

Charge Transport in Thermally Aged Paper Impregnated with Natural Ester Oil

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ABSTRACT

Samples of a dried composite oil-paper system were thermally aged in the laboratory under N_2 in a sealed container and analysed using their dielectric response. The contributing loss processes were separated using an equivalent circuit model containing constant high frequency capacitances, dc-conductances, a dispersive capacitance, and a low frequency quasi-dc (q-dc) dispersion process as circuit elements. This low frequency dispersion in oil-paper samples is a bulk property of the system that is often mistaken for a dc conduction process when only the imaginary capacitance or ac-conductance is measured. The Dissado-Hill response function is proposed for the frequency dependence of the dispersive capacitance in the mid-frequency range rather than the Debye function because its cluster concepts correlate with the sample morphology of a matrix of oil-filled cavities. The quasi-dc dispersion is assigned to the transport of mobile charges between charge-clusters associated with locally connected oil filled cavities, which is a process that can be represented on a global scale by the form of hierarchical circuit system that leads to constant phase angle responses, and is here modelled by the Dissado-Hill q-dc function. It was found that the two dispersions moved together to higher frequency with thermal ageing of the composite material. The characteristic frequency common to the dispersions had an activation energy that decreased with thermal ageing, whereas their amplitude increased. This behaviour is discussed in terms of possible changes to the oil-paper composite caused by the ageing process.

Index Terms — Oil impregnated paper insulation, dielectric measurements, equivalent circuits, charge transport, insulation ageing

1. INTRODUCTION

THE ageing of cellulose paper insulation in power transformers is a major factor in determining their life [1]. Cellulose is an organic polymeric material whose molecule is made of glucose rings that are linked to each other [2] as shown in Figure 1. It provides both electrical and mechanical support in transformers. Insulation paper possesses randomly distributed cavities of a range of sizes between its fibres. Impregnation of the paper with oil improves its electrical insulation strength by filling the cavities and preventing partial discharges. However

“Clusters” of charged particles may accumulate in the oil-filled cavities which may displace through randomly distributed connections in the amorphous region forming a matrix of clusters of connected cavities [3]. The distribution and connectivity within and between such clusters in the paper dominates the effective dielectric properties of the composite material and influence other properties such as electrical and mechanical strength.

Thermal stress, oxidation, and hydrolysis of the paper in service may lead to mechanical and electrical degradation of the oil-paper insulation and hence a reduction of its life

time. During this process the matrix of clusters of oil-filled cavities can be expected to alter. Therefore measurements of the dielectric response, i.e. of the permittivity and dielectric loss as a function of frequency and temperature, provide information on the ageing status of the oil-paper insulation system. From such measurements an equivalent circuit model may be derived that leads to a better understanding of the physical state of the material.

The clusters of connected oil-filled cavities contain dipolar entities that will yield a dielectric response, but can also be considered as acting as dipoles formed by bound positive and negative charges. These can be expected to contribute a dielectric dispersion to the response with a loss peak at a frequency associated with the relaxation time of the charge separation in the cluster of cavities. The amplitude will be related to the amount of charge separation in each cluster and the concentration of clusters. The clusters will also be sources for charge carriers that may move through the oil-paper material giving a low frequency contribution to the dielectric response. The cellulose paper and oil will also contribute a parallel capacitance at the high end of the frequency window. The analysis of the dielectric response carried out here therefore yields information on charge separation across clusters of connected cavities and its transport through the oil-paper material. Through the changes brought about by thermal ageing, it gives an indication of the way that the ageing has modified the material, and may be useful in diagnosing the extent of ageing itself. In this work, the dielectric response of thermally aged oil-paper samples has been examined to determine what information may be obtained about the nature of the ageing process and to assess the potential of the various response parameters as indicators for ageing diagnostics.

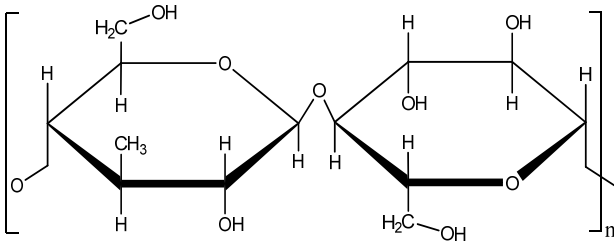


Figure 1. Cellulose structure taken from [2].

