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Effect of γ-irradiation on pasting and emulsification properties of octenyl succinylated rice starches

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ABSTRACT

Octenylsuccinylated (OS) starches from waxy rice or high-amylose rice (28.1% amylose) (DS 0.023 and 0.025, respectively) were gamma-irradiated at 10, 30, or 50 kGy and their pasting and thermal properties, crystallinity, and emulsification property were examined. When the OS starches were irradiated, the degrees of substitution gradually decreased as irradiation dose increased. A significant decrease in pasting viscosity was observed with an increase in irradiation dose, indicating the presence of chain degradation induced by the radiation. The melting temperature and enthalpy determined by differential scanning calorimetry increased slightly by irradiating at 10 or 30 kGy. Little change in crystallinity was observed in the X-ray diffraction analysis for the OS high-amylose rice starch regardless of irradiation doses, whereas a decrease in crystallinity was observed with the OS waxy starch irradiated at 50 kGy. Chain degradation induced by irradiation occurred mainly in the amorphous regions, but some loss of crystallinity occurred when the irradiation was excessive. The OS starches showed greater emulsion capacity and stability than the native counterparts due to their amphipathic nature. The irradiation further improved the emulsification properties of OS starches. The irradiation at 10 kGy was optimal, and treating at higher doses decreased the emulsion capacity and stability of the OS starches.

1. Introduction

Octenylsuccinylated (OS) starches, which were first introduced by Caldwell and Wurzburg (1953), have been used in various food products. The OS groups are covalently attached to starch chains through ester linkages, and impart surface activity to the starch molecules (Trubiano, 1986). OS starch has health benefits due to a high proportion of slowly digestible starch and low proportion of rapidly digestible starch (Han & BeMiller, 2007), which prevents a dramatic increase in blood glucose levels. In addition, OS starch or its dextrin may replace fat in foods reducing total calories (Cho, Lim, Park, Hwang, & Lim, 1999; Kim, Singh, Lee, Lim, & Lim, 2010) or be used in cholesterol-free foods (Tesch, Gerhards, & Schubert, 2002). The most attractive application of OS starch may be as an emulsifier in salad dressings, creams and coatings by producing strong films at the oil–water interface and emulsions that are resistant to reagglomeration (Liu et al., 2008). These properties result from bi-functional groups that are both hydrophilic (sugar part) and hydrophobic (octenyl part). Some OS starches and dextrins are used to encapsulate. Examples of the commercially available OS starches and dextrins are under the brand names of HiCap, Capsulm, NLokm, EmCap, and Cleargum, which are mostly used to encapsulate food ingredients (Bhosale & Singhal, 2006).

Dextrins are usually produced by hydrolyzing starches with acids or enzymes (Cho et al., 1999; Thomas & Atwell, 1999) or by dry heating (Kim et al., 2010; Chung, Lee, Han, & Lim, 2010). Acidic hydrolysis requires corrosion-resistant materials to prevent the formation of off-color and ash content increase (due to neutralization). Enzymatic hydrolysis is more costly and requires greater efforts to optimize the reaction (Liu et al., 2008). Alternative physical treatments such as microwave, UV and gamma-ray irradiation, ultrahigh hydrostatic pressure, and hydrothermal treatment could be used to produce starch dextrins. Among them, the advantages of gamma-irradiation are that it requires only a short treatment time, no chemicals, no additional processing, and produces no waste (Lotfy, 2009). Radiation energy generates a free radical at the C_1 position on the glucosyl unit, so induces scission of glycosidic linkages (Raffi & Agnel, 1983). Gamma-irradiated starch displays reduced viscosity and increased solubility due to depolymerization (Sokhey & Hanna, 1993). However, moderate irradiation doses induced no physical damage to the irradiated starch (Sonntag, 1980; Lee et al., 2006).

