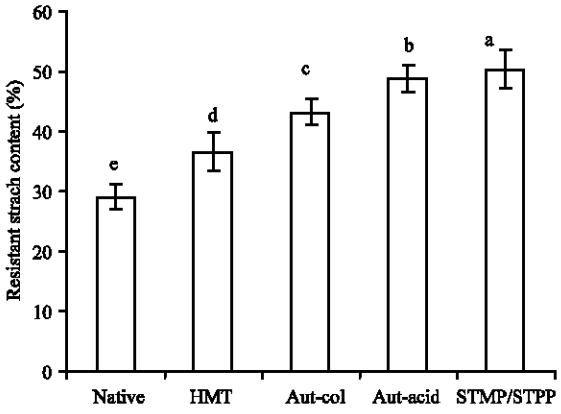

Fig. 3: Resistant starch percentage in native and modified parkia starch

favorable for the formation of extremely stable resistant starch (Sajilata *et al.*, 2006; Haralampu, 2000). This type of resistant starch is regarded as RS type-3. The reported levels of RS produced by autoclaving-cooling treatment were about 10% in cassava starch Onyango *et al.* (2006), 16.5% in amylomaize starch Kim and Kwak (2004) and 19% in banana starch (Aparicio-Saguilan *et al.*, 2005) reported a RS content of 18% in native corn starch, debranched for 4 h with pullulanase and then autoclaved. Digestible corn starch substituted with 18 % resistant starches was produced by autoclaving and cooling cycles reported by Pongjanta *et al.* (2009). Higher resistant starch was found in the crackers incorporated with 15 and 20%.

The RS concentration increased when debranching time increased to 48 h. With high amylose corn starch, the RS content of 36.4% was obtained by an autoclaving process. Similar as Corn starch (Hylon VII) was simultaneously heat-moisture treated and phosphorylated/cross-linked with a 99/1 (w/w) mixture of sodium trimetaphosphate/sodium tripolyphosphate reported by Yijin sang and Paul (2006). Cooked Hylon VII45-10 (43%) had a significantly higher level of RS than cooked Hylon VII (29), Hylon VII15-6 (32%), Hylon VII45-0 (32%) and Novelose 240 (29%). Novelose 240 is a commercial sample of RS made by heat-moisture treatment of Hylon VII. Cooked Hylon VII45-10 had about 35% more RS than either Hylon VII45-0 or Hylon VII15-6 and 48% more RS than the commercial RS Novelose 240.These values are less than the ones obtained in the present study.

Furthermore, chemical modifications by cross-linking with acetic acid/ acetic and STMP/STPP increased the yield of resistant starch up to (48.56; 50.16%).while, heat-moisture treatment and cross-linking with acetic acid/acid adipic and STMP/STPP after autoclaving-cooling cycles increased the yield of RS from 36.33% to above 50.16%.

Research with resistant starch is exciting because of so many possible health benefits. They include improved glucose regulation and better weight control, reduced constipation, reduced colon cancer risk and reduced blood cholesterol and triglycerides. Resistant starch has a low glycemic index. This may lower insulin demand by the body and benefit diabetics as they try to regulate their blood glucose within a normal range. As shown in Fig. 3, heat-moisture and autoclave - cooling cycles increased the RS content. Chung *et al.* (2009) stated that the amylose-amylose interactions which are much stronger than those of amylose-amylopectin or amylopectin-amylopectin, may have continued to exist after gelatinization and thereby partly restricting accessibility of starch chains to the hydrolyzing enzymes.

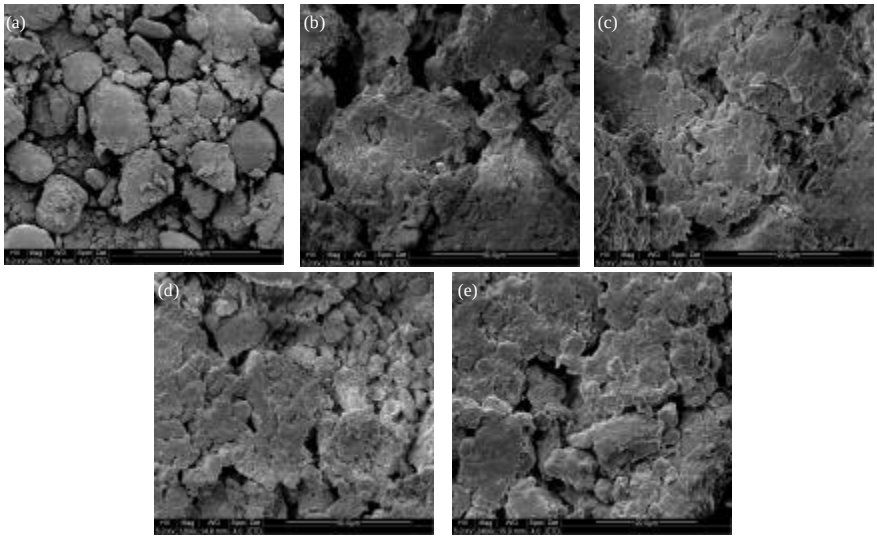

Fig. 4(a-e): Scanning electron micrographs of parkia starch; (a) Native, (b) HMT, (c) Aut-col, (d) Aut-acid, (e) STMP/STPP

