ORIGINAL ARTICLE

Effect of electron beam irradiation on physicochemical properties of sago starch

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Abstract

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The effect of electron beam irradiation on sago starch was determined in terms of physicochemical properties. The irradiation dose employed was in the range of 10 to 30 kGy. Peak viscosity was decreased when the irradiation dose was increased. The gel strength was found to be increased when the sago starch was irradiated at 10 to 15 kGy, thereafter, the value was decreased. Solubility was increased while swelling power was decreased. Redness, yellowness and free acidity were increased. Intrinsic viscosity, molecular weight and degree of polymerization were also decreased when the irradiation dose was increased.

Key words : sago starch, irradiation, pasting profile, swelling power, solubility

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ผลของการฉายรังสีด้วยลำแสงอิเลคตรอนต่อคุณสมบัติของแป้งสาคู

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การทดลองนี้เป็นการศึกษาคุณสมบัติของแป้งสาคูหลังจากผ่านการฉายรังสีด้วยลำแสงอิเลคตรอนในระดับ 10 ถึง 30 กิโลเกรย์ ซึ่งผลการทดลองพบว่าความหนืดสูงสุดของแป้งสาคูลดลงเมื่อระดับของการฉายรังสีสูงขึ้น ความแข็งของเจลมีค่าสูงขึ้นเมื่อระดับของการฉายรังสีมีค่า 10 และ 15 กิโลเกรย์ แต่เมื่อระดับการฉายรังสีสูงขึ้นอีกพบว่าค่าความแข็งของเจลมีค่าลดลง การฉายรังสีทำให้ค่าการละลาย ค่าสีเหลือง สีแดงและความเป็นกรดของแป้งสาคูเพิ่มขึ้นในขณะที่การพองตัวลดลง ค่าสัมประสิทธิ์ความหนืด น้ำหนักโมเลกุลและขนาดของโมเลกุลมีค่าลดลง

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Starch is found in a number of plant sources. It occurs in the form of granules, varied in shape and size and with different physico-chemical and functional characteristics (Tharanathan, 1995). It is composed primarily of amylose and amylopectin. Several minor components such as lipids, protein and phosphates are also present within the granules but at low concentrations (French, 1984). Amylose is an amorphous polymer linked by α-(1→4) linkages with chain lengths of 6,000 D-glucopyranose units and molecular weight between 150,000 and 600,000 Da (Kennedy and White, 1983). Amylopectin is a very large and highly branched polymer linked by α-(1→4) and α-(1→6) linkages with molecular weight around 1,000,000 D-glucose residues per molecule. It contains 4-5 percent of α-(1→6) branch-points, which has average chain length of 17-30 glucose units (Greenwood, 1979). X-ray crystallography and microscopy studies have revealed the amylopectin framework within the starch granule to be crystalline and organized in separated concentric rings as seen in cross-section (French, 1984). Sago starch, produced from pith of sago palm, is a useful resource for commercial raw material and food stuff (Takeda *et al.*, 1989). Sago is utilized in Malaysia mainly for the purpose of food production. This includes manufacture of vermicelli, bread, cracker, biscuits, sago pearls and many traditional foods. Although, native starches have many uses in food systems, modified starches have limitless for food and non-food application. The use of regular starch is limited by its physical and chemical properties. The granules are insoluble in cold water. Very often the viscosity of cooked native starch is too high to use in certain applications. Therefore, it should be modified to meet such application. The objective of starch modification is to alter the physical and chemical characteristics. Modified starch can provide a wide range of functions, from binding to disintegrating, imbibing or inhibiting moisture. Types of modification that are most often made, sometimes singly, but often in combinations, are crosslinking of polymer chains, non-crosslinking derivatization, pregelatinization and depolymerization (Shelton and Lee, 2000). Irradiation is known to degrade the starch (Rosenthal, 1992). Numerous studies have been carried out on gamma-irradiated starch (Greenwood and Mackenzie, 1963; Allah *et al.*, 1974; Scherz, 1974; Nene *et al.*, 1975; Roushdi *et al.*, 1983; Kume and Tamura, 1987; Ciesla and Zo'ltwski, 1991; Grant and D'Appolonia, 1991; Kang *et al.*, 1999). Effect of electron beam irradiation on potato starch was also studied by Greenwood and Mackenzie (1963). However, low irradiation doses were employed with potato starch. This study aimed at elucidating the effect of electron beam irradiation on the physicochemical characteristics of sago starch with higher doses in order to find its potential in an application.

