



# $^{17}\text{O}$ NMR and DSC for Studying Quality of Taro Paste as Affected by Processing and Storage

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Three kinds of taro paste were prepared with taro:water:sugar ratios of 1:0.33:1.4, 1:0.33:0.8 and 1:0.33:0.2. Results indicated that the paste with the highest sucrose concentration had the least hard texture. The retarding effect of sugar on hardness development and water mobility of the low concentration system was fructose > glucose > lactose > sucrose. The DSC characteristics showed that addition of 10 g/kg sodium stearoyl lactylate (SSL) or 10 g/kg monoglyceride (MG) decreased the hardening effect of low sugar content taro paste during storage under refrigeration. Taro paste with 10 g/kg SSL showed a larger endothermic peak for amylose-emulsifier complex but a smaller peak for retrogradation than did those with 10 g/kg MG or those without emulsifier. Results of  $^1\text{H}$ -decoupled  $^{17}\text{O}$  NMR  $R_2$  for taro paste with emulsifiers or sugars suggested that the changes in interactions among water, sugars, emulsifiers and starches were significantly related to the storage stability of taro paste.

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**Keywords:** taro paste; DSC; NMR; sugar; emulsifier

## Introduction

Taro (*Colocasia esculenta* (L.) Schott) belongs to the family Araceae and is grown mainly as an economic crop throughout the humid tropics in Africa and Pacific rims for its edible corms and leaves (1). It can be consumed as both a staple food and vegetable, and processed as a food ingredient, animal feed, etc. In Asia, taro paste is prepared with cooked taro and mashed into smooth paste, in which sweeteners, shortening and/or seasoning are usually added. Taro paste can be used as fillings for bakery products or desserts because of its unique texture and aroma. However, the quality of taro paste is easily degraded due to staleness, as with other starchy foods, and is also readily deteriorated by microorganism contamination.

As with most starchy foods, retrogradation of the starch component (predominantly the amylopectin fraction) may be the primary reason for the staling of taro paste during long-term storage (2–4). The rate of starch retrogradation is affected by many factors, including the sources (5–7), concentration of starch (8, 9) and storage temperature (10–12). To retard starch retrogradation during storage, addition of polyols (12–18) and emulsifiers (2, 19–21) has been examined with starch gel systems.

Differential scanning calorimetry (DSC) is frequently used to measure the degree of starch retrogradation (2, 7, 8, 12). Recently, nuclear magnetic resonance spectroscopy (NMR) and magnetic resonance imaging (MRI) have also been shown to be desirable techniques for the investigation of retrogradation of starch gels and starchy foods (22–26). Through measurements of the relaxation rate ( $R_1$  or  $R_2$ ) or time ( $T_1$  or  $T_2$ ) of water molecules, by probing the water nuclei (e.g.  $^1\text{H}$ ,  $^2\text{H}$ , or  $^{17}\text{O}$ ), information about the mobility of water molecules in the sample can be obtained and used to predict the storage stability of food products (24, 27, 28). In the present study, the effects of sugars and emulsifiers on the properties of taro pastes during storage were investigated using light microscopy, DSC and  $^1\text{H}$ -decoupled  $^{17}\text{O}$  NMR spectroscopy.

## Materials and Methods

### Materials

Taros (*Colocasia esculenta* var. Bin Lang Hsin Yu) harvested in southern Taiwan from September 1994 to January 1995 were used in this study. Sugars, including sucrose, dextrose (anhydrous), fructose and lactose, and emulsifiers, including monoglyceride (MG) (Amidan SDMT, Grindsted Products, Denmark) and sodium stearoyl lactylate (SSL) (Artodan SP 55M, Grinsted Products, Denmark), were used.

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## Methods

**Preparation of Taro Paste.** Taro paste was prepared according to the flow chart shown in **Fig. 1**. The cooked taro contained 675 g/kg moisture and 307 g/kg starch. Three kinds of taro paste, with high, medium, and low sugar content, were made from cooked taro:water:sugar ratios of 1:0.33:1.4, 1:0.33:0.8, and 1:0.33:0.2. The fresh-made taro paste was packaged and stored under refrigeration ( $4 \pm 1$  °C) or at room temperature ( $25 \pm 1$  °C).

