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Physicochemical properties and enzymatic digestibility of starch from edible canna (*Canna edulis*) grown in Vietnam

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Abstract

Edible canna starch and other root starches (cassava, potato and sweet potato) extracted from raw roots grown in Vietnam were used to determine physicochemical properties and enzymatic digestibility. The edible canna starch exhibited significantly higher blue value and amylose content than the other root starches. It also had a wide range of gelatinization temperature and a high transition enthalpy. The viscosity of hot paste from edible canna starch was quite low and stable, whereas the cool paste had high viscosity and weak resistance against retrogradation. The paste clarity of edible canna starch was also significantly higher than that of the others. During refrigeration and frozen storage, the paste of edible canna starch released so much expelled and absorbed water, which showed low stability during storage with high net syneresis. In this study, native edible canna starch was also found like potato starch to be highly resistant to hydrolysis by α-amylase.
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Keywords: Edible canna; Potato; Cassava; Sweet potato; Resistant starch

1. Introduction

Edible canna (*Canna edulis*) or Queensland arrowroot is a starchy root crop grown infrequently in the tropical highlands. In Vietnam, the edible canna is grown both in mountainous provinces and in some low-lying areas. The growing areas of edible canna were estimated to be from 20,000 to 30,000 ha in northern Vietnam (Hermann, 1996). The roots of edible canna are mostly used for isolating starch. Edible canna starches have large granules and high amylose content. The granular size is in a range of 6–100 μm and mostly above 50 μm (Hung & Le, 2001). Therefore, the edible canna starches are easily isolated by a simple sedimentation method. The edible canna starch in Vietnam is mostly used for preparing transparent starch noodles, a traditional oriental food of south-east Asia (Collado, Mabesa, Oates, & Corke, 2001; Galvez, Resurreccion, & Ware, 1994; Kasemsuwan, Bailey, & Jane, 1998; Mestres, Colonna, & Buleon, 1988; Miskelly, 1993; Takahashi, Hirao, & Kawabata, 1985). The noodle made from edible canna starch had excellent eating qualities such as high tensile strength, minor swelling and good transparency. The previous studies have reported on the properties of edible canna starches in Andean regions (Perez, Breene, & Bahnassey, 1998; Santacruz, Koch, Svensson, Ruales, & Eliasson, 2002; Soni, Sharma, Srivastava, & Gharia, 1990), in Taiwan (Inatsu et al., 1983) or in Thailand and Japan (Thitipraphunkul, Uttapap, Piyachomkwan, & Takeda, 2003). In Vietnam, physicochemical properties of edible canna starch are still poorly understood although this kind of starch was widely used for preparing the traditional noodle for a long time. Therefore, physicochemical properties of edible canna starch grown in northern Vietnam were studied in this study.

Recently, several studies have focused on using cassava, potato and sweet potato starches substitution for mungbeen or edible canna starches to produce the transparent noodle with reduction the price of product (Chen et al., 2002; Collado & Corke, 1997; Kasemsuwan et al., 1998; Kim & Wiesenborn, 1996; Kim, Wiesenborn, Lorenzen, & Berglund, 1996; Xu & Seib, 1993). Cassava and sweet potato are also abundant materials with low price in

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Vietnam. Therefore, these kinds of starches are hopefully used to partial substitution in starch noodle making in native or modified types. However, noodle made from native cassava or sweet potato starch had an inferior quality. Chemical modification might improve the noodle quality of these starches. So, it is necessary to characterize the differences in physicochemical properties between edible canna starch and other sources, which could orient for chemical modification. In this study, cassava, sweet potato and potato starches were also used to compare with edible canna starch for this purpose. In addition, starches in many foodstuffs were found to escape complete digestion by pancreatic α-amylase in the human small intestine (Englyst & Cummings, 1985). Various categories of such resistant starch have been reported (Englyst & Cummings, 1987). However, enzymatic digestibility of native edible canna starch has not been previously reported. Therefore, the susceptibility of this kind of starch towards α-amylase was also investigated in this study.

