Monitoring of must fermentation progress by polymer coated capacitive vapour sensor arrays

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Summary

Must fermentation is a complex enzymatic process where apart from the conversion of sugars to ethanol and CO_2, a variety of compounds (e.g. esters) responsible for the wine aroma, are produced in orders of magnitude lower concentrations. The present work deals with the challenging task to monitor the must fermentation progress and detect possible deviations from optimum fermentation with a gas sensor array. Each sensor is an interdigitated capacitor fabricated with 2.0μm conventional microelectronic technology and coated with polymeric film through a well formatted around the capacitor. The sensing performance of several polymers was evaluated in terms of sensitivity and selectivity. In general, hydrophilic polymers present a negative response while hydrophobic a positive response, if water is considered as reference. PCA analysis revealed that a successful fermentation follows a distinct curve not related to that obtained from a series of equivalent pure ethanol solutions.

Motivation

The development of a device for real-time control of must fermentation is a challenging task[1] requiring the use of a sensor array and further application of suitable algorithms for the signal discrimination. Furthermore, the solution to be developed should be able to perform the must fermentation process monitoring without pre-processing steps for enrichment of the sample under analysis[2].

Results

The sensor array implemented in the present study is an array of polymer coated InterDigitated Capacitors (IDC). The IDC array consists of 8 sensors and is fabricated on quartz wafers with 2.0um resolution conventional microelectronic processes (lithography, metal deposition, lift-off). Prior to the polymer application, suitable wells from SU-8 are formed around each IDC (1X1mm^2 sensing area) to define the sensing area in a reproducible way (fig.1). A large number of polymers were evaluated in terms of sensitivity and selectivity over model analytes (ethanol, mixtures of ethanol – water, ehylacetate etc.) and the most promising were deposited on the sensor array. In fig. 2 the dynamic response of two representative polymers is shown. The reference for these measurements is concentrated humidity. Standard chemical analysis was applied simultaneously to determine the evolution of alcohol content, and of several VOCs produced during fermentation. In the example of fig. 2, moisture-sensitive poly (2-hydroxyethyl methacrylate) [PHEMA] shows a negative response due to the lower volume fraction of water in the must, as compared to the reference. On the other hand, the response of a poly (dimethylsiloxane-co-diphenylsiloxane) hydroxy terminated [P(DMS- co-DPhS)-OH terminated] is positive. In Fig. 3 the response of two selected polymers over the entire fermentation period is shown. The response clearly depends on the fermentation day. Finally, in fig. 4 the PCA result of the capacitance responses is shown. The fermentation progress follows a path that is clearly different from that of a series of standard solutions of equivalent ethanol concentrations. This result clearly shows that the monitoring of fermentation is possible without using any preconcentration stage and could be potentially be applied for process control.

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[1] C. Pinheiro, C. M. Rodrigues, Th. Schafer, J. G. Crespo, "Monitoring the aroma production during wine-must fermentation with an electronic nose", Biotechnol. Bioeng. vol. 77, p. 632-640, 2002.

[2] M. Maciejewska, A. Szczurek, Z. Kerényi, "Utilisation of first principal component extracted from gas sensor measurements as a process control variable in wine fermentation", Sens. Actuat. B vol. 115 p. 170-177, 2006.

Figures

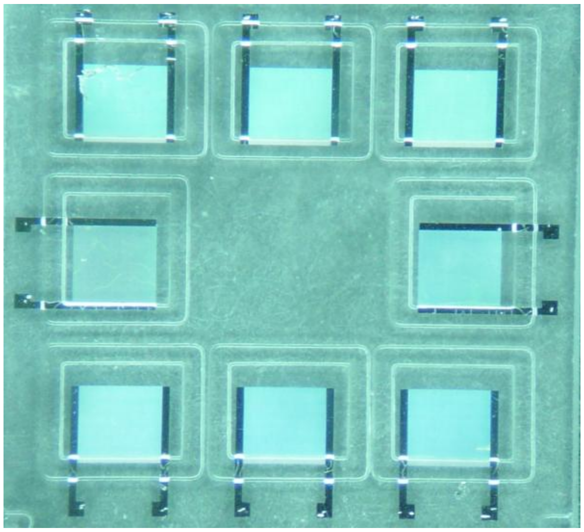

Fig. 1: Optical micrograph of the IDC sensor array prior to the deposition of the polymeric films and the wire bonding. The wells around each IDC are shown.

