Near infrared diffuse reflection spectroscopy and multivariate calibration for quality control and simultaneous quantification of multiple compounds in washing powders

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Introduction

Between 1991 and 2007 the consumption of washing agents in Germany per head and year was reduced by 1.1 kg to 7.6 kg and it is a clear aim to further reduce this amount. On an average a household (2.1 persons) performs seven washing procedures a week adding up to 600,000 tons of washing agents per year. Due to this enormous consumption, analytical techniques enabling fast, selective and sensitive determination and monitoring of ingredients before, during and after the production are required. In the present study we established a near-infrared (NIR) spectroscopic method for the fast qualitative identification of different composites and washing agent types on one hand, and a quantitative analytical model for the simultaneous determination of washing ingredients such as surface-detergents, phosphate, carbonate etc. in different types of full washing agents (FWA), compact washing agents (CWA) and colour washing agents (CoWA).

Materials and methods

NIR spectra were recorded with a scanning polarisation interferometer Fourier-transform NIR spectrometer (FT-NIR) (Büchi, Flawil, Switzerland). The FT-NIR instrument offers a resolution of 12 cm^{-1}, an absolute wavelength accuracy of ± 2 cm^{-1} and a relative reproducibility of 0.5 cm^{-1}. Chemometric software NIRCal 4.21 (Büchi) and "The Unscrambler" v9.6 (CAMO, Oslo, Norway) was used for creating the principal component analysis (PCA) and partial least squares (PLS) regression models. For testing the models the collected spectra were divided into a learning-set (c-Set, 67 %) and a test-set (v-Set, 33 %) both consisting of independent samples. Measurements

were carried out at room temperature (23°C) from 4000 – 10.000 cm^{-1}. 27 samples consisting of 9 FWA, 9 CWA, and 9 CoWA were purchased in stores around Innsbruck (Austria).

Results and discussion

Measurements in diffuse reflection mode, followed by a log (1/R) transformation, allowed assigning characteristic hydroxyl and alkyl absorption bands in the spectra (Figure 1).

Principal component analysis (PCA) (Figure 2) and PLSR calibration were computed over varying contents of washing powder ingredients. The washing agents were analysed for their

