OPTICAL ROTATION MEASUREMENTS IN FOOD INDUSTRY PROCESSES

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Abstract: An optical system was developed using commercial low-cost optical components to monitor food processes by measuring changes in optical rotation (OR) of chiral compounds. The OR signal as a function of processing time and the sample temperature were collected and recorded using a computer data acquisition system. Sucrose (1 g/100 ml solution) and distilled water were used to calibrate the instrument. Sucrose solutions (0.01 to 30 g/100 ml solution) were used to determine its sensitivity, linearity and stability. The instrument was validated by monitoring the hydrolysis of sucrose. System has been tested during a fermentation process and sugar-protein interaction as a function of temperature.

1. INTRODUCTION

High-sensitivity measurements based on the optical activity of specific food components can be used as feedback to control processes and improve product quality. In the food industry, sugar inversion and alcoholic fermentations are potential applications of high significance that could be monitored by following changes in optical rotation (OR). Sugar inversion or sucrose hydrolysis determines major quality factors of final products like candies or fermented beverages. Off-line measurements even after standardization of processes and product formulations are often insufficient to control sugar inversion. Sugar inversion must be monitored and controlled to obtain a desired sweetness, texture and stickiness in hard candies.

A potential application of OR measurements is the in-line/real-time monitoring of alcoholic fermentations to reduce quality variability by making timely adjustments to variables such as temperature. Several techniques to measure ethanol content have been standardized by the American Society of Brewing Chemists (ASBC), but they are all off-line measurements.

The objective of this work was to design, build and evaluate an OR measurement system to monitor and control processes such as alcoholic fermentations or sugar-protein interaction. This required the calibration of the instrument and to determine its sensitivity, linearity, and stability. It was also validated by monitoring the hydrolysis of sucrose and comparing initial and final OR values with those reported in the literature.

The instrument performance was evaluated by characterizing in-line the fermentation process of beer production and sugar-protein interaction as a function of temperature.

2. MATERIALS AND METHODS

2.1. Optical system description

The optical system set-up (Figure 1) used a polarized 652-nm semiconductor laser beam modulated at 50 KHz using a photoelastic modulator[1]. The modulated light beam passed through the solution under study contained in a temperature-controlled cell and then through a second polarizer. The cell path length used for a given experiment was the one best suited to maintain the optical signal within the range of the instrument.

Changes in the polarization of the light beam were monitored using a silicon detector, and its electric signal was filtered by a lock-in amplifier. This signal and solution temperature were collected and recorded in real-time using a computer data acquisition system (DAQ 6036E National Instruments Inc., Austin, TX).

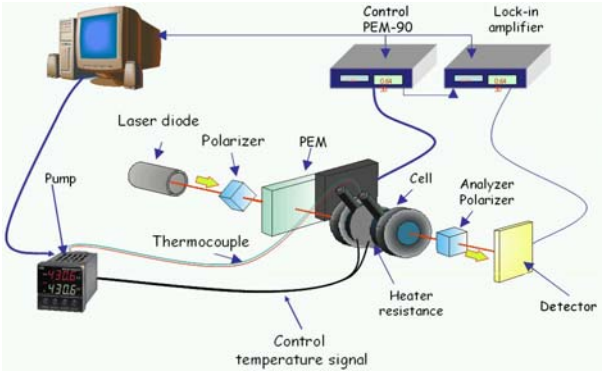

***Fig. 1** Optical rotation system*

The instrument was calibrated using distilled water for baseline and sucrose (1 g/100 ml solution) as a reference standard[1,2]. Sucrose (0.01 to 30 g/100 ml solution) was used to determine its sensitivity, linearity, and stability.

2.2. Applications

2.2.1. Brewing.

One-liter samples of fermenting wort were obtained from the OSU Pilot Brewing facility. Continuous monitoring of fermentation was accomplished by circulating fermenting wort through the optical instrument sample cell using a peristaltic pump (Figure 2).
A 1-cm path length cell was used for in-line/real-time monitoring of beer fermentation. The process was carried out at room temperature, as is typical for beer production.

2.2.2. Protein-sugar interaction

Denaturation of proteins can be prevented or retarded by using stabilizers, including sugars, polyols, aminoacids, methylamines, inorganic salts, carboxylic acids, quaternary amines, nucleotides, and surfactants[4]. During freeze- or air-drying, water molecules are removed from the protein surface and the cross-linking of adjacent proteins induces aggregation. To prevent such protein denaturation/ aggregation, the stabilizing agent might interact with hydrophilic sites on the protein surface released by the water molecules. Different sugars can be added to the protein solution, and their interaction can be studied as a function of temperature. In this work, trehalose was added to bovine serum albumin (BSA) solution, and heated during 30 minutes, from room temperature to around 85°C.

