Video Article

A High Performance Impedance-based Platform for Evaporation Rate Detection

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Abstract

This paper describes the method of a novel impedance-based platform for the detection of the evaporation rate. The model compound hyaluronic acid was employed here for demonstration purposes. Multiple evaporation tests on the model compound as a humectant with various concentrations in solutions were conducted for comparison purposes. A conventional weight loss approach is known as the most straightforward, but time-consuming, measurement technique for evaporation rate detection. Yet, a clear disadvantage is that a large volume of sample is required and multiple sample tests cannot be conducted at the same time. For the first time in literature, an electrical impedance sensing chip is successfully applied to a real-time evaporation investigation in a time sharing, continuous and automatic manner. Moreover, as little as 0.5 ml of test samples is required in this impedance-based apparatus, and a large impedance variation is demonstrated among various dilute solutions. The proposed high-sensitivity and fast-response impedance sensing system is found to outperform a conventional weight loss approach in terms of evaporation rate detection.

Video Link

The video component of this article can be found at http://www.jove.com/video/54575/

Introduction

Evaporation is a type of liquid vaporization and occurs along the gas-liquid interface of a collective body of water. The water molecules near the surface become capable of escaping from the liquid due to collision of water molecules. The evaporation rate is an important key factor during the process of evaporation. Generally, a balance or volumetric tube[1-3] is widely-used to detect the evaporation of solutions. However, it takes a long time to measure the evaporation rate due to the precision limitation of a balance or a volumetric tube. For this reason, a responsive and high-sensitivity instrument must be developed to probe into the details of the evaporation process.

Electrochemical impedance spectroscopy (EIS) is a fast-response, sensitive and effective experimental means in terms of *in-situ* impedance detection for electrochemical system characterization[4]. Therefore, EIS can be applied in various fields, such as recent studies on cellular behavior[5], bioanalytical sensing[6-7], electrolysis[8], conducting polymers[9], and electrochemical extraction[10]. Even though EIS systems had successfully been applied in a wide variety of disciplines, there exist an extremely small number of publications on its application to evaporation research.

Hyaluronic acid, a high molecular weight polysaccharide with strong water-binding potential, is a well-known humectant for cosmetic applications. One hyaluronic acid molecule can bind up to 500 water molecules[11] and reach 1,000 times its original volume[12]. An extremely small amount of hyaluronic acid can possess moisturizing function[13-14]. Due to the high moisture retention, hyaluronic acid has become an important component of cosmetic humectant products with high commercial value worldwide[15].

This study presents the method of a novel impedance-based apparatus featuring high speed detection, small volume sample requirement, and multiple sample measurements[16-19]. It is presented with a focus on the relative evaporation rate comparison among solutions as a way to validate the superiority of the innovative detection mechanism over a conventional weighing manner.

Protocol

1. Experimental Chip Module

1. **Fabricate the indium tin oxide (ITO) electrode chip by photolithography and chemical wet etching processes**
    1. Obtain an ITO substrate (370 mm x 480 mm x 0.5 mm (L x W x H)) with a 2,600 Å ITO layer commercially (See Materials List). Slice the ITO substrate to the dimensions of 90 mm x 90 mm x 0.5 mm with a glass cutter for the ITO electrode patterning process in a 4 inch aligner.
    2. Use an ultrasonic cleaner to clean the ITO glass with acetone and then with deionized water, for 15 min each. Dry the ITO glass with clean dry air.
    3. Dispense 5 ml of positive photoresist solution onto the surface of the ITO glass.
    4. Use spin coater at 500 x g for 30 sec to produce a uniform photoresist layer. Then bake on a hotplate at 90 °C for 5 min to drive off excess solvent in the photoresist.
    5. Expose the ITO glass to 14 mW of ultraviolet light at 436 nm for 3.1 sec through a film photomask with the designed pattern (See Materials List).
    6. Immerse the sample in 60 ml development solution at 23 °C for 30 sec to develop the patterned routes. Then bake on a hotplate at 120 °C for 10 min to harden the photoresist and improve photoresist adhesion.
    7. Immerse the sample for 3 min in 60 ml etching solution at 80 °C to etch the unprotected ITO layer.
    8. Immerse the sample for 1 min in 60 ml acetone to remove photoresist on surface of the ITO glass.
    9. Slice the ITO glass into the dimensions of 62 mm x 35 mm for the experimental ITO electrode chip (**Figure 1**) with a glass cutter.

