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Rheological study of starch and dairy ingredient-based food systems

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Abstract

An oscillatory probe rheometer was used to measure the basic rheological properties of food model systems consisting of starchwater, starch-protein, starch-sugar, and starch-sugar-protein mixtures. The mixtures were heated to 85 °C, and the rheological properties of the mixtures were measured during the cooling phase. The independence of complex modulus (G^*) on frequency of oscillation indicated that the starch and water system produced stronger gels than systems containing WPI due to its inactive filler. During the cooling, G' of starch-WPI (1:0.3) gels decreased but started to increase at warm temperature due to the effect of hydrophobic bonds. The addition of sucrose to the starch system lowered G' at 85 °C. During the cooling, G' of starch-sucrose gels increased, due to the effect of hydrogen bonds. Replacement of 50% sucrose with lactose caused different cooling, G' increased due to the different sugar. Addition of WPI to the starch and sucrose system decreased G' at 85 °C. During the cooling, G' increased due to the effects of hydrogen bonds. Substitution of 50% WPI with egg white protein did not change this, which might be beneficial in increasing elastic modulus and the rigid structure of bakery products during the later stage of baking and during cooling. In a pancake, addition of whey protein concentrate decreased the G', probably due to the limit of available water for gelatinization of starch and gelation of proteins.

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1. Introduction

Rheology is the study of the deformation and flow of matter (Rao, 1999). In food engineering, rheology helps to understand how food structure responds to applied force and deformation. Starch and protein play major roles in providing the desirable characteristics of food products from cereals, meat, and other sources. The texture of many bakery products depends upon the interaction of these constituents with water. Hence, from a practical point of view, the rheological characterization of foods and their constituents is very important, particularly in relation to structure and stability, and processing design. Knowledge of specific interactions and synergistic effects is highly desirable for creating new textures and to enable ingredient substitutions. The aim of this research was to study the rheological properties of dairy ingredients, such as whey protein and lactose, in starch-based food model systems with limited water. Small amplitude dynamic rheometry, which ensures both non-destructive measurements and simultaneous recording of viscous and elastic responses of a sample (Gunasekaran & Mehmet Ak, 2000), was used to monitor the gelation of starch-based dairy food models during cooling, by subjecting the sample to a very small stress.

Despite the commercial significance of starch pastes and food systems containing starch, there is relatively limited fundamental information available on the rheological properties of starch-based composite foods. Rheological properties of individual components, such as wheat starch and whey protein were studied (Aguilera,

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1995; Mangino, 1992; Wong & Lelievre, 1981); however, the basic rheological properties (complex modulus, G^* , storage modulus, G', and phase angle, δ) of these mixes were not studied. Differential scanning calorimetry (DSC) and viscometry were used to examine the effects of sugars on starch gelatinization and their effects on viscosity of starch (Kim & Walker, 1992; Spies & Hoseney, 1982).

Rheological properties of starch and cereal- or noncereal-derived proteins have been reported over the vears (Aguilera & Rojas, 1996; Chedid & Kokini, 1992; Madeka & Kokini, 1992; Muhrbeck & Eliasson, 1991). The viscoelasticity of interactions of sugar and starch has been widely studied. Basically, the sugar increases the gelatinization temperature of starches by reducing granular swelling. However, the rheological properties of starch and sugar mixtures in a waterlimited system were not addressed, except in the studies of Prokopowich and Biliaderis (1995); Sikora, Mazurkiewicz, Tomasik, and Pielichowski (1999), and Chiotelli, Rolée, and Le Meste (2000). At 50% water content, sucrose, maltotriose, and especially ribose, significantly delayed the G' development (Prokopowich & Biliaderis, 1995). Glucose greatly promoted rigidity development during the entire process while fructose enhanced it in the first 12 h and later retarded the process. Starch gelatinized in saturated sucrose solution more readily than in solutions of D-fructose and D-glucose (Sikora et al., 1999). Sugar delays starch gelatinization due to sugar-starch interactions in the amorphous and/or the crystalline regions of the starch granules (Chiotelli et al., 2000). Most of the previous works on rheological measurements were conducted at constant temperature.

