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Dielectric properties of apples in the range 0.1–100 kHz

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Capacitance and conductance spectra are measured between 100 Hz and 100 kHz for three varieties of apple. Analysis of the spectra for several types of electrode material indicates that electrode effects may be neglected for frequencies above 1 kHz. Power-law responses are observed with $C \sim f^{-0.3}$ for the capacitance and $G_{a.c.} \sim f^{+0.9}$ for the a.c. conductance. Activation energies were found to be of the order of 0.05–0.15 eV for the three apple varieties and found to be frequency dependent. Comparison is made with an apple in which the cellular structure has been damaged by freezing. Freezing produced a significant increase in the high-frequency activation energy for conductance and a decrease in the power-law exponent to +0.3 for a.c. conductance. It is suggested that the dielectric properties result from a combination of relatively free ionic diffusion in the extracellular medium and the hopping of counterions along trap sites on the cell wall.

1. Introduction

Measurement of the dielectric properties of living biomaterials can provide information regarding such systems, which is both practical and fundamental. Changes in the dielectric properties can be correlated with variations in physiological state for a wide range of animal [1–6] and plant [7–16] systems. Understanding the basic polarization and conduction processes in biogenic biomaterials may lead to the discovery of a mechanism for the reported effects of low-frequency electric and magnetic fields [17, 18].

Although some dielectric properties have been tabulated for animal tissues [19, 20], others, such as activation energy, are more difficult to obtain. Animals will generate a physiological response if attempts are made to change significantly the temperature of their tissues. Furthermore, living animal systems are difficult to maintain under constant conditions while dielectric measurements are made for extended periods of time. These difficulties are not present for plant systems. In the work reported here, the variations of capacitance and conductance with frequency (dielectric spectra) of three varieties of apple were measured over a range of temperatures to obtain activation energies for polarization and conduction processes. Although animal cells are surrounded by a membrane, not a wall as for plant cells, the activation energies should be of comparable magnitudes.

Use of a four-electrode technique [8] would permit the clear separation of bulk and electrode dielectric properties, but would require considerable analysis and very high-speed analogue to digital converters (ADC) to sample high frequencies. The two-electrode system used here is well-suited for obtaining a large number of rapidly taken spectra. An important consideration in this paper will then be the determination of the frequency range over which electrode effects may be neglected.

Finally, in addition to reporting the systematic examination of the apple dielectric properties, this paper relates them to those of other living biomaterials and to models developed for inorganic materials.

2. Experimental procedures

Two electrodes were inserted into each apple and connected to a Hewlett Packard 4192A low-frequency impedance analyser. Data acquisition and storage were controlled by a Hewlett Packard Model 310 microcomputer. Three varieties of apple (Granny Smith, Rome and Red Delicious) were studied. Each electrode consisted of a row of five needles (length 2 cm, separation 1 cm). The two electrode rows were separated by 3 cm. Four types of metal were used for the needles: (1) silver solder (60% silver), (2) nickel-plated steel, (3) soft steel and (4) chrome-plated brass.

The impedance analyser is capable of collecting capacitance and conductance spectra over the range 5 Hz–13 MHz, but requires zeroing at each frequency below 500 Hz in the parallel circuit mode. Measurements on known *RC* networks [21] indicated that for impedances characteristic of apple interiors the error introduced by not re-zeroing at each frequency above 100 Hz was less than 5%. Above 100 kHz, rigid fixation of the leads was required to avoid errors greater than 5%. To allow rapid measurements in this pilot study, the frequency range was restricted to 100 Hz–100 kHz.

The inter-electrode region may be regarded as a series combination of the electrode–apple interface and the apple interior. Use of a two-electrode system, such as the 4192A, necessitates the determination of the frequency range over which electrode effects are significant and how this range depends on the choice of electrode material.

Insertion of needle electrodes into an apple produces an electrochemical response due to interactions with the surrounding material. Some fluids are already present in the extracellular space. Others are released from the cell interiors as the walls are damaged by the insertion process. Complicated electrochemical reactions take place between the ions in these fluids and the electrode material. Proteins and other material become deposited on the electrodes and modify their conducting properties. A certain amount of time is required before equilibrium is established. This time may depend on both the type of electrode and the variety of apple. Progress of the system toward equilibrium can be monitored by measuring the dielectric spectra at various times after insertion of the electrodes.

Figs 1 and 2 illustrate, respectively, the capacitance and conductance spectra of a Granny Smith apple into which silver electrodes were inserted at time $t = 0$. Spectra are shown for times up to 22.5 h. Little change is apparent in the capacitance spectra below 1 kHz or in the conductance spectra at any frequency. This combination of apple variety and electrode type is the most favourable. Similar readings were taken using the other three electrode types in Granny Smith apples and all four types in Rome and in Red Delicious apples for times extending up to 65 h. For some combinations of apple and electrode, more than 48 h were required before equilibrium was approximately achieved.

In general, Granny Smith apples tended to equilibrate faster than Rome or Red Delicious. Silver electrodes tended to come to equilibrium more rapidly than the other three types. All subsequent spectra were taken after a time sufficient for the apple–electrode combination to reach equilibrium.

3. Results

The capacitance and conductance of the sample can be represented, respectively, as

$$C(f) = K(f)\varepsilon_0 A/d \qquad (1)$$

and

$$G(f) = g(f)A/d \qquad (2)$$

where the dielectric constant, K, and the conductivity, g, depend on the frequency, f. A is the effective area of the inter-electrode region and d is the electrode separation. Because of the irregular surface geometry of an apple, A varies from sample to sample even though the dimensions of the electrode needle sets are themselves fixed. In order to compare different types of electrode or varieties of apple, normalized spectra must be used.

Let

$$C'(f) = C(f)/C_{100\,\mathrm{kHz}} = K(f)/K_{100\,\mathrm{kHz}} \qquad (3)$$

and

$$G'(f) = G(f)/G_{100\,\mathrm{kHz}} = g(f)/g_{100\,\mathrm{kHz}} \qquad (4)$$

represent the normalized capacitance and conductance of an apple. Plots of $C'(f)$ and $G'(f)$ versus f should thus be independent of sample geometry and facilitate comparison across electrode type and apple variety.

