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Elena Curti[1,2*], Eleonora Carini[2], Elena Vittadini[2]

[1]Siteia.Parma Interdepartmental Centre, University of Parma, Parco Area delle Scienze, 181/a, 43124 Parma, Italy

[2]Department of Food Science, University of Parma, Parco Area delle Scienze, 47/A, 43124, Parma, Italy

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*Corresponding author. Parco Area delle Scienze 47/A, University of Parma, 43124 Parma, Italy; elena.curti@unipr.it; Tel. +39–0521–906234, Fax +39–0521-906028

Abstract

Bread staling reduction is a very important issue for the food industry. A fiber with high water holding capacity, extracted from potato peel, was studied for its ability to reduce bread staling even if employed at low level (0.4 g fibre / 100 g flour). Physico-chemical properties (water activity, moisture content, frozen water content, amylopectin retrogradation) and ^{1}H Nuclear Magnetic Resonance molecular mobility were characterised in potato fibre added bread over 7 days of storage. Potato fibre addition in bread slightly affected water activity and moisture content, while increased frozen water content and resulted in a softer bread crumb, more importantly when the optimal amount of water was used in the formulation. Potato fibre also reduced ^{1}H NMR molecular mobility changes in bread crumb during storage. Potato fibre addition in bread contributed to reduce bread staling.

1. Introduction

Bread staling is a process occurring during storage of the product, that results in crumb hardening, crust softening and loss of the characteristic fresh flavour of the product (Gray & Bemiller, 2003). Different phenomena contribute to bread staling: starch recrystallization is one of the factors contributing to crumb hardening, as well as gluten dehydration and its consequent loss of plasticity, and modified gluten-starch interactions. Water plays a fundamental role in bread staling and, hence, the study of water status and its dynamics is very important to better understand the bread staling phenomenon. Water migrates from crumb to crust at a macroscopic level and redistributes at a molecular level, becoming partially incorporated in starch crystals, looses phase separating capability (decreased "DSC freezable water" content) and is redistributed among bread domains (Baik & Chinachoti, 2001; Curti, Carini, Tribuzio, & Vittadini, 2014; Schiraldi & Fessas, 2001; Slade & Levine, 1991; Vittadini & Vodovotz, 2003).

Addition of large amounts of fibre into bread to produce high fibre products has been object of much research in an effort to improve customers' fiber intake but it is often detrimental to bread quality (Chen, Rubenthaler, Leung, & Baranowski, 1988; Katina, Salmenkallio-Marttila Partanen, Forssell, & Autio, 2006). Large amounts of fibre are known to negatively modify dough and bread properties, production process, and staling-related phenomena (e.g. gluten dehydration, amorphous starch recrystallization, water molecular redistribution among bread components; Collar, Santos & Rosell, 2007; Fadda, Sanguinetti, Del Caro, Collar & Piga, 2014; Gray & Bemiller, 2003). However, with the selection of the proper type of fibre and proper technological fibre treatment, fibre addition can improve bread properties and retard staling (Laurikainen, Harkonen, Autio, & Poutanen, 1998; Sangnark & Noomhorm, 2003; Sangnark & Noomhorm, 2004; Wang, Rosell & Benedito de Barber, 2002).

