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Published Version: Evaluation of the effect of edible coating on mini-buns during storage by using NIR spectroscopy / Nallan Chakravartula S.S.; Cevoli C.; Balestra F.; Fabbri A.; Rosa M.D.. - In: JOURNAL OF FOOD ENGINEERING. -ISSN 0260-8774. - ELETTRONICO. - 263:(2019), pp. 46-52. [10.1016/j.jfoodeng.2019.05.035]

Availability: This version is available at: https://hdl.handle.net/11585/723370 since: 2021-09-09

Published:

DOI: http://doi.org/10.1016/j.jfoodeng.2019.05.035

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(Article begins on next page)

This is the final peer-reviewed accepted manuscript of:

Nallan Chakravartula S. S.; Cevoli C.; Balestra F.; Fabbri A.; Dalla Rosa M. *"Evaluation of the effect of edible coating on mini-buns during storage by using NIR spectroscopy"*

which has been published in final form in JOURNAL OF FOOD ENGINEERING 2019, vol. 263, pp. 46-52

The final published version is available online at: https://doi.org/10.1016/j.jfoodeng.2019.05.035

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Contents lists available at ScienceDirect

Journal of Food Engineering



journal homepage: www.elsevier.com

Evaluation of the effect of edible coating on mini-buns during storage by using NIR spectroscopic

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ARTICLE INFO

Keywords: Mini-buns Staling Edible coating Near infrared

ABSTRACT

Moisture migration plays an important role in the stability and quality of bakery products. In this study, pectin, alginate and whey protein edible coating was used as an edible barrier to evaluate its effectiveness for retaining the quality of bread by reducing moisture loss and textural changes during storage. Mini-buns as model systems were applied with 1 layer and 2 layers of coating and evaluated for moisture and texture attributes by both mechanical and spectroscopic techniques. The coatings displayed a retaining effect with the percentage increase in crumb hardness lower for coated samples (1L: 126.5% and 2L: 231.2%) than control samples (Control: 271.8%). Further, the spectral data for the crust and crumb surfaces were pre-treated and subjected to Principal Component analysis (PCA) for the evolution along storage time. In particular for moisture content, discrimination at 8 h for crust and after 24h for crumb was noted for both 1L and 2L coating layers with a $R^2 > 0.85$. Subsequently, PLS models with spectral data described satisfactorily the evolution of hardness with low (<10.8) RMSE values.

1. Introduction

Bread is a widely consumed product, with hamburger buns and mini-buns being one of the fast moving products in both super markets and fast food market (Esteller et al., 2005). These are characterized by uniform crust, crumb characteristics and rapid staling depending on the baking and storage conditions, besides mould growth. The shelf-life of bread is extended usually by addition of anti-microbial agents alone or in combination with high barrier and modified atmospheric packaging (MAP).

Staling is a detrimental change resulting in food waste by altering the eating character and aesthetic value of bread. It is a complex phenomenon involving in starch, protein and water interactions that are further complexed by the re-crystallization of starch. The crust staling phenomenon is constantly linked to the re-distribution of moisture whereas the crumb staling is related to the complex interactions and is less understood (Fadda et al., 2014; Gray and Bemiller, 2003; He and Hoseney, 1990). Many studies investigated reduction of staling by use of additives and changes in formulation (Callejo et al., 1999; GomesRuffi et al., 2012; Gray and Bemiller, 2003). However, the control of moisture loss was observed to have a positive effect on the bread staling with higher softness perceived. Moisture desorption and absorption is regulated usually by packaging, with high barrier packaging being used leading to "*over-packaging*" (Piergiovanni and Fava, 1997). Reduced level and alternative packaging solutions are being explored to reduce the packaging impact apart from use of active packaging (Altan et al., 2018; Jideani and Vogt, 2016; Licciardello et al., 2014; Noshirvani et al., 2017; Sarinthip et al., 2018).

