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RELATIONSHIP BETWEEN WATER ACTIVITY OF CRISP BREAD AND ITS MECHANICAL PROPERTIES AND STRUCTURE

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The aim of this work was to determine the effect of water activity on structure and mechanical properties of rye crisp bread and fibre crisp bread containing wholegrain rye flour, wheat bran, oat meal and sesame seeds. Breads were stored at water activities in the range of 0-0.75. Their mechanical properties (deformability modulus, fracture stress and strain, fracture work) were measured by three-point bending tests. The microstructure of the crisp breads was studied at two water activities by scanning microscopy. Water activity significantly influenced mechanical properties of crisp bread. Increasing water activity caused an increase of the fracture stress at water activities ranging from 0.030 to 0.255 for rye bread and at a_w 0.039-0.319 for fibre bread. An increase of water activity above these values caused softening and a sharp decrease of the deformability modulus and fracture stress. Microscopic photographs showed that water activity had an influence on the structure of crisp bread. The rye bread had lower resistance to deformation than the fibre bread, probably due to differences in composition, and the added grain ingredients gave a more heterogeneous structure.

INTRODUCTION

Wholegrain foods such as whole meal or wholegrain breads, crisp breads, breakfast cereals and puffed whole grains are important sources of nutrients and photoprotective substances that are in a short supply in our diet, including dietary fibre, resistant starch, trace minerals, certain vitamins, and other components [Slavin *et al*., 1997; Richardson, 2000]. Wholegrain fibre is considered to be a significant factor contributing to reducing the risk of diseases such as diabetes, cardiovascular disease and certain cancers [Heiniö *et al*., 2003]. Despite the growing interest in the health aspects of wholegrain food, texture as the most important sensory attribute, remains a priority consumer choice criterion.

Texture is mostly related to physical, and especially mechanical properties of food products. Most of dry crispy/crunchy food products have a porous structure consisting of beams and films of solid material surrounding air cells [Luyten *et al*., 2004]. The mechanical and ultimate properties of these cellular solids depend on the composition and homogeneity of materials and also on the amount and structure of pores [Gibson & Ashby, 1997]. Non-uniformity in internal structure and surface characteristics of brittle and crunchy foods result in a very complex failure mechanism that involves repetitive deformation and fracturing of subsequent layers in a cell structure. The mechanical behaviour and structure of cellular solid foods give an irregular and irreproducible force-deformation relationship [Luyten *et al*., 2004]. Stokes & Donald [2000] reported that fracture in dry bread started at voids in the smallest beams between the open air cells. When the bread was moister, buckling and large deformation of these beams occurring before fracturing was clearly visible. Dry cereal-based baked and extruded products, such as crisp breads, wafers, crackers, and snacks, are hygroscopic due to their chemical composition, porosity and presence of starch in the amorphous state [Colonna *et al*., 1984; Marzec & Lewicki, 2006]. If the moisture of these crispy products increases, due to water sorption from the atmosphere or by mass transport from neighbouring components, it results in a soggy, soft texture [Nicholls *et al*., 1995]. Crispness is associated with pleasing textural contact and with freshness and good quality of low moisture cereal product; its loss caused by increased moistness of the material is a major cause of consumer rejection.

Water is a constituent of food which affects food stability, quality and physical properties. In solid foods water affects their response to force. Increasing water content can lead to plasticizing or antiplasticizing effects [Lewicki, 2004]. The plasticization of polymer chains facilitates deformation and brittle material becomes ductile and losses crispness.

The effect of water activity on crispness of cellular products has been studied by many authors. Katz & Labuza [1981] have described the texture of snack foods, such as crackers and chips, as a function of water activity. They demonstrated that baked saltine crackers, popcorn, and fried potato chips lost crispness when their water activity exceeded 0.35 to 0.50 depending on the product. A slight decrease in crispness of breakfast cereals occurred at a_w <0.5. Thereafter a rapid decrease of crispness was observed until a_w=0.8, at which the product lost its brittleness completely [Sauvageot & Blond, 1991]. A similar behaviour was observed when hardness

