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Research Article

Comparison of Physicochemical Characteristics of Starch Isolated from Sweet and Grain Sorghum

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The worldwide interest about sweet sorghum (*Sorghum bicolor* L. Moench) goes towards stem sugar, but little has been focused on its grain. The starches were isolated from the grains of eight sweet and four grain sorghum varieties, and their physical, chemical, and morphological properties were carefully compared. The results reflected that starch from sweet varieties usually had larger granule size than that from grain ones, especially from two sweet varieties GL-4 and GL-6 with the granule size of 15.49 μm and 15.67 μm, respectively. The amylose content of sweet varieties starch was lower than that of grain ones. For water solubility index, starch from sweet varieties ranked top, whereas that from grain varieties ranked top for swelling power. The starch from both sweet and grain had A-type crystalline pattern, while the data from ^{13}C NMR reflected pattern differences for C_1 and C_6 resonance between sweet and grain varieties. Chains length distribution from sweet varieties debranched starch was found a little different from grain one. The starch particles surface of sweet sorghum was smooth with some dents, while that from grain was smooth without appearance of dents. As sweet sorghum has ability to withstand harsh environments where other crops do not and is characterized by low production cost, the extensive potential existed for starch from sweet varieties to be used in starch industries.

1. Introduction

Sorghum (*Sorghum bicolor* L. Moench), mostly cultivated across the world in the warmer climatic areas, is quantitatively the world's fifth largest most important cereal grain after wheat, maize, rice, and barley [1]. Sorghum is usually classified into grain and sweet types according to their final utilization. The grain sorghum is mainly used as a principal food in tropical areas and sometimes as raw materials for alcoholic beverages, while the sweet type serves as a material for sweetener syrup [2, 3]. Taylor et al. (2006) mentioned that sweet sorghum thrived better under drier and warmer conditions than many other crops and is grown primarily for forage, silage, and syrup production due to the high biomass and sugar content [1]. Elkhalifa and Bernhardt (2013) defined sweet sorghum as a variety of common grain sorghum and the essence of sweet sorghum is not from its seed but from its stalk [2]. Most of the sweet sorghum variety is with low grain weight per spike and grain yield, and there is a significant negative relationship between grain yield and stalk sugar [4]. As more than 800 million people suffer from hunger and malnutrition in Africa, Asia, Latin America, and even Europe and USA, development of sweet sorghum will play an important role in promoting the development of agricultural production, livestock husbandry, and energy sources.

Among carbohydrate polymers, starch is currently enjoying attention owing to its usefulness in different food products. Sorghum, like other cereals, is rich in starch, a major storage form for carbohydrates, which makes up about 60–80% of normal kernels and has an excellent potential for industrial applications [5, 6]. Starch is composed almost entirely of the polysaccharides amylose and amylopectin. The physical arrangement of amylose and amylopectin and the interaction between starch molecules and other food components determine the physicochemical and functional properties of starch. These properties affect the quality of

starch based products and are essential to determine potential applications of starch [7]. Udachan et al. (2012) clarified that one way of using surplus sorghum is by way of producing starch and starch based sweeteners; the process is likely to be economical as sorghum is available on the large scale with low cost [8]. Singh et al. (2011) reported that sorghum starch had an excellent potential for global industrial applications. Therefore, further characterization is needed to understand the difference of physiochemical and functional properties between starches from sweet and grain sorghum starch [9]. For measurement of thermal properties, the differential scanning calorimetry (DSC) has been proven to be an extremely valuable tool to quantify the gelatinization of starch and has been widely used to study the thermal behaviors of starches [10–13]. The gelatinization of starch is very important in food processing and has been extensively studied in food science for decades [14–18], in particular with higher water content as mentioned by Liu et al. (2006) [18].

Much of the information about the crystalline properties of starch granule had been acquired from X-ray powder diffraction studies. According to several studies, starch could be classified into A, B, and C forms. In the native granular forms, the A pattern is associated mainly with cereal starches, while the B form is usually obtained from tuber starches. The C pattern is a mixture of both A type and B type but also occurs naturally, for example, smooth-seeded pea starch [19]. A widely accepted model of a typical cereal starch granule involves alternating amorphous and crystalline lamellae, in which the two main components, amylose and amylopectin, are embedded.

In recent years, ^{13}C solid-state Nuclear Magnetic Resonance (^{13}C-NMR) has been widely used to study native products because it can observe structural changes of starch samples in the solid, and it is essentially nondestructive. Although X-ray powder diffraction can monitor crystal structure and relative amounts of crystalline and amorphous phases in starch, it is only sensitive to long-range order, while NMR is sensitive to short-range order as discussed by Saitô et al. (1991), being especially suitable for fewer crystalline samples [20]. Combination of both techniques has provided much important information about secondary structure and molecular order of a number of molecular systems [21, 22], and the starch profiles of ^{13}C-NMR were also well studied by many researchers like Gidley and Bocick (1985) and Xu and Seib (1997) [21, 23].

Gel permeation chromatography (GPC) is a common technique to analyze starch structure including size exclusion chromatography as mentioned by Syahariza et al. (2010) and Yoo and Jane (2002) [24, 25], and the organic solvent most commonly used to dissolve starch is dimethyl sulfoxide (DMSO). DMSO disrupts the intramolecular and intermolecular noncovalent interactions in starch molecules by forming hydrogen bonding between its negatively charged oxygen atom and the hydroxyl groups on starch molecules as well as by forming hydrophobic interactions with starch through its methyl group [26].

All above-mentioned techniques have been introduced to test the grain sorghum starch, but the fine molecular structure of sweet sorghum starch and its physicochemical properties have not been fully investigated. Thus, in present study, eight varieties characterized as sweet type and other four characterized as grain type were used and their physicochemical characteristics were compared after investigation by the above-mentioned techniques. Such information will help identify uses for these sorghum starches in food and other industrial applications.

2. Materials and Methods

2.1. Plant Materials. Twelve sorghum genotypes from both China and Sudan were used in this study (Table 1). Among them, eight are sweet types, while four are grain types, and all were planted in the summer of 2013 in the experimental farm of Yangzhou University (32°N, 119°E) in Yangzhou, Jiangsu province, China, and the mature grains were harvested for following characterizations.

2.2. Starch Isolation. Sorghum starch was isolated from polished sorghum grain by an alkaline protease method with slight modifications [27]. The polished sorghum grains were soaked in three volumes of double distilled water, adjusted to pH 8.0–8.5 with 1.0 M NaOH, and held overnight. The supernatant was discarded, and three volumes of 0.001 M NaOH were added. The soaked grains were ground in a Warring blender (Blender ULTRA-TURRAX, T25 basic, IKA-WERKE, Guangzhou, China) at the medium speeds for three min and then a volume of 0.001 M NaOH was added to the slurry up to three volumes. The slurry was adjusted to pH 9.5 with 1 M NaOH by using a pH controller (Sartorius PB-10). Next, 5% of protease (Solarbio Science and Technology Co., Ltd., Beijing) was added and mixed with a magnetic stirring bar for 18 h at 40–45°C. After that, the slurry was passed through a 75 um screen, and the solids retained on the screen were removed. The sediment was centrifuged at 3800 rpm for 20 min at 4°C temperature, and the dark yellow supernatant was discarded. The sediment was washed with double distilled water and centrifuged for 20 min and the dark layer was removed. After the supernatant was discarded, the washing and centrifugation steps were repeated three times. Finally, the starch was freeze-dried for 18 h in the freeze dryer (Christ ALPHA 2-4LD Plus, Germany).

2.3. Amylose Content. Amylose content (AC) of starch was estimated by using the iodine blue value method based on the standard from Ministry of Agriculture, China [28]. The sample (50 mg, dry weight basis, done in triplicate) was dissolved in 0.5 mL of anhydrous ethanol and 4.5 mL of 1 M NaOH in 50 mL vials. The contents of the vials were vigorously agitated and then heated in a boiling-water bath for 20 min (with intermittent shaking). The vials were then cooled to ambient temperature. Distilled water was added up to volume and mixed gently and 5 mL was taken from each sample to opposite 100 mL volumetric flask containing distilled water. Sodium acetate (1.0 mL, 1.0 M) and iodine solutions (0.04%, 750 μL) were added, respectively. Volumetric flask was shaken gently after the distilled water was added up to volume.

Table 1: The information of sorghum varieties used in this study.