2. DIELECTRIC RESPONSE MODELS OF OIL-PAPER INSULATION

Other works have proposed a number of equivalent circuit models to represent the dielectric response of oil-paper insulation systems [4]. The models have been based on an extended Debye approach and have consisted of a parallel arrangement of branches each containing a series connection of a resistor and a capacitor [5] which behaves as an exponentially relaxing dipole in an electric field. The Debye response is the response of non-interacting dipoles to an applied alternating electric field and is equivalent to a parallel arrangement of identical resistor-capacitor series circuits. However, the dipoles or charges in the cavity –

cluster cannot in general be treated as non-interacting. The relaxation of a dipole or ion-pair within a cavity or cluster may affect the neighbouring dipoles/ions through (i) electrostatic forces, (ii) electro-mechanical forces arising from changes in the cavity shape and size, and/or (iii) the transport of charges between clusters at long times. This suggests that the extended Debye approach may not offer the best interpretation for the behaviour of the oil-paper system. We have therefore invoked the cluster model of relaxation [6] to describe the dielectric response of the oil-paper system at intermediate frequencies in our frequency window and we have used quasi-dc conduction [7] to describe the low frequency dispersion.

The expression given by the cluster model for the dielectric susceptibility of a system of relaxing dipoles is determined by the correlation between dipole motions within a cluster of dipoles and the correlation between dipole motions in different clusters, and is given by [6]:

$$\chi^*(\omega) = \chi_0 \left[\frac{\Gamma(1-n_1)}{m} \right] F(\omega/\omega_p) \quad (1)$$

and

$$F(\omega/\omega_p) = \left[\frac{\Gamma(1+m-n_1)}{\Gamma(2-n_1)\Gamma(m)} \right] \left[\omega_p/(\omega_p + i\omega) \right]^{1-n_1} \times {}_2F_1(1-n_1, 1-m; 2-n_1; \omega_p/(\omega_p + i\omega)) \quad (2)$$

Here $\Gamma(-)$ is the gamma function and ${}_2F_1(-, -; -; -)$ is the Gaussian hypergeometric function [8]. The amplitude factor χ_0 is defined by the static susceptibility, i.e. the dipole concentration of the dielectric dispersion. The loss peak frequency is ω_p , and the parameters n_1 and m lie in the range between zero and unity such that:

$$\chi''(\omega) \propto \chi'(\omega) \propto \omega^{n_1-1} \quad \text{for} \quad \omega > \omega_p \quad (3)$$

$$\chi''(\omega) \propto \chi'(0) - \chi'(\omega) \propto \omega^m \quad \text{for} \quad \omega < \omega_p \quad (4)$$

There also exists a different form of response variously termed quasi-dc (q-dc) conduction [7] or Low Frequency Dispersion (LFD) [9]. This type of response was termed anomalous low frequency dispersion (LFD) by Jonscher [9] since it commonly appears as the lowest frequency dispersion process observed, however the theoretical q-dc mechanism of Dissado and Hill [7] is not restricted to such low frequency for its operation [10] as it depends upon the speed of the charge carriers on restricted paths. The q-dc mechanism exhibits a characteristic (or threshold) frequency ω_c that defines the cross-over between a high frequency range where charge carriers are bound together as dipoles giving only a weak dispersion, and a low frequency range where the charge carriers become free to move along the restricted paths. The theoretical q-dc mechanism [7] gives the expression for the frequency dependent susceptibility, $\chi^*(\omega) = \varepsilon^*(\omega) - \varepsilon_0$, as:

$$\chi^*(\omega) = \chi_0 \left[\frac{\Gamma(1-p-n_2)}{\Gamma(1-p)\Gamma(1-n_2)} \right] F(\omega/\omega_c) \quad (5)$$

where

$$F(\omega/\omega_c) = \left[\frac{\omega_c}{(\omega_c + i\omega)} \right]^{1-n_2} \times {}_2F_1(1-n_2, 1+p; 2-n_2; \omega_c/(\omega_c + i\omega)) \quad (6)$$

