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IMPACT OF WET GLUTEN CONTENT ON NON-LINEAR VISCOELASTIC PROPERTIES OF WHEAT FLOUR DOUGHS

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ABSTRACT

The impact of wet gluten content in wheat flours on viscoelastic responses of the resulting wheat flour doughs under large deformations were studied using the Large Amplitude Oscillatory Shear (LAOS) tests. For this purpose, dough samples of hard red winter (HRW) wheat flour with 29.8±0.26% wet gluten and soft red winter (SRW) wheat flour with 23.9±0.15% wet gluten were obtained at the end of the Farinograph tests. Farinograph mixing stability and optimum water absorption capacity were higher for HRW wheat flour. LAOS tests revealed the contribution of gluten content to the resilience of wheat flour dough against the increasing deformations. Higher strain stiffening was found for HRW wheat flour dough with higher gluten content under large deformations with high frequency, resembling the deformations experienced during dough processing steps such as mixing or sheeting. Intracycle shear thinning behaviors of doughs were not affected by the gluten content at each frequency studied.

Keywords: wet gluten content, wheat flour dough, rheology, LAOS

YAŞ GLÜTEN MİKTARININ BUĞDAY UNU HAMURUNUN DOĞRUSAL OLMAYAN VİSKOELASTİK ÖZELLİKLERİ ÜZERİNDEKİ ETKİSİ

ÖZ

Buğday unlarındaki yaş glüten miktarının, bu unlardan elde edilen hamurların yüksek deformasyonlar altında göstermiş oldukları viskoelastik özellikleri üzerindeki etkisi Yüksek Genlikli Salınımlı Kayma (LAOS) testi ile belirlenmiştir. Bu amaçla, %29.8±0.15 yaş glüten içeren sert kırmızı kışlık buğday unu ve %23.9±0.26 yaş glüten içeren yumuşak kırmızı kışlık buğday unundan Farinograf testi ile hamur örnekleri elde edilmiştir. Sert kırmızı kışlık buğday ununun Farinograf stabilite ve optimum su kaldırma değerlerinin daha yüksek olduğu tespit edilmiştir. LAOS testleri, buğday unundaki glüten miktarının, artan deformasyonlar karşısında hamurun göstermiş olduğu mukavemete katkı sağladığını ortaya koymuştur. Daha yüksek yaş glüten miktarına sahip olan sert kırmızı kışlık buğday unu hamuru, yoğurma ve açma gibi hamur işleme aşamalarında görülen yüksek frekanslı deformasyonlar altında daha fazla gerinim katılaşması davranışı göstermiştir. Diğer taraftan, hamur örneklerinin her bir frekansta belirlenen döngü içi (salınım döngüsü) kayma incelmesi değerlerinde glüten miktarına bağlı olarak belirgin bir değişiklik gözlemlenmemiştir.

Anahtar kelimeler: Yaş glüten miktarı, buğday unu hamuru, reoloji, LAOS

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INTRODUCTION

Protein content of wheat varies between 7% and 18% and the proteins that form gluten constitute a large part (around 80%) of the total proteins (Shewry et al., 1997; Peña et al., 2002; Uthayakumaran and Wrigley, 2017; Guzmán et al., 2022). In general, the quality of wheat flour for bread making is evaluated by the amount of protein and the quality of gluten (Best et al., 2023), as gluten is the main network-forming protein in wheat flour with its ability to impart the desired viscoelasticity to dough (Yazar et al., 2017a). The viscoelastic properties of the gluten network formed upon hydration of flour particles during mixing help dough retain gas to obtain the desired loaf volume and crumb texture in bread (Shewry et al., 1997; Uthayakumaran and Wrigley, 2017; Yazar et al., 2017a). Thus, variations in the breadmaking abilities of different wheat flours have been found to relate to the differences in gluten proteins (Uthayakumaran et al., 2002). The differences in gluten content, gluten composition (i.e., gliadin to glutenin ratio), and gluten quality in different wheat flours have been suggested as the origin of textural differences between a wide variety of baked products (Bonilla et al., 2022). Therefore, gluten quality and quantity characterization constitute a major part of wheat flour quality assessment in terms of baking performance (Yazar, 2023).

Gluten properties and functionality can be determined either using the physicochemical tests such as Gluten wash, Zeleny sedimentation; or it can be measured by testing the rheological properties of the whole wheat flour dough through the empirical methods including Farinograph, Extensograph, Mixograph, and Alveograph tests (Uthayakumaran and Wrigley, 2017; Best et al., 2023). Empirical methods have demonstrated their usefulness in industry and research to relate the rheological behavior of dough to baking performance, they have the disadvantage of providing data in arbitrary units, which makes the fundamental interpretation of results difficult (Campanella and Peleg, 2002; Dobraszczyk and Morgenstern, 2003). Therefore, fundamental rheological methods, that enable an accurate comparison and interpretation of the obtained data, have gained attention for the characterization of dough's viscoelastic properties (Yazar and Demirkesen, 2023; Yazar, 2023). Fundamental rheological tests are classified as small deformation and large deformation tests based on the magnitude of the applied deformation (Duvarcı et al., 2019). Small deformation tests show little relationship with end-use performance as they are generally conducted under deformation conditions inappropriate for breadmaking (Amemiya and Menjivar, 1992; Dobraszczyk and Morgenstern, 2003; Yazar, 2023). Studies have shown that small strain rheology was more advantageous for understanding molecular interactions and microstructure, while fundamental large strain rheology could be used to differentiate different types of wheat flours as these tests can approach the deformations dough experiences during processing (Kim et al., 2008).

In general, the higher the protein content of a wheat flour, the greater the gluten formation (Best et al., 2023). In other words, the amount of gluten in wheat flour is an index of the protein content (Kulkarni et al., 1987). Different varieties of wheat vary in their protein content and in the composition and distribution of gluten proteins (Best et al., 2023). Hard wheat flours (both durum and common) tend to have higher protein content than soft wheat flours (Hoseney and Rogers, 1990). Therefore, hard red winter wheat and soft red winter wheat flours were used in this study to determine the impact of gluten content in wheat flours on non-linear viscoelastic properties of the resulting doughs. For this purpose, gluten functionality of the selected wheat flours was studied through the gluten wash method, Farinograph mixing, and Large Amplitude Oscillatory Shear (LAOS) tests. Thus, this study aims to reveal the possibility of using LAOS tests as a fundamental large strain rheological testing method to differentiate between wheat flours based on their gluten contents, rather than using empirical dough testing methods.