Whey protein isolate (WPI) and concentrate have commonly been used as food ingredients to control the quality of food products because of their various complementary functional properties (Morr & Ha, 1993; Phillips, Whitehead, & Kinsella, 1994). The gelatinization of starch with other food ingredients, such as whey protein, by heating in a mixing food system is different from that without them. Therefore, the study of interactions of starch and whey protein in food systems is very important. There were a few studies of corn starch (Shim & Mulvaney, 2001) and cassava starch (Aguilera & Rojas, 1996, 1997; Aguilera & Baffico, 1997) with whey protein. The stress relaxation test indicated that WPI gel, depending on pH, is a highly transient network structure while corn starch (CS) gel, depending on heating temperature, is very elastic (Shim & Mulvaney, 2001). Mix gels (CS:WPI = 0.5) at pH 9 showed a unique chemical compatibility with combination of the elasticity of CS and the internal stress dissipation of WPI. DSC measurement demonstrated independent thermal transitions in cassava starch/whey protein gels with 90% water content, respective to gelatinization and gelation (Aguilera & Rojas, 1996). With high cassava starch content (starch fraction >0.7), mixed gels showed low G' value due to formation of a weak cassava starch matrix with dispersed WPI regions. With low cassava starch content (0 < starch fraction < 0.4), mix gels showed a higher G' value than pure WPI gels. Destructive test (axial compression) result demonstrated similar effects of cassava starch contents on the structure of mix gels (Aguilera & Baffico, 1997).

Until now, most studies on rheological properties of food have focussed on interactions of two components in food systems, such as starch-water, protein-water, and starch-sugar. There is no study on rheological properties of interactions of more than two components in food systems, such as starch-sugar-protein systems, which are very common in food products. Basic rheological information during the transformation from sol-to-gel upon heating will be very important in determining the rheological characteristics of baked products. Such information will also be useful in understanding the flow behaviour of new starch-based composite food systems.

In this study, egg white protein was also selected to mix with starch and sugar because it has been extensively applied as an ingredient for food products due to its unique functional properties, such as gelling and foaming (Mine, 1995). The overall objective of this research was to study the rheological characteristics of starch and dairy-based food mixes (starch–sugar, starch–protein, and starch–sugar–protein), produced using wheat starch, whey protein, and sugars (lactose and sucrose). The specific objectives were: (1) to study basic rheological properties of starch–sugar–protein-based food model systems using small deformation rheometry and (2) to evaluate the rheological characteristics of a real food system.

2. Materials and methods

2.1. Materials

Wheat starch (AYTEX^R P) was obtained from a commercial source (ADM Milling CO., Keokuk, IA); Lactose-Grind 200 containing 95% of milk sugar was obtained from Land O'Lakes (Food Ingredients Division, Minneapolis, MN); whey protein isolate (WPI, Alacen 895) and whey protein concentrate (WPC, Alacen 878) were provided by New Zealand Milk Products, Inc. (Santa Rosa, CA); Sucrose was obtained from Mallinckrodt Baker, Inc. (Paris, KY). Pastry flour (Bob's Red Mill), butter, sugar, dry whole milk (Carnation) and baking powder (Clapped Girl) were purchased from a local grocery store (Smith's Grocery Logan, UT). Egg white protein was obtained from Marshall Egg Products (Marshall, MO).

2.2. Sample preparation

Two grammes each of wheat starch and water were used in all experimental mixtures. The amounts of starch and water were kept constant in all experiments. Ratios of WPI (dry weight basis) to starch examined were 0.15:1, 0.2:1, and 0.3:1. A combination of WPI and egg white protein was also used to study the effect of this composite model since most real foods contain these ingredients. The protein content was chosen, based on the typical protein content found in flour mixes. Based on the amount of sugar present in baked products, the approximate ratios of sucrose to starch used were 0.2:1, 0.4:1, and 0.6:1. The effect of lactose was examined by conducting a series of experiments in which 50% of sucrose was replaced by lactose in the starch-sugar mixture. For example, the 0.2:1 sucrose-starch complex was replaced by 0.1:0.1:1 sucrose-lactose-starch food model.

Analyses of a real food model with different levels of WPI were also done using a pancake mix. The mix consisted of water (47%), pastry flour (27%), dry whole milk (7%), butter (7%), WPI (7%), sugar (3%) and baking powder (2%). Rheological properties were assessed for whey protein concentrate (WPC) levels of 0%, 10%, 20% and 40%. Samples were prepared by adding the wet ingredients to the dry ingredients, gradually, and stirring continuously until the ingredients were mixed well. The samples were tested after mixing for 4 min.