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Rice starch has been variously used in industrial applications as well as in foods (Champagne, 1996) and the functional property of rice starch has been influenced by amylose–amylopectin ratio (Jane et al., 1999). It was reported that high amylose rice starch showed longer average chain length, higher portion of long B chain (DP > 37), and lower portion of short A chain (DP 6–12) than waxy rice starch (18.8 vs. 18.1; 8.7 vs. 7.9%; 26.9 vs. 33.8%, respectively) (Chung, Liu, Lee, & Wei, 2011). In this study, gamma-irradiation was applied to the OS waxy or high amylose rice starch with different amylose content to produce OS dextrins, and its effects on the physicochemical properties of the dextrins were examined.

2. Materials and methods

2.1. Materials

Milled grains from high-amylose and waxy rices harvested in 2010 (Chulwon, Korea) were purchased from a local store in Seoul, Korea. Rice starches from the grains were isolated using the alkali method (Lim, Lee, Shin & Lim, 1999). The apparent amylose content in high and waxy rice starch was 28.1% and 0%, respectively. Ocetenylsuccinic anhydride was purchased from Sigma–Aldrich Chemical Co. (Milwaukee, WI, USA).

2.2. Preparation of OS starch

Rice starches were octenylsuccinylated according to the procedure reported by Han and BeMiller (2007). An aqueous dispersion of starch (45% starch solids) was prepared, and the pH of the slurry was adjusted to 8.5–9.0 with 1 M NaOH soluiton. An aliquot of 2-octen-1-ylsuccinic anhydride (3.0% of starch solids) was added slowly to the starch slurry with vigorous stirring. The starch slurry was continually agitated for 6 h at room temperature while maintaining a pH of 8.5, which was neutralized to pH 6.5–7.0 with 1 M HCl solution, and then centrifuged (2500 rpm, 15 min). The precipitates were washed three times with water and finally with acetone, and then air-dried (40 °C) in a convection oven.

2.3. Gamma irradiation

The native and OS rice starches were packed in sealed polyethylene bags and irradiated with Cobalt-60 gamma-rays (AECL, IR-79, Nordion International Co. Ltd., Ontario, Canada, 100 kCi) in the Korea Atomic Energy Research Institute (Jeongeup, Korea). The irradiation was performed at 18 °C for up to 5 h at an irradiation rate of 10 kGy/h. The absorbed dose was verified using a Fricke dosimeter (ceric/cerous dosimeter) (Holm & Berry, 1970).

2.4. Degree of substitution (DS)

The DS of the OS starches was determined using the titration method (Kweon, Choi, Kim, & Lim, 2001) with slight modifications. Starch or dextrin was dispersed in a 2.5 M HCl-isopropyl alcohol solution (4.5 g dry solids in 25 mL), and then a 90% isopropyl alcohol solution (50 mL) was added. The suspension was filtered through a glass filter after 10 min with stirring, and the residue was washed with 90% isopropyl alcohol solution until no Cl^- was detected (using 0.1 M $AgNO_3$ solution). The starch was redispersed in 300 mL of water and gelatinized by heating in a boiling water bath for 20 min. The starch solution was then titrated with a 0.1 M NaOH solution, using phenolphthalein as an indicator. A blank of native starch was also titrated and used as a control. DS was calculated with the following equation, where A is the titration volume of the NaOH solution (mL), M is the molarity of the NaOH solution, and W is the dry weight (g) of starch or dextrin:

$$\mathrm{DS} = \frac{0.162 \times (A \times M)/W}{1 - \left[0.210 \times \frac{A \times M}{W}\right]}$$

2.5. Pasting viscosity

Pasting viscosity of the OS starches dispersed in distilled water (7%) was determined with a Rapid Visco-Analyser (Newport Scientific, Warriewood, Australia) using the Profile: equilibration to 50 °C for 1 min, heating to 95 °C at 10 °C/min, holding at 95 °C for 2 min, cooling to 50 °C at 10 °C/min, and holding at 50 °C for 1 min.