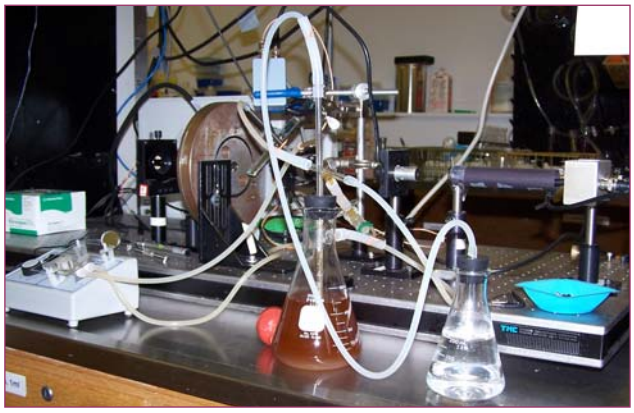

***Fig. 2** Optical instrument and peristaltic pump*

3. RESULTS

3.1. Optical system characterization and validation

3.1.1. Characterization

Using a 5-cm path length, the system linearity with respect to sucrose concentration gave a correlation factor of 0.999 (Figure 3). The OR sensitivity was determined to be 0.002° for low concentrations, as can be seen in the zoom-in graph in Figure 3, where error bars are also included.

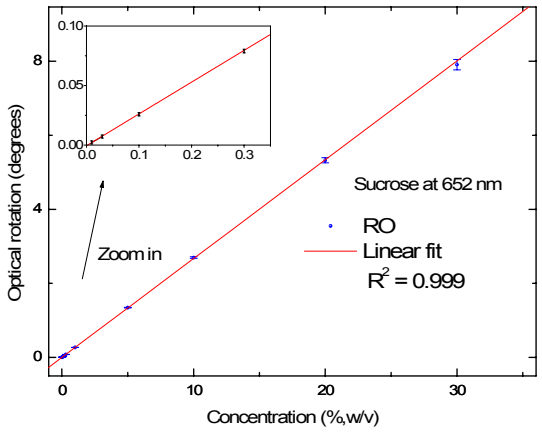

***Fig. 3** Optical rotation of sucrose solutions using a 5-cm cell path length. Error bars were obtained from 300 measurements made over 5 minutes*

A 24-h test showed stability values better than 2% of measured value. These three characteristics, i.e.,

sensitivity, linearity and stability, demonstrate that the optical system can obtain reliable data with high accuracy.

3.1.2. Validation

The hydrolysis at 60°C of a sucrose solution (5 g/100 ml, adjusted to pH 2.1 with 0.6 M HCl) was monitored by OR measurements to validate the performance of the system in applications based on sugar inversion measurements (Fig. 4). In this experiment a monochromator was used to adjust wavelength to 589 nm. Aliquots (15 ml) were collected in test tubes and cooled in an ice bath to stop the sugar hydrolysis. The solution was then neutralized to pH 7 with 0.1 M NaOH before OR measurements. The initial OR value of α = 1.65° was equivalent to a specific rotation [α] = -66°, which is close to the value reported in the literature ([α] = -66.5° at 589 nm). Initial measurements were made more often because the reaction is faster at the beginning, as previously reported[4]. The last sample reached a value of α = -0.39°, which is close to the literature inverted sucrose value of α = -0.5° for inverted sucrose, equivalent to specific rotation of [α] = -20° at 589 nm[5].

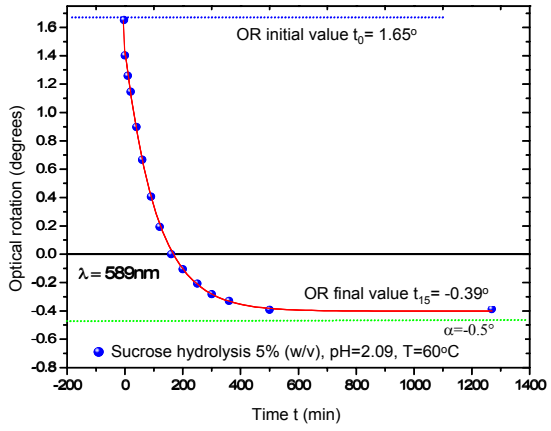

Fig. 4 *Sucrose hydrolysis showing the characteristic exponential decay of first-order reaction kinetics. (Sucrose solution at 5 g/100 ml, acid hydrolyzed at pH=2.09 and 60°C).*

3.2. Optical system application examples

Figure 5 shows OR values for wort during a fermentation process. An example of an experimental run is summarized, showing specific density, ethanol concentration and OR measurements as a function of time. As expected, the ethanol concentration, measured directly (Figure 5(a), increased over the time period where the density decreased. Optical rotation (OR) data closely followed the off-line density data in Figure 5(b). False "peaks" appeared in OR measurements close to time intervals where aliquots were taken for density characterization. They are produced by change from laminar flow to turbid flow-regime which remains for several minutes. This problem can be solved changing the place were samples are taken or eliminating sampling after system validation.