MATERIALS AND METHODS

Materials

Hard red winter wheat flour [11.54±0.14% moisture, determined according to the AACC Approved Method 44-15.02 (AACC, 2010)] and soft red winter wheat flour [12.7±0.11% moisture, determined according to the AACC Approved Method 44-15.02 (AACC, 2010)] were obtained from Siemer Milling Company (Hopkinsville, KY).

Methods

Wet gluten content

Wet gluten contents of wheat flours were determined according to the hand-washing method [AACC Approved Method 38-10.01 (AACC, 2010)]. The results were corrected to 14% moisture content and expressed in percentages (%).

Farinograph test and dough preparation for the LAOS tests

Farinograph tests according to the AACC Approved Method 54-21.02 (AACC, 2010) were conducted using the Brabender Farinograph (Duisburg, Germany) equipped with a 300-g capacity mixing bowl to determine the mixing properties and optimum water absorption capacities of the wheat flours used in this study. Mixing tests were performed in triplicates and a representative Farinogram for both dough samples were provided. Wheat flour samples (weighed based on 14% moisture) were mixed with added water at their optimum water absorption capacities for 20 minutes at 63 rpm. Samples for the LAOS tests were prepared in an identical manner.

Large Amplitude Oscillatory Shear (LAOS) tests

The non-linear viscoelastic properties of doughs from wheat flours with different wet gluten contents were measured with a DHR-3 Rheometer (TA Instruments, USA) using the LAOS (Large Amplitude Oscillatory Shear Test) mode. Measurements were conducted at strain amplitudes ranging from 0.01% to 200% at three different frequencies (1, 10, and 20 rad/s) at 25°C. A 40 mm sand-blasted plate and a gap of 2 mm were used. Dough samples were rested until the axial normal force relaxed to 1 N. LAOS tests were conducted in triplicates and the averages of oscillatory data were calculated using Fourier transforms as suggested by Ewoldt et al. (2008). LAOS data were analyzed using the TRIOS software (TA Instruments, USA) and plotted using OriginPro 8.6.

Statistical analysis

OriginPro 8.6 was used for statistical analyses with 95% confidence level. Tukey's comparison tests were applied ($P < 0.05$) to compare the data obtained for the dough samples with different gluten content. Rheology data were compared separately for each frequency applied (1 rad/s, 10 rad/s, 20 rad/s) at each LAOS strain. Lettering system was used to show significant difference between samples.

RESULTS AND DISCUSSION

Wet gluten contents of flours

Wet gluten content for HRW wheat flour was 29.8±0.15%, while it was 23.9±0.26% for SRW wheat flour, indicating a significantly higher wet gluten content for HRW wheat flour ($P < 0.05$). Hard wheat flours were suggested to have higher protein content, and thus higher gluten content compared to soft wheat flours (Hoseney and Rogers, 1990). Wet gluten contents of HRW wheat flour and SRW wheat flour were found to range between 15.8- 42.1% (Kulkarni et al., 1987; Ohm and Chung, 2002; Maghirang et al., 2006) and between 8.6- 32.8% (Finney and Bains, 1999; Ma and Baik, 2016), respectively in earlier studies that were in line with the findings of this study.

Mixing properties of wheat flours

Figure 1 shows the Farinograms for hard red winter wheat flour and soft red winter wheat flour for an evaluation of the mixing properties and optimum water absorption capacities. Hard wheat flour showed two hydration peaks, appearing at the 1st minute and at the 9th minute of Farinograph mixing (Figure 1a). Certain flours show an exceptionally large hydration peak followed by a second later peak. This second peak is considered as the true dough development time as it correlates more directly with the baking performance of the flour being tested (Bock, 2022). Therefore, the peak time or dough development time for the HRW wheat flour was determined as 9 minutes in this study. Besides, the optimum water absorption capacity of HRW wheat flour was found as 63.3±0.11% (v/w, flour weight basis), arrival time was 0.5 minutes, departure time was longer than 20 minutes, and its stability was equal to 16.0 minutes (Figure 1a). On the other hand, soft wheat flour dough showed only one development peak during the Farinograph mixing at 0.6 minute (Figure 1b). The optimum water absorption capacity of SRW wheat flour was 60.2±0.15% (v/w, flour weight basis), arrival time was 0.35 minutes, departure time was 3.0 minutes, and its stability was 2.65 minutes. According to the classification of wheat flour strength using the Farinograph tests that was brought by Bock (2022), HRW wheat flour can be classified as a strong flour (Figure 1a) with the stability ranging between 5- 10 minutes, dough development time ranging between 2.5- 14 minutes, and water absorption ranging between 60-63%. When the shape of the Farinogram is considered, HRW wheat flour can be defined as very strong due to the presence of double hydration peaks (Don, 2022). On the other hand, the strength of SRW wheat flour can be defined as weak to medium (Figure 1b) with its stability below 3 minutes, dough development time below 2.5 minutes, and water absorption ranging from 55% to 60% (Bock, 2022). Ultimately, all the Farinograph parameters suggested a less resilient and a weaker network for SRW wheat flour compared to HRW wheat flour. The optimum water absorption capacity determined for HRW wheat flour was significantly higher ($P < 0.05$) than that of SRW wheat flour. The water absorption for soft wheat lines were 50.7–59.0%, while it ranged between 54.2–63.4% for hard wheat lines (Cao et al., 2017), supporting the Farinograph results reported in this study.

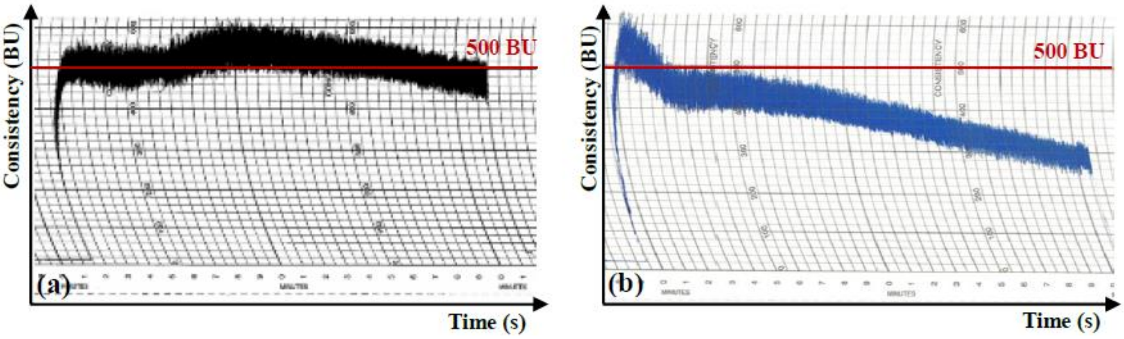

Figure 1. Farinograms for hard red winter wheat flour (a) and soft red winter wheat flour (b)

