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Miniature taste sensing system based on dual SH-SAW sensor device: an electronic tongue

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Abstract

We report on the design and performance of a novel analytical sensing system, a so-called electronic tongue; it is based upon a dual shear horizontal surface acoustic wave (SH-SAW) device that discriminates between liquids of different basic tastes. Sixty megahertz SH-SAW *dual* delay-line sensors were micro-fabricated on 36° rotated Y-cut X-propagating $LiTaO_3$ and placed below a miniature PTFE housing containing the liquid under test. In this SAW sensor one delay-line is electrically shorted and the other is free. Synthetic samples were analyzed with the four basic tastes of sour, salt, bitter, and sweet. The electronic tongue classified correctly all of the different basic tastes *without* a selective biological or chemical coating. Theory relating to the electro-acoustic properties represented by the relative permittivity and conductivity of the sample liquid is presented and related to experimental results. Dilution tests have also been performed in order to determine the detection limit of this physical taste sensor and hence its application potential.

Keywords: Electronic tongue; SAW; SH-SAW device; Taste sensor; Piezoelectric sensor

1. Introduction

In the food and beverage industries, and other areas such as environmental testing and medical diagnostics, the development of simple liquid/taste and gas/odour sensors is very desirable. In general, acoustic microsensors detect different physical properties, such as mass, density and viscosity of liquids and gases, and offer the benefits of real-time electronic read-out, small size, robustness, and low unit cost. In particular, surface acoustic waves (SAW) devices are of considerable interest for sensing applications in gaseous and/or liquid environment. As a SAW propagates on a piezoelectric crystal surface, the interaction of a gas or a liquid with the surface results in a change of the propagation characteristics of the wave. Thus, the detection of liquid or gaseous properties can be achieved by measuring changes in the velocity, frequency, amplitude or phase of the acoustic wave. Here we propose a shear horizontal surface acoustic wave (SH-SAW) *electronic tongue* sensor system for liquid testing, based upon a dual delay-line interdigital transducer (IDT) configuration. Previous studies have been reported in which piezoelectric devices have been used to distinguish between different beverages, such as fruit juices and whisky [1,2]. We utilize here a configuration that allows measurement of both mechanical and electrical characteristics of the liquid firstly to discriminate between different milk samples [3] and now to determine the different *taste* properties of liquids.

We believe that the term "electronic tongue" was first introduced at the Eurosensors X conference in 1996 [4] while the first concepts of taste sensors were perhaps published some years earlier, in 1990 [5]. The majority of the work reported on electronic tongues is based on electrochemical rather than acoustic principles. The two most common electrochemical techniques used are potentiometric [4–6] and amperometric (or voltametric) [7–9]. Potentiometric sensors employ zero-current and utilise either ion selective lipid membranes or ion selective electrodes with the potential at the working electrode measured, whereas amperometric sensors employ a potential to drive an electrode transfer reaction, and the resulting current is measured. In both electrochemical techniques ion selective electrodes/membranes are generally utilized to make them more specific to certain types of chemical species. Unlike electronic tongue devices reported to date, the devices described in this paper are miniature, low-cost, robust, durable, micropower devices based on *physical* rather than electrochemical principles. The main advantage of our proposed SH-SAW sensor is that

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it can detect various properties of an adjacent liquid *without* any membrane. Moreover, electrochemical sensors are extremely sensitive to the electrode impedance and thus susceptible to surface contamination or fouling. Our dual delay line SH-SAW design can ameliorate this problem (associated with Winquist et al. type electronic tongues) and thus make a more robust device. The approach adopted is based on a generic fingerprint correlated to key physical parameters and so does not employ a bio-chemical selective layer. This in turn increases the lifetime and durability of the resultant devices, albeit with a loss of specificity and, in some applications, sensitivity. However, if necessary, they can still be made more selective and sensitive to certain types of species by simply coating the sensing area with the selective membranes.

