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Investigation of the effects of psyllium powder addition on the quality of fresh and frozen gluten-free bread

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Abstract
The interest in gluten-free (GF) products has been growing since both the increase in prevalence of celiac disease and the preferences of GF diet. In this study, the contribution of psyllium powder (PP) to gluten-free (GF) bread quality, dough rheology and volatile compounds (VCs) profile was investigated throughout the frozen storage period of GF dough (-30 °C for 0, 7, 15, and 30 days). GF doughs containing 7.5% PP (PSY1) and 15% PP (PSY2) had lower tanδ value than GF control dough ($p<0.05$) according to the results obtained from fundamental rheological analysis. Frozen storage caused no effect on the tanδ value of PSY1 and PSY2 ($p \geq 0.05$). PP addition increased the specific volume (SV) of GF breads ($p<0.05$). No significant effect of frozen storage on SV was shown for PSY2 while SV values of GF control bread (GFB) and PSY1 decreased ($p<0.05$). Lower crumb hardness was shown for PSY1 and PSY2 on day 0. Significant effect of frozen storage on crumb hardness was observed for PSY1 on day 30 while harder crumb structure was shown for GFB throughout the frozen storage ($p<0.05$). Psyllium addition led to a significant reduction in both L* value of crust and crumb color ($p<0.05$). In the VCs analysis performed by HS/GC-MS, ethanol and 1-butanol, 3-methyl from alcohol group, butanal, 3-methyl- and hexanal from aldehydes were common for GFB and GF breads containing psyllium. 1-butanol, 3-methyl-, butanal, 3-methyl- and hexanal were the VCs of PSY1 and they were also shown after frozen storage. This study suggested that quality deterioration due to frozen storage was less in gluten-free breads containing psyllium.
Keywords: Frozen storage, Gluten-free dough, Fundamental rheology, Texture, Volatile compounds

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INTRODUCTION
The function of gluten in wheat based product has been known for years and excluding gluten from a bakery formulation causes a big challenge in terms of obtaining high-quality gluten-free (GF) product (Santos et al., 2020). A cohesive, extensible and elastic dough is formed thanks to the unique properties of gluten, and in turn the gas producing during fermentation is retained in the structure (Belorio & Gómez, 2020). However, the strict adherence to a GF diet is still an effective solution for people suffering from celiac disease. Also, GF diet is becoming a worldwide trend due to some claims that GF products are healthier. Therefore, the demand for gluten-free products has increased and extensive researches have been performed to improve quality and nutritional characteristics of GF products (Fratelli et al., 2021; Mezaize et al., 2010).

GF bakery products can be formulated by GF flour obtaining from cereals (mostly maize and rice), pseudo-cereals (mostly buckwheat) and legumes starch, hydrocolloids, enzyme and protein sources (Mezaize et al., 2010). Dietary fibers and minerals can also be incorporated into formulation to ameliorate the nutritional quality of GF products (Stantiall & Serventi, 2018). The use of dietary fibers in GF bakery products also provides better physical properties as well as lower glycemic response (Fratelli et al., 2018).

Recent literatures have focused on psyllium (Plantago ovata) as it is considered as a natural bioactive fiber. Addition of psyllium fiber into a wheat-based flat dough can restrict enzyme mobility behaving like a physical barrier, thus affecting in vitro starch digestion (Güler & Sensoy, 2023). Cappa et al. (2013) investigated the effects of psyllium and sugar beet fibre in GF bread formulation and indicated that psyllium had better performance on bread development. Hydroxypropylmethylcellulose (HPMC) is commonly used hydrocolloids that improves dough development and gas retention (Ylimaki et al., 1991). In a study of Mancebo et al. (2015), HPMC, psyllium and different levels of water were optimized in GF bread formulation and no synergistic effects between both hydrocolloids observed. Fratelli et al. (2018) obtained a 33% reduction in glycemic response by using 17.14% psyllium and 117.86% water in GF bread formulation. Similar studies were also performed to examine the combination effects of chickpea and psyllium (Santos et al., 2020) and HPMC, psyllium and xanthan gum (Belorio & Gómez, 2020).

Frozen storage of bakery products has been considered as a challenge due to the disruptive effects on dough matrix and yeast activity. However, food industry uses freezing process and frozen storage for bakery products that exhibit a faster rate of staling in order to provide fresh products to consumers at any time (Leray et al., 2010; Mezaize et al., 2010). Numerous studies have been performed to reveal the effects of frozen storage on wheat dough and the improvement strategies for its quality. As for GF dough, type of hydrocolloids, freezing and frozen storage conditions were investigated in some studies.

Lorenzo et al. (2009) investigated the effect of some hydrocolloids (HPMC, xanthan and guar gums) on refrigerated and frozen non-fermented gluten-free dough, suggesting that xanthan gum exhibited the best results in terms of dough elasticity while refrigerated and frozen storage for twenty days did not affect the quality of baked dough. Leray et al. (2010) compared the different formulations of non-yeasted wheat and gluten-free bread dough and studied the effects of freezing and frozen storage conditions. This comparison study showed that wheat dough was more sensitive than gluten-free dough to frozen storage. No differences were determined in terms of elastic and viscous moduli while consistency index and flow behaviour index were reduced by the presence of a freezing step in a study of Mezaize et al. (2010) who examined the gluten-free frozen dough in terms of rheological properties. Ozkoc and Seyhun (2015) evaluated the effects of gum type and flaxseed concentration on GF breads made from frozen dough and indicated that combination of flaxseed (5%) and guar gum (1%) had better characteristics in the quality of gluten-free breads. Hayıt and Gül (2019) developed a GF formulation with quinoa for partially baked GF bread and reported that 30% quinoa flour addition led to a part-baking 45 days of frozen storage without any significant changes in sensorial properties. The using of cryoprotectants is also another way to improve the quality of frozen dough. In a recent study, 9% fructo-oligosaccharide and 31 % hydrolyzed soy protein as a potential cryoprotectant have been incorporated into GF bread and the improved specific volume and crumb texture have been obtained (de Oliveira Teotônio et al., 2021).