2.6. Thermal transition properties

Thermal transition properties of the OS starches were determined by differential scanning calorimetry (DSC 6100, Seiko, Japan). Sample (approximately 3 mg) was weighed into an aluminum DSC pan and distilled water (sample:water = 1:4) was added. The sample pan was sealed and then heated from 25 to 160 °C at a heating rate of 10 °C/min.

2.7. X-ray diffraction

Crystallinity of the OS starches was examined using an X-ray diffractometer (X'Pert MPD, Philips, Almelo, Netherlands) at 40 kV and 30 mA, with nickel-filtered Cu-K_α radiation. Scattered radiation intensities were measured in a 2θ range between 3° and 40° at a rate of 0.02°/s.

2.8. Water solubility

The OS starches (200 mg, dry solids) were dispersed in 10 mL distilled water and then magnetically stirred for 50 min. The suspension was centrifuged at 1400 × g for 15 min. The carbohydrate content in the samples and supernatants was measured using the phenol sulfuric method (Dubois, Gilles, Hamilton, Rebers, & Smith, 1956).

2.9. Emulsion capacity and stability

Emulsion capacity (EC) was determined using the method described by Jiménez-Colmenero and García-Matamoros (1981) with slight modifications. The OS starches were dispersed in distilled water (300 mg dry solids in 100 mL), and the dispersion was heated at 85 °C for 20 min and then cooled to room temperature. The pH was adjusted to 5.5 by adding 0.1 M NaOH and then the dispersion was homogenized (Ace Homogenizer, Nissei AM-3, Tokyo, Japan), at 2000 rpm for 45 s. The mixer speed was switched to high speed (5000 rpm), and corn oil was gradually added. When the emulsion had collapsed, the volume of oil added was measured, and the EC was expressed in mL of oil/mL of the sample, The emulsion prepared with 2 g (dry solids) of starch, 20 mL water, and 20 mL corn oil was centrifuged at 800 × g for 10 min to assess emulsion stability (ES). The emulsion was then transferred to a mass cylinder (100 mL) and kept at room temperature for 24 h. ES was expressed as the volume percentage of the emulsion against the total solution.

2.10. Statistical analyses

Experimental data were analyzed using SPSS system v.8.2, (SPSS Inc., Chicago, IL, USA) and Duncan's multiple comparison tests. All analyses were performed in triplicate.

Table 1
Degree of substitution (DS)[a] of OS waxy and high-amylose rice starches after irradiation at different doses (0–50 kGy).

Starch	Dose			
	0	10	30	50
OS waxy	0.0231[b]	0.0192[cd]	0.0154[e]	0.0146[e]
OS high-amylose	0.0254[a]	0.0219[bc]	0.0207[c]	0.0186[d]

[a] All data were means of triplicates. Data with different letters are different with significance ($p < 0.05$) by the Duncan's least significant test.

3. Results and discussion

3.1. Degree of substitution (DS)

The appropriate DS for food applications of OS starches is 0.01–0.03 (Nilsson & Bergenståhl, 2007). The DS value of OS starches depends on both amylose content and reaction conditions: 0.024–0.03 (He, Song, Ruan, & Chen, 2006), 0.0149–0.0232 (Kim et al., 2010), and 0.011–0.018 (Song, He, Ruan, & Chen, 2006). The DS values of the OS rice starches prepared in this study are shown in Table 1. The OS high-amylose rice starch had a slightly higher DS value (0.0254) than that of the OS waxy rice starch (0.0231). Amylose content had a positive impact on the substitution with octenylsuccinyl anhydride (He et al., 2006), because the amorphous domains in which amylose mainly resides are more accessible by the reagent than are the crystalline lamellae.

DS values of the OS starches, decreased gradually by increasing irradiation dose, implying removal of OS groups as a result of irradiation. Starch chains were also degraded by irradiation (Sabularse, Liuzzo, Rao, & Grodner, 1992; Yu & Wang, 2007), mainly in the amorphous regions (Bao, Ao, & Jane, 2005; Lee et al., 2006).