2.3. Rheological experiments

The dynamic rheological properties of the food models were investigated by measuring the storage modulus (G'), complex modulus (G*), and phase angle (δ) with respect to stress, frequency, and temperature. The HAAKE Rheostress RS-75 rheometer (Paramus, NJ), with the parallel plate geometry probe (35-mm in diameter) and a thermostat-controlled water bath (DC5), was used for experiments. The gap between the plates was 1.0 mm.

Three types of tests were conducted. Stress sweep tests in the range between 100 and 1100 Pa were first conducted at room temperature (25 °C) at a frequency of 1 Hz to determine the linear viscoelastic region, necessary for the subsequent temperature and frequency sweep experiments. Prior to measurement, the sample was heated to 85 °C for 30 min at a rate of 1 °C/min and cooled immediately to 25 °C for 60 min at the same rate. Measurements were taken during the cooling phase. An average shear stress of 300 Pa was selected for starch and protein mixtures and 800 Pa was selected for starch and sugar systems after several trials with all the food models. The second set of experiments studied the rheological properties as a function of temperature. Temperature sweep tests were performed in the range between 85 and 25 °C at a constant shear stress value of 800 Pa and a frequency of 1 Hz. In order to prevent moisture loss from the sample during the heating processes, the edges of the plates were sealed with mineral oil. Lastly, frequency sweep experiments were conducted to characterize the degree of viscoelasticity of the sample within certain time limits. These experiments were completed immediately after the temperature sweep experiments. In a frequency sweep test, the response of a material is measured as a function of frequency (0.01–4.64 Hz) at a constant stress amplitude of 300 Pa at 25 °C. All experiments were replicated three times. Averaged values of replications were taken.

2.4. Data analysis

SAS (Version 6.09, Cary, NC) on VAX/VMS system was used for all statistical analysis. Data were analyzed using the general linear model (GLM) approach. Analysis of covariance was done separately for each of the dependent variables (storage modulus, complex modules, and phase angle) and also to evaluate the effects of sugar and protein on starch. Significance was accepted at the 5% confidence level.

3. Results and discussion

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3.1. Starch and WPI mixture

Starch and water dispersions heated above their gelatinization temperature behaved as viscoelastic pastes. Upon cooling, the paste thickens and may form an elastic gel if the dispersion has sufficient concentration (Ring, 1985). Fig. 1 shows the frequency independence of the complex modulus G^* for starch-protein mixtures within the testing range (0.01–4.64 Hz). Statistical analysis showed that addition of WPI decreased G^* of the starch system significantly (P = 0.0008 < 0.05). Mean G^* values of the 1:0.3 starch-WPI model were different for the systems with a lower WPI concentration



Fig. 1. Variation of complex modulus with frequency in starch–WPI model. Starch–WPI ratios are: (a) 1:0.0; (b) 1:0.3; (c) 1:0.2; and (d) 1:0.15.

(P < 0.05). The firmness of the gel depends on the extent of junction zone formation (BeMiller & Whistler, 1996). In this study, the frequency independence of the G^* , for all samples, indicated the presence of a network with a low possibility of rupture at junction zones within the frequency range (0.01–4.64 Hz) used. A frequency selected from 0.01 to 4.64 Hz for measurement was within a linear viscoelastic range.

Starch–water dispersion had higher values of G^* than those systems containing WPI (Fig. 1). This indicated that WPI weakened the gel structure of the starchprotein mixture, especially at low content (curves c and d in Fig. 1). The increase of WPI content in the mixtures caused the increase of the gel strength (curve b in Fig. 1), which was still lower than that of starch-water gel (curve a in Fig. 1). In this study, there were two gel systems in the mixture systems: WPI gel and starch gel. The water content is 50% for the starch-water system. When WPI was added, the water content decreased. The gelation of WPI competed for water in the mixture systems. It reduced the available water for the starch granule, so the starch gel was weakened (curve d in Fig. 1). Therefore, WPI acted as inactive filler and diluted gel strength of the starch fraction (Yang & Park, 1998). On the other hand, the addition of WPI to the starch-water system resulted in reduction of moisture in the starch-protein system. The reduction of moisture could increase the firmness of the starch-protein system, resulting in the increase of G^* as more WPI was added (curves b and c in Fig. 1). However, the reinforcement of the gelation of WPI and the reduction of moisture were not complementary for the reduction of gel strength of the starch-water system.