The highly significant linear correlations between in-line/real-time OR measurements and off-line ethanol and density measurements validated OR as an in-situ measurement technique for monitoring the progress of fermentation. In addition, the OR signal intensity could be used to obtain an indication of initial and final sugar concentration if combined with signal calibration, characterization of raw materials, and information on previously observed brewing process conditions. Overall, these findings suggest that an in-line/real-time data acquisition system based on OR measurements can improve beer brewing control as well as other alcoholic fermentation processes.

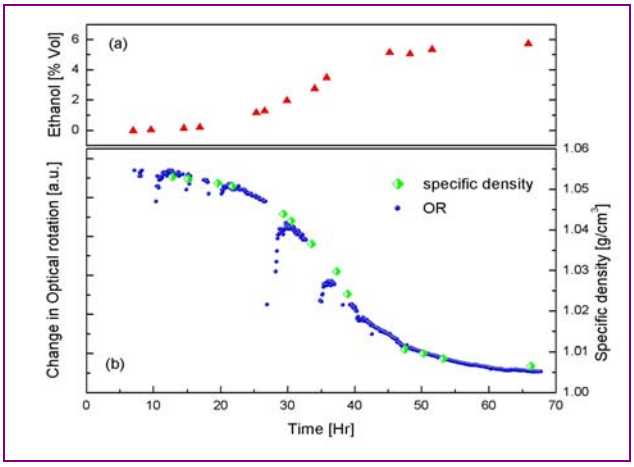

Fig. 5 *Optical rotation, specific density and ethanol content as a function of fermentation process time*

A second application experiment was the monitoring of protein-sugar interaction in dependence on temperature. Figure 6 shows a comparison of the optical rotation signal for protein solution and the corresponding protein-trehalose solution, during heating and cooling time.

Protein alone imparts the solution a levorotatory optical rotation. When trehalose, which is dextrorotatory, is added, the overall rotation produced is dextrorotatory. The plots show data points obtained during heating and cooling processes, with arrows indicating the progression of time. A clear difference in variability can be seen between plots, which can be attributed to some degree of stabilization produced by trehalose. The signal begins to change around 60°C, which is close to denaturation temperature for bovine serum albumin. A final average value is reached after some time around 80°C, but it is different for the two samples.

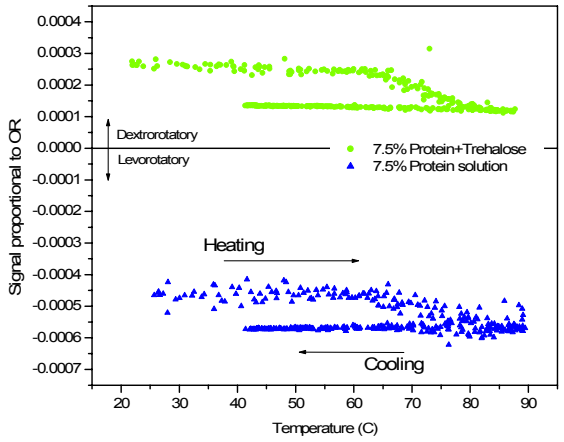

Fig 6 Comparison of optical rotation signal for protein and protein-trehalose solutions, during heating and cooling time.

Trehalose prevents denaturation over several degrees. During the cooling process, the signal does not change, indicating that proteins have been denatured in an irreversibly way.

4. CONCLUSION

The sensitivity and modular construction of the system demonstrated that OR measurements can be used as an in-line/real-time measurement technique for quality control in food processing. This optical system set-up has no moving parts and is more flexible than commercial polarimeters, providing the capability to be used in harsh environments. It could be implemented by the food and beverage industry for the in-line/real-time monitoring of industrial fermentations. This would facilitate detection of the fermentation endpoint and the optimization of process conditions for best product quality and cost control. The system should also be applicable to advantage in other industrial food processes which involve a significant change in optical rotation.

Finally, it can also be used as a research instrument to study protein denaturation by heat, acidification, pressure, and other factors used in food processing. Of particular interest would be studies on the interaction of proteins with sugars and other stabilizers of protein conformation.

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