

Research Article

Using MR-FTIR and Texture Profile to Track the Effect of Storage Time and Temperature on Pita Bread Staling

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Received 14 December 2017; Revised 12 February 2018; Accepted 22 February 2018; Published 29 March 2018

Academic Editor: Jorge Barros-Velázquez

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Textural deterioration of pita bread, PB, due to staling is an important quality parameter during storage. Loss of freshness due to PB staling results in significant quality and economic loss. PB staling was studied using both MR-FTIR spectroscopy and textural profile including time to rupture and tensile and compressive forces. The study was conducted at room temperature (23°C) and freezing temperature (−18°C) over 96 h of storage time after baking. Some physical properties of PB such as loaf weight, dimensions, water activity, and density were measured. MR-FTIR measurement spectra in the wavelength 900–1150 cm^{−1} corresponding to the “saccharides” regions and the range 3000–3600 cm^{−1} corresponding to O-H bond stretching vibration were able to clearly detect the PB deterioration at different storage times as shown by statistical significance test. Mechanical measurements of tearing force, time to rupture, and 25% compression force were also found to be good indicators of PB quality deterioration. Time to rupture, however, was found to be the best PB deterioration indicator. In addition, PB stored at room temperature showed a significant deterioration (toughening) compared to that stored at freezing temperature which showed little or nonsignificant deterioration during storage. High negative correlation, $r = -0.97$, was observed between the 25% compression force and the wavenumber 960 cm^{−1} at room temperature.

1. Introduction

Pita bread, PB, also known as Arabic bread or double-layer bread, is consumed widely in Middle East, the Nile Valley, and the Arab Gulf countries. PB is an important source of calories in a typical Middle Eastern diet. It has been reported that bread and other bakery products are responsible for about 85% of total calories in a typical Middle Eastern diet [1, 2]. The limited shelf life of PB due to mold growth and bread staling (firmness) results in some significant economic losses. The economic losses from bread waste and spoilage were estimated to be over one billion dollars per year [3]. In addition, wheat prices showed large increases in the global markets during the last two decades which contributed to further economic losses. Many Middle Eastern countries including Jordan are classified as a net-food importing countries. For example, statistics of agricultural production in Jordan for the

past few years showed that local wheat production was only adequate for few weeks. This resulted in heavy reliance on imported wheat flour which is sold to bakeries at a subsidized price. In 2015, for example, the Jordanian government spent about 214 M \$ in flour subsidy [4].

Pita bread is different from pan bread in terms of formulation, dough characteristics, baking time, and temperature. Pan bread formulation includes wheat flour, salt, sugar, yeast, shortening, oxidizing agents, and surfactant. PB has similar ingredients to pan bread, but with lower loaf thickness and a shorter baking time [5, 6]. Two types of bread quality deterioration are of particular interest to consumers including bread staling which results in the development of a progressively firm bread structure and mold growth resulting from high water activity of the bread (0.90 to 0.96) [5]. Bread deterioration due to staling is considered the most important factor in quality deterioration of bakery products

since it results in an increasingly firm bread structure during storage. Studies on pan bread staling reported a complex set of interfering processes that result from many factors. Starch retrogradation (recrystallization) is believed to be the most important factor causing negative textural changes in bread. It involves two processes: a process of rapid irreversible amylose gelation which takes place directly after baking and a slower amylopectin retrogradation responsible for subsequent bread staling during storage [7–12]. Other factors include starch-gluten interactions, existence and proportions of nonstarch polysaccharides (pentosans), moisture migration and redistribution, and storage temperature [8, 13].

Studies on PB showed that quality started to deteriorate 24 h after baking. Successful attempts were made to eliminate mold growth and extend shelf life in PB by adding antimolding agents such as fumaric acid and sodium propionate (Abou-Ghoush et al., 2007). Attempts to stop or slow down bread staling, however, were less successful. Such attempts included addition of gums and hydrocolloids (guar gum, locust bean gum, xanthan gum, and CMC) (Gavilloghi, 2006) [14] or addition of amylopectic and nonamylopectic enzymes to brown pan bread. Nevertheless, the results reported better bread quality and showed longer shelf life to some extent [15].

Applicability of various methods to measure bread staling has been reviewed extensively in literature. Texture-based methods and Fourier-transform infrared spectroscopy (FTIR) are some of the most common methods used. Texture-based methods use physical measurements of bread staling while FTIR spectral measurements are based on both physical and chemical parameters [16]. Fiszman et al. [15] used the penetration test and texture profile analysis to study the effect of enzyme addition on brown pan bread staling over 20 days of storage at room temperature. Mahmoud and Abou-Arab [17] compared the effectiveness of several methods to evaluate the bread staling in Egyptian flat bread. Aludatt et al. [18] used the compressive force test from a 13.8 mm diameter stainless steel probe to measure the texture of pita bread. They subjected two pita bread slices to compressive forces and measured the corresponding force at 25 and 50% deformation. Abou-Ghoush et al. (2007) used both tensile and compressive force test to measure textural changes in PB during 7 days of storage at room temperature. They concluded that tensile force was the most accurate method to measure bread firmness.