Changes in the storage modulus (G') of starch systems, as a function of WPI content, determined during cooling, are shown in Fig. 2. A significant difference (P = 0.0001 < 0.05) was observed between the 1:0.15 and 1:0.3 starch-WPI gels, whereas no significant difference (P = 0.089 > 0.05) was observed between the 1:0.15 and 1:0.2 gels. As the temperature decreased, G'of pure (wheat) starch gel increased (curve a in Fig. 2). A similar phenomenon occurs in corn and rice starches (Tsia, Li, & Lii, 1997). Starch gels are commonly regarded as composite systems, consisting of swollen particles embedded in a three-dimensional network of aggregated amylose chains. During the cooling, the effect of hydrogen bonds in the gel matrix gradually increased, especially at low temperature, so G' increased. When the temperature decreased to 50 °C, G' reached its peak, and then started to decrease, probably due to the contraction of gel volume during the cooling (Tsia et al., 1997).

When WPI was added to starch systems, the changes of G' of starch–WPI gels during the cooling were different from that of starch gel. At the low WPI content (1:0.15, curve d in Fig. 2), G' decreased during cooling.



Fig. 2. Storage modulus of starch–WPI food models as a function of temperature. Starch–WPI ratios are: (a) 1:0.0; (b) 1:0.3; (c) 1:0.2; and (d) 1:0.15.

When the content of WPI increased (1:0.2, curve c in Fig. 2), G' still decreased but the reduction was not as much as that at the 1:0.15 level. When more WPI (1:0.3) was added, G' was more than that of pure starch gel at 85 °C. When temperature decreased, G' initially decreased. But, at 65 °C, G' started to increase (1:0.3, curve b in Fig. 2). At low WPI content (1:0.15), the starch gel matrix was diluted due to inactive filler of WPI, so G' of gels decreased. Even during cooling processes, the diluting effect of WPI on the gels reduced the association of hydrogen bonds in the mixtures, so G' values are further weakened. However, as WPI content increased, the reinforcing effect of WPI on the gel matrix increased. In the starch-protein systems, swollen starch granules were embedded in the three-dimensional network of aggregated amylose matrix but also the protein (WPI) matrix. The reinforcement of WPI gelation overtook the weakening of the dilution, resulting in a mixed gel, stronger than pure starch gel at 85 °C. During cooling, G' decreased from 85 to 65 °C and then increased up to 30 °C (curve b in Fig. 2). This was probably caused by the hydrophobic bonds from protein-protein interactions. Unlike hydrogen bonds, hydrophobic interactions are enhanced in warm temperature (40-60 °C) (Cheftel, Cuq, & Lorient, 1985; Niwa, 1992). Therefore, when more WPI was added, the effect of hydrophobic bonds on the gel during the cooling from 65 to 30 °C, increased. However, it was not as high as pure starch gel, probably due to the dilution effect of WPI on the association of hydrogen bonds from water. After 30 °C, the effect of hydrophobic interactions decreased as temperature continually decreased, so G' decreased.

Fig. 3 shows a plot of the phase angle, δ , against decreasing temperature for starch with three levels of WPI. At 25 °C, a lower value of δ was obtained for starch solution; however, δ decreased as WPI increased. The lower value of δ indicates a stronger gel. While cooling, the phase angle of the sample with a starch–WPI ratio of 1:0.15 began to increase more markedly,



Fig. 3. Phase angles of starch–WPI food models as a function of temperature. Starch–WPI ratios are (a) 1:0.0; (b) 1:0.3; (c) 1:0.2; and (d) 1:0.15.

from approximately 65 °C, than the 1:0.2 and 1:0.3 samples. This signified a weakening gel structure during cooling processes for a mixed gel with low WPI content (1:0.15, curve d in Fig. 3). Low values of δ for pure starch gel (curve a in Fig. 3) and mixed gel with high WPI content (1:0.3, curve b in Fig. 3) indicated the formation of strong gel structure. At 85 °C, δ of the high WPI mixed gel was lower than that of pure starch gel. This confirms the reinforcement of WPI from the δ point.