Sample symbol	Original name	Type	Country	Source*
WAH	Wad Ahmed	Grain	Sudan	ACSP
ARG	Arfa gadamak	Grain	Sudan	ACSP
TAT	Tabat	Grain	Sudan	ACSP
GL-1	Bai Zhan Gao Liang	Grain	China	ICSCAAS
GL-4	Huang Zhan Gao Liang	Sweet	China	ICSCAAS
GL-6	Beijing Zhan	Sweet	China	ICSCAAS
GL-13	Tian Xuan 33	Sweet	China	ICSCAAS
GL-14	Tian Xuan 184	Sweet	China	ICSCAAS
ZS	Zao Shu	Sweet	China	JCAIAS
YT	Yan Tian	Sweet	China	JCAIAS
T-1	Tian Nong 1	Sweet	China	JCAIAS
ST	St008	Sweet	China	JCAIAS

*ACSP, Arab Company for Seed Production, Khartoum, Sudan. ICSCAAS, Institute of Crop Science, China Academy of Agricultural Sciences, Beijing, China. JCAIAS, Jiangsu Costal Area Institute of Agriculture Sciences, Yan Cheng, Jiangsu, China.

After 15 min, OD was measured at 620 nm (Ultrospec 2000, Pharmacia Biotech, Cambridge, England).

2.4. Gel Consistency. Measurement of starch gel consistency was performed by modifying a procedure described by Cagampang et al. (1973) [29]. The modified procedure consisted of combining 100 mg of starch with 0.2 mL of Thymol blue indicator (0.025%) in 12 cm × 13.35 mm culture tubes oscillated in Vortex Genie2 (Scientific industries, INC, USA). Then 2.0 mL of KOH (0.2 M) was added, and the tubes oscillated again. Very fast the mixture was cooked in a vigorously boiling-water bath (101°C) for 8 min. After cooking, the tubes were allowed to cool in room temperature for 5 min. The mixture was then placed in ice water in an upright position to cool for 20 min. After cooling, the tubes were placed horizontally over ruled logarithmic paper for 1 h at room temperature (25 ± 2°C) and the gel front migration was read to the nearest millimeter. The experiment was done in triplicate.

2.5. Water Solubility and Swelling Power of Starch. The estimation of swelling power (SP) and water solubility index (WSI) of starch was done according to the methods of Radosta et al. (1991) and Tang et al. (2004) with some modifications [30, 31]. A suspension of 0.1 g of starch and 10.0 mL of double distilled water (vortex for 10 sec) was heated in 95°C in a water bath for 30 min (with frequent shake every 2.0 min). The suspension was then cooled rapidly at room temperature (in the ice box) and centrifuged for 2000 rpm for 30 min. WSI is reported as the ratio of dry mater supernatant to dry starch sample, whereas SP is reported as the ratio of swelling starch granule's sediment to dry starch. The experiment was done in triplicate.

2.6. Starch Thermal Properties. The thermal property of starch samples was determined by using a differential scanning calorimeter (DSC 200F3, NETZSCH Company, Germany). The samples (5 mg) with excess water (1 : 2) were heated at 10°C/min from 20 to 120°C. Thermal transitions of samples for gelatinization were characterized by T_o (onset temperature), T_p (peak temperature), T_c (conclusion temperature), and ΔH (enthalpy of gelatinization). The enthalpy calculations were based on dry starch weight. The samples were analyzed twice, and the data were calculated with the software package (DSC 200F3, NETZSCH Company, Germany) [32].

2.7. X-Ray Powder Diffraction. The crystal structure of starch was studied with an X-ray diffractometer (Bruker AXS Model D8 ADVANCE, Germany). Samples were equilibrated in a saturated relative humidity chamber containing access sodium chloride for one week at ambient temperature and dark condition. X-ray diffraction was performed on an X-ray diffractometer with copper-cobalt radiation. Signals of the reflection angle of 2θ, from 3° to 40°, were recorded. The degree of crystallinity of samples was quantitatively estimated following the method of Nara and Komiya (1983) [33]. The ratio of the upper area (peaks area) to total diffraction area was taken as the degree of crystallinity [19, 32].

2.8. NMR Measurements. A solid-state ^{13}C NMR spectrum was measured on the Bruker Avance 500 solid-state NMR spectrometer at 125.8 MHz and 500.2 MHz, respectively. Samples were measured in four mm ZrO_2 rotors with spinning frequencies of eight kHz (^{13}C-NMR). NMR spectra were measured with a contact time of 1 ms and relaxation delay between two consecutive scans of 4 sec. Chemical shifts in the ^{13}C-NMR spectra were referred to the carbonyl line of glycine (with a signal at 176 ppm from TMS) by sample replacement. Spin-lattice relaxation times T1(C) of starch were measured with CP by the method of Torchia (1978) [34]. The experimental scheme with a variable spin-lock time within the range 0.1–10 ms after the proton signal excitation followed by constant contact time was used; the proton spin-locking field in frequency units was 80 kHz [35].

2.9. Morphological Properties of Starch Granules. Scanning electron micrographs (SEM) of starch were obtained with a scanning microscope (Environment Scanning Electron Microscope, model: XL-30ESM, Philips Company, Netherlands). Starch sample was suspended in ethanol (95%) to obtain 1% suspension and mounted on circular aluminum stubs with double-sided sticky tape. The starch granules were evenly distributed on the surface of the tape, and the ethanol was allowed to evaporate. The samples were then coated with gold-palladium (60 : 40), examined, and photographed at an acceleration potential of 10 kV with a magnification of ×1000, ×2000, ×4000, and ×8000 during micrography [36].

2.10. Gel Permeation Chromatography. Starch (10 mg) was mixed in 5 mL of sodium acetate buffer (0.1 M, pH 4.2) (in a 12 mL glass vial with a micro stir bar) and heated in a boiling-water bath for 1 h followed by cooling to room temperature.

The starch was treated with 13.4 μL of β-amylase (19 μ/L, Sigma product number A 7005) for 36 h at 40°C. The mixture was boiled for 10 min to deactivate the enzyme followed by cooling to room temperature. The mixture was kept at −20°C for 3-4 h and then lyophilized in freeze dryer (Christ ALPHA 2-4LD Plus, Germany) according to Asaoka et al. (1985) with some modifications [37]. Four milligrams of debranched starch was mixed with 4 mL dimethyl sulfoxide (DMSO) and stirred in a boiling-water bath for 24 h. The sample was filtered through a 2 μm filter and then 200 μL was injected into a PL-GPC 220 instrument (Polymer Laboratories, Inc., Amherst, MA, USA) equipped with three Phenogel columns of different pore sizes (100 Å, 103 Å, and 105 Å, Phenogel™ GPC, 10 μm, 300 × 7.8 mm), a guard column (Phenomenex, Inc., Torrance, CA, USA), and a differential refractive index detector. The eluent system was DMSO containing 5.0 mM $NaNO_3$ at a flow rate of 0.8 mL/min. The column oven temperature was controlled at 80°C. Standard dextran (American Polymer Standards Co., Mentor, OH, USA) with different molecular weights (MW) was used for MW calibration according to Zhu et al. (2010) [38].

2.11. Statistical Analysis. Data was analyzed using analysis of variance (ANOVA) procedures of the SPSS version 16.0. Variances were considered at a significant level of 95% ($p < 0.05$). Means were compared using Duncan's multiple range test (DMRT) [39]. Unpaired t-test was used to compare differences between means.

3. Results and Discussion

3.1. Amylose Content and Gel Consistency of the Starches. Amylose and amylopectin are important for food and bioindustry applications, as they have distinctively different structures and physicochemical properties. Amylose contents of starch from eight sweet and four grain sorghum varieties were shown in Table 2. Significant differences were observed among tested varieties ($p < 0.05$), and the AC from grain varieties (average 35.61%) was significantly ($p < 0.05$) higher from that of sweet varieties (average 23.52%) (Figure 1(a)). The GL-1 grain variety showed highest AC among all varieties, whereas GL-4 sweet variety showed the lowest AC. Starch from grain varieties had highest AC (ranged from 34.52% to 36.39%) compared with sweet varieties, which ranged from 5.39% to 33.50%. The current results reflected a vast range for AC (5.39–36.39%) compared with that of Udachan et al. (2012) (10.80–18.72%) for their study on four sorghum varieties [8] and Singh et al. (2010) (11.2–22.5%) for their study on sorghum cultivars grown in Indi [40] and Beta et al. (2001) (24.0–33.0%) from 95 Zimbabwean sorghum land races [41]. The vast range of AC observed in the current study was in accordance with that mentioned by Zhu (2014); he noticed existence of great genetic variation in AC of sorghum starch [42], and another study pursued by Hill et al. (2012) revealed a range of AC from 16.1% to 55.8% for 55 sorghum genotypes [43]. Starch with variable AC is of interest because of its utility in different applications and significant effect on the characteristics of final products. Schirmer et al. (2013) postulated that low- and high-amylose starches provide distinctively different structures and physicochemical properties during their various applications [44]. On the other hand, AC in the starch isolated from sweet sorghum juice was 16.4% [45], which was also different from AC of starch isolated from grain. Further efforts to look for the range of AC in sorghum juice in contrast with that of grain and the correlation between them are needed. Lu et al. (2013) reported that high amylopectin grains of waxy sorghum have a high economic value in the food and bioenergy industries because of their increased starch digestibility and higher ethanol conversion rate compared with wild-type sorghum grains [46]. Consequently, in this study, the starch from sweet varieties showed lower AC and higher amylopectin level compared with that of grain varieties, which make them with substantial potential for industrial uses.