In the spectrum of active packaging, recent studies evidenced that the use of edible coatings and films in combination with or without active components have improved the shelf-life of sliced bread (Balaguer et al., 2013; Otoni et al., 2014), mini panettone (Ferreira Saraiva et al., 2016) and cakes (Baeva and Panchev, 2005; Bartolozzo et al., 2016). Moreover, some researchers found positive effect of coatings on the texture and gluten network of cake type products and bread respectively (Baeva and Panchev, 2005; Galvão et al., 2018). In this aspect, edible films and coatings made up of one or more hydrocolloids with or without the addition of lipids with ability to act as gas and water va-

https://doi.org/10.1016/j.jfoodeng.2019.05.035 Received 18 January 2019; Received in revised form 10 April 2019; Accepted 27 May 2019 Available online xxx 0260-8774/ © 2019.

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Table 1

Weight loss and moisture content of control and coated (1 L, 2 L) mini buns during storage.

Sample		Storage time (hours)							
		0	2	4	6	8	12	24	
Weight loss (%)									
Whole bread	1 L	$0.0\pm0.0^{\mathrm{a}}$	$-1.5\pm0.3^{\rm b}$	$-3.0\pm0.5^{\circ}$	-3.9 ± 0.3^{cd}	$-4.7\pm0.4^{\rm d}$	-11.5 ± 0.9^{e}	-12.4 ± 0.6^{e}	
	2 L	$0.0\pm0.0^{\mathrm{a}}$	$-1.8\pm0.3^{\rm b}$	-3.6 ± 0.4^{c}	$-5.0\pm0.5^{\rm d}$	$-6.5\pm0.5^{\rm e}$	$-10.4 \pm 0.4^{\mathrm{f}}$	$-13.3\pm0.7^{ m g}$	
	Control	$0.0 \pm 0.0^{\mathrm{a}}$	$-2.5\pm0.1^{\rm b}$	$-4.1 \pm 0.1^{\mathrm{bc}}$	-5.6 ± 0.1^{bcd}	-7.4 ± 0.4^{bcd}	-10.8 ± 0.2^{bcd}	-15.7 ± 0.2^{bcd}	
Moisture conten	t (%)								
Top crust	1 L	22.1 ± 0.9^{a}	19.7 ± 0.3^{b}	19.6 ± 0.5^{b}	19.0 ± 0.8^{b}	18.6 ± 0.5^{b}	$16.5 \pm 0.8^{\circ}$	14.8 ± 0.5^{d}	
	2 L	$18.2\pm0.4^{\rm a}$	16.1 ± 0.8^{b}	$16.0\pm0.6^{\rm b}$	17.6 ± 1.4^{ab}	17.1 ± 1.2^{ab}	13.3 ± 0.9^{c}	12.6 ± 0.8^{c}	
	Control	18.7 ± 0.7^{abc}	18.2 ± 0.7^{abc}	$17.4 \pm 1.3^{ m abc}$	$17.2 \pm 1.1^{\mathrm{b}}$	16.4 ± 0.5^{b}	$20.5 \pm 3.9^{\circ}$	11.6 ± 0.5^{d}	
Bottom crust	1 L	$21.0\pm0.5^{\rm a}$	$20.7\pm0.8^{\rm a}$	$20.6\pm0.8^{\rm a}$	20.2 ± 1.5^{a}	18.6 ± 0.8^{b}	$17.8\pm0.8^{\mathrm{b}}$	14.6 ± 0.4^{c}	
	2 L	22.3 ± 1.4^{a}	19.4 ± 1.2^{b}	17.6 ± 1.1^{b}	19.1 ± 1.7^{b}	$18.5\pm0.6^{\mathrm{b}}$	14.5 ± 0.7^{c}	13.4 ± 0.9^{c}	
	Control	$18.8\pm1.7^{\rm a}$	17.8 ± 0.8^{a}	17.4 ± 0.4^{a}	18.0 ± 1.2^{a}	17.1 ± 1.1^{a}	16.8 ± 2.5^{a}	11.4 ± 1.0^{bc}	
Crumb	1 L	$31.4\pm0.9^{\rm a}$	30.6 ± 0.5^{a}	31.0 ± 0.6^{a}	$28.1\pm0.8^{\rm b}$	26.2 ± 0.8^{c}	$26.5 \pm 0.6^{\circ}$	19.7 ± 0.4^{d}	
	2 L	$30.4 \pm 0.9^{\mathrm{a}}$	30.7 ± 0.3^{a}	29.9 ± 0.6^{a}	25.5 ± 0.9^{b}	24.9 ± 0.6^{b}	24.9 ± 0.6^{b}	19.6 ± 0.7^{c}	
	Control	$31.1 \pm 0.7^{\mathrm{a}}$	$29.9\pm0.4^{\rm b}$	30.7 ± 0.5^{ab}	$27.5\pm0.6^{\circ}$	26.5 ± 0.4^{d}	22.4 ± 0.3^{e}	$17.3\pm0.3^{\rm f}$	