The use of psyllium in GF bread has been widely studied to improve the quality and nutrition content. In the present study, it was aimed to investigate the effects of psyllium in GF bread during frozen storage (-30 °C for 7, 15 and 30 days) in terms of rheological properties of doughs, quality characteristics and volatile compounds of breads.

MATERIALS AND METHODS

Materials

Gluten-free flour (Sinangil, Eksim Co., İstanbul, Türkiye), salt, sugar, compressed yeast, and oil were obtained from a local market. Gluten-free flour contains rice flour, sugar, pectin and xanthan gum as thickener, and baking powder (sodium bicarbonate and sodium acid pyrophosphate). Psyllium powder (PP) (81.1% fiber, 43% carbohydrate and 4.3% protein) was purchased from Talya Foods (Antalya, Türkiye).

Gluten-free bread making, freezing and thawing

Gluten-free bread formulation was shown in Table 1. Preliminary experiments were performed to determine the water absorption capacity in each formulation. Firstly, flour and salt were mixed in a kneader (Arzum, Türkiye) for 1 min at speed 1. Yeast and sugar were dissolved in water at room temperature, and then they were added into flour-salt mixture. After mixing for 1 min at speed 1, oil was added. Final mixing was performed for 3 min at speed 1 and 7 min at speed 2. 150 g of dough was placed on a baking pan, fermented for 1 hour at 30 °C and 85% relative humidity (Nuve TK 252, Ankara, Türkiye). Frozen storage (-30 °C for 7, 15 and 30 days) was performed for frozen dough samples. After storage, they were thawed at 30 °C for 1 hour. The baking conditions were at 190 °C bottom temperature and 230 °C top temperature for 40 min (Maksan MKF-4P, Türkiye).

Table 1. Gluten-Free Bread Formulations.

Bread Type	Gluten-Free Flour Mix (g)	Psyllium (g)	Salt; Sugar; Yeast; Liquid Oil (g)	Water (g)
GFB	300	0	4.5 ; 7.5 ; 12 ; 12	300
PSY1	277.5	22.5	4.5 ; 7.5 ; 12 ; 12	450
PSY2	255	45	4.5 ; 7.5 ; 12 ; 12	570

GFB: gluten-free control bread
PSY1: gluten-free bread containing 7.5% PP
PSY2: gluten-free bread containing 15% PP

Fundamental rheological analysis

A frequency sweep test (between 0.1 rad/s and 100 rad/s at 20 °C) was conducted to determine the rheological properties of the GF dough samples. A constant strain of 0.1% within linear viscoleastic region was applied. Antonpaar MCR 302 rheometer (Graz, Austria) was used with a PP50 probe by placing GF dough (~2 g) on the parallel plate with a 2 mm gap. The storage modulus (G′), loss modulus (G″) and tanδ (G″/ G′) values were obtained by triplicate measurements.

Determination of quality characteristics of gluten-free breads

The quality characteristics of samples (specific volume, texture and color) were performed after cooling for 2 h.

Specific volume

The volume of bread was determined according to the rapeseed displacement (AACC Method 10-05.01, 2000). The volume of bread (mL) was divided by the weight (g) of bread to calculate the specific volume (mL/g).
Two breads from each formulation were subjected to volume measurement and the means were calculated.

Texture

The bread samples were divided into two 12.5 mm slices for texture profile analysis (Stable Micro Systems TA.XT2 Plus, UK). A 36 mm cylindrical probe was used in the analysis, and a force of 50 N was applied to the samples at a speed of 55 mm/min. Hardness, springeness, cohesiveness, chewiness, and resilience parameters were evaluated. Triplicate measurements were performed.

Color

The lightness (L*), redness (a*) and yellowness (b*) parameters of the crust and crumb were determined by using a Chromameter (CR-100 Konica Minolta, Tokyo, Japan). The following equation was used to calculate the color differences (ΔE):

$$\Delta E = [(L^*- L_0^*)^2 + (a^*-a_0^*)^2 + (b^*-b_0^*)^2]^{1/2}$$

L^*_0, a^*_0, and b^*_0 are the values of fresh breads (day 0) while L*, a*, and b* values correspond to the days of frozen storage (7th, 15th and 30th). Three measurements were performed for crust color of each bread. Crumb color was determined by two measurements for each type of bread.

Determination of volatile compounds

The GF bread crumb (~3 g for each bread type) was subjected to HS/GC-MS analyses (GCMS-QP2010, Shimadzu, Milan, Italy) to determine the profile of volatile compounds (VCs). The details of the method was previously stated in the studies of Rzepa et al. (2009), Tulukcu et al. (2019) and Ozulku (2024). The detected volatile compounds showing 70% similarities with the commercial mass spectra libraries (NIST27 and WILEY7) were evaluated to exhibit VCs profile.

Statistical Analysis

One-way analysis of variance (ANOVA) followed by post hoc Tukey test was used to determine the difference between groups at 95% significance level with IBM SPSS Statistics 25 package program (Chicago, IL, USA).