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was considered. Force-deformation curves for an uniaxial compression test were recorded for crackers at various water activity (a_w =0.14-0.80) [Kohyama *et al*., 1997]. The curves became smoother and the maximum force decreased with the increase in water activity. Roudaut *et al*. [1998] studied texture properties of crispy bread as a function of water content using a compression test. They observed plasticizing effects of water in a range of water content between 3 and 9%; then up to 11%, there was an apparent hardening of the material. Beyond water content of 11%, the apparent modulus decreased and the softening effect of water became dominant. In some cases, the antiplasticizing effect is observed. Adsorbed water causes an increase in mechanical resistance of the material and reduces its brittleness. Marzec [2002] reported that failure stress of flat wheat and rye bread increased as moisture was adsorbed and reached a peak at an a_w between 0.5-0.6. Baked and extruded cereal products are generally in the glassy state, since cooking is accompanied by the disappearance of most crystalline structures of native starch. Cereal products stored above their glass transition temperature undergo changes which are manifested, among others, by alterations of their mechanical properties. Cellular products may thicken and their mechanical strength can increase [Roudaut *et al*., 1998].

The mechanical properties and fracture behaviour of the crispy food products are strongly affected by their structure. Microscopy can provide information about structure of cellular material. The most commonly used technique to determine bread structure is based on the light microscopy. The shape and size of gas cells is an important quality indicator of cellular structures of bread crumb [Scanlon & Zghal, 2001]. Aguilera *et al*. [2000] used hot stage light microscopy to elucidate changes in starch and simultaneous development in cellularity during frying potato chips. Electron microscopy is often used to study the morphology of cell walls and their thickness. SEM pictures of apple chips showed that the samples with thicker cell walls and larger internal voids were judged crispier [Sham *et al*., 2001]. Katz & Labuza [1981] studied the effect of water activity on the structural characteristics of crispy snacks. Micrographs of popcorn showed cellular collapse at a water activity as high as 0.75.

As demonstrated above many factors affect the texture of cellular food products. Moreover, the knowledge of those parameters is important in determining real material properties and also in predicting behaviour of products during storage. Extruded and baked cereal products are very popular. Their major attraction to consumers, apart from their nutritional advantages, is their crispy texture. The loss of crispness in foods can be caused by an increase of moisture due to water sorption during storage of dry crisp bread. The aim of this work was to obtain better understanding of the relationship between water activity of crisp bread and its mechanical properties and structure. The characteristics of the effect of water activity on mechanical parameters and structure can be used to predict shelf life and quality of crisp bread.

MATERIALS AND METHODS

Wholegrain rye crisp bread and fibre crisp bread (Barilla Wasa, Germany) made of wholegrain rye flour, wheat bran, oat meal and sesame seeds were purchased in a local market. Typical rectangular-shaped crisp bread had a size of 111 × 60 mm and was 4 mm thick. Samples were equilibrated over saturated salt solutions in desiccators to water activity in the range from 0 to 0.75 at 25°C. Water activity was measured with the use of a Hygroskop DT (Rotronic, Switzerland) with accuracy of ±0.001 after 49 days of storage, while water content of the samples was measured by drying according to the Polish Standard [PN-84/A-8636].

Mechanical properties of the breads examined were measured by a three-point loading bending test. Samples were placed on two supporting parallel bars situated 50 mm apart. The loading bar connected to a crosshead of a Zwick Machine 1445 (ZWICK GmbH, Germany) was used to deform the samples to the moment of their break. The bending test for crisp bread was done at a deformation rate of 20 mm/min with at least 9 replicates on the whole slice of crisp bread. Force *versus* deformation data was recorded, analysed and some mechanical indices were calculated.

Fracture stress σ_f was calculated from the equation [Kim & Okos, 1999]:

$$\sigma_f = \frac{3FL}{2bt^2}$$

where: L – distance between the supports (m); F – fracture force (N); b – width of the sample (m); and t-thickness of the sample (m).

Deformability modulus E_B was calculated as follows:

$$E_B = \left(\frac{dF}{d\delta}\right)\cdot\left(\frac{L^3}{4bt^3}\right)$$

where: $dF/d\delta$- – initial slope of force – deformation curve.

Fracture strain ε_f was calculated from the following equation:

$$\varepsilon_f = \frac{6\delta_{max}t}{L^2}$$

where: δ_{max} – deformation at fracture (m).

The fracture work (mJ) was calculated as the area under the bending curve: force (N)- deformation (mm).

Environmental scanning electron microscopy was used to investigate the microstructure of each commercial crisp bread type at water activities of a_w~0.28 and 0.56. The microstructure of the samples was observed in a scanning electron microscope Philips XL30ESEM TMP at accelerating voltage of 25 kV in high-vacuum mode (0.7 mm Hg). The microscopy study was done at the Laboratory of Electron Microscopy of Warsaw Agricultural University.