The mixing properties determined for hard and soft wheat flours using the Farinograph tests concurred with the wet gluten content values found for these wheat flours. This outcome indicates that the higher the gluten content, the higher the ability of the flours to absorb water. Besides the gluten properties, the higher water absorption capacity of HRW wheat flour has also been attributed to the higher levels of starch damage occurring during the milling of hard wheats (Ohm and Chung, 2002). However, as the Farinograph mixing proceeds, damaged starch granules lose their ability to hold all the water they absorbed in the early stages of mixing, leading to a progressive decrease in the stability (Sapirstein et al., 2007). This was not the case for the HRW wheat flour tested in this study, as evidenced by the double peak, and improved stability observed in the Farinograms for this flour (Figure 1a). Water absorption capacities determined through the Farinograph test have been reported to increase with respect to an increase in the grain protein content (Maghirang et al., 2006). Thus, the stronger network and higher water absorption found for HRW wheat flour compared to SRW wheat flour can be affiliated to the gluten contents of these flours. Starch damage was not included as a quality parameter in this study but should be considered in future work.

LAOS properties of wheat flour doughs with different wet gluten content

Linear and non-linear viscoelastic properties

Strain sweep data obtained in the linear region showed slightly higher G′ values for SRW wheat flour dough (Figure 2b) compared to HRW wheat flour dough (Figure 2a) at all frequencies studied, indicating a relatively elastic network for soft wheat flour dough as G′ was related to elastic properties. This result was interesting as HRW wheat flour had higher wet gluten content. However, it should be noted that gluten quality and gluten composition (i.e., gliadin to glutenin ratio) in wheat flours contributed to dough strength besides the gluten content (Khatkar et al., 1995; Uthayakumaran et al., 2002; Meerts et al., 2017). The higher G′ values obtained for SRW wheat flour dough in the linear region could be due to the higher gluten quality and/or higher glutenin to gliadin ratio in this flour. The impact of gluten quality and composition could be reflected on to linear viscoelastic properties of HRW and SRW wheat flour doughs as both wheat flour doughs were mixed at their optimum water absorption capacities. This difference in the viscoelastic behaviors of HRW and SRW wheat flours determined in the linear region conflicted with the Farinograph data as Farinograms indicated a weaker network for SRW wheat flour (Figure 1a,b). This is plausible as the deformations applied in the Farinograph mixing are large, complex, and non-uniform, while LAOS sweeps apply small and uniform deformations in the linear region (Yazar, 2023). Thus, for the hard and soft wheat flours tested in this study, the differences in the gluten composition became prominent under small deformations, while the difference in the gluten content dominated the viscoelastic behavior under large deformations.

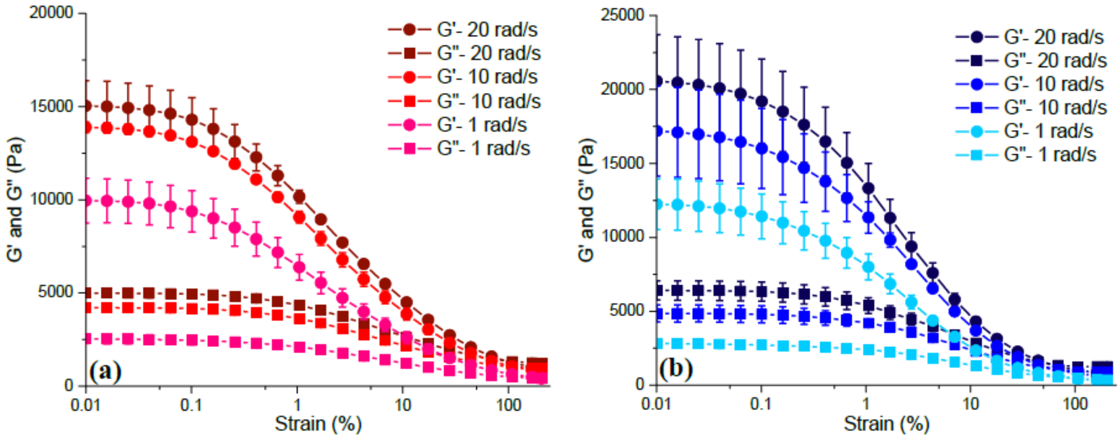

Figure 2. Strain sweeps: (a) hard wheat flour dough, (b) soft wheat flour dough [strain amplitude (γ): 0.01- 200%; frequencies (ω): 1 rad/s, 10 rad/s, 20 rad/s]

G′ and G″ values should be evaluated simultaneously for an in-depth analysis of the viscoelastic behavior. Thus, tanδ values resulting from the ratio of G″ to G′ (Duvarcı et al., 2019) were evaluated for both wheat flour doughs at selected strain amplitudes (Table 1). Even though, G′ values were higher for SRW wheat flour dough, tanδ values revealed no significant difference ($P > 0.05$) between the linear viscoelastic behaviors of both wheat flour doughs. As the amplitude of strain increased beyond 1.5%, the differences in tanδ values of HRW and SRW wheat flour doughs started to become more evident. In the non-linear region, tanδ values for HRW wheat flour dough were lower than those of SRW wheat flour dough ($P < 0.05$), suggesting a more elastic behavior for HRW wheat flour dough with higher gluten content against large deformations.

Table 1. Phase angle values for soft wheat flour dough and hard wheat flour dough at different LAOS strains (0.015%, 1.5%, 25%, 70%, 110%, 200%) and frequencies (1, 10, and 20 rad/s).

LAOS Strains (%)	Tangent δ					
	1 rad/sec		10 rad/sec		20 rad/s	
	Hard wheat flour dough	Soft wheat flour dough	Hard wheat flour dough	Soft wheat flour dough	Hard wheat flour dough	Soft wheat flour dough
0.015	0.25±0.00^{a}	0.23±0.02^{a}	0.30±0.00^{a}	0.28±0.01^{a}	0.33±0.01^{a}	0.31±0.01^{a}
1.5	0.35±0.01^{a}	0.33±0.01^{a}	0.42±0.01^{a}	0.40±0.01^{a}	0.45±0.00^{a}	0.43±0.01^{a}
25	0.55±0.03^{a}	0.72±0.01^{b}	0.66±0.03^{a}	0.79±0.00^{b}	0.71±0.02^{a}	0.83±0.01^{b}
70	0.68±0.04^{a}	0.91±0.03^{b}	0.85±0.03^{a}	1.01±0.02^{b}	0.91±0.05^{a}	1.08±0.04^{b}
110	0.79±0.05^{a}	1.04±0.04^{b}	0.98±0.03^{a}	1.17±0.03^{b}	1.06±0.07^{a}	1.25±0.06^{b}
200	0.99±0.06^{a}	1.31±0.06^{b}	1.24±0.04^{a}	1.50±0.02^{b}	1.34±0.10^{a}	1.58±0.03^{b}

tanδ values of hard and soft winter wheat flour doughs were evaluated separately at different LAOS strain amplitudes for each frequency applied.

