

Rheological Characteristics of Waxy Rice Starch Modified by Carboxymethyl Cellulose

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ABSTRACT: The effects of carboxymethyl cellulose (CMC) at different concentrations (0, 0.2, 0.4, and 0.6% w/w) on the rheological properties of waxy rice starch (WRS) pastes were evaluated under both steady and dynamic shear conditions. The flow properties of WRS-CMC mixtures were determined from the rheological parameters of power law and Casson models. All samples demonstrated a clear trend of shear-thinning behavior ($n=0.33\sim 0.34$), with a marginal difference shown between n values. The addition of CMC to WRS increased the apparent viscosity ($\eta_{a,100}$), consistency index, and Casson yield stress values. The dynamic moduli [storage modulus (G'), loss modulus (G''), and dynamic viscosity (η^*)] and ratio of G''/G' values of WRS-CMC mixtures also increased with an increase in CMC concentration; the higher dynamic rheological properties observed at higher CMC concentrations may be attributed to an increase in the viscoelasticity of the continuous phase in the starch-gum mixture system. Dependence of $\eta_{a,100}$ on temperature followed the Arrhenius model for all samples. The Cox-Merz rule was not applicable to WRS-CMC pastes with different CMC concentrations, demonstrating that there was a deviation between η^* and steady shear viscosities for all samples. Therefore, the synergistic effect of CMC on the rheological properties of WRS pastes appeared to be the result of coacervation.

Keywords: CLSM, waxy rice starch, carboxymethyl cellulose, synergistic effect, coacervation

INTRODUCTION

Waxy rice starch (WRS) is widely used in food products, either as a raw material or as a food additive. Similar to other starches, WRS has disadvantages, such as retrogradation, either from extended cooking, high shear, or acidic conditions, and may produce weak-bodied, cohesive, rubbery pastes, and undesirable gels (Shi and BeMiller, 2002). However, the addition of proper non-starch polysaccharides (gums) can overcome these limitations of starches (Kim et al., 2009). In general, starches modified by mixing with gums have improved rheological and textural properties. Previous studies have modified the rheological properties of WRS by adding small amounts of gums, such as guar gum, locust bean gum, and xanthan gum, which are widely used by the food industry as a favorable thickening agent in food systems (Kim et al., 2009; Kulicke et al., 1996). However, no comprehensive information is available about the effect of carboxymethyl cellulose (CMC) on the steady and dynamic rheological properties of WRS. CMC, which is a water-soluble heteropolysaccharides with a high molecular weight, is often

used together with starches to provide a desirable texture, control water mobility, and improve overall product stability by forming macromolecular structures through inter- and intra-molecular hydrogen bonds (Sun et al., 2017). Therefore, the main objective of this study was to investigate the rheological properties of WRS-CMC pastes supplemented with different concentrations of CMC under both steady and dynamic conditions. An understanding of phase separation and rheological properties of WRS-CMC mixtures could lead to improvements in the formulations of WRS-based products for further applications in product development.

MATERIALS AND METHODS

Materials and preparation of WRS-CMC mixtures

Commercial WRS was provided by the SMS Corporation (Pathum Thani, Thailand). The proximate composition of WRS was: 13.6% moisture, 0.7% protein ($N\times 6.25$), 0.8% fat, 0.2% ash, 84.7% carbohydrate (by difference), and the amylose content was 6.30%. CMC was purchased

Received 2 August 2019; Accepted 21 October 2019; Published online 31 December 2019

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from Shanghai Shengguang Edible Chemicals Co., Ltd. (Shanghai, China). WRS-CMC mixtures (5% w/w) for rheological measurements were prepared by dispersing WRS with distilled water and CMC to obtain 0%, 0.2%, 0.4%, and 0.6% (weight basis) CMC; 0% CMC (100% WRS) was used as the control. The dispersion was moderately stirred for 60 min at room temperature and heated at 95°C for 30 min with mild agitation using a magnetic stirrer (HS15-26P, Misung Scientific Co., Ltd., Daejeon, Korea). The hot paste samples were immediately transferred to a rheometer plate at 25°C to measure the rheological properties.