Table 2: Amylose content (AC), gel consistency (GC), water solubility index (WSI), and swelling power (SP) for starches from sweet and grain varieties.

Sample	AC (%)	GC (mm)	WSI (%)	SP (g/g)
Grain				
GL-1	36.39 ± 0.15^{a}	118.5 ± 0.35^{bc}	10.0 ± 0^{c}	11.22 ± 0.11^{ab}
WAH	35.25 ± 0.26^{ab}	112.0 ± 0.10^{de}	25.0 ± 5^{bc}	20.07 ± 0.93^{a}
ARG	36.28 ± 0.26^{a}	89.0 ± 0.11^{f}	30.0 ± 0^{bc}	16.21 ± 0.93^{ab}
TAT	34.52 ± 0.40^{ab}	125.5 ± 0.05^{bc}	10.0 ± 0^{c}	12.33 ± 1.89^{ab}
Sweet				
GL-4	5.39 ± 0.18^{e}	129.5 ± 0.05^{b}	95.0 ± 5^{a}	3.50 ± 3.50^{b}
GL-6	6.71 ± 0.11^{e}	157.7 ± 0.11^{a}	95.0 ± 5^{a}	7.00 ± 7.00^{ab}
GL-13	28.41 ± 0.66^{c}	126.0 ± 0.20^{bc}	35.0 ± 5^{bc}	14.85 ± 2.99^{ab}
GL-14	24.38 ± 1.17^{d}	106.5 ± 0.25^{e}	30.0 ± 0^{bc}	17.86 ± 1.57^{ab}
ZS	33.50 ± 0.04^{ab}	121.5 ± 0.05^{bcd}	40.0 ± 10^{bc}	15.69 ± 1.98^{ab}
YT	29.14 ± 1.24^{c}	120.0 ± 0.10^{bcd}	25.0 ± 5^{bc}	15.43 ± 1.43^{ab}
T-1	32.77 ± 0.04^{b}	121.0 ± 0.30^{bcd}	30.0 ± 0^{bc}	15.50 ± 0.21^{ab}
ST	27.82 ± 0.29^{c}	95.0 ± 0.21^{f}	15.0 ± 5^{bc}	11.72 ± 1.16^{ab}

All data represent the mean of three determinations. Means with the same superscript in each column are not significantly different ($p < 0.05$).

As reported previously, AC could play a major role in swelling, pasting properties, and gel firmness of starch [15, 16, 39, 47]. Gel consistency (GC) values ranged from 89.0 mm to 157.67 mm among tested varieties (Table 2). GL-6, a sweet variety, showed the highest GC (157.67 mm), whereas the grain variety ARG showed the lowest GC (89.0 mm). The range for grain varieties was from 89.0 mm to 125.5 mm, whereas for sweet varieties it was from 95.0 mm to 157.67 mm. The values and the range of GC from sweet varieties are generally higher than those from grain ones, while Figure 1(b) shows that the difference was not significant ($p < 0.05$). The highest values for GC observed in this study may be attributed to amylose level as sweet varieties showed lowest values compared with grain ones (Table 2). Furthermore, this notice is in accordance with that mentioned by Lindqvist (1979) and Chanapamokkhot and Thongngam (2007) [39, 47] and Beta et al. (2001) [41]; they noticed that gel hardness was largely determined by the AC of starch.

3.2. Water Solubility Index and Swelling Power. The ability to swell in excess water and solubility of the starches from

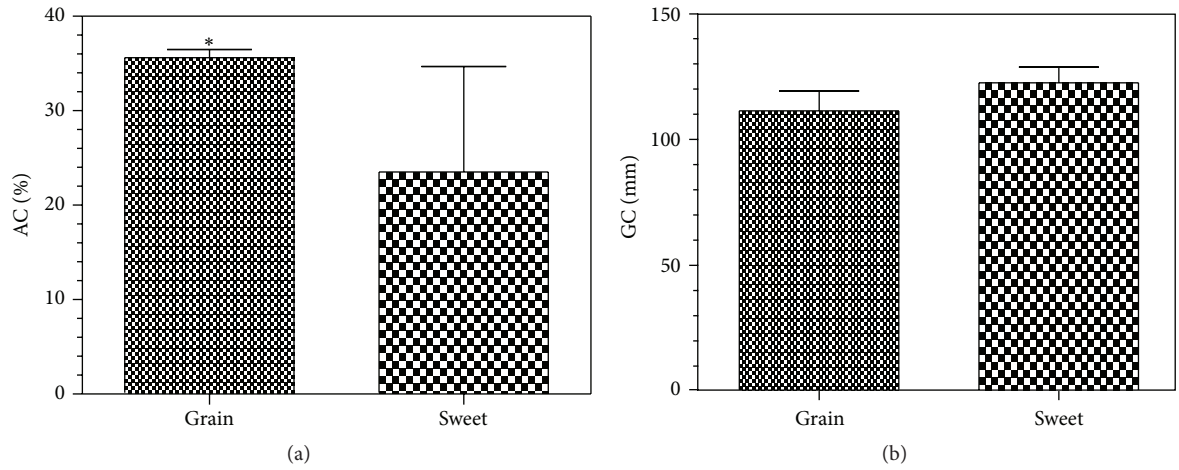


Figure 1: Comparison of the average amylose content (AC) (a) and gel consistency (GC) (b) of starches between sweet and grain varieties. $*$ indicates that significant difference was noticed ($p < 0.05$).

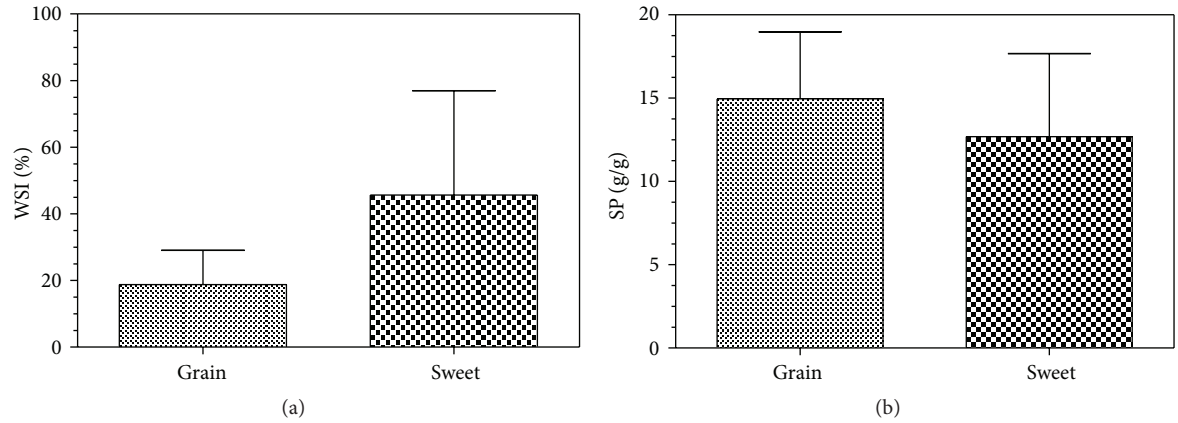


Figure 2: Comparison of water solubility index (WSI) (a) and swelling power (SP) (b) between sweet and grain sorghum varieties starch. No significant difference was noticed ($p < 0.05$).

sweet and grain varieties showed significant differences ($p < 0.05$) (Table 2). Water solubility index (WSI) for all varieties ranged from 10.0 to 95.0%, and the swelling power (SP) for all varieties had the range from 3.0 to 20.0 g/g. Among grain varieties, TAT showed the highest WSI (30.0%), whereas ARG showed the highest SP (20.07 g/g); but GL-1 showed the lowest values for WSI (10.0%) and SP (11.22 g/g). Among sweet varieties, GL-4 showed the highest WSI (95.0%) and the lowest SP (3.0 g/g), while GL-14 showed the highest SP (17.0 g/g) and ST showed the lowest WSI (15.0%). Starch with highest WSI (95.0%) was obtained from sweet varieties GL-4 and GL-6, whereas the lowest WSI value (10.0%) was obtained from grain varieties GL-1 and WAD (Table 2). Starch with highest SP was observed from a grain variety ARG, and the lowest SP was observed from a sweet variety GL-4. In current study, sweet varieties showed highest WSI compared to grain varieties, whereas grain varieties showed highest SP compared to sweet ones (Figures 2(a) and 2(b)). When starch molecules are heated in excess water, the crystalline structure is disrupted and water molecules become linked by hydrogen bonding to the exposed hydroxyl groups of amylose and amylopectin, which causes an increase in granule swelling and solubility. The differences in starch swelling power and water solubility index between grain and sweet varieties could be probably attributed to the amylose to amylopectin ratio and to the characteristics of amylose and amylopectin in terms of degree and length of branching and conformation as reported previously by Hoover (2001) [48]. Also the differences shown in the current study for morphological structure of starch granules were interplayed in the differences which have been noticed for SP and WSI [49]. The SP of starch indicates the degree of water absorption of starch granules, and the solubility reflects the degree of dissolution during the starch swelling procedure [50]. Consequently, the current study showed the lower degree of water absorption for sweet varieties compared with that of grain varieties. WSI and SP provide evidence of the magnitude of the interaction between starch chains within both the amorphous and crystalline domains as mentioned by Singh et al. (2003) [51].