Morphological characteristics by SEM: The SEM images of modified parkia starch samples were obtained and compared with that of native parkia starch. Figure 4 clearly illustrated that the modification of native parkia starch by heat-moisture treatment and autoclaving cooling cycles, cross-linking treatment altered the starch structure. While native starch exhibited a granular appearance (Fig. 4a), the granular structure disappeared and a continuous network with irregular shape was formed in the physically modified starch (Fig. 4b, c). On the other hand, the use of cross-linking agents appeared to make the starch structure more compact and dense. And it also looked like that the structure of starch cross-linked by acetic acid/acid adipic which was similar to that of starch modified by STMP/STPP (Fig. 4d, e).

These changes were attributed to interplay of factors such as: (1) amylose content, (2) interactions between starch chains, (3) arrangement of amylose chains within the amorphous domains and (4) lipid-amylose complexes. The morphology of chemically modified granules depends on botanical source of starches. Singh *et al.* (2004) reported that corn starch granules showed a higher resistance towards acetylation than potato ones. Most of the structural changes upon hydroxypropylation take place at the relatively less organized central core region of the starch granule, i.e., where the hydroxypropyl groups are most densely deposited (Kim *et al.*, 1992). Density of the amylopectin break down product after SCD (supercritical carbon dioxide) extraction, showed similar trends to amylopectin molecules, indicating aggregation, or completing with other molecules, or less degradation was also occurring (Stevenson *et al.*, 2007). However this change was also observed in the case of parkia starch. Likewise, Lawal and Adebowale (2005) working on acetylated jack bean starch reported no significant differences in shape and appearance of native and modified starch derivatives; this is probably attributed to the physiology of the starch granules and the modification level.

The role of anomalous amylopectin contributions in the control of enzyme resistance explains the value of parkia starches as raw materials used in autoclaving-cooling processes for

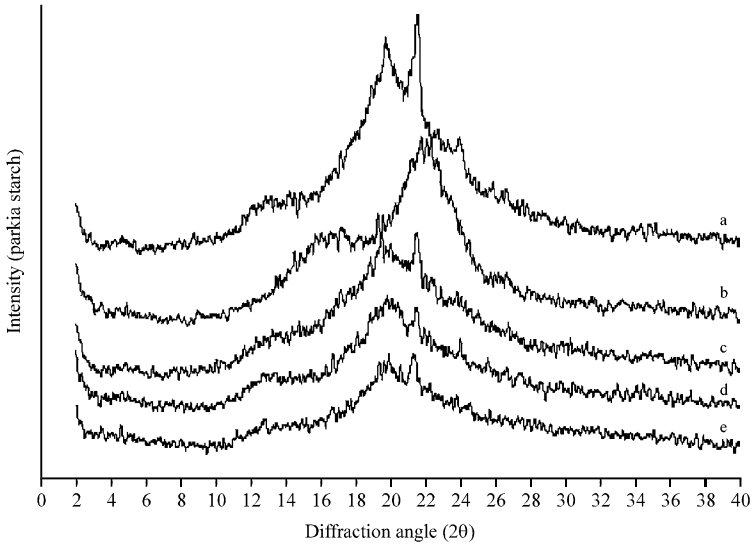

Fig. 5: Effect of different treatments on the X-ray diffraction spectra of parkia starch, (a) Native, (b) HMT, (c) Aut-col, (d) Aut-acid, (e) STMP/STPP

the training of RS. Increased RS content (Fig. 4). In this study, crystalline perfection (Fig 4a-e) and amylose-amylose and/or amylose-amylopectin interactions should have theoretically increased RS level. A similar observation was reported by Chung *et al.* (2009) on corn starch.

X-ray powder starch diffraction: The crystalline types of native and modified parkia starches were investigated based on X-ray diffraction patterns (Fig. 5). All parkia starches showed the characteristic C type pattern of legume starches which is mixture of A and B type starch (Hoover and Ratnayake, 2002). Native parkia starch showed a typical A-type pattern with strong reflections at 2θ degrees about three small peaks (5 and 16) two big peaks (19 and 22).