RESULTS AND DISCUSSION

In most literature references describing the effect of water activity on textural properties [Sauvageot & Blond, 1991;

Roudaut *et al*., 1998] it has been noticed that samples stored over salts solutions, which assure relative humidity of the atmosphere, achieved moisture equilibration within a particular period of time, usually between 1 and 3 weeks [Heidenreich *et al*., 2004]. Rye and fibre crisp breads placed in the desiccators had water activities in the range of 0.030 to 0.612 (Table 1). However, the samples did not reach their particular assumed water activity after 49 days of storage. A significant difference in water activities between values obtained by the product and of the environment in the desiccators was very evident at high relative humidity. Research conducted by Marzec [2006] demonstrated that rye extruded bread achieved water activity moisture equilibration after 21 days of storage. The sorption isotherms of wheat extruded bread have shown that 77 days of storage were enough for moisture equilibration. The presence of one ingredient affects the capacity of water adsorption by others ingredients. Water activity and moisture content of the purchased samples were measured immediately after removing them form packaging. As reported in Table 2, the a_w of rye and fibre breads was very similar but the samples exhibited differences in water content. This was probably due to the chemical composition of the breads. The fibre bread contained whole sesame seeds and oat meal, especially on the surface layer of the bread. A more heterogenous structure of fibre crisp bread, than that of the rye crisp bread, could influence its mechanical properties (Table 2). The higher values of the fracture stress and deformability modulus were observed for fibre crisp bread but the differences were not statistically significant (coefficient of variance 10-28%).

TABLE 1. Characteristics of crisp bread equilibrated to different water activity.

Type of bread	Water activity in desiccator	Water activity of crisp bread	Water content in crisp bread (%)	Fracture work of crisp bread (mJ)
Rye	0	0.030 (0.008)*	1.62 (0.36)	3.54 (0.85)
	0.225	0.255 (0.011)	5.69 (0.09)	5.54 (1.74)
	0.328	0.312 (0.015)	6.03 (0.34)	5.78 (1.32)
	0.432	0.380 (0.012)	7.32 (0.03)	6.01 (0.75)
	0.529	0.453 (0.011)	8.53 (0.10)	5.51 (0.76)
	0.648	0.531 (0.006)	10.44 (0.18)	5.44 (1.09)
	0.753	0.612 (0.002)	11.94 (0.09)	5.60 (0.91)
Fibre	0	0.039 (0.002)	2.14 (0.23)	4.04 (1.17)
	0.225	0.237 (0.003)	5.22 (0.03)	4.67 (0.91)
	0.328	0.319 (0.011)	6.16 (0.08)	8.50 (1.31)
	0.432	0.399 (0.008)	6.70 (0.14)	6.48 (1.13)
	0.529	0.389 (0.001)	8.03 (0.09)	5.80 (0.83)
	0.648	0.525 (0.011)	10.05 (0.01)	6.11 (1.54)
	0.753	0.560 (0.003)	10.85 (0.18)	8.00 (1.25)

*Standard deviations for average values of parameters are presented in brackets.

The influence of water activity on the bending-breaking behaviour of rye crisp bread is demonstrated in Figure 1. It shows the force *versus* deformation for a_w of 0.030, 0.255, 0.453 and 0.612. For clarity, only one of the replicates at a water activity has been selected for the figure. It can be seen from the curves that brittle behaviour observed for crisp bread at low water activities was characterised by multiple peaks. Saleem [2005] observed that curves obtained from three-point bending tests of biscuits gave more than one peak, which could be attributed to progressive breakdown of the cellular structure of the material. The low a_w resulted in jaggedness of the force-time curves. This phenomenon was strongly evident for extruded corn-rye bread at an a_w range 0.283-0.458 [Lewicki *et al*., 2004] and for extruded flat bread with 5.3 and 9% moisture [Fontanet *et al*., 1997]. In rye crisp bread increasing water activity from 0.030 to 0.453 resulted in a 2-fold increase of the deformation at which the sample broke. Increasing water activity smoothed out the bending curves, which indicated that less micro-breaking events occurred. Above a_w of ~0.4 increase of water activity decreased the breaking force and extended the deformation at fracture.

