Introduction

Frying is an ancient method of food preparation. Today, both industrial and catering/restaurant scale frying of fast foods and snacks represent a growing industry because of its speed, ease-of-use and ability to supply food with good appearance, flavor and texture. However, the physics and chemistry of frying process are still poorly understood.

During the frying process hundreds of chemical reactions take place, changing sensory and physical properties of the heating medium and finally of the food to be fried. A great amount of volatile and non-volatile compounds have been identified as thermal and oxidative reaction products, especially of oxidation and polymerisation. The composition of the degradation products depend on many variables such as temperature, use time and type of the oil, moisture content of food and contact with air. After frying, the originally present fat of the food or pre-fried products is nearly completely replaced with the used frying oil. Therefore, the quality of the frying oil is most important as it determines the quality of the fried products and its storability.

Several analytical indices for monitoring the degradation of frying oils have been proposed: para-Anisidine Value (AnV), Acid Value (AV), Total Polar Compounds (TPC) and di- and polyunsaturated Tricylglycerols (DPTG). TPC and DPTG remain the most reliable chemical indicators for the chemical analysis of used frying oils and fats. Therefore in industry and restaurants, electronic devices indicating the TPC content by measuring the change in the dielectric constant of the oil are used. These tests are simple to use but are not recommended by the German Society of Fat Sciences since the results are often influenced by both the moisture content and the type of the oil.

Industrial food producers should however understand the thermooxidative changes in their frying oils, especially far below the official legal limits of polar compounds (24%). The formation of undesirable oxidation products has to be avoided as the end result will be off-flavours and a shorter shelf life, even in frozen foods.

To sum up, there is a need of different tests which allow to monitor the oil degradation process including oxidized-by-products such as acid value, monomeric oxidized di-glycerides and the composition of the unused and heat-treated frying oil. No single parameter is sufficient to predict the oil quality and finally the food quality. The application of conventional analytical methods is however time-consuming and laborious, needs a well-equipped lab and skilled staff. Fourier-Transform Near Infrared Spectroscopy (FT-NIR) is a fast, non-destructive, and cost-effective technique for the routine analysis of fats and oils.

The aim of this study was to establish a validated method based on a large data set using FT-NIR spectroscopy in order to analyse both the fresh and the degraded frying oils for the relevant quality parameters.

Experimental

For the FT-NIR transmission measurements the oil samples were filled in 8 mm disposable vials (Figure 1). The measurements were performed using an FT-NIR spectrometer (MPA, Bruker Optik GmbH). The spectral range was 12,500 - 4,000 cm\(^{-1}\) co-adding 32 scans at a resolution of 8 cm\(^{-1}\).

The samples were analyzed for the relevant parameters Total Polar Compounds (TPC), Di- and Polyunsaturated Tricylglycerols (DPTG), para-Anisidine Value (AnV) and Acid Value (AV) using the standard DGF methods. A mathematical relationship between FT-NIR spectra and the analytical parameters was established using partial least squares regression (PLS algorithm).

The NIR calibration of each parameter has been set up with a large number of samples used frying fats collected from several fast food restaurants, bakeries, caterers or industrial producers by the food inspection authorities. Their compositions were very different: solid, semi-solid, liquid, containing low and high levels of mono-, polyunsaturated and saturated and trans-isomerized fatty acids.

The Iodine value varied from 16 to 150 (Figure 2), indicating the variety of the used oils. The validation of all methods was carried out using a test set (see Table 1).

Results and Discussions

The calibration and validation sets represent the whole concentration ranges of each analyte from the lowest to the highest values which can occur in practical life. High validation accuracies (Table 1) were obtained for all NIR determinations.

The precision of the methods TPC, DPTG, AV and AnV has been evaluated in an inter-laboratory test with 11 laboratories organized 2013 by the “Joint Committee for the Analysis of Fats, Oils, Fatty products, Related Products and Raw Materials (GA FETT)” and is adopted as the DGF standard method “C VI 21a (13)”. The comparison of the repeatability and reproducibility data of conventional and NIR data (Table 2) demonstrate that the performance of the NIR measurements is similar and for TPC and DPTG even better.

Furthermore, the Iodine Value, content of saturates, mono- and polysaturated fatty acids and trans fatty acid content are recognized parameters to control the oil quality and finally the food quality. They can also be used to monitor the structural changes in the composition of the used frying oil and thus the oil inside the finished product. The fatty acid composition has also often to be declared according the new regulations in Europe.

Conclusion

All proposed FT-NIR measurements correlate very well with the official standards methods. They are an objective index of the degradation process and help to optimize and control the frying process. FT-NIR is simple, quick, and easy to use by untrained operators anywhere. It is safe for use in food processing environment and correlates to the quality of food prepared and stage of degradation providing reliable results for quality assurance programs and food inspection.

References