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Exploiting an automated microfluidic hydrodynamic sequential injection system for determination of phosphate[☆]

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ABSTRACT

A microfluidic hydrodynamic sequential injection (μ HSI) spectrophotometric system was designed and fabricated. The system was built by laser engraving a manifold pattern on an acrylic block and sealing with another flat acrylic plate to form a microfluidic channel platform. The platform was incorporated with small solenoid valves to obtain a portable setup for programmable control of the liquid flow into the channel according to the HSI principle. The system was demonstrated for the determination of phosphate using a molybdenum blue method. An ascorbic acid, standard or sample, and acidic molybdate solutions were sequentially aspirated to fill the channel forming a stack zone before flowing to the detector. Under the optimum condition, a linear calibration graph in the range of 0.1–6 mg P L⁻¹ was obtained. The detection limit was 0.1 mg L⁻¹. The system is compact (5.0 mm thick, 80 mm wide \times 140 mm long), durable, portable, cost-effective, and consumes little amount of chemicals (83 μ L each of molybdate and ascorbic acid, 133 μ L of the sample solution and 1.7 mL of water carrier/run). It was applied for the determination of phosphate content in extracted soil samples. The percent recoveries of the analysis were obtained in the range of 91.2–107.3. The results obtained agreed well with those of the batch spectrophotometric method.

1. Introduction

The recent development of new methods has focused mainly on greener analytical systems which minimize the use of toxic chemicals and waste production to reduce exposure of humans to hazardous chemicals and environmental pollution [1]. Several flow-based analyses have been proposed for development of green chemistry such as flow injection (FI), sequential injection (SI), bead injection (BI), lab-on-chip (LOC), lab-on valve (LOV), and multi-syringe flow analysis (MSFA), etc. Despite many advantages, these techniques may require high cost instrumentation and/or consume high amount of chemicals. A microfluidic-hydrodynamic sequential injection (μ HSI) system is proposed to minimize these drawbacks.

The HSI was developed based on the hydrodynamic injection (HI) principle that was first proposed in the flow-based analysis field by Ruzicka and Hansen in 1983 as an alternative highly reproducible

sample introduction means for the FI system [2]. In HSI, reagent(s) and sample are sequentially introduced into the system to form stacked zones, while the carrier stream is stopped. Volume of each zone depends on the length of the zone which is designated by the length and size of the channel that accommodates each of the aspirated solutions, see Fig. 1. When the carrier flow is resumed, the zones of reagents and sample are mixed while moving to the detector. The operation and advantages of the hydrodynamic sequential injection (HSI) system is similar to that of a sequential injection (SI) system, but it does not require an expensive syringe pump and a selection valve. Precise loading volume, automatic operation, and low volume chemical consumption can be achieved in the HSI by using a simple peristaltic pump, low cost solenoid valves, and a simple controller. As compared to the FI system, which also employs simple and low-cost devices but operates in a continuous flow mode, the FI system usually consumes a higher amount of chemical solutions. Therefore, HSI can overcome some drawbacks of

[☆] This article is dedicated to Professor Gary D. Christian on the anniversary of his 80th birthday.

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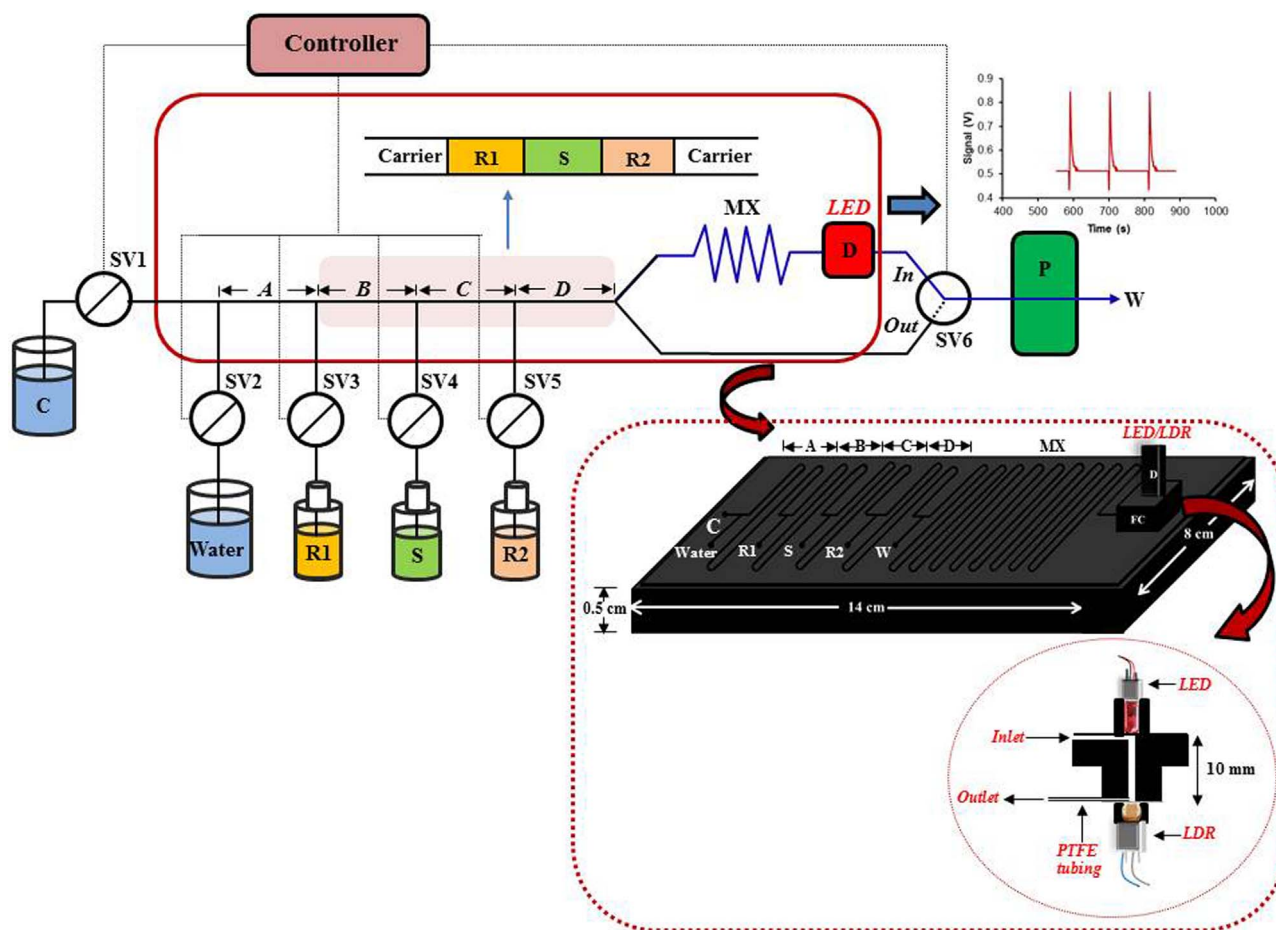