Values are mean \pm SD; Different letters row-wise indicate significant differences (p < 0.05).

Table 2

Mechanical parameters of control and coated (1 L, 2 L) mini buns during storage.

Parameter	Storage time (h)							
Hardness (N)	0	2	4	6	8	12	24	
1L	21.5 ± 1.5^{a}	$23.8 \pm 1.4^{\mathrm{ab}}$	27.5 ± 2.2^{ab}	$28.8 \pm 1.7^{\mathrm{b}}$	$35.6 \pm 3.4^{\circ}$	48.6 ± 4.6^{d}	100.2 ± 4.7^{e}	
2L	25.0 ± 3.1^{a}	29.6 ± 1.3^{ab}	28.2 ± 3.3^{ab}	39.8 ± 2.4^{abc}	43.0 ± 3.2^{abc}	82.8 ± 4.9^{bc}	$136.3 \pm 14.9^{\circ}$	
Control	19.4 ± 2.6^{a}	24.7 ± 3.6^{a}	25.6 ± 2.1^{a}	29.6 ± 3.1^{ab}	36.4 ± 3.1^{b}	72.3 ± 7.5^{c}	$128.8\pm9.9^{\rm d}$	
Cohesivity								
1L	$0.43 \pm 0.0^{\mathrm{a}}$	0.42 ± 0.0^{a}	0.40 ± 0.0^{a}	0.41 ± 0.0^{a}	0.40 ± 0.0^{a}	0.41 ± 0.0^{a}	0.42 ± 0.0^{a}	
2L	0.41 ± 0.0^{a}	0.40 ± 0.0^{a}	0.39 ± 0.0^{a}	$0.39\pm0.0^{\mathrm{a}}$	0.37 ± 0.0^{a}	0.36 ± 0.0^{a}	0.38 ± 0.0^{a}	
Control	$0.42\pm0.0^{\mathrm{a}}$	$0.38 \pm 0.0^{\mathrm{b}}$	0.39 ± 0.0^{ab}	$0.38\pm0.0^{\mathrm{b}}$	$0.37\pm0.0^{\mathrm{b}}$	$0.37\pm0.0^{\mathrm{b}}$	$0.38\pm0.0^{\rm b}$	
Chewiness (N)								
1L	6.34 ± 0.3^{a}	7.1 ± 0.6^{a}	$7.5\pm0.6^{\mathrm{a}}$	8.5 ± 0.9^{ab}	$10.6 \pm 1.2^{\mathrm{b}}$	15.1 ± 1.8^{c}	33.3 ± 1.8^{d}	
2L	7.0 ± 0.8^{a}	8.3 ± 0.3^{ab}	7.8 ± 0.9^{ab}	$11.1 \pm 1.0^{ m abc}$	11.4 ± 0.7^{abc}	$22.2\pm2.1^{\mathrm{bc}}$	39.3 ± 4.2^{c}	
Control	5.4 ± 0.9^{a}	6.1 ± 0.8^{ab}	6.7 ± 0.7^{ab}	$7.7\pm0.9^{\mathrm{ab}}$	9.1 ± 1.1^{b}	$19.9 \pm 0.8^{\circ}$	37.3 ± 3.8^{d}	

Values are mean \pm SD; Different letters row-wise indicate significant differences (p < 0.05).

