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Sequential injection system for simultaneous determination of sucrose and phosphate in cola drinks using paired emitter-detector diode sensor

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ABSTRACT

This work presents the simultaneous determination of sucrose and phosphate by using sequential injection (SI) system with a low cost paired emitter-detector diode (PEDD) light sensor. The PEDD uses two 890 nm LEDs. Measurement of sucrose in Brix unit was carried out based on the detection of light refraction occurring at the liquid interface (the schlieren effect) between the sucrose solution and water. Phosphate was measured from the formation of calcium phosphate with turbidimetric detection. With careful design of the loading sequence and volume (sample–precipitating reagent–sample), simultaneous detection of sucrose and phosphate was accomplished with the single PEDD detector. At the optimized condition, linear calibrations from 1 to 7 Brix sucrose and from 50 to 200 mg PO_4^{3-} L^{-1} were obtained. Good precision at lower than 2% RSD ($n=10$) for both analytes with satisfactory throughput of 21 injections h^{-1} was achieved. The method was successfully applied for the determination of sucrose and phosphate in cola drinks. The proposed method is readily applicable for automation and is found to be an alternative method to conventional procedures for on-line quality control process in cola drink industry.

1. Introduction

Light emitting diodes (LEDs) are commonly used as a light source for portable spectrometers due to the low power consumption and brightness [1]. Their emission spectra are rather narrow (about ± 40 nm), allowing specific selection of wavelengths without an expensive monochromator or filter. However, LEDs can also be used as a light detector [2,3]. Paired emitter-detector diode (PEDD) is an inexpensive optical sensor that consists of a pair of LEDs where one LED is used as a light emitter (LED emitter) and the other LED as a light detector (LED detector). Use of PEDD as detector of light is attractive in terms of costs, size and broad range of wavelengths from UV to near-infrared region (ca. 380–900 nm). Unfortunately, LED as detector generates very small photocurrent. Diamond's group [4–6] demonstrated the accurate and precise measurement of the photocurrent using a threshold detector and timer circuit. The principle is based on measurement of the time taken for the photon-induced current to discharge the reverse-biased LED from an initial 5 V to 1.7 V. Later, another approach was proposed by using direct measurement of the voltage generated at the LED detector [7,8]. This method is very simple and convenient employing a common pH-meter or a digital multimeter with high input impedance.

The use of PEDD for absorbance measurement has been reported as early as 2004 when Diamond et al. employed the device as optical sensor for colorimetric analysis of dyes for pH measurement [4]. Since then, application of PEDD detector has been broaden to environmental field such as detection of heavy metals [9] and phosphate [10] in water samples as well as to bioanalytical field, such as detection of hemoglobin [11] and alkaline phosphatase activity [12–14]. PEDD has also been adopted as a detector in post column HPLC [15] and IC [16,17]. Recently, PEDD compatible with optosensing films has been developed for sequential injection (SI) system [18,19]. Prussian Blue film was used as a model optical chemoreceptor to detect hydrogen peroxide [18]. Determination of glucose in serum was demonstrated for this application [19]. With different configurations, the PEDD can also be used for fluorescence detection. The first report

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of LED as fluorescence detector was in 2010 for the determination of quinine in beverage drinks [20]. Another application of fluorometric PEDD was the detection of calcium using complexation reaction with calcein [21,22].

The phenomena of light refraction and light scattering are alternative detection methods useful in analytical practice, especially with colored sample. This is because they can employ wavelengths at which that the colored solution does not absorb the light. Detection of light refraction at the liquid interface (the schlieren effect) was employed as detection in liquid chromatography using deflection of a laser beam [23] and in capillary electrophoresis [24]. In addition, the schlieren effect was also applied to flow analysis for quantitation of sucrose [25,26], alcohol [27] and glycol [28]. Another approach for analysis of colored sample is detection based on light scattering of colloidal particles (turbidimetry) [29] has been widely used through the formation of solid particles using suitable precipitating reagents. With coupling to flow-based system, it provides automation for several applications such as in environmental [30], biological [31] and food [32] samples.

In this work, implementation of PEDD for detection of the schlieren effect and the turbidity in liquid-flow system is presented. We selected sucrose and phosphate as model analytes to demonstrate this application for possible quality control in cola drink industry. The work used a sequential injection (SI) system [33]. With optimized selection of the loading sequence, simultaneous detection of the two analyte is carried out with one PEDD detector. The system is thus simple and compact.