Materials and methods

Sample

Sago starch was obtained from Nitsei sago industries Sdn.Bhd, Mukah, Sarawak. They were irradiated at 10, 15, 20, 25 and 30 kGy. The accelerate voltage and beam current of electron beam machine were 3 MeV and 10 mA. The moisture content of sago starch was 12.5%.

Pasting profile

Pasting profiles were measured using a Brabender Viskograph (PT100, Model no 801260, Duisburg, Germany) with a 700 cmg sensitivity cartridge. The bowl speed was 75 rpm. The starch slurry (400 ml at 6% starch solids) was pasted at a heating rate of 1.5°C/min heat from 30°C to 95°C, and held at 95°C for 30 min. It was then cooled down to the temperature of 50°C and held for 30 min. Peak viscosity indicates the paste viscosity at which the peak is attained during heating stage.

Gel strength

The gel strength was determined by the method of Jane and Chen (1992) with modification. The 8% starch paste was prepared. The sample was poured into a height of 2.7 cm in a cylindrical plastic container (diam. = 4.0 cm, height 5.5 cm) as a mold. The pastes were measured after aging at 25°C for 24 hours. The gel strength was determined using a Stable Micro System TAXT-2 Texture Analyser (Texture Technologies Corp., NY/Stable Micro System, Godalming, Surrey, UK). The paste in the mold was compressed at a speed of 1.0 mm/sec to a distance of 10 mm using a cylindrical probe (P0.5) and then retracted at the speed of 1 mm/sec to obtain the curves. The maximum force was termed gel strength.

Swelling power and solubility

Swelling power and solubility was determined as described by Schoch (1964). Starch was accurately weighed (2g, dry basis) into a dry tarred pre-weighed 250 ml centrifugal bottle. Distilled water was added to give a total volume of water equivalent to 180g. The starch was completely suspended by stirring at 200 rpm using magnetic stirrer. After taking out the stirrer, the bottle was immediately placed in constant temperature shaking water bath at 85°C±0.2 with continuous shaking at 200 rpm for 30 minutes. The centrifuge bottle was then dried and placed on a balance followed by the addition of distilled water to bring a total weight of 200 g. After capping, the bottle was centrifuged for 15 minutes at 1000xg. To measure solubility, 50 ml of the supernatant was then pipetted and transferred into an evaporating petri-dish and dried overnight in a hot air oven at 105°C. The dried residue was then cooled in a dessicator and weighed for soluble starch. To measure the swelling power, the supernatant was carefully removed and discarded. The bottle with the sediment paste was then weighed to give the weight of swollen starch granules. The result was expressed by the calculation:

$$\%\text{ solubility (on dry basis)} = \frac{\text{Weight of soluble starch}}{\text{Weight of sample on dry basis}} \times 400$$

$$\text{Swelling power} = \frac{\text{Weight of sediment paste} \times 100}{\text{Weight of sample on dry basis} \times (100 - \%\text{solubility})}$$

pH

The pH of each starch suspension was determined using a digital pH meter (model 3305 Jenway). Starch samples for pH measurements were prepared by suspending 1 g of starch in 25 ml of water at 25°C and agitating for 5-10 min (Sokhey and Chinnaswamy, 1993).

Free acidity

Free acidity was determined by titrating a starch suspension against 0.023 N standardized

NaOH solution. The starch suspension was prepared by suspending 1 g of starch sample in 10 ml of degassed, boiled distilled water in a beaker and continuously shaking for 5-10 min. The results were expressed as total neutralized acidity (Sokhey and Chinnaswamy, 1993).

Colour

The colour of sago starch was determined using a colorimeter (Hunter Lab Ultra Scan SN 7877, Reston, VA, USA) standardized with a white chromatic reflectance standard (L = 98.94; a = -0.17; b=0.04). Samples were put in transparence plastic bags. The L, a and b values denotes lightness, redness and yellowness, respectively.