The contribution of the electrodes to the measured dielectric properties can be determined by comparing the normalized spectra for a single variety of apple

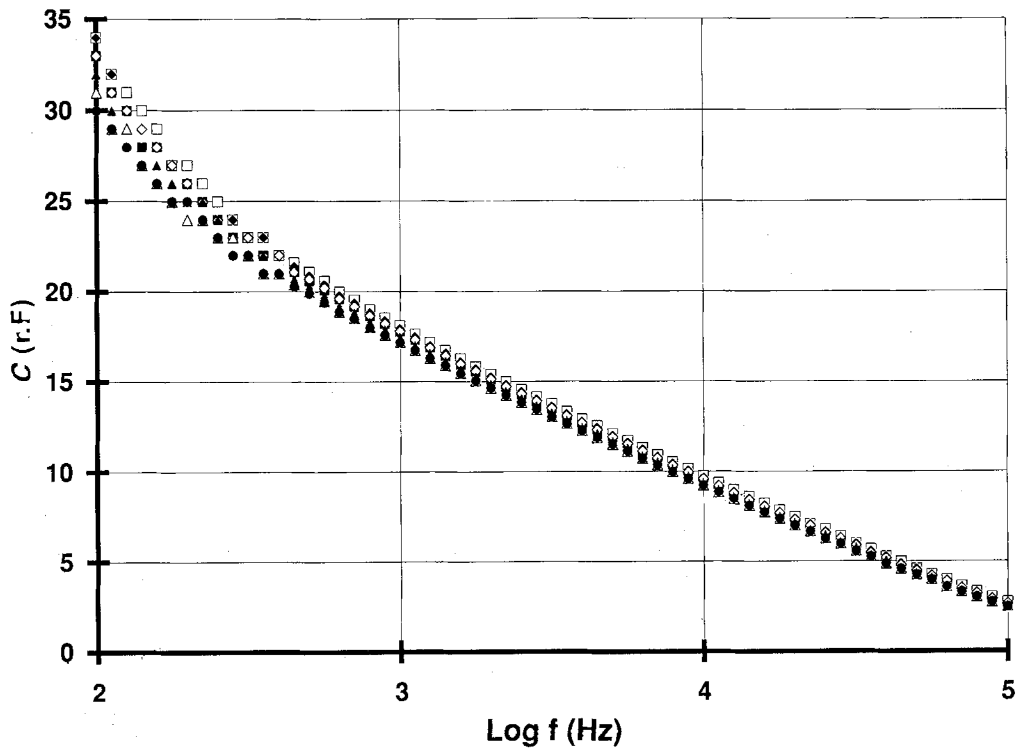

Figure 1 Change in the capacitance spectrum at various times, t, after insertion of silver electrodes into Granny Smith apples: (■) 0 h, (□) 2.5 h, (◆) 4.5 h, (◇) 6.75 h, (▲) 11.75 h, (△) 17.5 h, (×) 22.5 h.

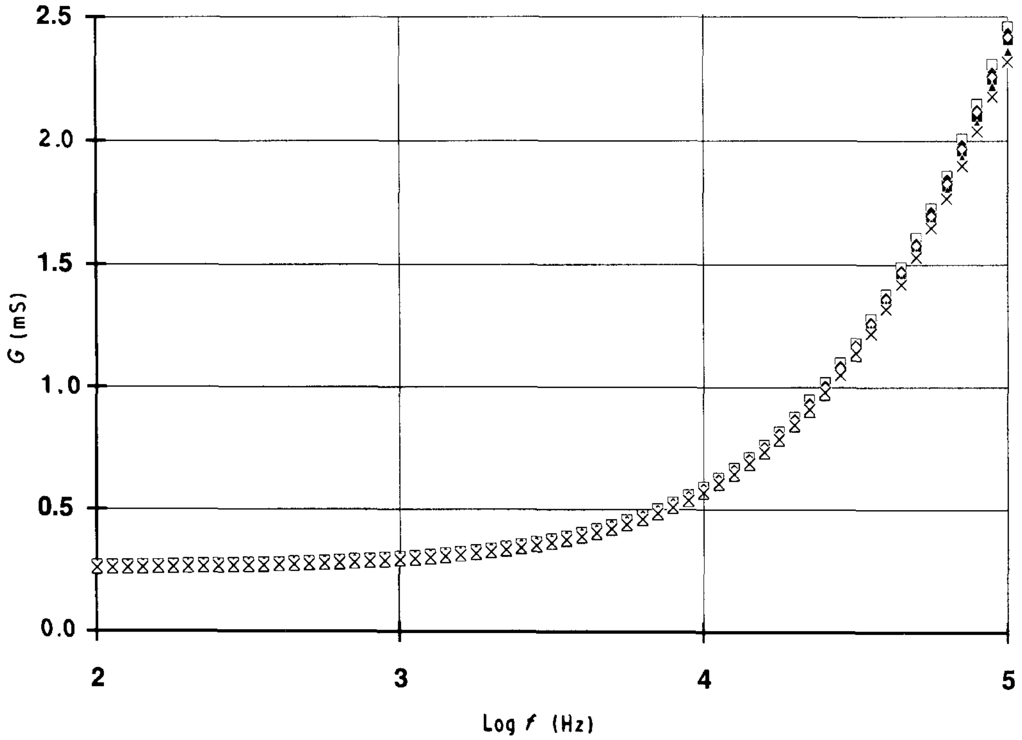

Figure 2 Change in the conductance spectrum at various times, *t*, after insertion of silver electrodes into Granny Smith apples: (■) 0 h, (□) 2.5 h, (◆) 4.5 h, (◇) 6.75 h, (▲) 11.75 h, (△) 17.5 h, (×) 22.5 h.

measured with each of the four electrode types. Figs 3 and 4 show the normalized capacitance and conductance spectra for each electrode type inserted into Granny Smith apples. Except for nickel electrodes, little difference is observed in the capacitance spectra. Significant deviations are present for nickel below about 5 kHz. Little difference is observed in the conductance spectra for any type of electrode. A very slight reduction is present for nickel below about 1 kHz. Similar results were obtained for Roma and for Red Delicious apples. Electrode effects appear negligible above 5 kHz.

Differences in the dielectric properties of the three apple varieties can be investigated by comparing their normalized spectra for a single electrode type. Figs 5 and 6 present the normalized capacitance and conductance spectra for silver electrodes inserted into each of the three varieties of apple. Although in both

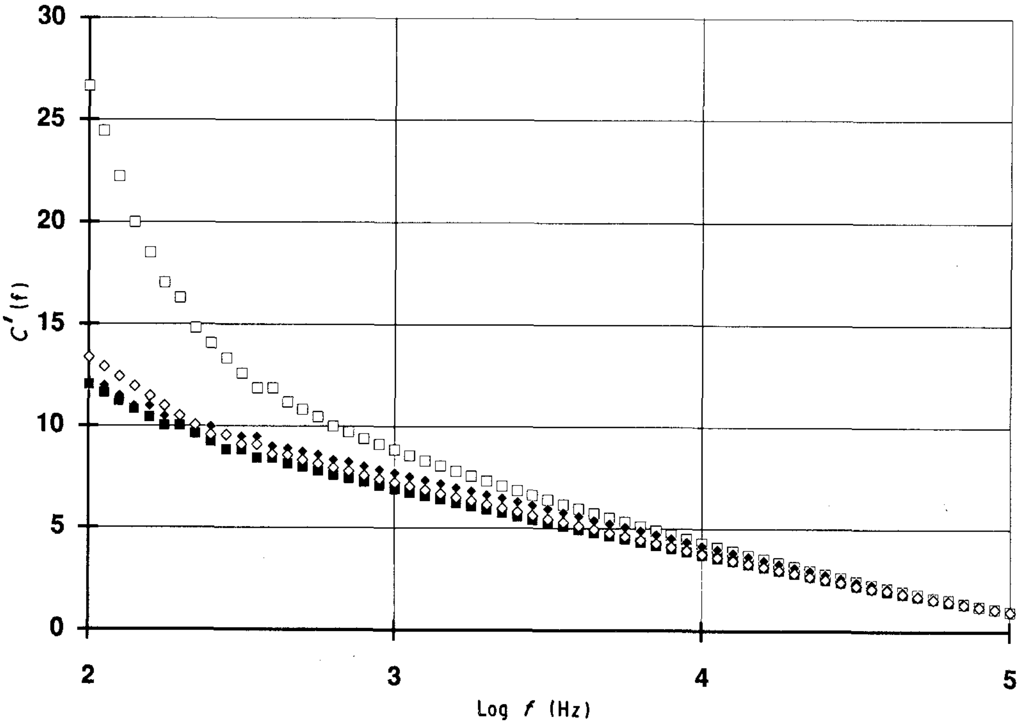

Figure 3 Normalized capacitance spectra for several types of electrode inserted into Granny Smith apples: (■) silver solder, (□) nickel-plated steel, (◆) soft steel, (◇) chrome-plated brass.

