Investigation of bread staling by handheld NIR spectroscopy in tandem with 2DCOS and MCR-ALS analysis

Marina De Géa Neves1\*, Heinz W. Siesler1, Isao Noda2

1 Department of Physical Chemistry, University of Duisburg-Essen D45117 Essen, Germany

2 Department of Materials Science and Engineering, University of Delaware, Newark, DE, USA
\*marina.de.gea.n@gmail.com

During the process of staling (aging) bread not only loses its texture, flavor, and freshness but also increases its hardness (Fadda et al., 2014). As a result of these palatable changes, consumer disapproval of bread grows quickly. It is known, that one of the critical factors in the staling process is the retrogradation (recrystallization) of amylopectin in starch. The decrease of water content by evaporation and diffusion from core to crust also contributes to aging. The present investigation was carried out using the technique of handheld near-infrared (NIR) spectroscopy. Apart from a considerable reduction of hardware price, this technique has recently become a powerful tool for in-the-field and on-site investigations of a broad range of materials. Time-resolved measurements were made in diffuse reflection of a fresh bread surface, thereby allowing the interpretation of the structural changes in bread as a function of the observed spectroscopic changes.

To quantitatively compare the patterns of spectral intensity variation over time, two-dimensional correlation spectroscopy (2DCOS) (Noda and Ozaki, 2004) was applied and the synchronicity and sequence of structural changes were proposed. Due to the variation of spectral changes, especially at the beginning of the aging process, the data was split into time-segmented sets. For the first six hours of staling white bread, the sequence of events is: crystallization of amylopectin (4703, 4346 cm-1) < evaporation of weakly hydrogen bonded water (5311 cm-1) < reorganization of starch OH-functionalities (5158 cm-1) < diffusion and evaporation of strongly hydrogen bonded water (4964 cm-1) and final crystallization of starch CH-functionalities (5851 cm-1). From 6 to 48 hours on the other hand, the sequence of events was: 4964, 5311 (OH water) < 5158 (OH starch) < 5851 (CH starch) < 4703, 4346 cm-1 (CH starch).

In addition, Multivariate Curve Resolution-Alternating Least Squares (MCR-ALS) (Piqueras et al., 2012) was used to investigate the changes in the spectra profile as a function of aging time. The cross-over points of the concentration profiles provide information regarding the progress of the above mentioned water evaporation/diffusion and the amylopectin crystallization processes.

**Keywords:** handheld NIR spectroscopy, bread staling, 2DCOS, MCR-ALS

REFERENCES

Fadda, C., Sanguinetti, A.M., del Caro, A., Collar, C., Piga, A., 2014. Bread staling: Updating the view. Comprehensive Reviews in Food Science and Food Safety.

Noda, I. (Isao), Ozaki, Y. (Yukihiro), 2004. Two-dimensional correlation spectroscopy : applications in vibrational and optical spectroscopy. John Wiley & Sons.

Piqueras, S., Burger, J., Tauler, R., de Juan, A., 2012. Chemometrics and Intelligent Laboratory Systems 117, 169–182.