RESULTS AND DISCUSSION

Rheological properties

The content and structure of ingredients significantly affect the rheological properties of GF dough.Therefore, rheological measurements have been widely performed to design the GF formulation (Yazar & Demirkesen, 2023). The effect of psyllium addition and frozen storage on the storage modulus (G′) and loss modulus (G″) of GF dough samples were presented in Figure 1., where G′ reflects the elasticity of dough, while G″ reflects the viscosity of dough. In this study, GF control bread (GFB) dough exhibited a lower G′ than the GF dough containing 7.5% PP (PSY1) and 15.0% PP (PSY2). PP addition into GF dough led an increase in G′ and a decrease in G″ on day 0 (Figure 1). In a study of Mancebo et al. (2015), psyllium addition (2-4%) increased the G′, particularly with reduced dough water levels. This was attributed to presence of ingredients with water-binding ability (Matos & Rosell, 2013). Also, a solid elastic-like behavior was shown in all GF dough samples since G′ was greater than G″ as stated in many studies (Lorenzo et al., 2009; Mancebo et al., 2015; Mezaize et al., 2010).

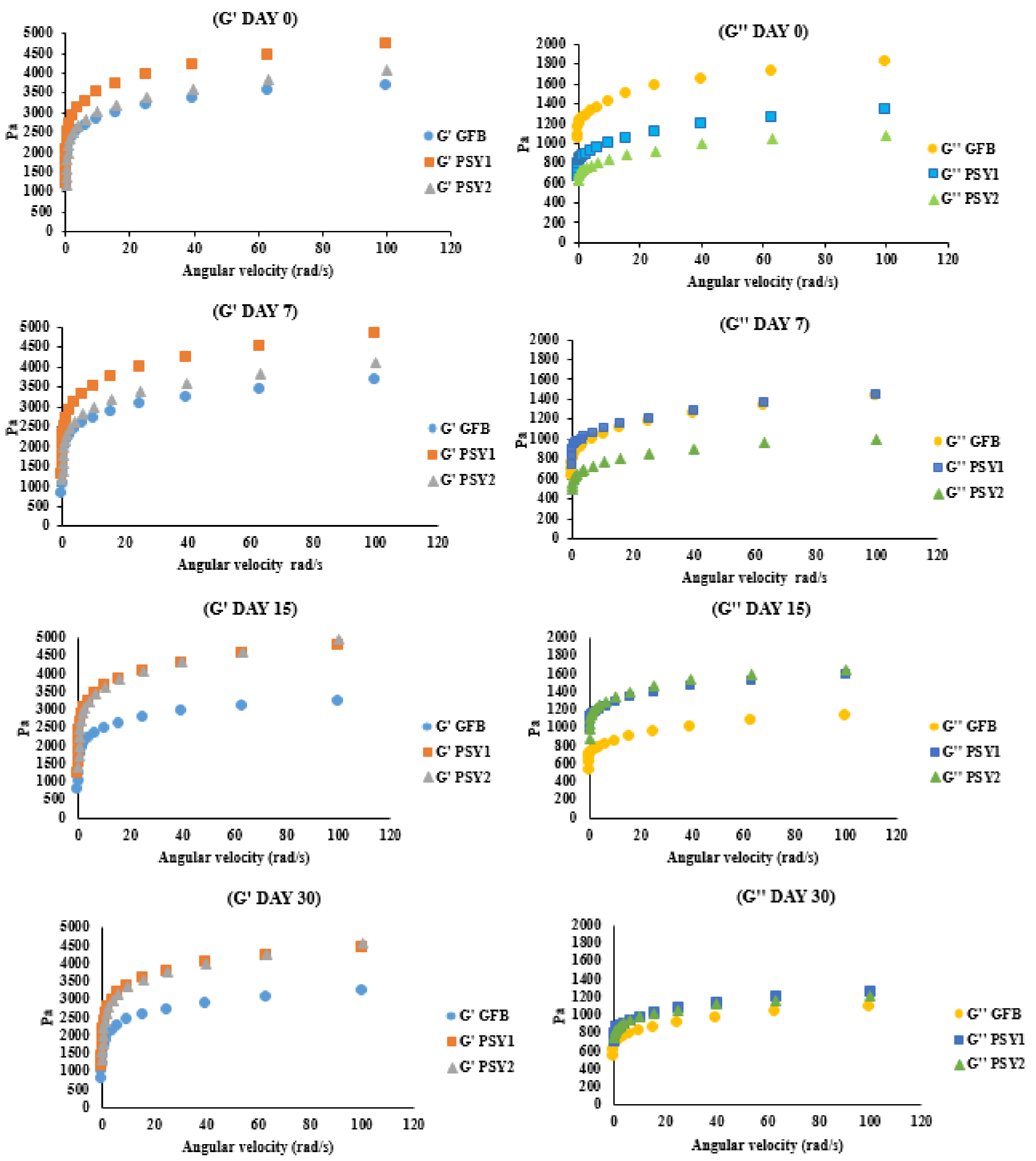

Figure 1. Rheological properties of gluten-free breads during frozen storage.
GFB : gluten-free control bread; PSY1: gluten-free bread containing 7.5% PP; PSY2: gluten-free bread containing 15% PP

The ratio of G″ to G′ (G″/G′) is corresponds to tanδ which shows the degree of dough viscoelasticity (Lu et al., 2023). Figure 2 shows the loss tangent (tanδ) value of all dough samples. The tanδ value was obtained lower than 1, suggesting that elastic behavior is more dominant than viscous behavior. PSY1 and PSY2 had lower tanδ values when compared to GFB dough ($p<0.05$, Figure 2). Mancebo et al. (2015) also reported that psyllium decreased the tanδ parameter. Frozen storage caused a significant reduction in tanδ value of GFB dough, but no significant differences were observed between 15 days and 30 days of frozen storage. Leray et al. (2010) reported that no significant effect of 28 days of frozen storage on the tanδ value of GF reference dough containing corn flour, starch, hydrocolloids (HPMC, guar gum, highly methylated pectin), etc. Frozen storage caused no effect on the tanδ value of PSY1 and PSY2 ($p>0.05$, Figure 2). Addition of psyllium decreased the impact of frozen storage when compared to GFB dough due to its high amount fiber.

