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Examining short-wavelength infrared hyperspectral imaging for do-it-yourself-for-you laundry detergent powder fablab quality control

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ABSTRACT

Do-it-yourself-for-you fablabs, autonomous mini factories responding to customer specific needs, require advanced cost efficient measurement systems for validating quality of end product. An example of end product is laundry detergent powder. The customer chooses raw materials with preferences related to for example odor, health and environmental aspects. Thus, the raw materials vary in each mixture. Operational risks include customized laundry detergent powder product missing a desired compound. To mitigate this risk, verification of each raw material existence in the mixture is essential. According to previous studies, optical measurement such as spectroscopy in short-wavelength infrared range is one promising way to identify raw materials in laundry detergent powders. For proving feasibility, we examined short-wavelength infrared hyperspectral imaging of laundry detergent powder samples - final products and raw materials. Additionally, we tested liquid soap samples with same method and experimental setup as in the detergent powder samples' case. This study shows, that existence of desired raw materials can be verified from detergent powder with short-wavelength infrared imaging. Final testing of customized laundry detergent powder product avoiding desired compound absence failure is enabled by spectroscopic measurement and analysis. Liquid soap is more challenging test subject because of strong water absorption in the short-wavelength infrared range. Further studies should cover testing and comparing more optical measurement and analysis methods for finding accurate and affordable do-it-yourself-for-you fablab solutions.

Keywords: SWIR, infrared, hyperspectral imaging, DIY4U, laundry detergent, fablab, quality control

1. INTRODUCTION

Recently, spectroscopy has offered new tools for quality measurements of detergent powders and other similar products. Asachi et al. has applied near-infrared (NIR) spectroscopy to analysis of homogeneity of binary and ternary mixtures[1]. They have demonstrated powder segregation analysis of low content level ingredients. Bittner's study recommends NIR as a method of choice for the analysis of washing powders[2] - providing extensive comparison of vibrational spectroscopic methods for the identification of washing powder brands as well as for an overall quantitative analysis of all ingredients of the analyzed laundry detergents. According to Zhang[3], chemometric calibrations combined with the NIR spectroscopy is suitable methodology for washing powder classification. Khanmohammadi et al. have proposed method based on mid-IR reflectance spectrometry with Doolittle[4,5] multivariate calibration being compared to partial least squares (PLS) method. Additionally, attenuated total reflectance Fourier transform infrared (ATR FT-IR)[6,7] spectrometry in wavenumber regions of 800–1,290 cm^{-1} and 1435–1342 cm^{-1} with PLS treatment of data is reported to be precise analytical procedure for quantitative determination of sodium percarbonate (SPC) in washing powder.

Related to liquid detergents and soaps, Brouckaert et al. have introduced in-line predicting of concentration levels of a surfactant and a polymer in a liquid detergent composition with a calibration model created from at-line acquired Raman spectra[8]. Wyllie report an experiment to determine the percentage of triclosan in a series of antibacterial soaps using ultraviolet- visible light (UV–VIS) spectroscopy following conversion of the triclosan to an azo-dye through reaction with nitrite and 4-sulfanilic acid[9]. For gasolines, Silva et al. proposes infrared spectroscopy as a promising technique for classification of gasoline with and without additives when associated with supervised pattern recognition methods and a pre-concentration step (distillation)[10]. In combination with PLS algorithm modeling, ATR-FTIR spectroscopy has been shown to be a simple, rapid and accurate technique for determining the quantity of Na4EDTA in aqueous solutions[11], Suárez et al. concludes. According to Carolei and Gutz, combination of ATR-FTIR with simple chemometrics like CLS

or ILS is very appropriate for the simultaneous analysis of three surfactants and water in shampoo and liquid soap during the production process[12].

1.1 DIY4U laundry detergent powder fablab and hyperspectral imaging

Fablabs are emerging to fast moving consumer goods (FMCG) market. Fablab is a digitally-enabled small-scale manufacturing machine or factory[13,14]. The fablab can exist for example in a supermarket. By utilizing the fablab, a consumer can produce detergent powder according to the customer's own preferences. The preferences might relate to e.g. allergies, environmental aspects or price of the detergent product. Additionally, a digital platform will enable the digital design and testing of new customized FMCG.

