DEVELOPMENT OF A PRE-CONCENTRATOR MICROSYSTEM FOR DETERMINATION OF PHOSPHORUS IN SPRING WATERS

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Abstract: Remote monitoring of waters from springs due to eutrophication was the objective of this work. For this purpose, a home-made production method was developed that made it possible to establish the appropriate conditions for the production of a pre-concentrator micro-device of phosphorus made in common glass. The process developed consists of the steps: layout definition, deposition time and temperature of the thermal press for the deposition of the mask, 3 minutes and 140ºC, followed by corrosion of the substrate, optimized in 90 min and, finally, the sealing. The microchip sealing step was performed by sol-gel process with the formation of zirconium oxide in the sealing step, Simultaneously by sealing and forming a porous layer for phosphate concentration, the best parameters being the proportion of Zr:OH 4:1 and Zr:CH_3COO^- de 0,48, which allowed the proper sealing of the glass microdevice.

Key-Words: *Microdevice, pre-concentrator, zirconium oxide, sol-gel, microfabrication.*

Introduction: The occupation of water source protection areas and the inadequate use of fertilizers continue to be the main causes of eutrophication in water reservoirs for public supply. In this sense, the use of a microdevice capable of analyzing the amount of phosphate in the water by means of a remote system, in real time, would bring benefits to the monitoring of the sources. This work constitutes a step for the development of an embedded system capable of performing water analysis, such as pH, rainfall index, nitrates and phosphates, among other aspects, capable of bringing a better understanding of these water bodies. In this way a microchip made in glass is being developed which will pre-concentrate phosphate allowing it to be determined by a photometric sensor in an embedded system.

Experimental: The microchip was made using a home made system, with the aid of a laser printer to define a resiste (or mask) on polyester films and subsequent deposition on glass slides, with the aid of a thermal press. Corrosion of the substrate to obtain the microchannels and preconcentration chamber (microsystem) were carried out using ammonium fluoride buffered hydrofluoric acid (NH_4F) solution in the molar ratio of 1: 1. The glass slides for the sealing were prepared and carefully cleaned with a solution-free detergent (salt-free detergent) and nitric acid solution (HNO_3), eliminating traces of interfering agents (contaminants) for zirconium oxide sealing. The glass slides are then immersed in a 4: 1 (V / V) solution of H_2SO_4: H_2O_2 for 10 min and then washed with distilled water. The second step of this process was to submerge the slides for 10 min in a 1: 1: 5 (V / V / V) solution of HCl: H_2O_2: H_2O, followed by washing with ultrapurified water. After these treatments the glass slides undergo a natural drying process and free from contact with dust. The sealing of the microsystem is conducted in a sol-gel system of zirconium oxide (ZrO_2) which will be the stationary phase for the retention of the species of interest, Besides naturally acting as a sealant agent due to the formation of covalent bond (cross-linking) with the substrate and its cover, also glass (cover slip). The sol-gel system is formed in a synthetic route consisting of a zirconium oxychloride solution octahydrate ($ZrOCl_2.8H_2O$) 2M, which was neutralized in alkali solution, under a temperature of 60ºC, leading to the formation of zirconyl hydroxychloride. Then a 5 M ammonium acetate solution is applied, leading to the gradual neutralization of the system and forming the zirconium oxide sol-gel.

Results and discussion: CorelDRaw X8 masks were obtained on 26 x 76 mm (L / C) glass substrate, 17 x 40 mm (L / C) oval preconcentration chamber and 100 μm width. The oval shape of the microchip favors microfluidic aspects and consequently greater efficiency in the preconcentration, since corners cause accumulation of species of interest. The adjustment of the pressing conditions (nominal pressure, time - 3 min, and temperature - 140 ° C) proved to be a critical parameter for the good transfer performance of the standard (decal). Substrate corrosion was performed by immersing the glass plate in a solution of HF / NH_4F (1: 1 HF: NH_4F) for 90 min with resin reinforcement (nailpolish) on the mask. The buffer solution formed by HF / NH_4F ensures a longer life for the corrosion solution and must be considered allowing the prediction of the depth of corrosion, in this case 100 μm. Finally the microchip sealing was obtained and flow, crystalline phase and porosity tests are important characterization steps. An experimental design was carried out to determine the optimum zirconium oxide sol-gel synthesis conditions, with Zr: OH 4: 1 and Zr: CH_3COO- proportions of 0.48, and finally the characterization of the phase by diffraction (XRD) and scanning electron microscopy (SEM).

Conclusion: From a home-made process it was possible to prepare a microdevice capable of pre-concentrating phosphorus (as phosphate), with the aid of a laser printer, a thermal press and a solution of buffered hydrofluoric acid and allowed to obtain pre-concentrating microsystems (Glass microchips). The studies allowed to define the best parameters for the deposition of the mascara and optimization of the sol-gel synthesis to obtain the microchip using the sealing element as a structural material and pre-concentrator phase. Preconcentration factor and real matrix performance tests are the steps to be followed, as well as the comparison of performance against microfabricated devices by conventional processes.

References and acknowledgements:

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