Intrinsic viscosity

Intrinsic viscosity was measured using the procedure described by Leach (1963) with a Haake Falling Ball Viscometer (Model KF10, Germany). Two hundreds ml of starch suspensions (1% w/v) in a 250 ml centrifuge bottle were heated in a water bath for 30 minutes. Following cooling, solutions (100 ml) with concentrations ranging from 0.02% to 0.01% were prepared from the cooked paste (1% starch solution), and the solvent used was 1M potassium hydroxide. Solubilization of the starch polymers by potassium hydroxide was carried out over a period of 16 hours. Obtaining a clear solution indicates complete solubilization of the starch solution. The solubilized starch solutions were centrifuged (1,500 rpm, 10 minutes) to remove any insolubles before the determination of viscosity takes place. Flow times of Ball No.2 between the ring marks were measured using a stopwatch (resolution 0.01s). The viscosity was calculated using the formula:

$$\eta = t(\rho_b - \rho_s).KF$$

where:

η = viscosity (mPas)
t = time measured between ring marks (s)
ρ_b = density of ball (g/cm^3)
ρ_s = density of sample (g/cm^3)
K = ball constant (mPa.g/cm^3/g)
F = factor for working angle

The intrinsic viscosity was obtained by extrapolating the linear curve of a series of reduced viscosity values at different concentrations (0.02% - 0.10%) to zero concentration. Reduced viscosity was given by the equation:

$$\eta_{red} = (1/C)\,(\eta_1 / \eta_2 - 1)$$

where:

η_{red} = reduced viscosity
C = concentration of starch solution
η_1 = viscosity of starch solution
η_2 = viscosity of 0.5M potassium hydroxide

From the intrinsic viscosity, the molecular weight of the starch was estimated using the Mark-Houwink equation:

$$[\eta] = KMa$$

where:

[η] = Intrinsic viscosity (g/ml)
K = 1.18 x 10^{-3} (Cowie, 1961)
a = 0.89 (Cowie, 1961)
M = average molecular weight

Statistical analysis

The data were analysed using the ANOVA procedure. Significant differences ($P < 0.05$) between means were further determined by Duncan's multiple-range test.

Results and discussion

Pasting profile

The pasting profiles of native and irradiated sago starch are shown in Figure 1. The peak viscosity of irradiated sago starch was drastically decreased as compared to the native sago starch when irradiation dose was increased. When the starch is heated in water, certain characteristic changes occur, the manifest one is a change in rheological behavior. Pasting of many starches exhibits a peak viscosity (P) followed by a mini-

