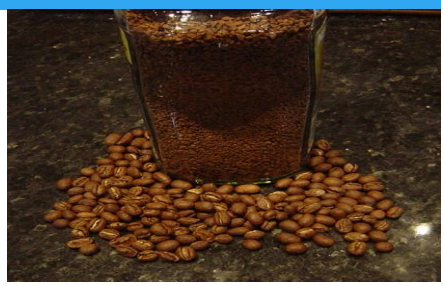
Lacerta Technology Ltd.

DETA Dielectric Thermal Analyser



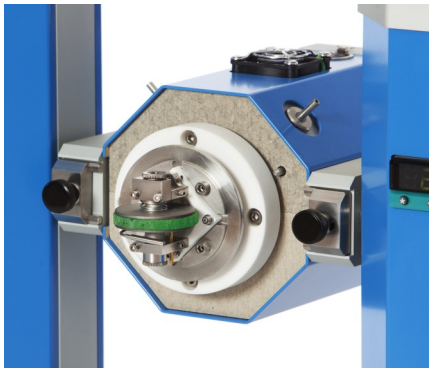
Application Note
Dielectric Thermal Analysis of Instant Coffee Powder

This application note will demonstrate the ability of the DS6000 DETA to investigate powder formulations of coffee. Both granulated instant coffee and granulated filter coffee can be analysed by DETA successfully. Examples of data in multi-frequency mode and down to -150ºC are shown.
As this material was powdered, the cup and plate arrangement was utilised for convenience although with care, simple parallel plates could also be used.

Introduction

Dielectric **Thermal** Analysis (DETA) is a powerful analytical tool for studying relaxation processes in materials or behaviour of polar species within a material. The glass transition (Tg) is a key process in any material, and can be observed with ease by DETA for many materials. This technique provides very revealing information about these relaxations through the tan δ vs temperature data. The form of the material can be anything from a thin film, sheet material, powder or a liquid.

Dielectric measurements are the electrical analogue of dynamic mechanical measurements. The mechanical stress is replaced by an alternating voltage across the sample (a.c.field) and the alternating strain becomes the stored charge (Q) in the sample. The sample in effect behaves as a simple capacitor. Q is always measured as its derivative dQ/dt = a.c.current.



The dielectric data is obtained from phase and amplitude measurements of current and voltage to resolve the components e* = Capacitance with sample/Capacitance with an identical air gap.

As in DMA, tan δ is the ratio of the loss factor(e″) to the storage component (e′ , dielectric constant or permitivity) . Tan δ is plotted against temperature and glass transition is normally observed as a peak since the material will absorb energy as it passes through the glass transition. The size of this peak quantifies the amount of amorphous material present in the sample.

Equipment

DS6000 DETA
1L 'Mini' Cryo

Experimental Conditions

Samples:	Instant Coffee powder and Filter Coffee
Geometry:	Cup and Plate 33mm diameter, thickness(gap) between 0.1 and 0.7mm
Frequency	1 and 10 kHz as indicated

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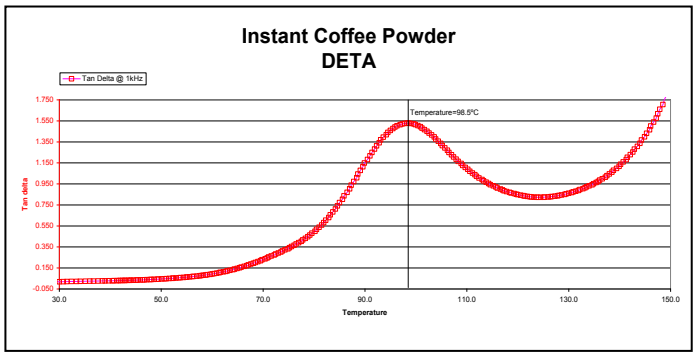
Experimental

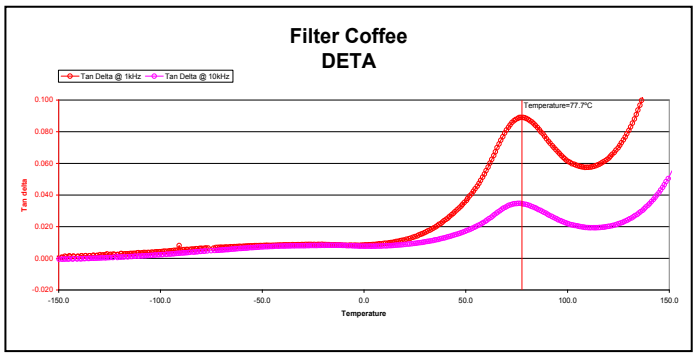
The granules in both cases were milled to a fine powder in a pestle and mortar immediately prior to analysis. Approximately 100mg was placed in the cup and spread evenly prior to compressing gently with the top plate. This was secured in place and the samples were cooled using the 1 litre 'Mini' cryo to the appropriate start temperature. The experiment was then started using the conditions indicated.

Results and Conclusion

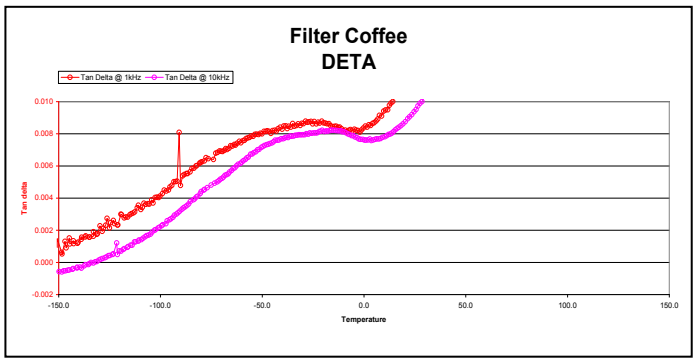
The first figure on this sheet shows a single frequency scan of instant coffee. This appears to be an extremely clean single relaxation. When the same material was run in a DMA powder holder, this peak appears less clean and it was thought that there may be several relaxation processes overlapping. The DETA data appears to suggest this is unlikely.



The second figure is the result of a multi-frequency run down to -150ºC. This shows a process at a slightly lower temperature than that observed in the instant coffee. It almost certainly is the same component but because filter coffee comprises of a very large insoluble fibrous base, the relaxation is very slightly plasticized by these components. Also, note the tand peak heights. In the filter coffee it is less than 10% compared to the instant coffee at 1 Hz. Note that this process appears not to be frequency dependant. However, with the issue of the fibrous matrix and proximity to the conductive region, the behaviour could appear unusual.



The third figure is the same as figure 2 with The tand expanded to reveal the small frequency dependant relaxations at low temperature. These correspond to data produced in a DMA reasonably well.



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