Both near and medium range infrared spectroscopy (NIR and MIR) were reported to provide good measurements of bread staling. Osborne [19] used NIR spectroscopy in the range of 400–2498 nm to study the effect of starch and water on bread staling. Osborne [20] used NIR spectroscopy between 1100 and 2500 nm to study starch crystallinity in bread. He reported that NIR spectroscopy can be used to follow three phenomena related to bread staling including starch structure changes due to inter- and intramolecular hydrogen bonds, crumb scattering, and moisture loss. Xie et al. [16] used NIR spectroscopy in the range 400 to 1700 nm to measure staling in white pan bread over 5 days of storage at room temperature. They compared the results with those obtained by compression test in texture analyzer. They reported that NIR measurements compared well with actual

storage time. They also reported a good agreement between NIR and texture analyzer measurements.

Cocchi et al. [21] reported that IR spectroscopy is more rapid, sensitive, and cheaper than other methods used to measure bread staling. They reported that mid infrared spectroscopy (MIR) is easier to interpret than NIR and more valuable in observing the molecular conformational changes, while NIR was better in reflecting chemical and physical aspects of bread staling especially those related to hydrogen bonding during starch crystalline network formation. Cocchi et al. [21] used MIR spectroscopy and principle component analysis (PCA) as a rapid and cheap method to monitor the bread staling during a storage period of one week. Ringsted et al. [22] reported the use of a 2D MIR-NIR correlation spectroscopy successfully to evaluate wheat bread crumb staling during aging.

The effect of storage time and temperature on the quality of pan bread has been reported by several researchers. Bread staling was observed to increase at refrigeration temperature compared to room temperature [23, 24]. Manzocco et al. [25] studied the crystallinity of bread stored at -18 , 4 , and 25°C over a period of 20 days using X-ray diffraction (XRD). They observed a significant correlation between firmness and relative crystallinity during storage. They found, however, that bread firmness was highest for bread stored at 25°C rather than at 4°C while firmness was lowest for bread stored at -18°C . Ronda et al. [26] studied the effect of prolonged storage time on staling of partially baked PB and fully baked (FB) breads. They used firmness, starch retrogradation, and moisture content in addition to glass-transition temperature (T_g) to evaluate bread aging. They concluded that the proper freezing storage temperature had to be sufficiently lower than T_g to slow down bread staling. Based on the obtained results, the authors proposed that hardening of bread during storage may not be only related to starch crystallization or water loss and developed a regression study describing how the combined effect of both variables could better explain the firming evolution.

No literature was found on the combined effect of storage time and temperature on the staling of PB. The objectives of this study were therefore

(1) to develop a fast and effective method for measuring PB texture quality deterioration using textural measurements and FT-MIR spectra,

(2) to study the effect of storage time (2 to 96 h) and temperature (-18 and 23°C) on staling of PB.

2. Material and Methods

2.1. Materials. Six lots of PB were prepared and baked. Bread was baked in a local automated bakery in Irbid, Northern Jordan, similar to the standard procedure reported by Faridi [27] and Quail [5]. A mixture contained 20 kg flour (14% moisture basis), 1% salt, 1% yeast, and 53% water approximately. This consistency is used by the bakery as it yields the most reliable baking absorption. The dough was mixed at medium speed for 6 min (1 min beyond development time) using planetary mixer. After mixing, the dough was allowed to ferment for 60 min at (30°C). After fermentation, the dough was scaled off

into 70–80 g pieces using an automatic cutter. The pieces were cut and rounded into balls and covered with plastic cover to prevent skin formation. The dough was passed through a two-stage roll sheeter. In the first stage, the gap between the rolls was 10 mm, and in the second stage the gap was set to 3 mm. Then, the bread was baked at 400°C for 90 s, on a stainless steel conveyor. After baking, the bread was cooled for 10 min and placed in bags. Two lots containing 10 loaves each were packaged in double zip lock polyethylene bags. Lots were packed during the first hour after baking and stored directly at room temperature (23°C) or at freezing temperature (−18°C) until used.

2.2. Methods

2.2.1. Physical Properties. Due to the possible variations in physical properties of PB, physical properties were measured, including loaf weight, thickness, volume, bulk density, moisture content, and water activity. Loaf weight was measured using a digital balance with 0.05 g accuracy (Denver Instruments, USA); loaf thickness (mm) and geometric mean diameter (mm) were measured using a digital caliper to 0.1 mm (ZZW Precision Tool Supply, China). Loaf volume was measured by water displacement method, where a 2 × 4 cm rectangular section of the loaf is first wrapped in a thin saran wrap, and the loaf was then immersed in water completely. The displaced water volume (cm³) was obtained using a 500 ml graduated cylinder. Loaf bulk density was calculated as loaf weight divided by volume. The moisture content of PB was measured using AACC method (AACC 44-15A, 1995). Water activity was measured using an AQUA LAB CX-2 digital water activity meter (Decagon Co., Pullman, WA, USA) after proper calibration. All measurements were obtained in triplicate.