The sense of taste may be presented in two ways. One aspect relates to the basic taste of sour, salt, bitter, sweet, and 'umami' that originates from different chemosensory receptors on the tongue called papillae. This aspect of taste is often referred to as the sensation of basic taste and stimulated by glutamate salts e.g. monosodium glutamate [10]. The other major aspect of taste called 'mouth feel' is related to the impression obtained when food enters the mouth and mastication. Here we use our sensor system to respond specifically to the basic tastes of the tongue and hence determine qualities of liquids in terms of sweetness, sourness, saltiness and bitterness.

2. Theory and operating principle

The majority of the work reported on SAW sensing involves the use of Rayleigh waves. For gas sensing applications, Rayleigh SAWs are ideal but they are not well suited to liquid sensing. This is because Rayleigh waves have both a surface-normal component as well as and a surface-parallel component with respect to the propagation direction and hence couple strongly with a liquid [11]. However, surface waves that have shear horizontal (SH) polarized displacement can propagate along the interface between the liquid and substrate without coupling strongly into the liquid, but will still be influenced by the liquid properties [12]. We have utilised this phenomenon in the development of our smart tongue device consisting of two SH-SAW delay lines designed using the software package L-Edit (Tanner Tools) and fabricated on a 36° rotated Y-cut X-propagating $LiTaO_3$ (36YX.LT) substrate. The device is used for simultaneous measurements of both mechanical (physico-acoustic) properties, and electrical (electro-acoustic) parameters of the liquid under test. This is achieved through a dual delay-line configuration, one shorted (metallised and electrically shielded) and the other left free (electrically active). This way, the shorted delay-line measures mechanical parameters predominantly mass loading and viscosity whilst the free delay-line additionally measures permittivity and conductivity of the liquid under test; electrical parameters can be related to certain taste properties, such as saltiness (sodium) and sweetness (sucrose) [13]. Fig. 1 shows a schematic of the basic arrangement of the dual delay-line sensing system.

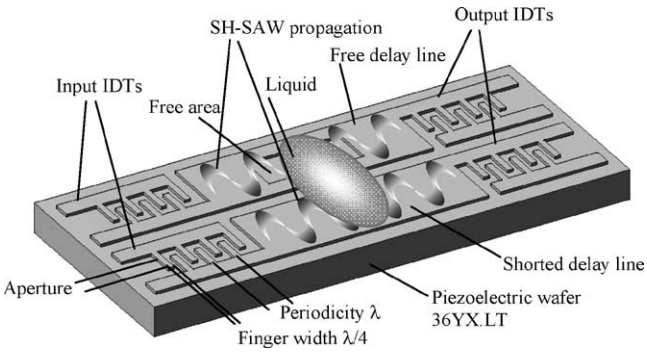

Fig. 1. Schematic of dual-delay line SH-SAW taste sensor.

The SH-SAW propagating on the surface of 36YX.LT substrate is sustained by both atomic displacements and electrical potentials due to the piezoelectric effect. When the surface of the substrate is metallised and electrically shorted the piezoelectric potential becomes zero at the surface and only the atomic/particle displacements interact with the adjacent liquid. This phenomenon is known as mechanical perturbation (or mechanical interaction) [14] and can be used to detect the mechanical properties of a liquid, e.g. its viscosity and density. On the other hand, when the surface is free and electrically active both the particle displacements and electrical potentials interact with the liquid. This is an electrical interaction (also known as the acousto-electric interaction/perturbation) with the liquid and affects the velocity and/or attenuation of SH-SAW propagation and it is utilised in sensing the electrical properties of the liquids, e.g. the relative permittivity and conductivity [14].

2.1. Electro-acoustic interaction

The electrical properties of a liquid are given by the relative permittivity, ε_r and its electrical conductivity, σ. By employing the perturbation theory proposed by Auld [14], and assuming the reference liquid is a non-conductive solution $\sigma = 0$ we have simply

$$\varepsilon_l = \varepsilon_r \varepsilon_0 \tag{1}$$

where ε_l and ε_0 are the permittivity of the reference liquid and that of free space, respectively, and ε_r is the relative permittivity of the unperturbed liquid. When perturbed, the permittivity of the liquid becomes a complex permittivity ε_l' given by

$$\varepsilon_l' = \varepsilon_r' \varepsilon_0 - j\frac{\sigma'}{\omega} \tag{2}$$