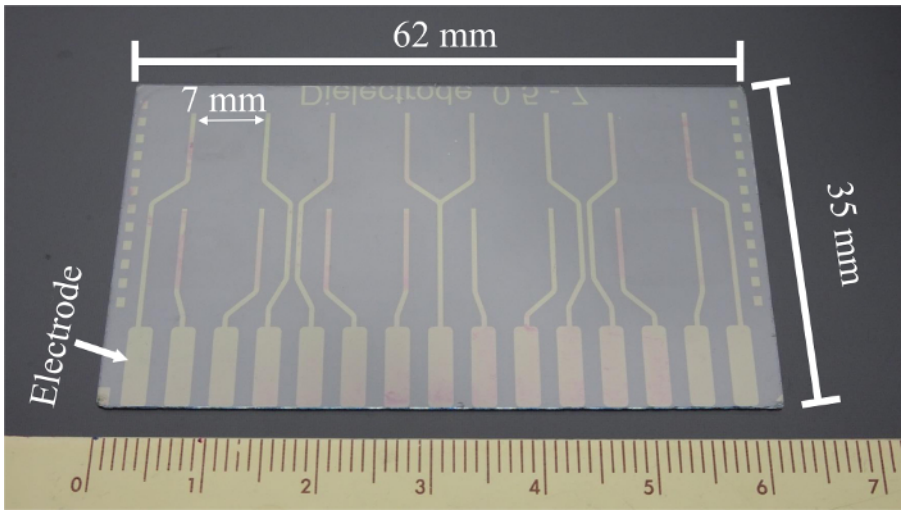

Figure 1: ITO electrode chip. The fabricated ITO chip with 8 pairs of electrode-patterned routes is shown. There are 15 electrodes measuring 2 mm x 8 mm at the side edge, and the central two routes share the same electrode. The distance between each pair of electrode fingers in a test well is 7 mm. Please click here to view a larger version of this figure.

2. **Construct the experimental chip module**
    1. Clean the commercial 8-well silicone array with an ultrasonic cleaner as shown in **Figure 2** with detergent, then deionized water, then 95% ethanol, and then deionized water, for 15 min each.
    2. Dry the 8-well silicone array by blowing clean dry air.
    3. Press the 8-well silicone array into the ITO chip in order to form the experimental chip module (**Figure 3**). Tightly bind the silicone array and ITO chip.

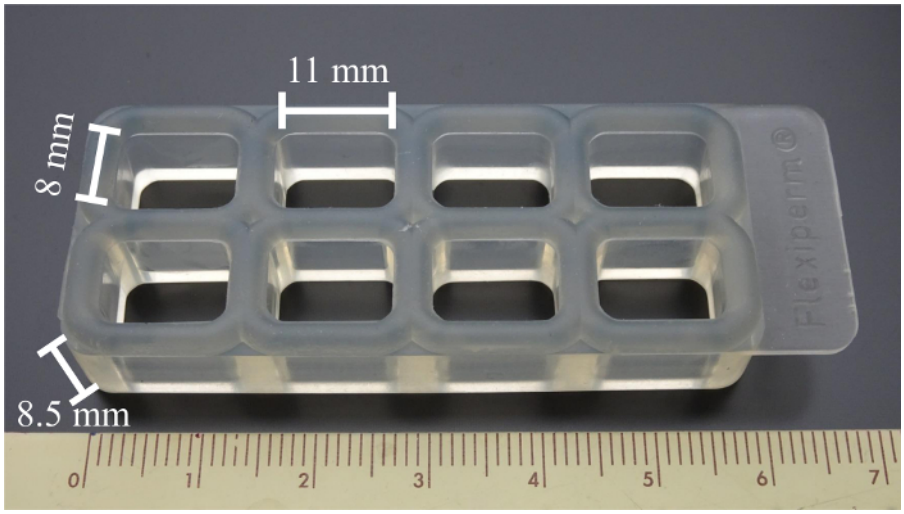

Figure 2: Silicone well array. The commercial 8-well silicone array can hold 8 tested samples simultaneously. The size of each well is 11 mm x 8 mm x 8.5 mm (L x W x H). Please click here to view a larger version of this figure.

