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Application of infrared portable sensor technology for predicting perceived astringency of acidic whey protein beverages

Ting Wang,* Siow-Ying Tan,† William Mutilangi,† Marcal Plans,* and Luis Rodriguez-Saona*[1]
*Department of Food Science and Technology, The Ohio State University, Columbus 43210
†Pepsi-Cola Company, Valhalla, NY 10595

ABSTRACT

Formulating whey protein beverages at acidic pH provides better clarity but the beverages typically develop an unpleasant and astringent flavor. Our aim was to evaluate the application of infrared spectroscopy and chemometrics in predicting astringency of acidic whey protein beverages. Whey protein isolate (WPI), whey protein concentrate (WPC), and whey protein hydrolysate (WPH) from different manufacturers were used to formulate beverages at pH ranging from 2.2 to 3.9. Trained panelists using the spectrum method of descriptive analysis tested the beverages providing astringency scores. A portable Fourier transform infrared spectroscopy attenuated total reflectance spectrometer was used for spectra collection that was analyzed by multivariate regression analysis (partial least squares regression) to build calibration models with the sensory astringency scores. Beverage astringency scores fluctuated from 1.9 to 5.2 units and were explained by pH, protein type (WPC, WPI, or WPH), source (manufacturer), and their interactions, revealing the complexity of astringency development in acidic whey protein beverages. The WPC and WPH beverages showed an increase in astringency as the pH of the solution was lowered, but no relationship was found for WPI beverages. The partial least squares regression analysis showed strong relationship between the reference astringency scores and the infrared predicted values (correlation coefficient >0.94), giving standard error of cross-validation ranging from 0.08 to 0.12 units, depending on whey protein type. Major absorption bands explaining astringency scores were associated with carboxylic groups and amide regions of proteins. The portable infrared technique allowed rapid prediction of astringency of acidic whey protein beverages, providing the industry a novel tool for monitoring sensory characteristics of whey-containing beverages.

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[1]Corresponding author: rodriguez-saona.1@osu.edu

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INTRODUCTION

The use of whey proteins in beverages is growing because of their high nutritional value, wide functional versatility, and association with health benefits such as weight control and muscle building. Beverage clarity increases at low pH (Miller, 2007); however, astringency perception also increases at low pH (Ye et al., 2011). The astringency development is a significant product challenge limiting consumer acceptance (Sano et al., 2005; Beecher et al., 2008).

Astringency is described as the complex of sensations due to shrinking, drawing, or puckering of the epithelium as a result of exposure to substances such as alums or tannins (Gibbins and Carpenter, 2013). Astringent molecules are commonly plant-based products, most commonly tannins, and the most usual form of exposure to these products worldwide is through polyphenols in drinking tea (Gibbins and Carpenter, 2013). Other astringent compounds include acids and metal alums and other dehydrating agents such as alcohols that create the subjective feeling of astringency (Gibbins and Carpenter, 2013). The mechanism of astringency is attributed to polyphenolic compounds (Haslam et al., 1988) binding to and precipitating specific salivary proteins (Baxter et al., 1997; Kallithraka et al., 1998) and limiting lubricity in the mouth (Clifford, 1996). Gibbins and Carpenter (2013) attribute astringency development to a loss of mucosal lubrication by altering the salivary bulk, saliva rheology, and saliva pellicle, leading to an increase of friction in the oral cavity. Studies to identify the mechanism driving astringency in acidic whey protein beverages have been inconclusive; however, some mechanisms have been proposed. Beecher et al. (2008) suggested that whey proteins become positively charged at low pH, and could bind and aggregate with salivary proteins (Sano et al., 2005; Vardhanabhuti et al., 2010). However, Lee and Vickers (2008) suggested that the astringency of acidic whey protein beverages was possibly caused by their high acidity. Ye et al. (2011) reported that protein aggregation that causes astringency at pH 3.4 and pH 2.0 can be attributed to whey protein and salivary protein interacting via electrostatic interactions at pH 3.4, whereas only salivary proteins undergo aggregation at pH 2.0.