**Sample Preparation for Light Microscopy.** Cooked taro or taro paste was stained with 50 g/kg I<sub>2</sub>-KI solution before microscopic examination. The sample was viewed under a bright-field light microscope (Leitz DMRB, Leica Ag, Switzerland) and photographed with a Wild MPS 52 photo system (Leica Ag, Switzerland).

**Hardness Measurement.** The texture profile analysis (TPA) of taro paste was measured with a TA-XT2 texture analyser (Stable Micro System, England) with a disc plunger (35 mm diameter). The sample of taro paste to be tested was filled into a plastic disc (42 mm diam  $\times$  12 mm height). The test speed was 5 mm/s and the pressing depth was 30% of the sample height. The

hardness of taro paste was expressed in terms of the height of the TPA curve.

**DSC Measurement.** A Setaram DSC 121 (Setaram Co., France) was used to measure the thermal characteristics of taro paste during storage. Taro paste ( $100 \pm 10$  mg) was packed into a stainless steel crucible with a stainless steel stopper sealed by an aluminum O-ring. Samples were scanned from 20 to 150 °C at a rate of 5 °C/min. The measurements were run in triplicate, and an empty stainless steel crucible was used as reference. For each endotherm, the onset ( $T_o$ ) and peak ( $T_p$ ) transition temperatures were determined using a computerized system developed by the Setaram Co. The transition enthalpies ( $\Delta H$ ) were determined from the peak area of the endotherm and expressed as joules per gram of dry matter.

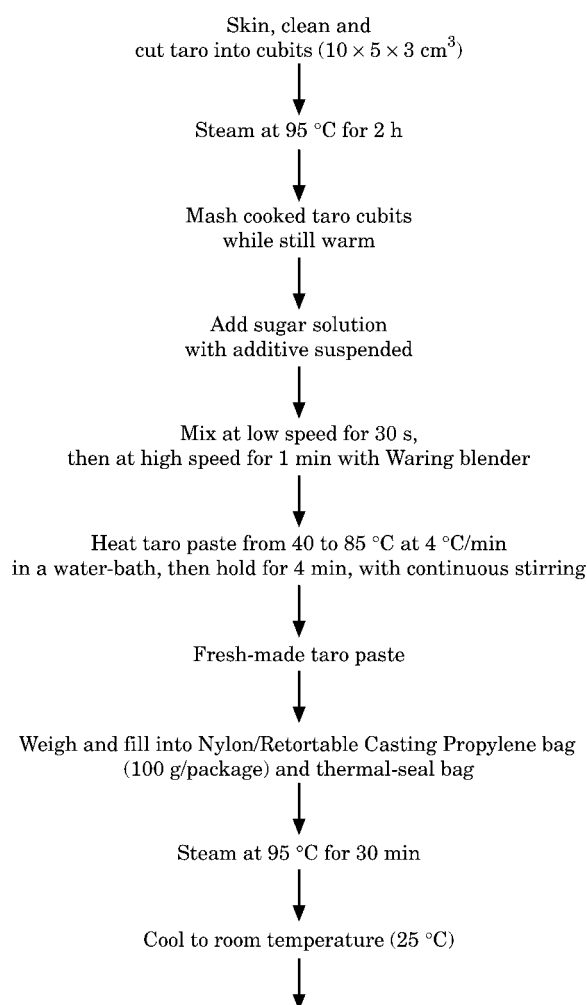
**<sup>1</sup>H-decoupled <sup>17</sup>O NMR R<sub>2</sub> measurement.** A Bruker MSL-200 spectrometer (Bruker Co., France), operating at 27.12 MHz <sup>17</sup>O NMR resonance frequency, was used for the <sup>17</sup>O NMR R<sub>2</sub> measurements. Single-pulse <sup>1</sup>H-decoupled <sup>17</sup>O NMR R<sub>2</sub> experiments were performed in duplicate at  $25 \pm 2$  °C. A 90° <sup>17</sup>O pulse width of 13 μs and a recycling time of 502.03 ms were used. Sweep width was  $\pm 2000$  Hz. The <sup>17</sup>O NMR R<sub>2</sub> measurements were made at natural abundance levels. The sample was placed in a 10 mm NMR tube and spun at  $12 \pm 1$  Hz. The number of scans was 1000 for each sample tested. The same sample tube was used to investigate the changes in water mobility for taro paste stored under refrigeration for 21, 63, 111, 159, 231, 375 and 616 h.