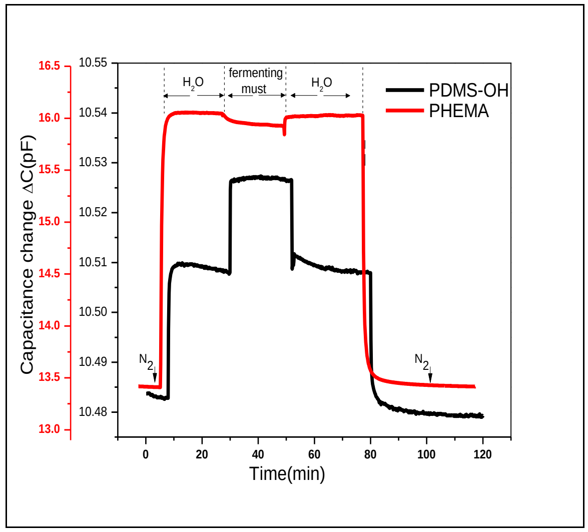

Fig. 2: Dynamic response of two representative polymers [P(DMS-co-DPhS)-OH terminated, PHEMA], upon successive exposures to pure water vapor and must fermented for 9 days.

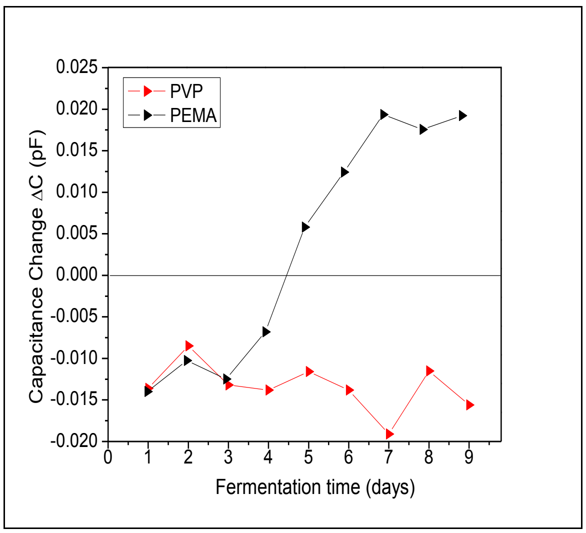

Fig. 3: Equilibrium responses of two polymers [hydrophilic Polyvinylpyrrolidone (PVP) and hydrophobic poly (ethyl methacrylate) (PEMA)] over the entire fermentation period.

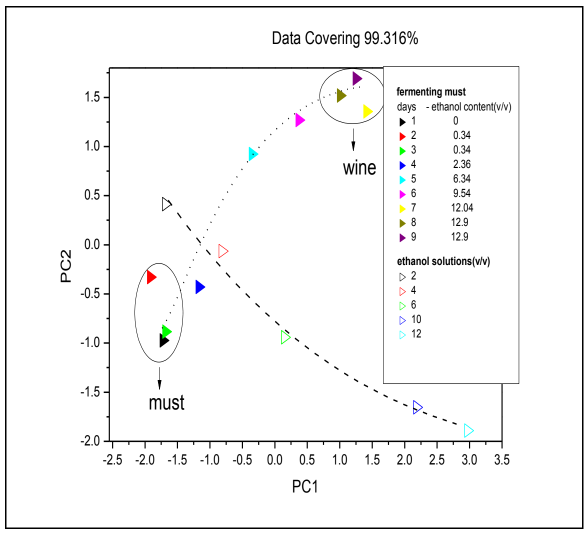

Fig. 4: PCA analysis of the sensor array equilibrium responses during fermentation progress. The PCA analysis of a series of equivalent pure ethanol solutions is also shown.