The amplitude factor χ_0 is defined by the dipole concentration at the time, $(\omega_c)^{-1}$, when the charge carriers of opposite sign just start to move independently, i.e. at the onset of the hierarchical transport process. The values of the exponents in this form of response are typically found to be in the range $0.5 < p < 1$ and $0.5 < n_2 < 1$, and it has been experimentally verified [11] to be a bulk response of the material rather than a dispersive electrode-material interface response [12] with which it is sometimes confused. The asymptotic behaviour of equation (5) for low and high frequency (relative to ω_c) is shown in equations (7) and (8) [7]:

$$\varepsilon'(\omega) \propto \varepsilon''(\omega) \propto \omega^{n_2-1} \quad \text{for } \omega > \omega_c \quad (7)$$

$$\varepsilon'(\omega) \propto \varepsilon''(\omega) \propto \omega^{-p} \quad \text{for } \omega < \omega_c \quad (8)$$

The q-dc response has specific features that distinguish it from DC conductivity even though in this form of response, χ'' has a similar frequency dependence, at low frequencies, to that which would be given by a DC-conductivity. In the q-dc response, polarization that is the result of charge carriers moving on restricted and partly-blocked paths in condensed matter leads to a constant phase angle form of dielectric response in which $\varepsilon''(\omega) \propto \varepsilon'(\omega) \propto \omega^{-p}$ where $0.5 < p < 1$ and p may approach arbitrarily close to unity [7]. When p is very close to unity the q-dc form of response dominates at low frequencies and can be easily confused with a DC conductance if $\varepsilon''(\omega)$ (or ac conductivity $\sigma(\omega) = \omega\varepsilon''(\omega)$) is the only component of the complex permittivity that is measured, since an ideal DC conduction in condensed matter gives a complex relative permittivity,

$$\varepsilon_r^*(\omega) = \frac{\varepsilon^*(\omega)}{\varepsilon_0}, \text{ which can be written as:}$$

$$\varepsilon_r^*(\omega) = \varepsilon_r'(\omega) - i \left[\varepsilon_r''(\omega) + \frac{\sigma_0}{\varepsilon_0\omega} \right] \quad (9)$$

The DC conductivity term in this expression contributes an exact inverse frequency response to $\varepsilon''(\omega)$ at low frequencies. The observation of an imaginary part of the permittivity (ε'') with an apparent slope of -1 in a $\log\{\varepsilon''(f)\}$ versus $\log\{f\}$ plot is usually taken as evidence for the existence of a dominating DC conductivity. Whilst a true DC conductance has real permittivity that is independent of frequency, the q-dc process exhibits the same frequency power law dependence in the real

permittivity as in the imaginary permittivity; it is this feature that distinguishes it from a true dc-conductance. The presence of a q-dc process can therefore be identified when $\varepsilon'(\omega)$ is measured and shown to follow the same frequency dependence as $\varepsilon''(\omega)$ [7].

The equation of motion for the response function corresponding to this q-dc susceptibility (equation (5)) reveals that when p is exactly unity, the response decomposes into a loss peak processes with relaxation frequency ω_c corresponding to bound negative and positive charges together with a DC conductivity [13]. This illustrates the nature of q-dc conduction (LFD) as a response due to negative and positive charges bound together at high frequencies that become separated independent carriers at frequencies $\omega < \omega_c$. In the limit of an ideal DC conductivity the charges are separated into two independent populations, one of free carriers and one of bound charge pairs, at all frequencies. It can be seen that the q-dc form of response would be expected if the ions trapped in the oil-filled cavities of the impregnated paper insulation are capable of becoming free to transfer from cavity to cavity through the material at long times (i.e. low frequencies), along specific routes on the paper fibres. For this reason, expressions (1) and (5) are used to define the frequency dependence of the complex susceptibility of the various circuit elements that are required to fit the experimental data where n_1 , n_2 , m and p are fractional and positive. The fitted circuit components are to be regarded as contributing to the capacitance through the relevant amplitude factor χ_0 multiplied by a geometrical factor (area/thickness when the circuit element is the whole sample) appropriate to the particular component.