with σ' being the conductivity. Following a perturbation, the changes in the velocity and attenuation may be approximated

by [15]

$$\frac{\Delta v}{v} \approx -\frac{K_s^2}{2}\frac{(\sigma'/\omega)^2 + (\varepsilon_r'\varepsilon_0 - \varepsilon_r\varepsilon_0)(\varepsilon_r'\varepsilon_0 + \varepsilon_P^T)}{(\sigma'/\omega)^2 + (\varepsilon_r'\varepsilon_0 + \varepsilon_P^T)^2} \quad (3)$$

$$\frac{\Delta \alpha}{k} \approx \frac{K_s^2}{2}\frac{(\sigma'/\omega)(\varepsilon_r\varepsilon_0 + \varepsilon_P^T)}{(\sigma'/\omega)^2 + (\varepsilon_r'\varepsilon_0 + \varepsilon_P^T)^2} \quad (4)$$

Here, K_s^2 is the electromechanical coupling coefficient when the unperturbed liquid is loaded on the free surface, ε_P^T is the effective permittivity of the SAW crystal, ε_r is the relative permittivity of the reference liquid (distilled water), ε_r' and σ' are the relative permittivity and conductivity (related to loss) of the measurand, respectively. The change in velocity $\Delta v/v$ and attenuation $\Delta\alpha/k$ (where k is the wave number: $k = 2\pi/\lambda$ and λ is the wavelength) can be determined from the phase difference and amplitude ratio measured using a vector voltmeter. By eliminating the permittivity or conductivity from Eqs. (3) and (4), the following circle formulae can be obtained. They can be used to create a permittivity-conductivity chart as shown in Fig. 2 and therefore to determine permittivity and conductivity of the liquid under test [13].

$$\left[\frac{\Delta v}{v} + \frac{K_s^2}{4}\frac{(2\varepsilon_r'\varepsilon_0 - \varepsilon_r'\varepsilon_0) + \varepsilon_P^T}{\varepsilon_r'\varepsilon_0 + \varepsilon_P^T}\right]^2 + \left[\frac{\Delta\alpha}{k}\right]^2 = \left[\frac{K_s^2}{4}\frac{\varepsilon_r\varepsilon_0 + \varepsilon_P^T}{\varepsilon_r'\varepsilon_0 + \varepsilon_P^T}\right]^2 \quad (5)$$

$$\left[\frac{\Delta v}{v} + \frac{K_s^2}{2}\right]^2 + \left[\frac{\Delta\alpha}{k} - \frac{K_s^2}{4}\frac{\varepsilon_r\varepsilon_0 + \varepsilon_P^T}{\sigma'/\omega}\right]^2 = \left[\frac{K_s^2}{4}\frac{\varepsilon_r\varepsilon_0 + \varepsilon_P^T}{\sigma'/\omega}\right]^2 \quad (6)$$

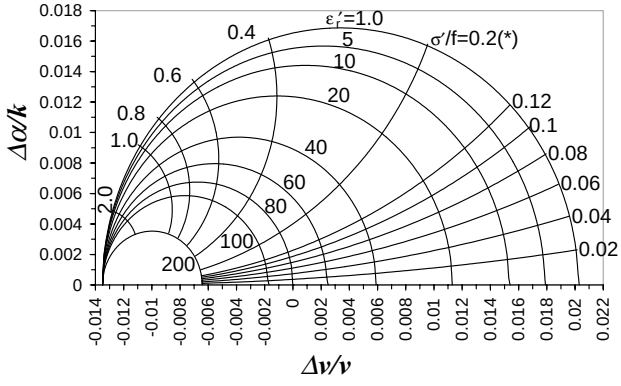

Fig. 2. Permittivity-conductivity chart (*: 1×10^{-8} (S/m)/Hz) showing theoretical behaviour.

