APPLICATION NOTE

Molecular Spectroscopy

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Rapid FT-IR-ATR and FT-NIR Spectroscopy Methods without Sample Preparation for Quality Control of Soap Noodle Parameters Using the PerkinElmer Frontier FT-MIR/NIR System

Introduction

Soaps are essential to personal and public health. They safely remove germs, soils and other contaminants and help us to stay healthy and make our surroundings more pleasant. Soaps are made from fats and oils or their fatty acids.

Soaps are sodium or potassium salts of long chain fatty acids. When triglycerides in fat/oil react with aqueous NaOH or KOH, they are converted into soap and glycerol. This is called alkaline hydrolysis of esters. Since this reaction leads to the formation of soap, it is called the saponification process (Figure 1). The saponification reaction is exothermic in nature, because heat is liberated during the process. The soap formed remains in suspension form in the mixture. Soap is precipitated as a solid from the suspension by adding common salt to the suspension. This process is called Salting out of Soap.

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If the saponification reaction is ineffective, free fatty acid (FFA) is presented. This would modify the alkalinity of the soap condition and affects its downstream applications.

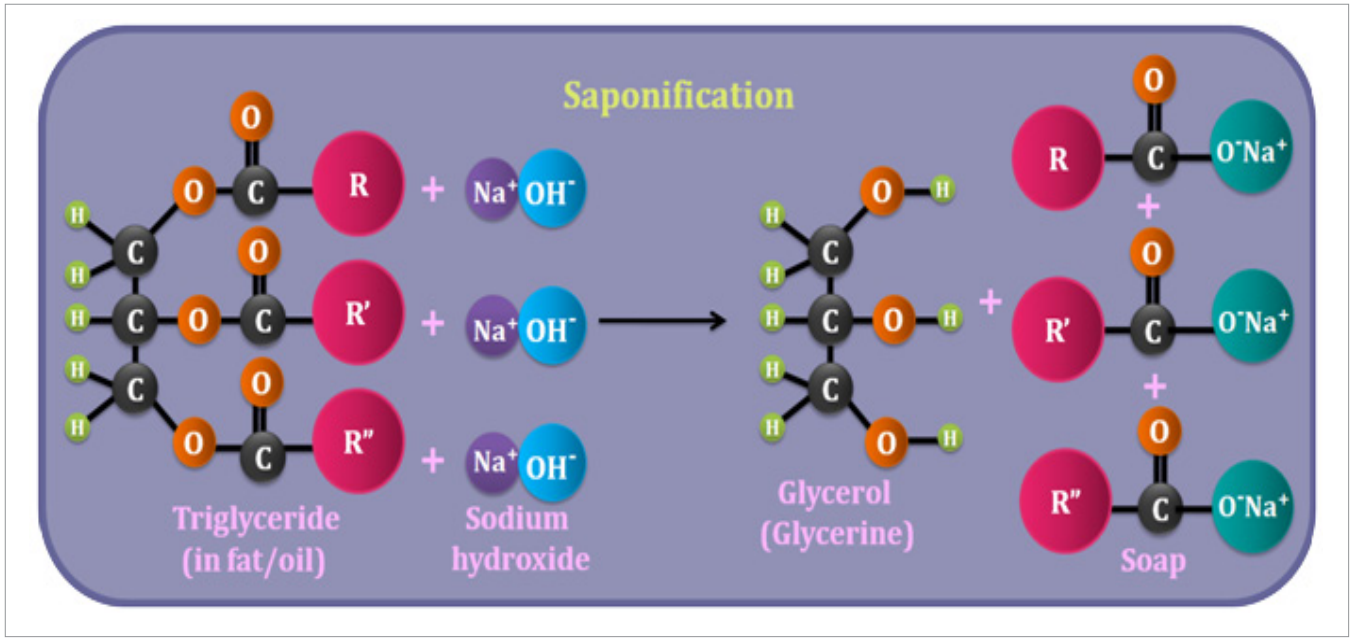

Figure 1. Saponification Process.

Various wet chemistry methods are used in the quality control of soap noodle parameters such as Fatty Acid composition (FAC), Free Fatty acid (FFA), Total Fatty Matter (TFM), Glycine (Gly), Moisture (MI), Ethylene Diamine Tri-acetic Acid (EDTA), etc.