2. Experimental

2.1. Chemicals and reagents

All solutions were prepared in deionized water (Barnstead EASYpure II, USA). The sucrose standard used in this work was commercial grade sugar (Mitr Phol, Thailand). A 50 Brix (Bx) stock sucrose solution was prepared by dissolving exactly 50.00 g of solid sucrose in 50.00 g of deionized water with stirring on a magnetic stirrer until the solid has completely dissolved. The stock solution was stored at 4 °C and used within one week.

A 5000 mg PO_4^{3-} L^{-1} stock phosphate solution was prepared by dissolving 0.7157 g of potassium dihydrogenphosphate (Fluka, Switzerland), previously dried at 60 °C for 2 h and kept in a desiccator, in 100.00 mL with deionized water. Working standard solutions used for calibration were mixed standards of sucrose and phosphate, prepared in deionized water by appropriate dilution of the stock solutions.

The precipitating reagent (R in Fig. 1) was a solution of 0.08 mol L^{-1} $CaCl_2$ in 0.1% (w/v) polyvinyl alcohol (PVA). This solution was prepared by weighing 0.888 g of calcium chloride (Merck, USA) and 0.10 g of PVA (Merck, USA), dissolving in approximately 90 mL of 0.3 mol L^{-1} ammonium buffer pH 10 and heating on a hot plate with magnetic stirring until all solids dissolved. After cooling to room temperature, ammonium buffer was added to make 100.0 mL. The buffer was prepared by dissolving 0.1338 g of ammonium chloride (Ajax Finechem, New Zealand) and 0.9 mL of 30%(w/w) ammonia (density 0.892 g L^{-1}, Panreac, Spain) in deionized water to give 100 mL.

2.2. Sample preparation

Regular and sugar-free cola drink samples were purchased from supermarkets in Bangkok, Thailand. All samples were degassed in an ultrasonic bath for 15 min. Dilutions of samples with water (1:1) were carried out prior to analysis. Five synthetic samples were also analyzed. These samples were prepared by dissolving analytical grade sucrose (UNIVAR, Australia) and potassium dihydrogenphosphate (Fluka, Switzerland) in deionized water (Table 2).

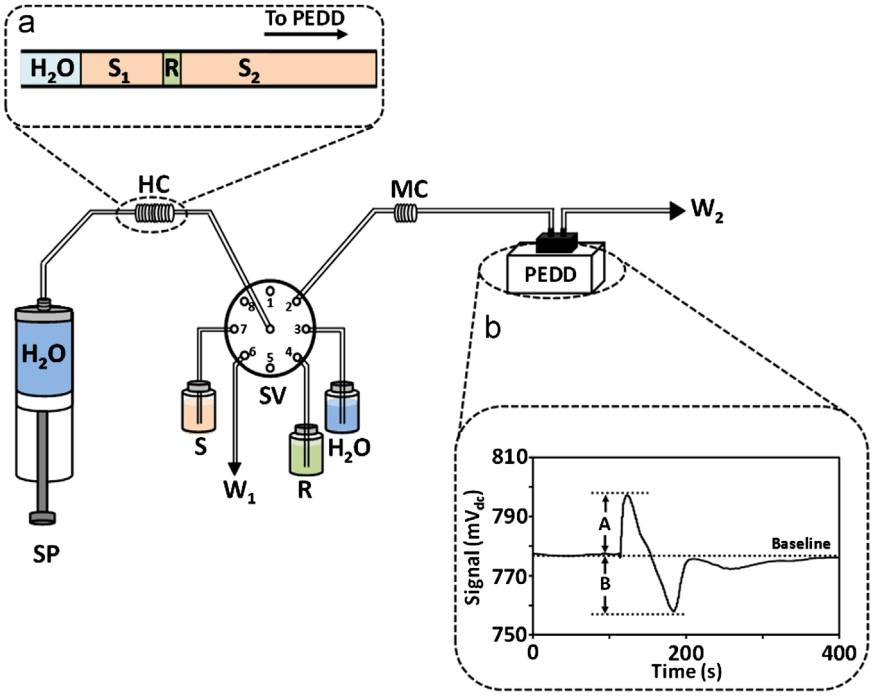

Fig. 1. Schematic diagram of the SI-PEDD system for simultaneous determination of sucrose and phosphate in cola drinks: S_1, 1st aspirated sample zone; R, precipitating reagent 0.08 mol L^{-1} $CaCl_2$ with 0.1%(w/v) PVA in ammonium buffer pH 10; S_2, 2nd aspirated sample zone; W, waste; SP, syringe pump; SV, selection valve; HC, 4.6-mL holding coil (i.d. 1 mm, 588 cm long); MC, mixing coil (i.d. 1 mm, 144 cm long); inset (a), sequence of the detection zone in HC; inset (b), signal profile of a mixed standard of sucrose and phosphate.