3.2. Pasting viscosity

Determination of pasting characteristics and the paste viscosity profile is an effective tool to evaluate the modification and cooking properties of starches (He et al., 2006). Pasting viscosity profiles of the OS rice starches are shown in Fig. 1. The OS starches exhibited higher peak viscosities and lower pasting temperatures than those of unmodified native starch. The OS waxy rice starch, prior to irradiation, displayed about four times greater peak viscosity than that of native starch. The increased viscosity and decreased pasting temperature of the OS starches could be explained by the bulky OS groups which may have caused steric hindrance inhibiting chain association, and increasing water penetration into starch granules (Bao, Xing, & Phillips, 2003). Additionally, an associative effect of bulky hydrophobic chains by network formation due to hydrophobic interactions could be another reason for the increased viscosity (Ortega-Ojeda, Larsson, & Eliasson, 2005; Thomas & Atwell, 1999). Irradiation decreased the pasting viscosity of the OS starches significantly. Yu and Wang (2007) reported that the decrease in viscosity of irradiated starch was due to starch chain degradation. The irradiation-induced degradation is different from the hydrolysis by acids or enzymes (Ciesla, Zoltowaski, & Mogilevski, 1991), although the exact mechanism is not clear. Because peak viscosity decrease resulted mainly from cleaving large amylopectin molecules into smaller fragments (Tester & Morrison, 1990), the overall decrease in viscosity after irradiation might be caused by amylopectin degradation as well as amylose degradation. It was noteworthy that almost no change was observed in the shape of pasting curves and pasting temperature by increasing irradiation dose. Wu, Shu, Wang, and Xia (2002) and Baik, Yu, Yoon, Lee, Byun, Baik, and Lim (2010) also reported no visible changes in gelatinization temperature of irradiated rice and maize starch, respectively. However, the time for peak was apparently shortened with increasing dose, and the increased

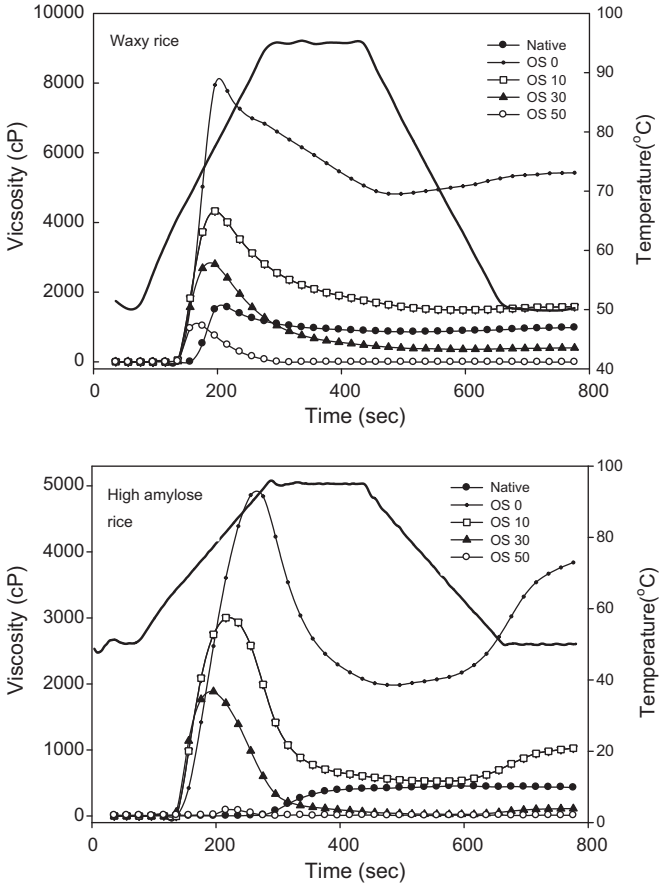

Fig. 1. Pasting viscosity of OS waxy and high-amylose rice starches irradiated at different doses (0–50 kGy).

starch solubility could be one reason for this result (Deschreider, 1960).