75 Potato peel, a by-product from the potato industry, has been reported to be a very rich
76 (higher than wheat bran) and good source of fibre with high water-holding capacity
77 (Camire & Flint, 1991; Camire, Violette, Dougherty & McLaughlin, 1997).
78 Few works considered the effect of potato peel as a source of fibre in bread (Toma, Orr,
79 D'Appolonia, Dintzis, & Tabekhia, 1979) and cakes (Sharoba, Farrag, & El-Salam, 2013),
80 reporting higher farinograph absorptions, reduced gas retention and volumes, as well as
81 increased hardness in the products. In all these reports potato peel was added in large
82 amounts (5-20%) to bread formulations in an effort to increase bread fibre content. Based
83 on its characteristics and properties, potato fibre may also have an effect on water status
84 and dynamics, possibly retarding and modulating bread staling.
85 The aim of the present work is to investigate a potential technological use of potato fibre in
86 improving bread physico-chemical properties and reduce bread staling. Potato fibre was,
87 therefore, added in small amount into a bread formulation, and its effect on physico-
88 chemical properties and water status of the product was studied during storage.
89 **2. Materials and methods**
90 *2.1. Bread formulation, production and storage*
91 Three breads were produced. The control sample was named STD and it was produced
92 with optimal water amount (500 Brabender Units). The formulations [wheat flour (Molino
93 Seragni, Cremona, Italy); sugar (Coprob S.C.A, Pavia, Italy); salt, (Italkali s.p.a., Palermo,
94 Italy); yeast (AB Mauri Italy s.p.a, Padova, Italy); sunflower seeds oil (Oleificio Zucchi,
95 Cremona, Italy)] are reported in Table 1.
96 Potato fibre (HI-FIBRE 115, HI-FOOD S.p.a. Collecchio, Italy), extracted from the potato
97 peel and very rich in soluble components, was added to the formulation at 0.4% on a flour
98 basis (g fibre/ 100 g flour) to produce two samples, P-W and P-STD. The water absorption
99 used for P-W was increased of 4%, according to preliminary trials aiming at identify the
100 conditions (fibre and water level) to obtain an optimized final product in terms of volume,

101 colour, and texture. P-STD was produced with the same amount of water used for STD to
102 clearly highlight the effect of fiber. Potato fibre composition (as indicated by the producer)
103 was as follows: ~ 6.0% (g / 100 g fiber) moisture, protein < 1.0 %, fat < 1.0%,
104 carbohydrates < 1.0 %, dietary fiber ~ 92.0 % (soluble fiber ~ 73.0 %; insoluble fiber ~ 19.0
105 %), ashes ~ 2.0 %.
106 Breads were produced with a home bread-maker (Backmeister 68511, UNOLD, Germany)
107 using a “basic” program (pre-heating 17 min; first kneading 5 min; second kneading 13
108 min; first fermentation 45 min; smoothing 1 min; second fermentation 18 min; smoothing 1
109 min; third fermentation 45 min; baking 55 min), cooled to room temperature, placed in
110 polyethylene bags sprinkled with about 3 ml ethanol, and stored at room temperature.
111 Samples (three loaves for each sample for each storage time) were analyzed fresh (day 0)
112 and after 1, 3, 5 and 7 days of storage.
113 *2.2. Volume and texture*
114 Volume was measured on three bread loaves for each sample following the American
115 Association Cereal Chemistry 10-05 method (Guidelines for Measurement of Volume by
116 Rapeseed Displacement).
117 Bread crumb hardness was measured with a TA.TX2 Texture Analyzer (Stable Micro
118 Systems, Goldalming, UK). At least eight cubic portions (2 x 2 x 2 cm^3) of crumb were
119 extracted from the central slices of the bread loaf and compressed (force = 0.1 N) to 40%
120 deformation using a cylindrical probe (P/35 Dia Cylinder Aluminium). Crumb texture was
121 described in terms of Hardness (maximum height of the first compression peak) and
122 Cohesiveness (ratio of the areas of the second to the first compression peak).
123 *2.3. Water activity and moisture content*
124 Water activity of crumb (from loaf centre) and crust was measured with a dew point
125 instrument (Aqualab 4TE, Decagon Devices, WA, USA). At least five measurements were
126 taken for each sample. Moisture content (MC) of crumb (from loaf centre) and crust were

127 determined in triplicate for each bread loaf by weight loss at 105°C (NSV 9035, ISCO,
128 Milan, Italy) to constant weight.

