Correlation studies between electronic nose response and headspace volatiles of *Tongkat Ali* extracts

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Introduction

Herbal medicines are special due to their neutraceutical and medicinal values, making quality control of raw herbs and their products essential to ensure consistency in quality, safety and efficacy. Traditionally odour analysis and food regulation involve gas chromatograph-mass spectrometry (GC–MS) and human taste panels which can give detailed information about the contents of the odour. GC–MS can separate, identify and quantify individual volatile chemicals, but difficult to correlate the data with sensory evaluation. Since odours are complex mixtures of many volatiles, the technique is highly impractical. Human sensory evaluation is useful but expensive, time consuming, subjective, as well as biased from illness and other factors.

As an alternative to both, electronic nose is gaining popularity in odour analysis which is essentially an instrument to mimic the human sense of smell and consists of chemical imaging and multiparameter sensing systems. The sensing system can be a single sensor device or an array of sensors providing "fingerprints" that are characteristics of a particular odour. The advantages of which are rapid, real-time detection of volatiles, lower cost and easy automation.

Medicinal plant formulations may consist of hundreds of phytochemical and is very difficult to identify most of these components by usual methods. Generally, only a few pharmacologically active components are employed for evaluating their quality and authenticity. The volatile components contained in extracts are normally lower than the detection limits of the detector. Thus some form of pre-concentration becomes mandatory. Among these, simultaneous distillation-extraction (SDE), dynamic headspace analysis or purge and trap methods using porous polymers and solid-phase micro-extractions (SPME) are the most popular methods in aroma analysis. Eurycoma longifolia Jack, commonly known as Tongkat Ali, belong to the Simaroubaceae family and grows wild in Southeast Asia. The plant contains a series of quassinoids, e.g., eurycomanone, 14,15_-dihydroxyklaineanone, eurycomanol and eurycomalactone; alkanoids, e.g., 9-methoxycanthin-6-one and together with others such as 13,21-dihydroeurycomanone, 13_,21-epoxyeurycomanone. We have developed a smell sensor using gas chromatographic stationary phases and lipid materials to mimic the olfactory system. In the present study, Tongkat Ali extracts are analysed with GC–MS using solid-phase micro-extraction sampling technique and correlated 12 identified compounds with a QCM array sensor.

Experiment

Sample

Twelve Tongkat Ali Jack samples were extracted using water and methanol and either freeze or spray dried. Some samples were commercial samples obtained from various manufacturers.

Electronic nose - Instrumentation

The frequency changes of the sensors were monitored by a Universal Sensor Array System (QTS-3) connected to a personal computer for data acquisition and processing.

Electronic nose - QCM array sensors

The array sensor consisted of eight AT-cut quartz crystals with gold electrodes on both sides. Eight different types of gas chromatographic stationary phases and lipid materials were used to fabricate the array sensor. The sensing materials consisted of polar, non-polar and amphiphilic materials. The sensing materials were coated on both sides of the quartz crystal either using simple ultrasonic spray coating device utilizing an ultrasonic atomizer. The coated crystals produced a relative standard deviation of less than 2 in terms of frequency changes. 100mg samples were taken in 20 ml vials and heated to 60°C for 15 min. The headspace vapours were funneled into the sensor chamber through a dried silica gel column, as shown in Fig. 1. The sequence of the sample injection was: baseline 60s, sample injection time 10s and recovery time 200s.

The response of the sensor was considered as the difference between the baseline and their maximum frequency change when samples were injected. Initially, compressed air was passed over the sensor array via valve 1 (valve 2 inactive) until a stable baseline was achieved (~ 1 min). Next the headspace volatiles in the vials were flowed over the sensor array by activating valve 2 and circulated for 1 min before the signals were recorded. The sensors were cleaned by compressed air for 3 min. A silica column was used to remove traces of moisture. Each sample was measured six times and the average was analysed. The relative standard deviations of the responses were less than 2.

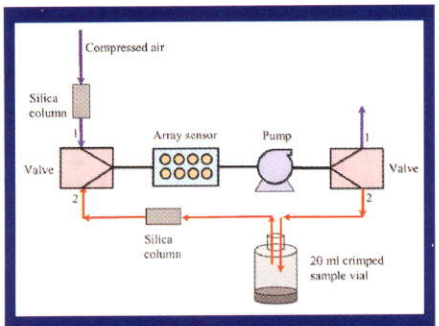

Fig.1 : Schematic diagram of the headspace sampling in quartz crystal array sensor.