Esterification with OS groups resulted in more substantial change in pasting viscosity for high-amylose starch than did for waxy starch (Fig. 1). For OS high-amylose rice starch, peak viscosity was ten times higher (453 cP vs. 4915 cP) than that of native starch. In addition, the OS starch started pasting at a much lower temperature than did the native counterpart (64.5 vs. 90.5 °C). These dramatic changes induced by the OS esterification might be related to the amylose content, because amylose is known to be more susceptible to OS esterification. The OS high-amylose rice starch also showed decreased pasting viscosity as irradiation dose increase, however, unlike OS waxy rice starch, OS high-amylose rice starch showed a gradual decrease in pasting temperature with the increase in irradiation doses, except at 50 kGy. The setback of OS high-amylose rice starch prior to irradiation was 1885 cP; and the value decreased to less than 100 cP at 30 or higher irradiation. The decreases in pasting temperature and setback were obvious evidences for the degradation of amylose chains.

3.3. Thermal transition properties

High-amylose rice starch showed a much higher onset temperature (T_o) and lower melting enthalpy (ΔH) than waxy rice starch in the thermal transition analysis (Table 2). The higher melting enthalpy reflects as greater loss in molecular order (Cooke & Gidley, 1992). Usually, high-amylose corn starch did not show a melting peak; however, high-amylose rice starch showed a distinct melting peak. This could be explained by the differences in amylose content: 50% or 70% in the high-amylose corn starches (Hylon V

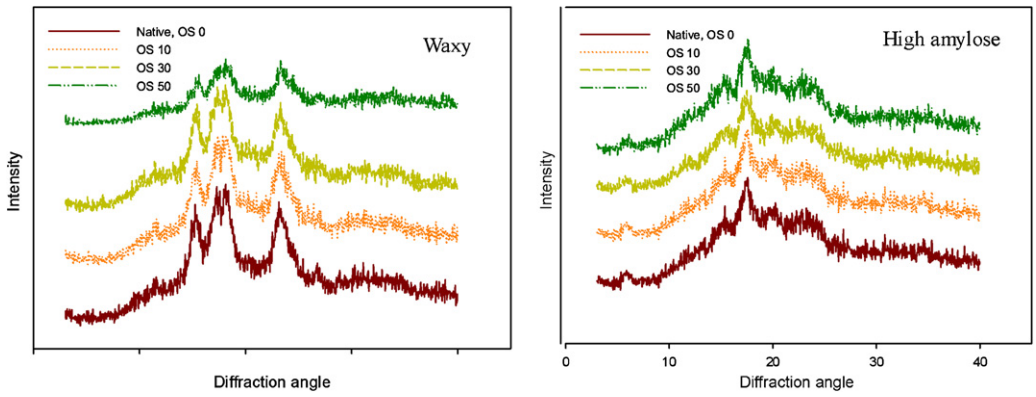

Fig. 2. X-ray diffraction patterns of OS waxy and high-amylose rice starches after irradiation at different doses (0–50 kGy).

or Hylon VII, respectively), and 26–35% in high-amylose rice starch (Choi & Shin, 2009; Sorada & Athapol, 2002; Wu et al., 2002).