A similar observation was reported by Pongjanta *et al.* (2009) shown that after debranching and Retrogradation, the high amylose rice starch molecules had rearranges and changes their crystal pattern from A to V-type pattern, as revealed by X-ray diffraction analysis. After the heat -moisture treatment and autoclaving-cooling cycles, the strongest peak at around (19 and 21°C) was observed and three small peaks at around 4 and 13°C(c), 3.5 and 13°C (d), 3 and 13°C (e) were generated, exhibiting the typical characteristics of B-type starch. In addition to B-type crystallinity, an additional peak at 22°C was clearly observed, indicating the presence of V-type crystallinity in the modified parkia starch X-ray diffraction similarly reported by Lee and Shin (2006). The coexistence of B- and V-type was also found in either acid hydrolyzed or autoclaving-cooling treated high amylose parkia starches, comparable with corn starch by Sievert and Pomeranz (1989), Chung *et al.* (2003). The cross-linked starch samples by acetic acid/adipic acid and STMP/STPP also exhibited similar X-ray diffraction patterns to the autoclaving-cooling treated corn starch.

Thermal analysis: In principle, fully gelatinized starch should produce a flat straight line with no absorption peak in DSC analysis. However, starch molecules rearrange and retrograde to form many crystal-like structures; breaking these crystal structures to re-solubilize starch molecules requires external energy. The gelatinization transition temperature [onset temperature (To), peak

Table 1: Effect of autoclaving-cooling and cross-linking treatments on the thermal properties of parkia starch

Samples	T_o (°C)	T_p (°C)	ΔH, (j/g)
Native parkia starch	83.65±3.03^{d}	84.31±2.18^{c}	6.56±2.45^{o}
HMT	69.22±2.04^{i}	89.31±3.12^{b}	13.45±2.45^{k}
Aut-col	64.11±4.12^{j}	69.62±4.03^{g}	9.28±3.36^{m}
Aut-acid	69.35±2.03^{h}	76.13±3.08^{e}	11.21±2.26^{l}
STMP/STPP	72.56±3.23^{f}	89.45±2.18^{a}	7.88±2.36^{n}

Values are means of three measurements±standard deviation, Mean values in the same column with different letters are significantly different ($p<0.05$)

temperature (Tp) and the enthalpy of gelatinization (ΔH) of native and modified starches are presented in (Table 1). It is suggested that, the endothermic transitions in the range of 42 and 72°C are probably due to melting of retrograded amylopectin, whereas other endothermic transitions appeared in approximately 120 to 170°C owing to melting of retrograded amylose (Sievert and Pomeranz, 1990). The rice starch was also acid modified and acetylated. Only endothermic change was noticed in zein protein, but on combination with starch and acetylation, the endothermicity decreased (Singh *et al*., 2007). The native parkia starch exhibited a clear gelatinization peak at 84.31°C with an endothermic enthalpy of 6.56 J g^{-1} (Table 1), whereas the modified starches suspended in water produce a flat straight line with no absorption peak in DSC analysis it was very difficult to identify the real peaks (data not shown). The gelatinization temperature range of native parkia starch (83.65-84.31) is comparable to those of other tuber starches, such as potato (Collado *et al*., 1999), cassava (Valetudie *et al*., 1995) and new cocoyam (Shi and Seib, 1992). Unlike that the high λ_{max} (of iodine complex of native) Fr-IIB indicated that carbohydrate content corresponding to this peak represented long linear chain fraction probably coming from external chains of the amylopectin molecule (Singh and Ali, 2006). The onset temperature for melting retrograded amylopectin is in the range of 69.62 to 89.45°C. In addition, (Table 1) shows that after parkia processing, its (ΔH, 6.56 to 13.45) increased in parallel with its content of RS which suggested that the decreased digestibility of the final parkia starch product may be attributed to retrograded amylopectin. Similar thermograms have been observed by Kasemsuwan *et al*. (1995) when they analyzed the retrogradation properties of high amylose 50 and 70% starches after storage at 4°C for 7 days. It has been rapport that the weaknesses of cassava starch as the raw material in food industries are low protein content, the ununiform of viscosity and gel forming ability, nos resistant to acidic condition and mechanical processes and prone to be syneresis (Julianti *et al*., 2011).

CONCLUSION

RS was prepared by subjecting native parkia starch to heat moisture and autoclaving-cooling cycles followed by cross-linking modifications. The effect of heat -moisture treatments and autoclaving-cooling followed by cross-linking significantly affected the formation of resistant starch, increasing the RS content up to maximal 50.16%.