Besides, the range for SP in present study was from 3.0 to 20.0 g/g, a little different from that reported by Singh et al. (2010). They reported that the SP of starches from

Table 3: Thermal properties of starch from sweet and grain sorghum varieties.

Sample	T_o (°C)	T_p (°C)	T_c (°C)	(Δ)H (J/g)
Grain				
GL-1	72.50 ± 0.00^{a}	76.15 ± 0.05^{a}	81.55 ± 0.05^{a}	12.43 ± 0.17^{abc}
WAH	70.60 ± 0.00^{c}	74.15 ± 0.05^{bc}	80.10 ± 0.20^{cd}	13.40 ± 0.21^{ab}
ARG	71.00 ± 0.00^{b}	74.4 ± 0.10^{bc}	80.30 ± 0.20^{bcd}	13.14 ± 0.63^{ab}
TAT	71.20 ± 0.00^{b}	74.65 ± 0.05^{b}	79.95 ± 0.05^{d}	12.68 ± 0.28^{ab}
Sweet				
GL-4	69.65 ± 0.05^{d}	74.35 ± 0.05^{bc}	81.15 ± 0.05^{ab}	13.50 ± 0.74^{ab}
GL-6	70.30 ± 0.00^{c}	73.95 ± 0.05^{c}	80.90 ± 0.10^{abc}	14.06 ± 0.25^{a}
GL-13	69.15 ± 0.05^{e}	72.20 ± 0.10^{d}	77.70 ± 0.20^{f}	12.63 ± 0.53^{abc}
GL-14	61.05 ± 0.05^{j}	66.40 ± 0.00^{h}	73.80 ± 0.30^{h}	8.85 ± 0.34^{de}
ZS	66.35 ± 0.05^{h}	70.85 ± 0.05^{e}	76.60 ± 0.20^{g}	10.48 ± 0.51^{cd}
YT	63.25 ± 0.05^{i}	68.20 ± 0.20^{f}	47.60 ± 0.10^{h}	7.85 ± 0.47^{e}
T-1	68.75 ± 0.05^{f}	72.65 ± 0.05^{d}	78.75 ± 0.05^{e}	12.04 ± 0.21^{abc}
ST	67.75 ± 0.15^{g}	71.20 ± 0.20^{e}	76.90 ± 0.20^{fg}	11.46 ± 0.12^{bc}

All data represent the mean of three determinations.
Means with the same superscript in each column are not significantly different ($p < 0.05$).

Table 4: The XRD and ^{13}C CP/MAS NMR chemical shifts of starches from sweet and grain sorghum varieties.

Sample	XRD		NMR chemical shifts (ppm)			
	RC (%)	Crystal pattern	C_1	C_4	C_2, C_3, C_5	C_6
Grain						
GL-1	16.42	A	102.23	82.40	72.61	62.27
WAH	15.00	A	102.13	82.15	72.71	62.32
ARG	15.27	A	102.08	82.10	72.66	62.27
TAT	15.22	A	102.81	82.60	72.76	62.47
Sweet						
GL-4	25.14	A	101.69	94.67	72.66	62.37
GL-6	24.82	A	101.73	95.45	72.71	62.37
GL-13	18.31	A	102.08	82.50	72.66	62.47
GL-14	15.34	A	102.18	82.40	72.66	62.12
ZS	17.82	A	101.93	81.90	72.61	62.37
YT	19.10	A	102.03	82.05	72.71	62.32
T-1	17.87	A	102.13	82.10	72.66	62.27
ST	16.91	A	101.98	82.50	72.66	62.27

different sorghum varieties had the range between 6.2 g/g and 15.3 g/g [40]. Subrahmanyam and Hoseney (1995) noticed the SP between 13.8 g/g and 15.2 g/g, which was also different from above-mentioned results [52]. For WSI, the range was between 10.0 and 95.0% in the current study and between 17.4% and 22.5% in the previously mentioned study for starches isolated from seven US sorghum cultivars [52]. Olayinka et al. (2008) mentioned SP of 8.79 g/g and solubility of 5.0% for starch isolated from Nigerian sorghum [53]. The above differences among different studies might be due to the different methods of starch isolation, different AC, different molar proportion of amylopectin A and B1, and different environmental condition [54].

3.3. Thermal Properties of Starch. The gelatinization properties of starch isolated from sweet and grain sorghum measured using DSC are summarized in Table 3. There had been significant difference among all varieties for DSC parameters studied ($p < 0.05$). For T_o, T_p, and T_c, the lowest degrees were noticed from sweet varieties compared with grain varieties. GL-1 grain variety ranked top among grain verities for those three parameters and even for sweet varieties showing the highest values of 72.5, 76.15, and 81.55°C, respectively. Sweet variety GL-14 showed lowest values for T_o and T_p (61.05 and 66.4°C, resp.), whereas sweet variety YT showed the lowest value for T_c (47.6°C) among all varieties. Among sweet varieties, GL-4 showed the highest values for T_p and T_c (74.35 and 81.15°C, resp.), whereas GL-6 showed the highest value for T_o (70.3°C). For enthalpy of gelatinization (ΔH) among all varieties, sweet variety GL-6 showed the highest degree (14.06 J/g), whereas another sweet variety YT showed the lowest degree (7.85 J/g), while grain varieties had the range from 12.43 J/g for GL-1 to 13.4 J/g for WAD, which revealed intermediate degrees. Figures 3(a) and 3(b) show that the degrees from grain varieties studied for T_o and T_p exhibited high significant ($p < 0.01$) differences from sweet varieties, whereas for T_c differences they were not significant ($p < 0.05$) (Figure 3(c)). ΔH of sweet varieties was a little bit higher than that of grain varieties, but the differences were not significant ($p < 0.05$) as presented in Figure 3(d). The highest ΔH which appeared by starch isolated from sweet varieties indicates a high level of the starch chain intramolecular bond. This high level may also be attributed to the increased rigidity of the starch granules as in current study sweet varieties showed starch granules size larger than that of grain ones. The enthalpy of starch gelatinization (ΔH) is the energy necessary to transform the granule structure from a crystalline to an amorphous state as discussed by Hasjim et al. (2013) [55]. As in the current study, this parameter showed higher degrees for sweet varieties starch compared with grain varieties starch and may relatively be attributed to the higher relative crystallinity of the former. Ji et al. (2004) hypothesized that T_o would be a measure of the perfection of starch crystallites, and the more perfect crystallites, the higher gelatinization onset temperatures [56]. The lowest degree noticed in this study for T_o from sweet varieties compared with that of grain varieties was due to the less perfect crystallites of sweet sorghum starch. The gelatinization temperature observed in the current study for sorghum starch is of similar range to that reported by Akingbala et al. (1988) (75.6 ± 0.9°C) for starch isolated from 24 sorghum varieties [57]. Also the current study is not far from that postulated by Sun et al. (2014) in their study on sorghum starch for T_o, T_c, T_p, and ΔH (64.60, 74.26, 69.42°C, and 9.02 J/g, resp.) [32].

3.4. Crystalline Pattern of Starch. Table 4 lists the crystalline pattern and relative crystallinity (RC), calculated from the ratio of diffraction peak area to total diffraction area, of starch from different sweet and grain sorghum varieties.