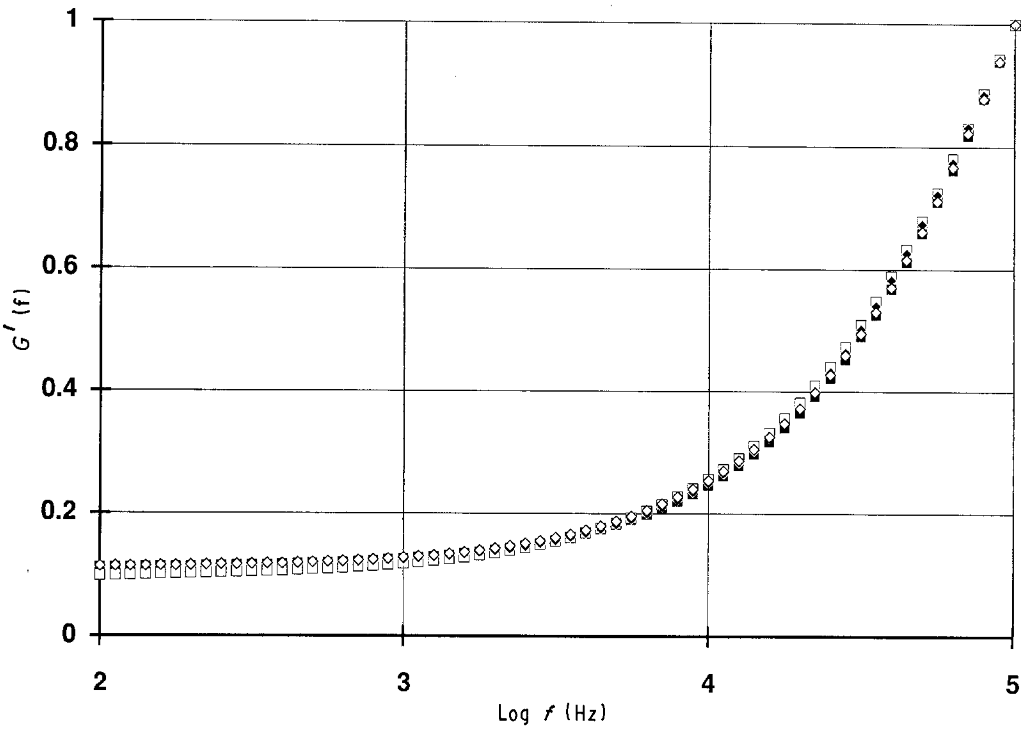

Figure 4 Normalized conductance spectra for several types of electrode inserted into Granny Smith apples: (■) silver solder, (□) nickel-plated steel, (◆) soft steel, (◇) chrome-plated brass.

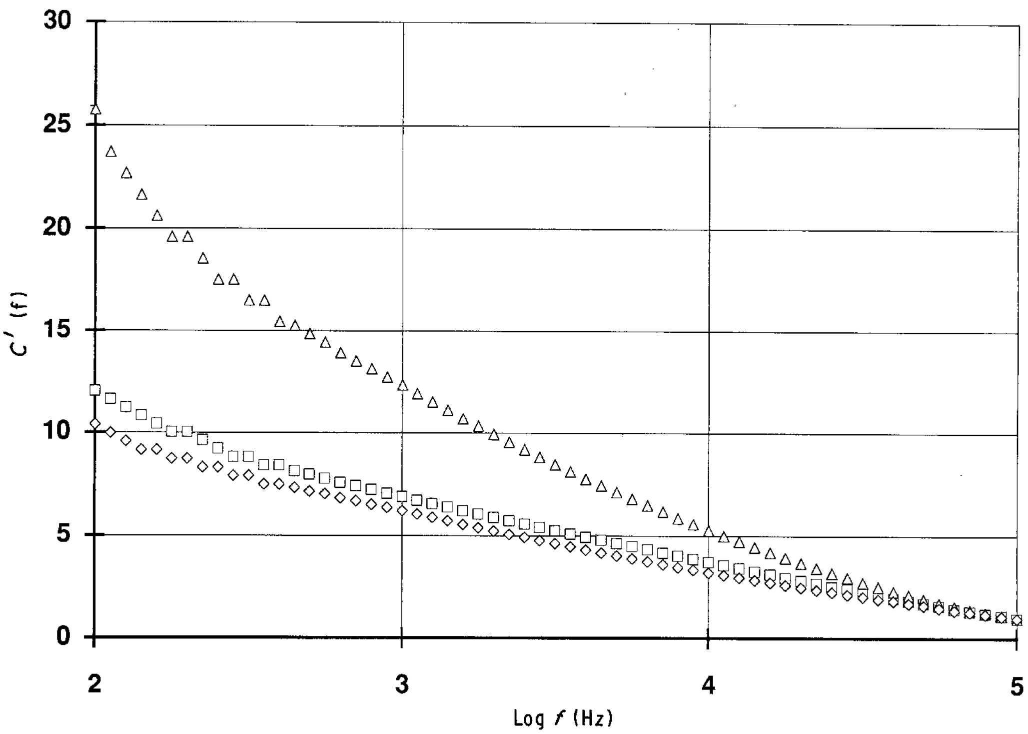

Figure 5 Normalized capacitance spectra for three varieties of apple with silver electrodes: (□) Granny Smith, (△) Rome, (◇) Red Delicious.

graphs the overall shape of the curves is similar for the three varieties, Rome apples have a significantly higher capacitance and conductance at low frequencies. Little difference is apparent between Granny Smith and Red Delicious apples. The same difference in Rome dielectric spectra was observed for each of the other three electrode types.

A series of measurements was conducted to determine whether there was evidence for thermal activation. A set of electrodes was inserted into an apple, which was then placed in a refrigerator. After sufficient time for electrode equilibration had passed, the apple was removed from the refrigerator and allowed to come to room temperature. The apple was then placed in a glass dish in a heated water bath. During this time dielectric spectra were taken at approximately 1 °C intervals between about 5 and 35 °C. In several cases spectra were recorded as the temperature was reduced back to about 5 °C to test for the presence of thermal hysteresis.

