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Original The use of potato fibre to improve bread physico-chemical properties dur Eleonora; Diantom, Agoura; Vittadini, Elena Giovanna Piera In: FOOD CH 195:(2016), pp. 64-70. [10.1016/j.foodchem.2015.03.092]	
Availability: This version is available at: 11381/2799269 since: 2021-12-02T11:11:52Z	
Publisher: Elsevier Ltd	
Published DOI:10.1016/j.foodchem.2015.03.092	
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1	The use of potato fibre to improve bread physico-chemical properties during
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12	Keywords : potato fibre, bread staling, ¹ H NMR molecular mobility, physico-chemical
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15	Running title: Potato fibre in bread staling
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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 ¹H 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 ¹H NMR molecular mobility changes in bread crumb during storage. Potato fibre addition in bread contributed to reduce bread staling.

1. Introduction

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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).

- 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
- on its characteristics and properties, potato fibre may also have an effect on water status
- and dynamics, possibly retarding and modulating bread staling.
- 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.

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: $\sim 6.0\%$ (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)
- using a "basic" program (pre-heating 17 min; first kneading 5 min; second kneading 13
- 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.
- Samples (three loaves for each sample for each storage time) were analyzed fresh (day 0)
- 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 cm³) of crumb were
- extracted from the central slices of the bread loaf and compressed (force = 0.1 N) to 40%
- 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
- instrument (Aqualab 4TE, Decagon Devices, WA, USA). At least five measurements were
- 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
- 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 FW = 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).
- 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 (¹H NMR)
- 146 A low resolution (20 MHz) ¹H 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
- the transverse (T₂) relaxation times of the samples. Crumb samples (10 mm high) were
- prepared in 10 mm NMR tube, sealed with Parafilm® to prevent moisture loss during the
- 150 NMR experiment.

- 151 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. ¹H FIDs were analyzed in the
- 153 time range 7 μs -100 μs where the homogeneity of magnetic field was assured. Fitting of
- 154 FID was carried out with a two components model (exponential and gaussian, LeBotlan &
- Helie-Fourel, 1995; Sigmaplot, v6, Systat Software Inc. USA):
- 156 $I(t) = y0 + A * \exp[-(t/T_A)] + B * \exp[-(t/T_B)^2]$
- 157 where y0 is the FID decay offset, A and B are the are intensities of each relaxation
- 158 component, T_A and T_B are the apparent relaxation times.
- 159 T₂ relaxation times were measured with a CPMG pulse sequence with a recycle delay of 3
- 160 s (≥ 5 ¹H T₁), an interpulse spacing of 0.04 ms and 4000 data points. Quasi-continuous
- 161 distributions of relaxation times were obtained from the experimental T₂ curves using a
- 162 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.
- 165 *2.6. Statistical analysis*
- One-way-analysis of variance (ANOVA, SPSS v.20, IBM, NJ, USA), followed by least
- significant difference test (LSD) at $p \le 0.05$, 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
- 171 3.1. Water activity and moisture content
- 172 Crumb and crust water activity and moisture content of samples are shown in Table 2.
- 173 Crust water activity (Figure 1a) was significantly different in the fresh breads: it was
- 174 significantly higher in P-W (~0.80) than in P-STD (~0.74) but statistically not different to
- 175 STD (~0.78). Crust water activity increased during storage in all samples, due to moisture
- 176 migration from crumb to crust.

- 177 Crumb water activity (Figure 1a) at day 0 showed very slight statistical differences (STD
- and P-STD: ~0.96, P-W: ~0.97), and samples were, therefore, considered not different for
- 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
- 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
- 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
- the crumb to the crust would come from the portion of crumb closer to the crust. At the end
- 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),
- followed by P-STD ($\sim 3.7 \pm 0.6 \text{ N}$) and STD ($\sim 4.5 \pm 0.7 \text{ N}$) 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.

213 3.3. Thermal analysis

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214 Thermograms of fresh bread crumbs (from -80 to 130°C, data not shown) showed a major 215 endothermic peak around 0°C and, in stored samples a second minor endothermic event 216 occurring at higher temperatures (50–80°C). The major DSC peak around 0°C was 217 attributed to ice melting and the relative enthalpy was used to calculate the frozen water 218 content (FW%) of samples (Table 2).

