

# Effect of cross-linking and esterification on hygroscopicity and surface activity of cassava maltodextrins

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## Abstract

The hygroscopicities of cassava maltodextrins, namely cassava maltodextrin (CMD), cassava cross-linked maltodextrin (CCMD), cassava octenyl succinic maltodextrin (COMD) and cassava cross-linked octenyl succinic maltodextrin (CCOMD), gradually increased as the dextrose equivalent (DE) and relative humidity (RH) increased. The hygroscopicities of four types of maltodextrins, with DE 10.2, were 13.42%, 9.89%, 11.80% and 8.91%, respectively, at 25 °C and RH 85%. It was observed that cross-linking and esterification decreased hygroscopicities by 26.5% and 12.1%, whereas the combination of them reduced it by 33.7%. The surface activities of cassava maltodextrins were estimated from the surface tension measurement. At 5%, the air–water surface tensions of CMD, CCMD, COMD and CCOMD solutions were 67.24 mN/m, 64.61 mN/m, 52.93 mN/m and 52.21 mN/m, respectively. The influence of esterification on surface activity was more effective than that of cross-linking.

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*Keywords:* Cross-linking; Esterification; Hygroscopicity; Surface activity

## 1. Introduction

Maltodextrins are enzymatic and/or acid hydrolysis products of starch, consisting of  $\alpha$ -(1,4) linked D-glucose oligomers and/or polymers, which are normally defined as having a dextrose equivalent (DE) value <20 (Klinkesorn, Sophanodora, Chinachoti, & McClements, 2004). They have many specific properties, such as low sweetness, and are odourless, within moderate viscosity. Thus, they are commonly used as spray-drying aids for flavours and seasonings, carriers for synthetic sweeteners, texture providers, fat replacers, film formers, and bulking agents in the food industry (Marchal, Beefink, & Tramper, 1999; Wang & Wang, 2000). Maltodextrins also have some disadvantages. Due to the absence of lipophilic groups, maltodextrins are unsuitable for oil-in-water emulsion systems. They are inconvenient in storage because their hygroscopicities

increase with increasing of DE value (Mollan & Celik, 1995; Viswanathan, 1999a).

With the development of food science and technology, attempts are made to improve maltodextrin properties via chemical modification, hydrolysis processes, and production units (de Miguel, Rieumajou, & Betbeder, 1999; Marchal et al., 1999). Chemical modification most commonly involves oxidation, esterification, etherification, or cross-linking (Richardson & Gorton, 2003). Waxy corn maltodextrin, esterified with octenyl succinic anhydrides (OSA), can provide emulsifying capacity and resistance to enzymatic degradation (Viswanathan, 1999a, 1999b). Cross-linking has been observed to decrease the solubility and water-binding ability of rice maltodextrin (Singha, Kaurb, & McCarthy, 2007). Up to now, little research has been conducted on the hygroscopicity or surface activity of cassava maltodextrin.

Cassava is one of the major starch sources in China; its annual production was estimated to be about 6 million tonnes in 2005. Cassava starch products are becoming increasingly available in many fields. (Huang, Li, Ye, & Cheng,

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2006). In this study, a cassava maltodextrin enzymatic hydrolysis product, was cross-linked with phosphorus oxytrichloride ( $\text{POCl}_3$ ) and/or esterified with OSA. Then, it was used to understand how cross-linking and esterification affected the hygroscopicity and surface activity of cassava maltodextrins.

## 2. Materials and methods

### 2.1. Materials

Commercial cassava starch was obtained from a local market. OSA was purchased from Heico Chemicals (Pennsylvania, USA). Heat-stable  $\alpha$ -amylase was obtained from Genencor International Inc. (Wuxi, China). Phosphorus oxytrichloride was purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). All other chemicals were of analytical grade.