Means that do not share a letter are significantly different ($P < 0.05$).

Significance level is 0.05.

G′ and G″ values for SRW wheat flour dough showed crossover (G′ = G″) between the strain amplitudes of 45% and 70% at the frequencies studied (Figure 2b). Beyond these strain amplitudes, SRW wheat flour dough had more viscous-like behavior (G″ > G′). On the other hand, G′ and G″ showed crossover for HRW wheat flour dough at the strain amplitudes of 70% and 110% when the frequency was 20 rad/s and 10 rad/s, respectively (Figure 2a). At the lowest frequency (1 rad/s), G′ and G″ did not show a crossover up to the highest strain amplitude (200%), suggesting higher resilience for HRW wheat flour dough under large deformations when compared to SRW wheat flour dough.

The strain amplitudes at which G′ and G″ crossover occurs increased for both wheat flour doughs as the frequency decreased. This revealed a delay in the viscous-like behavior against increasing deformations when the frequency was low. A similar trend was also captured through tanδ values, which increased as the amplitude of strain gradually increased, but decreased as frequency decreased (Table 1). This relatively more elastic behavior of wheat flour doughs at lower frequencies was attributed to the gluten network having more time to recover against the increasing magnitude of deformations at low frequencies (Yazar et al., 2022).

Strain sweep data showed similar linear viscoelastic properties for both HRW and SRW wheat flour dough. However, the higher wet gluten content in HRW wheat flour dough resulted in higher resilience against large deformations in comparison to SRW wheat flour dough. The information obtained regarding the viscoelastic behavior of wheat flour doughs in the non-linear region through the strain sweeps concurred with the Farinograph data that pointed out to a higher stability for HRW wheat flour dough against mixing deformations (Figure 1).

Hyun et al. (2002) provided a classification for the LAOS behaviors of materials. According to this classification, both wheat flour doughs showed type I non-linear behavior regardless of their wet gluten contents (Figure 3). Type I non-linear behavior (strain thinning) is described by the decrease in both normalized G' and G" versus increasing amplitude of strain (Hyun et al., 2002). The normalization of G' and G" in this evaluation helps us eliminate the redundancies and better observe the overshoots or decays (Yazar et al., 2023). In the linear region, the polymer chains are in a state of entanglement and thus G' and G" versus strain are constant, as observed in Figure 2 for both wheat flour doughs. As the amplitude of strain is gradually increased, polymer chains disentangle, and then align with the flow field resulting in a decrease in the moduli as seen in Figure 3. Yazar et al. (2022) characterized the non-linear viscoelastic behavior of hydrated gluten as a mixture of type III and type IV. In addition, hydrated wheat glutenin was found to have type III non-linear behavior (Yazar et al., 2023), while hydrated gliadin showed type IV non-linear behavior (unpublished data). Type III non-linear behavior (weak strain overshoot) is characterized by the decrease in G′ and the increase in G″ with respect to increasing strain amplitude. The complex structure of a materials with type III LAOS behavior resists against deformation up to a certain strain, where G″ increases. Then, the complex structure is destroyed by large deformation over the critical strain, after which the polymer chains align with the flow field, resulting in a decrease in G″. On the other hand, in type IV non-linear behavior (strong strain overshoot), the interaction between polymers was considered to be related with the intermolecular interaction between the hydrophobic groups and the interaction energy was suggested to be stronger than in type III (Hyun et al., 2002). Thus, wheat gluten consisting of almost equal portions of gliadin and glutenin proteins showed a mixture of type III and IV LAOS behaviors as suggested by Yazar et al. (2022). Although wheat gluten showed G′ and G″ overshoots at the onset of non-linearity, G′ and G″ values of both wheat flour doughs decreased as strain amplitude increased. Materials with type I non-linearity (i.e., wheat flour dough) were suggested to flow more readily compared to other materials with type III and IV non-linear behaviors (i.e., gluten, gliadin, glutenin). (Hyun et al., 2002). These findings revealed the contribution of starch to the decay observed in the moduli for wheat flour doughs in the non-linear region. There is an inverse relationship between amount of protein and starch. Thus, if protein content is higher in a flour, starch content must be lower (Hoseney and Rogers, 1990). As seen in Figure 3, the magnitudes of moduli for SRW wheat flour dough were slightly lower than those of HRW wheat flour dough at large strain amplitudes, indicating a more viscous-like behavior for SRW wheat flour dough under large deformations. This could be due to the presence of lower gluten content in SRW wheat flour dough, which eventually resulted in higher starch content compared to HRW wheat flour dough.

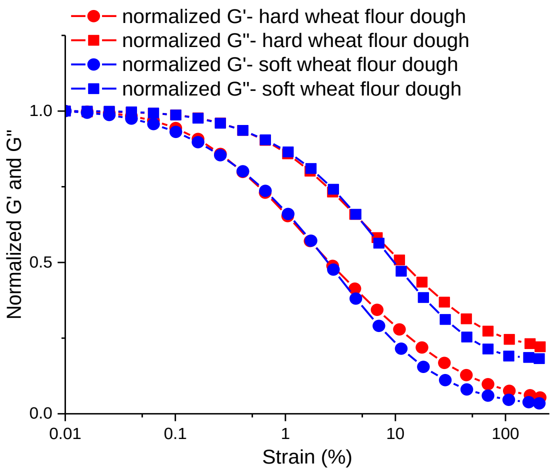

Figure 3. Normalized storage (G') and loss (G") moduli as a function of strain at 10 rad/s for soft wheat flour dough and hard wheat flour dough

Analysis of the Lissajous-Bowditch curves

Lissajous-Bowditch curves, which provide a unique rheological fingerprint of complex fluids (Ewoldt et al., 2007), are parametric plots of strain versus stress (elastic perspective) or strain rate versus stress (viscous perspective) (Macias-Rodriguez et al., 2018; Yazar et al., 2019). In this study, Lissajous-Bowditch curves were used to gain a qualitative insight into the impact of wet gluten content in wheat flours on non-linear viscoelastic properties of the resulting wheat flour doughs. Figure 4 shows the transition from linear to non-linear viscoelastic region for HRW and SRW wheat flour doughs through the elastic Lissajous-Bowditch curves at selected strain amplitudes including 0.015%, 1.5%, 25%, 70%, 110%, 200% and at low and high frequencies (1 rad/s and 20 rad/s). For both wheat flour doughs, linear viscoelasticity dominated the stress response at γ_0 = 0.015% as evidenced by the narrow elliptical trajectories for the elastic Lissajous-Bowditch curves (Figure 4a,b). At strain amplitudes $\gamma_0 \geq 1.5\%$, elliptical trajectories started to become wider gradually as the amplitude of strain increased gradually up to 200%, suggesting a transition from an elastically dominated to a viscously dominated viscoelastic behavior. These findings were concurrent with the strain sweep data indicating the onset of non-linearity to occur at strain amplitudes ranging from 0.1% to 0.25% (Figure 2a,b).

