



Preparation of novel alumina nanowire solid-phase microextraction fiber coating for ultra-selective determination of volatile esters and alcohols from complicated food samples



Zhuomin Zhang^{a,*}, Yunjian Ma^a, Qingtang Wang^b, An Chen^a, Zhuoyan Pan^a, Gongke Li^{a,*}

^a School of Chemistry and Chemical Engineering, Sun Yat-sen University, Guangzhou 510275, China

^b Key Laboratory of Analysis and Detection for Food Safety of Ministry of Education, College of Chemistry and Chemical Engineering, Fuzhou University, Fuzhou, Fujian 350002, China

ARTICLE INFO

Article history:

Received 26 January 2013

Received in revised form 18 March 2013

Accepted 19 March 2013

Available online 25 March 2013

Keywords:

ANW SPME fiber coating

Ultra-selectively sampling

Ester

Alcohol

Food

ABSTRACT

A novel alumina nanowire (ANW) solid-phase microextraction (SPME) fiber coating was prepared by a simple and rapid anodization-chemical etching method for ultra-selective determination of volatile esters and alcohols from complicated food samples. Preparation conditions for ANW SPME fiber coating including corrosion solution concentration and corrosion time were optimized in detail for better surface morphology and higher surface area based on scanning electron microscope (SEM). Under the optimum conditions, homogeneous alumina nanowire structure of ANW SPME fiber coating was achieved with the average thickness of 20 μm around. Compared with most of commercial SPME fiber coatings, ANW SPME fiber coatings achieved the higher extraction capacity and special selectivity for volatile esters and alcohols. Finally, an efficient gas sampling technique based on ANW SPME fiber coating as the core was established and successfully applied for the ultra-selective determination of trace volatile esters and alcohols from complicated banana and fermented glutinous rice samples coupled with gas chromatography/mass spectrometry (GC/MS) detection. It was interesting that 25 esters and 2 alcohols among 30 banana volatile organic compounds (VOCs) identified and 4 esters and 7 alcohols among 13 identified VOCs of fermented glutinous rice were selectively sampled by ANW SPME fiber coatings. Furthermore, new analytical methods for the determination of some typical volatile esters and alcohols from banana and fermented glutinous rice samples at specific storage or brewing phases were developed and validated. Good recoveries for banana and fermented glutinous rice samples were achieved in range of 108–115% with relative standard deviations (RSDs) of 2.6–6.7% and 80.0–91.8% with RSDs of 0.3–1.3% ($n = 3$), respectively. This work proposed a novel and efficient gas sampling technique of ANW SPME which was quite suitable for ultra-selectively sampling trace volatile esters and alcohols from complicated food samples.

© 2013 Elsevier B.V. All rights reserved.

1. Introduction

Due to the extremely high mobility and volatility of gas, it is hard to guarantee the efficiency and reproducibility of gas sampling projects. Gas sampling techniques still remain one of the weak spots in analytical chemistry nowadays, compared with sampling techniques for liquid and solid samples. Volatile organic compounds (VOCs) from all kinds of complicated samples such as biological and food samples are a special kind of gas samples, containing crucial information related with biological metabolism, food safety, quality control, etc. [1]. It is a tough task to sample trace VOCs from complicated samples entirely without efficient sampling techniques.

Efficient gas sampling techniques, especially for trace VOCs, should possess a wide sampling range, high extraction capacity and selectivity, and can be conveniently coupled with sequent analytical instruments [2]. The key to develop novel efficient and nondestructive sampling techniques for trace VOCs lies in the development of new enrichment media with higher extraction capacity and selectivity and establishment of new sampling techniques based on new enrichment media.

Nowadays, many novel gas adsorption materials such as nanometer-scale gas sensing material [3], nanoarray gas adsorption material [4], composite gas adsorption material [5] and metal-organic frameworks (MOFs) material [6] have been proposed as potentially effective enrichment media for trace VOCs. Among them nanometer-scale gas sensing material has received steadily growing attention for the trace VOCs detection due to its peculiar and fascinating gas adsorptive property due to their small particle size and large surface area. Most of nanometer-scale gas

* Corresponding authors. Tel.: +86 20 84110922; fax: +86 20 84115107.

