L.10: Raman spectroscopy for detection of urea adulteration in milk

Urea is often added to milk by unscrupulous elements for commercial benefits. Urea is an end product of nitrogen metabolism and is a normal constituent of milk. A cut off limit for urea concentration in milk is normally accepted to be ~70 mg/dl. Consumption of milk with a urea concentration above this cut off limit is believed to cause severe health problems for human beings. Hence, detection of urea and its quantitative estimation have great significance in dairy industries. Numerous types of urea sensors have been proposed in literature based on a variety of techniques including conduct-metric, potentiometric, thermometric, and optical methods. Each of these methods has been shown to be useful in certain situations and research aimed at improving the accuracy of these approaches is ongoing. In recent years, the advantages of Raman spectroscopy over other analytical techniques have increased its acceptance in various industrial fields. We, at OSDL, LBAID carried out an exploratory study to evaluate the applicability of Raman spectroscopy for rapid quantitative determination of urea adulteration in milk.

"Sanchi" milk, a product of Madhya Pradesh Cooperative Dairy Federation was procured for this study. Ten different sets of milk adulteration were prepared with each set containing milk samples added with urea at various concentrations ranging from 0-1000 mg/dl. The Raman spectra were measured with an in-house built Raman spectroscopy set up (Fig.L.10.1) that incorporates a 785 nm diode laser for the excitation and a TE cooled spectrograph cum CCD for the Raman signal measurement.

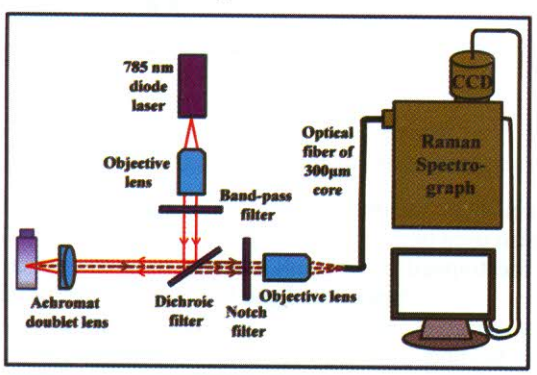

Fig.L.10.1: Experimental arrangement for urea adulteration detection in milk using Raman spectroscopy

For each urea concentration, five different spectra were recorded which were then averaged to get a mean Raman spectrum corresponding to that concentration. Standard normal variate (SNV) transformation was applied to these spectra to offset for any baseline shift.

An algorithm based on partial least square (PLS) regression was developed to quantitatively predict the concentration of urea where the set of pre-processed spectra was used as input. The accuracy of the regression algorithm was assessed by evaluating the root mean square error (RMSE), which measured the average difference between predicted and reference concentration values. The RMSE was separately estimated for the calibration and the validation data sets. The choice of the number of PLS components was based on the minimum RMSE (Fig.L.10.2).

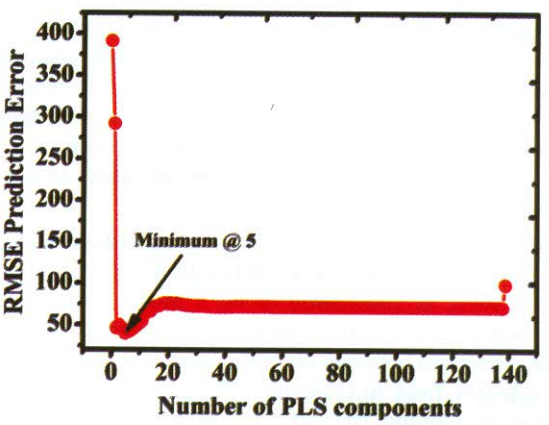

Fig.L.10.2: RMSE as a function of number of PLS components

A comparison of the concentration of urea estimated using the PLS algorithm with that of the reference concentration is shown in Fig. L.10.3. The predictive performance of the algorithm was measured by calculating the R^2 value that was found to be 0.99. The results suggested that the present set-up along-with the PLS algorithm could measure urea concentration with an accuracy of > 97 % above 100 mg/dl, while between 50 and 100 mg/dl, the accuracy lies within 90-95%. The details of these studies can be found in Food Analytical Methods, 8(1), 93-102, 2015.

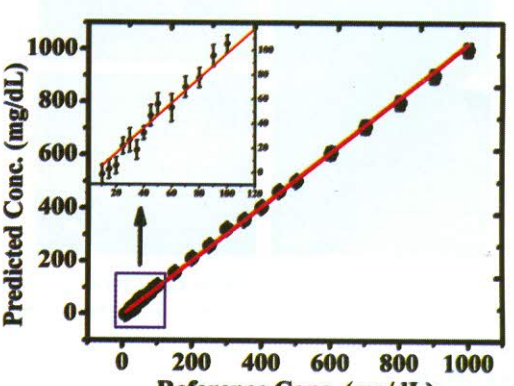

Fig.L.10.3: Prediction of urea concentration in milk.

Reported by:
K.M. Khan and S.K. Majumder(shkm@rrcat.gov.in)