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ABSTRACTS & PROCEEDINGS

TÜBİTAK

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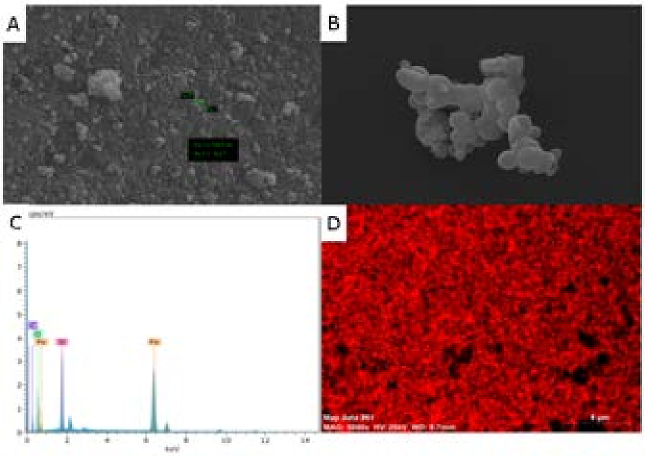
Characterization of Magnetic Particles



A) Fe3O4 nanoparticles B) Fe3O4 nanoparticles after modification C) SEM-EDX results D) Mapping analysis of Fe3O4 nanoparticles after modification

[Abstract:0520] OPY-058

Determination of volatile compounds in tea using vacuum-assisted headspace solid-phase microextraction

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Abstract

The widespread use of tea as a drink due to its pleasant smell, taste, as well as a mild stimulating effect and nutritional properties. The purpose of this work was to identify the characteristic composition of certain types of tea. The study of the chemical composition of tea helps to assess its quality and helps to control and manage the processing and storage conditions. Qualitative and quantitative analysis of volatile compounds, which gives the taste and aroma of tea, was carried out using gas chromatography gas chromatography-mass spectrometry (GC-MS). Low volatile organic compounds were analyzed using vacuum-assisted HS-SPME. This method was used to extract samples prior to determination by gas chromatography - detection using mass spectrometry. Chromatographic parameters were also optimized to determine the composition of the tea.

Keywords: Tea, vacuum-assisted headspace solid-phase microextraction.

I. Introduction

Currently, tea is one of the most consumed beverages in the world. Depending on the fermentation of tea leaves, there are three main types of tea: green tea, oolong tea and black tea. To this day, the health benefits of tea have been widely studied [1,2]. Many teas contain polyphenols, L-theanines, catechins, hydroxyaromatic acids, flavonols, theogallins, pigments, alkaloids, sugars, amino acids, vitamins, dicarboxylic acids, cations [3,4]. Almost all brands of tea in Kazkhastan are imported. Most people do not care about harmful substances contained in low-quality beverages, and consume them in large quantities. The most useful green tea becomes harmful and unhealthy if it is made from low-quality raw materials and poorly processed.

On the bushes, tea leaves contain thousands of chemical compounds. During the processing of tea leaves, chemical compounds inside them are destroyed, then complexes are formed with each other and form new compounds. Tea aromatic complex consists of hundreds of volatile aromatic and aromatic compounds that exist in trace amounts. Many of these aromatic compounds do not exist in fresh tea leaves; instead, they are derived from other substances during processing. The aromatic complex is sometimes divided into two parts: the primary aroma (from fresh tea leaves) and the secondary aroma (processed products) [5]. The study of the composition of various varieties of tea is of great interest.

II. Material and method

There are standard methods for determining for analysis of tea, which there are classical methods such as extraction. But such methods today are not very optimal, firstly, it requires a lot of funding, and this approach to analysis takes much time. And they do not allow for a complete analysis of the composition. Some samples of preparations include organic solvents (100-250 ml), reagents (concentrated). Therefore, to determine the composition of tea, we considered an environmentally safe and effective method. Considering the results of other articles, we will look at the benefits of SPME in identifying volatile and semi-volatile compounds in tea. This method has several advantages over classical methods. Solid phase micro extraction is one of the most common pretreatment methods for influencing volatile and semi-volatile compounds. This method does not require the use of reagents and combines the extraction and purification steps. As results the analysis time are reduced.

This method is one of the green methods for analysis, and the most important is to reduce the time spent on sample preparation. To further improve this method and optimize, scientists proposed their ideas. In 2001, Brinton proposed a way to speed up the extraction kinetics using vacuum conditions during HS SPME sampling. In 2005, the effect of the alternative method was confirmed by Dauzes and other scientist. However, this method has not yet been used for the analysis of tea.