The OS rice starches showed much lower T_o and ΔH than those of their native counterparts, because the OS groups reduced internal hydrogen bonding, helping starch granules swell at lower temperature (Bao et al., 2003). Additionally, the bulky hydrophobic groups enhance structural flexibility, contributing to the reduction in gelatinization temperature and melting enthalpy (Lawal, 2004). Irradiation at 10 or 30 kGy slightly increased the T_o and ΔH for both OS high-amylose and OS waxy rice starches. The ΔH increase might be related to the destruction of amorphous regions or relatively weak crystalline structure, inducing increased stability of the remaining crystallites (Singh, Singh, Ezekiel, & Kaur, 2011). Also, it has been reported that not only the cleavage of glycosidic bond, but new chemical bonds formation between anhydroglucosyl units could be induced as a result of transglucosidation after irradiation (Rombo, Taylor, & Minnaar, 2001). However, the enthalpy value decreased at 50 kGy for OS waxy rice starch, although no change was observed in OS high-amylose rice starch. The decrease in melting temperature and enthalpy at 50 kGy has been reported for both corn starch (Lee et al., 2006) and rice starch (Bao & Corke, 2002). It indicates that an excess irradiation induces the decomposition of crystalline structure.

Melting characteristics of amylose–lipid complex in high-amylose starches were also affected by gamma irradiation (Table 2). An amylose–lipid complex of OS starch melted at a lower temperature than that of native starch, although no change in melting enthalpy was found. The T_o value for the amylose–lipid complex in OS high-amylose rice starch slightly increased at 10 kGy, but decreased at higer kGy. Data showed that the irradiation affected not only the amylopectin crystals (low temperature endotherm) but also the amylose–lipid complex. Notably, the melting enthalpy was the highest at 50 kGy irradiation, suggesting that more amylose–lipid complex might be additionally formed by the intense irradiation. It could be expected that amylose chains were readily degraded by irradiation due to their accessibility, and the increased chain mobility by the chain degradation, in turn, possibly made the amylose more capable of complex formation with lipids.

3.4. X-ray diffraction

X-ray diffraction patterns of the irradiated OS starches are shown in Fig. 2. Native waxy rice starch exhibited a typical A-type diffraction pattern (Eliasson, 2004) with peaks at about 15 and 23° ($2\langle\theta\rangle$), and unresolved doubles at around 17 and 18° ($2\langle\theta\rangle$>). No differences in X-ray patterns were observed between native and OS starches, similar to the previous reports (Shogren, Viswanathan, Felker, & Gross, 2000; Song et al., 2006), indicating that OS esterification was located mainly in the amorphous regions of starch granules. High-amylose rice starch showed a typical B-type X-ray diffraction pattern with a peak at around 17° and a characteristic peak at about 5.6° ($2\langle\theta\rangle$>) as reported by Yano, Okuno, Kawakami, Satoh, and Omura (1985). As with waxy rice starch, esterification by OS group did not affect the crystalline pattern of high-amylose rice starch.

No change in the crystal pattern was observed when the OS high-amylose rice starch was irradiated at all doses tested (Fig. 2). For OS waxy rice starches, however, there were minor increases in crystallinity (peak intensity) by irradiating at 10 or 30 kGy; and, a decrease in crystallinity at 50 kGy. The overall changes in X-ray diffraction patterns of OS waxy rice starch appeared to be correlated to the changes in melting enthalpy (Table 2), showing that starch crystallinity may be increased by selective degradation of amorphous starch at

Table 2
Thermal transition properties of OS waxy and high-amylose rice starches after irradiation at different doses (0–50 kGy)[a].

Starches	Peak I				Peak II		
		T_o	T_c	ΔH	T_o	T_c	ΔH
Waxy	Native	65.3^{a}	79.5^{a}	20.9^{a}			
	OS 0	57.7^{c}	71.6^{c}	16.4^{c}			
	OS 10	59.6^{b}	73.6^{b}	18.7^{b}			
	OS 30	58.8^{bc}	72.5^{bc}	18.2^{b}			
	OS 50	56.4^{d}	71.7^{c}	15.8^{d}			
High-amylose	Native	73.4^{a}	88.8^{a}	15.6^{a}	97.4^{a}	111.2^{a}	0.44^{b}
	OS 0	63.8^{d}	79.1^{c}	11.1^{c}	93.2^{c}	107.8^{b}	0.43^{b}
	OS 10	67.5^{b}	81.2^{b}	12.6^{b}	95.1^{b}	106.5^{b}	0.41^{b}
	OS 30	66.9^{bc}	80.4^{bc}	11.9^{b}	93.8^{c}	97.5^{d}	0.35^{b}
	OS 50	65.9^{c}	79.5^{c}	12.3^{b}	93.0^{c}	104.6^{c}	0.71^{a}

[a] All data were means of triplicates. Data with different letters within same column are different with significance ($p < 0.05$) by the Duncan's least significant test.