Plotting the raw data from each strain cycle as σ(t) versus γ(t) reveals additional non-linear characteristics that are obscured by $G_1^{'}$ and $G_1^{''}$ (Ewoldt et al., 2008). The clockwise rotation of the elastic Lissajous-Bowditch curves with increasing strain amplitudes was reported to be an indication of gradual softening (Ewoldt et al., 2007). When the elastic Lissajous-Bowditch curves of the wheat flour doughs were evaluated (Figure 4a,b), the loops for SRW wheat flour dough showed a higher degree of clockwise rotation at $\gamma_0 \geq 25\%$, suggesting a higher degree of softening for wheat flour doughs with lower gluten content against the increasing deformations. The softening behavior was more pronounced at 1 rad/s (Figure 4b), compared to that probed under LAOS deformations at 20 rad/s frequency (Figure 4a). This finding pointed out to a higher degree of softening for SRW wheat flour dough as the strain amplitude increased gradually at low frequencies, which supported the tanδ values (Table 1).

As the amplitude of strain increased, the distortion from the elliptical shape in the elastic Lissajous-Bowditch curves was associated with strain stiffening (Ewoldt et al., 2007). Elastic Lissajous-Bowditch curves for both wheat flour doughs (Figures 4a and 4b) started to show distortion from elliptical shape with increasing strain amplitude at $\gamma_0 > 1.5\%$ (stress upturns within a cycle), indicating intracycle strain stiffening behavior. These stress upturns were more pronounced for both wheat flour doughs when the deformation frequency was 1 rad/s (Figure 4b), which suggested a higher degree of strain stiffening behavior under large deformations with low frequency regardless of the gluten content in wheat flour doughs. On the other hand, at the highest strain amplitude (200%) and the highest frequency (20 rad/s) combination studied, the distortion from the elliptical shape was more pronounced for HRW wheat flour dough, while the elastic Lissajous-Bowditch curve was more rounded for SRW wheat flour dough (Figure 4a). This finding obtained through the elastic Lissajous-Bowditch curves revealed a higher strain stiffening behavior for wheat flour doughs with higher gluten content under high frequency-large deformation processing conditions (Figure 4a), which could be experienced during dough processing steps such as mixing, or sheeting (Dobrazsczyk and Morgenstern, 2003).

Viscous Lissajous-Bowditch curves started to show narrower elliptical trajectories for both HRW and SRW wheat flour doughs as the amplitude of strain increased (Figure 5), suggesting viscous dissipation. The increase in the viscous-like behavior with increasing strain amplitudes was more pronounced for SRW wheat flour dough (Figure 5), as evidenced by the higher degree of decrease in the area of the curves from circular trajectories to narrow ellipses when compared to the change found for HRW wheat flour dough. The change in the viscous Lissajous-Bowditch curves of SRW wheat flour and HRW wheat flour dough was more pronounced at 1 rad/s (Figure 5b) compared to that at 20 rad/s (Figure 5a). This higher degree of difference between the viscous Lissajous-Bowditch curves of the wheat flour doughs at 1 rad/s concurred with the tanδ values (Table 1) and elastic Lissajous-Bowditch curves (Figure 4a,b), indicating a higher viscous decay for wheat flour dough with lower gluten content under large deformations with low frequency.