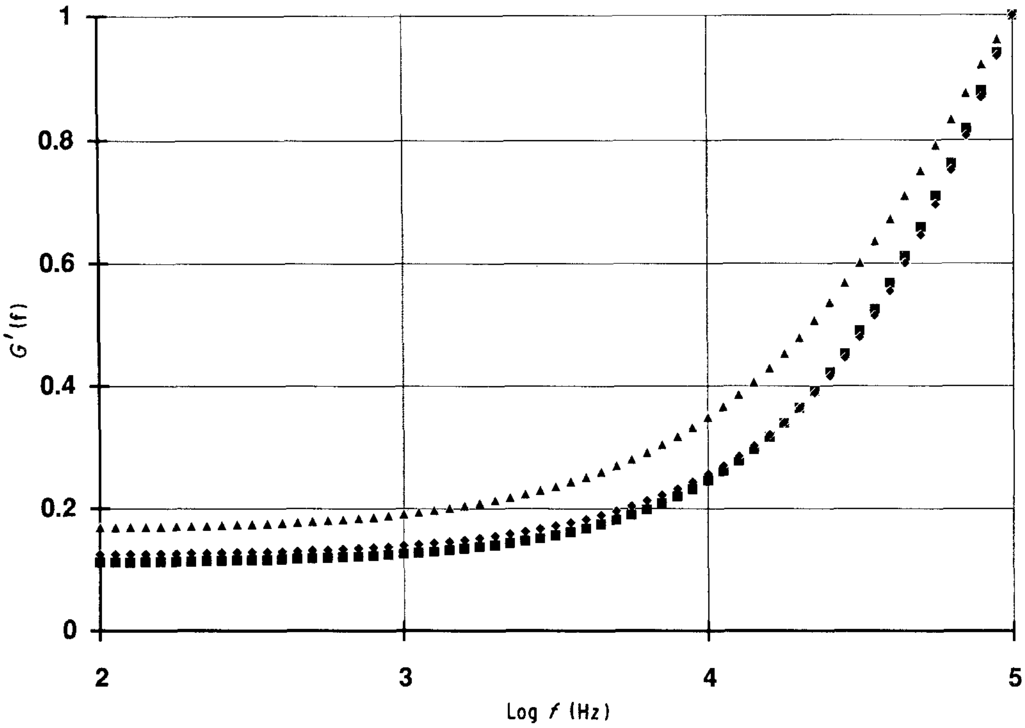

Figure 6 Normalized conductance spectra for three varieties of apple with silver electrodes: (□) Granny Smith, (△) Rome, (◇) Red Delicious.

The temperature of the apple interior could not be measured directly while dielectric spectra were taken because the presence of a probe within the apple interior would distort the spectra. Instead, similar electrodes were inserted into a second apple which was kept next to the first apple in the refrigerator and throughout the entire measuring process. A thermometer inserted into the core of the second apple provided the temperatures associated with the spectra recorded on the first apple. A check indicated that the core temperatures of the two apples agreed to within 0.1 °C.

Figs 7 and 8 illustrate, respectively, capacitance and conductance spectra for a Granny Smith apple with silver electrodes at several representative temperatures. Both the capacitance and conductance increase with temperature. Because the spectra obtained when the apple was cooled back to 4 °C are slightly higher

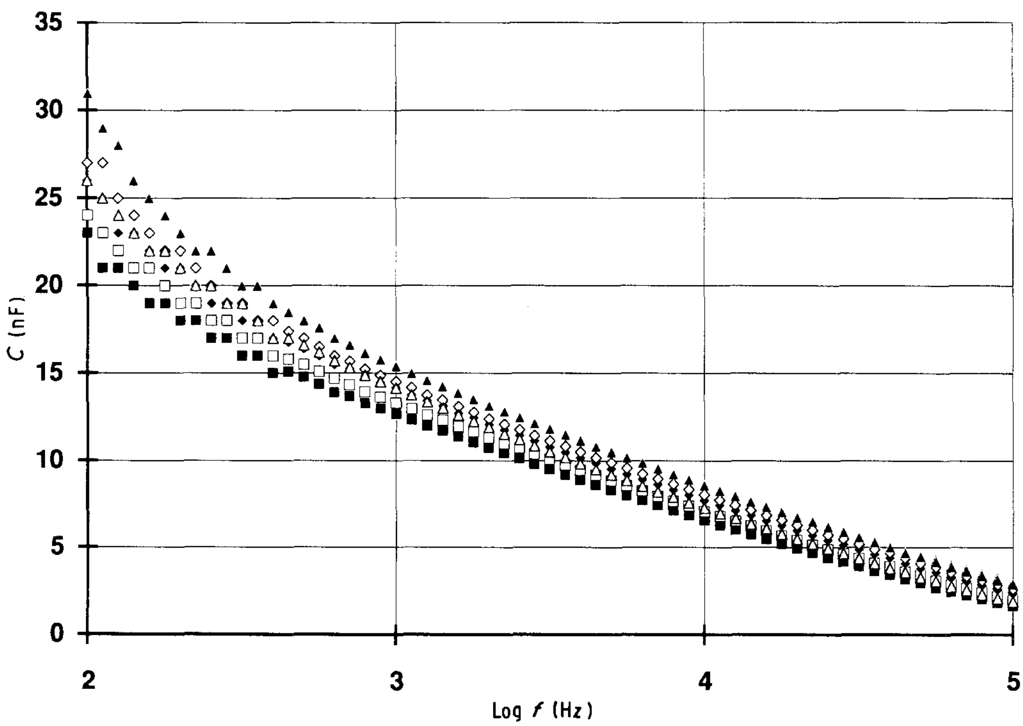

Figure 7 Capacitance spectra at several temperatures for Granny Smith apples with silver electrodes: (■) 5 °C, (□) 12 °C (◆) 19 °C, (◇) 26 °C, (▲) 34 °C, (△) 4 °C after second cooling.