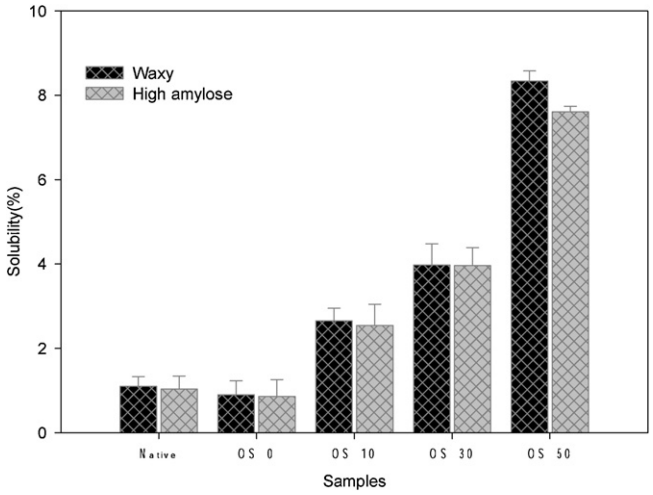

Fig. 3. Water solubility of OS waxy and high-amylose rice starches irradiated at different doses (0–50 kGy).

relatively low doses, but crystalline matrices could be decomposed when the dose was high (50 kGy in this case). It was reported that the crystalline regions of starch are protected from gamma irradiation (Loo, Ooi, & Boey, 2004; Williams et al., 2006) and that chain degradation by gamma irradiation occurs mostly in amorphous regions (Bao et al., 2005; Lee et al., 2006), but in this experiment, crystallinity could be destroyed by excess irradiation. Chung and Liu (2010) also observed a decrease in the relative crystallinity of potato and bean starches after gamma irradiation at 50 kGy.

3.5. Water solubility

Fig. 3 shows the solubility of OS starches after irradiation at different doses. Native and OS starches showed very low solubility (less than 1.0%). The solubility of OS starches was increased significantly by irradiation, and the increase was proportional to the doses. Kerf, Mondelaers, Lahorte, Vervaet, and Remon (2001) reported that the soluble fraction of corn and potato starch increased by irradiation. Sokhey and Chinnaswamy (1993) showed that the solubility of a high-amylose (70%) starch increased from 0.03 to 0.18% by irradiation at 30 kGy. Wootton, Djojonegoro, and Driscoll (1988) also demonstrated by spectrophotometric analysis of the amylose iodine complex that amylose solubility was increased as irradiation dose increased. It could be explained that the increased water solubility resulted from the chain degradation induced by irradiation. For an OS starch to function as an emulsifier or stabilizer of an emulsion, the starch should be soluble and distribute at the interface between fat globules and water (Cho et al., 1999). Thus, the increased water solubility of OS starches by irradiation could positively affect their functions in emulsions.

3.6. Emulsion capacity and stability

Both emulsion capacity and stability for the irradiated OS starches are shown in Fig. 4. The OS starches had a higher capacity for emulsion formation than that its native counterparts, because the OS substitution imparts surface active properties to starch (Trubiano, 1986). The starch chains provide viscosity and mixing properties which could impart stability to emulsions. When OS starch is used for an oil/water emulsion formation, the hydrophilic groups orient themselves in the water phase whereas the hydrophobic alkenyl long chains orient themselves in the oil phase, and the starch chains form a thick interfacial film at the oil/water interface. A mildly irradiated (10 kGy, in this case) OS