3.2. Starch and sugar mixtures

Figs. 4 and 5 show plots of G' against decreasing temperature for the starch-sucrose-lactose mixtures. At 85 °C, there was no significant difference (P > 0.05) between the starch-sucrose and starch-sucrose-lactose systems. However, at 25 °C, replacing sucrose with lactose resulted in a significant (P < 0.05) increase in G', compared to the starch-sucrose mixture. At 85 °C, at addition of low sugar (Fig. 4), G' of pure starch gels was higher than that of sucrose-starch gels but lower than that of sucrose-lactose-starch gels. At high sugar con-



Fig. 4. Storage modulus of starch–sucrose–lactose system as a function of temperature: (a) starch; (b) 1:0.2 starch–sucrose; (c) 1:0.1:0.1 starch–sucrose–lactose.



Fig. 5. Storage modulus of starch–sucrose–lactose system as a function of temperature: (a) starch; (b) 1:0.6 starch–sucrose; (c) 1:0.3:0.3 starch–sucrose–lactose.

tent (Fig. 5), G' of both mixed gels was lower than that of pure starch gels. This is due to the delay effect of sugar on the gelatinization and the fact that different sugars have different effects on the gelatinization of starches (Slade & Levine, 1987). When half of the sucrose was replaced by lactose, the gelatinization of starch was changed. The increasing gelatinization temperature of lactose is lower than that of sucrose (Slade & Levine, 1987). Therefore, when half of the sucrose was replaced by lactose, the effects of sugar on increasing gelatinization temperature of starch were reduced, so G' values of the mixtures with lactose were higher than those with sucrose. During the cooling, G' of all gels increased, due to the increase of hydrogen bonds at cold temperature.

3.3. Starch-sucrose-WPI mixtures

Rheological properties of starch and sucrose mixture, with the addition of WPI and egg white protein, were analyzed, since many food systems contain these three ingredients along with water. Fig. 6 shows the storage modulus of starch, starch–sucrose, and starch–sucrose– WPI systems during cooling. At 85 °C, the addition of



Fig. 6. Storage modulus of starch–sucrose–WPI system as a function of temperature: (a) starch; (b) 1:0.2 starch–sucrose system; (c) 1:0.2:0.3 starch–sucrose–WPI.

WPI to starch-sucrose mixtures did not affect G'(P > 0.05), but significantly did so at 25 °C (P < 0.05). Upon cooling, G' in the starch-sucrose-protein mixtures increased gradually due to the increase of hydrogen bonds in the mixed gels. On the one hand, the addition of WPI to starch and sucrose mixtures increased G', due to the reinforcement of the WPI matrix. On the other hand, sucrose affects protein-protein interactions in gels through enhancement of hydrophobic interactions (Dierckx & Huyghebaert, 2002). The reinforcement and enhancement resulted in the increase of values of G', which were even higher than G' for pure starch gels (curve c in Fig. 6).

To study the effect of interactions of egg white protein and WPI on starch-sugar-protein systems, the 1:0.6 starch-sucrose ratio was kept constant and 50% of the WPI was replaced by egg white. The variation of G' with temperature for the starch-sucrose system with WPI and WPI-egg white composition is shown in Fig. 7. Additions of sucrose and proteins significantly lowered G'(P < 0.05) at 85 °C. Throughout cooling, G' of mixed gels increased. Up to 25 °C, the values of G' were close (P > 0.05). High sucrose content (1:0.6, curve b in Fig. 7) delayed the gelatinization of starch compared to low sucrose content (1:0.3, curve b in Fig. 6). This was due to the effect of sucrose on starch gelatinization. Increasing sucrose can cause higher gelatinization temperature. Addition of WPI to a high sucrose content of the starch-sucrose system further reduces the value of G', due to the inactive filler of WPI (curve c in Fig. 7). At 85 °C, G' of the starch–sucrose–WPI gel was lower than that of the starch-sucrose gel. However, during the cooling, G' of the starch-sucrose-WPI gel increased gradually, even above that of the starch-sucrose gel after 70 °C. This was due to the enhancement of hydrophobic interactions due to the warm temperature (60–40 °C) and sucrose.