3. EXPERIMENTAL DETAILS

In this study we used palm oil as an impregnant for insulating paper and the samples were made as follows. Four hundred ml of un-branched Palm Kernel Oil Methyl Ester (PKOAE1) [14] was degassed and dried in an oven at 85°C for 2 hours. It was allowed to cool to ambient temperature and transferred to the ageing vessel. The water content left in the oil after this treatment was measured by coulometric Karl Fischer titration and was 201mg/kg. The acidity of the oil was measured using colour indication titration and is expressed in milligrams of potassium hydroxide required to neutralize the sample. Its value was 0.34mg KOH/g. Thermally upgraded paper was vacuum dried in an oven at 85°C for 24 hours. The ageing vessel containing the oil sample, 33 g of 0.255 mm thermally upgraded paper, 0.0150 m² galvanized steel strip, 0.01128 m² copper strip, and a 0.0150 m² aluminium strip, was placed in a vacuum oven for 30 minutes at ambient temperature. The galvanized steel represents the core of a transformer, while the copper and aluminium strip represent the conducting coils. The vessel was sealed with a lid at ambient temperature and placed in the ageing oven. The air in the vessel was purged out with dry nitrogen and the pressure in the vessel was increased to 600 kPa. The pressure was maintained at 600 kPa (6 bar) throughout the

ageing period. This pressure was chosen because the saturated vapour pressure of water at 150°C is 450 kPa and so the nitrogen pressure prevents evaporation of any residual moisture in the oil, forming water on the lid and getting back into the oil and paper as water droplets. The ageing was carried out at 150°C for 3 months. Samples of oil and paper were taken out of the vessel after 28 days, 56 days, and 84 days respectively for dielectric response measurement.

The frequency response of the ester-paper samples was measured using a three electrode solid test cell (Keithley model 6105) whose electrodes have a diameter of 50 mm. The test cell was placed in a temperature-controlled oven and was connected to a Solartron 1296 Dielectric Interface. The dielectric interface was connected to a Solartron 1255 Frequency Response Analyzer and controlled by a desktop computer in order to measure the dielectric response of the sample. The oven also provided a noise-suppressed environment for dielectric measurements because it is a grounded metal box. Frequency domain measurements were carried out using a 2 V rms sinusoidal excitation signal. The complex impedance of the sample under test was measured over the frequency range 10^{-3} - 10^6 Hz and at temperatures ranging from 20 to $50 \pm 1^\circ\text{C}$ at intervals of 10°C .

4. RESULTS

A master plot combining the dielectric responses at different temperatures has been made for each of the ester-paper samples by translating the individual responses along the $\log(\text{frequency})$ axis (scaling the characteristic frequency to a common value) and along the $\log(\text{capacitance})$ axis (scaling the response amplitude to a common value), Figures 2-5. In order to identify a possible power law behaviour (equations (3)-(4) and equations (7)-(8)) in the experimental data, it is necessary to plot the real and imaginary part of the complex capacitance on the same log-log graph axes. This follows from Kramers-Kronig relationship between the real and imaginary part of the complex susceptibility. The notation here is that EIP stands for Ester Impregnated Paper and XXD for the XX days of thermal ageing. Three different contributions to the response can be identified in these master plots. There is a frequency independent real component of the permittivity at the high frequency end of our frequency window. This response includes dielectric relaxations taking place at frequencies higher than 10^4 Hz, together with atomic and electronic polarizations from quantum resonances (which contribute an amount equal to the square of the refractive index to the relative permittivity). At slightly lower frequencies there is a dispersion in the capacitance $C'(\omega)$ associated with a loss peak that becomes more resolved as ageing progresses. At lower frequencies a constant phase angle response is observed with both $C'(\omega)$ and $C''(\omega)$ proportional to ω^p where p is close to unity. This corresponds to the q-dc response in the frequency range $\omega < \omega_c$, equation (8). The identification of a fractional power law dependence is not so clear for $C'(\omega)$ in the unaged ester-paper and an alternative behaviour such as a dispersive electrode-interface capacitance [12] may be