The chemicals used in these wet chemistry methods in laboratories in the soap making industry runs into millions of dollars every year. PerkinElmer has developed a rapid FT-IR-ATR method that does not require sample preparation using the PerkinElmer Frontier™ FT-MIR/NIR system with the Universal Attenuated Total Reflectance (UATR) accessory. The FT-IR-ATR method provides an effective environmentally friendly solution and savings in both time and cost for the palm oil soap industry.

Figure 2. PerkinElmer Frontier FT-MIR/NIR System with Near Infra-Red Accessory (NIRA-top) and Universal Attenuated Total Reflectance (UATR-bottom) Accessory.

Experimental

Instrument Settings and Chemometrics Calibration Models

Soap noodle samples were analyzed with both Near Infra-Red Accessory (NIRA) and Universal Attenuated Total Reflectance (UATR) accessory on a PerkinElmer Frontier FT-IR (Figure 2). The measurements were made with 32 scans at 8 cm-1 resolution for NIRA over the wavelength range of 10000 - 4000 cm-1 and 16 scans at 4 cm-1 resolution for UATR over the spectrum range of 4000 - 400 cm-1. Calibration models of FAC, FFA, TFM Gly and EDTA were performed using PerkinElmer Spectrum® Quant Advanced Chemometrics software. FT-IR calibrations were performed with the PerkinElmer proprietary Absolute Virtual Instrument (AVI) instrument feature, which allows calibration models to be shared and transferred from one instrument to another. This will resolve the re-calibration or re-validation of models issues that plague dispersive grating-type Near Infrared (NIR) instruments.

