Effect of Sucrose Fatty Acid Esters with Different Hydrophilic-lipophilic Balance Values on Pasting and Rheological Properties of Waxy Rice Flour

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Abstract Effects of sucrose fatty acid esters (SEs) with hydrophilic-lipophilic balance (HLB) values of 5 (S-570), 9 (S-970), and 15 (S-1570) on the pasting and rheological properties of waxy rice flour (WRF) were investigated. Rapid Visco Analyzer (RVA) showed that addition of SEs affected the pasting properties of WRF. Rheological experiment includes the steady shear flow characteristics, and dynamic viscoelastic properties were also determined using a controlled-stress rheometer. The steady shear tests demonstrated that the viscosity data fitted well with the power law model ($R^2 \geq 0.976$) and all WRF pastes exhibited typical pseudoplastic and shear-thinning properties. Dynamic rheological measurements revealed that the addition of S-970 and S-1570 significantly increased the values of G' and G'' of samples, whereas the addition of S-570 decreased these values. The addition of S-1570 reduced the value of $\tan \delta$, whereas S-570 and S-970 increased it.

Keywords: waxy rice flour, sucrose fatty acid esters, pasting, rheological properties, power law model

Introduction

Waxy rice is one of the main food grains that people often consume in Southeast and East Asia. Compared with normal rice, it contains very low amount of amylose, which contributes to its sticky and moist texture when cooked (1). Because of its special chemical composition and physical properties, waxy rice is regarded as one of the most important component of making traditional Chinese foods, such as rice cakes, gnocchi, and rice dumpling (2). However, during the storage of these waxy rice starch-based foods, the retrogradation of starch leads to an increase in hardness and roughness in product texture and makes it easy to be attacked by enzymes (3). Additionally, in common with other cereal flours, it also has negative aspects like tendency to syneresis and exhibition of breakdown, producing weak-bodied rubbery pastes and undesirable gels (4).

The additions of various emulsifiers and hydrocolloids to starch-based foods offer a good way to overcome these shortcomings (4–6). Over the last decades, many studies have already demonstrated that emulsifiers such as diacetyl tartaric acid esters of monoglyceride (7), glycerol monostearate (8), and sucrose esters (9) have significant impact on the rheological and pasting properties of starch and improve the quality of overall product during processing and storage. Different type of interactions between additives and starch molecule

and granules are believed to be responsible for these physical property changes. Compared with other starches, less effort has been devoted to investigate the effects of different emulsifiers or hydrocolloids on rice flour or starch (10,11). Samutsri and Supphantharika (12) studied the pasting and rheological properties of rice starch in the presence of guar gum and xanthan with salts. The effects of locust bean gum on the rheological properties of five varieties of rice with different amylose contents have also been studied (13). Very recently, Chen *et al.* (14,15) systematically studied the effects of pullulan on the physical and chemical properties of rice flour and its possible interaction models.

Sucrose fatty acid esters (SEs), commonly called sugar esters, are non-ionic emulsifiers that are often used in food, pharmaceutical, and cosmetic industries (16). They have the advantages of colorlessness, having non-off flavor, being biodegradable, and having excellent compatibility. Moreover, SEs can provide a wide range of hydrophilic-lipophilic balance (HLB) values by changing the esterification degree of hydroxyl groups in the sucrose molecule. Commercial SEs with HLB values ranging from 1 to 16 are mixtures with various esterification degrees and positional isomers of the sucrose stearic acid ester (16). The addition of SEs in dough formulations can retard amylopectin recrystallization (17) and produce bread with a fine crumb structure, extended shelf life, and improved freeze-thaw stability (18).

Although many studies have investigated the applications of different SEs in food industry, there are few studies that focus on the effect of SEs on the physical and chemical properties of starch. Addo *et al.* (19) found that the alveograph dough rheological properties of wheat flour improved as HLB value of SE increased. Our previous study (20) investigated the effect of SEs using different HLB values on the pasting, rheological properties, and freeze-thaw stability of rice flour. The result showed that SEs have a significant effect on the gelatinization and rheological properties of normal rice flour. It is reported that the amylose in rice flour mainly affects the texture and stickiness, whereas gelatinization temperature and pasting properties are mainly influenced by the amylopectin (13,21). Waxy rice flour (WRF) can be a good model for studying the influence of different additives on amylopectin (22,23). Therefore, despite its real applications, there is significant fundamental interest in understanding how SEs with different HLB values affect the rheological and pasting behavior of WRF whose composition consists of amylopectin, with almost no amylose.