Rheological analysis

The steady and dynamic shear rheological properties of the WRS-CMC mixtures were investigated using a Haake RheoStress 1 rheometer (Haake GmbH, Karlsruhe, Germany) and controlled stress rheometer (AR 1000, TA Instruments, New Castle, DE, USA), respectively. Plate-plate geometries with diameters of 35 mm (RheoStress 1) and 40 mm (AR1000) were used. Each sample was loaded between the parallel plates at 25°C and compressed to obtain a gap of 500 μm . After 5 min of equilibration to reach the measurement temperature of 25°C, rheological outcomes were measured.

The shear stress (σ , Pa) versus shear rate ($\dot{\gamma}$, s^{-1}) dependence was determined at $\dot{\gamma}$ values in the range of 0.4 \sim 500 s^{-1} . Further, to evaluate the time-dependent flow behavior, the samples were sheared first in ascending order and then in descending order. The up-flow curves were described by the power law model (Eq. 1) and the Casson model (Eq. 2):

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

$$\sigma^{0.5} = K_{oc} + K_c\dot{\gamma}^{0.5} \quad (2)$$

where K is the consistency index ($\text{Pa}\cdot\text{s}^n$), n is the flow behavior index (dimensionless), and K_c is the Casson plastic viscosity. The Casson yield stress (σ_{oc}) according to the Casson model [Eq. (2)] was determined as the square of the intercept (K_{oc}) that was obtained from linear regression of the square roots of the $\dot{\gamma}$ - σ data. By using the magnitudes of K and n , we calculated the apparent viscosity ($\eta_{a,100}$) at 100 s^{-1} . The temperature dependence was assessed by fitting the Arrhenius model (Eq. 3) to the experimental data using $\eta_{a,100}$ at different temperatures (25 \sim 70°C) for all samples:

$$\eta_{a,100} = A \cdot \exp(E_a/RT) \quad (3)$$

where A is a constant ($\text{mPa}\cdot\text{s}$), T is the absolute temper-

ature (K), R is the gas constant (8.3144 J/mol/K), and E_a is the activation energy (J/mol).

Dynamic rheological tests were conducted using small-amplitude oscillatory rheological measurements at 25°C. A dynamic oscillatory test was performed as a function of the angular frequency (ω) (0.63 \sim 62.8 $\text{rad}\cdot\text{s}^{-1}$) at 2% strain. The 2% strain was within the linear viscoelasticity limit. The TA rheometer data analysis software (version VI. 1.76, TA Instruments) was used to obtain the experimental data and calculate the storage (or elastic) modulus (G'), loss (or viscous) modulus (G''), complex viscosity (η^*), and loss tangent ($\tan \delta = G''/G'$). The $\tan \delta$ value allows the evaluation of viscoelastic behavior: $\tan \delta < 1$, predominantly elastic behavior; $\tan \delta > 1$, predominantly viscous behavior. All rheological measurements were performed in triplicate.

Correlations between the values of dynamic shear parameters (η^* and ω) and steady shear parameters (η_a and $\dot{\gamma}$) were estimated using the Cox-Merz rule (Eq. 4). To examine the applicability of the Cox-Merz rule, the η_a and η^* of all WRS-CMC mixtures were plotted versus $\dot{\gamma}$ and ω , respectively (Cox and Merz, 1958).

$$\eta^*(\omega) = \eta_a(\dot{\gamma}) \mid_{\omega=\dot{\gamma}} \quad (4)$$

Statistical analysis

All experiments were conducted in triplicate, with data are reported as the mean \pm standard deviation (SD). For multiple comparison analysis, analysis of variance (ANOVA) followed by Duncan's multiple range test was performed using the Statistical Analysis System program (version 9.2; SAS Institute, Cary, NC, USA). $P < 0.05$ was considered statistically significant.

