THE INVESTIGATION OF AVAILABILITY OF ENERGY DISPERSIVE X-RAY FLUORESCENCE SYSTEM TO OBSERVING BREAD STALING

M. Erdem Sağsöz[1], H. Gürbüz Kotancılar[2], M. Murat Karaoğlu[2], K. Emre Gerçekaslan[2], Salih Z. Erzeneoğlu[3]

[1]Division of Radiodiagnostic, Faculty of Medicine, Atatürk University, 25240 Erzurum, Turkey
[2]Department of Food Engineering, Faculty of Agriculture, Atatürk University,25240, Erzurum, Turkey
[3] Department of Physics, Faculty of Science&Arts, Atatürk University, , 25240 Erzurum, Turkey

ABSTRACT

Bread staling mechanism has been investigated for a long time but, because of the mechanism of bread staling is a rather complex phenomenon, it is not entirely understood. The economic losses caused by bread staling are incredibly important for the world economy. It is generally accepted that starch reorganization is major cause of bread staling. Starch crystallization in bread increases with time during staling. The crystallites formation due to retrogradation of starch (mainly amylopectin) has been studied by both X-ray diffraction (XRD) and differential scanning calorimetry (DSC). In this study, N_{coh}/N_{Comp} (R) intensity ratios are measured as functions of storage time with a Si(Li) detector using Am–241 annular source. The texture measurements have also been performed. Our experimental results are presented and discussed in this study.

1. INTRODUCTION

Bread staling is one of the most common problems in bread storage. The mechanism of bread staling is a rather complex phenomenon and not entirely understood [1-6]. When bread stored in aseptic conditions, it is an biochemically inactive colloidal system [7]. The economic losses caused by bread staling are incredibly important for the world economy [1,8,9]. The limited shelf life of bread, resulting from bread staling, has a great economical impact of millions of dollars in Türkiye and possibly of billions of dollars for the world per year.

Bread staling mechanism has been investigated for a long time. In the beginning, it was thought that bread staling was result from only loss of water. But, Boussingault (1852) showed that bread stored in hermetically sealed containers also exhibited the staling process [10]. After about 150 years, it is generally accepted that starch reorganization or recrystallization (called by retrogradation) is major cause of bread staling [2,10, 11]. For that reason, in recent studies on bread staling have been focused on starch retrogradation.

Starch is major and very important component in bread. Starch consists of two polymer chains which are amylose and amylopectin. Amylose has a linear form while amylopectin has a branched form. This polymers exhibit different retrogradation kinetics. According to Jagannath *et al* [12] bread staling is due to starch retrogradation including the following two process: a time dependent retrogradation of amylose and time independent retrogradation of amylopectin. Because of the amylose rapidly became retrogradate, it is suggested that amylopectin retrogradation responsible for bread staling [13].

Starch crystallization in bread increases with time during staling. The crystallites formation due to retrogradation of starch (mainly amylopectin) has been studied by both X-ray diffraction (XRD) and differential scanning calorimetry (DSC). DSC give an information about amount of recrystallization (quantitative data), while XRD show crystal type [9,13-18]

Scattering of photons by atoms, molecules and solids is an important method for obtaining information about the structural properties of materials. Accurate determination of ratio of coherent/Compton scattering for different materials is important, for they have been widely used in the fields of atomic and radiation physics and non-destructive elemental analysis of materials and compared with theoretical predictions based on atomic models and tested the validity of these models. There have been a few investigations on ratio of coherent/Compton scattering. For instance, İçelli and Erzeneoğlu [19] investigated effects on measurement of photon-atom scattering of applied pressures on sample at different thickness. The experimental study on ratios of coherent/Compton scattering differential cross sections have been studied for some elements with atomic number $26<Z<82$ in 59.5 keV for 55° and 115° [20]. Analysis of binary systems with coherent/Compton intensity ratio have been performed by [21]. This study shows that the Rayleigh/Compton scattering method can be used to analyze binary systems with sufficient accuracy. X-ray emission lines measurable with common instruments have been not produce at environmental, geological, and biological

samples including a major part of the matrix consists of light elements such as carbon, oxygen, and nitrogen so, the scattered photons are the only way to characterize the matrix directly. Duvauchelle et.al., [22] have studied the effective atomic number in the Rayleigh to Compton scattering ratio. Erzeneoğlu [23] has measured ratios of Rayleigh scattering to Compton scattering differential cross section through angles from 55^{o} to 105^{o} for Mo, Ag and Sn.