The objective of this work was to investigate the effect of different SEs on the properties of WRF. Three SEs with HLB values varying from 5 to 15 were studied. The pasting and rheological properties were determined and discussed. The influences of SEs with different HLB values on WRF were compared with that of normal rice flour, which was previously studied. The results have theoretical and practical implications in choosing suitable SEs with different HLB for different requirements in normal and WRF products.

Materials and Methods

Materials WRF purchased from Brothers Foodstuff Technology Co., Ltd (Bengbu, China). Proximate composition of WRF was determined using the standard method (3). Results show that the WRF contains approximately 79.6% crude starch, 8.4% protein, and 9.8% moisture. All SEs used in this study were kindly supplied by Mitsubishi-Kagaku Foods Corporation (Tokyo, Japan). The “S” series is short for the sucrose stearate. Details for different SEs used in this experiment are shown in the Table 1 (20). Each sample was prepared using ultra-pure water with a resistivity of 18.2 MΩ·cm.

Preparation of WRF-SE suspensions Different amount of SEs (0.3, 0.5, or 0.7% in mass fraction on the basis of 3 g WRF) were suspended in 25-g ultra-pure water at room temperature, followed by heating under stirring to completely dissolved. Then, 3 g of WRF was weighed into Rapid Visco Analyzer (RVA) special aluminum canisters, and the SE aqueous solution was added. The mixture was thoroughly stirred by magnetic stirring apparatus at room temperature. After completely mixing, samples were sonicated for 5 min to remove air bubbles and disperse SEs in suspensions. For comparison, control sample was prepared using the same methods, but no SEs were added.

RVA measurements The pasting properties of WRF and WRF-SEs mixtures were determined by RVA (Model RVA4; Newport Scientific Pty. Ltd., Warriewood, Australia). The slurries were manually stirred using plastic paddles for 10 s before inserting into the RVA instrument. The heating and cooling program is conducted according to the general pasting methods (20). The suspension was first equilibrated at 50°C for 1 min, then heated to 95°C within 3 min 45 s, and held at 95°C for 2 min 30 s. It was subsequently decreased to 50°C in 3 min 45 s at a constant rate and then held at 50°C for 1 min 30 s, while the revolution speed of the plastic paddle was set at 960 rpm in the first 10 s to disperse the sample and then maintained at 160 rpm during the following measurement.

Some pasting properties of WRF including the pasting temperature (PT), peak viscosity (PV), trough viscosity (TV), breakdown value (BDV), final viscosity (FV), and setback value (SBV) were obtained from the RVA curves. Each sample was tested in triplicate, and the results were presented as means±standard deviation. At the end of RVA measurement, the sample gels were cooled to 25°C spontaneously and then immediately transferred to the rheometer plate for determining the rheological properties (24).

Rheological measurements The rheological properties of WRF-SE pastes were obtained using a controlled-stress rheometer (AR-G2; TA Instruments, New Castle, DE, USA) equipped with the parallel plate system. All the rheological properties were determined using a 40-mm-diameter parallel plate at 25°C, and the determination gap was set at 1 mm. The samples were placed on the rheometer plate and equilibrated for 2 min before measurements. A thin layer of low-viscosity silicon oil was smeared around the edge of the sample to prevent the evaporation of moisture during measurement. For each sample, steady flow and dynamic viscoelastic properties were determined. All rheological determinations were performed in triplicates for each sample.

Steady flow measurements were performed to obtain shear rate versus shear stress (flow curves) data (25). The cone was programmed to increase the shear rate from 0 to 100 s⁻¹ (upward curve) after equilibrated 1-min decrease from 100 to 0 s⁻¹ (downward curve). Data obtained from the flow curves of the shear cycle were used to characterize the flow properties of the mushy samples. The steady shear rheological properties of gels were analyzed using the well-known power law model as

$$\sigma = K \times \dot{\gamma}^n \quad (1)$$

where σ is shear stress (Pa), $\dot{\gamma}$ is shear rate (s⁻¹), K is the consistency coefficient (Pa·s ^{n}), and n is flow behavior index (dimensionless). Experiment data with shear rate ranged from 0.1 to 100 s⁻¹ are selected for analysis.