Table 3

Correlation between moisture content and the mechanical parameters.

1 L	M_T	M_B	M_C	Н	Со	Che
M_T	1.00					
M_B	0.86	1.00				
M_C	0.86	0.89	1.00			
H	-0.85	-0.85	-0.94	1.00		
Со	-0.16	-0.16	-0.23	0.32	1.00	
Che	-0.82	-0.82	-0.91	0.98	0.48	1.00
2L						
M_T	1.00					
M_B	0.67	1.00				
M_C	0.52	0.71	1.00			
H	-0.65	-0.77	-0.91	1.00		
Со	0.25	0.40	0.46	-0.38	1.00	
Che	-0.65	-0.77	-0.91	1.00	-0.32	1.00
Control						
M_T	1.00					
M_B	0.74	1.00				
M_C	0.51	0.76	1.00			
H	-0.56	-0.80	-0.96	1.00		
Со	0.15	0.31	0.39	-0.31	1.00	
Che	-0.57	-0.81	-0.95	1.00	-0.25	1.00

^{*1} L- 1 layer; 2 L- 2 layers; M_T – moisture top crust (%); M_B – moisture bottom crust (%); M_C – moisture crumb (%); H- hardness (N); Co- Cohesivity; Che- Chewiness (N).

por barrier properties (Falguera et al., 2011; Galus and Kadzińska, 2015; Passarinho et al., 2014) can reduce crumb hardening by minimizing the migration of moisture.

The effectiveness of the coatings can be measured in terms of the moisture and textural changes which are strongly correlated to sensor-

Table 4

Parameters of the power law equation $(y = ax^b)$ obtained between top moisture content (*x*) and mechanical parameters (*y*).

Sample	Mechanical parameter	Equation		
		R^2	а	b
1L	Hardness	0.967	3.23E+07	-4.7204
	Chewiness	0.966	4.80E+07	-5.275
2L	Hardness	0.904	2.36E + 07	-4.799
	Chewiness	0.896	1.05E + 07	-4.969
Control	Hardness	0.889	4.47E+05	-3.3203
	Chewiness	0.895	2.24E + 05	-3.5449

ial acceptance. The textural attributes are frequently evaluated with texture profile analysis and compression tests industrially and as observed in literature (Conte et al., 2018; Licciardello et al., 2017). However, other techniques like near infrared spectroscopy (Cevoli et al., 2015; Piccinini et al., 2012), vibrational spectroscopy (Czaja et al., 2018; Nouri et al., 2017; Ringsted et al., 2017) are being explored for higher reliability and non-destructive nature. In particular, near infrared (NIR) spectroscopy in range of 1100 nm and 2400 nm was found to be related to the bread staling and was comparable to the texture results as observed by Cevoli et al. (2015) and Xie et al. (2003). Also, it is cost effective in comparison to traditional methods with information on both physical and chemical aspects of staling related to the hydrogen bonding. In relation to staling, the spectral wavelengths relating to the amylo-pectin retrogradation was observed to be the main factor for differentiating the staling. Moreover, the absorption bands of water

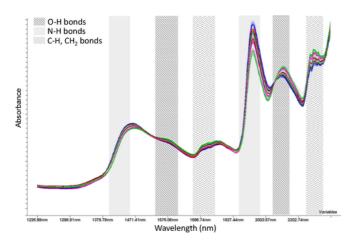


Fig. 1. Representative spectra of coated samples during storage.

molecules reflect the evolution of changes in starch and protein along the storage time, with successful application of NIR in products like bread and flat breads (Büning-Pfaue, 2003; Osborne, 1996; Wilson et al., 1991; Xie et al., 2003).

In light of the findings, in this work, NIR spectroscopy was used to evaluate the moisture content and physico-mechanical parameters during short storage of commercial mini buns applied with edible coatings.