129 *2.4. Frozen water content and retrograded amylopectin*

130 Crumb thermal properties were measured with a Differential Scanning Calorimeter (DSC
131 Q100 TA Instruments, New Castle, DE, USA), calibrated with indium and mercury. Bread
132 crumb (4 g, from loaf centre) was properly compressed to obtain a flat and compact crumb
133 sample to maximize heat transfer within the DSC cell during the experiment. Samples (5-
134 10 mg) were taken and placed in stainless steel pans (Perkin Elmer, USA) that were then
135 hermetically sealed, quench cooled to -80°C and heated at 5°C/min to 130°C. DSC
136 thermograms were analysed (Universal Analysis Software 3.9A, TA Instruments, New
137 Castle, DE). “Frozen” water (at the given experimental conditions; FW) was calculated
138 from the endothermic peak around 0°C (ice melting) using the following equation:

139 $$\text{FW} = \text{Enthalpy Ice Fusion} \times \left(\frac{1}{latent\ heat\ ice\ fusion}\right) \times \left(\frac{1}{MC}\right) \times 100$$

140 Where FW is Frozen water at the given experimental conditions (g frozen water/100 g
141 water), Enthalpy Ice Fusion (J / g product), Latent heat of ice fusion is 334 J / g ice and
142 MC is Moisture Content (g water/ g product).

143 Retrograded amylopectin (J/g sample) was obtained by the integration of the endothermic
144 peak in the 50-80°C temperature range.

145 *2.5. Molecular mobility (^{1}H NMR)*

146 A low resolution (20 MHz) ^{1}H NMR spectrometer (the MiniSpec, Bruker Biospin, Milano,
147 Italy) operating at 25.0 ± 0.1 °C was used to measure the Free Induction Decay (FID) and
148 the transverse (T_2) relaxation times of the samples. Crumb samples (10 mm high) were
149 prepared in 10 mm NMR tube, sealed with Parafilm® to prevent moisture loss during the
150 NMR experiment.

FIDs were acquired using a single 90° pulse, followed by a dwell time of 7 μs, 32 scans and a recycle delay of 3 s and a 10 ms acquisition window. ^{1}H FIDs were analyzed in the time range 7 μs -100 μs where the homogeneity of magnetic field was assured. Fitting of FID was carried out with a two components model (exponential and gaussian, LeBotlan & Helie-Fourel, 1995; Sigmaplot, v6, Systat Software Inc. USA):

$$I(t) = y0 + A * \exp[-(t / T_A)] + B * \exp[-(t / T_B)^{\wedge 2}]$$

where y0 is the FID decay offset, A and B are the are intensities of each relaxation component, T_A and T_B are the apparent relaxation times.

T_2 relaxation times were measured with a CPMG pulse sequence with a recycle delay of 3 s (≥ 5 ^{1}H T_1), an interpulse spacing of 0.04 ms and 4000 data points. Quasi-continuous distributions of relaxation times were obtained from the experimental T_2 curves using a UPENWin software (Alma Mater Studiorum, Bologna, Italy). Default values for all UPEN parameters were used with the exception of one (LoXtrap) that was set to 1 to avoid extrapolation of relaxation times shorter than the first experimental point.

2.6. Statistical analysis

One-way-analysis of variance (ANOVA, SPSS v.20, IBM, NJ, USA), followed by least significant difference test (LSD) at $p \leq 0.05$, was used to verify significant differences of evaluated parameters of the same sample during storage and among the samples at the same storage time.

3. Results and discussion

3.1. Water activity and moisture content

Crumb and crust water activity and moisture content of samples are shown in Table 2.

Crust water activity (Figure 1a) was significantly different in the fresh breads: it was significantly higher in P-W (~0.80) than in P-STD (~0.74) but statistically not different to STD (~0.78). Crust water activity increased during storage in all samples, due to moisture migration from crumb to crust.