2.3. Texture Analysis

2.3.1. Tensile Test (Tearing Force and Time to Rupture Test). Tearing force (N) and time to rupture (s) were tested using a procedure similar to that developed by Abu-Ghoush et al. [28] with some modifications. Bread sections were cut into a dump-bell shape with dimensions (115 mm × 20 mm × 6 mm). All sections were obtained from the central region of the loaf avoiding the exterior peripheries. Each end of the section was placed in a special clamp designed for tensile testing (Tensile Grips) with a Universal Testing Machine (UTM, XWW-2W, China). The tensile test with grip probe had the following settings: pretest speed: 1 mm s^{−1}; test speed: 1.7 mm s^{−1}; posttest speed: 10 mm s^{−1}; and 40% strain force: 10 g. Tearing force and time to rupture were recorded when the sample failed (ruptured). Measurements were first conducted 2 hrs after baking (fresh baked sample). The remaining bread loaves were equally divided into two batches: one was stored at −18°C (frozen) while the other was stored at 23°C (room temperature) for later testing. Measurements were then done at 24, 72, and 96 hrs after baking for the samples stored at both storage temperatures. For the frozen samples and prior to measurements, the samples were removed from freezer one hour before testing to allow reaching the room

temperature. All measurements were conducted in triplicate at room temperature. The samples were kept in double, tightly sealed zipper bags until the beginning of the test. Test time was generally less than 3 minutes. After the test is complete, the samples were directly returned to the zipper bag. To check for possible significant moisture loss, the samples' moisture was checked before and after the test for the samples at room and frozen temperatures. The results showed less than 1% difference in moisture.

2.3.2. Compression Test (Deformation Test). Compression test was also conducted at the same storage temperatures and times as above, using the procedure proposed by Dahle and Sambucci [29]. The procedure was originally adapted by American Association of Cereal Chemists (AACC 74-10A) for evaluating freshness of sliced white bread. Compression force was obtained using a Universal Testing Machine (UTM, model xww-2A, China). A 13.8 mm stainless steel cylindrical probe was used to compress two slices of PB at a test speed of 1 mm/s. Preliminary testing showed that compression force values at 25 and 50% deformation (equivalent to 25% and 50% of the bread sample thickness) were better indicators of bread toughness than maximum compression force (force at failure) proposed in the original AACC 74-10A procedure. The compression force (N) was therefore recorded at 25% and 50% deformation in addition to maximum compression force. Testing was conducted at 2, 24, 72, and 96 hrs after baking for the bread samples stored at −18°C and 23°C. All measurements were conducted in triplicate at room temperature. The samples' moisture was also checked before and after the test for the samples at room and frozen temperatures. The results showed less than 1% difference in moisture.

2.4. FTIR Analysis. FT-IR spectra of bread samples stored at both room and frozen conditions over 5 days of storage were obtained using Bruker FTIR spectrometer, model Tensor II operating in the middle IR region (4000–400 cm^{−1}) with attenuated total reflection (ATR) mode. The spectrophotometer is equipped with Bruker Platinum ATR accessory with pressure applicator. Each bread sample stored at room conditions was removed from the polyethylene bags and placed in room conditions for 2 minutes before the start of each measurement. Bread samples stored in frozen conditions were removed from freezer an hour before the test to allow it to reach room temperature. The measurements were obtained directly close to the middle of the bread loaf without any further treatments except for the pressure applied by the ATR accessory. All spectral measurements were made at a nominal resolution of 2 cm^{−1}, with 32 interferograms that were coadded and zero-filled to double the number of data points and then apodised with a Blackman-Harris 3-term function before Fourier transformation. The single-beam spectra of the samples were divided by the single-beam spectra acquired without sample (reference spectra) in order to yield transmission spectra [21]. Similarly, the samples moisture was checked before and after the test for the samples at room and frozen temperatures. The results showed less than 1% difference in moisture.

TABLE 1: Physical properties of PB loaves.

Parameter	Mean \pm SD
Weight (g)	59.91 \pm 2.76
Thickness (mm)	5.76 \pm 0.17
Moisture content (% w.b.)	25.91 \pm 2.11
Water activity	0.95 \pm 0.02
Geometric mean diameter (mm)*	148.18 \pm 2.59
Bulk density (kg/m ³)	336.90 \pm 22

* Average of major, medium, and minor diameters.

2.5. Data Analysis. A single factor Analysis of Variance (ANOVA) was used to detect the effect of storage time and temperature for tensile and compression tests in addition to FT-MIR spectra. For FT-MIR data, however, the spectra were first modified by estimating unit absorbance area under the band 900–1150 cm⁻¹ which corresponds to the “saccharides” regions and the unit absorbance area under the band 3000–3600 cm⁻¹ which corresponds to O-H bond stretching vibration. Both bands were reported to be useful in tracking bread staling during storage [21]. In addition the software SmartPLS (SmartPLS 3.2.7, GmbH) was used to explore the correlation between texture and FTIR measurements.

