3D PRINTED CENTRIFUGAL MICROFLUIDIC PLATFORM FOR SOLID-PHASE-EXTRACTION AND FLUORESCENT DETECTION OF SPILLED OIL IN WATER

Yong Zhang
Ph.D. Candidate

Faculty Advisor: Dr. Wanjun Wang

ABSTRACT

In order to realize a lower detection limit of the target molecules, solid phase extraction (SPE) is widely used to pre-concentrate the target molecules in the liquid sample. Fluorescent detections are often following the SPE process to determine the existence and concentration of these molecules. However, conventional SPE method usually have to be conducted in the lab because professional equipment are required and the operations are complicated and time consuming. Recently, our research group developed a novel centrifugal microfluidic platform based on 3D printing technology. Special designed mechanical pinch valves, which were simply controlled by the rotation frequency of the platform, were applied to operate the flow of the reagent and sample in the device. The prototype of the proposed system was fabricated with PLA with FDM method. In the experiment, different stationary sorbent, such as C18, activated charcoal, and 3D printable porous polymer, were used and compared. The experimental results showed that the fluorescence intensity of the sample was significantly increased. This SPE platform is easy to operate and can be potentially used as a portable on-situ spilled oil enrichment and detection device.

The BP offshore crude oil spill accident occurred in the Gulf of Mexico shocked the world. The massive residual oil in coastal water has negative long-term effects on the environment. Therefore, portable and efficient instruments are highly desired to monitor the residual crude oil in the coastal water system [1]. The Fluorescence method is one of the most popular approach to detect the oil in water. In practice, crude oil emits light in the range of 400 nm to 650 nm when stimulated by a UV light source [2]. To enhance the detecting sensitivity, enrichment of the target molecules is often the first step. Lab-on-CD is a CD like centrifugal microfluidic platform based on the idea that the liquid can be transported from an inner reservoir to an outer one via a microchannel connecting them with the centrifugal force generated by rotation. Lafleur et al [3] tried to mount the C18 column into a lab-on-a-CD system and utilize the centrifugal force to drive the sample through the sorbent. In recent years, additive manufacturing technology, also commonly called 3D printing technology, experienced phenomenal development and become readily available. Many 3D printed microfluidic devices were reported [4]. With 3D printing technology, complex microfluidic devices (including lab-on-CD cartridge) can be fabricated easily with lower cost and shorter time. Attempts of 3D printing Lab-on-CD system have been made by our group [5].

In this paper, a novel centrifugal microfluidic platform with mechanical pinch valves had been demonstrated for detecting trace amount of oil pollution in water. The pinch valves used to manipulate the reagent and sample flows in the system can be simply controlled by the spinning speed of the centrifugal platform. SPE method is adopted and different stationary sorbent were compared.

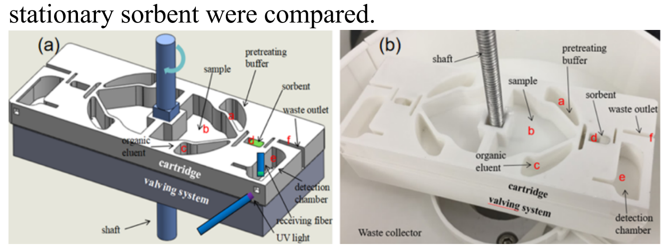

Fig 1. The (a) design and (b) fabricated centrifugal platform.

As shown in Figure 1, the system was based on solid phase extraction (SPE) principle. It consists of two major parts. The first part was the valving disc which is a permanent componen. The second part is the disposable fluidic cartridge containing all the fluidic channels and chambers. The specially-designed valving disc helps to synchronize the releasing sequence of the reagents and their flow direction. When oil enrichment is completed, the system stops spinning and the enriched sample is collected in detection chamber. UV light is introduced through a window designed on the side of the detection chamber and fluorescence was detected by a perpendicular fiber. Figure 2 shows the working principle of the fluidic cartridge. As shown in Figure 2(a), valves V2, V3, and V5 are normally-closed, while valves V1 and V4 are normally-open. As the operation starts, the pretreating buffer solution goes through valve V1 to condition the sorbent, then flow through the waste outlet as shown in Figure 2(b). Valve V2 is then opened to permit the sample in chamber b to go through the sorbent and flows out. In this process, oil molecules are extracted by the sorbent as shown in Figure 2(c). Valve V4 is then closed, and valve V3 and V5 are opened. Eluent in chamber c is released as shown in Figure 2(d) and flows through the sorbent and elutes the oil particles to chamber e.

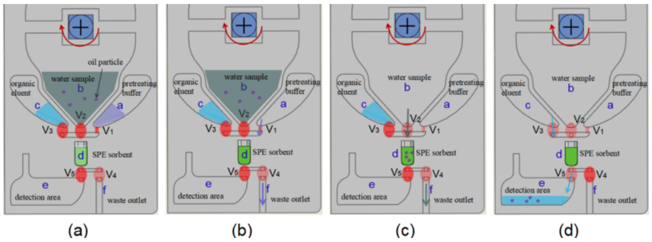

Fig 2. Working principle of the fluidic cartridge.