The aim of this study is to investigation of availability of energy dispersive X-ray fluorescence system in the evaluation of bread staling during storage. Because of the staling mechanism is very complex, only one parameter or experimental technique is not adequate in evaluation of staling rate. For that reason, texture measurements have also been performed in this study. Additionally, Ribotta *et al.* [10] designated that both crumb firming and amylopectin retrogradation increased with storage time. Because of the moisture content of bread crumb changes with storage time, moisture factor have completely removed with a view to follow only changes in the molecular structure of bread.

2. EXPERIMENTAL PROCEDURE

2.1. Experimental arrangement

The schematic arrangement of the experimental setup in the present work is shown in Fig.1. The experiment was performed using a annular source of Am-241 of intensity 100 mCi which essentially emits monoenergetic (59.5 keV) gamma rays. A Si(Li) detector was used to detect the scattered gamma photons. The resolution of the detector (FWHM) was found to be 160 eV at 5.96 keV and data were collected into 1024 channels of a multichannel analyser. To obtain reasonable scattering peak and reduce the statistical errors in measurement, the spectra were collected for a period of 500 s.

2.2. Sample procedure

Bread was baked in a conventional oven according to straight dough method [24]. Two different dough formulations, which are with shortening and without shortening, were used in bread preparation. The recipe and breadmaking processes are currently employed in our country in the preparation of white bread. Producted breads were stored at the room temperature (20 ± 2°C) and refrigerator temperature (4 ± 1°C). For X-ray system, freeze dried (Iyophilized) bread crumbs were ground and compressed into pellets for 300 s at 5 ton by using a manual hydraulic press after sieving.

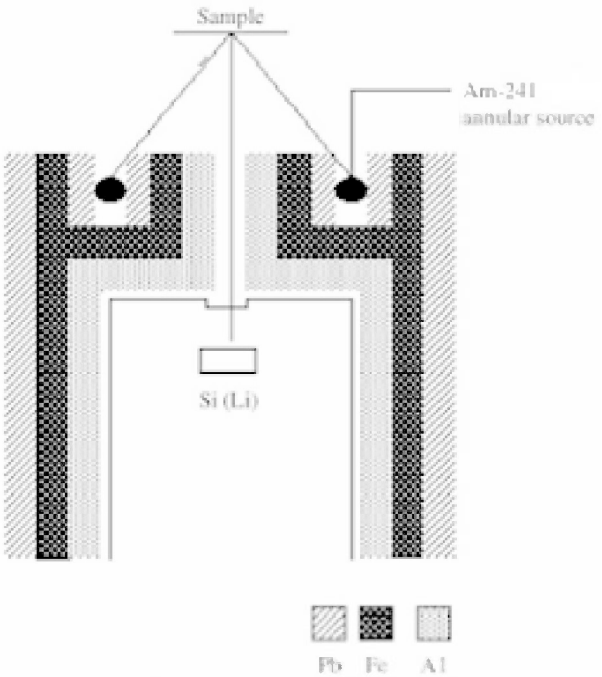


Fig. 1. Experimental arrangement and geometry.

2.3. Texture Analysis

For the texture analysis, Carr and Tadini's [25] method was modified. The analysis of texture was carried out using the texture analyser SMS (model TA-XT.plus, Stable Micro System, England) with a 36mm probe (P/36). The measured parameters were firmness, springiness and cohesiveness. Because of these parameters

have strong correlation to the sensorial parameters, they were chosen. The extremities of the bread were removed, resulting in 5 cm thickness of slices. Fig.2 shows generalized texture profile curve obtained using the texture analyser model TA-XT.plus.

