



Handheld electrochemical device for the determination of the strength of garlic



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ARTICLE INFO

Article history:

Received 7 January 2016

Received in revised form 26 February 2016

Accepted 15 March 2016

Available online 26 March 2016

Keywords:

Garlic

Screen printed electrode

Food quality

Electrochemical instrumentation

Bromination

Organosulfur compounds

ABSTRACT

A handheld electrochemical sensor has been demonstrated for the quantification of the strength of garlic. The device is based on the enhanced voltammetric response in the presence of organosulfur compounds extracted from garlic. Convenient and disposable platinum screen-printed electrodes are employed. All measurements and data analyses are performed within the device. A linear response of the voltammetric peak current enhancement as a function of garlic concentration was observed, indicating the ability of the device to be applied to garlic samples of any strength. Importantly, this portable sensor can be used by non-scientifically skilled personnel and does not require expensive laboratory equipment. It is thus suitable for application in the food industry.

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1. Introduction

Garlic belongs to the genus *Allium* which is well-known for its antibiotic activity [1,2], pungency and strong odour. These properties stem from their organosulfur components such as disulfides (R–S–S–R') and sulfoxides (R–S(=O)–R'). [3] Upon cell damage, the sulfoxides are biologically converted to thiosulfonates (R–S(=O)–S–R') and it is this family of compounds that are responsible for the pungent flavour of garlic. [4] The painful and burning sensation of garlic is induced by thiosulfonates from the alliums activating the pain receptor and triggering inflammation. [3]

Thanks to its unique odour and properties, garlic in both raw and processed forms is widely used as a cooking ingredient. However, long term storage of the bulbs prior to processing is known to alter the sulfoxide composition [5,6]. Moreover, different cooking methods [7], sub-species, sources and harvest time of garlic results in a variability of the flavour and strength of garlic [8–10].

For consistency of the quality of the final consumer products, the strength of garlic and the amount required must be reviewed frequently on a batch-to-batch basis before they can be used in industrial-scale food production. At present, the 'strength' of garlic is assessed in the food industry via organoleptic testing. However,

in the preparation of ready-made meals, cooking sauces, garlic powder, medicinal supplements and many other garlic containing products, large quantities of garlic have to be tested. This demands a more reliable, quicker and accurate method than taste testing.

Scientifically, various techniques are available for the analysis of organosulfur concentration such as mass spectrometry [11], high performance liquid chromatography [12–14], gas chromatography [15,16], capillary electrophoresis [17] and electrochemical methods [18,19]. To date, electrochemical bromination [19] is the most appropriate method for industrial usage due to its relatively low cost, short experimental time and ease of use for non-scientifically trained operators. The electrochemical method uses the bromide/bromine redox system as the homogeneous redox mediator at a platinum macro-electrode. Organosulfur is extracted from garlic using the less toxic solvent, ethyl acetate. The method has been proved to be able to cover the whole range of garlic strength and the results showed an excellent correlation with organoleptic tests [19].

In this paper, we develop a small, portable device to carry out the electrochemical bromination at point-of-use. The device can perform cyclic voltammetry measurements using a custom-made potentiostat. Data analysis is done within the device hence no prior knowledge of electrochemistry or data processing is required. Disposable screen-printed electrodes are employed instead of conventional macro-electrodes to discard the step of electrode preparation. The novelty of this paper lies in the design of the

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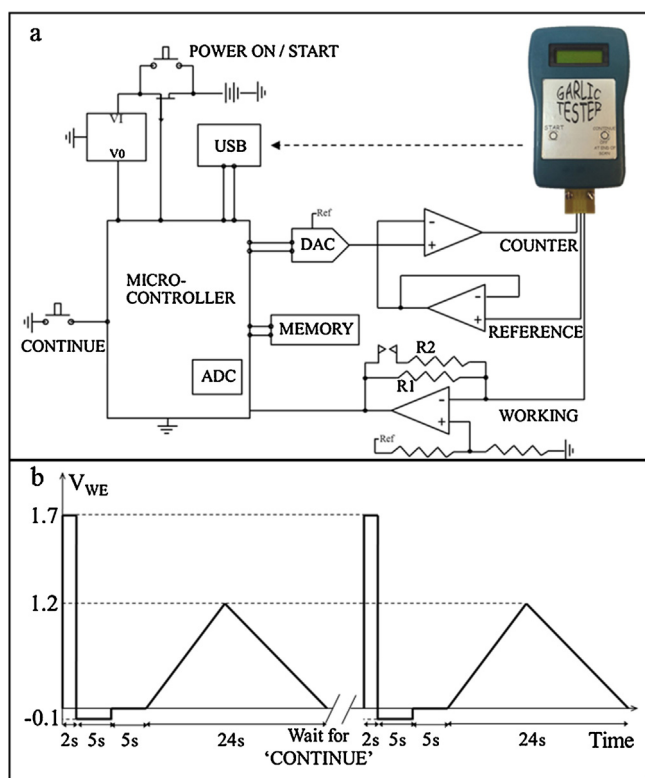


Fig. 1. a) Schematic diagram of the electronic circuit of the Garlic Meter with the picture of the meter showing at the top right corner; b) the voltage (V_{WE}) applied as a function of time.

device, which below we term the 'Garlic Meter.' The investigation of the differences between screen-printed electrodes and macro-electrodes, and the origins underlying them is also evidenced. We show how limitations and difficulties in the use of screen-printed platinum electrodes can be overcome and demonstrate the capability of this Garlic Meter to authentic chopped garlic samples of different concentrations.