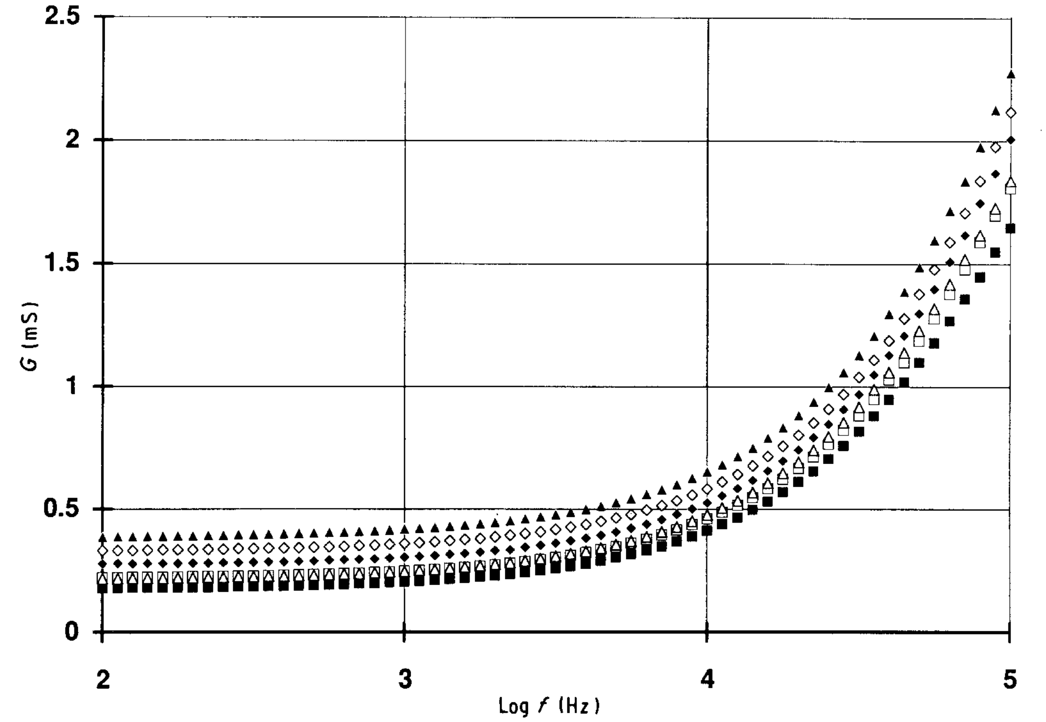

Figure 8 Conductance spectra at several temperatures for Granny Smith apples with silver electrodes: (■) 5 °C, (□) 12 °C, (◆) 19 °C, (◇) 26 °C, (▲) 34 °C, (△) 4 °C after second cooling.

than those taken originally at 5 °C, some thermal hysteresis is present. Evidently, slight changes were produced in the apple by the thermal cycling.

If the capacitance and conductance are thermally activated, their variation with absolute temperature, T, can be represented as

$$C(f, T) = C_0(f)\exp[-E_{ac}(f)/kT] \quad (5)$$

and

$$G(f, T) = G_0(f)\exp[-E_{ag}(f)/kT] \quad (6)$$

where k is Boltzman's constant. The activation energies for capacitance, E_{ac}, and conductance, E_{ag}, may vary with frequency. Figs 9 and 10 are Arrhenius plots for capacitance and conductance of a Granny Smith apple with silver electrodes for several frequencies. A linear slope, which is frequency dependent, is apparent in both figures.

Table I lists the activation energies for the three varieties of apple with silver electrodes at several frequencies. Table II shows the activation energies for Granny Smith apples with the four types of electrode

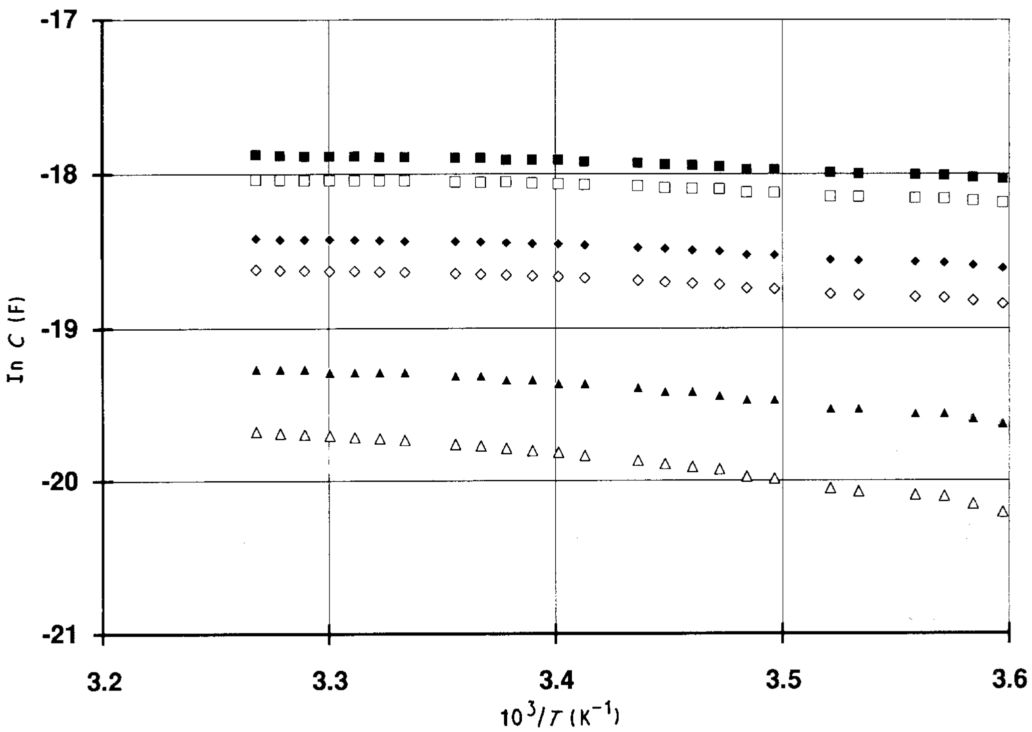

Figure 9 Arrhenius plots for capacitance of Granny Smith apples with silver electrodes at several frequencies: (■) 0.5 kHz, (□) 1 kHz, (◆) 5 kHz, (◇) 10 kHz, (▲) 50 kHz, (△) 100 kHz.

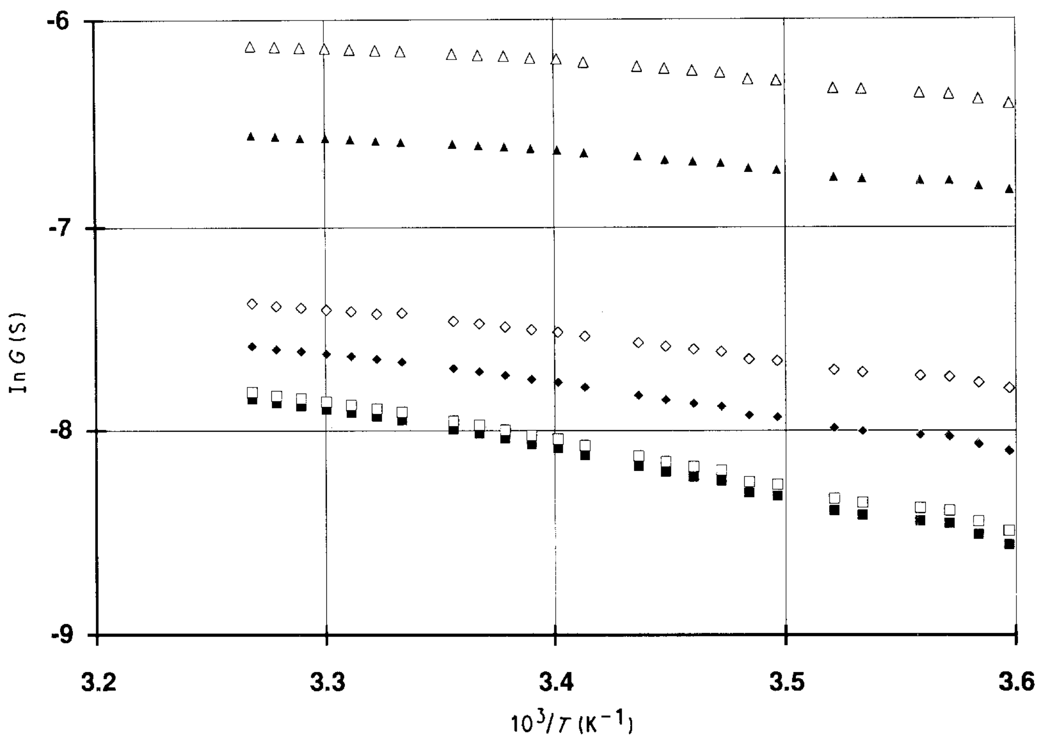

Figure 10 Arrhenius plots for conductance of Granny Smith apples with silver electrodes at several frequencies: (■) 0.5 kHz, (□) 1 kHz, (◆) 5 kHz, (◇) 10 kHz, (▲) 50 kHz, (△) 100 kHz.