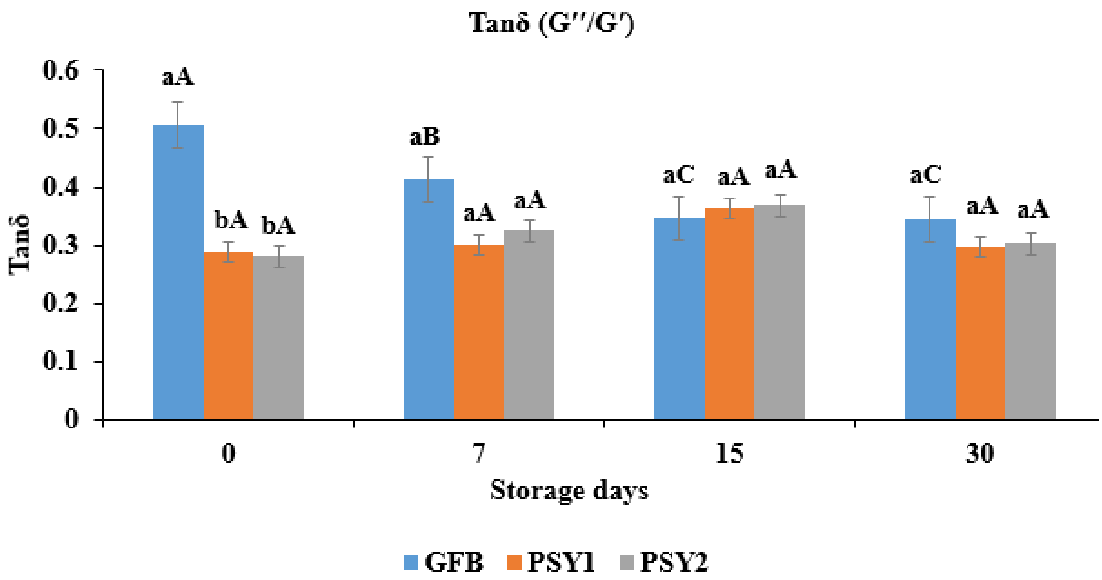

Figure 2. The changes of tanδ values of gluten-free breads during frozen storage.
Different uppercase letters show significant differences during the frozen storage of same bread type ($p < 0.05$). Different lowercase letters show significant differences between bread types for the same frozen storage time ($p < 0.05$). GFB : gluten-free control bread; PSY1: gluten-free bread containing 7.5% PP; PSY2: gluten-free bread containing 15% PP

Quality Characteristics of GF breads

Specific volume and texture

Specific volume (SV) and crumb properties are the main criteria to evaluate bread quality. Table 2 shows SVs and textural characteristics of GF bread samples. Highest SV values were obtained in PSY1 and PSY2 on day 0. Psyllium addition increased SV when compared to control formulation, similar with the study of Fratelli et al. (2021). However, some studies reported that psyllium didn't improve the volume of GF bread (Mancebo et al., 2015; Santos et al., 2020). But, it was suggested that the hydration level and amount of psyllium in the formulation were important in order to be shown the effect of psyllium on GF bread quality (Fratelli et al., 2018; Santos et al., 2020). No significant effect of frozen storage on SV was shown for PSY2 while SV values of GFB and PSY1 decreased ($p<0.05$). The bread SV is inversely correlated with crumb hardness as stated in many studies (Capriles and Arêas, 2014; Gallagher et al., 2004; Sabanis et al., 2009). Therefore, lower crumb hardness was shown for PSY1 and PSY2 on day 0. Fratelli et al. (2021) also observed lower crumb firmness value with the addition of psyllium when compared to control. This has been explained that interactions between psyllium and water enhanced the formation of gel network, and in turn improved the gas retention capacity during baking, volume and texture of GF bread (Fratelli et al., 2018). Moreover, water adjustment was recommended for fibre-enriched GF formulations to achieve optimum dough viscosity and bread characteristics (Capriles et al., 2021; Conte et al., 2019). Harder crumb structure was shown for GFB throughout the frozen storage. Significant effect of frozen storage on crumb hardness was observed for PSY1 on day 30 while it was on day 7 for PSY2. Springeness and cohesiveness values of GF breads weren't significantly different on day 0. Frozen storage had no effect on springeness, cohesiveness and chewiness values of GFB sample, but its resilience value increased on storage day 15 (Table 2). Resilience value is a kind of indicator of crumb elasticity and a reduction of this value is associated with loss of elasticity (Onyango et al., 2011). Resilience value of PSY2 remained stable throughout the frozen storage while there was a significant reduction in resilience value of PSY1 at the end of frozen storage. Chewiness value which is defined as energy to masticate a solid food was influenced by frozen storage of PSY2 (Table 2). 15 days of frozen storage was better in terms hardness, chewiness and resilience value of PSY1 and PSY2 while 7 days of frozen storage is more suitable for GFB due to its sharp increase in hardness value.