177 Crumb water activity (Figure 1a) at day 0 showed very slight statistical differences (STD
178 and P-STD: ~0.96, P-W: ~0.97), and samples were, therefore, considered not different for
179 this parameter. Crumb water activity generally decreased during storage without important
180 changes in all samples, at day 7 P-STD had a water activity slightly lower than STD and P-
181 W.
182 Crust moisture content was significantly higher in potato fibre added breads (~15%, g
183 water/100 g sample) as compared to STD (~13%), increased during storage in all
184 samples, and was not statistically different at the end of storage (Figure 1b). Crumb
185 moisture content of fresh breads was only slightly higher in P-W and STD (~41%) than in
186 P-STD (~39%), as previously reported for other fibres (Dalgetty, & Baik, 2006).
187 Crumb moisture content significantly decreased in all samples, as a consequence of the
188 macroscopic migration of water from the wetter crumb to the drier crust. However, this
189 decrease was limited due to the fact that crumb represented, weight wise, the majority of
190 the bread loaf and crust only a small portion. Moreover most of the water migrating from
191 the crumb to the crust would come from the portion of crumb closer to the crust. At the end
192 of storage STD (~39%) and P-W (~40%) had significantly larger crumb moisture content
193 as compared to P-STD (~37%).
194 *3.2. Crumb texture*
195 All bread samples (STD, P-STD and P-W) had comparable loaves volume (data not
196 shown).
197 Hardness and cohesiveness of bread crumbs during storage are shown in Figure 1a and
198 1b. Crumb hardness was comparable in fresh samples, and it significantly increased in all
199 products during storage, as expected. Crumb hardness of STD and P-STD increased
200 more pronouncedly during storage than in P-W. P-W was the softest bread (~2.6 ± 0.6 N),
201 followed by P-STD (~3.7 ± 0.6 N) and STD (~4.5 ± 0.7 N) at the end of storage. Only slight
202 differences in MC were observed in the 7-days old STD and P-W, hence the improved

texture observed in P-W was not relatable to MC. Cohesiveness was comparable in fresh samples and decreased similarly in all samples during storage.

Previous works investigated bread formulations where soluble fibre was added in limited amounts (1-5%) with the aim to improve bread properties. Soluble fibre has been reported to increase crumb firmness and amylopectin retrogradation, at moisture contents larger than in the control sample (Gómez, Ronda, Blanco, Caballero, & Apesteguía, 2003; Skendi, Biliaderis, Papageorgiou, & Izydorczyk, 2010; Zhou et al., 2009). In our samples, softer crumb was observed also in absence of larger MC (P-STD), suggesting that the high water holding capacity of the potato fibre may have positively influence the texture of the product.

3.3. Thermal analysis

Thermograms of fresh bread crumbs (from −80 to 130°C, data not shown) showed a major endothermic peak around 0°C and, in stored samples a second minor endothermic event occurring at higher temperatures (50–80°C). The major DSC peak around 0°C was attributed to ice melting and the relative enthalpy was used to calculate the frozen water content (FW%) of samples (Table 2).

At day 0 FW was significantly larger in P-W (~57 ± 5 %, g frozen water / 100 g water) and P-STD (~61 ± 4 %), than in STD (~51 ± 4 %), indicating that, with very slight differences in the MC, the availability of water, as measured by DSC, was influenced by the presence of potato fibre, that possibly acted on water-solids interactions (as observed in this time-frame experimental window). FW significantly decreased in all samples, to ~55%, ~52% and ~47% in P-W, P-STD and STD, respectively, as previously reported (Ribotta & Le Bail, 2007). The differences observed in macroscopic water parameters (MC and FW) indicated that water molecules interacted more loosely in the bread matrix in the presence of potato fibre (P-W and P-STD), suggesting that potato fibre enhanced water macromolecular mobility (Curti, Carini, Bonacini, Tribuzio, & Vittadini, 2013).