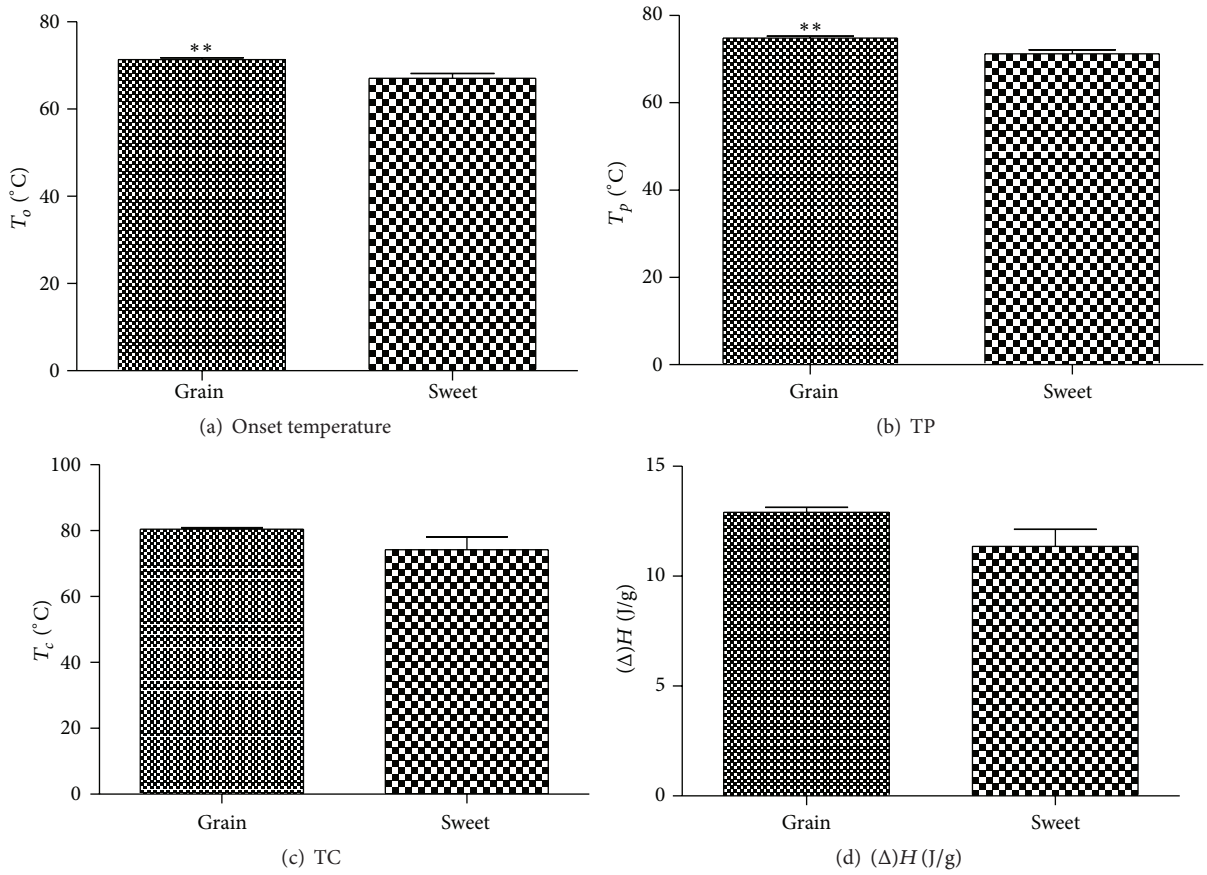


FIGURE 3: Comparison of starch thermal properties between sweet and grain sorghum varieties. (a)–(d) represent T_o, T_p, T_c, and ΔH, respectively. ∗∗ indicates that high significant difference ($p < 0.001$) was noticed.

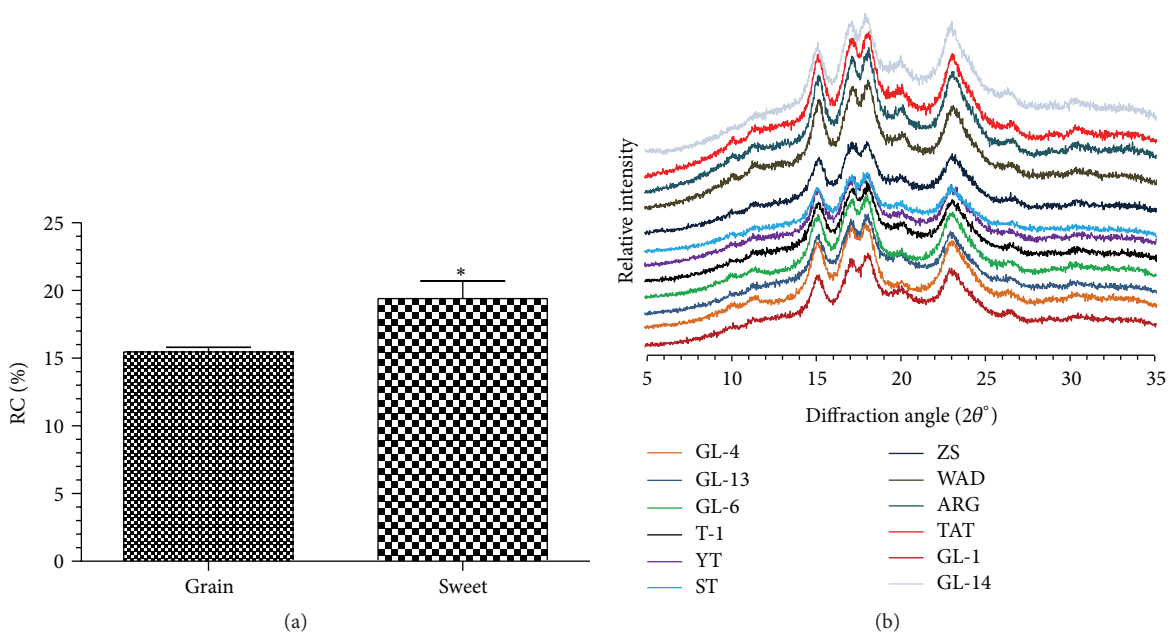


FIGURE 4: Comparison of relative crystallinity (RC) (a) and XRD spectrograph (b) of starches from sweet and grain varieties. ∗ indicates significant difference ($p < 0.05$) was noticed.