TABLE I Activation energies with silver electrodes

f (kHz)	E_{ac}(eV)			E_{ag}(eV)		
	Granny Smith	Rome	Red Delicious	Granny Smith	Rome	Red Delicious
0.1	0.06	0.08	0.08	0.19	0.15	0.16
0.5	0.04	0.03	0.04	0.18	0.14	0.15
1	0.05	0.03	0.04	0.17	0.14	0.14
5	0.05	0.04	0.05	0.13	0.10	0.11
10	0.06	0.05	0.05	0.10	0.08	0.09
50	0.10	0.08	0.10	0.06	0.05	0.05
100	0.14	0.12	0.15	0.08	0.07	0.07

TABLE II Activation energies for Granny Smith apples

f (kHz)	E_{ac}(eV)				E_{ag}(eV)			
	Silver	Nickel-plate	Soft steel	Chrome-plate	Silver	Nickel-plate	Soft steel	Chrome-plate
0.1	0.06	0.12	0.06	0.11	0.19	0.19	0.20	0.20
0.5	0.04	0.05	0.04	0.04	0.18	0.19	0.19	0.19
1	0.05	0.05	0.04	0.05	0.17	0.18	0.18	0.18
5	0.05	0.07	0.06	0.06	0.13	0.13	0.13	0.13
10	0.06	0.07	0.07	0.07	0.10	0.10	0.11	0.10
50	0.10	0.13	0.12	0.13	0.06	0.08	0.08	0.08
100	0.14	0.16	0.16	0.17	0.08	0.10	0.09	0.10

at several frequencies. Similar results were obtained for the other combinations of apple variety and electrode type, but are not shown here for simplicity. For comparison, at room temperature $kT \approx 0.026$ eV. At any one frequency the activation energies for capacitance and conductance do not seem to depend on apple variety. The slightly higher values in the table for Granny Smith apples with silver electrodes did not consistently appear for the other electrode types. At any one frequency the activation energy for conductance was independent of electrode type for all three apple varieties. For frequencies up to 1 kHz the activation energy for capacitance did depend on the electrode for all three varieties. These results are consistent with Figs 3 and 4, which show that electrode effects appear only in the capacitance spectra below about 5 kHz.

In contrast, a consistent dependence of activation energy on frequency appeared for all combinations of apple variety and electrode type. The activation energy for conductance decreases with frequency up to about 50 kHz, where it becomes approximately constant. A more complicated behaviour is observed for capacitance. The initial decrease is presumably due to the transition from electrode–apple interface to bulk properties. The activation energy is independent of frequency between 0.5 and 10 kHz, but increases thereafter.

The conductance spectra may be represented in the form

$$G(f) = G_{d.c.} + G_{a.c.} \quad (7)$$

where $G_{d.c.}$ is the low-frequency (100 Hz) conductance. Fig. 11 plots $G_{a.c.}$ for the conductance spectra of Fig. 8. The linearity of this log–log plot indicates a power-law behaviour for $G_{a.c.}$: $G_{a.c.}(f) = Af^n$, where A is a constant. Least-square fits for frequencies above 1 kHz yielded n values which increased from 0.89 at $T = 5\,°C$ to 0.91 at 34 °C. For Rome and Red Delicious apples, n increased from 0.86 to 0.88. Exponents in this range are characteristic of hopping processes. It is not clear that the variations with temperature or apple variety are significant.

The dielectric properties of the apples can be expressed in terms of the real, K', and imaginary, K'', parts of the dielectric constant by using

$$K'(f) = C(f)d/\varepsilon_0 A \quad (8)$$

and

$$K''(f) = [G(f) - G_{d.c.}]/2\pi f \varepsilon_0 A \quad (9)$$

The electrode area can be approximated as the product of the needle length and the row width. All electrode types had the same dimensions so that $A \approx 8 \times 10^{-4}$ m^2. This approximation tends to overestimate A because the needles are not fully embedded to their entire length, but also to underestimate A because within the apple the current path will spread somewhat beyond the borders of the needle array. Fig. 12 presents the variation of K' and K'' with frequency for all three apple varieties with silver electrodes. The weak dispersion in K'' is consistent with the exponent $n \approx 0.9$ for $G_{a.c.}$. A power-law response is apparent for K' below about 10 kHz. For $K(f) = Bf^{-p}$, $p = 0.24$ for Granny Smith and Red Delicious apples and $p = 0.32$ for Rome. A break from linearity appears above 10 kHz.

To determine whether any features of the capacitance or conductance spectra could be associated with structural elements of the fruit, an activation energy series was carried out on a Granny Smith apple with silver electrodes, which was then placed in a freezer for several days. Upon removal, another activation energy series was performed as it warmed to room temperature. Figs 13 and 14 show, respectively, the capacitance and conductance spectra for the apple before and after freezing. The low-frequency capacitance was increased markedly and the high-frequency capacitance decreased slightly by freezing. A very large increase in conductivity occurred at all frequencies. Although not shown here, $G_{a.c.}$ continued to show power-law behaviour. The exponent n for this apple, however, was reduced from 0.90 to 0.29 by the freezing. The Arrhenius plots for capacitance were not as linear after freezing. The activation energies varied from 0.17–0.27 eV for $f \leq 1$ kHz, but were essentially 0 above this frequency. In contrast, the activation energies for conductance only varied between 0.14 and 0.16 eV for all frequencies. The Arrhenius plots were also more linear than those for capacitance. Freezing thus produced a significant increase in the activation energies for conduction at high frequencies. The changes in capacitance activation energies were more complex.