In this work, the volatile and semi-volatile components were extracted separately by solid-phase microextraction in head space (HS-SPME) and vacuum-assisted HS-SPME (Vac-SPME), and then analyzed by gas chromatography mass spectrometry. Desorbed volatile compounds were analyzed using 7890A/5975C

GC-MS (Agilent, USA). A DB-35MS capillary column (30 m × 0.25 mm, 0.25 μm) was employed with high purity helium as the carrier gas at a flow rate of 1 mL/min.

To control the gas chromatography system for recording and processing the results and data software Agilent MSD ChemStation (version 1701EA) was used. Processing data included determination of retention times, peak areas, and processing the spectral data obtained by mass spectrometric detector. To decrypt the received mass spectra Wiley 7th edition and NIST'02 library (total number of spectra in the library - over 550 th.) were used.

III. Results and discussion

Samples weighing 1 g were placed in 20 ml vials. The vial was closed and the air was evacuated for 2 minutes with a syringe. Then after removing the syringe, 2 ml of tea is injected into the vial, and then kept in the mixer for 20 minutes. After that, SPME fiber was introduced into the vial and the analytes were extracted for 30 minutes. After extraction, the SPME fiber was immediately removed from the free space wick and inserted into the GC injector for desorption at 260°C for 5 minutes.

The vacuum assisted headspace solid phase microextraction was optimized for studying the volatile matter of tea. During optimization, were selected the optimal fibers for extraction. For analysis, were used 5 types of fibers: CAR/PDMS, PA, PDMS, PDMS/DVB and DVB/CAR/PDMS. The use of DVB/CAR/PDMS made it possible to detect more number of compounds in chromatograms. The selection of the extraction time and the incubation time was also carried out. During Vac-SPME, the extraction temperature is one of the most important parameters (Fig.1). Thus, the extraction time was optimized for 5, 10, 15, 20, 30, 45 and 60min at 80°C. The optimal extraction time was 20 minutes, during which most of the compounds extracted. Tea samples were pre-incubated for 5, 10, 15, 20, 30 and 40 min at 80°C using DVB / CAR / PDMS fiber. The optimal pre-incubation time was 10 minutes - during this time equilibrium was established between the phases of gas and liquid.

Also during the analysis, the moisture content of the tea matrix (tea leaf) was studied. Moisture in dry tea was about 5%. The determination of the moisture content continued in 5, 10, 30, 50, and 70,100 %. The results showed that more components are released at a humidity of 70% (Fig.2).

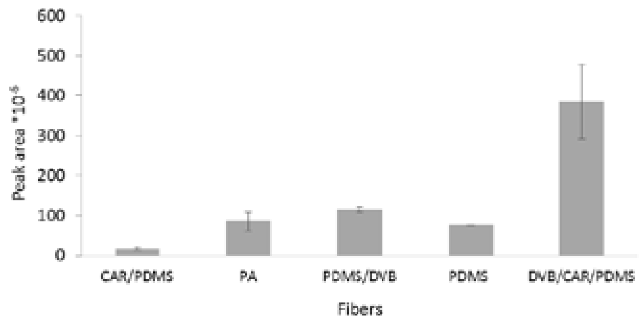

Fig. 1: Fiber selection

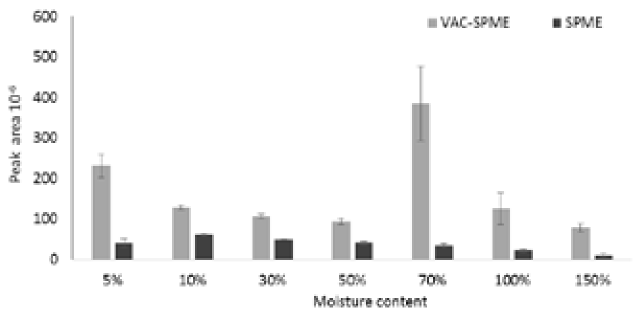

Fig. 2: Optimization of moisture content

Conclusion

A simple, fast, and reliable solid phase microextraction procedure was developed enhanced by the use of a vacuum in an extraction vial. It was used to extract tea samples prior to determination by gas chromatography - detection using mass spectrometry.

As a result, the work done was first used the method of vacuum- assisted SPME for the analysis of tea. The parameters of the vacuum-assisted SPME GC-MS were optimized. This method improved the conventional SPME method. Using this method, commercial teas were analyzed and their component composition was determined. Finally, for the evaluation of the vacuum-assisted SPME method, all experimental parameters, that affect the extraction efficiency of the analytes have been optimized, including the time and temperature of extraction, the preincubation time and incubation temperature, the vacuum time and the moisture content of the tea matrix (tea leaf). The optimal VAC-SPME parameters for tea samples are DVB/CAR/PDMS fiber coating, an extraction temperature of 80°C and a pre-incubation time of 10 minutes. The optimal extraction time for tea samples is 20 minutes.

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