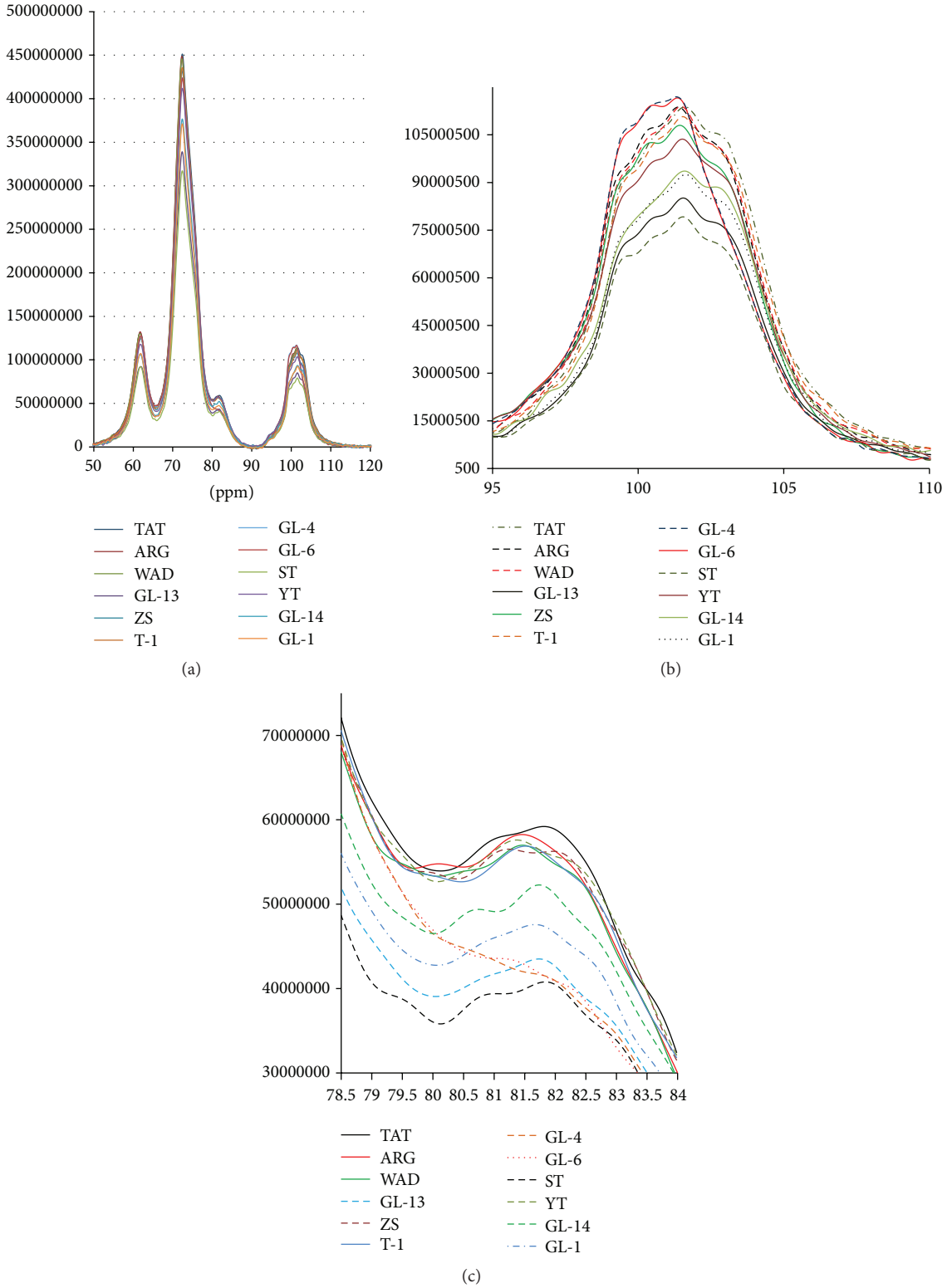
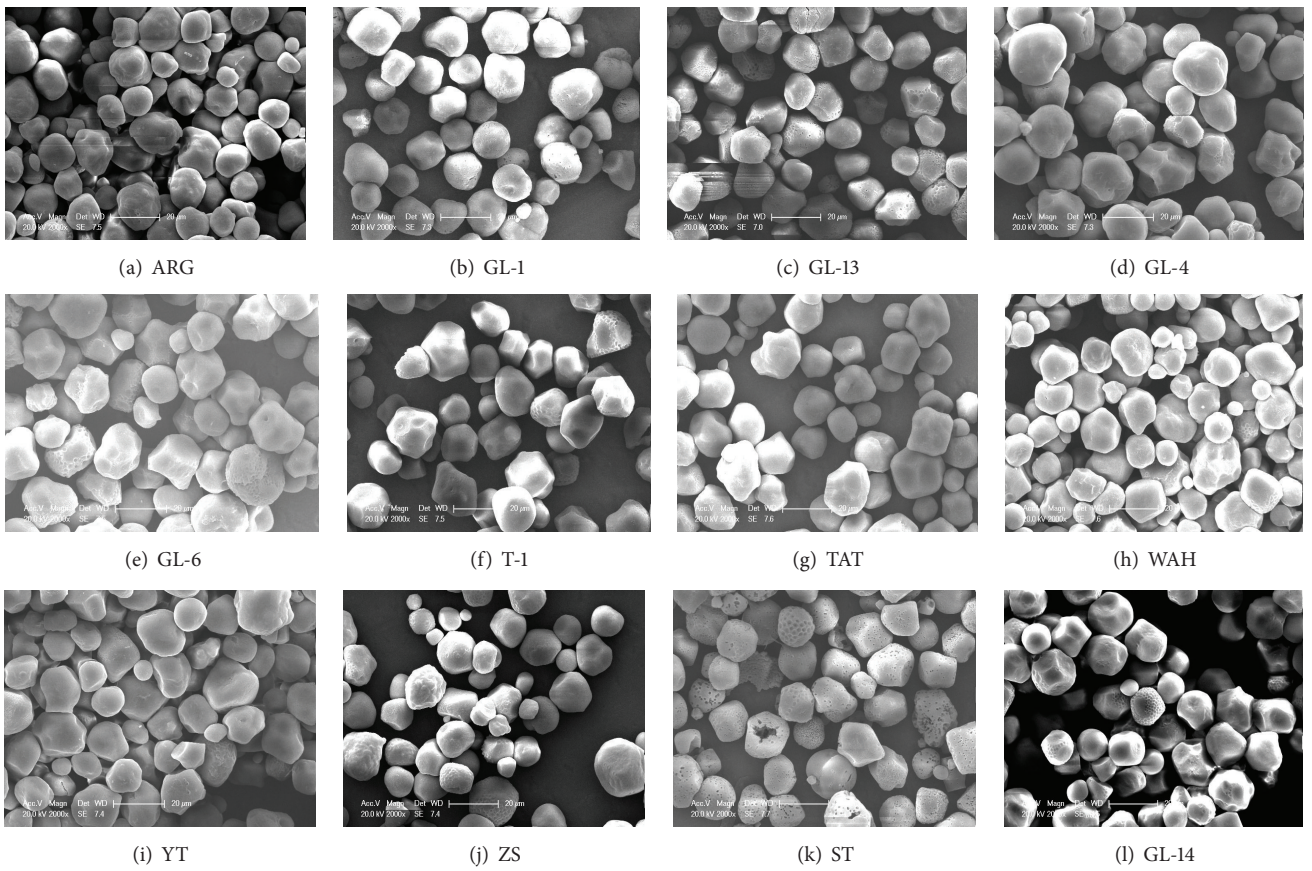


Figure 5: The ^{13}C CP/MAS NMR spectrograph (a) and C_1 resonance (b) and C_4 resonance (c) of starches from different sweet and grain sorghum varieties.



(a) ARG (b) GL-1 (c) GL-13 (d) GL-4

(e) GL-6 (f) T-1 (g) TAT (h) WAH

(i) YT (j) ZS (k) ST (l) GL-14

Figure 6: SEM images (2000x) of starch granules from different sweet and grain sorghum.

Sweet varieties studied showed high RC from grain varieties. Grain varieties had the range of RC from 15.00% to 16.42%, whereas sweet varieties ranged from 15.34% to 25.14%. The sweet variety GL-4 showed the highest RC (25.14%) among all varieties, whereas the grain variety WAD showed the lowest one (15.0%). Clearly sweet varieties studied reflected RC significantly ($p < 0.05$) higher than that from grain varieties (Figure 4(a)). Figure 4(b) shows starch crystalline properties of sweet and grain varieties studied using XRD. The results showed that both sweet and grain sorghum starches presented type A crystalline pattern as described by Zobel (1988) with strong peaks at around 15° and 23° and an unresolved doublet at around 17° and 18° of diffraction angle 2θ [58]. The peaks noticed for the current starches that appeared at 15°, 17°, 18°, and 23° (2θ) were also found in previous studies, such as Shin et al. (2004) for their study about Korean raw waxy sorghum starch [59], Boudries et al. (2009) for their study about sorghum starch cultivated in the Algerian Sahara [7], and Alvesa et al. (2014) for their study about starch isolated from sugarcane and sweet sorghum juice [45]. The A-diffraction pattern is typical of cereal starches and is characterized by double helix formed by packed amylose and amylopectin molecules in a monocyclic arrangement [60]. The current range for RC is in accordance with that reported by Zobel (1988); he mentioned that the degree of crystallinity of natural starch granules had a range from 15 to 45% [58].

NMR was used for further investigations of crystalline structure for isolated starch. Chemical shifts of ^{13}C CP/MAS NMR for the starch isolated from different sweet and grain varieties studied are shown in Table 4. Molecular order in starch granule is composed of two types of helices from the amylopectin branch chain. The helices that are packed in short-range order are defined as the double helical order, which can be detected by ^{13}C CP/MAS NMR but not by XRD. The helices that are packed in long-range order are related to the packing of double helices forming crystallinity, which can be measured by both ^{13}C CP/MAS NMR and XRD [61]. Figure 5(a) shows spectral feature of NMR spectra for different sweet and grain sorghum varieties starch. Spectral resonance differences For C_1 (which contains information about both crystalline nature as well as noncrystalline nature) were noticed [62]. Currently studied grain varieties and 6 out of 8 of sweet varieties showed a triplet model at C_1 resonance, as described by Atichokudomchai et al. (2004) [61] and, therefore, showed C_A type starch as discussed in Bogracheva et al. (2001) for the long-range order of starch [63]. C_6 resonance did not clearly exhibit differences between sweet and grain varieties as much as those at C_4 resonance (Figure 5(c)) and C_1 resonance (Figure 5(b)). As shown in Figure 5(b), C_1 resonance of grain varieties had two shoulders at the left side of the abundant peak and one shoulder at the right side. Sweet varieties GL-4 and GL-6 had two shoulders