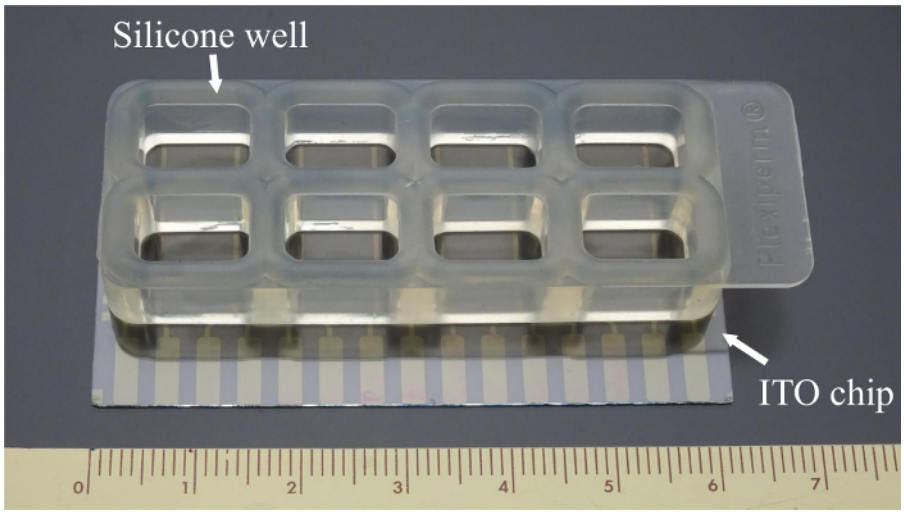

Figure 3: Experimental chip module. The ITO electrode chip is attached with the 8-well silicone array to form the experimental chip module. The adhesion between the silicone array and the ITO chip is strong. Therefore, the silicone array and the ITO chip can bond together for use without any adhesive substance. Please click here to view a larger version of this figure.

2. Impedance Measurement

1. Connect the personal computer, lock-in amplifier, and switch relay to form the impedance readout module as shown in **Figure 4**.

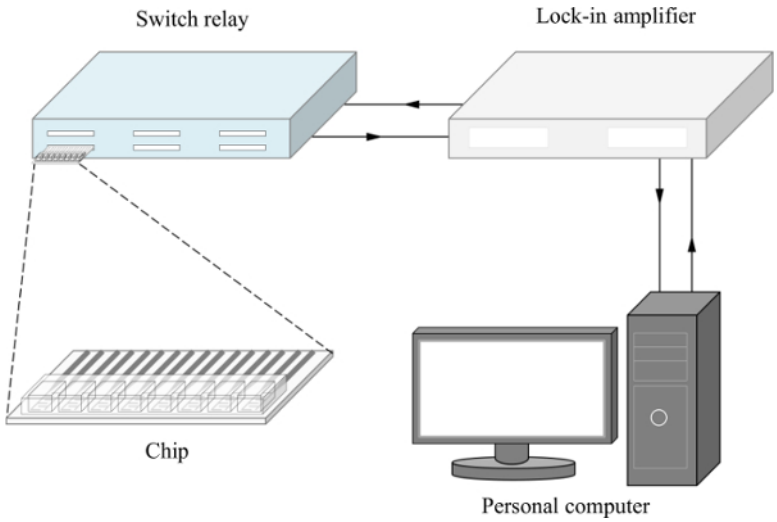

Figure 4: Schematic of the impedance-based apparatus. The lock-in amplifier, switch relay, and personal computer comprise the impedance readout module. The commercial phase-sensitive lock-in amplifier is used to send and extract the electrical signals. The homemade switch relay circuit connecting various ITO chips is used to specify which well and which ITO chip to be tested. A total of 6 chips can be connected to the switch relay specifying 48 samples in a time sharing manner. The real-time in-phase resistance and the signal phase shift of the tested solution are recorded continuously on a personal computer for the whole evaporation process. Please click here to view a larger version of this figure.

2. Put the experimental chip module into the socket of the switch relay.
3. Input parameters in the computer program. Input the signal frequency (1 kHz), the specified well number (0-7), the execution cycle (100), and the filename (HA).

3. Evaporation Experiments

1. Prepare four 2.5 ml hyaluronic acid solutions at 0, 0.05, 0.5 and 1 w/v% in water. Place each 2.5 ml sample solution in a vial measuring 14.75 mm x 45 mm x 8 mm (O.D. x H x I.D.).
2. For each solution, add 0.5 ml sample solution to a single well of the ITO chip module.
3. Weigh and record the initial weight of each vial by the electronic balance machine.
4. Execute the computer program to automatically measure and record the real-time in-phase resistance and the signal phase shift of specified wells on the ITO chip.
5. Start the evaporation experiments simultaneously in the same place by both the weighing method and impedance method.
6. Weigh and record the weight of each vial by the electronic balance machine at scheduled time points.
7. Analyze collected data in the weighing method and impedance method.[19]

Representative Results

During the evaporation process, the conductive ions in the tested solution became concentrated with the decreasing solution volume, and the impedance of this solution decreased. The rates of weight loss and impedance decrease in the evaporation progress for each tested solution were measured. For comparison purposes, the data in the rates of weight loss and impedance decrease were normalized to water and then plotted together in **Figure 5**. As illustrated in **Figure 5**, the weight loss demonstrates the same tendency as impedance, and shows that the relative evaporation rate to water evaporation decreases with the hyaluronic acid concentration. However, a large amount of variation is found in the proposed impedance-based approach than in the conventional weighing method for the evaporation examinations. The normalized data only had a 0.06 fall from 0% to 1% hyaluronic acid concentration intervention in the weighing approach, while a tremendous drop of 0.84 was found in the impedance-based apparatus. The simple linear equation is used to relate the normalized rates of weight loss and impedance decrease.