The mechanical properties of fibre crisp bread were similar to those observed for rye crisp bread. The increase in force and deformation at which breaking occurred was the result of increasing water activity. Although, the shape of the curves and the general tendency to smoothing the curves were similar to the previously described for rye bread, the plastic behaviour of fibre crisp bread occurred at higher water activity than for rye bread. Decreasing the breaking force with increasing water activity was observed above water activity of ~0.3 for fibre bread.

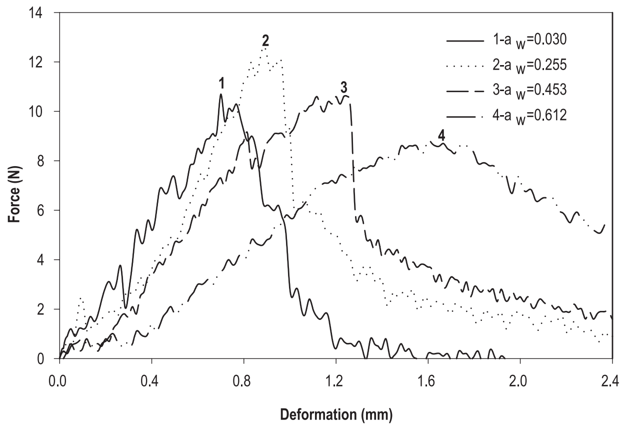

FIGURE 1. Breaking curves of rye crisp bread at different water activity.

TABLE 2. Mechanical properties of crisp bread determined directly for purchased sample.

Type of bread	Water activity a_w	Water content (%)	Fracture stress (kPa)	Fracture work (mJ)	Deformability modulus (MPa)	Fracture strain (%)
Rye	0.274 (0.008)*	6.36 (0.01)	1032.8 (106.0)	7.51 (1.89)	95.5 (27.1)	1.04 (0.27)
Fibre	0.280 (0.002)	8.03 (2.29)	1069.5 (155.1)	7.21 (0.90)	100.6 (31.5)	0.92 (0.25)

*Standard deviations for average values of parameters are presented in brackets.

The fracture stress as a function of crisp bread water activity for rye and fibre crisp breads is shown in Figure 2. The fracture stress of fibre and rye crisp bread was strongly related to water activity. At low water activities, increasing the water activity yielded a higher fracture stress. The fracture stress generally describes the fracture strength of products. Hence, the fracture strength was the largest at critical water activity, *i.e.* 0.255±0.011 for rye bread and at a_w = 0.319±0.011 for fibre bread. After these values, the samples became more deformable and pliable and the higher the water content, the lower values of the fracture stress were observed.

Adsorption of water resulted in strengthening of the investigated materials and reduced their brittleness. An increase in strength along with an increase in water activity was explained by Harris & Peleg [1996] as a result of partial plasticization of air cell wall material, which increases the structure's cohesion, hence, hardness. The hardening of extruded crisp bread in the range of water contents from 9 to 11% was also observed by Fontanet *et al*. [1997] and Marzec & Lewicki [2006]. These authors suggested that the mechanical behaviour of extruded bread depended on the short range structural reorganization caused by the increased molecular mobility. The changes of starch and the mechanical behaviour of flat crisp bread were a result of the extrusion process. In our work, the investigated crisp breads were produced by baking a dough consisting of a mixture of flour and water, with small amounts of salt and some other ingredients (*e.g.* wheat bran). Therefore, the level of critical water activity after which products lose toughness was stipulated as 0.255 for rye and 0.319 for fibre crisp breads. Water activity above these values affected the texture of crisp breads by softening the starch/protein matrix. The fibre bread was also made of rye flour, but the added sesame seeds and oat meal altered the strength of the material. The fracture stress at critical water activity for fibre crisp bread was about 12% higher than that for the rye product.

The effect of water activity on the deformability modulus and fracture strain of rye and fibre bread is presented in Figures 3 and 4. Figure 3 shows that for rye bread the relationships between deformability modulus, fracture strain and water activity were linear. The deformability modulus of rye bread decreased as water activity increased. The adsorbed water affected the loss of stiffness and brittleness of the samples. A similar effect of water activity on Young' modulus and

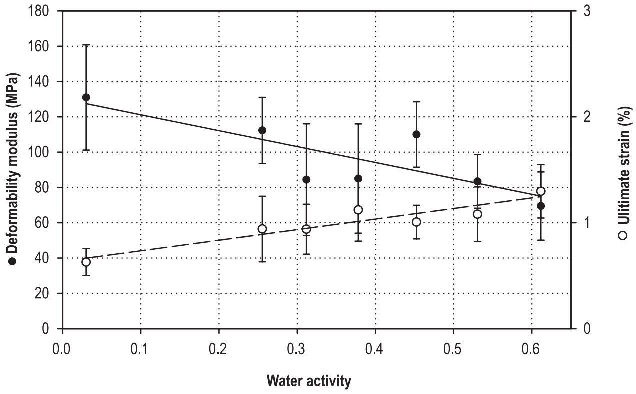

FIGURE 3. Effect of water activity on deformability modulus and fracture strain of rye bread.