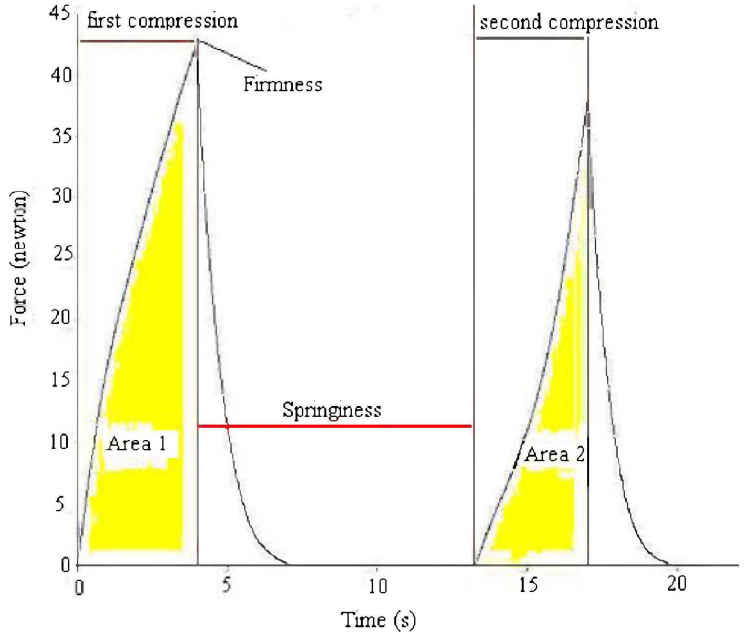


Fig. 2. Generalized texture profile curve obtained using the texture analyser (TA-XT.plus).

The TPA method was conducted under these conditions: pre test speed: 2.0 mm/s, test speed: 5.0 mm/s, post-test speed: 5.0 mm/s, distance: 20 mm, trigger type: auto-20 g, time: 5 s.

Firmness: the peak force during the first compression cycle (height of curve 1), N.

Springiness: the height that the food recovers during the time that elapses between the end of the first bite and the start of the second bite, mm.

Cohesiveness: The ratio of the positive force area during the second compression portion to that during the first compression (Area 2/Area 1), excluding the areas under the decompression portion in each cycle, dimensionless. According to results were obtained by using texture analyser, the diagram of firmness vs. storage period relating to bread crumb stored at room temperature is shown Fig.3 and the general diagram of firmness vs. storage period relating to bread crumb is shown Fig.4.

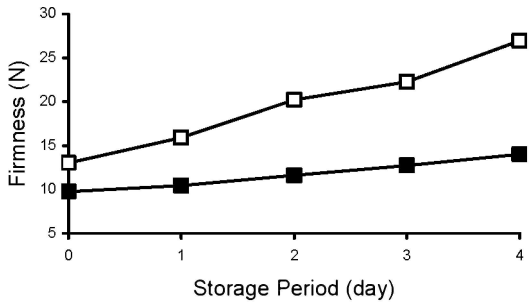


Fig. 3. The diagram of firmness vs. storage period relating to bread crumb stored at room temperature.

read with shortening (■), without shortening (□).

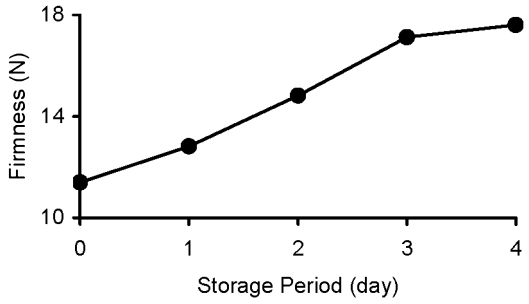


Fig. 4. The general diagram of firmness vs. storage period relating to bread crumb.