Fig. 1. The μ HSI system for determination of phosphate: C = Carrier (Water), R1 = Ascorbic acid, S = Standard/sample, R2 = Molybdate solution, P = Peristaltic pump, SV = Solenoid valve, MX = Mixing zone, FC = Flow cell, D = LED/ LDR colorimetry and W = Waste.

both SI and FI, especially in terms of cost and chemical consumption.

HSI was presented for the first time by J. Jakmuneč at the International Conference on Flow Injection Analysis (ICFIA 2005) which was organized by Gary Christian and Sue Christian [3]. Later on, many HSI systems were developed for determination of various analytes. For example, an automated HSI was developed for the determination of manganese [4], phosphate and silicate [5], nitrite and nitrate [6] in environmental samples such as soil, water, wastewater and fish pond water. In addition, the HSI system can be applied to biological samples for screening of beta-thalassemia trait [7,8]. These researches have focused mainly on the development of fully automated cost-effective systems that also reduce amounts of chemicals consumption and toxic wastes. However, the development of a durable, compact/portable, and rapid analysis system is also important for more applications, especially for field analyses such as auto-analyzer/monitoring system for the “fit to purpose” applications.

In this work, we developed a miniaturized μ HSI system and demonstrated its performance for the determination of phosphate which was selected as a model analyte. Phosphate is a major plant nutrient that should be monitored in agricultural soil in order to regulate the appropriate amount of fertilizer to add to the soil. Excess phosphate in soil may leach into the aquatic environments resulting in the eutrophication phenomenon [5]. Standard methods for the determination of phosphate are based on the reaction of phosphate with acidic molybdate, producing heteropolymolybdate [5,9] that is reacted with a reducing agent such as stannous chloride [10] or ascorbic acid [11] to form a molybdenum blue compound.

Several flow-based analyses with colorimetric/spectrophotometric detection have been proposed for determination of phosphate. Micro-

flow injection analysis (μ FIA) was developed for determination of orthophosphate as a molybdenum blue complex [12]. SI system has been applied for the determination of phosphate and silicate using vanadomolybdate [13] or molybdenum blue method [14]. A stopped-flow SIA with lab-on-valve has been proposed for determination of phosphate that is based on the reduction of molybdenum yellow to molybdenum blue by ascorbic acid in the presence of antimonyl tartrate serving as a catalyst. In addition, the FIA with a stopped-flow mode has been proposed for determination of phosphate and silicate simultaneously using molybdenum blue [15,16] or using oxalic acid to improve the selectivity of the formation of both reduced molybdophosphoric and molybdosilicic acids [14]. Moreover, the use of an inexpensive homemade long path length spectrophotometric flow cell has been proposed to measure phosphate at trace level in natural water based on the formation of an ion association complex of hetero polymolybdophosphate with bismuth [17].

In this work, the automated μ HSI system was developed aiming at a more compact, more durable, and more cost-effective device with low consumption of the chemicals. The μ HSI platform was fabricated by laser engraving an acrylic block to form a pattern of the manifold of the desired length, width, and depth on the surface of the block, then sealed with another acrylic plate to create a closed channel. Solutions were introduced into the platform through the solenoid valves embedded onto the acrylic plate to obtain a portable setup that can be controlled by the software program to allow sequential aspiration of various solutions into the system. The system was successfully demonstrated for the determination of phosphate with analytical features comparable to other flow-based systems. In addition, the automated μ HSI system provides sample throughput of 30 h^{-1} and consumed chemical reagents

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