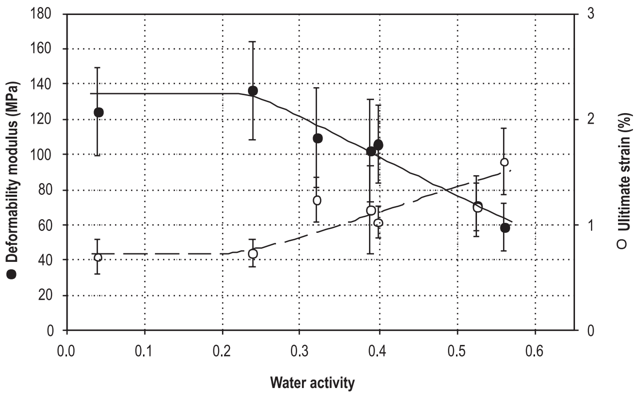

FIGURE 4. Effect of water activity on deformability modulus and fracture strain of fibre bread.

fracture strain was observed for biscuits [Saleem *et al*., 2005] and wafers [Martinez-Navarette *et al*., 2004]. The decrease of Young's modulus was explained by increasing moisture induced by structural degradation. The small fracture strains of the samples at low water activities were characterised by an initial elastic behaviour at fracture. The increase in fracture strain reflected the plasticization of crisp bread by water. The higher the water activity the more plastic the material was. From a water activity of 0.039 the deformability modulus and fracture strain of fibre bread remained constant until a water activity of 0.237 (Figure 4). Above this water activity a decrease in deformability modulus values and a gradual increase in fracture strain were observed as a result of increasing water activity. Gondek & Marzec [2006] observed a similar effect of water activity on sensory attributes of rye crisp bread. The material having water activity in the range from 0 to 0.65 was subjected to Quantitative Descriptive Analysis (QDA). A relationship between the majority of kinesthetic attributes (overall quality and plasticity) and water activity remained constant in the water activity range of 0.198–0.308. The increase of water activity above ~0.31 caused a gradually decrease of total quality and an increase in plasticity which described the level of plasticity perceived at the first bite.

The average and standard deviation in the values for fracture work at different water activities are presented in Table 1.

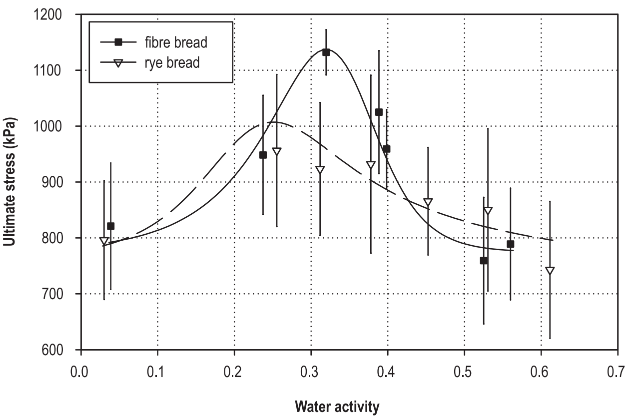

FIGURE 2. Fracture stress as a function of crisp bread water activity.

The general trend of a change of work with a change in water activity was not consistent. An increase of water activity of crisp breads caused an increase of the fracture work, and the work reached a maximum value at $a_w = 0.432$ for rye crisp breads and at 0.328 for fibre bread, and after that the values of work varied.