2. Materials and methods

2.1. Materials

Sodium bromide (M&D Laboratory Chemicals, >99%), sodium perchlorate monohydrate (Sigma-Aldrich, 98%) and ethyl acetate (Sigma-Aldrich, $\geq 99.7\%$) were used as received. Garlic samples were provided by Beacon Foods Ltd. All solutions were prepared using deionised water (Millipore) with a resistivity of $18.2 \text{ M}\Omega \text{ cm}$ at 25°C . Measurements were conducted using disposable screen-printed electrodes (Dropsens ref. 550, 4 mm diameter [20]) consisting of platinum working electrode, platinum counter electrode and silver pseudo reference electrode.

2.2. Design of the garlic meter

The electronic circuit of the device is outlined schematically in Fig. 1a. The device includes i) a three-electrode potentiostat built from three OP196 operational amplifiers to conduct electrochemical measurement; ii) PIC microcontroller programmed in PICBASIC PRO Compiler to operate the device; iii) 12-bit digital-to-analog converter (DAC) to supply the voltage set by microcontroller to the amplifier; iv) 12-bit analog-to-digital converter (ADC) to deliver the signal obtained from the amplifier to the microcontroller; v) a switch labelled as 'START' to turn on the device and commence the

electrochemical measurement; vi) a switch labelled as 'CONTINUE' to start the second measurement (discussed further below); vii) a voltage regulator to maintain a constant voltage level; viii) two 12 V DC relays; ix) 10k ohm resistor labelled as 'R1' in the diagram allows the flow of currents up to the maximum of ca. $130 \mu\text{A}$; x) The lower resistor R2 is put in the circuit during the conditioning phase, this allows larger currents through the working electrode to allow enough charge to pass for surface oxidation of the platinum. In addition, the second relay reconfigures the inputs to allow more voltage between the counter and reference electrodes during the oxidizing activation phase. The normal three-electrode configuration is used for the reducing activation phase as the potentials are less. The device is powered by four 1.5 V AAA batteries. The values of voltage applied throughout the measurement are summarized in Fig. 1b as a function of time.

The Garlic Meter is designed to perform three major functions: electrode activation, two cyclic voltammetry measurements and data analysis; described next.

a) Electrode activation

Pre-treatment of the platinum screen-printed electrodes (SPEs) is required prior to performing electrochemical measurements, as will be evidenced later. This is done by holding the potential at 1.7 V for 2 s to electrochemically oxidize platinum and impurities on the electrode surface, followed by the application of -0.1 V for 5 s. At this potential, all platinum oxides formed during the electrode activation at 1.7 V would be reduced back to platinum as reported by Vetter and Schultze [21] that complete reduction of platinum oxide occurs at 0.6 V vs SHE ($\sim 0.85 \text{ V}$ vs Ag). The system is then left at 0.0 V for 5 s prior to the cyclic voltammetric measurement.

b) Cyclic voltammetry

The voltage at the working electrode is varied linearly as a function of time, relative to the reference electrode where the potential is scanned from 0 V to 1.2 V and back to 0 V at a scan rate of 100 mV s^{-1} . The Garlic Meter first runs a cyclic voltammogram for the control solution (no organosulfur is present), and then the same voltammogram is repeated in the presence of the garlic sample. Both scans are required for the quantification of garlic strength as described in the following section.

c) Data acquisition and analysis

Due to the partial onset of the oxidation of the metal surface, it is likely that the currents at the anodic end of the measurement may be higher than the diffusion-limited peak currents of the redox couple under study. The Garlic Meter is therefore designed to run the scan up to 1.2 V to obtain the appropriate range for cyclic voltammogram, but only allows the detection of the peak current up to 1.1 V; refer to Fig. 2a.

At the end of the measurements, the raw data of the current as a function of time (or voltage) can be obtained by connecting the Garlic Meter to a personal computer via USB connection. For industrial use and hence for the use by non-scientifically skilled personnel, analysis is performed internally by the garlic meter. Here, the screen is programmed to display the peak currents of the first and second scans as well as the baseline currents of both scans taken at 0.7 V as a first review point. The screen shows these values of peak currents for three seconds, then advances to the next page displaying the results of baseline corrected peak currents for both scans. The result of final data analysis which is the normalized current enhancement ($i_p - i_{p,0}/i_{p,0}$) calculated by a microcontroller is shown at the bottom line of this screen. This current enhancement quantifies the amount of organosulfur in the garlic sample.

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