3. RESULTS AND DISCUSSION

The experimental results obtained in the present work are graphically given in Fig. 5-8. Because there are no experimental studies on the investigation of availability of energy dispersive X-ray fluorescence system to observing bread staling in the literature, our findings in the present study could not be compared with the literature values. To the best of our knowledge, the present investigation constitute the first experimental measurements. Firstly, the investigation were applied to the bread without freeze drying in our experiment. The undried breads has more value of scattering than the freeze dried breads. This shows us the effect of water factor. During staling, water moves from crumb to the crust. Therefore, the water factor was eliminated to watch just the molecular changes. As seen from Fig 5-8, the ratios of coherent/Compton scattering vary with increasing storage period. This results may be attributed to the variation in the crystal structure of starch. Similar results have also been reported by earlier investigators [10,14,16,26]. Also, Fig 5,6 is shown that the ratios of coherent/Compton scattering for bread stored at refrigerator temperature is more values than these stored at room temperature. In addition to this, the ratios of coherent/Compton scattering for bread supplemeted with shortening is more values than without shortening in Fig.7,8

According to TPA results, it was determined that firmness increased with storage time. This increase was lower in bread with shortening and stored at refrigerator temperature than bread without shortening and stored at room temperature, respectively. It has been reported that the addition of shortening was crumb softener and to be used an anti-staling agent for bakery products [18,24]. Because of the bread structure become firm but crumbly with staling, the value of Area 1 increased and thus cohesiveness (Area 2 / Area 1) decreased with storage time. Because of the moisture migrate from crumb to crust during the staling, crust lost fragility and become leathery. Therefore, springiness values weren't change relatively. Firmness of the bread is so important for consumer acceptance; therefore, only firmness graphic is used in this paper.

As a result, further experimental studies on this matter will be useful to confirm, understand and interpret these results. Also, in order to reach more definitive conclusions on scattering intensity ratios influenced by storage time, we project to extend these measurements for various bread formulations, different scattering angle and primer energies.

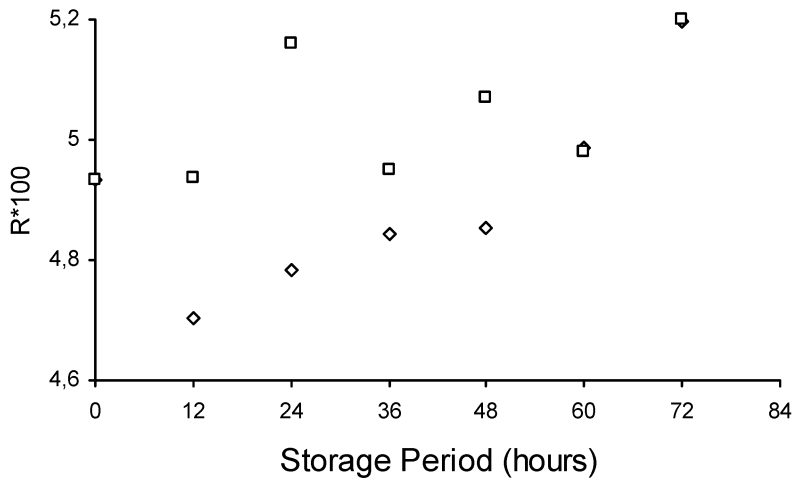


Fig. 5. The diagram of N_{coh} / N_{Comp} (R) intensity ratios*100 vs. storage period relating to bread crumb without shortening. Storage temperature; 20±2°C (◊), 4±1°C (□).