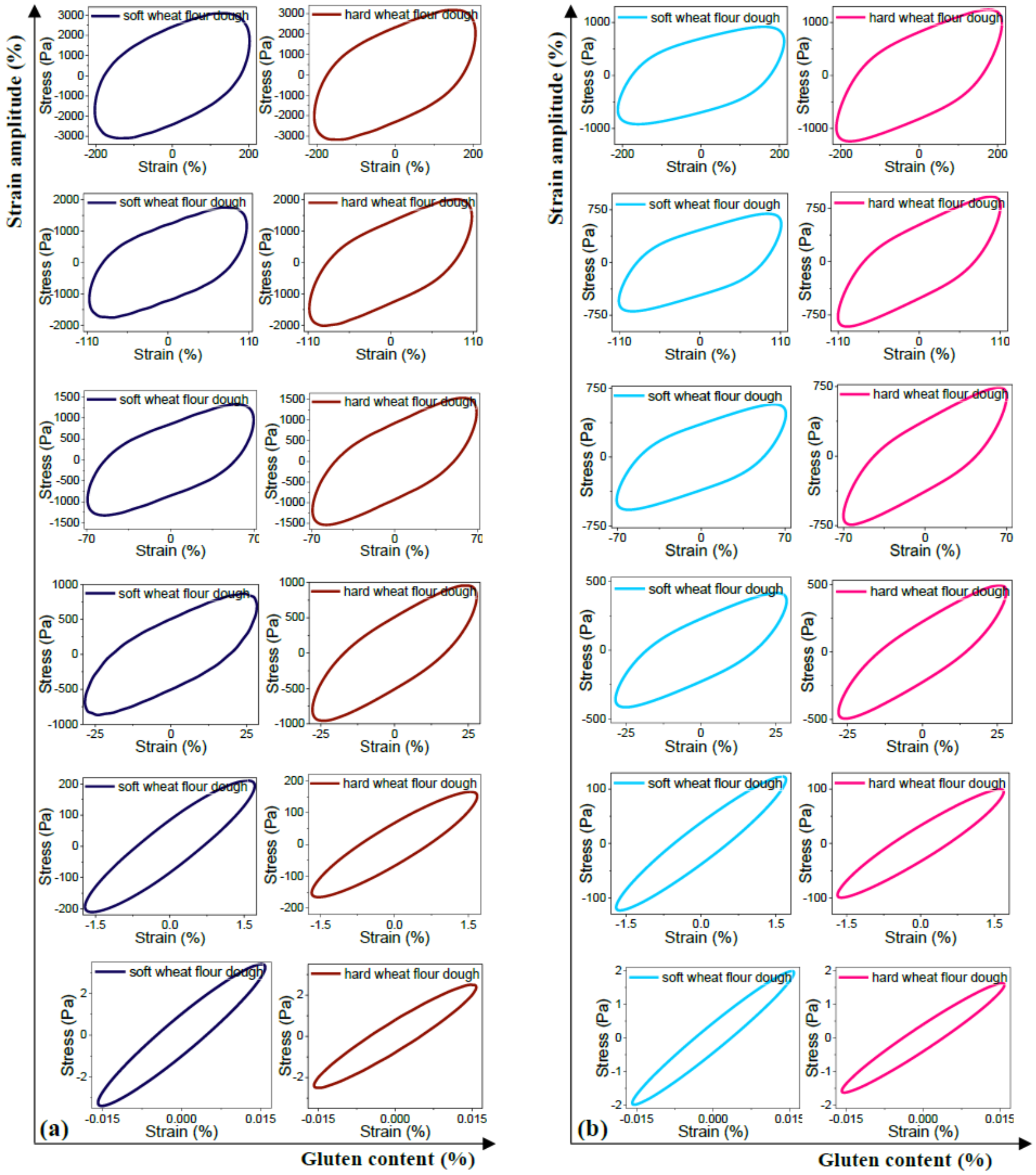

Figure 4. Raw elastic Lissajous-Bowditch curves for HRW and SRW wheat flour doughs at selected strains of 0.015%, 1.5%, 25%, 70%, 110%, 200%. Dark color indicates high frequency [(a): 20 rad/s] and light color indicates low frequency [(b): 1 rad/s].

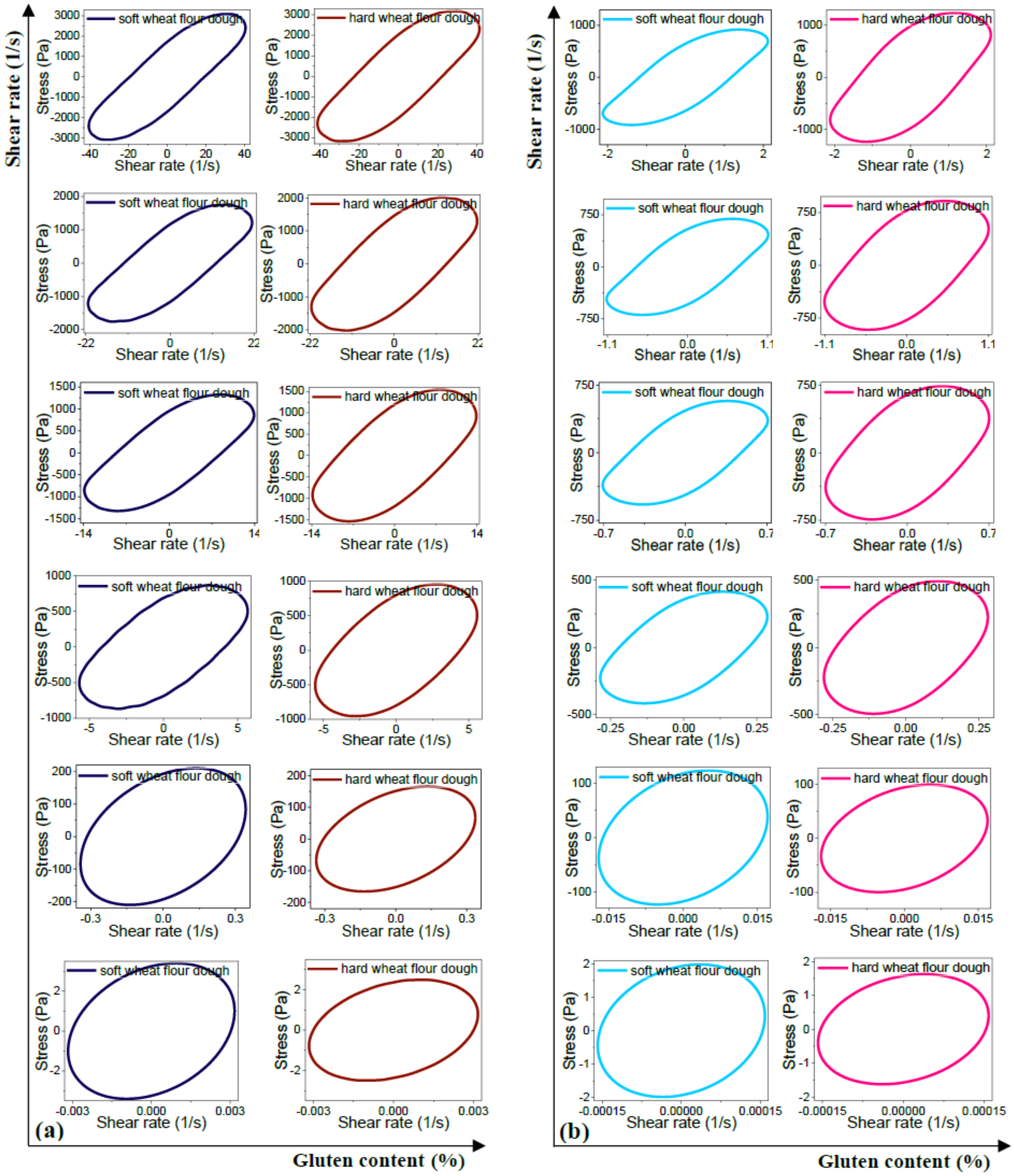

Figure 5. Raw viscous Lissajous-Bowditch curves for HRW and SRW wheat flour doughs. Shear rates correspond to strains 0.015%, 1.5%, 25%, 70%, 110%, 200%. Dark color indicates high frequency [(a): 20 rad/s] and light color indicates low frequency [(b): 1 rad/s].