The oscillatory tests were performed over a frequency range of 0.1–10 Hz (0.628–62.8 rad/s) at a constant strain 1%, which was in the linear viscoelastic region for all samples. The storage modulus (G'), loss modulus (G''), and mechanical loss tangent ($\tan \delta$) as a function

of angular frequency (ω) were obtained in this measurement using TA rheometer Data Analysis software (version 5.7.1).

FTIR spectroscopy Measurement of SEs in the dry state were conducted using a FTIR spectrometer (model Nicolet™ 380; Thermo Electron Corporation, Waltham, MA, USA) in the range of 4000–400 cm^{-1} at room temperature. Sieved powder sample of 2 mg and potassium bromide of 200 mg were taken into an agate mortar and grounded to completely mix them together. The obtained spectra were analyzed by EZ OMNIC Spectra Software.

Statistical analysis All the experiment data were reported as mean value $\pm$ standard deviations. The results were analyzed by analysis of variance and Duncan's multiple range tests ($p < 0.05$) to determine significant differences between samples of different components. All calculations and analyses were performed with SPSS version 17.0 for Windows.

Results and Discussion

FTIR spectra for different SEs Details of different SEs used in this experiment are shown in the Table 1. The determinations of infrared spectrum were conducted to obtain more information on the structures of different SEs. Figure 1 shows the FTIR spectra for three kinds of SEs (S-570, S-970, and S-1570).

As can be seen from Fig. 1, the infrared spectra of three types of SEs are very similar to each other; wavelengths of 2921.8 and 2854.3 cm^{-1} infer the C-H asymmetric and symmetric stretching vibration, respectively. The bands near 1730 cm^{-1} (1737.6 cm^{-1} for S-570, 1735.7 cm^{-1} for S-970, and 1733.8 cm^{-1} for S-1570) are attributed to the saturated catenulate C=O stretching vibration of esters. It can be seen that compared with the other two sucrose esters, S-570 has the maximum peak intensity near 1737.6 cm^{-1} , which indicates that S-570 has a larger number of polyesters. These results are further confirmed by the fact that the intensity of wavelength at 721 cm^{-1} decreases in the following order: S-570 > S-970 > S-1570. It is known that the wavelength between 720 and 725 cm^{-1} infers the wag vibration of $-(\text{CH}_2)_n-$ group when $n > 4$. This polymethylene group only exists in the side chain of sucrose ester of stearic acid. This indicates that the S-570 mixtures have more hydrophobic side chains in the molecule and can give a lower HLB value.

Pasting properties The pasting curves of WRF alone and WRF-SEs

Fig. 1. The IR spectra for three types of SEs (S-570, S-970, and S-1570).

mixtures at various SE concentrations determined by RVA are depicted in Fig. 2. It is well known that when the heating-up temperature is less than the gelatinization temperature, waxy rice starch granules only shows limited swelling, and the starch paste has a lower viscosity at that moment. With the increase of temperature and heating time, amylopectin granules swelled. As shown in Fig. 2, these changes result in a rapid increase in viscosity of the starch paste. Starch paste has the maximum viscosity value when granules are swollen to a maximum extent. Thereafter, the further extension of amylopectin chains in WRF causes the disintegration of starch granules and leads to decrease in mixture viscosity. Finally, with the decrease of heating temperature, the viscosity of WRF paste takes on an evident downward trend.

The extraction values of pasting parameters for WRF as affected by various SEs are shown in Table 2. The results show that all SEs used affected the pasting properties of WRF, although the influence degree was highly dependent on the kinds of SEs. It can be found that for the same kinds of additive, PV and TV increase with the increase of SE concentrations. All SEs used improved PV and TV of WRF with varying degrees and moved up the pasting curve along the +Y-axis. Compared with the control sample, the effects of S-970 and S-1570 on the pasting curve are most significant. As can be seen from Fig. 2A, the influence of different SEs on the pasting properties of WRF becomes more and more significant with the increase of SE concentrations. In addition, similar phenomena are observed for the effects of S-1570 on the pasting curve of WRF as shown in Fig. 2C. It is indicated that after the addition of 1-mono-cis-13-docosenoyl glycerol, the viscosity of starch pastes slightly increases in the RVA profile (24). Therefore, we have a reason to speculate that the interactions