Results

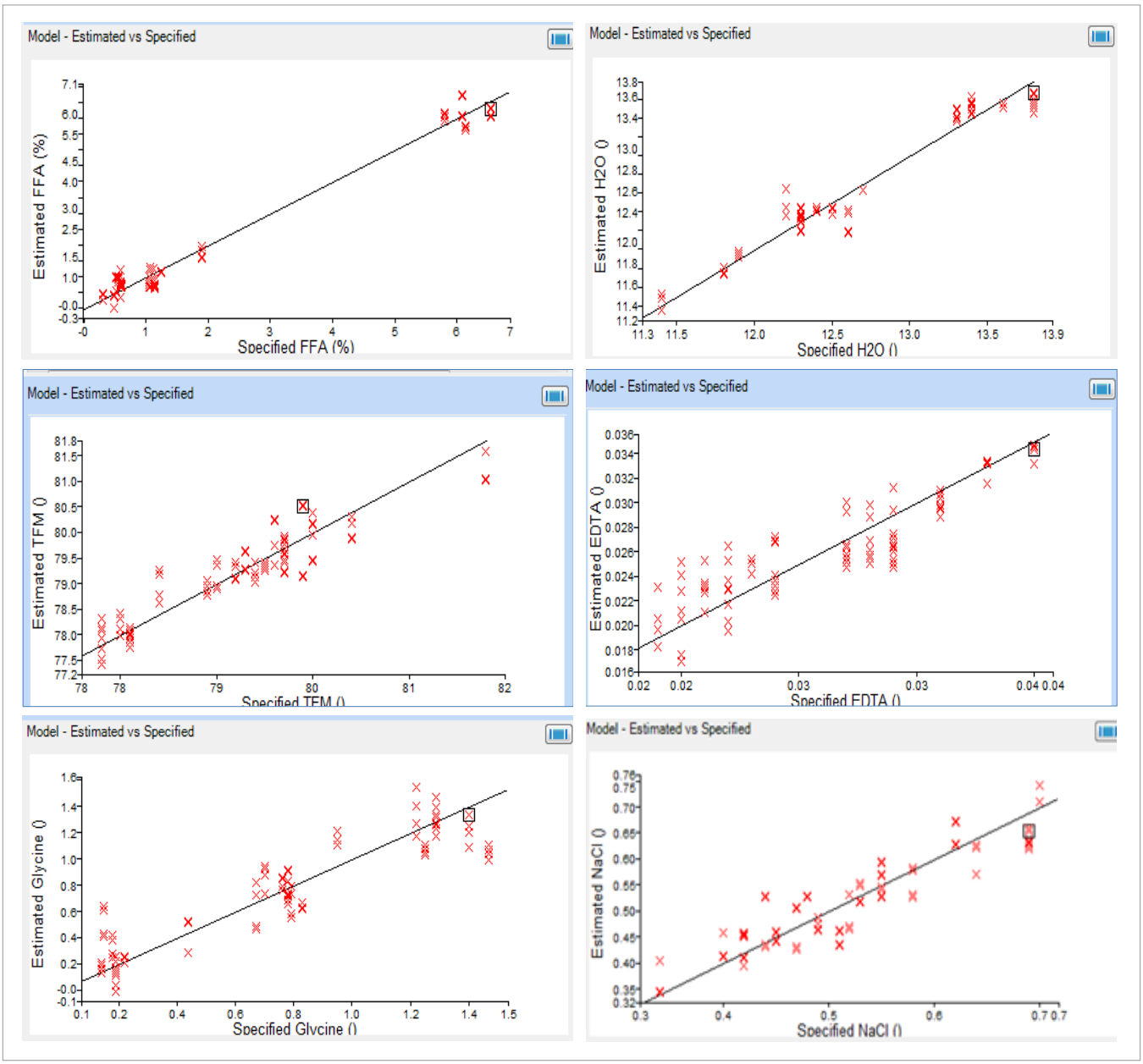

Figure 3. Calibration Models for Soap Noodle Parameters- FFA, TFM, Gly, H_2O, EDTA and NaCl.

Table 1. FT-NIR-NIRA and FT-IR-UATR Method Calibration Summaries for Soap Noodle Application.

Regression Summary		
Property	**FT-NIR-NIRA Method % Variance (R Squared)**	**FT-IR-UATR Method % Variance (R Squared)**
FFA	98.08%	97.98%
EDTA	66.60%	75.57%
Moisture	82.18%	79.45%
Glycine	86.64%	83.06%
TFM	79.33%	87.04%
NaCl	83.76%	82.59%

Table 2. Fatty Acid Composition (FAC) Calibration for C12, C14 and Summaries for Soap Noodle.

Regression Summary		
Property	**Number of PCs**	**% Variance (R squared)**
C12	5	95.9
C14	5	94.9
C16	5	90.3
C18:0	4	79.3
C18:1	8	96.5
C18:2	4	83.2

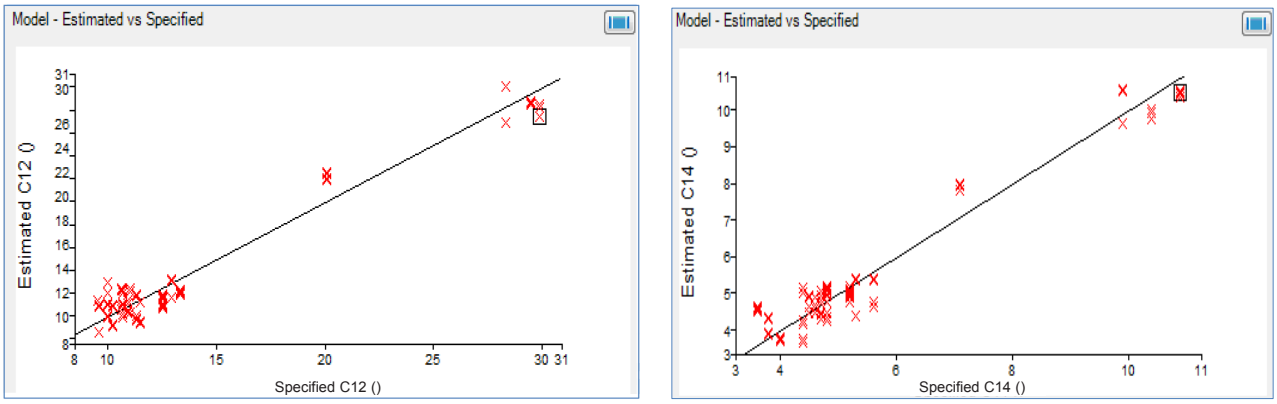

Figure 4. Calibration Models for Fatty Acid Composition (FAC) for C12 C 14.

Both FT-NIR-NIRA and FT-IR-UATR methods worked well for the soap noodle application. The calibration models of these quality control parameters shown above depicted a high percent variance. It could be further improved with more samples to cover the gaps and with a wider composition range (Figure 3 and 4, Tables 1 and 2).

Conclusion

PerkinElmer Frontier FT-MIR/NIR system with an UATR accessory provides an alternative rapid analytical tool to the wet chemistry reference methods for quality control soap noodles parameters. The calibration models for parameters of FFA, TFM, Gly, H_2O, FAC and EDTA are good and can be improved with more samples to cover the gaps and a wider composition range.

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