3. Results and Discussion

3.1. Physical Properties. The physical properties of PB are shown in Table 1. The weight, thickness, and diameter were 59.9 g, 5.76 mm, and 148 mm, respectively. The results were higher than those reported by Toufeili et al. [30] for PB who reported the thickness and diameter of PB to be 1–2 mm and 26–30 cm, respectively. This can be attributed to the possible variations in PB production process, including differences in formulation, dough characteristics, temperature-time combination during baking, and addition of shortenings, oxidizing agents, and surfactants [6]. Water activity was notably high (0.95) which explained the reason behind the fast molding of PB, normally after 5 days of baking at room temperature. Abu-Ghoush et al. [28] reported also similar water activities for Arabic flat bread (0.94–0.96). Bulk density is 336 \pm 22 kg/m³, which is larger than corresponding values for white pan bread (290 to 340 kg/m³) [31]. This may also explain why PB has more chew-ability compared to pan bread.

3.2. Tearing Force and Time to Rupture Test. The effect of storage time (2 to 96 hrs) and storage temperatures (23°C, and -18°C) on tearing force of the PB samples is shown in Figure 1. Tearing force decreased with increase in storage time for both storage temperatures. At room temperature, tearing force decreased from 0.85 N after 2 hrs (fresh samples) to 0.45 N after 96 hrs of storage after baking. A lesser decrease of 0.85 to 0.6 N, however, was observed for bread stored in freezing temperature. Decrease in tearing force is an indicator of loss of freshness in PB. As expected, bread samples stored at room temperature showed a faster loss of freshness compared to those stored in freezing temperature. This might be attributed to the limited mobility and interactions between

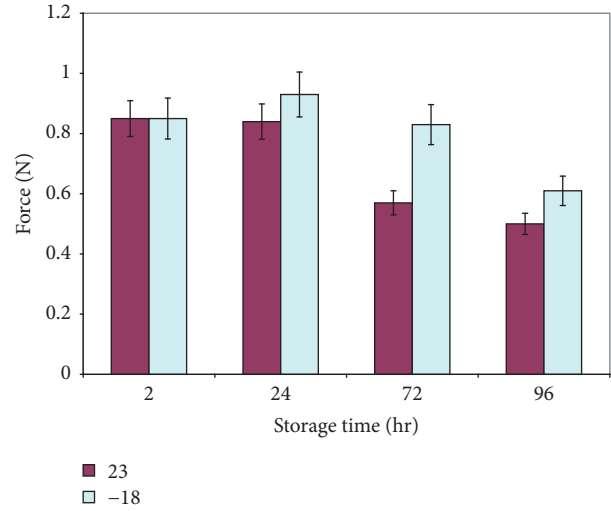


FIGURE 1: Relationship between tearing force and storage time for PB at different storage temperatures.

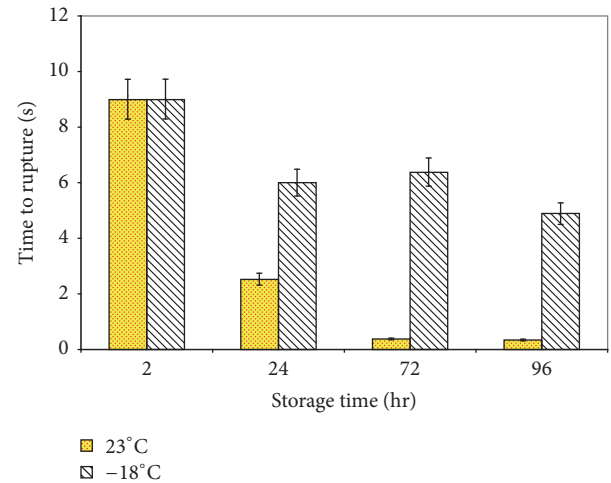


FIGURE 2: Relationship between time to rupture and storage time for PB at different storage temperatures.

water, starch, and protein in bread matrix during freezing storage of bread samples resulting in an overall slowdown of the bread staling and retarding negative quality changes. The relationship between time to rupture and storage time is shown also in Figure 2. Time to rupture is a measure of the time needed for bread samples to fail under tension force. Time to rupture decreased from 9 s for fresh bread samples (2 hrs after baking) to 0.5 s after 96 hrs of storage at room temperature. ANOVA test showed significant differences (at $p < 0.05$) among storage times for bread stored at room temperature, while no significant difference was observed for samples stored at freezing temperature (at $p < 0.05$). This shows that time to rupture is better indicator of PB freshness than tearing force. Toufeili et al. [30] reported similar findings for Arabic flat bread. They reported the probing extensibility, which is equivalent to the time to rupture, to decrease significantly with bread aging (staling). These results agreed also with earlier studies on Arabic flat bread

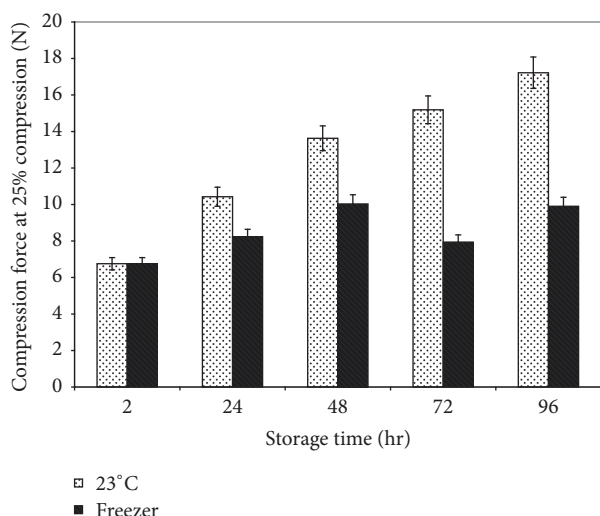


FIGURE 3: Relationship between compression forces at 25% compression and storage time at 23°C (room temperature) and -18°C (freezer temperature) for pita bread (source: 3rd batch).

treated with texture enhancers which concluded that time to rupture was the best method to track PB bread staling over storage time [28]. The longer bite of pita bread as compared to pan bread could be due to the absence of the shortening in the pita bread formula and the higher bulk density.