RESULTS AND DISCUSSION

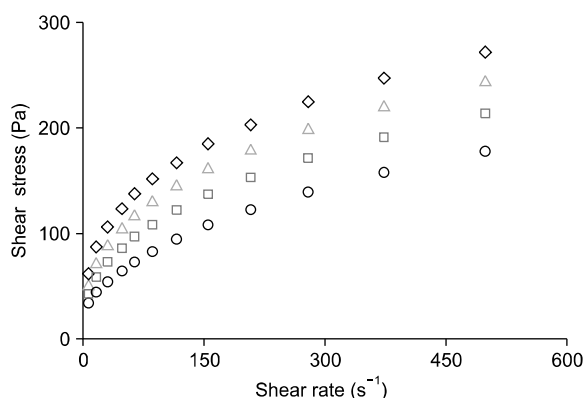
Steady shear properties

The results of σ versus $\dot{\gamma}$ of WRS-CMC mixtures containing different CMC concentrations (0.2% \sim 0.6%) fitted well to the power law (Eq. 1) and Casson (Eq. 2) models with high R^2 (0.98 \sim 0.99) values (Table 1). All samples had a non-Newtonian (pseudoplastic) nature and demonstrated shear-thinning behavior with flow behavior index (n) values as low as 0.33 \sim 0.34 (Fig. 1). This type of flow behavior is typical of starch-gum mixture systems (Kim et al., 2009; Lee et al., 2017; Kim and Yoo, 2011; Kim and Yoo, 2006; Yoo et al., 2005). There were no significant differences between the n values of the control (0% CMC) and WRS-CMC mixtures, indicating that the presence of CMC in the WRS-CMC mixtures had no effect on the n values. Similar results were reported in a previous study of WRS-guar gum mixtures (Lee et al.,

Table 1. Apparent viscosity ($\eta_{a,100}$), consistency index (K), flow behavior index (n), and Casson yield stress (σ_{oc}) of waxy rice starch-carboxymethyl cellulose (CMC) mixtures with different CMC concentrations

Concentration (%)	$\eta_{a,100}$ (Pa·s)	K (Pa·s ⁿ)	n	σ_{oc} (Pa)
0 (control)	0.84±0.05 ^d	17.7±0.84 ^d	0.34±0.01 ^a	23.7±0.71 ^d
0.2	1.14±0.03 ^c	25.4±2.11 ^c	0.33±0.02 ^a	31.5±0.65 ^c
0.4	1.35±0.03 ^b	28.5±1.36 ^b	0.34±0.01 ^a	38.9±0.59 ^b
0.6	1.57±0.01 ^a	34.7±1.50 ^a	0.33±0.01 ^a	46.9±0.50 ^a

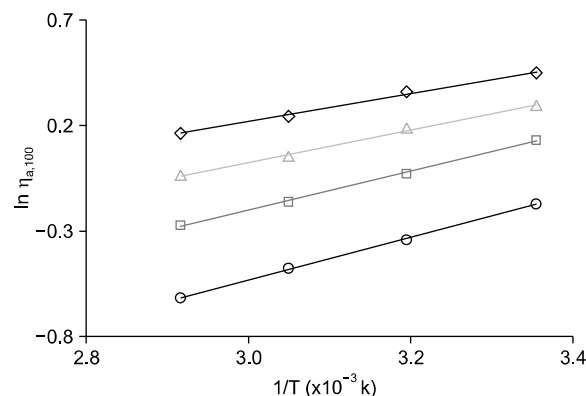
Mean values in the same column with different letters (a-d) are significantly different ($P < 0.05$).

**Fig. 1.** Plots of shear stress and viscosity versus shear rate for waxy rice starch-carboxymethyl cellulose (CMC) mixtures with different CMC concentrations at 25°C: (○) 0%, (□) 0.2%, (△) 0.4%, and (◇) 0.6%.