Water activity is an important factor influencing mechanical behaviour of brittle food. Adsorbed water is supposed to behave as a lubricant at high water activities and reduce the friction between surfaces, which results in low strength. This can be explained by differences in the microstructure of the investigated products and composition of breads. Figures 5, 6, 7 and 8 demonstrate the effect of water activity on the structure of rye and fibre crisp breads, respectively. The environmental scanning microscopy was used to analyse structure of bread samples. This microscopic method is suitable for moist samples due to nondestructive preparation technique. A photograph of a typical cross section of commercial rye bread, which was taken directly from a package at an a_w of 0.274, is shown on Figure 5. In rye crisp bread the pores were irregular, but most of them had characteristic sharp edges. The structure of bread equilibrated to water activity of 0.560 (Figure 6) contained more smooth structures. In the moist rye bread the pores became rounded, and thickness of cell walls increased significantly. Crisp bread adsorbed about 6 g of water per 100 g of solids in the range of activities between 0.274 and 0.560. This amount of water was sufficient to transition from a crisp to a non-crisp state. Katz & Labuza [1981] observed that only 3 g of water per 100 g d.m. were needed to be absorbed to lose the crispness of cellular snacks. Adsorbed water softened the structure, which was shown by the loss of mechanical resistance of rye bread.

The inner structure of commercial fibre bread (Figure 7) was similar to that observed for rye bread, both breads were very porous but fibre bread seemed to contain more irregular and larger cells. The composition of fibre bread was more complex, some ingredients as wheat bran, oat meal, sesame seeds, fragmented and whole grain of rye could play an important role in the formation of a harder structure. The microstructure of fibre bread at a_w=0.280 differed form that observed at water activity of 0.560. As expected, moist fibre bread showed more round cells and thicker cell walls than those observed for the material at low water activity (Figure 8). Moreover, the moist fibre bread was visibly collapsed in the outer layer of the sample. The fibre bread was plasticized by the adsorbed water, which resulted in decreasing crispness of the material. Katz & Labuza [1981] postulated that water adsorbed by popcorn dissolved some of intercellular glue-like

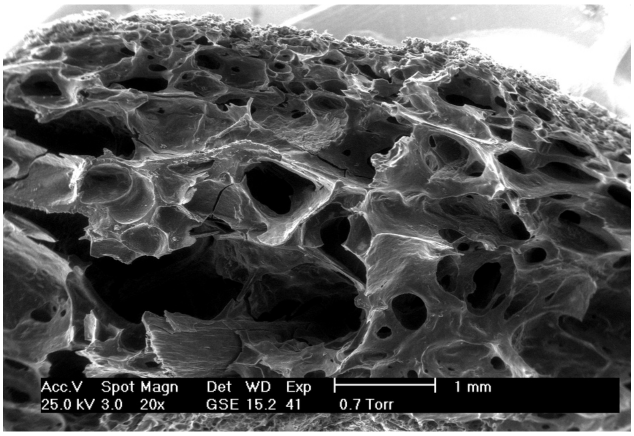

FIGURE 5. Scanning electron micrograph of rye crisp bread at a_w=0.274.

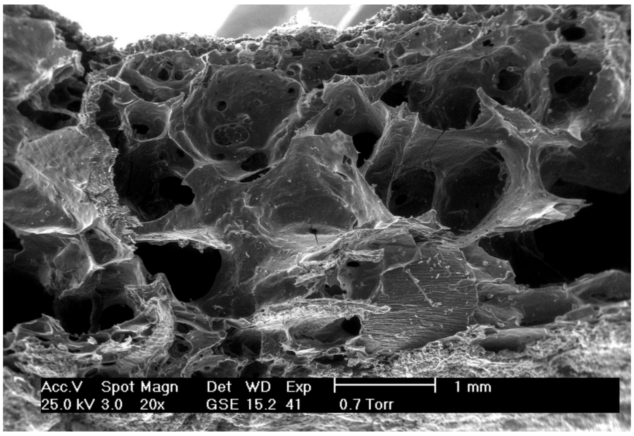

FIGURE 7. Scanning electron micrograph of fibre crisp bread at a_w=0.280.

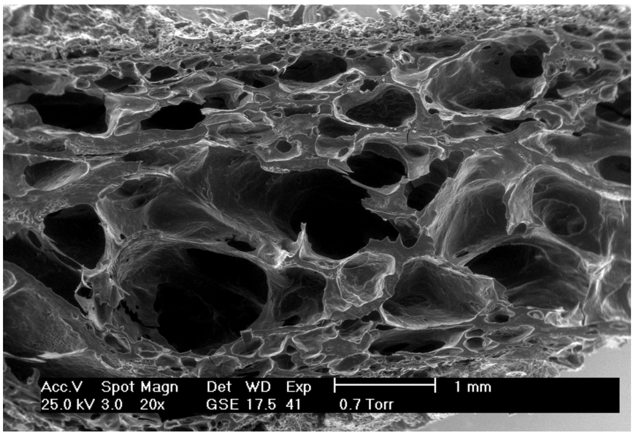

FIGURE 6. Scanning electron micrograph of rye crisp bread at a_w=0.560.