3.3. Compression (Deformation) Test. The compression test measured compression force at 25% of total bread thickness. The results of compression test for PB at room and freezer temperature are shown in Figure 3. The values varied between 7 and 17 N and between 7 and 10 N at room and freezer temperature, respectively. Measurements of compression force at 50% deformation were also obtained (not shown) and less variation was observed for bread samples compressive force with storage time. It is observed that compression force at 25% of total bread thickness increased significantly with storage time, while increase in 50% compression was insignificant. The values of 25% compression values were therefore found to be better indicator of bread deterioration than those obtained at 50% compression. ANOVA test showed significant differences at ($p < 0.05$) among storage times for bread stored at room temperature and no significant difference was observed for samples stored at freezing temperature at the same level. Furthermore, multiple linear regression statistical analysis showed that the relationship between compression force at 25% thickness and storage time (t_s) in hr is highly significant ($p < 0.001$) for quadratic and logarithmic relationships:

$$F_{25\%} = 5.92 + 0.39(t_s) - 2.81 \times 10^{-3}(t_s)^2 \quad \text{with } R^2 = 0.981, \quad (1)$$

$$F_{25\%} = -2.31 \ln(t_s) - 10.4 \quad \text{with } R^2 = 0.988,$$

where $F_{25\%}$ is the compression force at 25% of total bread sample thickness in N; t_s is storage time in hr at room temperature (23°C).

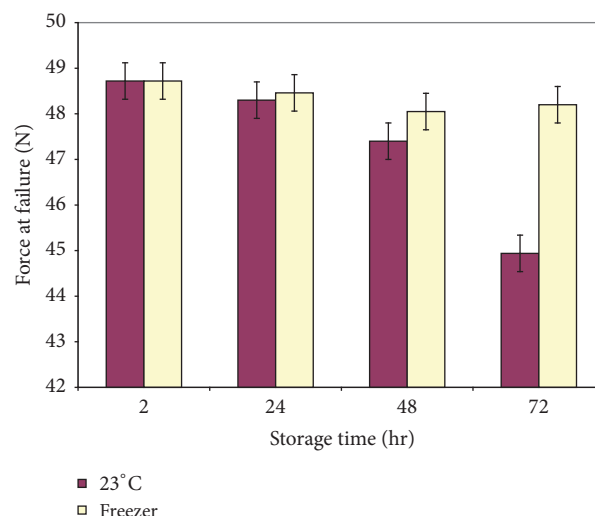


FIGURE 4: Relationship between compression forces at failure and storage time at 23°C (room temperature) and -18°C (freezer temperature) for pita bread (source: 4th batch).

The compressive force at failure was also measured. The relationship between compressive force at failure and storage time for PB stored at room and freezing temperatures is shown in Figure 4. The compressive force at failure decreased with storage time between 44.5 and 48.5 N and between 48 and 48.5 N for room and freezing temperatures, respectively. The relationship between failure force and storage time, however, was found not to be statistically significant at both storage conditions.

3.4. FTIR Spectroscopy. Mid infrared spectroscopy measurements of PB staling were obtained to compare with textural measurements. The normalized unit absorbance versus wavelength for PB samples stored under room temperature for 5 days is shown in Figures 5 and 6. Unit absorbance increased with storage time in the band 900–1150 cm^{-1} , which corresponds to the “saccharides” group indicating progressive starch crystallization with storage time which may partially explain PB staling due to starch retrogradation. In addition, the second spectra band, 3000–3600 cm^{-1} , which corresponds to O-H bond stretching vibration showed also some increase in unit absorbance with storage time which may also explain the effect of water mobility on water-starch-protein interaction and therefore on PB staling. ANOVA was further used to investigate the statistical significance among the different storage times. The ANOVA analysis was carried out by calculating the area under each of the five storage times and for both bands (900–1150 cm^{-1} and 3000–3600 cm^{-1}), separately. ANOVA was then used to separate variation within each storage time to variation between storage times. A significant variation was observed between storage times as compared to variation within storage time for both bands ($p < 0.001$). The results obtained are in agreement with Cocchi et al. [21] who observed an increase in absorbance unit with storage time in pan bread. Ringsted et al. [22] reported similar results where a 2D MIR-NIR correlation spectroscopy