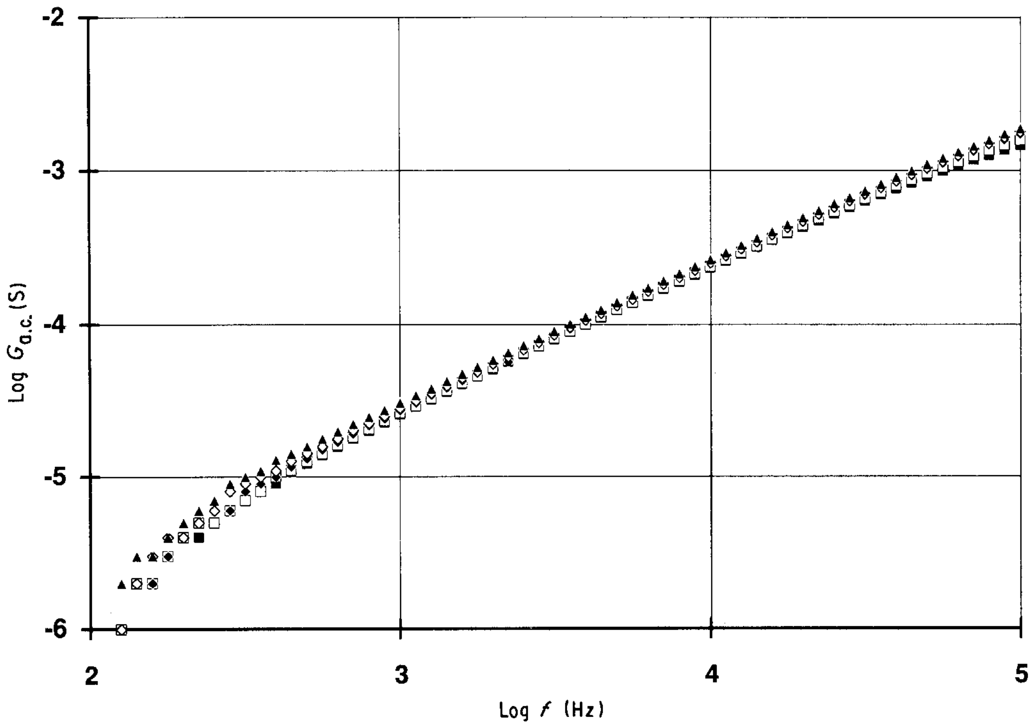

Figure 11 Variation of a.c. conductance with frequency for Granny Smith apples with silver electrodes at several temperatures: (■) 5 °C, (□) 12 °C, (◆) 19 °C, (◇) 26 °C, (▲) 34 °C.

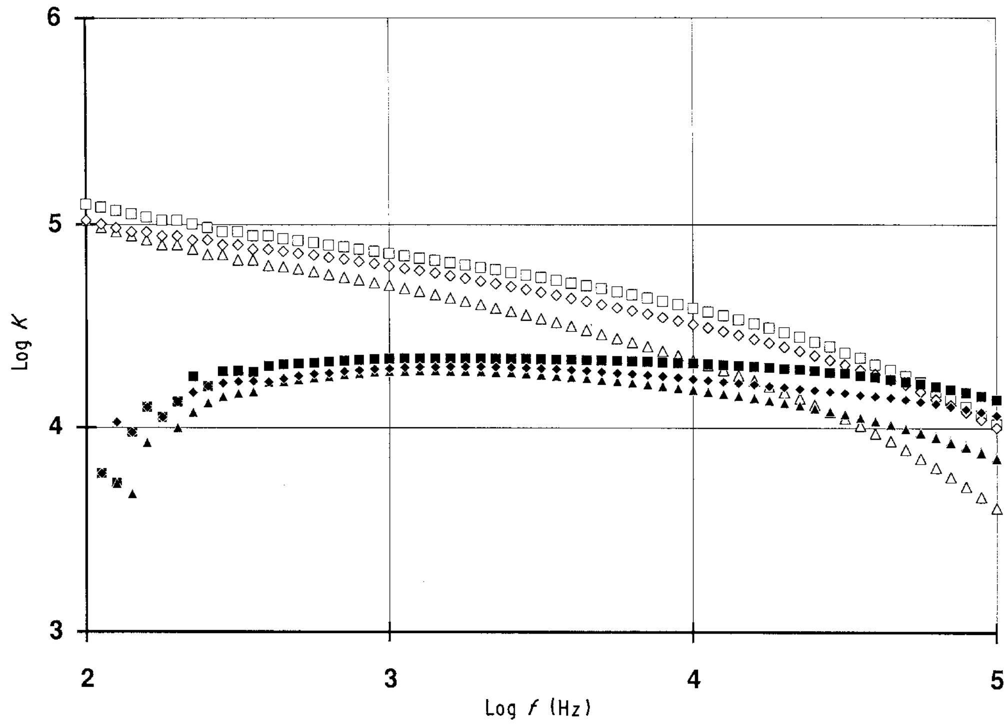

Figure 12 Variation of the real, K', and imaginary, K'', parts of the dielectric constant of several varieties of apple with silver electrodes. (□, △, ◇) K', (■, ▲, ◆) K''. (□, ■) Granny Smith, (△, ▲) Rome, (◇, ◆) Red Delicious.

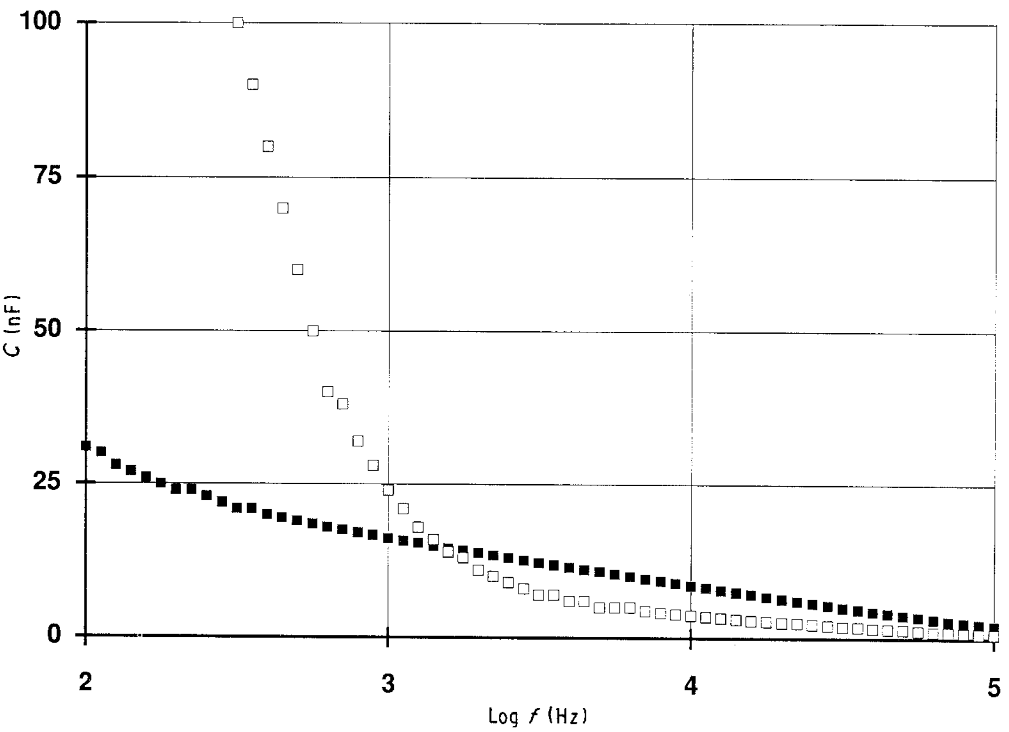

Figure 13 Capacitance spectra for Granny Smith apples with silver electrodes (■) before and (□) after freezing.

4. Discussion

The results shown in Figs 3 and 4 and in Table II indicate that, once equilibrium has been established, the contribution of the electrode–apple interface is relatively unimportant to the conductance spectra and plays a major role in the capacitance spectra only below about 1 kHz. It should be noted, however, that the time required for equilibrium to be established depends on both the variety of apple and the nature of the electrode material and must be determined before other spectra are taken. Similar considerations obviously must apply to measurements made on animal systems.