LAOS parameters for wheat flour doughs as a function of gluten content

Elastic LAOS parameters

To further describe the non-linear viscoelastic properties captured by the elastic and viscous Lissajous-Bowditch curves for wheat flour doughs with different gluten content, meaningful LAOS parameters described by Ewoldt et al. (2008) were also analyzed. Non-linear elastic properties of SRW wheat flour dough and HRW wheat flour dough were shown in Figure 6a through the elastic Chebyshev coefficients. The magnitudes of the third-order elastic and viscous Chebyshev coefficients e_3 and v_3 can be used to indicate SAOS to LAOS transitions. Moreover, e_3 and v_3 can reveal the underlying causes driving the nonlinear elastic and viscous intracycle stress response (Yazar et al., 2019). The sign of the third-order elastic Chebyshev coefficient indicates if the material shows strain stiffening ($e_3 > 0$) or strain softening ($e_3 < 0$). Ultimately, the magnitude of e_3/e_1 provides a quantitative measure of the degree of non-linearity (Ewoldt et al., 2007). A positive value for the ratio of the third-order elastic Chebyshev coefficient to the first-order elastic Chebyshev coefficient ($e_3/e_1 > 0$) was associated with intracycle strain stiffening behavior (Ewoldt et al., 2008). Figure 6a indicated positive values (> 0) for the magnitudes of e_3/e_1 for both HRW and SRW wheat flour doughs, suggesting intracycle strain stiffening behavior in the non-linear region at all frequencies studied. Strain stiffening behavior for HRW wheat flour dough (Yazar et al., 2016a) and for SRW wheat flour dough (Yazar et al., 2016b) obtained at different stages of Farinograph mixing were found earlier. At the highest frequency (20 rad/s), as the amplitude of strain increased, a decrease was observed in the magnitude of the strain stiffening behavior of SRW wheat flour dough at a strain amplitude of 70%; while it occurred at around 110% for HRW wheat flour dough (Figure 6a). These critical strain amplitudes indicating a decay in the strain stiffening behaviors of wheat flour doughs emphasized the resilience of the HRW wheat flour dough against the increasing deformations due to its higher gluten content when compared to SRW wheat flour dough. At these critical strain amplitudes, the gluten network starts to weaken with increasing strain at high frequencies and the resulting mechanical energy introduced in the dough (Yazar et al., 2016b).

As the frequency of the LAOS deformation decreased to 10 rad/s, the decrease in e_3/e_1 values was observed at 110% strain for SRW wheat flour dough and at 170% strain for HRW wheat flour dough (Figure 6a). When the frequency of deformation further decreased to 1 rad/s, the magnitude of e_3/e_1 for SRW wheat flour dough started to decrease at the strain amplitude of 170%, while no decrease was observed in the strain stiffening behavior of HRW wheat flour dough up to the strain amplitude of 200% (Figure 6a). The change found in the e_3/e_1 values elucidated the impact of deformation frequency on the strain stiffening behavior of wheat flour dough (Yazar, 2023). Due to its viscoelastic nature, wheat flour dough is known to partially recover after being stretched rapidly, followed by the sudden removal of the force (Delcour and Hoseney, 2010). The gluten network might undergo bond ruptures as the amplitude of strain kept increasing at high frequencies (Yazar et al., 2016a,b), leading to a more limited recovery. On the other hand, Chebyshev coefficients also showed that the gluten network had time to stretch and reached its limit in terms of its ability to elastically deform at the lower frequencies, as evidenced by the strain amplitude where the decay in the strain stiffening behavior started to increase with the decrease in frequency (Figure 6c). In other words, the energy delivery from the applied strain at low frequencies was quite slow. Thus, gluten filaments found enough time to recreate network junctions that had been lost during stretching. And the higher rate of network junction creation than the rate of loss under low frequency LAOS deformations resulted in higher strain stiffening behavior (Yazar et al., 2019). When e_3/e_1 values of HRW and SRW wheat flour doughs were evaluated at the highest LAOS strain applied (200%), a significant difference ($P < 0.05$) was found at 20 rad/s (Figure 6a). However, the difference was not significant ($P > 0.05$) at lower frequencies (Figure 6b,c), which revealed that the contribution of gluten content in wheat flours to the strain stiffening behavior of doughs being more pronounced at high frequencies under large deformations.

Strain stiffening behavior of wheat flour dough is considered to occur due to entanglement coupling of large glutenin molecules (Sroan et al., 2009). The higher degree of strain stiffening behavior found for HRW wheat flour dough especially at high frequencies (Figure 6) is indicative of stronger entanglements in the presence of higher gluten content in wheat flours. Strain stiffening can be simply defined as the stress developed by the protein–starch matrix against the deformation resulting from the expanding gas cells (van Vliet et al., 1992; Yazar et al., 2017a). Expansion of gas cells during fermentation causes thinning of the dough film surrounding the gas cells (Sroan et al., 2009), and the extended gluten-starch matrix around the gas cells was suggested to be prevented from rupturing by the strain stiffening response (van Vliet et al., 1992; van Vliet, 2008). If the strain stiffening behavior of a dough system is above or below the optimum, a decrease is expected to occur in the loaf volume of the resulting baked product (Yazar et al., 2017a). Therefore, strain stiffening is an important phenomenon in terms of baking performance of wheat flours. The viscoelastic nature of wheat flour dough determines the degree of strain stiffening behavior under large deformations and, thus, the degree of dough expansion and loaf volume (Yazar, 2023). Gluten is the origin of the viscoelastic behavior in wheat flour doughs (Uthayakumaran et al., 2002; Yazar et al., 2017b). And the higher gluten content in HRW wheat flour compared to SRW wheat flour enabled HRW wheat flour dough to show higher degree of strain stiffening behavior against high frequency-large deformations (Figure 6a), that shed light on to the differences in the quality characteristics of baked products formulated with HRW and SRW wheat flours.

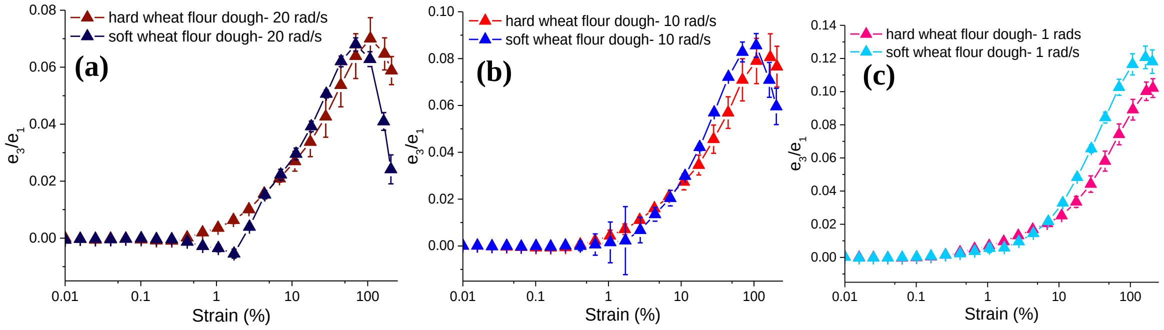

Figure 6. e_3/e_1 values for hard red winter wheat flour dough and soft red winter wheat flour dough [γ: 0.01%- 200%, ω: 20 rad/s (a), 10 rad/s (b), 1 rad/s(c)]

A constant increase in e_3/e_1 values of dough systems against the increasing LAOS strain at 10 rad/s frequency was found to deteriorate loaf volume in the resulting breads. However, an increase in e_3/e_1 followed by a decrease at larger strain amplitudes, as in the case of wheat flour doughs (Figure 6), was reported to contribute to loaf volume (Yazar et al., 2017a). The delay observed in the strain stiffening behavior of HRW wheat flour dough against the increasing strain amplitudes (Figure 6) suggested an understanding of the reason behind the decrease in the loaf volume for breads prepared with strong wheat flour with high gluten content and quality (Sapirstein et al., 2007).