Table 1. Details of different SEs used in this experiment

SEs type	HLB	Ester composition (%)		Melting temperature	
		Monoester	Polyester	Start point (°C)	Peak point (°C)
S-570	5	30	70	50	57-65
S-970	9	50	50	49	56
S-1570	15	70	30	49	55

Fig. 2. RVA pasting profiles of WRF with and without different concentrations of S-570 (A), S-970 (B), and S-1570 (C)

between starch granules and emulsifier molecules can alter the swelling power of granules during the pasting procedure.

Figure 3 shows the pasting curves of WRF-SE mixtures with different kinds of SEs at the same concentration, i.e., 0.7%. It is obviously the addition of SEs that increased PV of WRF. As indicated by previous studies (2), the addition of thickening additives may enhance the forces that are exerted on the granules under shear condition and increase PV of starches. On the other hand, the influence of different SEs on the pasting properties descends in the following order: S-970>S-1570>S-570. It is interesting to find that S-970 affects the pasting properties of WRF most significantly. This may

be attributed to the fact that S-970 has an optimum content of monoesters to polyesters. It was also observed that at the highest concentration (0.7%), S-570 has the lowest effects on FV and SBV of WRF. This is probably due to the fact that S-570 has more side chains in the molecular structure and could interact with more starch granules at high concentrations. Chen *et al.* (14) also found that the addition of pullulan to normal rice starch decreased FV, SBV, and BV at a higher concentration of additive. These behaviors indicate that SEs containing both monoester and polyester may have some synergistic interactions including the effects on continuous phase for the formation of gel network and the intertwinement between SEs

Table 2. Effects of SEs at different concentrations on the pasting properties of waxy rice flour

Sample	SE%	PV	TV	Breakdown	FV	Setback	PT (°C)
control	0	2496.3±2.1 ^{e1)}	1259.0±3.6 ^g	1237.3±3.1 ^b	1645.3±3.1 ^e	386.3±3.2 ^a	70.5±0.5 ^{a,b}
S-570	0.3	2496.3±15.0 ^e	1271.7±6.5 ^g	1224.7±13.3 ^{b,c}	1641.0±6.1 ^e	369.3±9.5 ^{a,b}	69.4±1.2 ^b
	0.5	2515.7±15.9 ^e	1271.3±6.4 ^g	1244.3±19.4 ^b	1637.7±8.5 ^e	366.3±3.2 ^{a,b}	69.4±0.5 ^b
	0.7	2684.3±12.7 ^b	1347.0±26.9 ^e	1337.3±15.3 ^a	1670.0±8.2 ^d	323.0±21.9 ^{d,e}	69.4±0.5 ^b
S-970	0.3	2548.0±17.1 ^d	1308.0±16.1 ^f	1240.0±14.4 ^b	1658.7±11.6 ^{d,e}	350.7±8.1 ^{b,c}	70.2±0.9 ^{a,b}
	0.5	2776.3±12.2 ^a	1542.3±8.1 ^b	1234.0±15.9 ^b	1695.0±9.9 ^c	152.7±5.9 ^g	70.7±0.1 ^a
	0.7	2797.0±20.7 ^a	1718.3±18.0 ^a	1078.7±38.6 ^f	1826.7±14.6 ^a	108.3±6.1 ^h	70.5±0.5 ^{a,b}
S-1570	0.3	2540.7±17.2 ^d	1347.0±9.0 ^e	1193.7±13.4 ^{c,d}	1682.0±8.2 ^{c,d}	335.0±2.0 ^{c,d}	70.2±0.4 ^{a,b}
	0.5	2554.3±14.9 ^d	1392.7±6.4 ^d	1161.7±8.6 ^{d,e}	1705.0±24.3 ^c	312.3±23.4 ^e	70.5±0.4 ^{a,b}
	0.7	2654.3±10.1 ^c	1499.0±18.3 ^c	1155.3±27.0 ^e	1789.0±21.7 ^b	289.0±5.3 ^f	70.5±0.5 ^{a,b}

¹⁾Values with different superscript letters within the same column are significantly different at $p<0.05$.