Table 2. Specific volume and textural characteristics of gluten-free breads

	Frozen Storage Times (Days)	BREAD TYPES		
		GFB	PSY1	PSY2
Specific Volume (mL/g)	0	2.46±0.01^{bA}	3.28±0.01^{aA}	3.10±0.17^{aA}
	7	2.29±0.08^{bAB}	2.93±0.06^{aB}	2.83±0.22^{abA}
	15	2.13±0.01^{cB}	2.95±0.01^{aB}	2.62±0.001^{bA}
	30	2.27±0.01^{bB}	2.73±0.003^{aC}	2.51±0.17^{abA}
Hardness (N)	0	4.34±0.48^{aB}	2.57±0.67^{bB}	1.78±0.54^{bC}
	7	6.56±1.16^{aA}	3.91±0.55^{bAB}	3.17±0.48^{bB}
	15	6.25±0.38^{aA}	3.13±0.47^{bB}	3.22±0.2^{bB}
	30	6.53±0.44^{aA}	5.49±1.03^{aA}	5.2±0.62^{aA}
Springeness	0	0.98±0.05^{aA}	0.99±0.01^{aA}	0.99±0.02^{aA}
	7	0.97±0.02^{aA}	0.98±0.01^{aA}	0.98±0.01^{abA}
	15	0.99±0.05^{aA}	0.99±0.01^{aA}	0.99±0.003^{aA}
	30	0.96±0.01^{aA}	0.99±0.16^{aA}	0.97±0.01^{aA}
Cohesiveness	0	0.8±0.01^{aA}	0.85±0.03^{aA}	0.84±0.04^{aA}
	7	0.79±0.02^{aA}	0.8±0.03^{aB}	0.84±0.02^{aA}
	15	0.81±0.01^{aA}	0.84±0.03^{aAB}	0.82±0.01^{aA}
	30	0.8±0.02^{aA}	0.83±0.03^{aAB}	0.82±0.03^{aA}
Chewiness (N)	0	3.42±0.36^{aA}	2.96±0.28^{aA}	1.83±0.34^{bC}
	7	6.87±3.71^{aA}	3.54±2.06^{aA}	2.59±0.37^{bB}
	15	4.98±0.69^{aA}	3.25±0.44^{bA}	2.93±0.5^{bB}
	30	5.00±0.34^{aA}	4.28±0.81^{aA}	4.12±0.49^{aA}
Resilience	0	0.47±0.01^{bBC}	0.53±0.02^{aA}	0.48±0.01^{bA}
	7	0.45±0.01^{cC}	0.54±0.02^{aA}	0.51±0.01^{bA}
	15	0.52±0.01^{aA}	0.52±0.01^{aA}	0.5±0.01^{aA}
	30	0.49±0.02^{aAB}	0.47±0.02^{aB}	0.48±0.03^{aA}

Different uppercase letters show significant differences during the frozen storage of same bread type ($p < 0.05$). Different lowercase letters show significant differences between bread types for the same frozen storage time ($p < 0.05$). GFB : gluten-free control bread; PSY1: gluten-free bread containing 7.5% PP; PSY2: gluten-free bread containing 15% PP

Color

The color is another criterion that affects the consumer preferences. The water content, pH and presence of reducing sugars and amino acid in dough formulation besides relative humidity, temperature, mode of heat transfer as baking conditions determine the color characteristics of bakery products. GF bakery products often present lighter color than control bread. The dietary fibers used in GF formulations generally provides better color properties (Arslan et al., 2019). The crust and crumb color characteristics of GF breads were shown in Table 3. Psyllium addition led to a significant reduction in both L* value of crust and crumb color. The increasing amount of psyllium in GF breads also caused a darkening effect on crumbs as seen in Figure 3. These darker characteristics of PSY1 and PSY2 than GFB were probably due to the brownish color of psyllium (Fratelli et al., 2018). A significant impact of frozen storage on wheat bread crust color was reported by Sharadanant and Khan (2003), which reducing the crust L* value. In the present study, frozen storage caused no regular changes in L*, a* and b* values of all GF breads (Table 3). However, total color changes (ΔE) in the crust of GFB increased significantly throughout the frozen storage ($p<0.05$). Also, PSY1 and PSY2 breads showed higher ΔE values on day 15 and 30 of frozen storage.

Table 3. Color properties of gluten free breads

Bread type	Storage time (days)	Crust				Crumb			
		L*	a*	b*	ΔE	L*	a*	b*	ΔE
GFB	0	54.81±1.59^{aB}	11.48±1.35^{aA}	29.13±2.47^{aB}		76.93±2.54^{aA}	-1.73±0.11^{bA}	11.16±1.12^{aA}	
	7	56.89±0.5^{aB}	10.44±1.04^{aA}	31.87±0.88^{aA}	3.59±1.96^{bC}	71.09±1.08^{aB}	-2.28±0.09^{cC}	8.15±0.43^{aB}	6.84±1.62^{aC}
	15	68.38±2.97^{aA}	3.68±1.04^{cB}	17.55±1.53^{aC}	19.47±1.70^{aB}	64.68±0.4^{aC}	-1.79±0.08^{cB}	6.23±0.37^{aC}	13.41±2.28^{aA}
	30	43.38±1.14^{bC}	10.37±0.47^{aA}	12.66±0.7^{aD}	20.08±2.03^{aA}	71.24±1.71^{aB}	-1.9±0.09^{bB}	7.11±0.94^{aAB}	7.29±0.85^{aB}
PSY1	0	46.52±2.17^{bA}	10.87±0.99^{aA}	17.73±1.18^{bA}		61.97±1.57^{bA}	5.63±0.29^{aA}	6.11±0.78^{bA}	
	7	43.23±1.93^{bB}	11.26±0.44^{aA}	18.27±1.48^{bA}	3.35±0.67^{cC}	56.57±0.5^{bB}	5.64±0.19^{bA}	1.97±0.26^{bB}	6.81±1.19^{aB}
	15	38.94±1.04^{cC}	8.65±0.96^{aB}	10.43±0.5^{bB}	10.76±1.32^{cA}	52.8±0.9^{bC}	4.87±0.25^{bB}	1.68±0.14^{bB}	10.21±0.92^{cA}
	30	42.7±1.07^{bC}	8.17±0.38^{bB}	9.65±0.57^{bB}	9.34±1.40^{cB}	57.67±1.76^{bB}	5.71±0.18^{aA}	1.32±0.28^{bB}	6.44±0.55^{bC}
PSY2	0	35.31±0.63^{cC}	8.25±0.37^{bB}	7.96±0.66^{cBC}		50.87±1.26^{cA}	5.7±0.37^{aB}	4.09±0.17^{cA}	
	7	43.56±0.33^{bB}	8.62±0.41^{bA}	10.07±0.31^{cA}	8.52±0.47^{aC}	49.71±1.5^{cA}	6.62±0.24^{aA}	-0.56±0.33^{cC}	4.88±0.31^{bB}
	15	47.01±0.9^{bA}	5.32±0.22^{bC}	7.24±0.27^{cC}	12.08±0.5^{bA}	41.63±0.93^{cB}	5.28±0.06^{aB}	-0.5±0.28^{cC}	10.33±0.47^{bA}
	30	45.6±2.00^{aA}	7.89±0.52^{bB}	9.10±1.66^{bAB}	10.36±0.47^{bB}	49±2.65^{cA}	5.76±0.59^{aB}	0.09±0.41^{cB}	4.41±1.42^{cC}