2017). The K, $\eta_{a,100}$, and σ_{oc} values of WRS-CMC mixtures were much higher than those of the control (without CMC) and increased with increased CMC concentration from 0 to 0.6% (Table 1), indicative of a higher synergism with CMC. These results demonstrate that WRS-CMC mixtures were high shear-thinning fluids with high magnitudes of K and σ_{oc} .

Temperature dependence (Arrhenius equation)

The temperature dependence of $\eta_{a,100}$ of the WRS-CMC mixtures was determined by fitting the data to the Arrhenius model (Eq. 3), where $\eta_{a,100}$ decreases to an exponential function with temperature. The Arrhenius temperature relationship has been confirmed experimentally in previous studies of starch-gum mixtures (Lee et al., 2017; Kim and Yoo, 2011; Kim and Yoo, 2006; Yoo et al., 2005). From a plot of $\ln \eta_{a,100}$ (ordinate) versus $(1/T)$ (abscissa), $E_a = (\text{slope} \times R)$, and A is the exponential of the intercept. E_a values were determined from regression analysis of $1/T$ versus $\ln \eta_{a,100}$ (Fig. 2), and were in the range of 5.71 ~ 8.70 kJ/mol with high determination coefficients ($R^2 = 0.96 \sim 0.98$) (Table 2). The E_a values (5.71 ~ 7.59 kJ/mol) of the WRS-CMC mixtures were much lower than the value (8.70 kJ/mol) of the control; further, E_a values decreased with increased CMC concentration, indicative of a lower effect of temperature on the rheological parameter at higher CMC concentrations. Similar patterns between E_a variation and concentration

**Fig. 2.** Arrhenius plots of $1/T$ (K) versus apparent viscosity ($\eta_{a,100}$) at 100^{-1} for waxy rice starch-carboxymethyl cellulose (CMC) mixtures with different CMC concentrations: (○) 0%, (□) 0.2%, (△) 0.4%, and (◇) 0.6%.**Table 2.** Activation energies (E_a) of wash rice starch-carboxymethyl cellulose (CMC) mixtures with different CMC concentrations

Concentration (%)	A (Pa·s)	E_a (kJ/mol)	R^2
0	0.03	8.70	0.98
0.2	0.05	7.95	0.98
0.4	0.09	6.67	0.96
0.6	0.16	5.71	0.97

have been previously observed for the rice starch-xanthan gum (Lee et al., 2017) and rice starch-tara gum mixtures (Kim and Yoo, 2011). Therefore, the E_a values of WRS-CMC mixtures in a temperature range of 25 ~ 70°C were significantly affected by the concentration of CMC.

Time-dependent flow behavior

Measurement of increasing and decreasing $\dot{\gamma}$ of WRS-CMC mixtures showed a hysteresis loop pattern in the range of 0.4 ~ 500 s^{-1} , indicating that all samples exhibited time-dependent shear-thinning (thixotropic) flow behavior (Fig. 3). The area enclosed by the hysteresis loop is known to indicate the degree of breakdown due to shearing (Weltmann, 1943). Thixotropic flow behavior was less pronounced in the presence of CMC based on the area of the loop between the up and down flow curves (Fig. 3), demonstrating that CMC addition enhanced re-association after structure breakdown accelerated by high $\dot{\gamma}$. The hysteresis loop area of WRS-CMC mixtures de-