2. Materials and methods

2.1. Materials

Edible canna (*Canna edulis*) grown in northern part of Vietnam was used. Starches were isolated and purified from raw root at the Laboratory of Post-harvest Technology, Hanoi University of Technology, Vietnam. The raw roots were ground using an engine-driven drum grater. Then the slurry was kept in a solution of 4.5% $NaHSO_3$ and passed through the sieves (0.232 and 0.105 mm in aperture size). Resultant starches were washed thoroughly in clean water to remove the light and color substances. Finally, the starch sediment was recovered by centrifugation and dried in an oven at 40 °C to 10–11% moisture. Cassava, potato and sweet potato starches, which were also isolated and purified from raw roots in Vietnam as described above, were used to compare with the edible canna starch.

2.2. Methods

2.2.1. Measurements of iodine absorption spectra, blue value and amylose content

Iodine absorption spectra of native starches were determined at room temperature based on the procedure of Morrison and Laignelet (1983) with a slight modification. The starch (40 mg) was completely dispersed in 10 ml of 10%-urea-containing dimethyl sulfoxide (UDMSO) solution by standing at room temperature for 10 min and then heating in a boiling water bath for 20 min. After cooling to room temperature, an aliquot (1 ml) of dispersion was weighed into a 100-ml volumetric flask. About 95 ml of distilled water and 2 ml of 0.2% iodine solution were added and then filled up to 100 ml and mixed immediately. The mixture was stood for 20 min at room temperature and an absorption curve of the mixture was measured from 450 to 800 nm with a spectrophotometer (UV-160A, Shimadzu, Japan).

Blue value (BV) of starches was measured at 680 nm as described previously (Takeda, Takeda, & Hizukuri, 1983). The amylose content of starches were determined according to the approved method 61-03 (AACC 1995) with a slight modification (Hung & Morita, 2005).

2.2.2. Determination of physicochemical characteristics of starches

X-ray diffraction analysis was performed using an X-ray diffractometer (Rigaku Co., Ltd, Rint-2000 type, Tokyo, Japan) operated at 40 kV and 80 mA. Diffractograms were obtained from 4° 2θ to 40° 2θ with a scanning speed of 8°/min and scanning step of 0.02°.

Thermal characteristics of starch and stored paste were determined using a differential scanning calorimeter (DSC) (DSC-60, Shimadzu, Japan). Sample (3 ± 0.1 mg) was directly weighed into an aluminum vessel and then 10 μl of distilled water was added. The vessel was sealed and kept at room temperature for more than 30 min for equilibration. Then the sample-containing vessel was heated from 30 to 100 °C at a rate of 10 °C/min. After heating in DSC, the samples were stored at 4 °C for 5 days. Then they were rescanned as described above. An empty vessel was used as a reference. The initial, peak and recovery temperatures and transition enthalpy were automatically calculated from the chart.

Pasting properties of starch suspension (8%, w/v) were tested using an amylograph (Brabender, Germany) as described elsewhere (Hung & Morita, 2005).

Swelling power of starches was measured according to the method of Sasaki and Matsuki (1998) with a slight modification. Isolated starch (0.16 g, db) was directly weighed into glass tubes with coated screw caps and then added with 5 ml of distilled water. The tubes were placed in a shaking apparatus and heated from 50 to 90 °C at 10 °C intervals. After keeping at those temperatures for 30 min, the heated sample was quickly cooled to room temperature in cold water and centrifuged at 3000*g* for 15 min. The supernatant was carefully removed and swelling power was determined as described by Hung and Morita (2005).

The paste clarity of starch was determined according to the method of Craig, Maningat, Seib, and Hoseney (1989). Stach (0.05 g, db) was suspended with distilled water (5 ml) in a glass-stoppered tube and heated at 95 °C for 30 min with occasional shaking every 5 min. After cooling, clarity of starches was measured on a spectrophotometer (UV-160A, Shimadzu, Japan) at 650 nm against a water blank.

Net syneresis of gels from the pastes of cooked starches during refrigeration and frozen storages was determined according to the method of Zheng and Sosulki (1998) as follows. The pastes after the amylograph measurement were divided into two samples (60 g/sample). The samples were kept in 100-ml bottles sealed with screw caps and stored at either 4 °C (in refrigerator) or −18 °C (in freezer). Net syneresis was determined as described by Hung and Morita (2005)

2.2.3. Enzymatic hydrolysis

The procedure was essentially that of Knutson, Khoo, Cluskey, and Inglett (1982) with a slight modification. Starch (100 mg) was suspended in 10 ml of distilled water containing 1200 units of α-amylase. Then the suspensions were placed in a water bath at 37 °C and shaken continuously. A 1.0-ml aliquot was withdrawn at appropriate time intervals (0, 3, 6, 12, 24, and 48 h), added with 2 ml of 95% ethanol and then filled up to 10 ml with distilled water. After centrifugation of the solution at 3000*g* for 10 min, the supernatant was analyzed for soluble carbohydrate according to the procedure of Hizukuri, Takeda, Yasuda, and Suzuki (1981). Percent hydrolysis was expressed as amount of maltose released per 100 mg of dry starch. Appropriate controls without the enzyme were prepared.