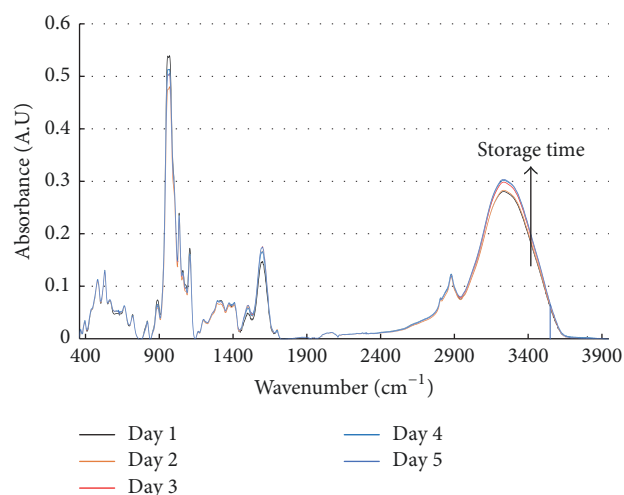


FIGURE 5: Mean spectra of the normalized bread spectra over 5 days of storage in room conditions.

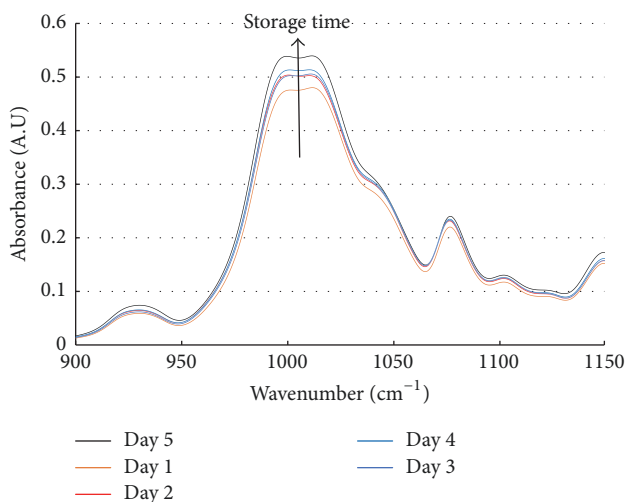


FIGURE 6: Amplified mean spectra of the normalized bread spectra over 5 days storage at room temperature in the band 900–1150 cm^{-1} .

was successfully used to track bread crumb aging. They observed that MIR band at 1047 cm^{-1} which was related to amylopectin retrogradation was highly correlated with bread hardness measured by texture analyzer. They also found a high covariance between MIR band 1047 cm^{-1} and NIR band 910 nm . They concluded that major bread staling processes such as water loss and amylopectin retrogradation can be easily followed by both MIR and NIR spectra. Similarly, van Soest et al. [32] reported that MIR spectroscopy has been successfully used in bread to track changes from more amorphous to more structured and crystalline starch form in fresh bread to a stale bread by increase in intensity at 1047 cm^{-1} and decrease in intensity at 1022 cm^{-1} . They also reported that changes in starch structure studied by MIR spectroscopy was related to the double helix content and alignment in starch or the so-called short-range order.

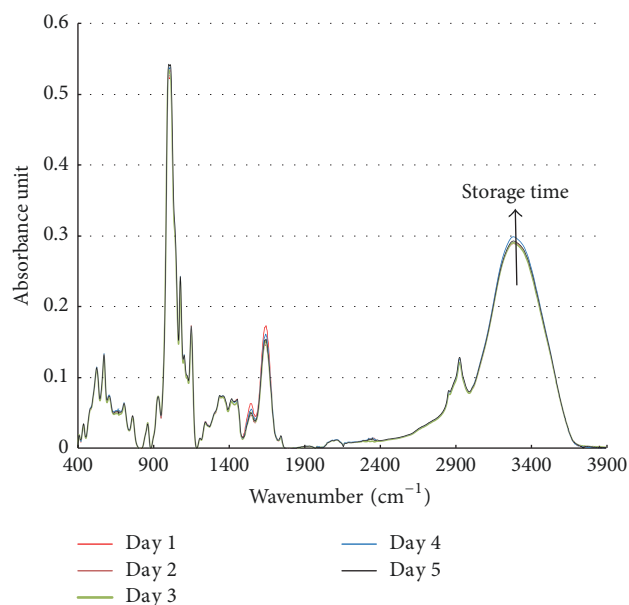


FIGURE 7: Mean spectra of the normalized bread spectra over 5 days of storage in frozen condition.

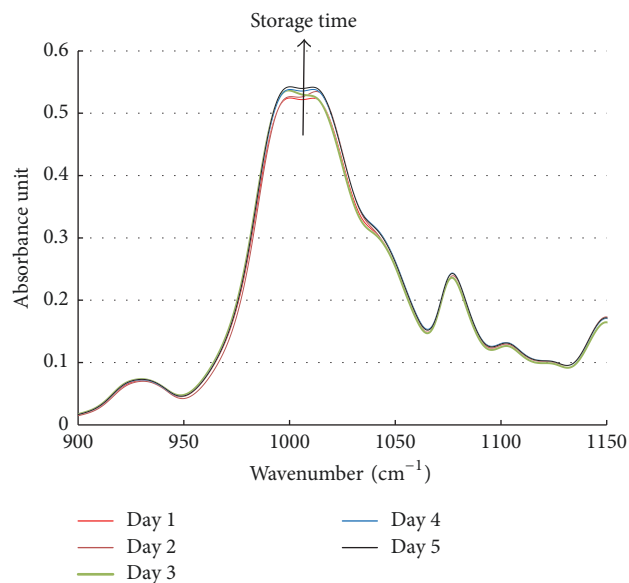


FIGURE 8: Amplified mean spectra of the normalized bread spectra over 5 days of storage at freezing temperature in the band 900–1150 cm^{-1} .