E-mail addresses: cesgkl@mail.sysu.edu.cn (Z. Zhang), zzm@mail.sysu.edu.cn (G. Li).

sensing materials have ordered structures, high thermal stability, adjustable chemical functionality, ultra-high porosity and the availability of crystalline and well-characterized porous structures [7,8]. Furthermore, nanoarray gas absorption material possesses the larger surface area and extraction capacity for VOCs, since regular nanoarray structural material with the proper interspace could usually offer larger surface area than randomly oriented material and was suitable for the adsorption of proper-sized gas molecules [9–12]. Especially, one dimensional (1D) nanoarray material such as nanorod [4], nanotube [13], nanowire [14], etc., can further improve the surface area and gas sensing property, since regular nanoarray structure can improve material thickness and density. However, until today they are seldom used as extraction media for the real gas sampling projects, since it is difficult to fabricate these excellent 1D nanoarray materials as appropriate and stable gas sample techniques. Only several kinds of 1D nanoarray materials such as ZnO nanorod array [4], ZnO nanorod array polydimethylsiloxane composite [15] and nanoporous array anodic alumina (NAAA) [16] have been fabricated as solid-phase microextraction (SPME) fiber coatings for the real VOC extraction and analysis. They possessed the large surface area and demonstrated the excellent gas absorption capacity and selectivity. Especially, NAAA as an aluminum-substrate nanoarray SPME fiber coating showed the special selectivity to the biological VOCs. Apart from NAAA, aluminum-substrate nanoarray material usually involves alumina nanowires (ANW) which possesses higher material density and extraction capacity to biological VOCs owing to its smaller nanometer-scale [17]. Several synthetic methods have been applied for the preparation of ANW including catalyst assisted vapor-liquid-solid deposition [18,19], catalyst-free vapor-solid deposition [20,21], transition metal catalysis [22,23], template method [24,25], chemical etching method [26,27] and mechanical cleavage of porous aluminum oxide membranes [28] and so on. Among these methods, chemical etching method has aroused the attention of many chemists for ANW preparation based on chemical etching ordered porous anodic alumina template in NaOH aqueous solution recently, since the operation procedures of chemical etching method are relatively simple with low preparation cost.

Conventional sampling techniques for VOCs mainly involve liquid-liquid extraction (LLE), steam distillation (SD), simultaneous distillation extraction (SDE), supercritical fluid extraction (SFE) and purge-and-trap (P&T) [1,2]. Microextraction techniques such as SPME [29] and stir bar sorptive extraction (SBSE) [30] are simple, miniaturized, rapid and environment-friendly, which represent a major part of modern sampling technique for trace VOCs and have been successfully used for the real sampling projects of trace VOCs. Especially headspace SPME (HS-SPME) is solvent-free, efficient and suitable for *in vivo* sampling trace VOCs and has been proved as an excellent VOC sampling technique [31]. The limited sampling capacity and selectivity of commercial SPME fiber coatings usually limited its application for the analysis of trace VOCs from complicated matrixes. Many novel SPME fiber coatings have been developed for the enhancement of extraction capacity and selectivity. However, there is still few fiber coatings developed for the selective VOC sampling projects. Chemical etching NAAA produced by anodic oxidation would result in a novel ANW SPME fiber coating in the aluminum substrate with homogeneous alumina nanowire structure and higher surface area. It can be convenient to be coupled with sequent detection techniques such as gas chromatography/mass spectrometry (GC/MS) for selectively sampling and analyzing trace VOCs from complicated sample matrix.

In this work, a novel ANW SPME fiber coating was directly fabricated and prepared on aluminum substrate by a simple anodic oxidation-chemical etching method. Preparation conditions of ANW SPME including corrosion solution concentration and corrosion time were optimized in order to achieve the better

extraction efficiency. The extraction capability and selectivity for typical volatile esters and alcohols by ANW SPME fiber coating has been investigated in detail. Finally, ANW SPME fiber coating was applied for selectively sampling and analyzing volatile esters and alcohols from banana and fermented glutinous rice samples coupled with GC/MS detection.