3. Electronic tongue design and fabrication

The interdigital transducer (IDT) is an essential component of the SAW device. The IDTs convert electrical energy into mechanical energy, and vice versa, for the generation and detection of the surface acoustic waves. Several requirements were taken into consideration in the design of the new SH-SAW liquid sensors. In order to measure easily the delay time difference between the two delay lines of the SH-SAW device long delay times, long delay lines and thus large devices are desirable. However, both the sensitivity and response time (due to thermal properties) improve with smaller devices. The waves interfere constructively when the distance between the adjacent finger pairs of the IDTs is equal to one half the wavelength of operation, with maximum positive interference occuring at the resonant frequency. At this frequency, the efficiency of the transducer in terms of converting the electrical energy to mechanical acoustical energy, or vice versa is maximised. By choosing the aperture and the number of finger pairs, the IDT can be matched to a given input line, thus giving low insertion loss for a device. The bandwidth of the transducer is also dependent upon the number of transducer fingers; increasing the number of fingers decreases the bandwidth [16]. Conversely, in order to minimize the device capacitance the number of fingers should be kept low. Moreover, to minimize conversion losses the number of fingers should be high and diffraction losses can be kept to a minimum when the aperature is large [17]. Considering these various constraints, the SH-SAW device's transmit and receive transducers were optimally designed to have 28 finger pairs with 17 μm width of electrode and 17 μm separation between each electrode, i.e. a periodicity of 68 μm. The IDT aperture is 2 mm with the IDT centre-to-centre separation of 7.5 mm and a free area of 2.0 mm × 1.5 mm. The device operating frequency is approximately 60 MHz.

The devices were fabricated using a simple, well established wet etching process. Firstly, the 3 in. $LiTaO_3$ wafers were carefully cleaned of any surface contaminats, such as dust and grease, in order to obtain good adhesion and uniform coating of metallic layers. The cleaning was performed by immersing the wafers in trichloroethylene at 60 °C for 10 min, followed by an acetone bath at 60 °C for 10 min. The wafers were rinsed with methanol and finally with distilled and deionised water. The wafers were then dried using compressed filtered (0.2 μm filtration) dry nitrogen. Next, thin metal films of chromium and gold with thicknesses of 20 and 120 nm, respectively, were deposited onto the wafers by dc sputtering using a commercial CVC 601 system. The thickness uniformity of the metal layers across the profile of the wafer is very important to ensure consistent behaviour from device to device. The wafer was then coated with a photoresist solution (Hoechst AZ5214E (28% PGMEA)) and spun at 4500 rpm for 45–60 s to produce a thin film over the wafer. The wafer was then given a soft bake on a hotplate at 90 °C for 2 min. After this soft bake it was exposed to ultraviolet

light for 1.5 s, using a mask alligner (Karl Suss MA-6 system), through a 4 in. photomask (1.5 mm thick white crown flint glass coated with anti-reflective chrome and resolution of 1 μm), which defines the geometry of the devices to be produced on the wafer. This photomask was designed using an L-Edit (Tanner Tools Inc.) commercial software package, and then the mask design files were converted into a GDS II format (offers compatibility with E-beam writer) and supplied to Compugraphics International Limited (UK) who fabricated the mask. Following exposure, the positive resist was chemically developed by immersing the wafer in developer (1:4 mixture of AZ 400 K and DI water) for 45–60 s, so that the regions exposed to light were removed leaving exposed areas of metal. Finally, chemical etching (using standard ISO clean gold and chrome etch) of the exposed unwanted metal was performed, thus leaving the device structures on the wafer. The photoresist was kept on to protect the devices during dicing of the wafer. The wafers were diced using a diamond saw (Sola Basic Tempress model 602) leaving the edges parallel to the IDT fingers fairly rough to mimise normal reflection of the backward travelling waves from the edge of the device back into the IDTs. The final device dimensions were approximately 8.0 mm × 10.5 mm. Fig. 3 illustrates the main steps of the fabrication process.

In order to test the sample liquids, the devices were mounted on a custom designed printed circuit board (PCB) below a PTFE cell that contained the liquid under test. The cell is 32 mm in length by 20 mm wide with a central reservoir of 6.8 mm × 2.5 mm × 8.0 mm and a volume of approximately 136 μl. The liquid cell is positioned accurately over the sensing area between the IDTs with the aid of guiding pins that fit into holes in the PCB as shown in Fig. 4. The cell rests on the device without any sealant. This enables easy removal of the cell to clean the device and yet holds the liquid without leakage.