The rotational mobility of water in each sample was monitored by measuring the <sup>1</sup>H-decoupled <sup>17</sup>O NMR R<sub>2</sub>, which was calculated from the line width at half-height of each spectrum multiplied by  $\pi$  (29). If the water molecules, i.e. <sup>17</sup>O in this measurement, relax faster, that generally indicates a larger R<sub>2</sub> (or a shorter T<sub>2</sub>) and less mobility of the water in the sample.

## Results and Discussion

### Light microscopy

**Figure 2** shows the light micrographs of a fresh-cooked sample and a cooked taro sample stored under refrigeration for 1 d. More nondamaged cells with many starch granules inside (tiny dark blue spots) were detected in the micrograph of the fresh-cooked taro than in that of the stored cooked taro. Abe (30, 31) found that the physical properties of Ann (red bean paste) were changed notably, and starch granules were released from the damaged cells of Ann during kneading of cooked red bean. Since the raw material and the processing of red bean paste and taro paste are similar, the amounts of free starch granules released from damaged cells during processing or storage of the taro paste should also contribute to the change in quality of the product. Therefore, the preferred prepa-



Store taro paste under refrigeration or room temperature for testing

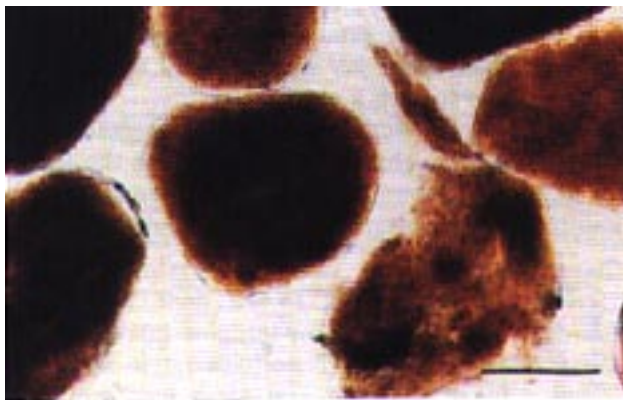
**Fig. 1** Flow chart of the preparation of taro paste sample

ration of taro paste uses fresh-cooked taro, to avoid damaged cells and the release of free starch granules. After 15 d storage of the sample prepared with low sugar content (130.72 g/kg), some cells shrunk slightly, and clusters of starch granules (possibly the amyloplasts) inside the cells were not as clear (Fig. 3b) as those shown in the 1 d-old sample (Fig. 3a). Furthermore, more damaged cells and free starch granules were also found in a sample stored for 15 d than in those after 1 d of storage.

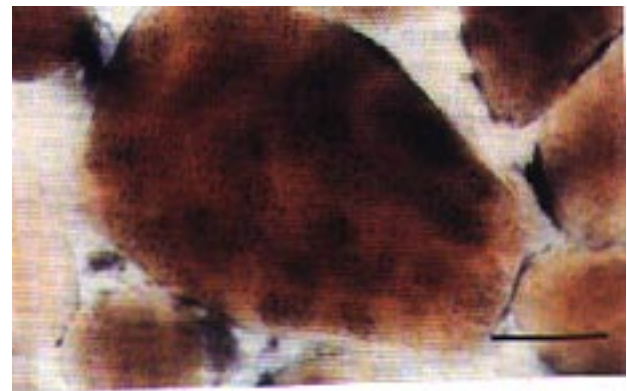
#### *Effects of concentration and variety of sugar*

The effects of sucrose concentration on the hardness of taro paste stored under refrigeration and at room