A comprehensive valving disc was designed and used in the centrifugal microfluidic platform. As shown in Figure 2, V_1 was a common capillary valve, and the rest are mechanical pinch-valves. The upper microfluidic cartridge and a slide-based valving disc at bottom were fixed together on a rotating shaft, which was driven by a DC motor. Figure 3 shows the design of the slide-based valving disc. The operational principle of the mechanical pinch valves is shown as Figure 3. By carefully design the grooves on the valving chip, multiple valves can be "mechanically-programmed". A detailed discussion of the design and test of the mechanical valving system has been presented in previous paper published by our group [6].

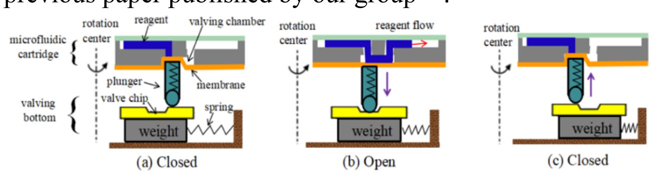

Fig 3. The operational principle of the mechanical pinch valves.

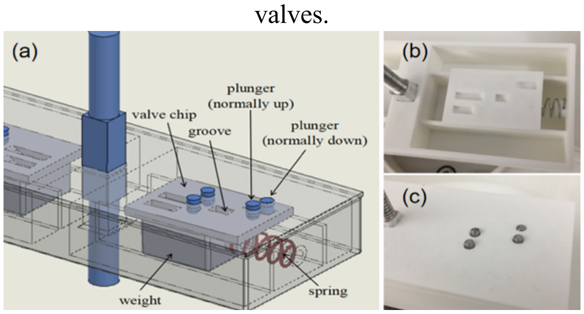

Fig 4. The (a) design and (b) (c) fabricated valving system.

Different sorbents were tested and compared. Table 1 shows detailed information of the three sorbents used in the experiments.

Table 1. The sorbents used in our experiment.

Sorbent	State	Size	Volume/Weight	Printability	Cost	Vendor and model
C18	powder	40-60μm	60mg	No	high	Thermo Scientific, HyperSep
Activated charcoal	granular	250-850μm	25mg	No	low	Sigma-Aldrich, C3014
Porous filament	filament	1.75mm	4×4×6mm	Yes	low	PORO-LAY, LAY-FELT

The prototype of the proposed system was 3D printed with PLA material. The platform was tested with 10ppm standard oil-water sample. In the experiment, C18, activated charcoal, and 3D printed porous polymer were used as stationary sorbent respectively and compared as shown in Fig 5(a). In the SPE experiments, 0.6ml methanol was loaded in chamber *a* as the pre-treating buffer to condition the stationary sorbent. 6 mL of 10ppm oil-water mixture sample was prepared and loaded in chamber *b*. 0.6ml nonane was loaded in chamber *c* as the eluent to desorb. The 3D printed porous polymer was proved to have the best SPE performances with about six times of enrichment efficiency. To further study its performance, more samples with lower oil concentration were tested. The experiments were conducted following the foregoing procedure. But the testing sample were replaced as 5ppm and 2ppm, respectively. Fig 5(b) and (c) shows the experimental results. The experiment results demonstrated functionality of the prototype lab-on-CD system.

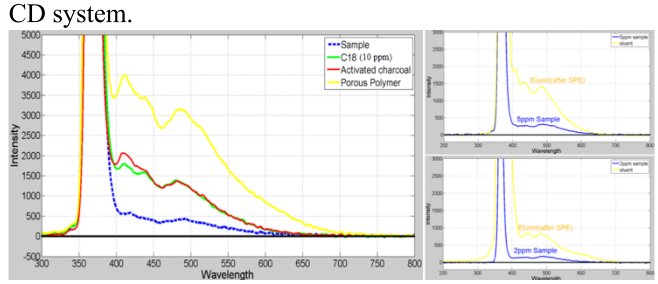

Fig 5. Experiment results of SPE with the centrifugal platform.

ACKNOWLEDGMENTS

This work was supported by the "Development of Portable Lab-on-CD Instrument for In-Situ and High Precision Detection of Spilled Oil Particles in Coastal Water System" project funded by the Gulf of Mexico Research Initiative.

REFERENCES

[1] Groton, C. T., and V. A. Herndon. "UPDATING THE SMART DISPERSANT MONITORING PROTOCOL: Review of Commercial-Off-The-Shelf Instruments." (2008).
[2] Steffens, Juliana, et al. "Application of fluorescence to the study of crude petroleum." Journal of fluorescence 21.3 (2011): 859-864.
[3] Lafleur, Josiane P., et al. "Miniaturised centrifugal solid phase extraction platforms for in-field sampling, pre-concentration and spectrometric detection of organic pollutants in aqueous samples."Talanta 81.1(2010):722-726.
[4] Bhattacharjee, Nirveek, et al. "The upcoming 3D-printing revolution in microfluidics." Lab on a Chip 16.10 (2016): 1720-1742.
[5] Xiang, Jiwen. "Microfluidic Technology and Application in Urinal Analysis." (2018).
[6] Xiang, Jiwen, et al. "Mechanically programmed valving technology and the active flow switching application in centrifugal microfluidics." Sensors and Actuators B: Chemical259 (2018): 325-331.