### 2.2. Preparation and chemical modification of maltodextrins

Cassava native starch was hydrolyzed with heat-stable  $\alpha$ -amylase at 95 °C and spray-dried to obtain cassava maltodextrin (CMD). Cassava cross-linked maltodextrin (CCMD) was prepared by treating native starch with  $\text{POCl}_3$  (Woo & Seib, 1997) and then heat-stable  $\alpha$ -amylase before spray-drying. For cassava octenyl succinic maltodextrin (COMD), native starch was treated with OSA (Bao, Xing, Phillips, & Corke, 2003; Shih & Daigle, 2003) and heat-stable  $\alpha$ -amylase, respectively, and spray-dried. Cassava cross-linked octenyl succinic maltodextrin (CCOMD) was produced, first by treating native starch with  $\text{POCl}_3$ , OSA and heat-stable  $\alpha$ -amylase and spray-drying.

Degradation of starch in maltodextrin was indicated by the dextrose equivalent (DE) that expresses the content of reducing-end groups. DE values of all maltodextrins were measured using Fehling's solution, with methylene blue as internal indicator (Lane & Eynon, 1923).

The cross-linking of starch was expressed by sedimentation volume (SV) for low level modification. One gram of modified starch (dry basis) was added to 100 ml of distilled water, heated in a boiling water bath for 20 min with continuous stirring. Once cooled, the sample was placed in a measuring cylinder (100 ml) at room temperature for 24 h. The volume of sedimentation layer was recorded in triplicate and the data are the means of three sets of experiments (Li, Liu, Liu, Li, & Chen, 2005).

The degree of substitution (DS) is the average number of hydroxyl groups substituted per glucose unit. The DS values of COMD and CCOMD were determined by a neutralization titration method. (Joint FAO/WHO Expert Committee on Food Additives, 2001).

### 2.3. Determination of the hygroscopicity

To obtain expected relative humidity (RH), different saturated aqueous solutions were used as described (American

Standard Test Methods, 1992). All the samples (CMD, CCMD, COMD and CCOMD) were dried in vacuum oven at 70 °C for 24 h. About 1 g sample was weighed and spread as a thin layer in an aluminium pan (30 × 20 mm), and placed in a desiccator with saturated salt solution. Then, the desiccator was sealed and held in the thermostat (Ketong Inc., China) at 25 °C for 24 h. Percent moisture absorption of each sample was calculated with respect to its initial dried weight. Each run was performed in triplicate and thus the values of hygroscopicity presented were averages of three sets of experiments (Chandumpai, Singhpi-bulporn, Faroongsarng, & Sornprasit, 2004).

### 2.4. Determination of the surface tension

The air–water surface tensions,  $\gamma$ , of various maltodextrin solutions, were measured by the Wilhelmy plate principle, using a dynamic contact angle analyser (DCA-315, Thermo Cahn, USA) at 25 ± 0.5 °C. The maltodextrin solutions were equilibrated for 12 h, and then a glass plate (18 × 18 mm) was used in measuring the air–water surface tension. The value of surface tension between double-distilled water and air did not differ from the standard value within the range of the experimental error and was 71.92 mN/m. The values presented are the equilibrium surface tensions and averaged data from at least two repetitions (Semenova, Belyakova, Antipova, & Jubanova, 1999).

### 2.5. Statistical analysis

Analysis of variance (ANOVA), Spearman correlation analyses (SAS Institute, Cary, NC, USA), and the Duncan multiple range test were used to compare means of data. Significance levels were used at  $p < 0.05$  and  $p < 0.01$ .

## 3. Results and discussion

### 3.1. The hygroscopicity of maltodextrins

We focussed on the effects of DE value, cross-linking and esterification on the hygroscopicity of various cassava maltodextrins in the present paper. The hygroscopicities of CMD gradually increased with increasing DE (Table 1), and there is a positive correlation between DE value and hygroscopicity at different RH values (RH 11%,