2. Materials and methods

2.1. Materials

Sodium alginate (Sigma-Aldrich, St. Louis Missouri USA), pectin (derived from Citrus peel, Sigma-Aldrich, St. Louis Missouri USA), and whey protein concentrate (80% protein, Products-Gianni SRL, Milan Italy) were used for preparation of edible coating solution. Glycerol (\geq 99.5%) (Sigma-Aldrich, St. Louis Missouri USA) and Tween[®] 20 (Sigma-Aldrich, St. Louis Missouri USA) were used as plasticizing and emulsifying agents respectively. The 'mini buns' (Roberto Industria alimentare s.r.l, Italy) were procured from a local market (IperCoop, Cesena, IT). The ingredients of the mini buns (diameter of 70 mm and height of 30 mm).as per the packaging are wheat flour, water, rapeseed oil, yeast, Glucose-Fructose syrup, sugar, salt, and emulsifiers.

2.2. Preparation of coated bread

The coating solutions and the coated bread were prepared as described by Nallan Chakravartula et al. (2019). The coating solution was then spread onto the surface of bread with the aid of a confectionary brush as a single layer (1 L) or two layers (2 L) with 3-min interval between applications. The samples coated with single or two layers were initially dried at 60 $^{\circ}$ C for 25 min and 35 min respectively. The control samples consisted of uncoated samples randomly selected from the packages.

2.3. Storage evaluation

To study the effect of edible coating on bread dehydration kinetics and texture evolution, the samples were stored at 25 °C, 50% RH in climatic chambers (Constant Climate Chamber with Peltier technology, model HPP 108/749- Memmert, Germany) for 48 h, without any secondary packaging. The samples were evaluated periodically at 0, 2, 4, 6, 8, 12, and 24 h for the selected parameters. Furthermore, 36 h and 48 h samples of storage were also considered for NIR spectroscopy.

2.4. Weight loss and moisture content

The weight loss (%) during storage was recorded using an analytical balance (KERN and Sohn GmbH) up to the nearest 0.001 g. Moisture content on wet basis of the crust and crumb were separately determined by oven drying method (AOAC, 2000) at 105 °C until constant weight was achieved.

2.5. Mechanical characteristics

Mechanical characteristics were evaluated by Texture profile analysis (TPA) using a Texture Analyser (Mod.TA.HDi 500, Stable Micro Systems, Surrey, UK) according to Balestra et al. (2011) with modifications of using a P/75 mm cylindrical aluminum probe attached to a 25 kg load cell and whole bread similar to Guadarrama-Lezama et al. (2016). The data were acquired by associated software (Textexceed) and the texture parameters namely; hardness, chewiness and cohesivity were calculated. The sample hardness was determined by evaluating the maximum force of the first compression peak and is expressed in Newton's (N). Cohesivity was calculated as the ratio of work during the second compression cycle to that of first compression cycle. Finally

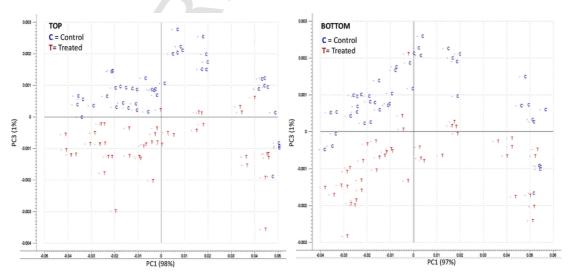


Fig. 2. PCA score plots for the top and bottom surface of the control (C) and treated (T) samples with 1 L coating.

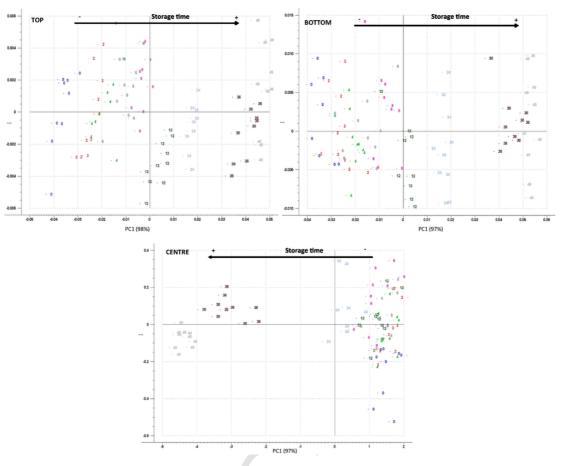


Fig. 3. PCA score plots for the samples (1 L) along storage time for different bread surfaces.