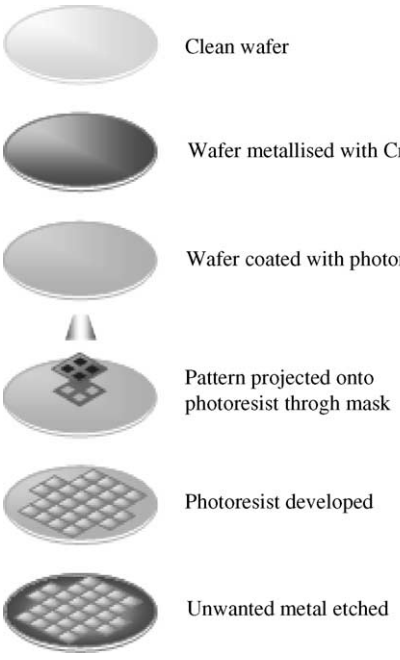

Fig. 3. Photolithography fabrication process used to make IDT SAW sensors.

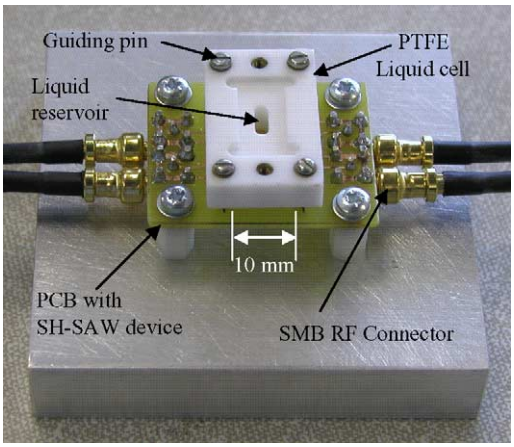

Fig. 4. Photograph of miniature total analysis taste system or electronic tongue.

4. Experimental

The experimental procedure for the SH-SAW devices involves the measurement of both the phase velocity and attenuation of the SH-SAW signals propagating on the two delay lines of the sensor. The set-up includes a signal generator (HP 8648C), the SH-SAW sensor and a vector voltmeter (HP 8508A). In this, an electrical signal is fed from the signal generator to the input IDTs. The amplitude ratio and phase difference between the input and output signals of each delay line, and the amplitude ratio and phase difference between the output of the shorted and free delay lines, were monitored by the vector voltmeter. The fractional velocity shift $\Delta v/v$ and attenuation change $\Delta\alpha/k$ of the SH-SAW can be determined from the phase difference $\Delta\phi$ and amplitude ratio A_f/A_s (subscripts f and s represent the free and shorted delay line, respectively) according to Eqs. (7) and (8) below.

$$\frac{\Delta v}{v} = \frac{v\Delta\phi}{2fl} \tag{7}$$

$$\frac{\Delta\alpha}{k} = -\frac{v\ln(A_f/A_s)}{2\pi fl} \tag{8}$$

where $\Delta\phi$ is the phase shift in degrees and A_f/A_s the amplitude ratio between the free and shorted delay lines, f the operating frequency, v the phase velocity of the SAW, and l the sensing (or interaction) length between the waves and liquid.

The experiments were performed at a temperature of (23 ± 0.1) °C using a commercial Techne Dri-Block® (DB-2D) heater. The solutions used for the typical ground tastes were HCl (sour), NaCl (salty), quinine (bitter) and sucrose (sweet). Equal volumes of the mixtures (60 μl) were

dispensed into the liquid cell using a micro-pipette (Gilson Pipetman P200) and the device and cell cleaned and dried after each sample was tested using de-ionised (DI) water. The samples were sequenced randomly, and five replicate measurements were conducted on each sample.

5. Results and discussion

Fig. 5 shows a 3D principal components analysis (PCA) of the set of attenuation and phase parameters for the different taste sample solutions. The amplitude ratio and phase difference on each of the delay lines (electrical shorted and free) were used as the four parameters for the PCA. The concentrations used here were 0.1 M for HCl, NaCl and sucrose, and 1.3×10^{-4} M for quinine. We can see from the plot that 100% linear separation of the basic tastes was achieved with the control, pure DI water, clearly distinct.