219 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 220 221 the MC, the availability of water, as measured by DSC, was influenced by the presence of 222 potato fibre, that possibly acted on water-solids interactions (as observed in this time-223 frame experimental window). FW significantly decreased in all samples, to ~55%, ~52% 224 and ~47% in P-W, P-STD and STD, respectively, as previously reported (Ribotta & Le Bail, 225 2007). The differences observed in macroscopic water parameters (MC and FW) indicated 226 that water molecules interacted more loosely in the bread matrix in the presence of potato 227 fibre (P-W and P-STD), suggesting that potato fibre enhanced water macromolecular 228 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 \pm 0.2, ~1.5 \pm 0.1 and ~1.5 \pm 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 ¹H
- 244 components, with a ¹H FID NMR experiment, while slower relaxing protons were
- 245 characterised in terms of ¹H T₂ relaxation times distributions.
- 246 Representative ¹H FIDs of bread object of this study are reported in Figure 2a. At day 0 ¹H
- 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 ¹H FIDs
- 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
- recrystallised amylopectin content. At day 7, ¹H FID were comparable in STD and P-W,
- 252 while P-STD still showed the steepest decay.
- ¹H 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

percentage of protons belonging to the more rigid and more mobile proton populations detectable within the FID experimental time-frame. In fresh breads, the more rigid component (population A, relaxing at 0.016-0.018 ms, T_A) represented ~16-17% of the total protons in STD and P-W while it was larger (~20%) in P-STD although with comparable relaxation time, indicating a reduced molecular mobility in this sample. The more mobile component (population B, relaxing at 1.5-2.0 ms, T_B) was, specularly, less represented in P-STD than in STD and P-W. T_A and T_B relaxation times remained constant during storage while the amount of protons belonging to population A increased in all samples with increasing storage time, indicating an increasing molecular rigidity of bread crumb. At the end of storage P-STD was the product with a larger degree of rigidity, as its population A increased to ~36%, while it represented only ~30% of the protons of P-W and STD. The presence of two proton populations in ¹H FID was previously reported model systems (heated dough) and bread (Bosmans, et al., 2012): the fastest relaxing protons were associated to the protons of crystalline and amorphous starch not in contact with water and to protons of amorphous starch and gluten in little contact with water, respectively. The differences observed at day 7 in the ¹H FID populations of the breads object of this study appeared not to be attributable to both moisture content loss and amylopectin retrogradation (that was comparable), as previously reported (Farhat, Ottenhof, Marie, & De Bezenac, 2003; Sereno et al., 2007), but only to the different moisture contents. A representative ¹H T₂ distribution is shown in Figure 3a. Three ¹H T₂ protons populations were observed in all samples and they were named starting from the shortest to the longest relaxation time C, D and E, respectively (Figure 3b and 3c). The overall shape of the ¹H T₂ relaxation time distributions did not change in all samples during storage. Population C represented protons relaxing at ~ 0.5 ms (T_{2C}), population D protons relaxed at \sim 9-11 ms (T_{2D}) while population E protons were characterised by relaxation times of

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about 130 ms (T_{2E} ; Figure 3b). Comparable relaxation times were found among all fresh samples. In STD, population C encompassed ~ 29% of total protons, population D ~ 66 %, and population E ~ 5%. P-STD showed a comparable (~30%) presence, to STD, of the more rigid protons while these protons were less abundant in P-W (~26%) (Figure 3c). On the contrary, a smaller presence of protons belonging to population D was observed in P-STD (~65%) than in P-W (~70%). No significant changes in mobility of populations C and E were detected (comparable T_{2C} and T_{2E} at all storage times) during storage in all samples, while population D shifted towards shorter relaxation times (T_{2D} significantly decreased to ~ 6-8 ms) in all samples (Figure 3b and 3c). Protons population abundances showed slight changes in STD, where population C decreased to ~26% and population D increased to ~69% (Figure 3b and 3c) during 7 days of storage.

Previous studies reported the presence of multiple proton populations in white bread. Changes in proton mobility have been related to macroscopic and mesoscopic staling phenomena, such as water migration, starch recrystallization and crumb firming. In particular increasing rigidity (larger amount in the FID fast relaxing protons and T₂ population D) has been attributed to water loss, starch crystals formation, gluten dehydration (Bosmans et al., 2012; Bosmans, Lagrain, Ooms, Fierens, & Delcour, 2013;

298 Engelsen et al., 2001; Sereno et al., 2007; Wang, Choi, & Kerr, 2004).

It was hypothesised that potato fibre, especially when the optimum water amount was used in the formulation (P-W), contributed to reduce molecular mobility changes during storage, possibly limiting crumb firming.

4. Conclusions

Potato fibre (extracted from potato peel) was found to have an important effect, when added in limited amounts (0.4%), on bread properties during storage. Texture of the products was improved (bread was softer), in particular when the optimum amount of 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. ¹H NMR mobility was affected by the presence of potato fibre: the fast-relaxing protons (¹H FID population A and ¹H T₂ 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.

Acknowledgements

This work was partially supported by Emilia-Romagna Region (POR FSE 2007-2013). The authors would like to thank Maryam Asadzadeh for carrying out part of the analysis.

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- 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

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

- 443 **Fig. 3**. Representative ¹H T₂ relaxation time distribution of samples (a); ¹H T₂ relaxation
- 444 times (b) and populations (c) of STD (black), P-W (white) and P-STD (grey) during
- 445 storage.

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^aStatistical 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)