Native and 12-h hydrolyzed starch granules were observed using a scanning electron microscope (SEM). The starches after hydrolysis for 12 h by α-amylase as described above were washed with 95% ethanol, collected on a glass-filter, dried in a vacuum desiccator and then observed by SEM according to the procedure of Hung and Morita (2005).

2.2.4. Statistical analysis

Analysis of variance (ANOVA) was performed using Duncan's multiple-range test to compare treatment means at $P<0.05$ (Steel & Torrie, 1960). All tests were carried out at least in duplicate.

3. Results and discussion

3.1. Iodine absorption spectra, blue value and amylose content

The results of measurement of iodine absorption spectra and blue value are shown in Table 1. The maximum absorption wavelength (λ_{max}) of edible canna starch was higher than that of potato starch, but lower than that of cassava and sweet potato starches, whereas the blue value of edible canna starch was the highest, followed by decreasing order of potato, sweet potato and cassava starches. The amylose content of edible canna starch was 37.1%, significantly higher than that of other root starches. Thitipraphunkul et al. (2003) reported that the amylose content of Japanese-green, Thai-green and Thai-purple edible canna starches ranged from 19 to 25%, whereas edible canna starch from the Andean region was 23.8% (Santacruz et al., 2002). Thus, these results indicated that the structure of edible canna starch in Vietnam was significantly different from those grown in other countries.

Table 1
Absorbance of starch–iodine complex and amylose content of various Vietnamese root starches[a]

Sample	λ_{max} (nm)	Blue value (nm)	Amylose content (%)
Edible canna	602.3 ± 1.3	0.476 ± 0.008	37.1 ± 2.3
Cassava	607.0 ± 3.9	0.337 ± 0.010	17.0 ± 1.0
Potato	598.7 ± 1.0	0.460 ± 0.014	27.4 ± 3.7
Sweet potato	610.0 ± 2.8	0.400 ± 0.002	20.0 ± 1.5

[a] Values are the mean ± SD, $n=6$.

3.2. X-ray diffraction pattern

X-ray diffraction patterns of native starches are shown in Fig. 1. Edible canna starch grown in northern Vietnam exhibited B-type crystal with a peak at 15.8 Å (line 1), a broad medium intensity lines at about 5.9 Å (line 3a, 3b), a strong line at 5.2 Å (line 4a) and a medium intensity double at 4.0 and 3.7 Å (lines 6a, 6b) as classified by Zobel (1988). This result agreed with the previous studies (Jane et al., 1999; Thitipraphunkul et al., 2003). As a result, B-type crystal is the characteristic of edible canna starch. Cassava, used as a reference, showed A-type crystal with the major peaks at around *d*-spacings 5.8 (line 3b), 5.2 and 4.8 (line 4a, 4b) and 3.8 (line 6a) as previously reported (Zobel, 1988).

3.3. Thermal characteristics

Thermal characteristics of starches and paste from various root starches are shown in Table 2. A range of gelatinization temperature of edible canna starch was 67.4∼76.1 °C, which was not significantly different from that of cassava and potato starches, but significantly higher than that of sweet potato starch. Transition enthalpy of edible canna and potato starches, which had higher amylose contents than the others, was significantly higher than that of cassava and sweet potato starches, agreeing with the previous reports (Gunaratne & Hoover, 2002; Jane et al., 1999). While Thitipraphunkul et al. (2003) reported that the enthalpy of edible canna starches (both Japanese and Thai cultivars) was similar to that of cassava starch. Thus, the root starches containing the higher amylose contents had the larger transition enthalpy than those containing the lower amylose contents. In contrast, waxy wheat starches which contained dominantly amylopectin had larger transition enthalpy than did regular wheat starches (Abdel-Aal, Hucl, Chibbar, Han, & Demeke, 2002; Fujita, Yamamoto, Sugimoto, Morita, & Yamamori, 1998; Lee, Swanson, & Baik, 2001). These results indicate that thermal characteristics of starches depended on various factors such as molecular architecture of the crystalline region, amylose to amylopectin ratio, chain length distribution, amount of double-helical order, etc. (Hoover, 2001; Jane et al., 1999; Sasaki & Matsuki, 1998). After storage for 5 days in a refrigerator, the range of gelatinization temperature and transition