Table 1  
Hygroscopicity (%) of CMD with different DE value at 25 °C<sup>a</sup>

DE	RH (%)			
	11	53	85	100
6.5	4.81 ± 0.26 <sup>d</sup>	10.08 ± 0.09 <sup>d</sup>	11.76 ± 0.28 <sup>d</sup>	13.23 ± 0.30 <sup>d</sup>
8.0	5.50 ± 0.54 <sup>cd</sup>	10.72 ± 0.12 <sup>d</sup>	12.28 ± 0.50 <sup>d</sup>	13.95 ± 0.23 <sup>c</sup>
9.3	6.09 ± 0.19 <sup>c</sup>	11.77 ± 0.46 <sup>c</sup>	12.64 ± 0.44 <sup>c</sup>	14.93 ± 0.15 <sup>b</sup>
10.2	6.84 ± 0.53 <sup>b</sup>	13.27 ± 0.53 <sup>b</sup>	13.42 ± 0.46 <sup>b</sup>	15.29 ± 0.65 <sup>b</sup>
11.8	8.65 ± 0.34 <sup>a</sup>	15.41 ± 0.42 <sup>a</sup>	15.80 ± 0.23 <sup>a</sup>	18.12 ± 0.32 <sup>a</sup>

Different letters within each column show significant difference at  $p < 0.05$ .

<sup>a</sup> Results are expressed as means ± standard deviation.

$r = 0.96$ ,  $p < 0.01$ ; RH 53%,  $r = 0.98$ ,  $p < 0.01$ ; RH 85%,  $r = 0.93$ ,  $p < 0.01$ ; RH 100%,  $r = 0.95$ ,  $p < 0.01$ ). At RH 85%, the hygroscopicity of CMD with DE 11.8 was  $15.80 \pm 0.23\%$ , which was about 34% higher than that of CMD with DE 6.5. Hygroscopicity is the ability to absorb moisture from the surrounding atmosphere and is affected by the type and content of low molecular weight saccharides in maltodextrin (Wang & Wang, 2000). Glucose, maltose and maltotriose are very hygroscopic in their amorphous states and are responsible for strong interaction with the water molecule (Jaya & Das, 2004; Marchal et al., 1999). Maltodextrins with higher DE are characterized by a considerable presence of low molecular weight saccharides, which are the major factors responsible for hygroscopicity. The hygroscopicities of CMD with the same DE gradually increased as RH increased because of more moisture adsorption (Wang & Wang, 2000). Maltodextrin of about DE 10 is adapted for use as a wall material with gum Arabic, mesquite gum providing very good encapsulating capacity, owing to its suitable viscosity and good film-forming ability (Perez-Alonso, Baez-Gonzalez, Beristain, Vernon-Carter, & Vizcarra-Mendoza, 2003; Perez-Alonso, Beristain, Lobato-Calleros, Rodriguez-Huezo, & Vernon-Carter, 2006). Maltodextrin with DE 10.2 was further modified to improve the surface activity and to decrease the hygroscopicity; then it might provide greater availability in scientific and commercial areas.

CCMD possesses phosphodiester linkages, and the degree of cross-linking (DC) can be assessed with the sedimentation volume (SV) of cross-linked starch. With a lower SV, the DC was higher. As the SV decreased, the hygroscopicities of CCMD gradually decreased as shown in Table 2. A positive correlation between SV and hygroscopicity was observed at different RH values (RH 11%,  $r = 0.91$ ,  $p < 0.01$ ; RH 53%,  $r = 0.97$ ,  $p < 0.01$ ; RH 85%,  $r = 0.95$ ,  $p < 0.01$ ; RH 100%,  $r = 0.95$ ,  $p < 0.01$ ). Cross-linking lowered the hygroscopicity of maltodextrins because it probably prevented amylopectin or amylose from dissociating and reinforced the hydrogen bonds with chemical bonds (Craig, Manigat, Seib, & Hosene, 1989). At RH 85%, the hygroscopicity of CCMD with SV 13.70 ml was  $9.89 \pm 0.16\%$ , which was about 26.5% lower than that of CMD with SV 88.68 ml. At the same SV value, the hygroscopicities of CCMD were gradually increased with higher RH.

