

Abstract

The importance of determining phase changes when developing near infrared spectroscopic calibrations

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Introduction

When one does not know where the data come from, it is a relatively straight-forward matter to simply line up the X and Y data blocks and then build a relationship. The *SECV* can be determined and the results used or compared to other techniques or instrumentation.

However the data, the spectra represent real materials, and real materials can violate the fundamental assumptions under the calibration process. One of those assumptions is that the analyte stays in the same form no matter the concentration. This is frequently not the case. For example, if one calibrates for percent alcohol in water the material does not stay in the same chemical phase from zero percent alcohol to 100%. At the beginning one has alcohol dissolved in water and at the other extreme, one has water dissolved in alcohol.

This type of phase change has been observed in the feed ingredient DDGS (dry distiller's grain and syrups). The phase and bonding pattern of the moisture changes as one goes from very dry to very wet material. The shape of the water molecules, their bonding patterns and their spectra all change. Therefore, it is not reasonable to expect a calibration to remain linear across this range. In fact the NIR spectra show this and show at what concentrations the moisture in DDGS changes phase.

Experimental

The NIR spectra of several hundred DDGS samples were obtained on a Perten DA7200. These spectra were compared to the moisture as determined by weight loss on drying. Results are presented comparing calibrations over the entire range and calibrations over a single and multiple split range.

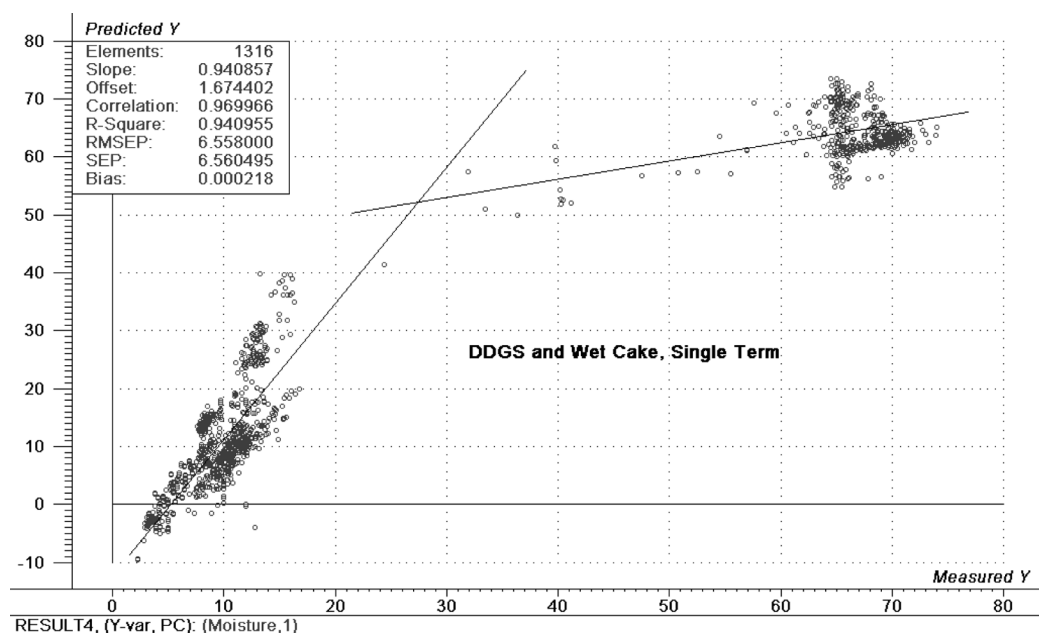


Figure 1. Scatter plots illustrating the splitting of samples based on their chemistry and spectroscopy.

Results

The idea of splitting calibrations into ranges is not new. The work shown here demonstrates that there is a natural or best point for such a split based on the chemistry and spectroscopy of the situation.