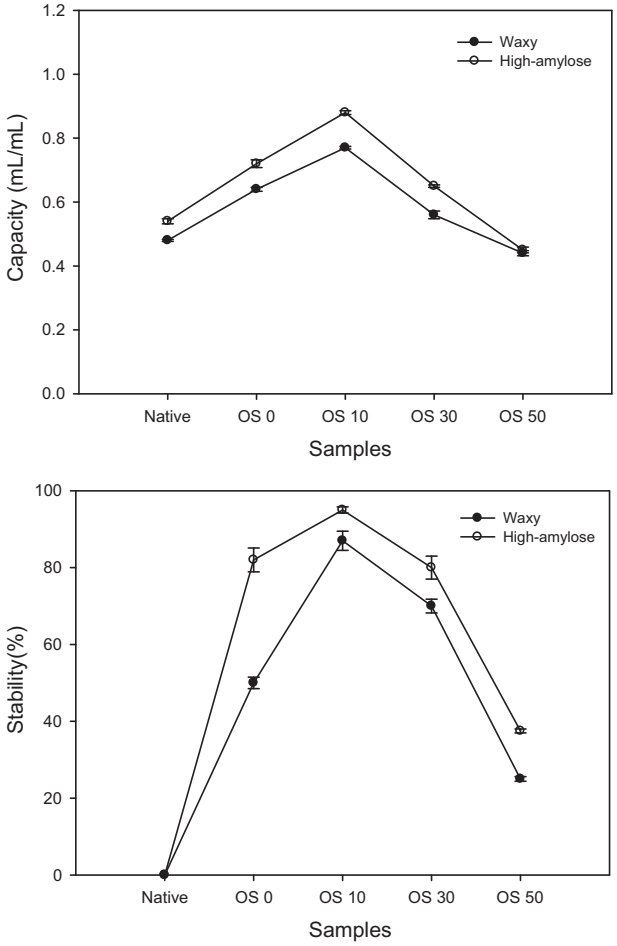

Fig. 4. Emulsion capacity and stability of OS waxy and high-amylose rice starches after irradiation at different doses (0–50 kGy).

starches showed more effective emulsifying capacity than that of non-irradiated OS starch. Liu et al. (2008) also reported that the emulsion capacity of an OS waxy corn starch increased after being hydrolyzed by α-amylase. However, if emulsifiers are too small in molecular size, single-molecule interfacial film is formed and thus emulsion stability may not be maximized (Shogren et al., 2000). Accordingly, the excessive degradation by high doses of irradiation (>10 kGy) decreased both emulsion capacity and stability of the OS starches (Fig. 4). Viswanathan (1999) examined the effect of DS on emulsion capacity and found that high DS did not necessary for maximum capacity. Based on the literatures, emulsifying properties are often functions of amphipathic material size. In this study, OS rice starch irradiated at 10 kGy had the highest emulsion capacity and stability than those of the other starches tested, and this could be explained that the most suitable chain size for emulsification was formed at this dose.

The OS high-amylose starches exhibited higher capacity and stability for emulsion than those of waxy OS starches at the same irradiation dose (Fig. 4). Because the starch functions at the interface between oil and water, linear amylose chains might be more favored than branched amylopectin chains. Therefore, the presence of amylose which is highly capable of film formation could result in improving emulsion capacity and stability of the starch.

4. Conclusions

Gamma-irradiated OS starches exhibited a significant decrease in pasting viscosity. The degradation seemed be occurred mainly

in the amorphous regions of starch granules for OS waxy and high-amylose rice starches, however, decrease in crystallinity observed at 50 kGy might be explained by decomposition of crystalline region. The mild chain degradation of starch chains induced by irradiation (10 kGy) was effective in enhancing the capacity of octenylsuccinylated rice starches for the formation and stabilization of an oil/water emulsion. High-amylose rice starch exhibited superior functions in emulsification to waxy rice starch, possibly due to the existence of linear amylose chains.

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