Table 5 PLS regression parameters for moisture content.

Sampl	Sample		Calibration		Cross-Validation		Test Set Validation	
		R ²	RMSEC	R ²	RMSECV	R ²	RMSET	
1 L	Тор	0.949	0.87	0.948	0.9	0.938	0.9	
	Bottom	0.957	0.72	0.955	0.77	0.947	0.85	
	Centre	0.969	1.31	0.959	1.4	0.946	1.52	
2L	Тор	0.915	1.01	0.852	1.31	0.8451	1.36	
	Bottom	0.901	1.32	0.899	1.35	0.862	1.45	
	Centre	0.957	1.51	0.945	1.72	0.921	1.88	

chewiness (N) was calculated from product of hardness, cohesivity and springiness.

2.6. NIR spectroscopy

The control and coated samples were submitted to near-infrared spectroscopy (NIR) for acquisition of the spectra using a FT-NIR spectrophotometer (MATRIX TM –F, Bruker Optics) in diffuse reflectance in the range of 800 and 2500 nm (8 cm^{-1} resolution). An average of 32 scans was obtained for each sample at intervals of storage by placing the optical fiber probe (IN 261, Bruker Optics, Mass., USA) in direct contact with the bread surface. The probe is characterized by a diameter of 10 mm with a bifurcated fiber bundle to illuminate the sample and collect the refracted light. For each spectral wave, the background was acquired by placing the probe in the specific support covered with quartz under the same environmental conditions as the sample (25 °C) and subtracted from sample spectra. The bread was scanned at 3 central points of the upper crust, lower crust and crumb; subsequently for

Table 6

PLS regression parameters for mechanical parameters.

Sample	Mechanical parameter	Calibration	Calibration		Cross-Validation		Test Set Validation	
		R ²	RMSEC	R ²	RMSECV	R ²	RMSET	
1L	Hardness (N)	0.947	5.4	0.886	8.1	0.878	8.8	
	Chewiness (N)	0.974	1.4	0.901	2.9	0.879	3.4	
2 L	Hardness (N)	0.973	6.3	0.924	10.6	0.915	10.7	
	Chewiness (N)	0.969	2.2	0.915	3.7	0.892	3.7	

each surface, the average spectrum of the three acquisitions was calculated.

2.7. Data analysis

Significant differences between means of mechanical parameters, weight loss and moisture content at different storage times were explored by using ANOVA (post-hoc Tukey HSD, p-level <0.05). Kruskal-Wallis test was used if significant differences emerged between variance means of the Levene test (Statistica-StatSoft, version 7). Pearson's analysis, (p-level <0.05) was performed to evaluate the correlation between moisture content and mechanical parameters. Furthermore, on the base of the results obtained by the Pearson's analysis, specific power law relations between moisture content and mechanical properties were evaluated (p < 0.05).

The NIR spectra acquired on different bread samples were analyzed by multivariate analyses (The Unscrambler ver. 9.7, CAMO, Oslo, Norway). The first part of the spectra until 1200 nm was deleted as it contains no useful chemical information, but only instrumental noise. The absorbance data were normalized by using the Standard Normal Variate (SNV) technique. To remove the effects of light scattering, spectra were pre-treated with multiplicative scattering correction (MSC). To reduce the noise and obtain more band information, the absorbance data were treated by applying the first derivative (Savitsky-Golay). Furthermore, spectral data were subjected to Principal Component Analysis (PCA) and Partial Least Square (PLS) regression to discriminate the samples as function of different storage times, and to predict moisture content and mechanical parameters, respectively. The predictive power of the obtained models were tested by analyzing the calibration results and by performing the full cross and test set validation (30% of the samples). Randomly selecting the samples, the models were solved 5 times and the mean results were analyzed in terms of determination coefficient (R²) and Root Mean Square Error (RMSE). To avoid the model over-fitting, optimal number of latent factors was identified by plotting the root mean square error as a function of the number of factors (minimum of the curve).