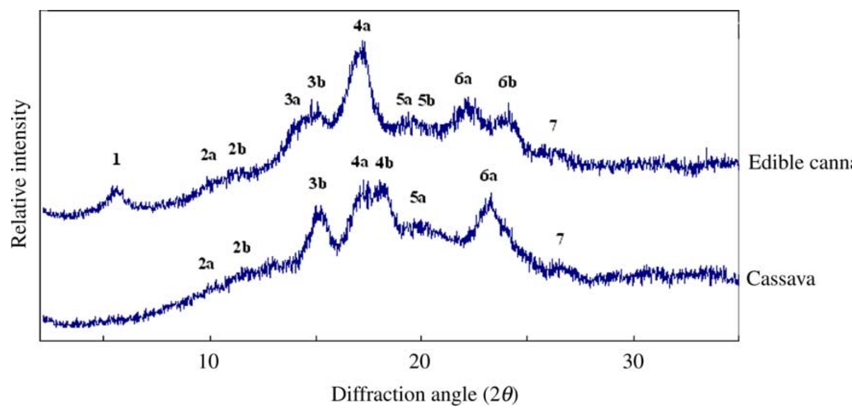

Fig. 1. X-ray diffraction pattern of native edible canna and cassava starches.

Table 2
Thermal characteristics of various Vietnamese root starches[a,b]

Sample	Starch				Paste after 5 days of the storage			
	T_i (°C)	T_p (°C)	T_r (°C)	ΔH (J/g)	T_i (°C)	T_p (°C)	T_r (°C)	ΔH (J/g)
Edible canna	67.4c	70.8c	76.1b	14.5b	46.3a	62.6b	72.7b	5.4c
Cassava	66.9c	72.0d	77.0c	12.4a	46.0a	54.5a	56.8a	0.3a
Potato	64.9b	69.6b	76.4b	14.1b	46.9a	67.5c	72.1b	2.7b
Sweet potato	57.4a	65.9a	74.5a	12.3a	46.6a	61.6b	66.1b	1.6b

[a] T_i, T_p, T_r: initial, peak and recovery temperatures; ΔH, enthalpy of the endothermic peak.
[b] Means by the same letter in the same column is not significant difference ($P<0.05$), $n=3$.

enthalpy of pastes decreased significantly as compared with those of native starches. Retrogradation of different kinds of starches also depends on the structure of amylose and amylopectin in the starches. The stored pastes of cassava and sweet potato starches had lower range of gelatinization temperature and transition enthalpy than those of the pastes of edible canna and potato starches. Especially, the stored paste of edible canna starch had significantly larger transition enthalpy than the other root starches resulting in high level of retrogradation, which is similar to the properties of edible canna grown in other countries (Jane et al., 1999; Thitipraphunkul et al., 2003). Although amylose content mainly contributed to the retrogradation of starches, chain length distribution of amylopectin affected the retrogradation rate of starches (Jane et al., 1999). In addition, the presence of other compositions in starches such as phosphate monoesters, lipids and phospholipids also affected the retrogradation of starches (Lim, Kasemsuwan, & Jane, 1994).

3.4. Pasting properties

The gelatinization and peak temperatures of edible canna and potato starches were considerably higher than those of cassava and sweet potato starches (Table 3). The paste prepared from edible canna starch had significantly lower peak viscosity and breakdown than did the other root starches. This means the paste of edible canna starch exhibited a greater gel consistency and hot paste stability as compared with the others. However, the final viscosity and setback consistency of paste from edible canna starch was significantly higher than those of the others. This result showed that the paste of edible canna starch had a weak resistance against retrogradation caused by high amylose content in starches. In contrast, the lower final viscosity and setback and higher breakdown of cassava starch paste indicated that this starch paste was not stable at high temperature and had high resistance against retrogradation.