Viscous LAOS parameters

In the non-linear region, a positive value for the ratio of the third-order viscous Chebyshev coefficient to the first-order ($v_3/v_1 > 0$) is associated with intracycle shear thickening, while a negative value ($v_3/v_1 < 0$) indicates intracycle shear thinning behavior (Ewoldt et al., 2008). Figure 7 shows the v_3/v_1 values for the HRW and SRW wheat flour doughs. Both wheat flour doughs showed intracycle shear thinning behavior in the non-linear region as evidenced by the negative values obtained for v_3/v_1 values (< 0) at all frequencies studied (Figure 7). Intracycle shear thinning behavior for HRW wheat flour dough (Yazar et al., 2016a) and SRW wheat flour dough (Yazar et al., 2016b) obtained at different stages of Farinograph mixing was also reported. The magnitude of intracycle shear thinning behavior in the non-linear region was higher at higher frequencies for both wheat flour doughs, as evidenced by the lower v_3/v_1 values observed at 20 rad/s (Figure 7a) compared to those at 10 rad/s (Figure 7b) and 1 rad/s (Figure 7c).

The degree of shear thinning behavior for both wheat flour doughs started to decrease once a certain strain amplitude was reached. At the highest frequency applied (20 rad/s), this decrease occurred at 110% strain amplitude (Figure 7a). As the frequency decreased to 1 rad/s (Figure 7c), the decrease in the shear thinning behaviors of wheat flour doughs was observed at the strain amplitude of 170%. A difference at this strain amplitude was found between HRW and SRW wheat flour doughs at 10 rad/s frequency (Figure 7b), where the decrease in shear thinning occurred at 110% for SRW wheat flour dough and at 170% for HRW wheat flour dough. This difference probed in the shear thinning behaviors of the tested wheat flour doughs under large deformations indicated that lower gluten content in wheat flour doughs induced shear thinning behavior at smaller deformations with high frequencies. The shear thinning behavior started to occur at larger deformations in the presence of higher gluten content in wheat flours, which was revealed for the first time in this study through the LAOS tests.

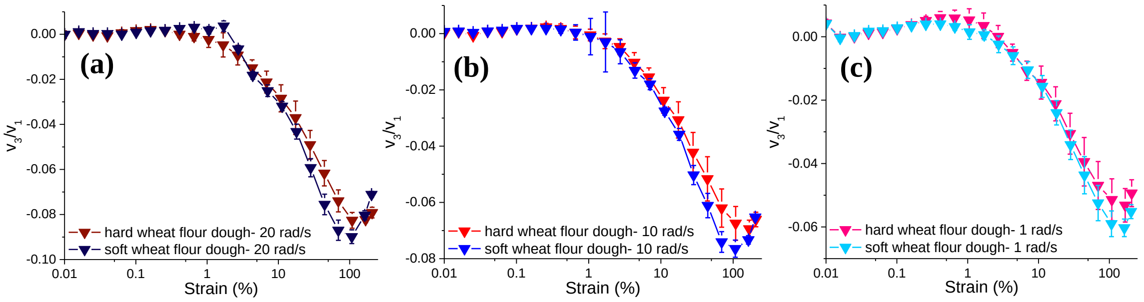

Figure 7. v_3/v_1 values for hard red winter wheat flour dough and soft red winter wheat flour dough (γ: 0.01%- 200%, ω: 1 rad/s, 10 rad/s, 20 rad/s)

Viscous LAOS properties revealed that the gluten content of wheat flours did not significantly affect the shear thinning behaviors of the resulting doughs at each frequency studied. This finding showed that the differences in the gluten content of wheat flours affected the elastic properties of the resulting doughs in the non-linear region, rather than their viscous properties. Similarly, a more pronounced non-linearity was found in the elastic component for hard wheat flour doughs obtained at different stages of mixing, where the gluten network development characteristics were different (Yazar et al., 2016a).

CONCLUSIONS

LAOS tests conducted on HRW and SRW wheat flour doughs were shown to successfully differentiate between wheat flours in terms of wet gluten content. As an empirical dough testing method, Farinograph tests indicated a stronger network for HRW wheat flour as evidenced by the higher stability, formation of double hydration peak, and higher water absorption capacity when compared to SRW wheat flour. It was possible to obtain deeper insight into the impact of gluten content on the non-linear viscoelastic properties of wheat flour doughs through the LAOS tests. In the linear viscoelastic region, tanδ values revealed similar viscoelastic network properties for both wheat flour doughs, indicating no significant impact of gluten content in flours on the linear viscoelastic properties of the resulting doughs. However, as the amplitude of strain increased into the non-linear region, differences in the viscoelastic properties of doughs resulting from the different gluten contents of the wheat flours became evident. Both wheat flour doughs showed strain stiffening and shear thinning behaviors under large deformations, which was clearly observed by the distortion in the elliptical trajectories of the elastic and viscous Lissajous-Bowditch curves. Among the elastic LAOS parameters, e_3/e_1 values were able to better differentiate between different wheat flour doughs based on their gluten contents. The higher gluten content in HRW wheat flour dough compared to SRW wheat flour dough resulted in higher degree of strain stiffening behavior for HRW wheat flour dough against high frequency-large deformations, as evidenced by the higher e_3/e_1 values at 200% strain with 20 rad/s frequency. These results highlighted the prominent influence of gluten content in wheat flours on the non-linear viscoelastic properties of the resulting doughs at high frequencies, resembling those experienced during certain dough processing steps, leading to differences in baked product quality. On the other hand, viscous LAOS parameters did not point out to a significant change in the shear thinning behaviors of HRW and SRW wheat flour doughs at each frequency studied. Elastic and viscous LAOS parameters indicated that the by the gluten content in wheat flours affected the elastic properties of wheat flour dough under large deformations more than the viscous properties. This revealed the contribution of gluten content in wheat flours to the resilience of the resulting wheat flour doughs against the increasing deformations.

This study only focused on the impact of gluten content on LAOS properties of wheat flour doughs. However, future studies should focus on the gluten quality and composition of wheat flours and discuss these results through the LAOS properties of the resulting doughs to provide a better understanding of the influence of gluten characteristics on the mechanical properties of wheat flour doughs.

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CONFLICT OF INTEREST

The author has declared no conflict of interest.

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