The DSC also showed that the formation of amylose double helices could contribute to the changes in the X-ray diffraction patterns. Even though an experimental procedure to increase the content of RS and its physicochemical properties were suggested in this study, further textural and rheological studies would be necessary for the use of the produced RS as a possible functional ingredient in food products.

ACKNOWLEDGMENTS

This article is supported by 111 project-B07029 and PCSIRT0627. The authors are grateful to Fatoumata Sylla Manager Ministry of Foreign Affairs, Conakry, guinea who sent the sample down to Wuxi, China.

REFERENCES

Aparicio-Saguilan, A., E. Flores-Huicochea, T. Tovar, F. Garcia-Suarez, F. Guiterrez meraz and L.A. Bello-Perez, 2005. Resistant starch-rich powder prepared by autoclaving of native and lintnerized banana starch: Partial characterization. Starch Starke, 57: 405-412.

Aziah, A. A.N., L.H. Ho., C. A. Komathi and R. Bhat, 2011. Evaluation of resistant starch in crackers incorporated with unpeeled and peeled pumpkin flour. Am. J. Food Technol., 6: 1054-1060.

Bobboi, A.A., J.L. Yefon and A.A. Gidado, 2004. Comparative studies of non-digestible polysaccharides: Wheat and potato resistant starch and pectin on glycemic, lipemic, blood urea and intestinal parameters in growing rats. J. Medical Sci., 4: 331-339.

Bobboi, A.A., U.A. Ismaila, S.H. Margaret, A.Y. Hajjara and G. Aboubakar, 2003. Changes in carbohydrates and lipid metabolisms and gastrointestinal function parameters in rats fed amylase resistant corn starch. J. Med. Sci., 3: 298-306.

Champ, M., L. Noah., G. Loizeeau and F. Kozlowski, 1997. Definition, Functionality and Analysis. In: Complex Carbohydrates in Foods, Cho, S., L. Prosky and M. Dreher (Eds.). Marcel Dekker, Inc., New York, Basel, pp: 157-168.

Chung, H.J., H.Y. Jeong and S.T. Lim, 2003. Effects of acid hydrolysis and defatting on crystallinity and pasting properties of freeze-thawed high amylose corn starch. Carbohydrate Polym., 54: 449-455.

Chung, H.J., Q. Liu and R. Hoover, 2009. Impact of annealing and heat-moisture treatment on rapidly digestible, slowly digestible and resistant starch levels in native and gelatinized corn, pea and lentil starches. Carbohydrate Polym., 75: 436-447.

Collado, L.S., R.C. Mabesa and H. Corke, 1999. Genetic variation in the physical properties of sweet potato starch. J. Agric. Food Chem., 47: 4195-4201.

Haralampu, S.G., 2000. Resistant starch: A review of the physical properties and biological impact of RS_3. Carbohydr. Polym., 41: 285-292.

Hoover, R. and W.S. Ratnayake, 2002. Starch characteristics of black bean, chickpea, lentil, navy bean and pinto bean cultivars grown in Canada. Food Chem., 78: 489-498.

Jiang, B., Z. Tao and C. Wokadala Obiro, 2009. Starch blocking stability of the *Phaseolus vulgaris* Alpha-Amylase inhibitor (α-AI1). Am. J. Food Technol., 4: 9-19.

Julianti, E., Z. Lubis, Ridwansyah, E. Yusraini and I. Suhaidi, 2011. Physicochemical and functional properties of fermented starch from four cassava varieties. Asian J. Agric. Res., 5: 292-299.

Kasemsuwan, T., J. Jane, P. Schnable, P. Stinard and D. Robertson, 1995. Characterization of the dominant mutant amylase- extender (Ael-5180) maize starch. Cereal Chem., 72: 457-464.

Khondkar, D., R.F. Tester and J. Karkalas, 2009. Effect of cross-linking on the resistance to enzymatic hydrolysis of waxy maize starch and low-methoxy pectin. Food Hydrocolloids, 23: 387-393.

Kim, H.R., A.M. Hermansson and C.E. Eriksson, 1992. Structural characteristics of hydroxypropyl potato starch granules depending on their molar substitution. Starch - Starke, 44: 111-116.

Kim, N.H., J.H. Kim, S. Lee, H. Lee, J.W. Yoon, R. Wang and S.H. Yoo, 2010. Combined effect of autoclaving-cooling and crosslinking treatments of normal corn starch on the resistant starch formation and physicochemical properties. Starch-Starke, 62: 358-363.