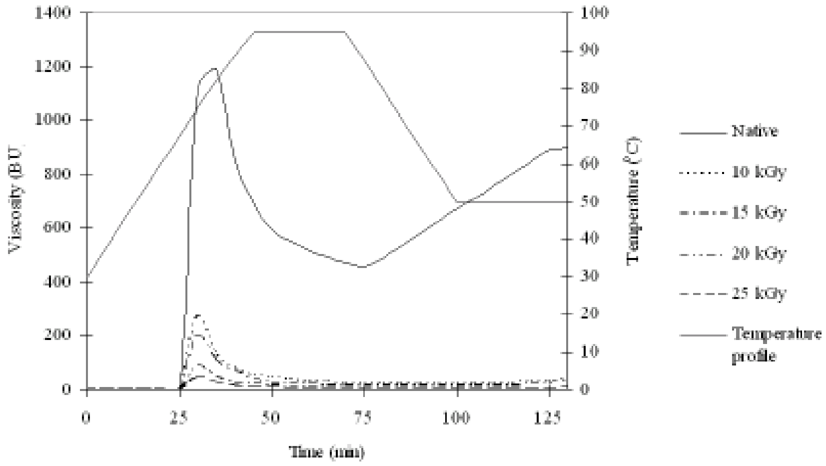

Figure 1. Pasting profile of native and irradiated sago starch

mum viscosity. The peak viscosity is related to the situation where the granules show an optimum balance between swelling and rigidity. Further heating and shear promote the weakening and disruption of the swollen granules. The difference between the peak and minimum viscosity value is defined as the breakdown. Breakdown viscosity (H) is regarded as a measure of the degree of disintegration of the swollen granules or paste stability. The lower pasting temperature and lower breakdown of irradiated sago starch imply a higher hot paste stability (i.e. more resistant to shear thinning during cooking) (Qain and Khun, 1999) due to the irreversible change in the structure. This drastic reduction in viscosity were caused by cleavage of the glycosidic bond through free radical formation to from smaller carbohydrate units of dextrins of varying lengths, leading to a reduction in the molecular weight (MacArther and D'Appolonia, 1984). Therefore, scissions of the chains probably produce short amylose chains, short linear chains from the branches of amylopectin or small branches of amylopectin. When a high energy electron strikes an organic molecule, it dislodges a valence electron from the molecule, producing a cation radical. Electron bombardment transfers so much energy to the molecules or cation radicals that most of the cation radicals fragment after formation via degradation of H-bond (McMurry, 1999). During cooling of the hot paste, the viscosity increases again to a more or less constant value (setback). Setback viscosity (C) is regarded as a measure of gelling ability or the retrogradation tendency (Shelton and Lee, 2000). The lower setback of irradiated sago starch as compared to the native sago starch exhibited the slower retrogradation as reported by Yu *et al.* (1999). Upon cooling, the irradiated sago starch showed almost similar pattern as in the hot paste. The lower viscosity of irradiated sago starch suggested strongly a small tendency to retrograde and implies that they may have a potential application at low temperature (Qain and Khun, 1999). Amylopectin is responsible to promote swelling. Therefore, the decreased viscosity reveals the inability of amylopectin to hold the granule when water is imbibed. The reason for this was possibly the amylopectin branches being cleaved by energy from the electron beam.

Gel strength

Starch paste after gelatinisation consists of solubilised carbohydrates almost all of which are amylose and of swollen starch granules or their fragments, and it exhibits viscoelastic properties. On cooling of concentrated starch paste, starch gel quickly develops; solubilised carbohydrates form a continuous network which links swollen starch granules (Hikone, 1998). The gel strength of the irradiated sago and native sago starch is shown

Table 1. Characteristics of irradiated sago starch

Characteristics	Irradiation dose (kGy)					
	Native	**10**	**15**	**20**	**25**	**30**
Gel strength	39.85±4.19c	53.60±3.85a	47.90±1.92b	36.05±5.02c	40.13±4.85c	26.38±3.45d
Swelling power	13.27±0.27a	8.74±0.52b	8.59±0.45b	8.32±0.70b	4.67±0.95c	4.07±0.42c
Solubility	13.09±3.23d	22.32±0.47c	31.56±2.43ba	37.47±5.90a	22.22±3.56c	24.95±3.34bc
pH	4.39±0.04a	3.71±0.07b	3.28±0.11c	3.25±0.08c	3.25±0.11c	3.17±0.06c
Free acidity	0.20±0.04d	0.23±0.02d	0.32±0.01c	0.35±0.03c	0.40±0.01b	0.47±0.02a
Colour						
L value	92.88±0.17b	93.91±0.01a	91.74±0.04e	92.88±0.17b	92.50±0.07c	92.09±0.15d
a value	2.90±0.03d	2.26±0.01e	3.63±0.02b	3.22±0.02c	3.60±0.01b	3.81±0.01a
b value	5.91±0.03f	8.84±0.08e	11.98±0.06c	11.76±0.08d	12.84±0.07b	13.59±0.04a

Each value represents the mean±standard deviation of three analyses from two replications. Means within each column with different letters are significantly different (P<0.05)

in Table 1. The gel strength of sago starch was increased when sago starch was irradiated at 10 and 15 kGy. This might be due to the linear glucans derived from the broken amylopectin as an effect of irradiation. When the irradiation dose was further increased, the gel strength was decreased. This might be due to more amylopectin fractions having leached out from the granules into the continuous phase. The reduction in chain length in amylose and amylopectin causes difficulty for the linear molecules to form hydrogen bonds as the distance between ends of the molecules is too far apart (Takeda *et al.*, 1986). Rosenthal (1992) reported that on irradiation, hydrolysis and oxidative degradation of carbohydrate molecules may occur. Smaller saccharides may be oxidized at the end of the molecule to form acid, and as a result of ring scission aldehydes may be formed. Large carbohydrate molecules are split into smaller units by cleavage link, resulting in depolymerization. Thus, chains of carbohydrates may be shorten with a loss of gelling power. It was reported by Fiedorowicz *et al.* (1999) that amorphous zones of starch granule underwent radiolysis by gamma-ray irradiation before their crystallite areas did.