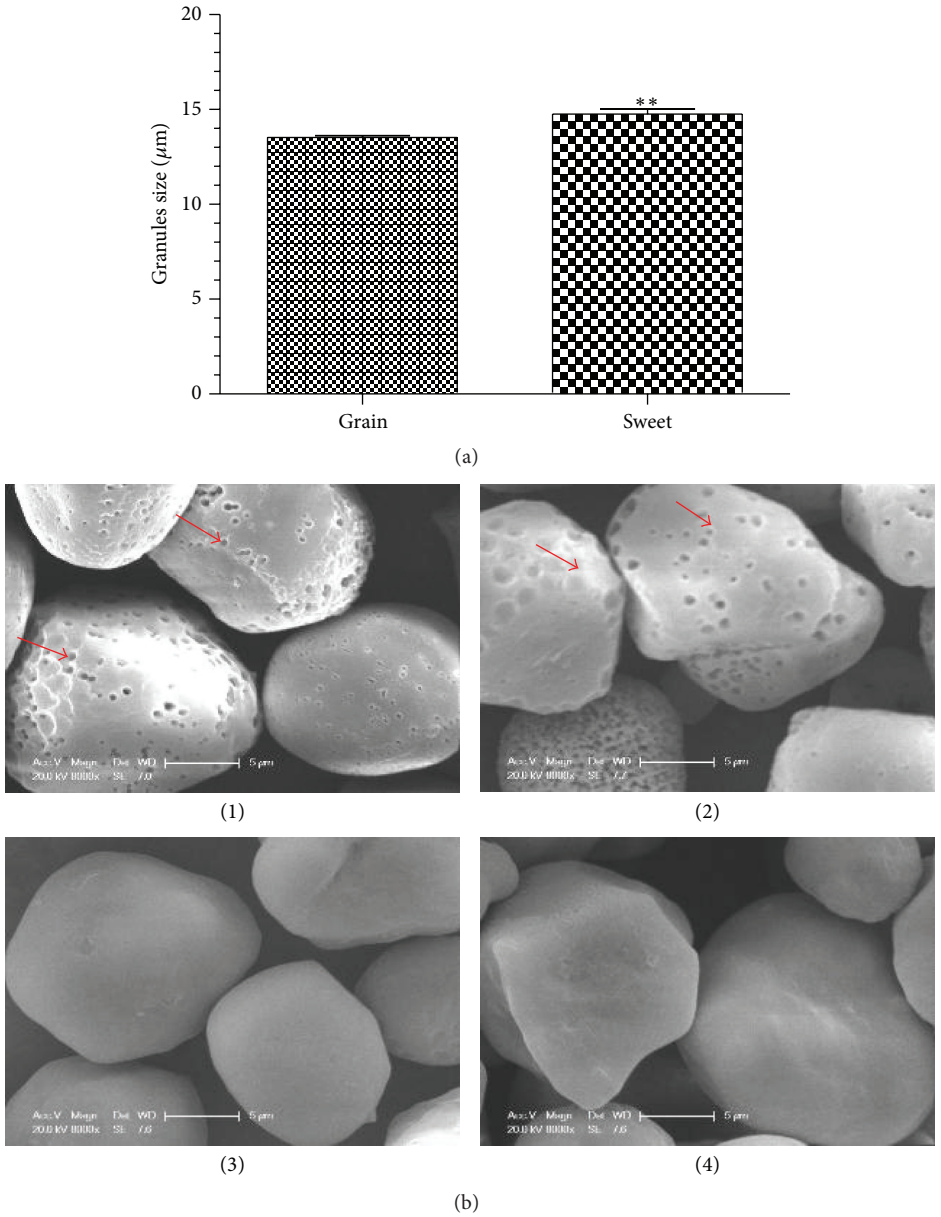


Figure 7: Comparison of starch granule size (a) and SEM images at magnification of 8000x (b) between sweet and grain varieties. ∗∗ means that high significant ($p < 0.01$) difference was noticed. In panel (b), 1–4 indicate GL-13, ST, TAT, and WAH, respectively.

at the left side, while at the right side they did not. Sweet varieties ZS and YT look like grain one but the right shoulder showed short extension. Sweet variety GL-13 revealed very short extension for the lower left shoulder, whereas for T-1 and ST the upper left shoulder showed short extension. GL-14 had no clear shoulder on the right side, but the left side showed one more extension (Figure 5(b)). For C_4 resonance, all the varieties showed clear differences for appearance of shoulders and peaks (Figure 5(c)). Sweet varieties GL-4 and GL-6 had no peaks appearing. Shoulders at the left side for TAT (81.0 ppm), WAD (80.9 ppm), and GL-1 (80.1 ppm) 1 ppm occur as distinct peaks for ST, GL-14, ARG, and ZS at 80.87, 80.72, 80.12, and 79.83 ppm, respectively (Figure 5(c)). Sweet variety ZS had clear shoulder at the right side at 82.00 ppm and ST had shoulder at 79.58 ppm (Figure 5(c)).

3.5. Morphological Properties of Starch Granule. Scanning electron microscope (SEM) was used to investigate morphology of starch from different sweet and grain sorghum as shown in Figure 6. SEM for starch granules from sweet and grain varieties showed polygonal or spherical shape without clear differences between them in the shape. These results for starch granule shape are in accordance with those reported by Benmoussa et al. (2006) for their study about sorghum starch in America [64] and by Singh et al. (2010) for their study about sorghum starch in India [40] but are little different

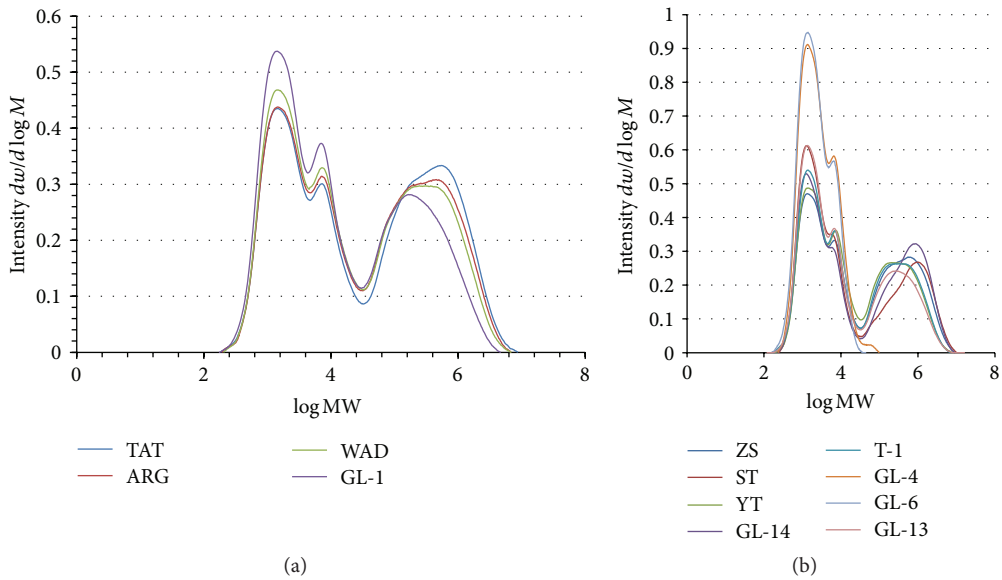


Figure 8: GPC profile for debranched starches from grain (a) and sweet (b) sorghum varieties.

Table 5: The starch granules size for different sweet and grain sorghum varieties.

Sample	Granule size μm			
	Max.	Min.	Mean	SD±
Grain				
GL-1	22.51	5.23	13.63	2.63
WAD	21.35	6.040	13.38	2.86
ARG	22.45	5.718	13.35	3.07
TAT	20.58	7.345	13.74	2.69
Sweet				
GL-4	25.03	6.43	15.49	3.57
GL-6	24.44	7.45	15.67	3.58
GL-13	22.95	6.64	14.59	2.54
GL-14	21.59	6.02	13.95	2.74
ZS	22.19	4.73	13.83	3.62
YT	27.44	6.61	14.46	3.61
T-1	23.01	9.17	15.48	2.88
ST	21.69	5.47	14.66	2.80

from those reported by Alvesa et al. (2014) for their study on sweet sorghum juice starch; they noticed predominantly an irregular polyhedral shape and generally an irregular shape [45]. The granules diameters of both sweet and grain varieties starch are presented in Table 5, and their range was from 13.35 to 15.67 μm. The sweet varieties granule diameter had the range from 13.83 μm to 15.67 μm. Starch from grain varieties had granule size from 13.35 to 13.74 μm. The granule size of the current study was in the range with that reported in Lindeboom et al. (2004); they summarized the size of starch granules from conventional sources ranges from less than 1 μm to more than 100 μm [65]. In the same way of comparing current results, Ai et al. (2011) mentioned starch granule size from 4 to 35 μm in their study for five genotypes of sorghum [66], whereas Gaffa et al. (2004) reported granule size from 4 to 26 μm for their study about white and red sorghum [67]. These differences clarified that this parameter is under genetic control, varying with the variety and environment. Starch from sweet varieties GL-6 and GL-4 showed the highest granule diameter (15.67 and 15.49 μm, resp.) in spite of the fact that they had the lowest AC% (6.71% and 5.39%, resp.). From the current study, starch granule size of sweet varieties exhibited significantly ($p < 0.05$) larger size compared to that of grain varieties (Figure 7(a)). Also Gaffa et al. (2004) noticed that the higher amount of large molecules was shown from red sorghum for their study about white and red sorghum starch [67]. The starch from both sweet and grain varieties showed smooth surface in accordance with that reported previously by Sun et al. (2014) [32]. Consequently, sweet varieties showed some dents in surface (Figure 7(b)) which was early mentioned in Sang et al. (2008) [68]. There appeared to be more dents on the surfaces of sweet variety GL-13 (indicated by arrows) (Figure 7(b)). Zhang et al. (2010) [69] and Sun et al. (2014) [32] clarified the existence of holes on the surface of starch granule, but they attributed that to the heat-moisture treatment which led to structural changes. Consequently, in the present study, in which there was no heat-moisture treatment, these dents may indicate that the structure of starch granule is different from those which had no dents, and further investigations are sharply needed. Comparing the current results from sorghum with other starch sources we noticed the following: it was a little different from that of wild *Trapa* species (*Trapa* spp.) as reported by Huang et al. (2015) (long axis length was from 15.02 to 16.51 μm) [62], it was even higher than that for rice (2.0–7.0 μm) as mentioned by Vandeputte and Delcour (2004) [70], and it was near to that of barely (2.0–18.1 μm) as reported by Tang et al. (2004) [31].