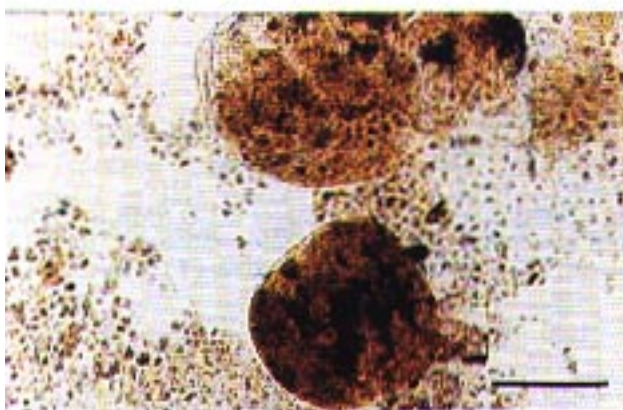
temperature were investigated. The degree of hardness was used as an index for the staleness of a sample. The results showed that sucrose was an effective anti-hardening agent for taro paste, especially at high concentration and under refrigeration (Fig. 4). Sugars are known to retard the rate of retrogradation of starch gel by reducing the rate of crystallization of starch molecules during storage (12, 14, 18). Hence, the decrease in hardness of taro paste with increase in sucrose concentration might be ascribed to less mobile water available for reorganizing amylose and amylopectin molecules in the free starch granules during storage (32), and the effect of high osmotic pressure of the sucrose solution on the cells would restrict swelling of the starch granules. The ability of sucrose to



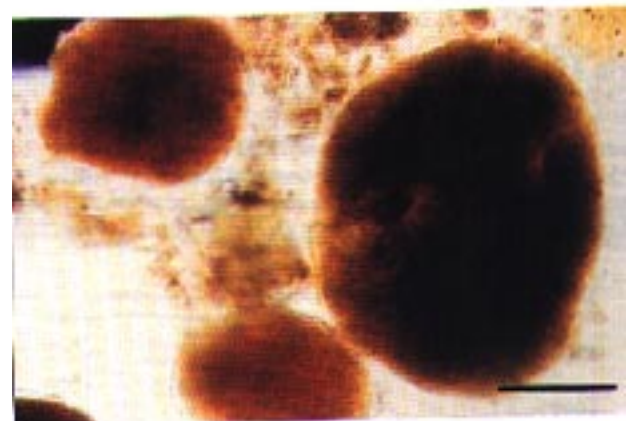
(a)



(a)



(b)



(b)

**Fig. 2** Micrographs of cooked (a) fresh-cooked taro and (b) taro stored under refrigeration for 1 d (bar = 50  $\mu\text{m}$ )

**Fig. 3** Micrographs of low sucrose content (130.72 g/kg) taro paste (a) stored for 1 d, and (b) stored for 15 d under refrigeration (bar = 50  $\mu\text{m}$ )

penetrate the starch granule and interact with starch chains might stabilize the amorphous regions inside the starch granule, which might also slow the rate of hardening (33).

The rate of increasing hardness was much higher under refrigeration temperature than at room temperature (Fig. 4). This result suggested that starch retrogradation was the major factor in the hardening of the taro paste. For most starch gels or starchy foods, the retrogradation rate is inversely proportional to the storage temperature, due to the accelerating effect on the rate of nucleation of crystals at low temperature (10–13).

Figure 5 shows the results for the effects of lactose, fructose and glucose on the hardness of taro paste made with low sugar content and stored at refrigeration temperature. The antistaling effect of the sugars on taro paste stored for up to 20 d was: fructose > glucose > lactose > sucrose. These data were different from those representing the influence on the retrogradation of sweet potato starch gel stored at 5 °C, which showed sucrose > glucose > fructose (19). This discrepancy might be attributed to the fact that taro paste is a