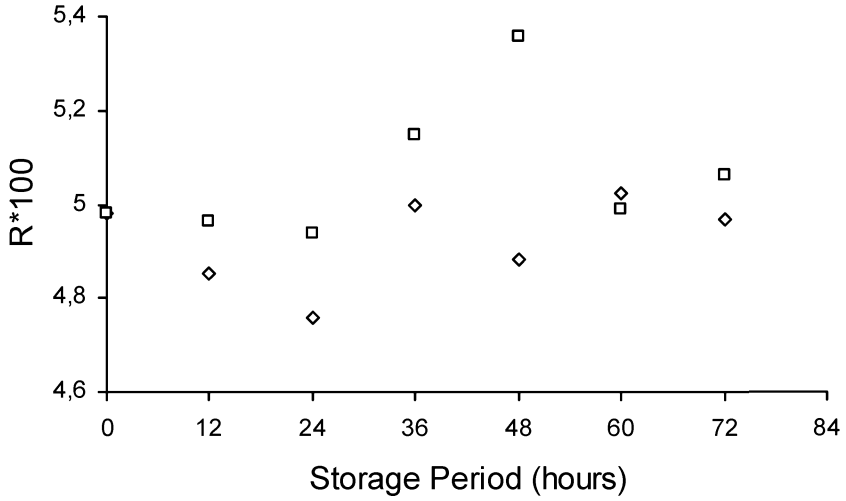


Fig. 6. The diagram of N_{coh} / N_{Comp} (R) intensity ratios*100 vs. storage period relating to bread crumb with shortening. Storage temperature; 20±2°C (◊), 4±1°C (□).

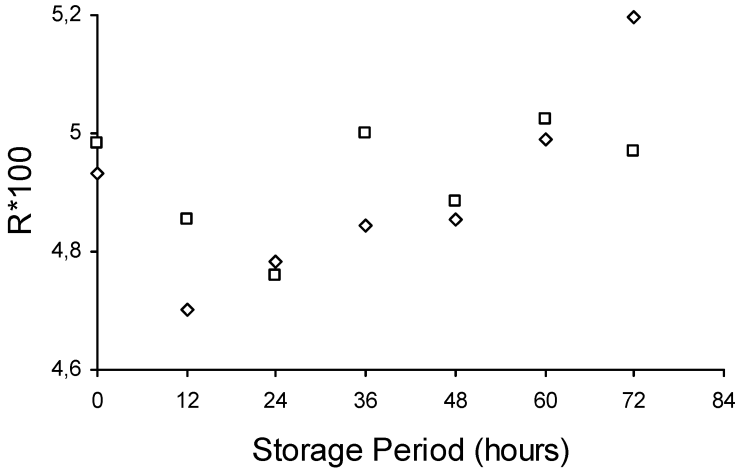


Fig. 7. The diagram of N_{coh} / N_{Comp} (R) intensity ratios*100 vs. storage period relating to bread crumb stored at room temperature. Bread without shortening (◊), with shortening (□).

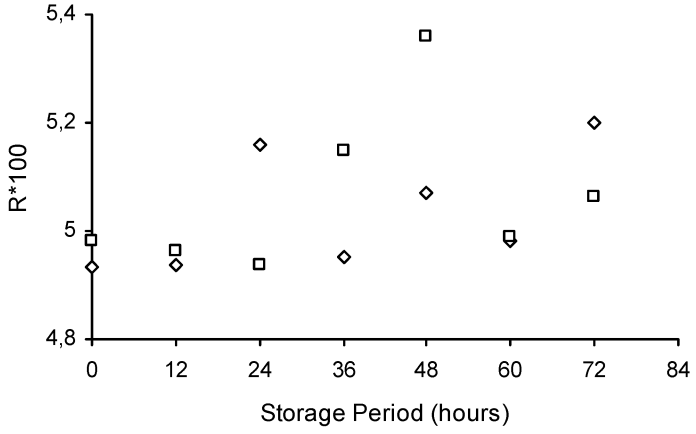


Fig. 8. The diagram of N_{coh} / N_{Comp} (R) intensity ratios*100 vs. storage period relating to bread crumb stored at refrigerator temperature. Bread without shortening (◊), with shortening (□).

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