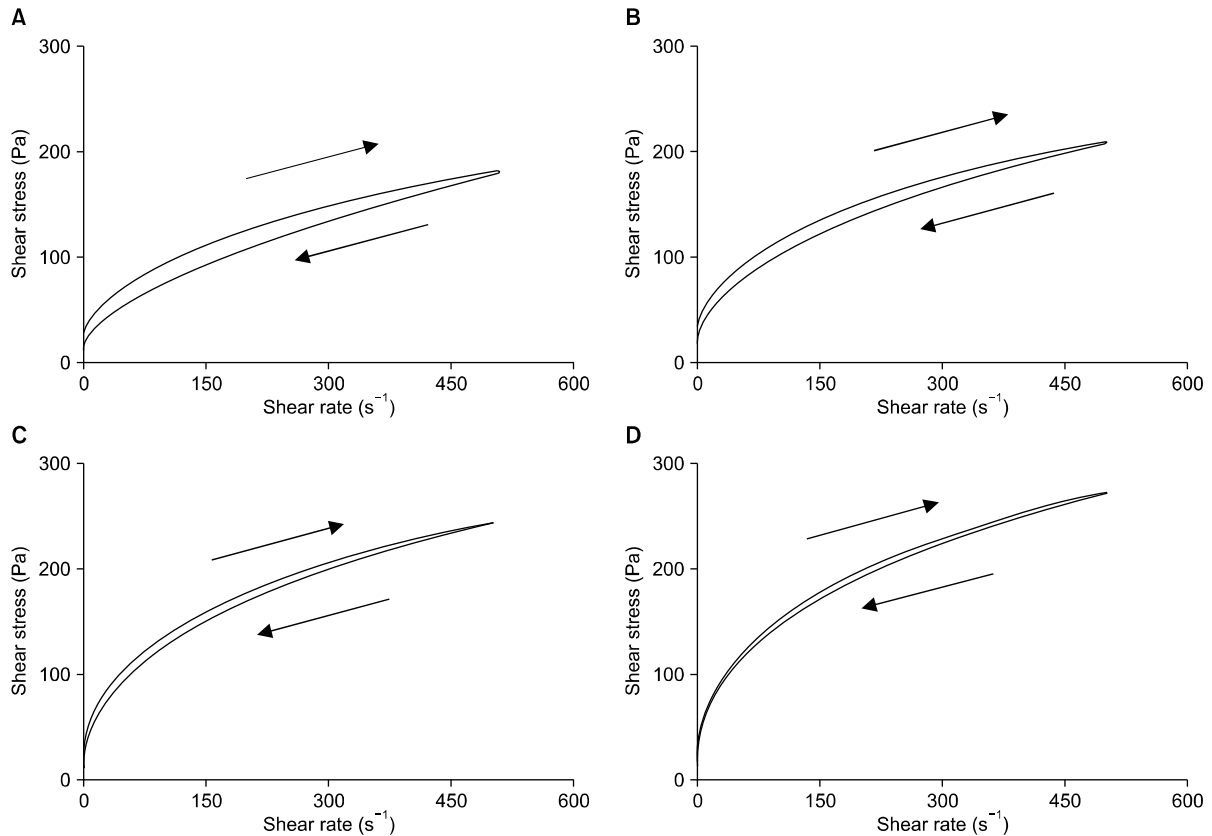


Fig. 3. Thixotropic flow curves of waxy rice starch-carboxymethyl cellulose (CMC) mixtures with different CMC concentrations at 25°C: (A) 0%, (B) 0.2%, (c) 0.4% and (d) 0.6%.

creased with increased concentration of CMC. Specifically, the loop area of WRS with 0.6% CMC was markedly decreased compared with that of WRS alone, suggesting that the addition of CMC at high concentrations enhanced the formation of the shear-induced network structure of the WRS-CMC mixtures and reduced the breakdown of the network at a high $\dot{\gamma}$ (Lee et al., 2017). This may be attributed to the synergistic effects between WRS and anionic CMC molecules in the formation of typical cross-linkages found in biopolymer gels (Michailova et al., 1999). Similar findings have also been reported for other starch-gum mixtures (Lee et al., 2017; Sikora et al.,

2008; Korus et al., 2004).