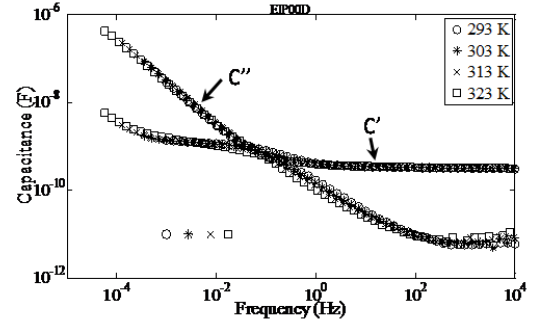


Figure 2. Dielectric Response of Unaged Ester-paper. The scales are correct at $T = 20^\circ\text{C}$ and the isolated points describe the translation locus necessary to bring the four sets of data into coincidence.

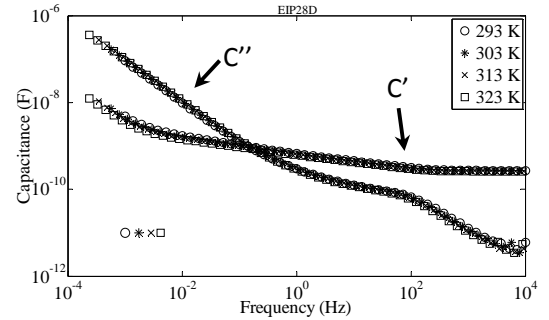


Figure 3. Dielectric Response of Ester-paper after 28 days. The scales are correct at $T = 20^\circ\text{C}$ and the isolated points describe the translation locus necessary to bring the four sets of data into coincidence.

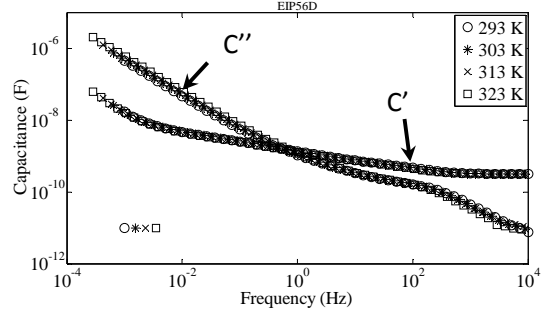


Figure 4. Dielectric Response of Ester-paper after 56 days. The scales are correct at $T = 20^\circ\text{C}$ and the isolated points describe the translation locus necessary to bring the four sets of data into coincidence.

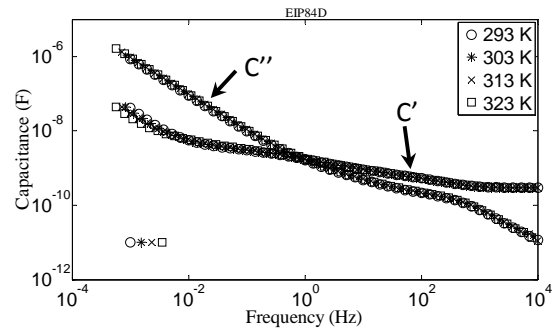


Figure 5. Dielectric Response of Ester-paper after 84 days. The scales are correct at $T = 20^\circ\text{C}$ and the isolated points describe the translation locus necessary to bring the four sets of data into coincidence.

responsible, however the q-dc identification becomes very clear in the case of the aged samples and for this reason we

have assumed that it applies to the unaged sample as well as the aged ones albeit reaching dominance at lower frequency.