Currently, astringency of whey protein beverages is determined by trained sensory panelists, a benchmark in qualifying and quantifying sensory properties; however, it is laborious, expensive, and time consuming. The demand for protein-fortified foods and beverages is increasing steadily. A quick and reliable method to predict astringency in low pH beverages will enable efficiency in early ingredient screening and product development. Mid-infrared spectroscopy (4,000–700 cm^{-1}) is a rapid, sensitive, and high-throughput method commonly used in analysis of food components. The output is a molecular "fingerprint" spectrum of the absorption bands corresponding to frequencies of vibrations between the bonds of the atoms making up the material. Optical technology is been rapidly developed and instruments are already available commercially as portable, handheld, and micro-devices (Ellis and Goodacre, 2006). Ellis et al. (2015) summarized the developments in portable and handheld or remote sensor devices (or both) for on/at-line analysis at points of vulnerability along the complex food supply networks. Advantages of these sensor devices include low cost, small size, compactness, robustness, and ease of operation for in-field routine analysis. Furthermore, specific bands arising from group vibrations may be assigned to known specific chemical entities in most cases. Developments in the field of multivariate techniques have been prompted by the need of reliable, accurate, robust, and simple methods for routine analysis of spectroscopic data (Udelhoven et al., 2000). Partial least squares regression (**PLSR**) method is the most commonly used regression algorithm in molecular spectroscopy. The algorithm seeks for the best decomposition of the X matrix that will best describe the Y matrix (Roggo et al., 2007). The PLSR method derives its usefulness from its ability to analyze noisy and collinear data (Wold et al., 2001) combining the features of principal component analysis and multilinear regression to compress a large number of variables into a few latent variables [partial least squares (**PLS**) factors] by attempting to explain the maximum variance in both the spectra and reference data sets (Karoui and De Baerdemaeker, 2007).

Mid-infrared spectroscopy has been widely used for assessing milk quality such as predicting milk composition (Lynch et al., 2006), protein composition (De Marchi et al., 2009a; Bonfatti et al., 2011), milk coagulation properties (De Marchi et al., 2009b), milk fat composition (Rutten et al., 2009; Soyeurt et al., 2011), and major mineral contents (Soyeurt et al., 2009). Because the value and functionality of whey protein products are largely affected by their composition, infrared spectroscopy combined with chemometrics has shown potential in correlating various functional properties of whey and casein hydrolysates (van der Ven et al., 2002) and characterizing whey protein powders [whey protein concentrate (**WPC**), whey protein isolate (**WPI**), and whey protein hydrolysate (**WPH**)] based on their unique infrared profile showing well-separated clustering among the powder samples (Wang et al., 2015). In contrast, a limited number of infrared applications have been reported for predicting perceived astringency (Ferrer-Gallego et al., 2013) with major emphasis directed toward quantification of tannins in wine (Edelmann and Lendl, 2002; Fernandez and Agosin, 2007; Noypitaka et al., 2015).

Our objective was to develop a predictive model for estimating astringency in acidic whey protein beverages based on the relationship between the perceived intensity of the target astringency stimuli and unique spectral fingerprints from a portable infrared spectrometer. In addition, we will explore the protein chemical modifications associated with perceived astringency in acidic beverages based on the unique PLS regression vector information associated with the underlying chemistry of the models.

MATERIALS AND METHODS

Formulation of Whey Protein Beverages and Sensory Astringency Scores

Whey protein beverages were produced from WPI, WPC, or WPH powders obtained from 9 different commercial manufacturers for a total of 19 different whey protein products. Beverages contained 5 g of protein per 8 oz (21.1 g/L) equivalent, sucrose (8% wt/wt) was added in each formulation, and phosphoric acid (40%) or potassium carbonate (20%) solutions were used to adjust the pH of individual beverage sample to target. Beverage samples were formulated from whey protein powders [WPI (9), WPC (6), and WPH (4)] and adjusted to different pH values ranging from 2.2 to 3.9 (Table 1). Each sample was coded with a 3-digit number and vacuum packed in a glass bottle. Beverage samples were maintained at 4°C until spectra collection using FTIR. A validation set was produced with 3 different powder sources of WPI, and one WPH along with sucrose (8% wt/wt), and their pH were adjusted to 3.0 and 3.8.

Quantitative sensory test on all whey protein beverage samples was conducted at the Sensory Service Center in North Carolina State University. Ten panelists