The fablabs need to have quality assurance methods. Process analytical technologies (PAT) and hyperspectral imaging in short-wavelength range offer opportunities to develop novel tools for the quality measurements of detergent powders. Hyperspectral imaging also increases possibilities of spectroscopy enabling e.g. fast area scanning. In this study, we examined short-wavelength imaging to both detergent powder final product as well as raw materials of detergent powders.

2. METHOD OF HYPERSPECTRAL IMAGING

We executed numerous detergent sample measurements with a short-wavelength infrared hyperspectral camera in Optics laboratory in VTT Kuopio, Finland. The samples consisted of final detergent powder product bought from local a supermarket in Kuopio and raw materials of detergent powders from Procter & Gamble Co., United Kingdom. Additionally, we tested one liquid detergent sample, which originated from a product bought from the local supermarket in Kuopio. We prepared the samples by pouring detergent or raw material into Petri dishes or equivalent holder dishes.

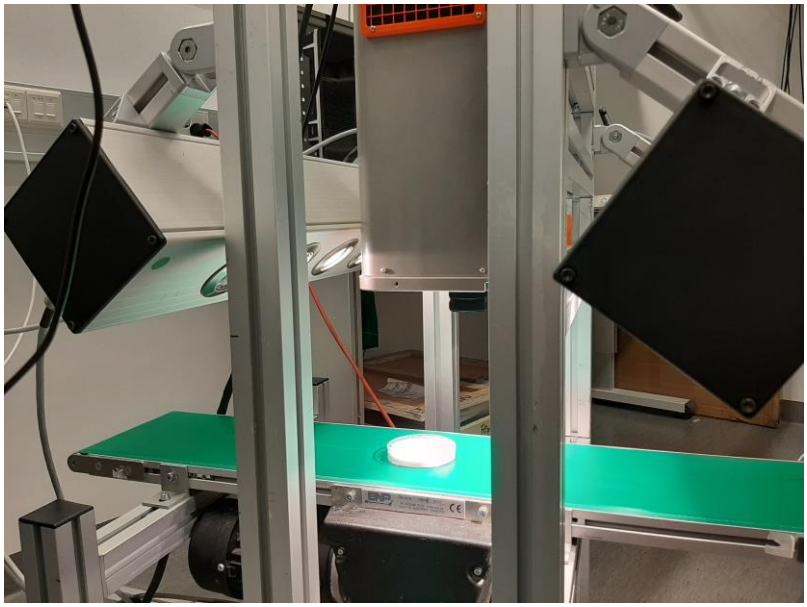

Figure 1. A detergent sample on a Petri dish on conveyer belt in measurement location of Specim Specam short-wavelength infrared hyperspectral camera. Tilted halogen lamps on both sides of the camera form broad-wavelength irradiance to the measurement location.

We measured the samples in the Petri dishes with Specim Specam short-wavelength infrared hyperspectral camera shown in Figure 1. The Specim Specam utilizes so-called "push-broom" or "line-scanning" technology. Wavelength range of the hyperspectral camera covers about 900-2500 nanometers. A conveyer belt scans the samples by moving them through measurement location or measurement line. Successful measurement requires powerful active lighting. Halogen lamps on both sides of the camera form broad-wavelength irradiation to measurement location. The scanning produces three-dimensional hyperspectral data cube, which holds two-dimensional spatial data. The third dimension is spectrum in each pixel. The spectrum acts as a fingerprint of certain chemical compound or mixture of compounds, because certain compound 'owns' certain absorbance lines or bands in wavelength dimension.

3. RESULTS OF SWIR EXAMINATIONS

In this study, we report selected remarkable findings of our numerous hyperspectral imaging tests. We emphasize, that we are only beginning to understand hyperspectral imaging of detergent powders and different analysis tools related to the resulting data cubes. Consequently, our results and analysis in this field are very preliminary.