The use of flexible, surface electrodes was investigated in a pilot study. Aluminium foil electrodes with foam backing were pressed against the apple surface with a spring mechanism. Consistent contact was difficult to achieve, so that measurements made after

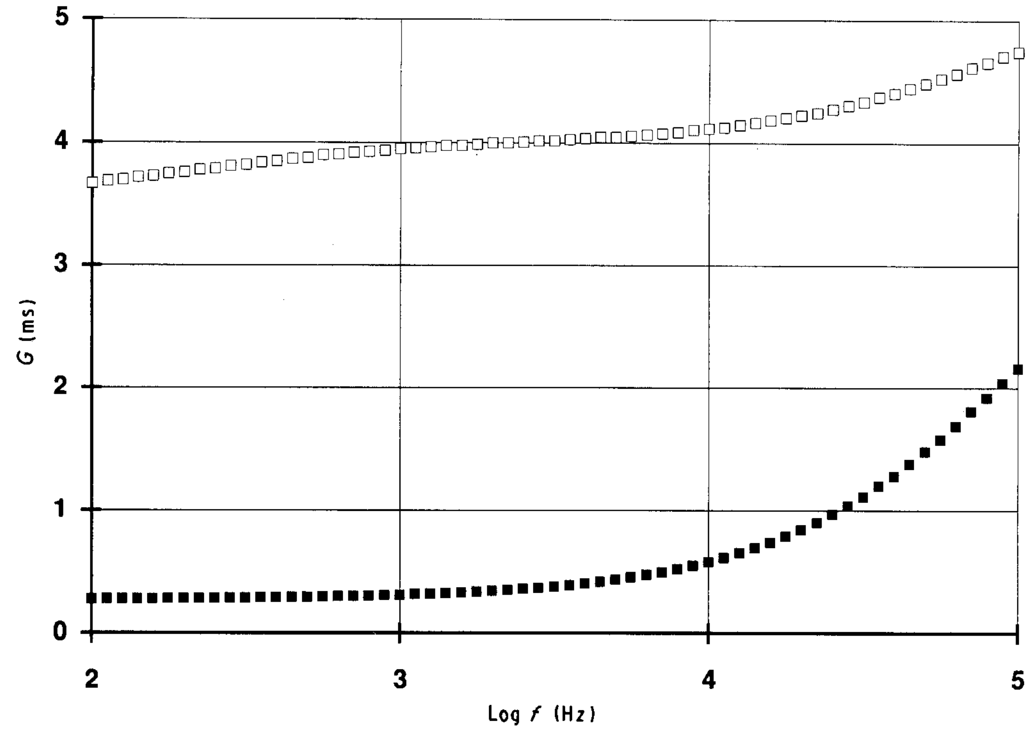

Figure 14 Conductance spectra for Granny Smith apples with silver electrodes (■) before and (□) after freezing.

the electrodes were removed and then reapplied on the same apple were not reproducible. Moreover, the dielectric properties were determined almost entirely by the skin and not the interior of the fruit.

The low-frequency capacitance and conductance of Rome apples appear to be relatively higher than those of Granny Smith and Red Delicious apples. Rome apples are softer in texture than the other two varieties. This difference in spectra is consistent with the very large increases in the low-frequency capacitance and conductance produced by freezing, during which the cell structure was severely damaged and the apple became very soft. Kato [13] has observed a similar large increase in conductance at all frequencies for spoiled and damaged apples. He also shows an increase in the low-frequency capacitance and a decrease in the high-frequency capacitance for damaged apples.

Future experiments will investigate the correlation between the dielectric and mechanical properties of fruit. If softening is associated with a preferential increase in low-frequency conductivity, then the ratio of high- to low-frequency conductivities might serve as an objective measure of fruit ripening. In addition, attempts will be made to determine whether differences in the internal structure of the fruit (e.g. peripheral cells compared with deeper cells) are detectable in the spectra. The experiments reported here used electrodes with a length of 2 cm and thus sampled primarily the deeper tissue.

The dielectric spectra presented here are similar to those of other biological materials. Power law behaviour is apparent in data reported on bone [22, 23] and muscle [24]. In plant systems, Hart has shown power-law responses at low frequencies for Coleus and Poinsettia stems [7]. Hill *et al.* [10–12] have demonstrated similar behaviour for a wide variety of plant leaves.

Previous work [8] with a four-electrode system at frequencies somewhat lower than those reported here also indicated a power-law (f^{-p}) response for apples. At frequencies below about 500 Hz the exponents for K' and K'' were found to be 1.3 and 1.0, respectively. In the present paper, the corresponding exponents are about 0.2–0.3 for K' and 0.1 for K'' at frequencies in the kilohertz region. Further work is planned in which the present system using the HP 4192A at high frequencies will be combined with a 12 bit ADC, four-electrode system for low frequencies to make measurements on a single apple over a very wide frequency range. Confirmation of a significant decrease in the exponents for K' and K'' near 1 kHz would be evidence for a strong low-frequency dispersion [25–27].

The low-frequency dielectric properties in living biomaterials are produced by ionic transport in the extra-cellular space. Only at frequencies on the order of 100 kHz will capacitive coupling across the cell membrane or wall begin to take place. Below 100 kHz living biological materials may be modelled as being composed of insulating inclusions in a conducting medium. Analogous physical systems exhibit dielectric responses similar to those in plant and animal tissues. Grosse has shown [28–30] that the very large low-frequency permittivity in such systems is produced by counterion polarization at the fluid–insulator interface. Cell membranes and walls, however, are not smooth. Polar groups extending into the extracellular fluid provide trapping sites at which counterions may be bound. The dielectric response of living biomaterials may consist of two parts: (1) steady-state (d.c.) diffusion of ions in the extracellular space, and (2) hopping of counterions along the disordered wall or membrane surface. The fractal nature of such diffusion along a disordered interface has been discussed by Dissado and Hill [31] and by Niklasson [32].

The activation energies reported here may represent trap depths for counterions hopping along the exterior of the cell wall. High frequencies correspond to shorter diffusion times and distances. The increase in conduction activation energy with decreasing frequency may be due to the higher probability of an ion encountering a deeper trap as the diffusion time increases. Freezing living biological materials produces severe damage to cell membranes and walls. Cell contents leak into the extracellular fluid and increase greatly its conductivity as is apparent in Fig. 14. The damaged walls provide deeper traps for the counterions and thereby increase the high-frequency activation energy for conductance.

The increase in activation energy for capacitance, which does not appear until about 50 kHz, may be related to the onset of capacitive coupling across the cell wall. The apparent disappearance of the high-frequency capacitive activation energy and the decrease in the high-frequency capacitance itself after freezing are consistent with this hypothesis. For comparison, Stout [16] has reported an activation energy on the order of 0.2 eV for the resistance and reactance of plant stems. No clear frequency dependence was apparent.

5. Conclusion

The results reported here indicate that the dielectric properties of apples, and presumably those of other biogenic materials, can be understood in terms of principles established for inorganic materials. Conduction and polarization processes involve the activated hopping of counterions along cell walls or membranes superimposed on steady-state charge transfer in the extracellular spaces.

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