Colour

Colour of starch may differ from one to another. The electron beam irradiation may cause an effect on the colour as it is involved with the high energy. The colour change of native and irradiated sago starches are shown in Table 1. It was found that the a (redness) and b (yellowness) values were increased. These results were slightly different from Kang *et al.* (1999) who reported that there was no noticeable changes in the a value was observed among the corn starch sample while the b value increased with gamma radiation. The difference might be due to different radiation technique and type of starches employed. The change of colour seemed mainly due to the caramelization reaction of monosaccharides obtained from starch polysaccharide degradation as a result of the electron beam machine is capable of hydrolyzing chemicals bonds, thereby cleaving large molecules of starch into smaller fragments of dextrin, sugar, and sugar acids that may be either electrically charged or uncharged as free radicals (Greenwood and Mackenzie, 1963).

Swelling power and solubility

In general, swelling of most starch granules begins at the least organized, amorphous, intercrystallite regions of the granule. As this phase swells, it exerts a tension on neighboring crystallites and tends to distort them. Further heating leads to uncoiling or dissociation of double helical regions and breaks up of amylopectin crystallite structure. The liberated side chains of amylopectin become hydrated and swell laterally, further disrupting crystallite structure. The starch molecules are unable to stretch longitudinally, and actually may have a tendency to contract to approach a random coil conformation (French, 1984). The swelling power of sago starch was decreased as the irradiation dose was increased (Table 1). This was due the scission of the amylose and amylopectin chains in starch molecules (Rosenthal, 1992). Andr'ea *et al.* (2000) reported that the reduction of pasting viscosity could result from a decrease in swelling due to partial depolymerization of amylopectin and/or amylose in amorphous regions. The solubility of irradiated starch is shown in Table 1. It was increased when the irradiation dose was increased. Irradiation of corn, potato, and sweet potato starches resulted in an increase in water solubility, reducing power, and acidity (Radley,1960; El' Saadany *et al.*,1974; Duarte and Rupnow, 1994). Several researchers reported the increment in material soluble in water as evidence of molecular degradation of starch by irradiation (Radley, 1960; Duarte and Rupnow, 1994).

pH

pH of irradiated sago starch was decreased while the acidity was increased (Table 1). Sokhey

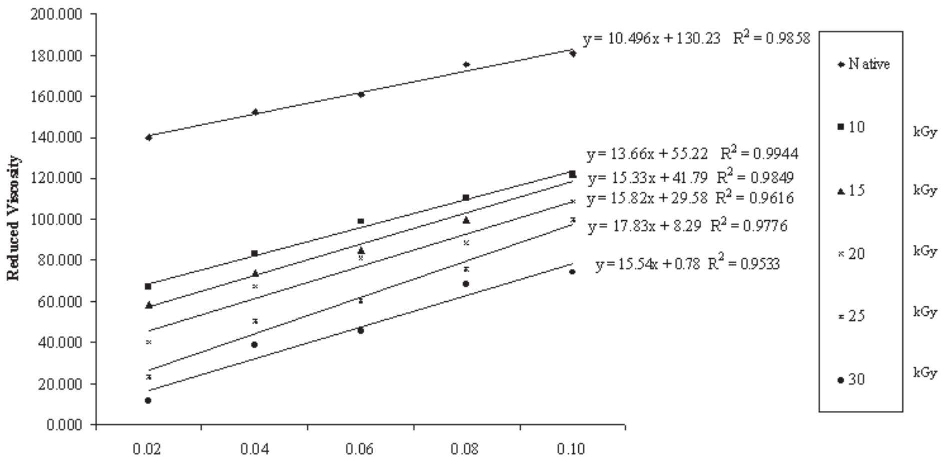

Figure 2. Reduced viscosity of native and irradiated sago starch

Table 2. Intrinsic viscosity, molecular weight and degree of polymerisation of irradiated sago starch