3.1 Principal component analysis

We measured multiple spectra of different raw materials of detergent powders. Principal component analysis verified, that the raw materials separate spectrally from each other, as shown in PCA diagram in Figure 2. The raw materials: Hilas33, Tinopal, Sokalan, TAED, ProvoxC, SodaSolvay, CitricAcid and others formed clear groups in the PCA diagram.

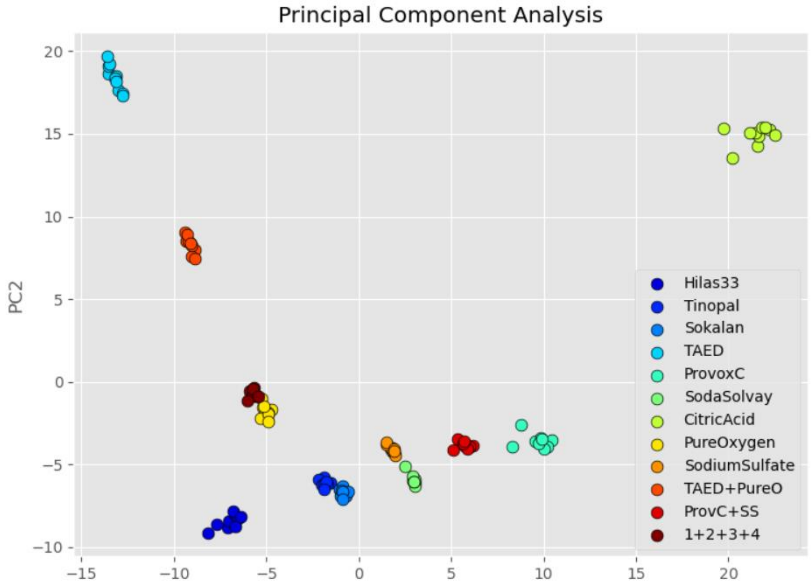

Figure 2. PCA diagram of detergent raw materials showing two first principal components: PC1 in x-axis and PC2 in y-axis. The raw material spectra form groups verifying that they are clearly distinguishable from each other.

3.2 Partial least squares analysis

Table 1. Calibration test set: 'Bulk' mixture had four ingredients: Sodium sulphate 150g, Hilas33 112,5g, TAED 15g and Tinopal 0,75g. SodaSolvay as fifth component mixed into the bulk creating fourteen sample concentrations 0-60%.

Sample	Bulk weight, g	SodaSolvay weight, g	SodaSolvay Concentration, %
1	5,0	0	0
2	4,8	0,2	4
3	4,6	0,4	8
4	4,4	0,6	12
…	…	…	…
11	3,0	2,0	40
12	2,8	2,2	44
13	2,5	2,5	50
14	2,0	3,0	60

We prepared a calibration test set as follows. ‘Bulk’ mixture had four ingredients: Sodium sulphate mass was 150 grams, Hilas33 112,5 grams, TAED 15 grams and Tinopal 0,75 grams. Into the bulk, we mixed SodaSolvay as fifth component with different masses - creating fourteen mixtures with SodaSolvay concentrations ranging from 0 to 60%, as shown in Table 1. We took ten measurement points from each sample. Our calibration model utilized three of them per sample and cross-validation verified the model. First derivate of the measured spectra and ‘loadings’ revealed wavelength areas important for the calibration, as shown in Figure 3.