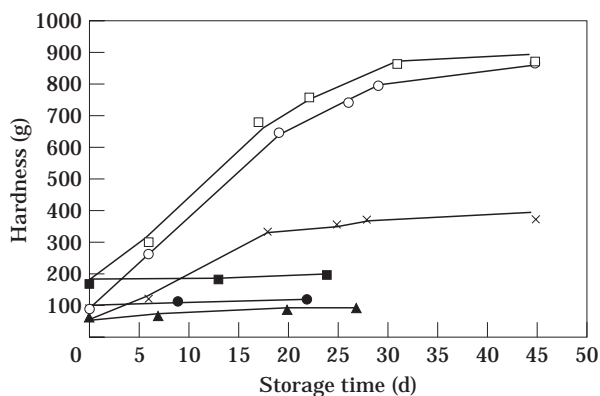


Fig. 4 Effect of sucrose concentration on the hardness of taro paste stored at room temperature and under refrigeration. (□) = low sucrose content (130.72 g/kg); (○) = medium sucrose content (375.59 g/kg); and (X) = high sucrose content (512.82 g/kg) taro paste stored under refrigeration. (■) = low sucrose content; (●) = medium sucrose content; and (▲) = high sucrose content taro paste stored at room temperature

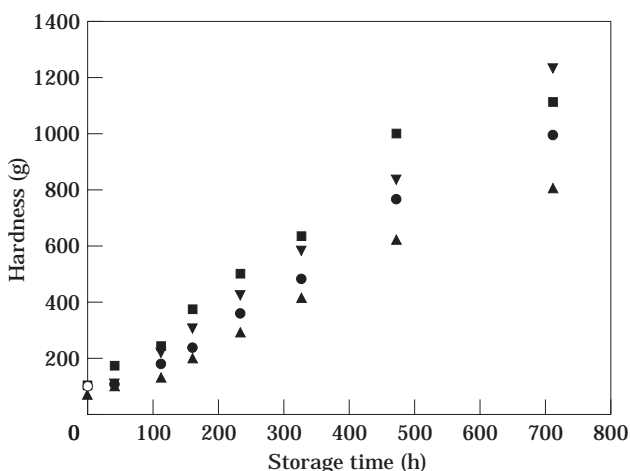


Fig. 5 Effects of various sugars on the hardness of low sugar content taro pastes stored under refrigeration. (■) = sucrose; (●) = glucose; (▲) = fructose; (▼) = lactose

complex system, while sucrose–water–sweet potato starch is a simpler system, and/or that retrogradation of starch was one of the factors affecting the hardness of the sample. It had been shown that the amount of solubilized pectin increased and insolubilized pectin decreased during kneading of red bean paste (34). Hence, nonstarch polysaccharides could also influence the texture of taro paste.

To investigate the changes in water mobility for taro pastes during storage, the <sup>1</sup>H-decoupled <sup>17</sup>O NMR R<sub>2</sub> was measured, and the results are shown in Fig. 6. The R<sub>2</sub> of the sample increased, i.e. the mobility of the water decreased, as storage time increased up to 15 d, then remained steady. Lai *et al.* (28) showed that the decrease in water mobility of modified corn starch paste only lasted for 5 d when the identical technique was applied. This discrepancy was attributed to a relaxation effect caused by structure rearrangement of the sheared starch polymers to relieve the mechanical stress created during sample preparation. The interactions between sugar *per se*, water and starch might also contribute to such a result.

The mobility of water in the taro paste made from fructose was the highest, and from sucrose the lowest (Fig. 6). Lai and Schmidt (29) found that the mobility of water in a sugar solution was only dependent on sugar

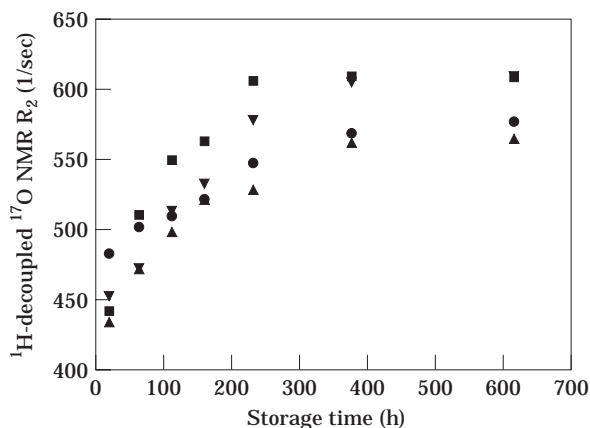


Fig. 6 Effect of sugars on the changes in <sup>17</sup>O NMR R<sub>2</sub> for low sugar content taro pastes during refrigeration storage. (■) = sucrose; (●) = glucose; (▲) = fructose; (▼) = lactose