$Y = 0.0852X + 0.9166$, $R^2 = 0.97$

where X and Y represent the normalized rates of impedance decrease and weight loss, respectively. The rate of weight loss, *i.e.,* the evaporation rate of interest, in hyaluronic acid solution can be found correspondingly by way of the measured data in the impedance decrease. In practical applications, the measured impedance data can be quickly converted into the weight loss of hyaluronic acid solution by this linear equation.

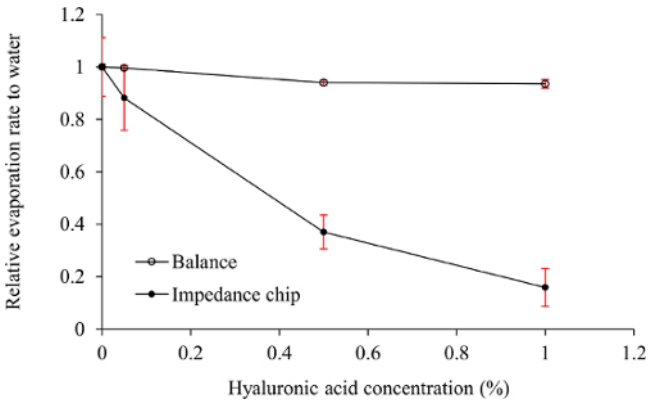

Figure 5: Relative evaporation rates to water of hyaluronic acid solutions at different concentrations. The relative evaporation rate to water is defined as the evaporation rate of a solution normalized by water. The relative evaporation rate to water against hyaluronic acid concentration by tests of balance and impedance chip are shown together for comparison. There is a larger change in the testing of impedance chip as compared the testing of balance. The error bar is the standard deviation in three experiments. Please click here to view a larger version of this figure.

Discussion

The critical step for evaporation measurement in this impedance-based detection is the preparation of the tested solutions. Deionized water cannot be used due to its enormous impedance. Instead, tap water containing conductive ions was used to prepare hyaluronic acid solutions for experiments. However, the electrical properties of tap water were not constant for use. Therefore, normalization, such as the relative evaporation rate to water in this study, was adopted as an alternative index for evaporation. The limitation of this technique is that tested solutions must have conductive ions for electrochemical characterization.

Very recently, a graphene-based impedance chip has been proposed for the modification of this technique[20]. With exceptional electronic and optoelectronic properties, graphene has attained considerable attention as an alternative to ITO for various electrode or conductor applications. The graphene-based finger-like electrode chip was successfully demonstrated in examining the stability of emulsion products by electrochemical impedance spectroscopy.

This study revealed that a 0.05% hyaluronic acid solution can reduce the relative evaporation rate to water by 12% as measured by the impedance. Therefore, topical application of 0.1% hyaluronic acid cream can lead to a significant improvement in skin hydration[21]. The molecular weight of hyaluronic acid plays an important role in its applications. For example, hyaluronic acid with a higher molecular weight could have better analgesic effects[22]. The application of low-molecular-weight hyaluronic acid had a significant reduction of wrinkle depth due to better penetration abilities[21]. In the future, the effects of the molecular weight on the moisturizing capacity of hyaluronic acid can be studied simultaneously on this impedance-based platform with multiple sample measurements for comparison purposes. A total of 6 chips can be connected to the homemade switch relay specifying the well to be tested for a real time test on 48 samples in a time sharing manner.

Although the conventional weight change approach stands as a simple and the most straightforward way to measure the moisturizing capacity of a solution, it is a time-consuming approach for observing enough weight change to determine an accurate evaporation rate. For example, it took about half a day to detect the desired evaporation rate of hyaluronic acid solution due to the detection limit of a precision balance with reasonable experimental error in this study. However, the electrical property of a solution is more sensitive than weight. The change in electrical properties can be detected sooner than weight loss in the evaporation process. In this study, the change rate in electrical impedance of hyaluronic acid solution at the end of a one hour observation period of evaporation was sufficiently determined. Therefore, the presented impedance-based detection apparatus is found to outperform the conventional weighing method in terms of detection sensitivity and response time.

Corresponding to the previous publication[23] and commercial device for the assessment of transdermal water loss, the electrical property can be treated as an index to reflect the evaporation rate. However, this presented impedance-based detection apparatus shows following advantages over the former: (i) a small sample volume requirement, (ii) parallel detection, (iii) easy disassembly for cleaning and reuse, and (iv) multiple applications such as bio molecular detection, cellular behavior, and phase separation[16-19]. The proposed high-sensitivity and fast-response impedance-based apparatus is validated as a superior candidate to handle evaporation tests relative to a conventional weight loss approach. In the future, this proposal impedance-based apparatus can also potentially be applied in any intrinsic property of a material or a specific process that could affect the conductivity of an electrochemical system[24].

Disclosures

The authors have nothing to disclose.

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