Irradiation dosage (kGy)	**Characteristics**		
	Intrinsic viscosity[a]	**Molecular weight**[b] **(10^5)**	**Degree of polymerization**[b]
Native	130.23	4.57	963.70
10	55.22	1.74	408.62
15	41.79	1.28	309.24
20	29.58	0.87	218.89
25	8.29	0.08	61.34
30	0.78	0.02	5.77

[a] **Obtained from extrapolation of reduced viscosity curve**
[b] **Obtained from calculation as mentioned in methods**

and Chinnaswamy (1993) reported that the increment in acidity of irradiated starches could be due to the breakdown of starch molecules, and presumably induce COOH formation. Similar results were also reported by the action of free radicals which may cause an increase in the starch acidity.

Intrinsic viscosity

The reduced viscosity of native and irradiated sago starch is shown in Figure 2 and the intrinsic viscosity, taken from the extrapolation of curve of the reduced viscosity is shown in Table 2. According to the relationship between number average degree of polymerisation (DP_n) and intrinsic viscosity $[\eta]DP_n = 7.4[\eta]$ Cowie (1961), the DP of native and irradiated sago starch was estimated (Table 2). Mw and DP of irradiated sago starch were decreased when the irradiation dose was increased. This result confirmed the reduction of pasting viscosity and swelling power due to electron beam irradiation capable in ionising the amylose and amylopectin chain. Table 3 shows the correlation coefficients between physicochemical characteristics of native and irradiated sago starch. The increase in solubility of starches could be a good indication of the reduction in molecular weight because it had significantly negative correlation as shown in Table 3. The reduction in DP value indicated the degradation of starch polymer chains. However, the determination of the DP value from the intrinsic viscosity only provides a rough estimation of the DP of starch as a whole and does not give any information of the individual chain length of amylose and amylopectin.

Conclusion

The physicochemical characteristics of sago starch were affected by the electron beam irradiation. The degradation of amylose and amylopectin resulted in the change in physicochemical characteristics. The peak viscosity, yellowness, redness, solubility and free acidity were increased when the irradiation dose was increased. The gel

Table 3. Correlation coefficients between physicochemical characteristics of native and irradiated sago starch

Main effect	Dose	peak viscosity	gel strength	swelling power	solubility	pH	Free acidity	L value	a value	b value	[η]	Mw	DP
Dose	1												
peak viscosity	-0.90626*	1											
gel strength	-0.24414	-0.11527	1										
swelling power	-0.81331	0.97684*	-0.32076	1									
solubility	0.98394*	-0.93795*	-0.18827	-0.84917	1								
pH	-0.93285*	0.96199*	0.04319	0.89789*	-0.09801*	1							
Free acidity	0.96658*	-0.79037	-0.40704	-0.66243	0.94971*	-0.88179*	1						
L value	-0.3131	0.13688	0.29976	0.03187	-0.3746	0.38611	-0.50852	1					
a value	0.59127	-0.3364	-0.49653	-0.1906	0.60967	-0.56432	0.76825	-0.92659*	1				
b value	0.95925*	-0.91693*	-0.13241	-0.8357	0.98729*	-0.98209*	0.94434*	-0.49021	0.68352	1			
[η]	-0.96948*	0.97838*	0.0124*	0.9379*	-0.97445*	0.96336*	-0.88672*	0.22006	-0.45884	-0.9557*	1		
Mw	-0.96488*	0.98096*	-0.00569	0.93712*	-0.97096*	0.96259*	-0.87864*	0.21229	-0.44779	-0.95281*	0.99983*	1	
DP	-0.96948*	0.97838*	0.01239	0.9308*	-0.97445*	0.96336*	-0.88672*	0.22006	-0.45884	-0.9557*	1.0000*	0.99983*	1

*** Significance at 0.05 level (P<0.05)**

strength, swelling power and pH were decreased when the irradiation dose was increased. The intrinsic viscosity was decreased when the irradiation was increased.

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