Further experiments were then performed in order to determine the effect of diluting the different taste solutions using DI water and repeating the tests. The 0.1 M solutions were diluted in steps by a factor of 2^n in which the exponent n varied from 1 to 5. Each time the volume of DI water was increased by a factor of 2^n and added to a fixed volume of the solutions. Fig. 6 shows the PCA plot for the lowest concentration solutions in the dilution test. Again from this plot 100% linear separation of the taste samples is clearly observed.

Fig. 7 presents a PCA plot of six different concentrations of these taste samples with DI water as the reference. From this plot it can be seen that as the solutions were diluted, the clusters of the individual concentrations tended towards that of DI water. These results show that the limit of detection of the different tastes is typically 0.1% or 1 part in 10^3.

Measurements made on the sample liquids of the different taste concentrations used in the dilution test were also analysed to determine their electrical properties. These measurements were performed using the same setup as for the other experiments except that the amplitude ratio and phase shift measured were between the outputs of the delay lines and not between the input and output of each delay line as before. The reference liquid used was again DI water and the attenuation change and velocity shift were determined from the phase difference and amplitude ratio. The results were plotted on the conductivity-permittivity chart from which the values can be obtained according to Eqs. (3) and (4). Fig. 8 shows specifically the results for the sour (HCl) and salty (NaCl) solutions. We can see, as expected, decreasing the concentration of the solutions decreases the conductivity with a small increase in the permittivity. The electrical conductivity of the HCl solutions varied approximately from 1.4 to 3.7 S/m and the electrical permittivity between 76 and 74, with increasing concentration. The conductivity of NaCl increased from 0.08 to 1.1 S/m with increasing analyte concentration (5.9×10^{-3} to 0.1 M). The sweet (sucrose) and bitter (quinine sulphate) solutions are very weak electrolytic

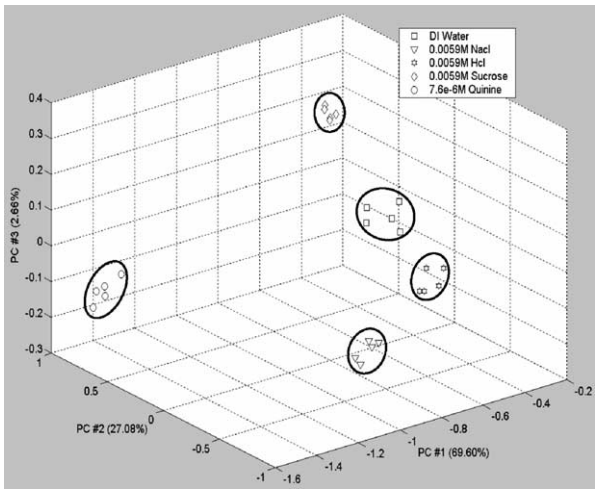

Fig. 6. 3D PCA plot for the lowest concentrations of the diluted taste sample solutions again showing 100% linear separation.

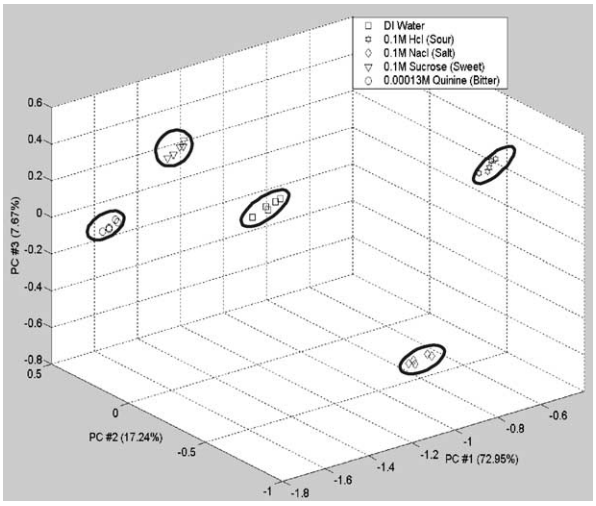

Fig. 5. 3D PCA plot for the four different taste solutions and reference liquid (DI water) showing 100% linear separation.