Different uppercase letters show significant differences during frozen storage of same bread type ($p<0.05$). Different lowercase letters show significant differences between bread types on the same frozen storage time ($p<0.05$). GFB : gluten-free control bread; PSY1: gluten-free bread containing 7.5% PP; PSY2: gluten-free bread containing 15% PP

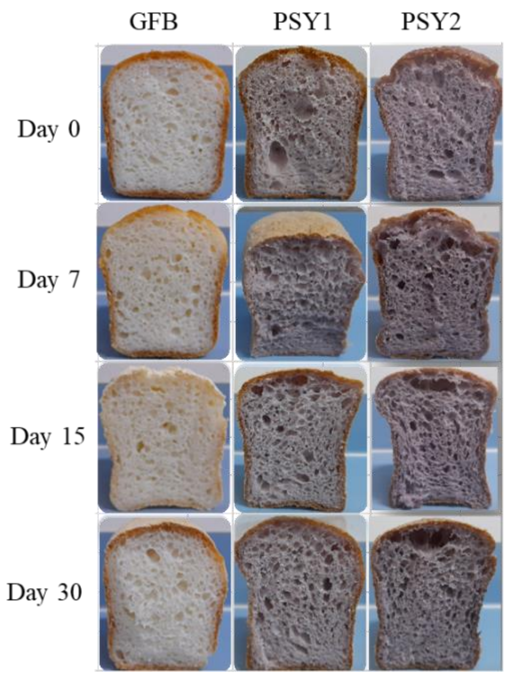

Figure 3. Images of gluten-free breads during frozen storage. GFB : gluten-free control bread; PSY1: gluten-free bread containing 7.5% PP; PSY2: gluten-free bread containing 15% PP

Volatile compounds

The final aroma of bread is also important for the choice of consumers. The type of flour or flour mixtures used in GF formulations are the primary factors that influence the aroma characteristics. Both the effects of psyllium addition and frozen storage were evaluated and presented in Table 4. GFB contained more volatile compounds (VCs) when compared other GF breads. This maybe explained by the compositional characteristics of psyllium affected the generation of VCs from Maillard reaction. The amino acid profiles and sugar types of added ingredients determine the rate of Maillard reaction (Pico et al., 2019). Psyllium is a kind of dietary fibre consisting primarily of highly branched arabinoxylans, composing of arabinose 22.6%, xylose 74.6%, only traces of other sugars with about 35% of non-reducing terminal residues. This profile can cause a lower extent of Maillard reactions and less detected VCs than GFB (Singh, 2007; Pico et al., 2019). Ethanol and 1-butanol, 3-methyl- from alcohol group, butanal, 3-methyl- and hexanal from aldehydes were common for GFB and GF breads containing psyllium (Table 4). Butanal, 3-methyl- and hexanal have been described apple-like and fruit-like, respectively (Öncel, 2023; Pico et al., 2017). 2-Propanol, 1-methoxy-, which gives sweet ether-like aroma, was only found in GFB and not detected after frozen storage. 2,3-pentanedione, butanal, 2-methyl-, 3-hepten-2-one, 2-propen-1-one,1-cyclopropyl-, and formic acid, ethenyl ester were the VCs of GFB which were not found after frozen storage (Table 4). 2,3-pentanedione and butanal, 2-methyl- have been reported as pleasant descriptors like caramel and fruity-almond, respectively (Pico et al., 2017). 1-butanol, 3-methyl-, butanal, 3-methyl- and hexanal were the VCs of PSY1 and they were also shown after frozen storage (Table 4). 1-butanol, 3-methyl-, butanal, 3-methyl- and hexanal have been known to exhibit organoleptic characteristics such as balsamic-alcohol, apple-like and green grass, respectively (Pico et al., 2017). Butanal, 3-methyl- was the preserved VC of PSY2 after frozen storage. A change of VCs intensity during frozen storage was reported in a study of He et al. (2023). Therefore, freezing treatment can cause both decrease and increase in signal intensities of some VCs by affecting the aromatic compounds in the dough by the yeast cells and the interaction of dough components with VCs (He et al., 2023).

Table 4. Volatile compounds of gluten- free breads during frozen storage

		Bread type											
		GFB				PSY1				PSY2			
		Day 0	Day 7	Day 15	Day30	Day 0	Day 7	Day 15	Day 30	Day 0	Day 7	Day 15	Day 30
Alcohol	Ethanol	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
	1-Butanol, 3-methyl-	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓	
	1-Hexanol		✓	✓							✓		
	2-Propanol, 1-methoxy-	✓											
	1,3-Butanediol		✓										
Aldehydes	Butanal, 3-methyl-	✓	✓		✓	✓	✓	✓	✓	✓	✓	✓	✓
	Butanal, 2-methyl-	✓						✓	✓	✓		✓	✓
	Hexanal	✓	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓
	Pentanal			✓				✓					✓
	Heptanal		✓	✓	✓	✓	✓						
	Octanal				✓								
	Nonanal		✓	✓	✓								
	2-heptenal			✓									
	Valeraldehyde			✓									
Ketones	2,3-Pentanedione	✓						✓		✓			
	Acetoin	✓		✓	✓			✓					
	3-Hepten-2-one	✓											
	2-Propen-1-one,1-cyclopropyl-	✓											
Esters	Formic acid, 2-propenyl ester				✓	✓	✓						
	Formic acid, ethenyl ester	✓											
Furans	Furan, 2 pentyl-												
	Furan<2-amyl>		✓	✓	✓	✓		✓	✓				
Terpenes	dl-Limonene		✓	✓				✓	✓				