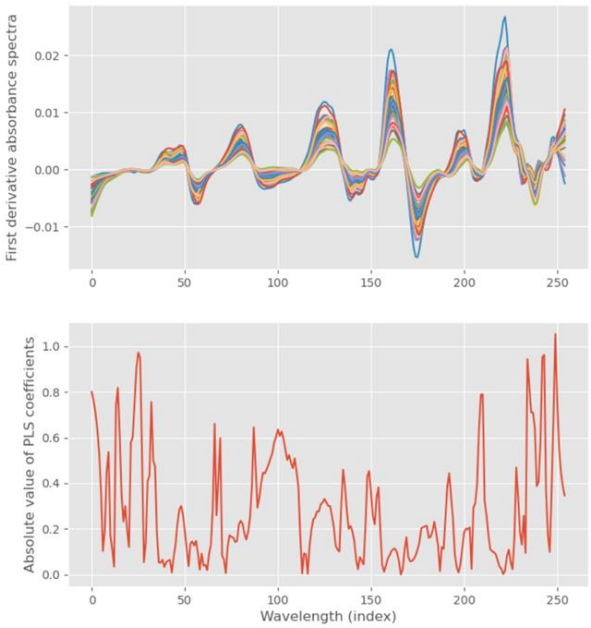

Figure 3. First derivative of absorbance spectra (above) and loadings (below). The loadings show important spectrum ranges.

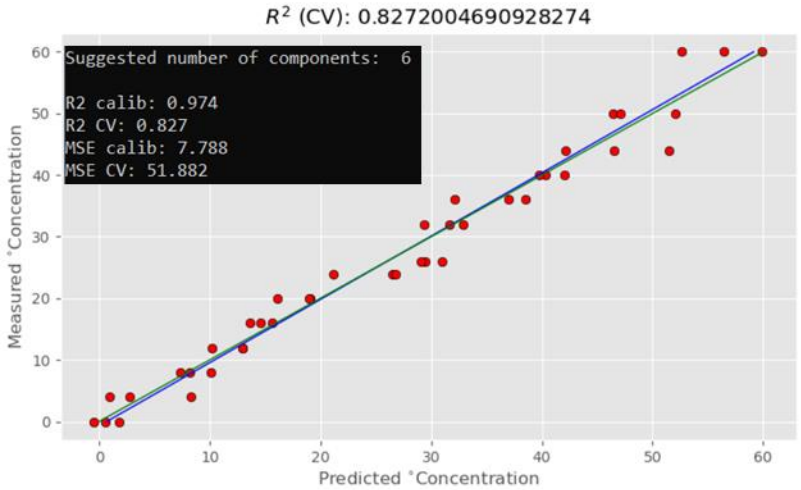

Figure 4. PLS analysis with six components showed rough correlation between predicted and measured SodaSolvay concentration.

Partial least squares (PLS) analysis with whole spectra is shown in Figure 4. Analysis result indicate, that there is correlation between predicted and measured SodaSolvay concentration. Successfully fitted calibration line describes the correlation. Nevertheless, mean square error of cross validation (MSE CV) remains large. As the MSE CV reduces, the more accurate calibration is achieved. Optimal number of PLS components was found to be six.

Additionally, we tested calibration with optimized wavelengths utilizing the loadings bands. Applying the optimized wavelengths, we were able to reduce the MSE CV value. Contradictory, number of PLS components rose significantly. Large amount of PLS components might lead to calibration overfitting, which magnifies risk in credibility of prediction. As a summary, this preliminary calibration test showed quite promising results. However, many aspects must be still enhanced and optimized:

- Testing different pre-treatments of spectra
- Better mixing of the substances
- Taking mean spectra from larger area of sample
- Outlier detection and removal
- Testing other methods than PLS
- Studying more than one substance's concentration varying
- Inspecting substances with only minor concentrations in the mix

With liquid soap, we did not achieve successful data for spectroscopic analysis. Measurement readings showed strong - almost total absorbance in about 1400-2500 nanometers range. This strong absorption was due to water content in the mixture.

4. CONCLUSIONS

Existence of desired raw materials can be verified from detergent powder with short-wavelength infrared imaging. Final testing of customized laundry detergent powder product avoiding desired compound absence failure is enabled by spectroscopic measurement and several possible analysis methods including PCA and PLS. Liquid soap is more challenging test subject because of strong water absorption in the short-wavelength infrared range. Further studies should cover testing and comparing more optical measurement and analysis methods for finding accurate and affordable do-it-yourself-for-you fablab solutions.

5. ACKNOWLEDGEMENTS

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