Table 3
Pasting properties of cooked starch prepared from various Vietnamese root starches[a,b]

Sample	T_g	T_p	V_p	V_f	BD	SB
Edible canna	69.0b	93.0c	620a	1420d	20a	800d
Cassava	68.5b	85.0a	765b	730a	420d	−35a
Potato	69.0b	93.0c	875c	1270c	100b	395c
Sweet potato	67.0a	88.5b	880c	1150b	185c	270b

[a] T_g, T_p: gelatinization and peak temperatures; V_p, V_f: peak and final viscosities; BD, breakdown; SB, setback.
[b] Means by the same letter in the same column is not significant difference ($P<0.05$), $n=2$.

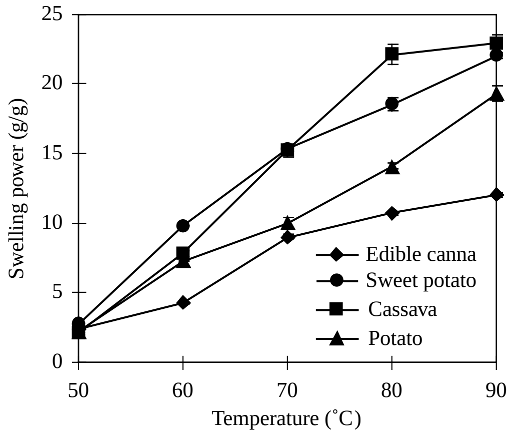

Fig. 2. Swelling powers of various Vietnamese root starches —♦—, edible canna; —■—, cassava; —▲—, potato; —●—, sweet potato.

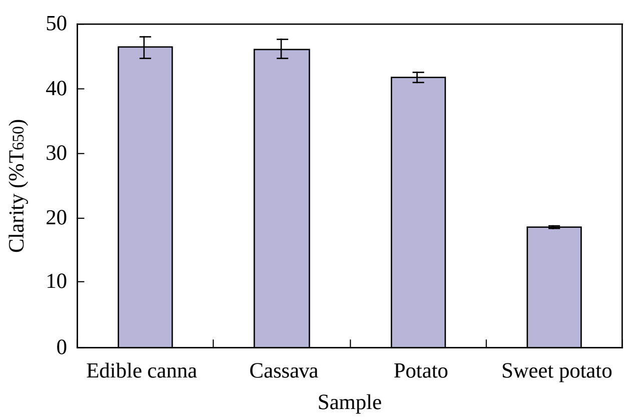

Fig. 3. Paste clarity of various Vietnamese root starches.

3.5. Swelling power and paste clarity

Swelling power of the root starches increased rapidly in a range of 50–90 °C (Fig. 2). Swelling power of edible canna starches was the lowest, followed by potato starch. The cassava starch paste had lower swelling power than sweet potato starch at temperature of less than 70 °C. However, swelling power of this kind of starch increased rapidly at higher temperature. These results showed that edible canna starch had the smallest water-holding capacity during heating because of high amylose content. This property is responsible for the excellent quality of transparent noodle made from this kind of starch.

The paste clarity of root starches measured by using light transmittance is shown in Fig. 3. The paste cooked from edible canna starch was significantly clearer than that of the other root starches. The paste clarity of sweet potato starch was the most opaque, whereas the paste clarity of cassava starch was higher than that of potato starch. These results were caused by a fine structure of compositions in starch granules rather than amylose to amylopectin ratios or starch granule sizes (Craig et al., 1989).

3.6. Gel stability

The stability of starch gels under refrigeration and frozen storages is given in Table 4. The free water of edible canna starch, which was separated from fresh paste, was not significant difference from potato and sweet potato starches, but significantly lower than that from cassava starch. The paste from edible canna starch released so much the expelled water in the first week of refrigeration storage, whereas a small amount of expelled water was released in next weeks (data not shown) resulting in net syneresis of edible canna was the highest in the first week of refrigeration storage and decreased in next weeks. This result was caused by high amylose content of edible canna starch, which retrograded rapidly during storage. The net syneresis of edible canna starch paste was significantly higher than that of the others during refrigeration and frozen storages. Among the other starches, the paste of sweet potato starch had lower net syneresis during refrigeration storage, whereas the paste of cassava starch exhibited more stable during frozen storage. These results showed that the paste of edible canna starch retrograded more quickly than did the other root starch pastes during storage. The paste of cassava starch in frozen storage was more stable, whereas the pastes of potato starch and sweet potato starches were less stable in frozen storage. Consequently, the paste of all native starches is not stable during refrigeration or frozen storage.