Table 2  
Hygroscopicity (%) of CCMD with different SV value at 25 °C<sup>a</sup>

SV (ml)	RH (%)			
	11	53	85	100
88.68	$6.84 \pm 0.44^a$	$13.27 \pm 0.18^a$	$13.41 \pm 0.09^a$	$14.81 \pm 0.39^a$
37.33	$6.40 \pm 0.14^{ab}$	$12.04 \pm 0.24^b$	$13.23 \pm 0.37^a$	$13.59 \pm 0.54^b$
32.75	$6.21 \pm 0.29^{bc}$	$11.42 \pm 0.45^c$	$12.66 \pm 0.11^b$	$13.18 \pm 0.60^b$
19.31	$5.73 \pm 0.31^c$	$10.42 \pm 0.42^d$	$11.54 \pm 0.25^c$	$12.01 \pm 0.40^c$
13.70	$4.83 \pm 0.24^d$	$9.23 \pm 0.27^c$	$9.89 \pm 0.16^d$	$10.50 \pm 0.43^d$

Different letters within each column show significant difference at  $p < 0.05$ .  
<sup>a</sup> Results are expressed as means  $\pm$  standard deviation.

COMD is introduced with an octenyl succinic group, which possesses a hydrophilic carboxyl and hydrophobic octenyl tail end. Thus, the hygroscopicity of COMD depends on interaction with moisture. Table 3 shows that the hygroscopicity of COMD was negatively correlated to the DS at different RH values (RH 11%,  $r = -0.63$ ,  $p < 0.05$ ; RH 53%,  $r = -0.75$ ,  $p < 0.05$ ; RH 85%,  $r = -0.86$ ,  $p < 0.01$ ; RH 100%,  $r = -0.85$ ,  $p < 0.01$ ). The incorporation of an OSA group resulted in the reduction of moisture affinity, and the effect of the octenyl chain was more important than the carboxyl group for the hygroscopicity (Tarvainen et al., 2003). At RH 85%, the hygroscopicity of COMD with DS 0.017 was  $11.80 \pm 0.32\%$ , which was about 12.1% lower than that of CMD. At the same DS value, the hygroscopicities of COMD were gradually increased as the RH increased.

To investigate the effect of combination of cross-linking and esterification on hygroscopicity, four types of maltodextrins were compared. As seen in Table 4, there were significant differences among four types of maltodextrins at various RH values ( $p < 0.05$ ). Both CCMD and CCOMD had the same SV, 13.70 ml. COMD and CCOMD had the same DS 0.017. CCOMD had a lower hygroscopicity than had CMD, CCMD and COMD at different RH values. At RH 85%, the hygroscopicity of CCOMD was 33.7%, 11.0% and 24.5% lower than those of CMD, CCMD and COMD, respectively. The results suggest that the combination of cross-linking and esterification had more influence on the hygroscopicity than had individual modification. Maltodextrins with approximately the same DE 10.2, but composed of different saccharide structures, do indeed show different levels of moisture uptake (Kearsley & Birch, 1975).

Table 3  
Hygroscopicity (%) of DS with different DS value at 25 °C<sup>a</sup>

DS	RH (%)			
	11	53	85	100
0.006	$6.05 \pm 0.26^a$	$12.53 \pm 0.37^a$	$13.42 \pm 0.28^a$	$13.88 \pm 0.18^a$
0.009	$5.92 \pm 0.18^a$	$12.42 \pm 0.48^{ab}$	$13.27 \pm 0.30^a$	$13.86 \pm 0.29^a$
0.012	$5.84 \pm 0.59^a$	$12.12 \pm 0.19^{ab}$	$13.01 \pm 0.38^{ab}$	$13.40 \pm 0.56^{ab}$
0.015	$5.69 \pm 0.33^a$	$11.63 \pm 0.14^{ab}$	$12.46 \pm 0.51^b$	$12.76 \pm 0.45^{bc}$
0.017	$5.48 \pm 0.26^a$	$11.01 \pm 0.29^b$	$11.80 \pm 0.32^c$	$12.14 \pm 0.24^c$

Different letters within each column show significant difference at  $p < 0.05$ .  
<sup>a</sup> Results are expressed as means  $\pm$  standard deviation.