3.6. Chains Length Distribution of Starch. The results of chains length distribution for debranched starch from both sweet and grain varieties using gel permeation chromatography (GPC) are presented in Figures 8(a) and 8(b). Both

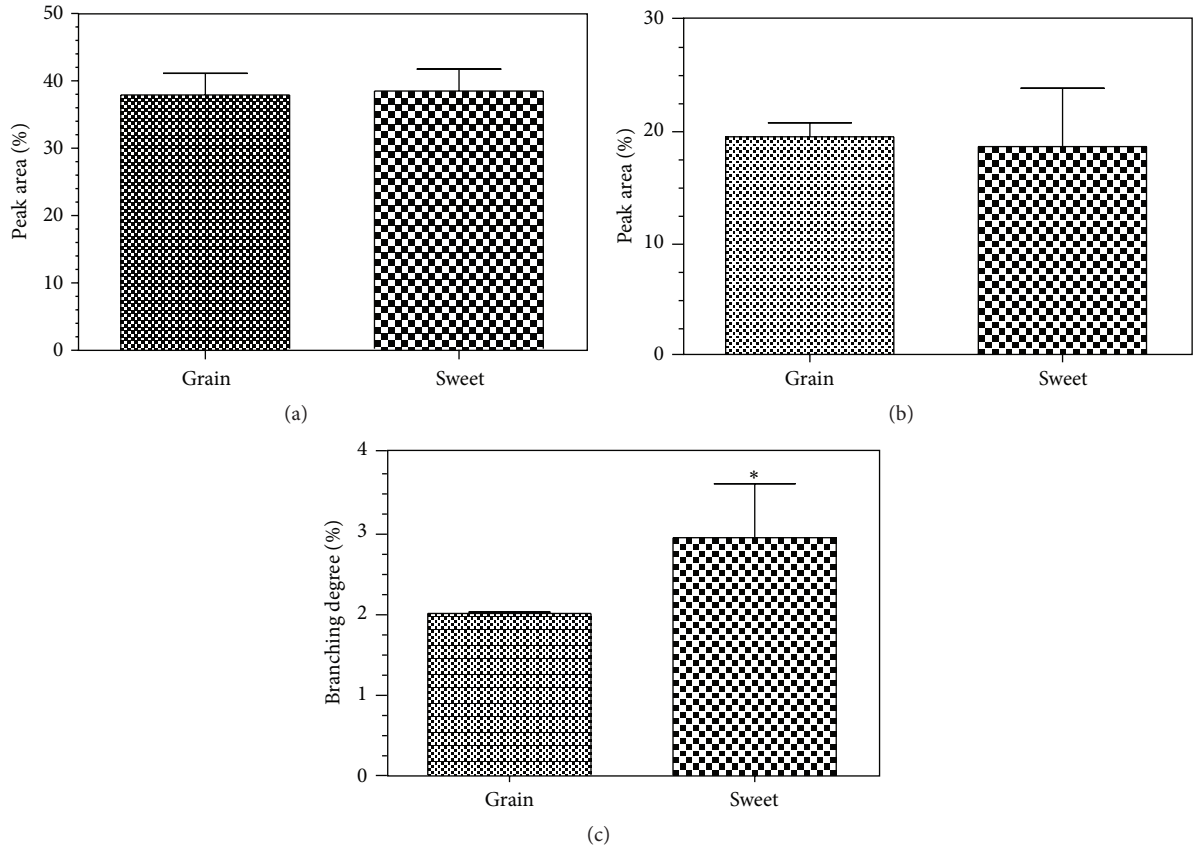


Figure 9: Comparison of peak 1 area (a), peak 2 area (b), and the branching degree (peak 1/peak 2) (c) from GPC profile between sweet and grain varieties starches. ∗ indicates that significant difference was noticed ($p < 0.05$).

Table 6: Molecular weight distribution of sweet and grain sorghum starch.

Sample	Peak area (%)		
	Peak 1	Peak 2	Peak 1/peak 2
Grain			
GL-1	42.53 ± 2.50^{b}	21.17 ± 0.40^{bc}	2.01 ± 0.16^{c}
WAD	39.90 ± 5.69^{b}	19.57 ± 0.38^{bcd}	2.04 ± 0.33^{c}
ARG	38.47 ± 5.48^{b}	19.17 ± 1.49^{cd}	2.00 ± 0.13^{c}
TAT	36.81 ± 3.91^{b}	18.17 ± 1.74^{cd}	2.02 ± 0.02^{c}
Sweet			
GL-4	72.78 ± 0.58^{a}	27.22 ± 0.58^{a}	2.68 ± 0.08^{abc}
GL-6	76.02 ± 0.47^{a}	23.98 ± 0.47^{ab}	3.17 ± 0.08^{abc}
G-13	47.03 ± 0.45^{b}	17.81 ± 0.52^{cde}	2.64 ± 0.05^{abc}
GL-14	47.28 ± 4.92^{b}	11.96 ± 0.48^{f}	3.96 ± 0.57^{a}
ZS	43.11 ± 7.02^{b}	15.44 ± 3.23^{def}	2.90 ± 1.06^{abc}
YT	42.84 ± 5.20^{b}	20.63 ± 0.22^{bc}	2.08 ± 0.23^{c}
T-1	44.24 ± 4.02^{b}	18.68 ± 0.39^{cd}	2.37 ± 0.26^{bc}
ST	51.13 ± 1.19^{b}	13.26 ± 0.63^{ef}	3.86 ± 0.09^{ab}

All data represent the mean of two determinations.
Means with the same superscript in each column are not significantly different ($p < 0.05$).

sweet and grain varieties showed typical GPC chromatogram of debranched starch. A tri-modal of peaks for chains length distribution was observed and designated as peak 1, peak 2, and peak 3, respectively. Peak 1 and peak 2 refer to amylopectin (AP) fraction representing the short branch chains (A and short B chains) and long branch chains (long B chains), respectively, while peak 3 refers to amylose fraction (AM) as mentioned in Song and Jane (2000) [71]. The area percentage of peaks from the GPC chromatographs was used to compare the weight distribution of chains for AP and AM fractions. All varieties showed significant differences ($p < 0.05$) as shown in Table 6. Peak 1 showed that short chains are the major group and exhibited the range from 36.81 to 76.02% for TAT and GL-6, respectively, while peak 2 showed the range from 11.96 to 27.22% for GL-14 and GL-4, respectively. The branching degree of AP (peak 1/peak 2) was from 2.00 to 3.96% for ARG and GL-14, respectively. Sweet varieties studied were not significantly ($p < 0.05$) different from grain varieties for peak 1 (Figure 9(a)) and peak 2 (Figure 9(b)), whereas for branching degree significant difference ($p < 0.05$) was noticed (Figure 9(c)). For the appearance of peaks and shoulders for the AM fraction, sweet varieties were categorized in four groups: (GL-13, YT, and T-1), ZS, (ST and GL-14), and (GL-6 and GL-4) (Figure 8(b)), whereas grain varieties were categorized in three groups: WAD, GL-1, and TAT and ARG (Figure 8(a)). As shown in Figure 8(a) grain varieties WAD, ARG, and TAT have two AM components, while GL-1 only has one. For sweet varieties Figure 8(b) shows

that all varieties almost have two components for AM fraction except GL-4 and GL-6. The existence of two components of AM fraction is in accordance with what was reported by Wang et al. (2014) [72]. Sweet variety ZS looks like grain varieties (ARG, WAD, and TAT) showing left shoulder and a little peak for AM fraction.

4. Conclusion

From the above-mentioned data, it can be concluded that sweet varieties exhibited little differences from grain varieties and some of them look like grain ones, which indicates the possibility to use their starches for industrialization as their grains are not preferable in food. All grain varieties showed high AC content, while some sweet varieties reflected very low AC content and others showed intermediate content. The current results showed that the essence of sweet and grain in sorghum plant beside accumulation of high sugar in stems was also affected by amylose and amylopectin fractions which affect the physical and chemical properties of their grain starch. Wide range shown in current study for AC content keeps sorghum out of altering the proportions of amylose and amylopectin, which makes it preferable for commercial uses.

Competing Interests

The authors declare that they have no competing interests.

Acknowledgments

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