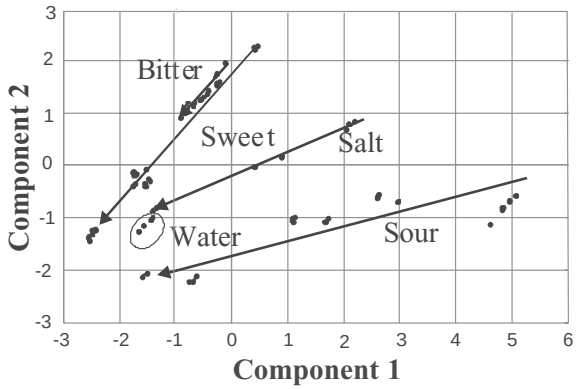

Fig. 7. 2D PCA plot for the dilution testing of the four different taste liquids. The stationary cluster of DI water is also shown.

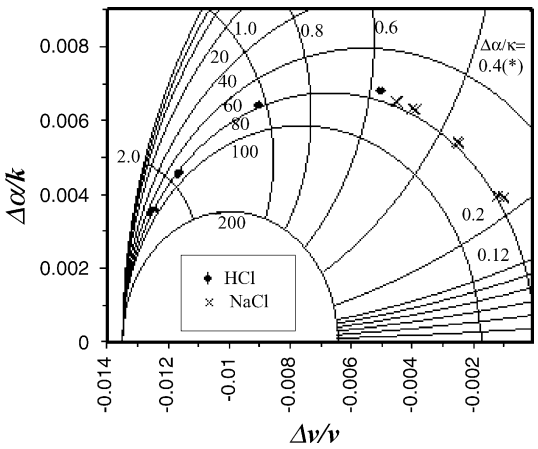

Fig. 8. Experimental results for salty (NaCl) and sour (HCl) solutions of different concentrations on the permittivity-conductivity chart. Separation is clearly visible.

solutions with conductivities close to that of DI water (i.e. practically zero) and so very small changes in conductivity with a change in concentration were noticed.

6. Conclusions

A taste sensing system has been developed and is based upon a dual SH-SAW sensor configuration that removes common mode effects e.g. drift/fouling. We have demonstrated the ability of the sensing system to discriminate between four basic tastes without the use of either electrochemical sensors or selective membranes.

The sensor responds to various *physical* parameters of the liquid, and so in an indirect rather than direct measure of taste. A detection limit of about about 0.1% has been achieved. The theory of the electro-acoustic interactions has been used to relate the experimental results to electrical properties of the liquids under test and promising results shown. Two physical parameters, conductivity or pH, were studied and cannot be used to classify uniquely both the type of taste and its concentration—whereas the four acoustic parameters can do so. We believe that viscoelastic coupling to the liquid is a critical parameter and should relate to "mouthfeel" (viscosity) as well. Here we have used single synthetic samples each related to a single basic taste, however the system has also been used to analyse complex samples, such as cow's milk, and the ability to determine milk freshness has been reported before [3]. In [3] we have shown that we are able to discriminate between different levels of sourness in milk, which is a complex mixture. The results showed that we could measure subtle changes in taste as this is related to significant cellular damage. Therefore, we believe that there are real world applications in which we can measure key flavours. Here we have shown that the sample vectors are simply linearly separable—a trivial pattern recognition problem. Thus there is scope to improve the discrimination power through the use of pre-processing (i.e. feature selection) and non-linear pattern recognition classifiers (e.g. support vector mapping). In another study our devices were used for the discrimination of commercial soft drinks such as Coke and Pepsi [18].

A microsystem version is under design that will work at higher frequencies (433 and 869 MHz) and should significantly improve the sensitivity. Higher sensitivities can also be achieved by the use of selective layers, as known from previous publications on biosensors. We believe that our system could eventually be used in certain applications as a simpler, more practical low-cost taste sensor than those sensors employing either electrochemical sensors or biological membranes.

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