The spectra obtained for PB stored for 5 days under freezing temperature are shown in Figures 7 and 8. The figures show much less variation in absorbance unit with storage time for both saccharides and O-H bands. ANOVA for the area under both bands showed nonsignificant variation (at $\alpha = 0.05$) between storage days when compared to variation within storage days for both the O-H and saccharides bands. The effect of freezing on slowing PB deterioration was therefore evident for both water “O-H” band and starch “saccharides” band. This may be explained by the limited mobility

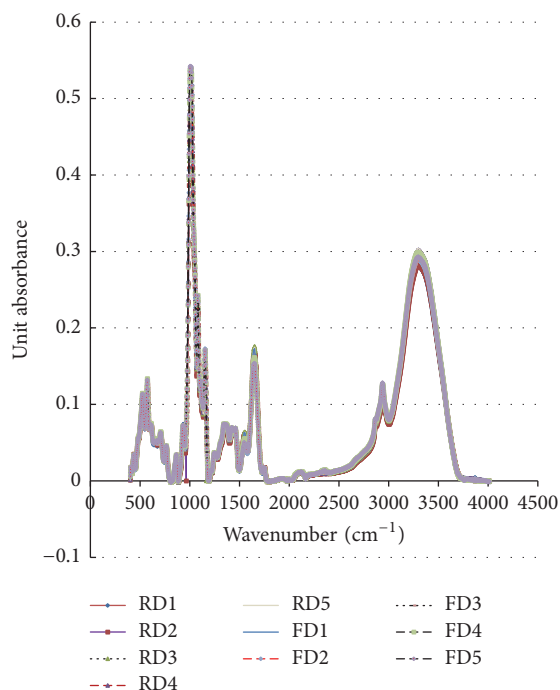


FIGURE 9: Mean spectra of the normalized bread spectra over 5 days of storage for room and frozen conditions.

of water during freezing which hindered water-starch-protein interactions and therefore prevented PB staling. In order to compare room and frozen conditions spectra, Figure 9 shows unit absorbance versus wavenumber for both room and freezing storage temperatures over the period of storage. The result did not, however, provide much information since all spectra appeared to be overlapping. Ronda et al. [26] reported a slowdown of bread staling stored at temperature sufficiently below glass-transition temperature T_g . Manzocco et al. [25] reported also that bread crystallinity was lowest under frozen temperature of -18°C .

Generally, results showed that time to rupture test provided the best results in terms of tracking the effect of storage time and temperature on PB staling followed by the 25% deformation test which showed less sound but significant results. MR-FTIR measurement spectra in the wavelengths $900\text{--}1150\text{ cm}^{-1}$ and $3000\text{--}3600\text{ cm}^{-1}$ were also able to clearly detect and track PB staling at different storage times as shown by statistical significance testing. While the first two tests were destructive and required longer preparation and testing times, the last one was nondestructive and required shorter testing time and minimum test preparation. Therefore, FTIR would be the optimal choice for quick or online tracking of PB staling but still more expensive instrumentation is needed. The first two tests, on the other hands, will be cheaper to conduct but more appropriate for offline testing.

A summary of the textural properties of PB is shown in Table 2. The data show that 25% compressive force varied between 6.3 and 17.2 N and 6.3 and 10 N for room and freezing storage, respectively. Time to rupture decreased from 9 to 0.29 s and 9 to 3.4 s for room and freezing storage, respectively. Tearing force (N) and force at failure showed

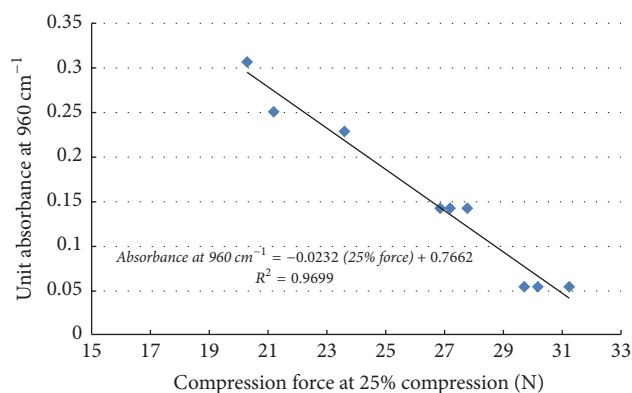


FIGURE 10: Correlation between compression force at 25% compression and absorbance at 960 cm^{-1} during 96 hr of storage at room temperature (23°C).

less pronounced decrease with storage time for both room and freezing storage. The results show clearly that 25% compressive force and time to rupture were better indicators of PB staling during storage.