3. Results & discussions

3.1. Weight loss and moisture content

Water is an important aspect of bread which directly determines the consumer perception of freshness. Water within the product is redistributed from crumb to crust and subsequently lost to the environment during storage, causing changes in crust/crumb moisture and weight. Table 1 presents the weight loss data for the Control (C), 1L and 2L coated mini-buns increasing significantly for all samples along the storage time. However, the control samples registered a marginal of 1-2% higher weight loss than the coated samples irrespective of the number of layers. Subsequently, the moisture content decreased following an exponential trend with the final moisture loss being 66.1% for control and 62.8%–64.2% for 1 L and 2 L coated samples, respectively with 2 L coating exhibiting significantly lower moisture loss. It is expected that the moisture content decreases along the storage time and specific behavior with respect to the surface was not observed concerning the top and bottom surfaces. However, a slight decrease in the moisture loss by 2L coating was observed that might be indicative of retaining effect, as observed in dietetic cakes covered with pectin films by Baeva and Panchev (2005).

3.2. Mechanical parameters

The effect of the coatings in controlling the staling has been evaluated by textural parameters namely hardness, cohesivity and chewiness

as presented in Table 2 until 24h of storage. Texture is an important characteristic for the acceptance of bread and the parameter hardness is strongly correlated to the consumer purchase decision. The redistribution of moisture from crumb to crust and the retrogradation of starch are the main factors for hardening of the crumb (Fadda et al., 2014; Gray and Bemiller, 2003). A significant increase in the bread hardness and chewiness has been observed with the increase in storage time as expected (He and Hoseney, 1990; Licciardello et al., 2014). The hardness of the coated sample was significantly higher than their respective control at time '0h'. This can be probably due to the fact that whole bread was considered including the crust, to have similar effect as to when a bun is consumed. The addition of coating as expected formed an individual layer on the top of the crust which might have contributed to the additional hardness. Although the coated sample had higher initial hardness, considering the degree of increase in hardness until 24h the control sample registered higher value (Control: 271.8%) than those of the coated samples (1L: 126.5% and 2L: 231.2%), confirming the retaining effect of the coating. Similar inferences on hardness have been observed by Eom et al. (2018) with starch/gum based edible coating for rice cakes. However, cohesivity decreased during storage, irrespective of the presence or absence of coating. Subsequently, chewiness related to the reinforcement and dehydration of bread increased along storage time. This trend is similar to that of the hardness values with a higher increase in the control in comparison to the coated samples.

Furthermore, the moisture and textural parameters correlation was investigated by using Pearson test (Table 3). As can be observed the moisture content of crust and crumb are positively correlated for both 1 L (r = 0.86-0.90) and for 2 L (r = 0.51-0.76). Hardness and chewiness were negatively correlated to the moisture, with a stronger correlation to crumb moisture (r = 0.91-0.96) for both the layers studied. However, cohesivity (Co) was observed to be negatively correlated to the hardness and chewiness irrespective of sample. This observation is similar to that observed by Chin et al. (2011) in sweet buns with different types of glazing. Power law relations $(y = ax^b)$ between top moisture content (x) and hardness or chewiness (y) were evaluated for 1 L, 2L and control samples, respectively. The equation parameters are reported in Table 4. The best fit was obtained for the samples with one layer of coating (R2 = 0.97), while the worst for the control samples (R2 = 0.89). Considering the strong correlation between hardness and chewiness (r = 0.98-1), very similar results, in terms of determination coefficients, were obtained for these two parameters belong to the same sample.

3.3. NIR spectroscopy

The characteristic near infrared spectra of coated bread at different storage times are shown in Fig. 1. In particular, resonance bands of O–H bonds related to water and starch were observed at about 1450 and 1940 nm, and N–H bonds vibrations at 1500–1570 nm and 2050–2070 nm. The absorption peaks at 1730, 1770 and 2310 nm were ascribed to the first overtone of the C–H stretch and to C–H₂ group of the lipids (Cevoli et al., 2015; Giangiacomo, 2006; Nallan Chakravartula et al., 2019).

