Quantitative Analysis of Solutions Using a High Resolution Portable Raman Spectrometer

Raman is a well-developed spectroscopic tool for molecular identification and is widely accepted for qualitative analysis where it can provide rapid identification of materials, often at their point of use or delivery. Due to the high resolution that Raman can provide, it is also used for structural elucidation as well as quantitative analysis, (ref M.J. Pelletier Applied Spectroscopy 57, 20A-42A (2003).) A benefit of Raman over other molecular spectroscopic tools is that it can be used to measure aqueous solutions. In this study, we have used B&W Tek's portable i-Raman® Plus and BWIQ® chemometric modeling software to develop a quantitative calibration model to determine the glucose, fructose and sucrose concentrations in a tertiary aqueous mixture.

BWIQ is a multivariate analysis software package for spectral data analysis and can be used explore the relationships between spectra and response data or spectra and sample classes. BWIQ combines traditional chemometric methods such as Partial Least Squares Regression (PLSR) and Principal Component Analysis (PCA), with new methods such as adaptive iteratively reweighted Penalized Least Squares (airPLS) and Support Vector Machine (SVM) algorithms.

Experiment and Results:

For this study, the i-Raman Plus from B&W Tek was chosen because it is the most sensitive portable Raman spectrometer on the market. A fiber optic probe was used to collect spectra through glass scintillation vials in this study.

A set of 31 standard samples with known amounts of glucose, fructose and sucrose in distilled water were prepared following an experimental design for development of a calibration curve. The total concentration in each sample was 0.4M of sugar. Raman spectra were collected over the range of 176 – 3200cm^{-1} under the following conditions: 785nm excitation wavelength set to ~300mW power output, and an integration time of 50 seconds. Representative spectra of the solutions of the individual sugars are shown in Figure 1.

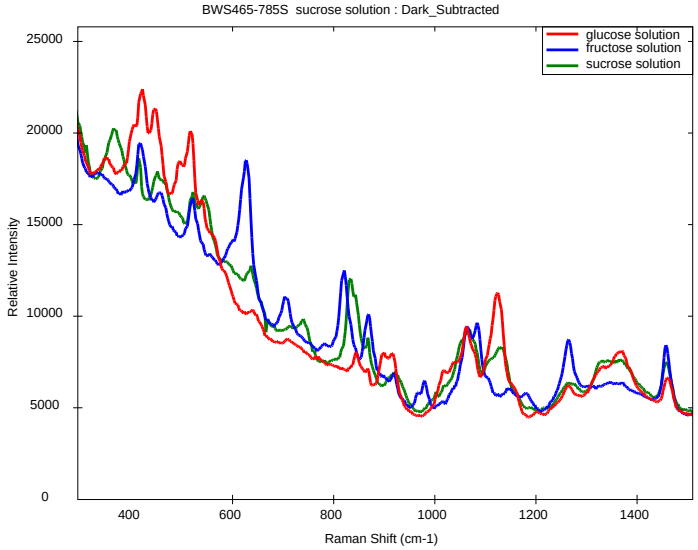

***Figure 1**: Raman spectra of aqueous sugar solutions*

A total of 62 spectra (each sample in duplicate) were collected with varying concentrations of the three components and imported into BWIQ software for analysis. Fifty spectra (25 samples scanned in duplicate) were chosen as the calibration set and the remaining 12 were used as a prediction set. The data was used without any preprocessing. A calibration curve was created using PLS1 regression on the data over the spectral region from 250 -1500cm^{-1}. An overlay of samples with varying concentration is shown in Figure 2.

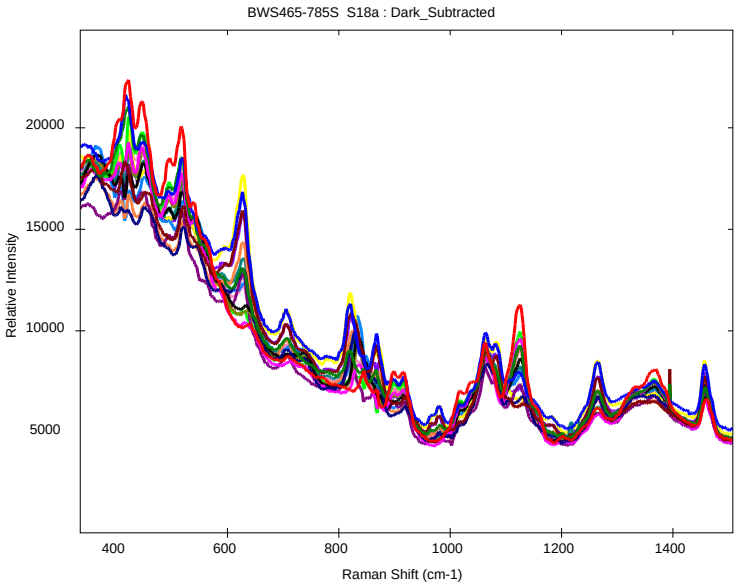

***Figure 2:** Overlay of Raman spectra of tertiary solutions of glucose, fructose and sucrose*

A three factor PLS model was developed for each of the three components. The concentrations were plotted to show the accuracy of the model. The results for glucose are shown in Figure 3, and have an R^2 value greater than 0.999 for both the measured and predicted concentrations. Similarly, the RMSEC and RMSEP were calculated to be less than 0.008.

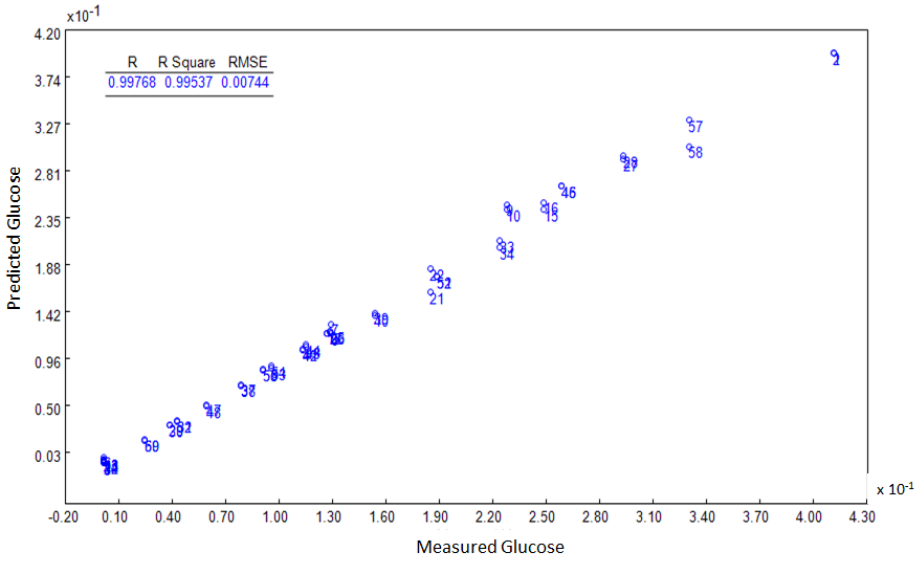

***Figure 3**: Measured vs. predicted plot of concentration percentage for Glucose 3 Factor PLS model*

Samples that were not used in the calibration were used as a prediction set using the calibration model developed in the BWIQ software. The models were used to successfully determine the concentration in these samples.

Conclusions

From this experiment, we have shown the successful use of the i-Raman Plus portable Raman spectrometer with BWIQ to develop a quantitative model to determine the concentration of glucose in tertiary aqueous mixtures of glucose, sucrose, and fructose as shown in Figure 1. For more information please visit www.bwtek.com or call +1 (855) BW-RAMAN.

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