3.7. Enzymatic hydrolysis of native starches

Degrees of hydrolysis by α-amylase of native starches are shown in Fig. 4. At the first 6 h, all native starches were

Table 4
Net syneresis of gels prepared from various Vietnamese root starches during refrigeration and frozen storage[a,b]

Sample	Free water (%, wt)	Net syneresis (%, wt)							
		Refrigeration				Frozen			
		W_1	W_2	W_3	W_4	24 h	48 h	72 h	96 h
Edible canna	2.1a	42.3c	28.4c	30.0c	24.0c	72.0d	74.0d	77.0c	76.7c
Cassava	5.3b	4.3a	3.2a	18.0b	22.0bc	2.9a	4.3a	3.0a	22.5a
Potato	3.2a	7.3b	20.3b	20.0b	20.0b	59.2c	59.0c	63.0b	68.9b
Sweet potato	2.7a	3.8a	3.5a	5.6a	9.2a	40.4b	47.0b	67.0b	68.2b

[a] W_1, W_2, W_3, W_4: 1 week, 2 weeks, 3 weeks and 4 weeks.
[b] Means by the same letter in the same column is not significant difference ($P<0.05$), $n=4$.

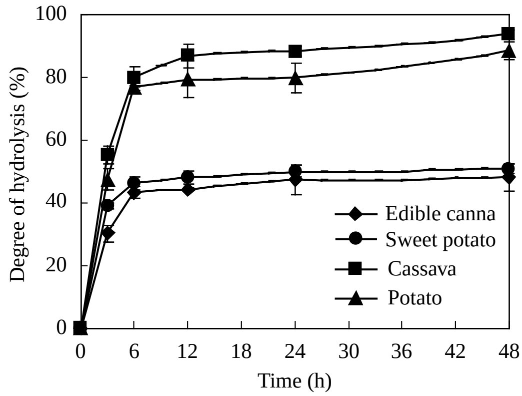

Fig. 4. Enzymatic hydrolysis of various Vietnamese root starches.

rapidly hydrolyzed and then the rate of hydrolysis decreased significantly. Degrees of hydrolysis of cassava and sweet potato starches (93.6 and 88.5%, respectively) were significantly higher than those of edible canna and potato starches (51 and 48%, respectively) after 48 h of hydrolysis. Thus, cassava and sweet potato starches were more susceptible to α-amylase than edible canna and potato starches. The previous study found that banana and potato starch contained large amounts of resistant starches (Englyst, Kingman, & Cummings, 1992). However, little information about the level of resistant starch in other starch sources is available except high amylose maize starch (Brown, 1996). In this study, edible canna starch was highly resistant to the hydrolysis of α-amylase. The large amount of resistant starch was considered to relate with B-type crystal structure of native edible canna starch like raw potato, banana and high amylose maize starches.

3.8. Scanning electron microscope

Appearances of native starch granules before and after enzymatic hydrolysis are observed in Fig. 5. Edible canna starch granules were significantly larger than those of the other root starch granules. All native starch granules had smooth surface and no evidence of cracks because of the low level of starch damage during fractionation. After 12 h of hydrolysis by α-amylase, the different patterns of enzyme attack among the root starch granules were observed. In hydrolyzed edible canna starch, the large granules were broken to fragments, caused by exposing their interior structure, while some granules exhibited disc-like depressions on surfaces and the others were still intact with no evidence of α-amylse attack. Likewise, the hydrolyzed potato starch had some intact granules though the interior of other granules were exposed. In contrast, all granules of cassava and sweet potato starches were attacked by α-amylase with the deep pin-holes of hydrolyzed cassava starch or roughened surfaces of hydrolyzed sweet potato starch. Thus, the patterns of enzyme attack were different among the root starches due to the structure of these kinds of