2. Experimental

2.1. Chemical reagents and materials

In this work, isoamyl acetate (99%) was purchased from Alfa Aesar (A Johnson Matthey company, USA). 1-Octanol (99%) was obtained from Aldrich (St. Louis, MO, USA). Isoamyl butyrate (99%), 1-butyl butyrate (99%), methyl butyrate (99.5%), ethyl propionate (99%), propyl butyrate (99%), ethyl hexanoate (99%), 1-amyl butyrate (99%), 2-propanol (99%), 1-propanol (99%), 2-methyl-1-propanol (99%), 2-furanmethanol (98%), 1-butanol (99%), 1-hexanol (99%), 1-heptanol (98%), 1-nonanol (98%) and 1-decanol (98%) were purchased from Aladdin (Shanghai, China). 1-Hexyl butyrate (98%) and 3-methylbutyl 3-methylbutanoate (98%) were purchased from TCI (Tokyo, Japan). Oxalic acid, acetone, ethanol, sodium hydroxide, phosphoric acid, chromium trioxide, 1-hexane and perchloric acid were all analytical reagent-grade and obtained from Tianjin Chemical Reagent Plant (Tianjin, China). Ultra-pure helium (99.999%) was purchased from Guangzhou Xicheng Gas Factory (Guangzhou, China).

Stock solutions of the mixed standard for the study of extraction capacity of ANW SPME fiber coating compared with commercial SPME fibers were prepared by dissolving 2.6 mg of isoamyl acetate, 2.6 mg of 1-butyl butyrate, 2.6 mg of isoamyl butyrate, 2.5 mg of 3-methylbutyl 3-methylbutanoate, 2.5 mg of 1-hexyl butyrate, 2.4 mg of ethanol, 2.3 mg of 2-propanol, 2.4 mg of 1-propanol, 2.4 mg of 2-methyl-1-propanol and 3.4 mg of 2-furanmethanol in 10 mL of 1-hexane. The evaluation for the extraction selectivity of ANW SPME fiber coating was conducted based on two mixed standard solutions of typical VOCs. One mixed standard solution was prepared by dissolving 10 esters in 10 mL of 1-hexane including 2.6 mg of ethyl propionate, 2.7 mg of methyl butyrate, 2.6 mg of isoamyl acetate, 2.6 mg of propyl butyrate, 2.6 mg of 1-butyl butyrate, 2.6 mg of ethyl hexanoate, 2.6 mg of isoamyl butyrate, 2.5 mg of 1-amyl butyrate, 2.4 mg of 3-methylbutyl 3-methylbutanoate and 2.5 mg of 1-hexyl butyrate. Another mixed standard solution was prepared by dissolving 6 alcohols in 10 mL of 1-hexane including 2.4 mg of 1-butanol, 2.4 mg of 1-hexanol, 2.4 mg of 1-heptanol, 2.5 mg of 1-octanol, 2.5 mg of 1-nonanol and 2.5 mg of 1-decanol. Working solutions were diluted from the related stock solutions above in the study.

Aluminum wire (99.999% purity, 0.25 mm diameter, annealed) used in this work was purchased from Alfa Aesar (A Johnson Matthey Company, USA). Commercial 100 μ m polydimethylsiloxane (PDMS), 65 μ m polydimethylsiloxane/divinylbenzene (PDMS/DVB), 50/30 μ m carboxen/divinylbenzene/polydimethylsiloxane (CAR/DVB/PDMS) and 75 μ m carboxen/polydimethylsiloxane (CAR/PDMS) SPME fiber coatings (Supelco, USA) were used for the comparison study.

2.2. Instruments

A Zhaoxin Linear Adjustable DC Power Supply, Model RX-602D (Shenzhen Zhao Xin Yuan Electronics Co., Ltd., Shenzhen, China), was used to provide the constant voltage for anodic oxidation during the preparation of ANW SPME fiber coatings. YX2000A entire intelligent ultrasonic cleaner (Guangzhou Xinyue electronic technique Ltd. Inc., China) and HS-4 magnetic stirrers (IKA, Germany)

Download English Version:

<https://daneshyari.com/en/article/1204023>

Download Persian Version:

<https://daneshyari.com/article/1204023>

[Daneshyari.com](https://daneshyari.com)