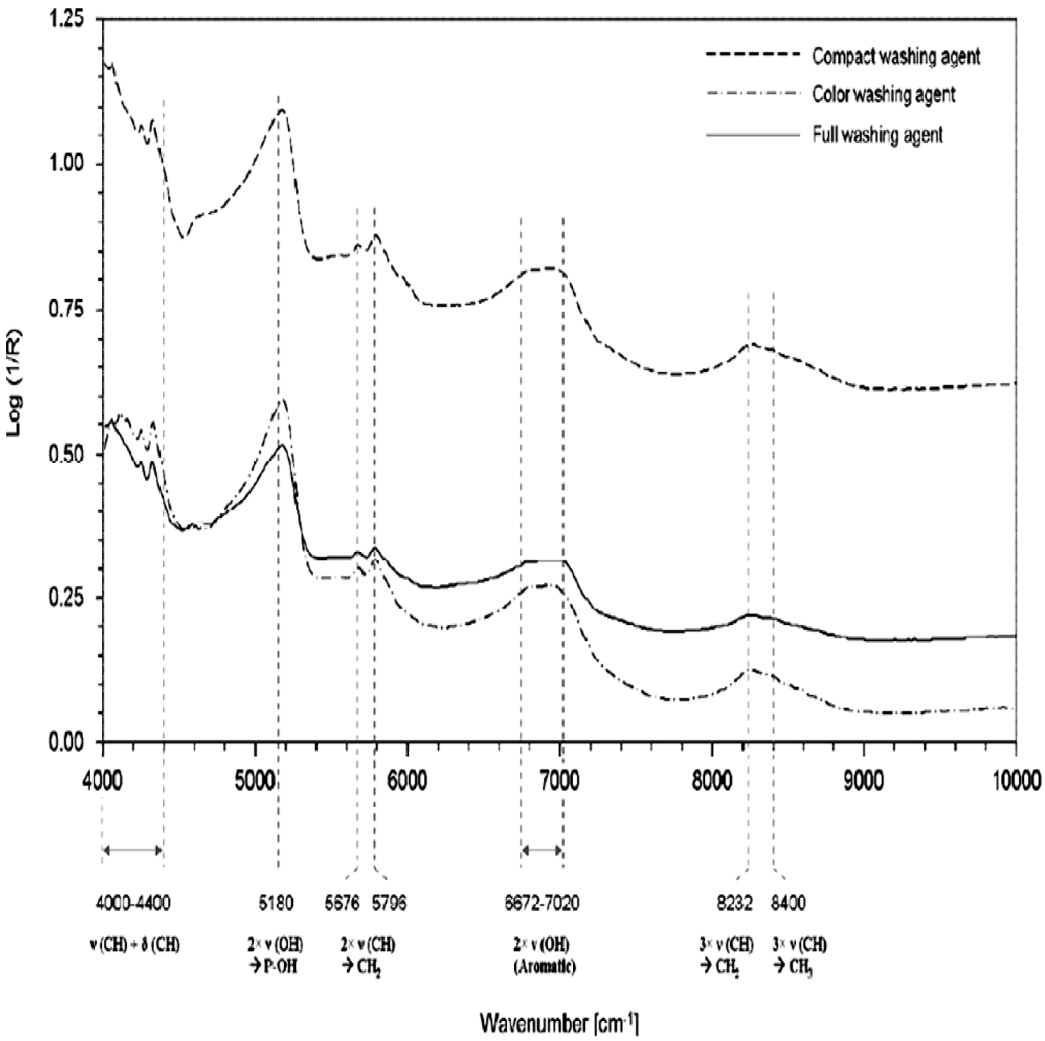

Figure 1. Averaged NIR absorption spectra of different types of washing powders.

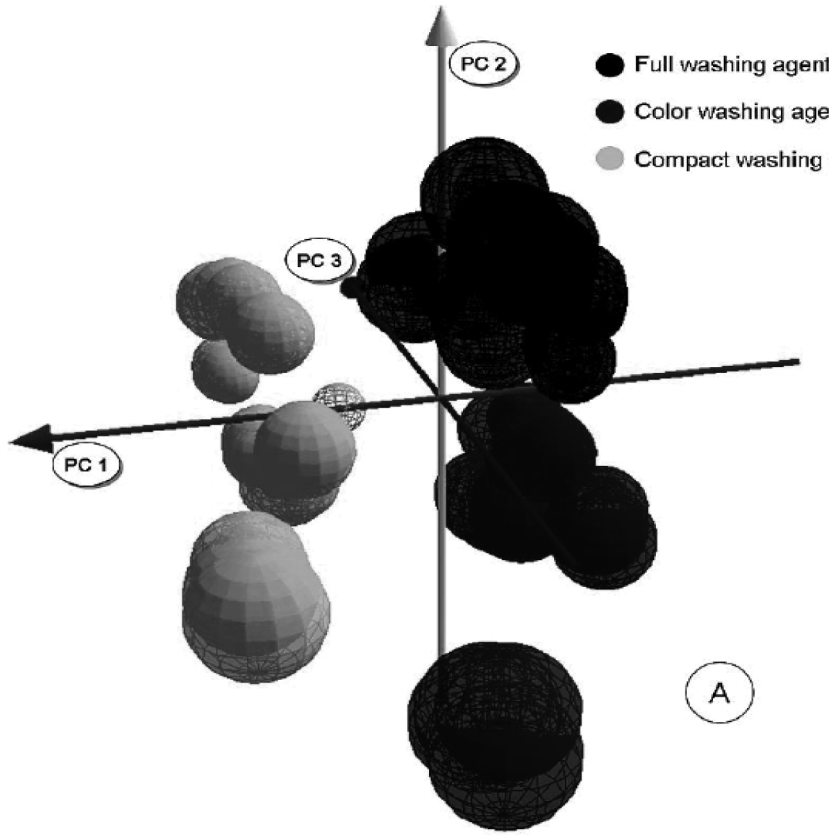

Figure 2. 3-dimensional factor plot of the different washing powder types.

contents [%] of silicon, phosphorus, carbonate, methylene blue active substance (MBAS), and non-ionic tensides (agents that modify the surface tension characteristics of water) to build the quantitative multivariate models. Silicon and phosphorus were analysed by inductively coupled plasma (ICP), carbonate was determined as $CaCO_3$, anionic detergents were analysed by the MBAS (methylene blue active substance) method, and the total amount of non-ionic tensides was determined by high-performance liquid chromatography (HPLC). By applying various data pre-treatments five partial least squares regression (PLSR) models were calculated to predict the amounts of the mentioned parameters simultaneously, by only one measurement. The standard errors of prediction (*SEP*) showed 0.59 % for silicon (range 0.65–5.11 %), 0.05 % for phosphorous (range 0.03–0.45 %), 3.61% for carbonate (range 8.60–49.90 %), 1.40 % for MABS (range 1.87–10.7 %), and 0.79 % for non-ionic tensides (range 0.15–4.00 %), respectively. For the first time a validated NIR-spectroscopic method is presented, offering an easy to apply, high- precision and high sample throughput method, not only for industrial quality assurance applications, but also for rapid quality control, e.g. by applying NIR handheld instruments.