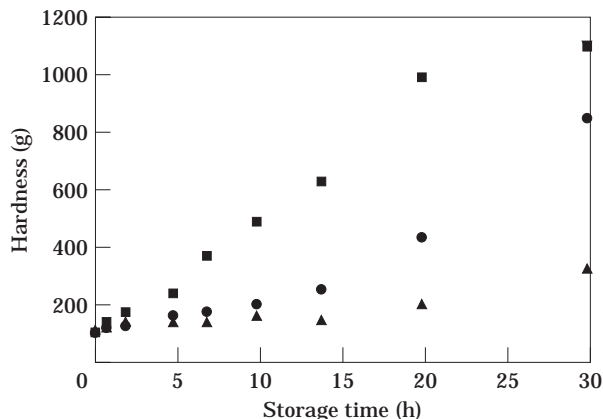


Fig. 7 Effect of emulsifiers on the hardness of low sugar content taro paste during refrigeration storage. (■) = without emulsifier; (▲) = 10 g/kg SSL; (●) = 10 g/kg MG

**Table 1** Effects of emulsifiers on the DSC characteristics of low sucrose content taro paste under refrigeration storage

Additive and storage time	Amylopectin crystallization			Amylose-emulsifier complex		
	Melting temp. (°C)		Enthalpy, $\Delta H$ (J/g, DM)	Melting temp. (°C)		Enthalpy, $\Delta H$ (J/g, DM)
	$T_o$	$T_p$		$T_o$	$T_p$	
<b>Control</b>						
17 h	ND	ND	ND	ND	ND	ND
162 h	50.3	59.5	0.3	ND	ND	ND
328 h	51.5	60.4	1.9	ND	ND	ND
496 h	50.3	59.5	3.1	ND	ND	ND
688 h	50.2	58.8	4.0	ND	ND	ND
<b>10 g/kg SSL</b>						
17 h	57.9	61.1	0.4	100.0	104.8	0.7
162 h	51.9	58.7	0.5	101.3	105.0	0.7
328 h	48.6	59.1	1.0	100.5	105.0	0.7
496 h	50.8	59.7	1.4	98.7	102.6	0.7
688 h	51.7	59.2	1.9	93.5	97.9	0.7
<b>10 g/kg MG</b>						
17 h	57.6	60.0	0.4	100.4	104.5	0.6
162 h	46.3	51.9	0.7	100.3	104.6	0.6
328 h	46.1	52.6	2.0	100.1	104.9	0.6
496 h	47.9	54.5	2.2	100.2	104.2	0.6
688 h	47.4	56.8	2.9	99.6	103.5	0.6

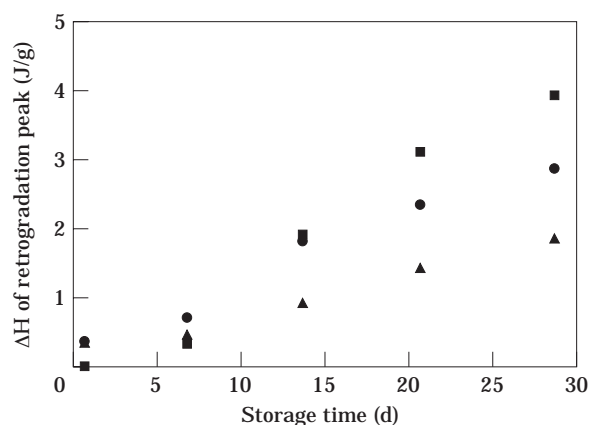
ND=not detectable.

concentration and was independent of the kind of sugar, for concentrations below 444.44 g/kg (sugar/(sugar + water)). However, when the concentration was higher than 444.44 g/kg, water molecules in fructose-water solution were more mobile than those in sucrose-water solution. The sugar solution used for preparing taro paste was only 377.36 g/kg, but the components of taro paste would further absorb water during the heating process, thus greatly increasing the concentration of sugar in the system. Consequently, the interaction of sucrose with water reduced water mobility.