Table 4  
Hygroscopicity (%) of different type maltodextrins at 25 °C<sup>a</sup>

Type	RH (%)			
	11	53	85	100
CMD	$6.84 \pm 0.53^a$	$13.27 \pm 0.53^a$	$13.42 \pm 0.46^a$	$15.29 \pm 0.65^a$
CCMD	$4.83 \pm 0.24^{bc}$	$9.23 \pm 0.27^c$	$9.89 \pm 0.16^c$	$10.5 \pm 0.43^c$
COMD	$5.48 \pm 0.26^b$	$11.01 \pm 0.29^b$	$11.80 \pm 0.32^b$	$12.14 \pm 0.52^b$
CCOMD	$4.49 \pm 0.43^c$	$8.79 \pm 0.46^c$	$8.91 \pm 0.36^d$	$9.98 \pm 0.38^c$

Different letters within each column show significant difference at  $p < 0.05$ .  
<sup>a</sup> Results are expressed as means  $\pm$  standard deviation.

The higher RH greatly increases the hygroscopicity of maltodextrins and it is beneficial for maltodextrin products to store them at lower RH (11%).

### 3.2. The surface tension of maltodextrin solutions

COMD and CCOMD, with the incorporation of hydrophobic octenyl succinic groups into a normally hydrophilic backbone, can be used as surfactants in several fields, such as food and pharmacy. Their surface activity generally shows reduction of air–water surface tension. The surface tensions of different DE maltodextrin solutions at concentrations of 1.25% and 5% are shown in Fig. 1. The results indicate that DE had no influence on the surface tension (Semenova et al., 1999). At the same DE value, the surface tensions at 5% were lower than those at 1.25%, which might result from the protein residues and lipids in maltodextrins (Semenova et al., 2003). The effects of SV on the surface tension of CMD are shown in Fig. 2. Surface tension was slightly decreased with a lower SV. It is noteworthy

that the phosphorus bridge bond has excellent flexibility at the air–water interface. Furthermore, the phosphate group, in cross-linking, confers a certain emulsifying capacity because of its ionic property (Singha et al., in press; Solarek, 1986). The surface tension of COMD with DS 0.017 was 52.93 mN/m (Fig. 3). The results indicate that the DS had significant influence on the air–water surface tension, ascribed to the incorporation of the OSA group. The molecules of COMD could orient themselves at the air–water interface and form a monomolecular layer. The reduction of surface tension gave evidence of the strong surface activity of COMD (Tesch, Gerhards, & Schubert, 2002; Trubiano, 1986). To investigate the effect of combination of cross-linking and esterification on surface activity, the air–water surface tensions of four types of maltodextrin solutions were compared (Fig. 4). At 5%, the air–water surface tensions of four types of maltodextrin solutions were 67.24 mN/m, 64.61 mN/m, 52.93 mN/m and 52.21 mN/m, respectively. The surface tensions of COMD and CCOMD solutions were significantly lower

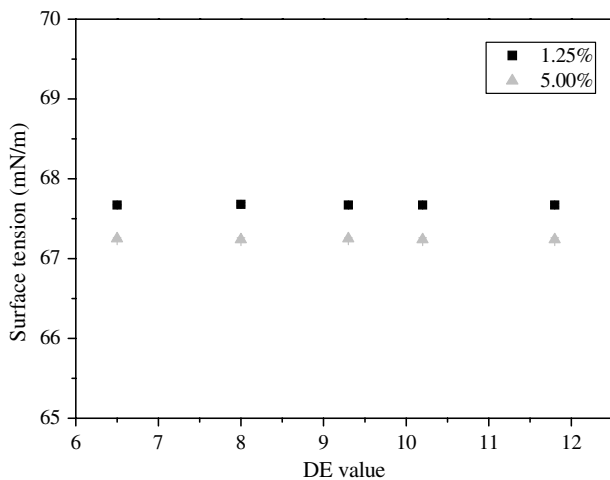


Fig. 1. The surface tensions at different DE values of CMD solutions.