Fig. 3. Pasting profiles of WRF in the presence or absence of different SEs at the same concentration (0.7%)

molecules and starch granules (26).

It was found that with the addition of the same HLB values of SEs, PV in RVA curve increased more significantly for normal rice flour, whereas the addition of S-970 and S-570 decreased TV (20). In addition, S-1570 and S-970 both enhanced FV, SBV, and PT, whereas S-570 induced an evident reduction of FV and PT of normal rice flour. By comparing the results obtained above for WRF, S-970 with the same concentration has the greatest impact on pasting properties, namely, BDV, FV, and SBV, which are inconsistent with that for normal rice flour. The reason could be attributed to the fact that rice starch has a large amount of amylose, whereas waxy rice mainly consists amylopectin and no amylose.

It is known that the pasting and rheological properties of starch are influenced by many factors, including the distribution of amylopectin branch chain length and amylose content and lipid content. In this study, the WRF paste after gelatinization can be seen as a mixture of continuous phase, i.e., mostly low molecular weight amylopectin in aqueous solution, and dispersed phase comprising swollen amylopectin granules. The properties of starch paste depend on the nature and interactions of these two phases with different SEs. During the procedure of continuous heating, small molecular weight amylopectin will leach out from the starch granules (27). PV increasing with the addition of SEs can be attributed to the formation of complexes between the emulsifier and leached amylopectin. These complexes can cover the surface of starch granules, hinder the exudation of amylopectin, and delay the swelling and disintegration process of starch granules (28), which also leads to the decrease of breakdown viscosity. On the other hand, because of the hydrophilic properties of SEs, they could form hydrogen bonding network with water molecules in the continuous phase. It is reasonable that S-970 and S-1570 with more hydrophilic hydroxyl groups could form more extensive hydrogen bonding network with water molecules than around starch granules. Therefore, the chemical properties of SEs to form hydrogen bonding network may become a significant factor in case of WRF. For normal rice starch, it was stated that the interactions between monostearate and helical amylose molecule are the main reason that affect the pasting properties (20).

Fig. 4. Flow curves of WRF paste with and without SEs

Steady shear properties The typical flow curves of WRF gel with and without different SEs are presented in Fig. 4. The flow curves of all the samples exhibited clockwise hysteresis loop with variation in shear rate, which suggested that shear stress obtained from upward curve is larger than that of downward curve, and demonstrated thixotropic behavior of the starch gel. This may attribute to the fact that the original molecular structure of gel was damaged with the increase in shear rate, which resulted in the reduction of fluid apparent viscosity (29). When the shear stress is removed, starch gel is restored to the original apparent viscosity, and restoration of molecular structure will take some time. In addition, our previous studies (20) indicated that the thixotropic loop area of rice flour paste with the addition of SEs is smaller than that of control group, which illustrates that SEs can weaken the thixotropy of rice paste effectively.

The power law equation is used to describe flow behavior parameters of upward and downward flow curves. Consistency coefficient (K), flow behavior index (n), and apparent viscosity ($\eta_{a,100}$) at a shear rate of 100 s^{-1} for each paste are presented in Table 3. All experiment data fit well with the power law model with R^2 between 0.976 and 0.996. It can be seen that all samples exhibited typical pseudoplastic and shear-thinning properties because the flow behavior index (n) is in the range of 0 to 1. Generally speaking, most pseudoplastic fluid contains ramose polymer particles, such as swollen amylopectin granules. Flow behavior index (n) can characterize the rigidity of granules in fluid: the smaller the value is, the greater the flexibility of the molecule. The addition of SEs enhances the n value of upward flow curves and the amplitude of effect gradually reduces with the increase of HLB value.

The addition of SEs increases the value of the consistency coefficient in downward flow curves. Influence of SEs on the apparent viscosity ($\eta_{a,100}$) of upward and downward curves is consistent with each other. Compared with the control sample, addition of SEs increases the apparent viscosity ($\eta_{a,100}$) of all the samples, and the larger the HLB value is, the greater the apparent viscosity ($\eta_{a,100}$). The apparent viscosity of the starch gel decreases with increasing shear rate, which indicates that the fluid exhibits shear thinning characteristics. The reason should be ascribed to the fact that the starch granules are

Table 3. Influence of SEs on the steady shear rheological properties of waxy rice flour