GFB : gluten-free control bread; PSY1: gluten-free bread containing 7.5% PP; PSY2: gluten-free bread containing 15% PP

CONCLUSION

In this study, the improvement effects of psyllium addition into gluten-free bread were observed as stated in previous studies. Quality deterioration in terms specific volume reduction and crumb hardness increments due to frozen storage was less in gluten-free breads containing psyllium than gluten-free control bread. Especially, the gluten-free bread with 7.5% psyllium showed no significant differences in terms of crumb hardness throughout 15 days of frozen storage. The increase in crumb hardness due to frozen storage was more notable on day 7 of frozen storage for gluten-free bread with 15% psyllium when compared to the 7.5% psyllium addition level.Therefore, 7.5% addition level and the frozen storage for 15 days were more suitable. According to the results of volatile compounds analysis, gluten-free control bread exhibited more volatile compounds than the breads containing psyllium. Nevertheless, further studies are required in order to reveal the effects of both psyllium addition and frozen storage on volatile compounds of gluten-free breads.

Compliance with Ethical Standards

Peer-review

Externally peer-reviewed.

Declaration of Interests

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Author contribution

This study is the part of the master thesis of SCK. She (SCK) conducted experiments, data analysis. GÖ supervised SCK and wrote the manuscript. All the authors reviewed the manuscript.

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REFERENCES

AACC. (2000). Approved Methods of AACC, 10th ed.; American Association of Cereal Chemists: St. Paul, MN, USA.

Arslan, M., Rakha, A., Xiaobo, Z., & Mahmood, M. A. (2019). Complimenting gluten free bakery products with dietary fiber: Opportunities and constraints. Trends in Food Science & Technology, 83, 194-202. https://doi.org/10.1016/j.tifs.2018.11.011

Belorio, M., & Gómez, M. (2020). Effect of hydration on gluten-free breads made with hydroxypropyl methylcellulose in comparison with psyllium and xanthan gum. Foods, 9(11), 1548. https://doi.org/10.3390/foods9111548

Cappa, C., Lucisano, M., & Mariotti, M. (2013). Influence of Psyllium, sugar beet fibre and water on gluten-free dough properties and bread quality. Carbohydrate Polymers, 98(2), 1657-1666. https://doi.org/10.1016/j.carbpol.2013.08.007

Capriles, V. D., Santos, F. G., & Aguiar, E. V. (2021). Innovative gluten-free breadmaking. Trends in Wheat and Bread Making, 371-404. https://doi.org/10.1016/B978-0-12-821048-2.00013-1

Conte, P., Fadda, C., Drabińska, N., & Krupa-Kozak, U. (2019). Technological and nutritional challenges, and novelty in gluten-free breadmaking-a review. Polish Journal of Food and Nutrition Sciences, 69(1). 10.31883/pjfns-2019-0005

de Oliveira Teotônio, D., da Costa, B. A. F., Gomes, P. T. G., Santos, M. P., Amaral, E. F. G., Clerici, M. T. P. S., Leoro, M. G. V., & Schmiele, M. (2021). Fructo-oligosaccharides, hydrolyzed soy protein and yeast (*Saccharomyces* sp.) extract as potential cryoprotectans in gluten-free frozen dough and bread quality. Research, Society and Development, 10(3). https://doi.org/10.33448/rsd-v10i3.13556

Fratelli, C., Muniz, D. G., Santos, F. G., & Capriles, V. D. (2018). Modelling the effects of psyllium and water in gluten-free bread: An approach to improve the bread quality and glycemic response. Journal of Functional Foods, 42, 339-345. https://doi.org/10.1016/j.jff.2018.01.015

Fratelli, C., Santos, F. G., Muniz, D. G., Habu, S., Braga, A. R. C., & Capriles, V. D. (2021). Psyllium improves the quality and shelf life of gluten-free bread. Foods, 10(5), 954. https://doi.org/10.3390/foods10050954

Gallagher, E., Gormley, T. R., & Arendt, E. K. (2004). Recent advances in the formulation of gluten-free cereal-based products. Trends in Food Science & Technology, 15(3-4), 143-152. https://doi.org/10.1016/j.tifs.2003.09.012

Güler, N., & Sensoy, I. (2023). The effect of psyllium fiber on the in vitro starch digestion of steamed and roasted wheat based dough. Food research international, 168, 112797. https://doi.org/10.1016/j.foodres.2023.112797

Hayıt, F., & Gül, H. (2019). Kinoa ununun ve kısmi pişirilerek dondurma yönteminin glutensiz ekmek kalitesi üzerine etkisi. The Black Sea Journal of Sciences, 9(2), 406-427 (in Turkish). https://doi.org/10.31466/kfbd.647249

He, N., Xia, M., Zhang, X., He, M., Li, L., & Li, B. (2024). Quality attributes and functional properties of whole wheat bread baked from frozen dough with the addition of enzymes and hydrocolloids. Journal of the Science of Food and Agriculture, 104(4), 1928-1941. https://doi.org/10.1002/jsfa.13077

Leray, G., Oliete, B., Mezaize, S., Chevallier, S., & de Lamballerie, M. (2010). Effects of freezing and frozen storage conditions on the rheological properties of different formulations of non-yeasted wheat and gluten-free bread dough. Journal of Food Engineering, 100(1), 70-76. https://doi.org/10.1016/j.jfoodeng.2010.03.029