Dynamic rheological properties

Fig. 4 shows the changes in G' and G'' as a function of the ω for WRS-CMC mixtures at 25°C. The G' and G'' values were increased with increasing ω , and the G' was much higher than G'' depending on the frequency, demonstrating weak gel-like behavior of the WRS-CMC mixtures. This type of behavior has been interpreted as an entangled network among macromolecules (Dublier and Curvelier, 1996) and is in good agreement with that observed for other waxy starch pastes mixed with gums (Michailova

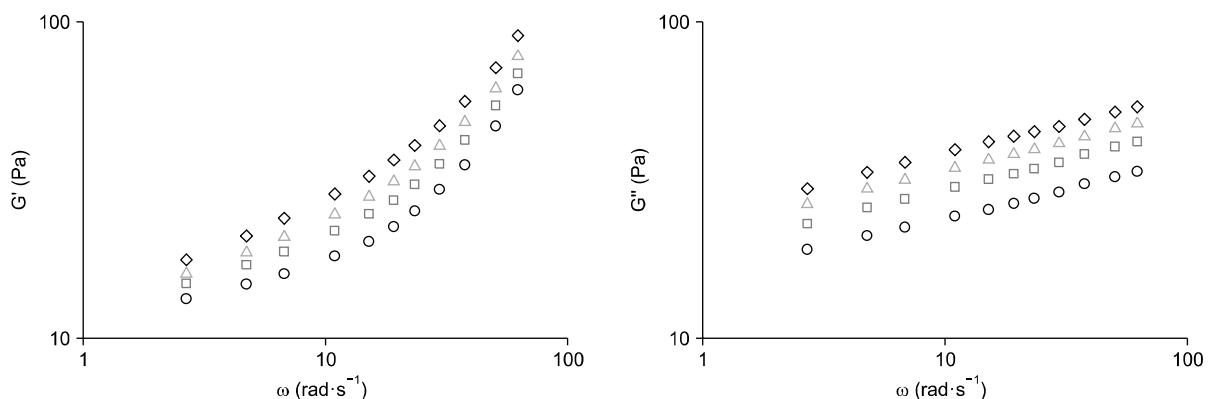


Fig. 4. Plots of modulus (G') and loss modulus (G'') versus ω for waxy rice starch-carboxymethyl cellulose (CMC) mixtures with different CMC concentrations at 25°C: (○) 0%, (□) 0.2%, (△) 0.4%, and (◇) 0.6%.

Table 3. Storage modulus (G'), loss modulus (G''), complex viscosity (η^*), and $\tan \delta$ values at $6.28 \text{ rad} \cdot \text{s}^{-1}$ for waxy rice starch-carboxymethyl cellulose (CMC) mixtures with different CMC concentrations

Concentration (%)	G' (Pa)	G'' (Pa)	η^* (Pa·s)	$\tan \delta$
0	9.29 ± 0.64^d	4.54 ± 0.26^d	1.65 ± 0.08^c	0.49 ± 0.06^d
0.2	14.4 ± 0.77^c	7.35 ± 0.12^c	2.57 ± 0.12^c	0.51 ± 0.02^c
0.4	17.0 ± 0.38^b	10.0 ± 0.16^b	3.14 ± 0.04^b	0.59 ± 0.02^b
0.6	19.9 ± 0.37^a	13.0 ± 0.33^a	3.78 ± 0.07^a	0.66 ± 0.01^a

Mean values in the same column with different letters (a-d) are significantly different ($P < 0.05$).