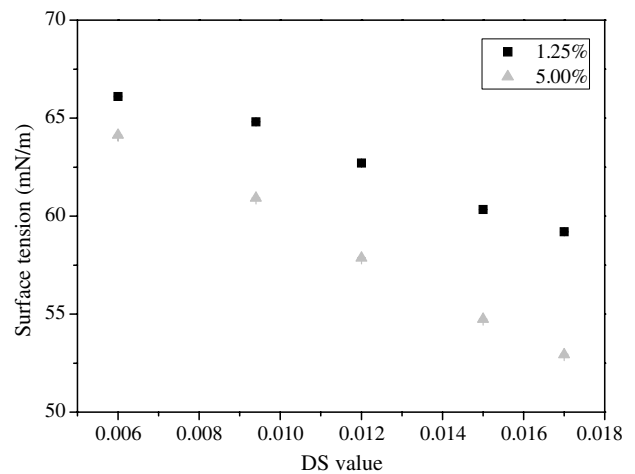


Fig. 3. The surface tensions of different DS COMD solutions.

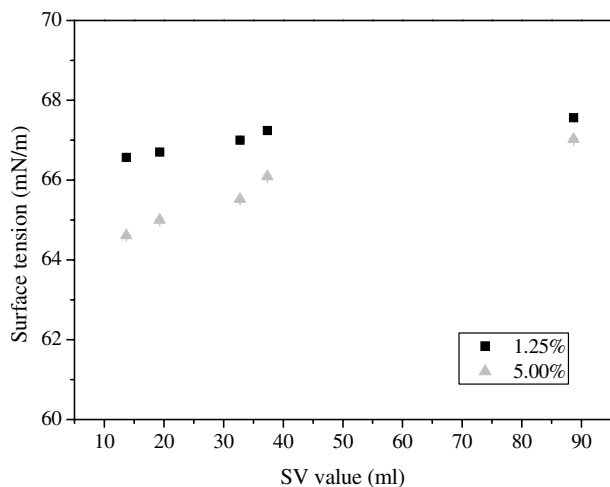


Fig. 2. The surface tensions of different SV CCMD solutions.

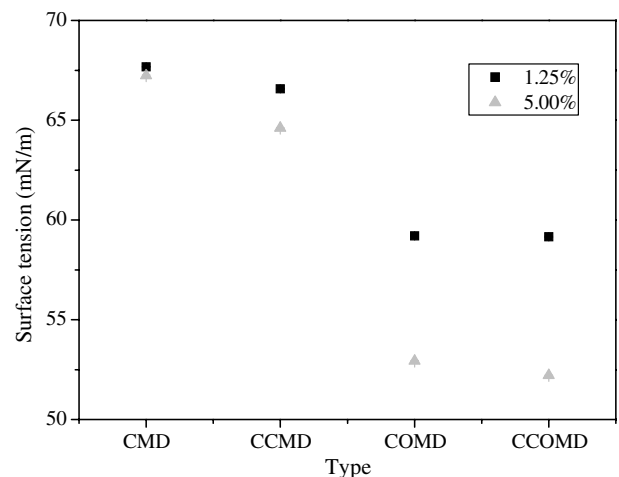


Fig. 4. The surface tensions of different maltodextrin solutions.

than those of CMD and CCMD solutions, and the surface activity of CCOMD was similar to that of COMD ( $p < 0.05$ ). This suggested that the influence of esterification on the surface activity was more effective than was that of cross-linking.

#### 4. Conclusions

In this study, the effects of cross-linking and esterification on the hygroscopicity and surface tension of cassava maltodextrins were investigated. The hygroscopicities of all maltodextrins were gradually increased with the increase of DE and RH. Cross-linking and esterification decreased hygroscopicities by 26.5% and 12.1%, respectively. However, the combination of them reduced it by 33.7%. Higher RH greatly increases the hygroscopicity of maltodextrin and it is beneficial for maltodextrin product to store them at lower RH (11%). At 5%, the air–water surface tensions of four types of maltodextrin solutions were 67.24 mN/m, 64.61 mN/m, 52.93 mN/m and 52.21 mN/m, respectively. The influence of esterification on the surface activity was more effective than was that of cross-linking.

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