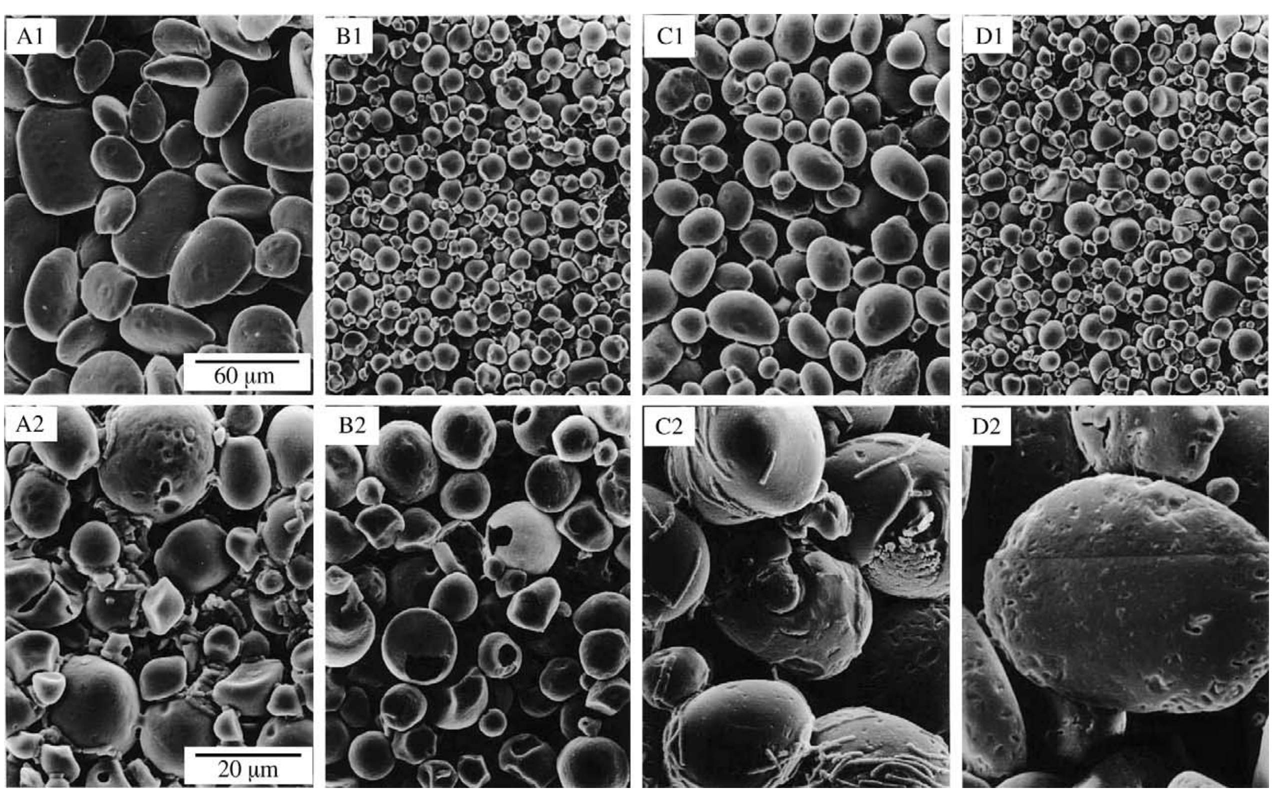

Fig. 5. SEM of various Vietnamese root starch granules before and after enzymatic hydrolysis for 12 h. A1, B1, C1, D1 and A2, B2, C2, D2: native and hydrolyzed starch granules of edible canna, cassava, potato and sweet potato, respectively.

starches was varied and these results reflect the different hydrolysis rates as shown in Fig. 4.

4. Conclusion

The edible canna starch was a kind of high-amylose starch, which exhibited significantly higher blue value and amylose content than the other root starches. The edible canna starch also had a high range of gelatinization temperature and a large transition enthalpy. The viscosity of hot paste from edible canna starch was quite low and stable, whereas the cool paste had high viscosity and weak resistance against retrogradation. The paste clarity of edible canna starch was significantly higher than that of the others. During refrigeration and frozen storage, the paste of edible canna starch released so much expelled and absorbed water. It showed a low stability during storage with high net syneresis. In addition, native edible canna starch was also found to resist highly to hydrolysis of α-amylase like potato starch. The physicochemical properties of edible canna starch in Vietnam for producing excellent transparent noodle, which were different from the other root starches, were found in this study. In further study, fine structures of amylose and amylopectine and granules are being characterized for more understanding of this kind of starch.

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