Sample	Up curve			Down curve		
	$\eta_{a,100}$ (Pa·s)	K (Pa·s ⁿ)	n (-)	$\eta_{a,100}$ (Pa·s)	K (Pa·s ⁿ)	n (-)
control	1.400±0.278 ^{b1)}	19.643±0.539 ^b	0.419±0.019 ^c	1.363±0.135 ^b	13.440±1.529 ^b	0.496±0.003 ^a
S-570	1.486±0.053 ^{a,b}	11.863±1.126 ^c	0.551±0.015 ^a	1.452±0.049 ^{a,b}	13.877±0.413 ^b	0.503±0.003 ^a
S-970	1.677±0.004 ^a	18.163±0.397 ^b	0.482±0.005 ^b	1.631±0.004 ^{a,b}	16.173±0.180 ^{a,b}	0.495±0.004 ^a
S-1570	1.751±0.005 ^a	24.380±0.559 ^a	0.421±0.006 ^{b,c}	1.697±0.008 ^a	18.046±0.347 ^a	0.478±0.004 ^b

¹⁾Values with different superscript letters within the same column are significantly different at $p < 0.05$.

Table 4. Storage (G') and loss (G'') moduli, and $\tan\delta$ at 1 Hz for waxy rice flour with different SEs

Sample	G' (Pa)	G'' (Pa)	$\tan\delta$
Control	13.99±0.17 ^{c1)}	5.99±0.13 ^b	0.428±0.004 ^b
S-570	9.04±0.33 ^d	4.87±0.09 ^c	0.539±0.006 ^a
S-970	15.08±0.62 ^b	6.61±0.12 ^a	0.439±0.009 ^b
S-1570	18.96±0.20 ^a	6.41±0.11 ^a	0.338±0.009 ^c

¹⁾Values with different superscript letters within the same column are significantly different at $p < 0.05$.

intertwined with each other so tightly at rest or low shear and the system exhibits larger viscosity. However, when the flow speed or shear rate increases, the polymer particles that were originally distributed dispersedly rotate and shrink into lumps, and these changes reduce the entanglement between granules and thus form the fluids showing the shear-thinning properties (30,31).

Dynamic viscoelastic properties Frequency sweep is the measure of the variation of sample viscoelastic behavior with the change of frequency under constant stress or strain. It's known that storage modulus (G') and loss modulus (G'') represent the elastic properties of energy storage and the viscous properties of energy dissipation, respectively. The G' , G'' , and $\tan\delta$ values at an oscillatory frequency of 1 Hz for fresh WRF gels with and without SEs at 25°C are shown in Table 4. The increase in angular frequency increases the G' and G'' over the whole frequency range. Furthermore, the addition of S-970 and S-1570 significantly increases the value of G' and G'' compared with the control sample, whereas S-570 decreases that for paste. Slightly different from our earlier findings (20), the current results show that S-970 maximally increased the G'' of WRF, which is inconsistent with the influence of SEs on pasting properties. Meanwhile, a crossover between storage modulus and loss modulus was not observed at all in the process of dynamic oscillatory shearing, which demonstrated that the paste shows typical weak gel properties (31). As can be seen from Table 4, G' is always greater than G'' , which indicates that the elastic properties of flour gel are much stronger than viscous properties. Moreover, the addition of SEs has a greater influence on G' compared with G'' , which shows that the effect of SEs on the elastic properties of WRF gel is greater than that on viscous properties. The value of $\tan\delta$ reflects the relative intensity between elastic properties and viscous properties of the gel. With $\tan\delta$, we can judge whether the material behaves like a solid or liquid. As

shown in Table 4, S-1570 reduced the value of $\tan\delta$, whereas S-570 and S-970 increased the $\tan\delta$ value with varying degree.

It was pointed out that the overall dynamic viscoelastic properties of starch gels were determined by the density of crosslinks in the continuous phase, the entanglements between the amylopectin moieties, and effective contacts between the dispersed phases (32). The addition of S-1570 increases the G' value of starch gel greatly. The largest increase in G' by S-1570 indicates that the junction zones of starch gel can be strengthened by the interaction of monostearate in the continuous phase to form a stronger crosslinking network. S-570 has high content of hydrophobic polyester and can form a larger steric hindrance, which will weaken the gel network structure and reduced the viscoelastic of starch gel (25).

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