When half of the WPI was replaced by egg white protein, the gel structure was additionally weakened, so egg white protein was also an inactive filler. The presence of WPI and egg white protein in starch-sugar



Fig. 7. Storage modulus of starch-sucrose-protein food model as a function of temperature: (a) starch; (b) 1:0.6 starch-sucrose; (c) 1:0.6:0.3 starch-sucrose-WPI; (d) 1:0.6:0.15:0.15 starch-sucrose-WPI-egg white.



Fig. 8. Phase angle of starch–sucrose–protein food model as a function of temperature: (a) starch; (b) 1:0.6 starch–sucrose; (c) 1:0.6:0.3 starch–sucrose–WPI; (d) 1:0.6:0.15:0.15 starch–sucrose–WPI–egg white.

systems reduced the availability of water for starch granules to swell during gelatinization for the reinforcement of the gel structure. At such low water content, it also delayed the gelation of proteins. During cooling processes, a more elastic gel was formed. This was caused by the increase of hydrophobic interactions due to the warm temperature (40–60 °C) and sucrose, as well as the increasing effect of hydrogen bonds at the cold temperature.

Fig. 8 demonstrates the phase angle of starch, starch– sucrose, and starch–sucrose–protein systems during cooling processes. At 85 °C, the values of δ increased upon addition of WPI and egg white protein, indicating that mixed gels were weaker than pure starch gel (P < 0.05). Values of δ decreased as the temperature decreased, indicating that gels became stronger during the cooling, due to the effects of hydrophobic interactions at warm temperature and hydrogen bond interactions at cold temperature. At 25 °C, phase angles among mixed gels were not significantly different (P > 0.05).

3.4. Real food model – pancake

Pancake is a complex system because of the interaction between starch and all the other ingredients, such as proteins, sugar and fat. In pancake batter, whey protein concentrate (WPC) can help to stabilize the emulsion by preventing migration and coalescence of dispersed fat globules. Evenly dispersed fat, throughout the pancake batter, gives the final product a uniform texture and flavour. The rheological properties of pancake mix were studied at a constant stress value of 300 Pa and frequency of 1 Hz. Fig. 9 shows the storage modulus of pancake mix during cooling. At 85 °C, values of G' for all the mixed gels were very close (P > 0.05) but were significantly different at 25 °C (P < 0.5). As the temperature decreased, G' increased gradually upto about 70 °C and then reached an almost a steady state. After 70 °C, the general trends of curves were similar for all



Fig. 9. Storage modulus for pancake mix as a function of decreasing temperature.

the samples tested. As the content of WPC increased, G' values were decreased by further cooling of mixed gels and were lower than the values for the standard pancake mix.

Experiments (Fig. 9) indicated that an increased amount of WPC significantly affected the G' values of the standard pancake mix. The decreased values of G'with higher concentration of WPC were probably due to the increased amounts of lipid and lactose present in the WPC, since adding lipids or sugars delays starch gelatinization, swelling, and leaching of amylose from the starch granule (Kim & Walker, 1992; Spies & Hoseney, 1982; Tester & Morrison, 1990). The available water also decreased with increase of WPC, so the effect of hydrogen bonds at the cold temperature, on the mixed gels, decreased. Therefore, WPC was an inactive filler.

4. Conclusions

The starch–WPI gel had a lower G^* and higher δ values than the starch food model during cooling processes. As the concentration of WPI increased, the gel strength increased, due to the enhancement of hydrophobic interactions. However, with lower WPI concentration, the network exhibited a lower strength, due to the inactive filler of WPI. Replacement of 50% of sucrose by lactose in the starch–sucrose food model affected the storage modulus of the starch–sucrose system. Hence lactose could be used in bakery products with lower levels of sugar, to impart strength and rigidity.

Addition of WPI to the starch-sucrose system increased G' during cooling. When egg white was used to replace 50% of the WPI, a decrease in G' and increase in δ were observed. However, there was a continued increase of gel strength due to hydrophobic interactions at warm temperature and hydrogen bond interactions at cold temperature, during the cooling. Results indicated that WPI replacement for egg white protein might be beneficial in increasing elastic modulus and the rigid structure of bakery products during the later stage of baking and during cooling. The storage modulus decreased with increase in the concentration of WPC in the pancake mix, probably due to the limit of available water for gelatinization of starch and gelation of proteins.

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