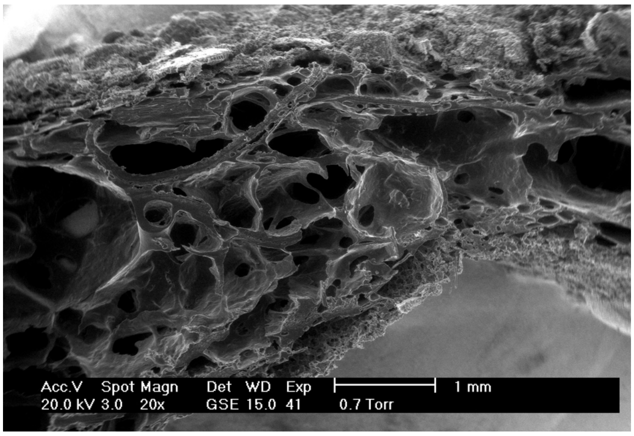

FIGURE 8. Scanning electron micrograph of fibre crisp bread at a_w=0.560.

material and gelatinised starch on the cell walls. At higher water activity the starch granules were probably more swollen and more amylose leached out.

CONCLUSIONS

The changes of water activity were responsible for the mechanical properties of rye and fibre crisp breads. Increasing water activity caused an increase of the fracture stress at water activities in the range form 0.030 to 0.255 for rye bread and from 0.039 to 0.319 for fibre bread. The hardening of samples was probably connected with structural rearrangements of biopolymers and adsorbed water seemed to induce the new matrix of proteins and carbohydrates. It seems that differences in the structure of crisp bread could play an important role in the formation of more harder texture. An increase in water activity above these critical values caused softening and flowability of crisp breads. The decrease of deformability modulus accompanied by an increase of the fracture strain were explained by increasing moisture inducing structural degradation. The high water activity affected the more plastic behaviour of samples and loss of brittleness. At high water content, texture became soft and rubbery. Moreover, at a macroscopic level water had a plasticizing effect which resulted in smoothing cell structures and increasing thickness of the cell walls. The rye bread had a lower resistance to deformation than the fibre bread, probably due to differences in composition, for the added grain ingredients yielded a more heterogeneous structure.

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ZALEŻNOŚĆ MIĘDZY AKTYWNOŚCIĄ WODY A WŁAŚCIWOŚCIAMI MECHANICZNYMI I STRUKTURĄ PIECZYWA CHRUPKIEGO

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Celem pracy było określenie wpływu aktywności wody na strukturę i właściwości mechaniczne pieczywa chrupkiego: żytniego i pieczywa żytniego wysokobłonnikowego z mąki pełnoziarnistej, z dodatkiem otrąb pszennych, płatków owsianych i ziaren sezamu. Pieczywo było przechowywane w środowisku o aktywności wody w zakresie 0-0,75. Właściwości mechaniczne (moduł odkształcalności, naprężenie i odkształcenie łamiące oraz pracę łamania) przeprowadzono przy wykorzystaniu trójpunktowego testu zginania. Mikrostrukturę pieczywa chrupkiego dla dwóch aktywności wody zbadano za pomocą mikroskopii skaningowej. Aktywność istotnie wpływała na właściwości mechaniczne pieczywa chrupkiego (rys. 1). Wzrost aktywności wody w zakresie od 0,030 do 0,255 dla pieczywa żytniego i dla pieczywa wzbogaconego w błonnik 0,039-0,319 wpływał na wzrost wartości naprężeń łamiących (rys. 2). Wzrost aktywności wody powyżej tych wartości powodował mięknięcie pieczywa i gwałtowny spadek wartości modułu i naprężenia łamiącego (rys. 3, 4). Zdjęcia mikroskopowe wskazują istotny wpływ aktywności wody na strukturę pieczywa chrupkiego (rys. 5-8). Pieczywo żytnie charakteryzowało się mniejszą odpornością na odkształcenie niż pieczywo o wyższej zawartości błonnika, które zawierało w swoim składzie otręby i ziarna, to wpływało na uzyskanie pieczywa o niejednorodnej strukturze.