229 The endothermic peak (50-80°C temperature range) observed in the thermograms of
230 stored bread crumb was attributed to recrystallised amylopectin melting (Baik &
231 Chinachoti, 2000; Russell, 1983). No endothermic peak was observed in the fresh and 1-
232 day stored samples while at longer storage times the peak became increasingly larger
233 (Figure 1c). Recrystallised amylopectin at day 7 was comparable among the samples (1.7
234 ± 0.2, ~1.5 ± 0.1 and ~1.5 ± 0.4 J/g sample, in STD, P-W and P-STD respectively)
235 indicating that potato fibre had no particular influence on amylopectin retrogradation,
236 despite its high water holding capacity, as previously reported in soluble fibre enriched
237 bread (Dalgetti & Baik, 2006; Skendi, Biliaderis, Papageorgiou, & Izydorczyk, 2010). Softer
238 crumbs in P-W and P-STD were not related to retrograded amylopectin (that was
239 comparable) and MC (lower in P-STD as compared to P-W), while it might be associated
240 to a stronger water retention of potato fibre and an 'higher water availability' as suggested
241 by the larger FW.

242 *3.4. Molecular mobility*

243 Molecular mobility characterization was carried out for, the fastest-relaxing ^{1}H
244 components, with a ^{1}H FID NMR experiment, while slower relaxing protons were
245 characterised in terms of ^{1}H T_2 relaxation times distributions.

246 Representative ^{1}H FIDs of bread object of this study are reported in Figure 2a. At day 0 ^{1}H
247 FIDs of STD and P-STD were sharper than in P-W, despite their comparable MC,
248 suggesting an increased molecular mobility in this sample. During storage all ^{1}H FIDs
249 increased their steepness as previously reported in bread (Curti et al., 2014; Sereno, Hill,
250 Mitchell, Scharf, & Farhat, 2007), due to moisture loss from the crumb and increasing
251 recrystallised amylopectin content. At day 7, ^{1}H FID were comparable in STD and P-W,
252 while P-STD still showed the steepest decay.

253 ^{1}H FID curves were fitted with a two components model (exponential and Gaussian
254 function; Figure 2b and 2c) to obtain quantitative information about the relaxation time and

255 percentage of protons belonging to the more rigid and more mobile proton populations
256 detectable within the FID experimental time-frame. In fresh breads, the more rigid
257 component (population A, relaxing at 0.016-0.018 ms, T_A) represented ~16-17% of the
258 total protons in STD and P-W while it was larger (~20%) in P-STD although with
259 comparable relaxation time, indicating a reduced molecular mobility in this sample. The
260 more mobile component (population B, relaxing at 1.5-2.0 ms, T_B) was, specularly, less
261 represented in P-STD than in STD and P-W. T_A and T_B relaxation times remained constant
262 during storage while the amount of protons belonging to population A increased in all
263 samples with increasing storage time, indicating an increasing molecular rigidity of bread
264 crumb. At the end of storage P-STD was the product with a larger degree of rigidity, as its
265 population A increased to ~36%, while it represented only ~30% of the protons of P-W and
266 STD.
267 The presence of two proton populations in ^{1}H FID was previously reported model systems
268 (heated dough) and bread (Bosmans, et al., 2012): the fastest relaxing protons were
269 associated to the protons of crystalline and amorphous starch not in contact with water
270 and to protons of amorphous starch and gluten in little contact with water, respectively.
271 The differences observed at day 7 in the ^{1}H FID populations of the breads object of this
272 study appeared not to be attributable to both moisture content loss and amylopectin
273 retrogradation (that was comparable), as previously reported (Farhat, Ottenhof, Marie, &
274 De Bezenac, 2003; Sereno et al., 2007), but only to the different moisture contents.
275 A representative ^{1}H T_2 distribution is shown in Figure 3a. Three ^{1}H T_2 protons populations
276 were observed in all samples and they were named starting from the shortest to the
277 longest relaxation time C, D and E, respectively (Figure 3b and 3c). The overall shape of
278 the ^{1}H T_2 relaxation time distributions did not change in all samples during storage.
279 Population C represented protons relaxing at ~ 0.5 ms (T_{2C}), population D protons relaxed
280 at ~ 9-11 ms (T_{2D}) while population E protons were characterised by relaxation times of