et al., 1999; Achayuthakan and Suphantharika, 2008). Therefore, the dynamic rheological properties of the starch paste were determined by the structural properties of the dispersed and continuous phases, which could also provide useful information about its viscoelastic properties. Table 3 shows G' , G'' , and η^* at $6.28 \text{ rad} \cdot \text{s}^{-1}$ for the WRS-CMC mixtures at 25°C . In general, G' , G'' , and η^* for the WRS-CMC mixtures were higher than those for the control (without CMC) and increased with increased concentration of CMC, indicating that the addition of CMC to WRS pastes had a synergistic effect on viscoelastic properties. The synergistic effect of CMC on the rheological properties of WRS pastes may be interpreted as an increase in the viscoelastic properties of CMC due to the increase of its local concentration in the continuous phase in the starch-gum mixture system (Alloncle and Doublier, 1991). The $\tan \delta$ values of all WRS-CMC mixtures were in the range of $0.49 \sim 0.66$, indicating that the WRS-CMC mixtures were predominantly more elastic than viscous. The $\tan \delta$ values increased with increasing CMC concentration from 0.2% to 0.6%. In addition, samples containing 0.6% CMC showed a much higher $\tan \delta$ value (0.66) compared with other samples ($0.49 \sim 0.59$), indicating that WRS-CMC mixtures exhibited a greater viscous behavior at higher CMC concentrations. Based on these observations, WRS-CMC mixtures may be viscoelastic depending on the CMC concentration.

The mechanism for the changes in the viscoelastic properties of starch pastes containing gums can be largely explained by an incompatibility phenomenon between dissimilar polysaccharides called coacervation (Kim and Yoo, 2006; Yoo et al., 2005; Lai et al., 2003). Coacervation is a type of phase separation, defined as a process during which a homogeneous colloidal solution separates into two immiscible liquid phases: the dense liquid phase (coacervate), which is relatively concentrated in colloidal molecules, and the diluted liquid phase containing less colloidal components in equilibrium with the coacervate phase (Zhao and Wang, 2017). According to Alloncle and Doublier (1991), the starch dispersions may be regarded as a composite material consisting of swollen granules dispersed in a continuous biopolymer matrix. Therefore, when the gum is located within the continuous phase, the volume of this phase is reduced, which causes a

marked increase in gum concentration in the continuous phase, resulting in a very high viscosity. The results are in agreements with those reported for mixtures of gums with acorn flour and rice starch (Kim and Yoo, 2011; Kim and Yoo, 2006).

The effect of CMC on the viscoelastic properties of WRS pastes can be explained by the formation of a thermodynamically incompatible network structure considering that addition of CMC results in a small increase in G' compared with G'' with $\tan \delta$ values ($0.51 \sim 0.66$) higher than that of the control (0.49). Therefore, the mechanism of the synergistic effect of CMC in the WRS-CMC mixture system can be predicted by the changes in dynamic rheological parameters, as highlighted by Kim et al. (2009) and Funami et al. (2008).

Applicability of Cox-Merz rule

The applicability of the Cox-Merz rule (Cox and Merz, 1958) (equivalence between steady shear rheological properties and small-amplitude dynamic rheological properties at equal frequency values ($\omega = 0.63 \sim 62.8 \text{ rad} \cdot \text{s}^{-1}$) and $\dot{\gamma}$ ($0.4 \sim 100 \text{ s}^{-1}$) for WRS-CMC mixtures with different CMC concentrations was evaluated (Fig. 5). The magnitudes of η^* were lower than those of η_a , demonstrating that WRS-CMC mixtures did not obey the Cox-Merz rule over the entire range of $\dot{\gamma}$ and ω investigated. This behavior may be attributed to the heterogeneous nature of the polysaccharide dispersions that undergo aggregation and the highly branched structure of the polysaccharides, as noted by Da Silva and Rao (1992) and Xu et al. (2006). In addition, Silva et al. (2017) stated that the $\eta^* < \eta_a$ behavior may be associated with differences in structure formation when samples are subjected to dynamic oscillatory shear and steady shear measurements. This phenomenon was also observed for other starch or gum dispersions (Da Silva and Rao, 1992; Xu et al., 2006; Chun and Yoo, 2007). Similar differences between the two lines (η^* and η_a) were observed for all samples, indicating that the deviation from the Cox-Merz rule was not affected by the CMC concentration. These results suggest that the applicability of the Cox-Merz rule may be dependent on the type of starch or gum used in the starch-gum mixture system.