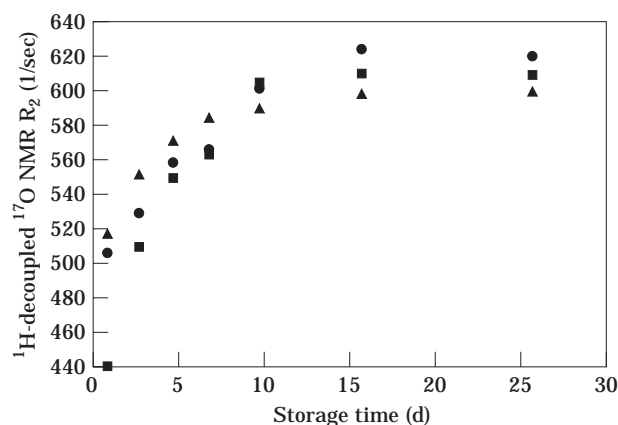
#### Effects of emulsifiers

The influence of emulsifier (10 g/kg MG SSL) on the hardness of low sucrose content taro paste under refrigeration storage is illustrated in Fig. 7. Taro pastes

with emulsifiers showed a significant retarding effect on hardness development during 30 d storage. SSL had a greater effect on decreasing the hardness of taro paste than did MG. From the DSC characteristics, two endothermic peaks were observed for the samples with emulsifier added, but only one peak was detected for the samples without emulsifier (Table 1). The first peak occurred at 46–63 °C and corresponded to the retrogradation of amylopectin; the second peak occurred at 93–105 °C and corresponded to the formation of amylose-emulsifier complex (2). Taro paste with added emulsifier showed a higher enthalpy change due to retrogradation during the first 7 d of storage, but the rate of retrogradation was much slower than that for the sample without added emulsifier (Fig. 8). These results also showed that more amylose-SSL than amylose-MG complexes were formed in the samples. The amount of amylose-emulsifier complex rose



**Fig. 8** Effect of emulsifiers on the change in the retrogradation peak for low sugar content taro pastes during refrigeration storage.  $322 \times 2$  (■) = without emulsifier; (▲) = 10 g/kg SSL; (●) = 10 g/kg MG



**Fig. 9** Effect of additives on the change in  $^{17}\text{O}$  NMR  $R_2$  for low sugar content taro pastes during refrigeration storage. (■) = without emulsifier; (▲) = 10 g/kg SSL; (●) = 10 g/kg MG

slightly with increased storage time. This finding suggested that amylose molecules were gradually leached-out or solubilized from damaged cells during storage. The effect of emulsifier on water mobility in the taro paste during storage was also examined by  $^1\text{H}$ -decoupled  $^{17}\text{O}$  NMR  $R_2$  measurements. In contrast to the taro paste made with sugar, the water mobility in taro paste with added emulsifier did not change significantly (Fig. 9). This indicated that the mechanisms involved in the loss of quality during storage were different for emulsifier-taro and sugar-taro pastes.

Since the emulsifier would have formed a complex with the hydrophobic moiety of the starch fraction, the interactions among water, sucrose and starch were not significantly affected. Thus, the change in water mobility for taro paste made with emulsifier was similar to that for paste made without emulsifier. The slight decrease in water mobility for the taro paste with emulsifier during the first 10 d storage could be due to an increase in viscosity of the taro paste (28).

## Conclusion

Changes in taro paste properties during storage appear to be related to the rate of retrogradation of free starch granules released by the heating and kneading processes. Sucrose at high concentration may interact with starch molecules or may restrict the swelling of starch granules, and result in a retardation of starch retrogradation during refrigeration. From the DSC measurements, we conclude that starch retrogradation may be one of the major factors affecting the hardness of taro paste during storage. The mobility of water in taro paste, measured by the  $^{17}\text{O}$  NMR  $R_2$ , can be used to demonstrate the interactions among water, solutes, emulsifiers and starches in a food system.

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