Kim, S.K. and J.E. Kwak, 2004. Estimation of resistant starch content of high-amylose corn starch. Food Sci. Biotechnol., 13: 71-74.

Koksel, H., S. Ozturk, K. Kahraman, A. Basman, O.O. Ozbas and G.H. Ryu, 2008. Evaluation of molecular weight distribution, pasting and functional properties and enzyme resistant starch content of acid-modified corn starches. Food Sci. Biotechnol., 17: 755-760.

Lawal, O.S. and K.O. Adebowale, 2005. Physicochemical characteristics and thermal properties of chemically modified jack bean (*Canavalia ensiformis*) starch. Carbohydrate Polymers, 60: 331-341.

Lee, H. and M. Shin, 2006. Comparison of the properties of wheat flours supplemented with various dietary fibers. Food Sci. Biotechnol., 15: 746-751.

Onyango, C., T. Bley, A. Jacob, T. Henle and H. Rohm, 2006. Influence of incubation temperature and time on resistant starch type III formation from autoclaved and acid-hydrolysed cassava starch. Carbohydrate Polym., 66: 494-499.

Pongjanta, J., A. Utaipattanaceep, O. Naivikul and K. Piyachomkwan, 2009. Effects of preheated treatments on physicochemical properties of resistant starch type III from pullulanase hydrolysis of high amylose rice starch. Am. J. Food Technol., 4: 79-89.

Roberts, S.B., 2000. High-glycemic index foods, hunger and obesity: Is there a connection? Nutr. Rev., 58: 163-169.

Sair, L., 1967. Heat-moisture treatment of starch. Cereal Chem., 44: 8-26.

Sajilata, M., R.S. Singhal and P.R. Kulkarni, 2006. Resistant starch: A review. Compre. Rev. Food Sci. Food Saf., 5: 1-17.

Shi, Y. and P.A. Seib, 1992. The structure of four waxy starches related to gelatinization and retrogradation. Carbohydr. Res., 227: 131-145.

Sievert, D. and Y. Pomeranz, 1989. Enzyme-resistant starch. I. Characterization and evaluation by enzymatic, thermoanalytical and microscopic methods. Cereal Chem., 66: 342-347.

Sievert, D. and Y. Pomeranz, 1990. Enzyme-resistant starch. II. Differential scanning calorimetry studies on heat-treated starches and enzyme-resistant starch residues. Cereal Chem., 67: 217-221.

Singh, N., D. Chawla and J. Singh, 2004. Influence of acetic anhydride on physicochemical, morphological and thermal properties of corn and potato starch. Food Chem., 86: 601-608.

Singh, V. and S.Z. Ali, 2006. *In vitro* hydrolysis of starches by α-amylase in comparison to that by acid. Am. J. Food Technol., 1: 43-51.

Singh, V., S. Isobe, H. Okadome and K. Ohtsubo, 2007. Preparation of high degree substituted acetylated acid modified rice starches, intermediate degree substituted derivatives of rice starch and zein proteins and preparation of biodegradable films (Part II). Trends Applied Sci. Res., 2: 103-114.

Sira, E.E.P. and M.L. Amaiz, 2004. A laboratory scale method for isolation of starch from pigmented sorghum. J. Food Eng., 64: 515-519.

Stevenson, D.G., F.J. Eller, J.L. Jane and G.E. Inglett, 2007. Starch structures and physicochemical properties of a novel β-glucan-enriched oat hydrocolloid product with and without supercritical carbon dioxide extraction. Am. J. Food Technol., 2: 248-256.

Topping, D.L. and P.M. Clifton, 2001. Short chain fatty acids and human colonic function: Roles of resistant starch and non starch polysaccharides. Physiol. Rev., 8: 1031-1064.

Unlu, E. and J.F. Faller, 1998. Formation of resistant starch by a twin-screw extruder. Cereal Chem., 75: 346-350.

Valetudie, J.C., L. Guadeloupe, P. Colonna, B. Bouchet and D.J. Gallant, 1995. Gelatinization of sweet potato, tania and yam tuber starches. Starch - Starke, 47: 298-306.

Vonk, R.J., R.E. Hagedoorn, R. de Graaff, H. Elzinga, S. Tabak, Y.X. Yang and F. Stellaard, 2000. Digestion of so-called resistant starch sources in the human small intestine. Am. J. Clin. Nutr., 72: 432-438.

Warren, J.M., C.J.K. Henry and V. Simonite, 2003. Low glycemic index breakfasts and reduced food intake in preadolescent children. Pediatrics, 112: e414-e419.

Woo, K.S. and P.A. Seib, 2002. Cross-linked resistant starch: Preparation and properties. Cereal Chem., 79: 819-825.