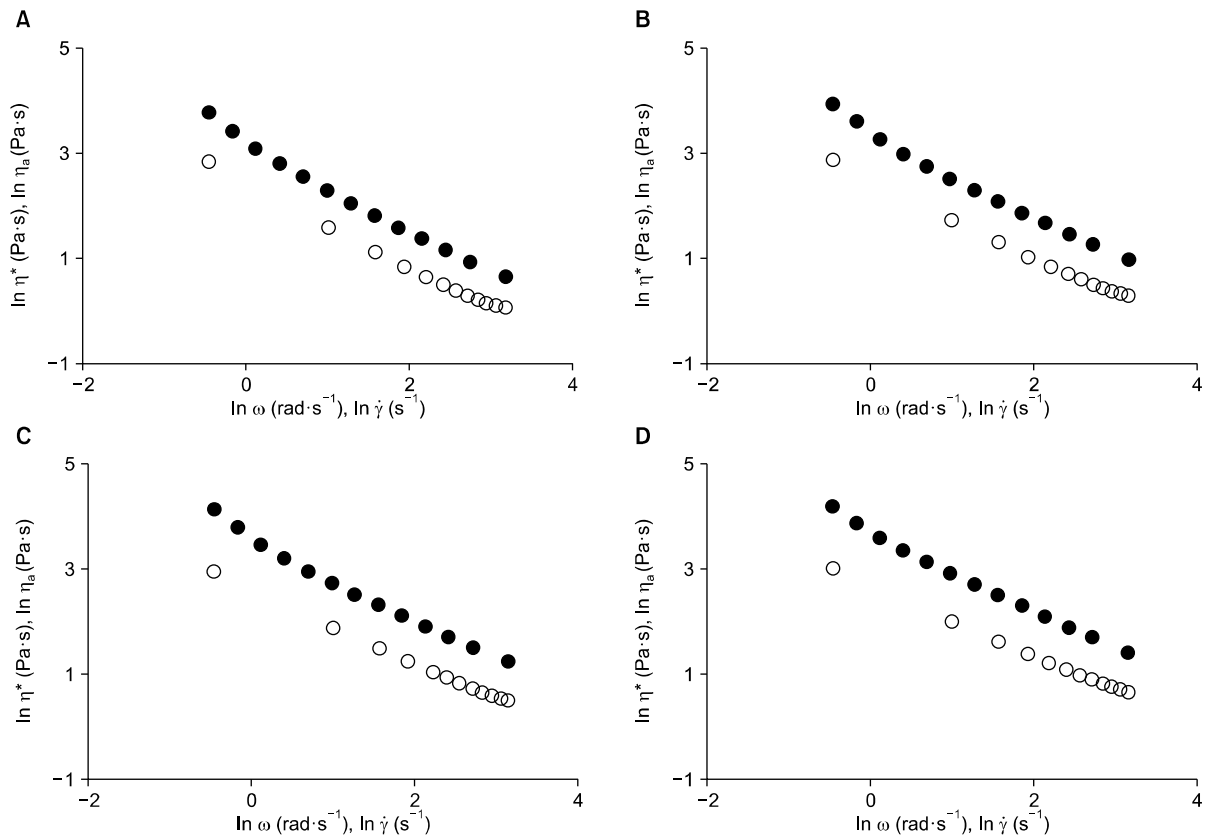


Fig. 5. Combined plot of apparent viscosity (η_a , ●) and complex viscosity (η^* , ○) versus angular frequency/shear rate (Cox-Merz plot) for waxy rice starch-carboxymethyl cellulose (CMC) mixtures with different CMC concentrations at 25°C: (A) 0%, (B) 0.2%, (C) 0.4% and (D) 0.6%.

ACKNOWLEDGEMENTS

This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIT) (NRF-2019R1H1A2080055). This work was also supported by the Dongguk University Research Fund.

AUTHOR DISCLOSURE STATEMENT

The authors declare no conflict of interest.

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