CHAPTER I
INTRODUTION

1.1 Statement of purpose

Aldehydes, an organic compound containing carboxaldehyde group (-CHO), are known to be mutagenic and carcinogenic substances. Low-molecular mass aldehydes (LMMAs) are high water-soluble and classified to be harmful organic pollutants [1, 2]. LMMAs can found in air, rainwater, dew, and fog. In addition, they are one of the main ozonation disinfection byproducts (DBPs) in drinking water [3-5]. DBPs are substances from the water disinfection process which occurred from the reaction between organic and inorganic matter with chemical treatment agents. Although no legislation has been established, it is necessary to measure aldehydes in drinking water due to their health effects.

Aldehydes are usually determined by chromatography [6, 7] and most methods are based on derivatization. Many derivatizating reagents were used such as 2,4-dinitrophenylhydrazine (DNPH), dansyloxyamine and o-(2,3,4,5,6-pentafluorobenzyl) hydroxylamine (PFBHA) [8-12]. In recent years, the DNPH-HPLC-UV method in which aldehydes were derivatized with DNPH and analyzed by high-performance liquid chromatography (HPLC) with ultraviolet detection (UV) has been established as the most widely used standard procedure for the analysis of aldehydes in aqueous samples [13, 14]. The DNPH-derivatives formed are generally extracted and preconcentrated by solid-phase extraction (SPE) before analyzed by HPLC-UV [15-17]. However, many approaches have been reported including (i) derivatization before SPE extraction, (ii) derivatization after SPE extraction and (iii) derivatization while extraction (extraction with SPE sorbent-coated with derivatizing agent). Nowadays, the approach of derivatization while extraction is widely used method because it can derivatize, preconcentrate and extract aldehydes in one step. Mastsumoto et al. [18] reported the determination of aldehydes in rain, fog, dew water and the gas phase using DNPH coated on silica cartridge as sorbent for analysis with HPLC-UV. Cancho et al. [19] determined aldehydes in drinking water by solid-phase microextraction (SPME) using pentafluorobenzylhydroxylamine coated on polydimethylsiloxane/divinylbenzene. Moreover, extractions of aldehydes by functionalized silica particles in cartridge format were studied. Basheer et al. [20] compared the extraction efficiency of functionalized silica with various hydrocarbon chain such as ethyl (Si-C_2,), octyl (Si-C_8) and octadecyl (Si-C_{18}) for determination of

aldehydes in rainwater. Si-C_2 showed the best efficiency for extraction of aldehydes because of their high polarity. Later, Zhang et al. [21] developed poly (methacrylic acid-ethylene glycol dimethacrylate) monolith to be a sorbent and used for the determination of DNPH derivatives of aldehydes in human saliva. Polymer monolith was beneficial for analyzing small amounts of sample. Furthermore, polymer monolith can synthesize for required pore size and arrangement of pore. As a result, porosity had a good distribution and therefore, extraction efficiency was better than particles.

There are two main types of sorbent used in SPE which are cartridges and disks [22]. SPE disks had smaller particle size and lower backpressure allowing faster flow rate through the sorbent. This type was suitable for large volume sample. In order to achieve higher enrichment efficiency for extraction of analytes, the choice of suitable adsorbents for SPE was very important. Moreover, SPE disk that fabricated from electrospun fibers can improve a sorptive capacity and surface area. In fact, electrospun fibers were usually obtained as a form of membrane. Therefore, the use of electrospun fibrous membrane as sorbents for SPE may be a good choice.

In present, many researches have reported the electrospun polymer nanofiber-based SPE devices [23, 24] such as nylon6, polystyrene (PS) and polyacrylonitrile (PAN) [25, 26]. The advantage of nanostructured materials was the large specific surface area allowing the reduction of sorbent bed mass while high extraction efficiency was achieved. Chigome and Torto [27] reviewed that electrospun polymer nanofibers were applicable as SPE sorbents. For example, polystyrene nanofibers were packed into micro-pipette tips as micro-column SPE and nylon6 nanofibers were fabricated into sheet or disk form. Xu et al. [28] reported the use of electrospun nanofibrous nylon6 membrane as sorbent for solid-phase extraction. The membrane was arranged as a disk and determined three estrogens in environmental water samples. Moreover, nanofibrous nylon6 membrane was applied for the determination of phthalate esters in water samples [29] and the determination of bisphenol A in plastic bottled drinking water [30]. A comparison of nanofibrous nylon6 membrane with commercial microporous nylon membrane and C18 cartridges were carried out. The results indicated that nanofibrous nylon6 membrane were better enrichment, higher extraction efficient and lower limits of detection than others.

In this work, electrospun fibrous polymer membrane was fabricated and used as sorbents in SPE. The membrane was coated with DNPH and then used for derivatization, preconcentration and extraction of aldehydes in one step. Important

parameters influencing the extraction efficiency were optimized. The applicability of the proposed SPE disk for determination of aldehydes in drinking water was also examined.

1.2 Objective of this research

The objective of this work is to prepare DNPH-coated polymer fibers in membrane format and used as a sorbent in solid-phase extraction for determination of aldehydes in water.

1.3 Scopes of this research

1.3.1 Fibrous polymer membrane such as nylon6, polystyrene and polyacrylonitrile were prepared by electrospinning technique.

1.3.2 Electrospun fibrous polymer membranes were coated with DNPH and used as a sorbent for solid-phase extraction.

1.3.3 DNPH-coated electrospun fibrous polymer membranes were studied for extraction of aldehyde in water.

1.3.4 DNPH-coated electrospun fibrous polymer membranes were applied for the determination of aldehydes in drinking water.

1.4 Benefits of the research

The research aimed to get an electrospun fibrous polymer membrane for extraction of aldehydes in water.