281 about 130 ms ($T_{2E;}$ Figure 3b). Comparable relaxation times were found among all fresh
282 samples. In STD, population C encompassed ~ 29% of total protons, population D ~ 66 %,
283 and population E ~ 5%. P-STD showed a comparable (~30%) presence, to STD, of the
284 more rigid protons while these protons were less abundant in P-W (~26%) (Figure 3c). On
285 the contrary, a smaller presence of protons belonging to population D was observed in P-
286 STD (~65%) than in P-W (~70%). No significant changes in mobility of populations C and
287 E were detected (comparable T_{2C} and T_{2E} at all storage times) during storage in all
288 samples, while population D shifted towards shorter relaxation times (T_{2D} significantly
289 decreased to ~ 6-8 ms) in all samples (Figure 3b and 3c). Protons population abundances
290 showed slight changes in STD, where population C decreased to ~26% and population D
291 increased to ~69% (Figure 3b and 3c) during 7 days of storage.
292 Previous studies reported the presence of multiple proton populations in white bread.
293 Changes in proton mobility have been related to macroscopic and mesoscopic staling
294 phenomena, such as water migration, starch recrystallization and crumb firming. In
295 particular increasing rigidity (larger amount in the FID fast relaxing protons and T_2
296 population D) has been attributed to water loss, starch crystals formation, gluten
297 dehydration (Bosmans et al., 2012; Bosmans, Lagrain, Ooms, Fierens, & Delcour, 2013;
298 Engelsen et al., 2001; Sereno et al., 2007; Wang, Choi, & Kerr, 2004).
299 It was hypothesised that potato fibre, especially when the optimum water amount was
300 used in the formulation (P-W), contributed to reduce molecular mobility changes during
301 storage, possibly limiting crumb firming.

302 **4. Conclusions**

303 Potato fibre (extracted from potato peel) was found to have an important effect, when
304 added in limited amounts (0.4%), on bread properties during storage. Texture of the
305 products was improved (bread was softer), in particular when the optimum amount of
306 water was used in the bread formulation (P-W). The addition of potato fibre slightly

affected water parameters, in terms of water activity and moisture content. DSC analysis showed a larger presence of frozen water and reduced retrograded amylopectin in potato fibre added breads. ^{1}H NMR mobility was affected by the presence of potato fibre: the fast-relaxing protons (^{1}H FID population A and ^{1}H T_2 population C) were less represented in the bread produced with the optimal water amount, indicating a decreased rigidity, that was maintained during storage.

Potato fibre improved the texture of bread, possibly by a retention of water allowing for the maintenance of a softer crumb texture during storage.

More information should be collected to better understand the nature of potato fibre interactions with other bread components (e.g. starch). The effect of higher amounts of potato fibre on bread staling could also be considered, to evaluate the optimal level of addition that represents the compromise between technological and sensory quality. The implementation of potato fiber addition at industrial level should be in last instance carried out.

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434

435 **Figure Captions**

436 **Fig.1**. Hardness (a), Cohesiveness (b) and Amylopectin retrogradation (c) of STD (black),
437 P-W (white) and P-STD (grey) during storage[a]

438

439 **Fig.2**. Representative ^{1}H FIDs (a) of STD (black), P-W (white) and P-STD (grey) at day 0
440 (circles) and day 7 of storage (squares); ^{1}H FID relaxation times (b) and populations (c) of
441 STD (black), P-W (white) and P-STD (grey) during storage.

442

443 **Fig. 3**. Representative ^{1}H T_2 relaxation time distribution of samples (a); ^{1}H T_2 relaxation
444 times (b) and populations (c) of STD (black), P-W (white) and P-STD (grey) during
445 storage.

[a]Statistical significance: small letters indicate significant differences among samples with the same formulation at different storage times; capital letters indicate significant differences among samples with different formulation at the same storage time; n.d. Not detectable; (-) No significant difference)