The PCA score plots for the NIR spectra were performed to discriminate between control samples (C) and coated bread samples (T). Both top and bottom crust surfaces along with crumb were considered. The best results were obtained by using the SNV spectral pre-treatment followed by the Savitzky–Golay. As reported in Fig. 2, a clear separation between samples was observed along the PC3 (1%). Through the analysis of the X-loading it was possible to observe that the highest variance (PC3) corresponds to 2050–2070 nm spectral range (N–H bonds). This is probably due to the protein contributed by the coating on the treated samples. Similar results were obtained for both one and two layers of coating (data not shown).

Considering the storage time, it can be observed that the spectral absorbance decreases in the region related to O–H bonds and reciprocatively increases in the typical spectral range of the fats and proteins, (N–H, C–H and C–H₂ bonds). The PCA score plots (PC1 *vs.* PC2) developed to discriminate the samples on the basis of the storage time are reported in Fig. 3, for the top, bottom crust and centre crumb, respectively. The samples are arranged along the PC1 (explained variance 98%, 97% and 97%) on the basis of the storage time. A clear separation between samples was observed, for the top and bottom surface, especially after 8 h of storage. With regard to the centre, discrimination was detected only after 24 h of storage as constant moisture content was observed for two days of storage. Following this, the spectral data were subjected to PLS to estimate the moisture content and mechanical parameters, respectively.

In general, for the moisture content, satisfactory results were achieved for one and two coating layers and bread position (top, bottom and centre) with a R^2 in test set ranging from 0.938 to 0.947, and from 0.845 to 0.921 respectively. Analyzing the calibration, the full cross and the test set validation, the best models concern the sample characterized by one layer of coating (top: $R^2 = 0.938$, RMSET = 0.9%; bottom: $R^2 = 0.947$, RMSET = 0.85%; centre: $R^2 = 0.946$, RMSET = 1.52%).

Concerning the mechanical parameters, hardness and chewiness were taken into account and PLS models were developed analyzing the NIR spectra acquired on the bread top. Also in this case, good results were obtained in terms of R² and RMSE for calibration, cross-validation and test set validation, respectively. The best results were reached for the hardness of the samples with 2 layers of coating ($R^2 = 0.915$, RM-SET = 10.7 N). Recently, Ringsted et al. (2017) reported correlation values (R²) between bread hardness measured during 7 days of storage, and near-infrared bands ranging from 0.88 to 0.97. More weak results were obtained by Xie et al. (2003) developing a PLS model (NIR spectra) to predict the firmness of bread stored at 27 °C for 5 days ($R^2 = 0.80$ in calibration). With regard to flat bread tensile tests and near-infrared reflectance spectroscopy were used to monitor flat bread ageing treated by preservatives. Al-Mahsaneh et al. (2018) reported high R² values and confirmed that the NIRS along with texture analysis are valuable tools to detect the effect of the preservatives on shelf-life and quality of flat bread.

4. Conclusions

The results of the presented study show that the application of coating has a retaining effect on the product to some extent. The NIR spectroscopy was useful to predict the textural responses during storage on basis of both changes in moisture and protein bands. The principal component analysis of the NIR spectra was useful for differentiating the samples based on coating application. Further, PLS models were developed to estimate the moisture and texture which might be useful to track staling. The practical knowledge on coatings effect on staling is useful to develop potential solutions to preserve bread with higher efficiency and lower packaging impact.

Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

Declarations of interest

None.

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Glossary

- 1L : Single layer of coating
- 2L: Two layers of coating
- C: Control bread samples
- T: Coated bread samples
- *MC* : Moisture Content *PC* : Principal Components
- *RH* : Relative humidity
- *MSC*: Multiple scatter correction
- NIR : Near Infra-red
- PCA : Principal Component Analysis
- PLS : Partial Least Square
- SVN: Standard normal variate
- RMSE : Root mean square error
- RMSEC : Root mean square error Calibration set
- RMSECV : Root mean square error Cross Validation set

RMSET : Root mean square error Test set