The relationship between textural properties and FTIR measurements was further explored. The results were obtained using the software Smart PLS which calculates the correlation matrix among all variables. The best correlation ($r = -0.97$, negative correlation) was observed between compression force at 25% compression and FTIR absorbance corresponding to the wavenumber 960 cm^{-1} over the period of storage at room temperature as shown in Figure 10. Similarly, lower negative correlation ($r = -0.85$) was observed between holding until rupture time and absorbance at 982 cm^{-1} . However, much lower correlations were observed for frozen PB (r values were less than 0.52).

3.5. Effect of Storage Time and Temperature on PB Quality.

The effect of storage temperature on PB quality deterioration was found to be significant. Both texture measurements and FTIR spectra measurements showed that bread stored at freezing temperature of -18°C retained better quality during storage. These findings were best observed by time to rupture test (Figure 2) followed by FTIR measurements (Figure 5) and then by 25% compression force (Figure 3) and finally by tearing force (Figure 1). High proportion of free water (water activity) of PB explains why freezing was effective in slowing bread staling as freezing limits the mobility of water and prevents water-starch-protein interaction and therefore slower PB staling. Aguirre et al. [33] observed that moisture equilibration occurred between crust and crumb during bread storage, and they reported that freezing storage at -18°C resulted in very limited water mobility when compared to breads stored at 4 and 25°C . They also concluded that free water measured by water activity remained almost constant in breads stored at -18°C for 23 days. In addition they showed that starch molecules reassociated during storage to produce a new crystalline structure characterized by the typical B-type XRD structure and that storage at -18°C slowed down but did not retard the recrystallization process. freezing storage of partially baked PB Indian chapati flat

TABLE 2: Various textural properties of PB.

Storage time (h)	25% force (N)		Time to rupture (s)		Tearing force (N)		Force at failure (N)	
	(23°C)	(−18°C)	(23°C)	(−18°C)	(23°C)	(−18°C)	(23°C)	(−18°C)
2	6.3 (0.12)*	6.3 (0.12)	9.0 (1.20)	9.0 (1.20)	0.85 (0.09)	0.85 (0.08)	48.5 (4.7)	48.7 (4.7)
24	10.4 (1.38)	8.2 (0.91)	2.5 (0.21)	6.0 (0.52)	0.84 (0.07)	0.93 (0.07)	48.7 (5.2)	48.6 (4.2)
48	15.2 (1.14)	7.9 (1.04)	-	-	-	-	47.8 (6.2)	48.6 (3.9)
72	13.6 (3.09)	10 (0.81)	0.30 (0.02)	4.9 (0.42)	0.50 (0.04)	0.61 (0.05)	44.8 (5.3)	48.1 (6.2)
96	17.2 (4.27)	9.9 (2.57)	0.29 (0.03)	3.4 (0.31)	0.57 (0.06)	0.39 (0.04)	-	-

* Mean (standard deviation).

bread was reported to be useful in maintaining its quality. The extensibility of PB chapatti after rebaking was found similar to the fresh, conventionally baked bread [34]. Ronda et al. [26] showed that freezing storage time resulted in a significant decrease in firmness of partially baked bread crumb. Gray and Bemiller [13] reported that mobility of water and other macromolecules in bread matrix could be responsible in part for bread staling and bread quality deterioration. Further investigations on the effect of adding water binders, hydrocolloids, and surface active agents to PB are needed to explore the effect of reducing available water on staling at room temperature given the fact that freezing was effective in reducing PB by limiting water availability [35].

4. Conclusions

- (i) Textural parameters including time to rupture and tearing force decreased significantly with increase in storage time and PB staling for bread stored at room temperature while 25% compression force and unit absorbance of FTIR increased significantly with increase in storage time and bread staling at room temperature.
- (ii) PB stored at −18°C showed nonsignificant changes in shearing force, time to rupture, or 25% compression force during storage. Therefore, freezing was found to be effective method for slowing down PB staling and therefore quality deterioration.
- (iii) Time to rupture, the 25% deformation, and MR-FTIR measurement spectra in the wavelength 900–1150 cm^{−1} corresponding to the “saccharides” regions and the range 3000–3600 cm^{−1} corresponding to O-H bond stretching vibration were able to clearly detect the PB deterioration at different storage times as shown by statistical significance test.
- (iv) High negative correlation ($r = -0.97$) was observed between the 25% compression force and the wavenumber 960 cm^{−1} during storage at room temperature.

Additional Points

Practical Application. Pita bread, PB, is an important part of the Middle Eastern, Arabian Gulf, and Nile Valley diet. Improvements of quality of PB to slow down bread quality deterioration by staling and reduce the associated economic

losses are of great interest. The effect of storage time and temperature on PB has not been addressed appropriately in literature. In addition, an accurate and reliable analytical method for measuring PB quality deterioration has not been explored yet. The objectives of this study were to establish reliable instrumental methods for measuring PB quality deterioration and to study the effect of storage time and temperature on bread quality.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

The authors would like to acknowledge the Deanship of Scientific Research at Jordan University of Science and Technology for supporting this research project.

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