Lorenzo, G., Zaritzky, N. E., & Califano, A. N. (2009). Rheological characterization of refrigerated and frozen non-fermented gluten-free dough: Effect of hydrocolloids and lipid phase. Journal of Cereal Science, 50(2), 255-261. https://doi.org/10.1016/j.jcs.2009.06.003

Lu, P., Guo, J., Fan, J., Wang, P., & Yan, X. (2023). Combined effect of konjac glucomannan addition and ultrasound treatment on the physical and physicochemical properties of frozen dough. Food Chemistry, 411, 135516. https://doi.org/10.1016/j.foodchem.2023.135516

Mancebo, C. M., San Miguel, M. Á., Martínez, M. M., & Gómez, M. (2015). Optimisation of rheological properties of gluten-free doughs with HPMC, psyllium and different levels of water. Journal of Cereal Science, 61, 8-15. https://doi.org/10.1016/j.jcs.2014.10.005

Matos, M. E., & Rosell, C. M. (2013). Quality indicators of rice-based gluten-free bread-like products: relationships between dough rheology and quality characteristics. Food and Bioprocess Technology, 6, 2331-2341. https://doi.org/ 10.1007/s11947-012-0903-9

Mezaize, S., Chevallier, S., Le-Bail, A., & De Lamballerie, M. (2010). Gluten-free frozen dough: Influence of freezing on dough rheological properties and bread quality. Food Research International, 43(8), 2186-2192. https://doi.org/10.1016/j.foodres.2010.07.030

Onyango, C., Mutungi, C., Unbehend, G., & Lindhauer, M. G. (2011). Modification of gluten-free sorghum batter and bread using maize, potato, cassava or rice starch. LWT-Food Science and Technology, 44(3), 681-686. https://doi.org/10.1016/j.lwt.2010.09.006

Ozkoc, S. O., & Seyhun, N. (2015). Effect of gum type and flaxseed concentration on quality of gluten-free breads made from frozen dough baked in infrared-microwave combination oven. Food and Bioprocess Technology, 8, 2500-2506. https://doi.org/ 10.1007/s11947-015-1615-8

Ozulku, G. (2024). Monitoring the Dough Properties, Quality Characteristics and Volatile Compounds of Whole Wheat Bread Made by Different Sourdough Types during Frozen Storage. Foods, 13(9), 1388. https://doi.org/10.3390/foods13091388

Öncel, B. (2023). Determination of the aroma profile and bioactive properties of gluten-free bread with legumes flour. Çukurova University, Graduate School of Science, Department of Food Engineering, PhD Thesis, Adana, Turkiye, 276 pp.

Pico, J., Hansen, Å. S., & Petersen, M. A. (2017). Comparison of the volatile profiles of the crumb of gluten-free breads by DHE-GC/MS. Journal of Cereal Science, 76, 280-288. https://doi.org/10.1016/j.jcs.2017.07.004

Pico, J., Antolín, B., Román, L., Bernal, J., & Gómez, M. (2019). Selection of the most suitable mixture of flours and starches for the improvement of gluten-free breads through their volatile profiles. European Food Research and Technology, 245, 1755-1766. https://doi.org/10.1007/s00217-019-03279-z

Rzepa, J., Wojtal, Ł., Staszek, D., Grygierczyk, G., Labe, K., Hajnos, M., Kowalska, T., & Waksmundzka-Hajnos, M. (2009). Fingerprint of selected Salvia species by HS-GC-MS analysis of their volatile fraction. Journal of Chromatographic Science, 47(7), 575-580. https://doi.org/10.1093/chromsci/47.7.575

Sabanis, D., Lebesi, D., & Tzia, C. (2009). Effect of dietary fibre enrichment on selected properties of gluten-free bread. LWT-Food Science and Technology, 42(8), 1380-1389. https://doi.org/10.1016/j.lwt.2009.03.010

Santos, F. G., Aguiar, E. V., Centeno, A. C. L., Rosell, C. M., & Capriles, V. D. (2020). Effect of added psyllium and food enzymes on quality attributes and shelf life of chickpea-based gluten-free bread. LWT, 134, 110025. https://doi.org/10.1016/j.lwt.2020.110025

Sharadanant, R., & Khan, K. (2003). Effect of hydrophilic gums on the quality of frozen dough: II. Bread characteristics. Cereal Chemistry, 80(6), 773-780. https://doi.org/10.1094/CCHEM.2003.80.6.773

Singh, B. (2007). Psyllium as therapeutic and drug delivery agent. International Journal of Pharmaceutics, 334(1-2), 1-14. https://doi.org/10.1016/j.ijpharm.2007.01.028

Stantiall, S. E., & Serventi, L. (2018). Nutritional and sensory challenges of gluten-free bakery products: a review. International Journal of Food Sciences and Nutrition, 69(4), 427-436. https://doi.org/10.1080/09637486.2017.1378626

Tulukcu, E., Cebi, N., & Sagdic, O. (2019). Chemical fingerprinting of seeds of some salvia species in Turkey by using GC-MS and FTIR. Foods, 8(4), 118. https://doi.org/10.3390/foods8040118

Yazar, G., & Demirkesen, I. (2023). Linear and non-linear rheological properties of gluten-free dough systems probed by fundamental methods. Food Engineering Reviews, 15(1), 56-85. https://doi.org/10.1007/s12393-022-09321-3

Ylimaki, G., Hawrysh, Z., Hardin, R., & Thomson, A. (1991). Response surface methodology in the development of rice flour yeast breads: sensory evaluation. Journal of Food Science, 56(3), 751-755. https://doi.org/10.1111/j.1365-2621.1991.tb05374.x