The equivalent circuit description that fits these responses is divided into two rows of parallel circuit elements, with the rows in series with one another, see Figure 6. The rows are denoted by the subscripts 1 and 2 and each are comprised of a dc-conductance (G_1 and G_2), a constant high frequency capacitance ($C_{1\infty}$ and $C_{2\infty}$), and a frequency dependent response element ($C_1^*(\omega)$ and $C_2^*(\omega)$). Here $C_1^*(\omega)$ is the Dissado-Hill loss peak response function (equation (1)) with its capacitance contribution ($C_1(0)$) denoted by C_{10} given by χ_0 (of equation(1) multiplied by a geometrical factor appropriate to its area and thickness). Similarly $C_2^*(\omega)$ stands for a q-dc response (equation (5)) [6,11,15], with a capacitive amplitude factor denoted by C_{20} given by the relevant χ_0 in equation (5) multiplied by its appropriate geometrical factor. The equivalent complex capacitance ($C_{tr}^*(\omega)$) of the top row (row 1) of the circuit is:

$$C_{tr}^*(\omega) = C_{1\infty} + C_1^*(\omega) - iG_1/\omega \quad (10)$$

That of the bottom row (row 2) is $C_{br}(\omega)$:

$$C_{br}^*(\omega) = C_{2\infty} + C_2^*(\omega) - iG_2/\omega \quad (11)$$

And the total complex capacitance, $C_t^*(\omega)$ is given by:

$$[C_t^*(\omega)]^{-1} = [C_{tr}^*(\omega)]^{-1} + [C_{br}^*(\omega)]^{-1} \quad (12)$$

These equations are fitted to the data using a non-linear least squares technique to find the optimum combination of parameters. The frequency dependence of $C_1^*(\omega) = C_1(\omega)' - iC_1(\omega)''$ and $C_2^*(\omega) = C_2(\omega)' - iC_2(\omega)''$ is given by equations (2) and (6) respectively.

An example of the fit to a specific set of data is shown in Figure 7 and the fit parameters of each sample are given in Tables 1 and 2 for a temperature of 20°C.

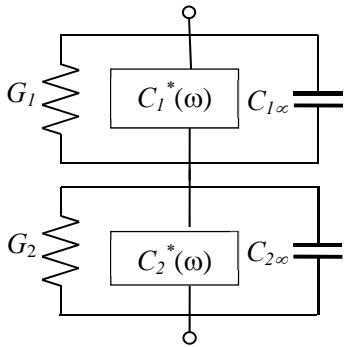


Figure 6. Equivalent circuit model of dielectric response of Ester-paper system. $C_{1\infty}$ and $C_{2\infty}$ are frequency independent capacitances that combine to give the high frequency 'infinite' frequency capacitance of observed response; G_1 and G_2 are dc conductances. $C_1^*(\omega)$ is a loss peak dispersion), and $C_2^*(\omega)$ is a quasi-DC contribution, whose frequency dependences are given by equation (1) and equation (5) respectively.

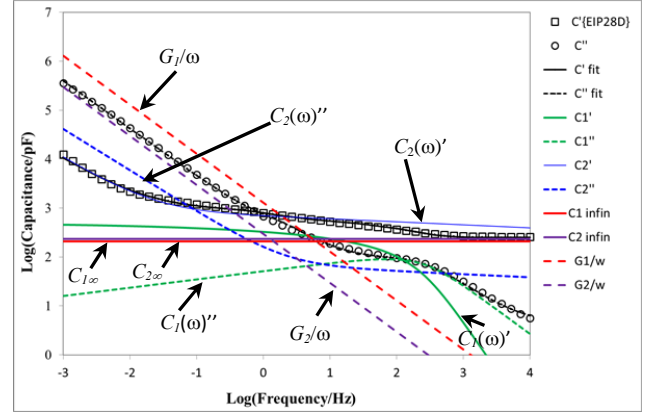


Figure 7. Equivalent Circuit Fit of 28 Days Aged Ester-paper Dielectric Response at 50°C. Experimental data: real (square markers) and imaginary (circle markers) parts of measured capacitance; Fitted data: real (solid line) and imaginary (dashed line). Frequency dependent circuit elements have real components indicated by a prime and imaginary components by a double prime.

Because a master plot can be formed, the shape parameters, n_1 , m , n_2 , p , will be the same at all temperatures and only the characteristic frequencies and response amplitudes will change. The contribution of the various circuit elements to the overall response are separately identified in figure 7. It can be noticed that at low frequencies the frequency dependence of the response is determined by that of the Q-DC process (i.e. equation (8) with p given in Table 1). The two dc-conductance circuit elements are electrically in series and their contribution to the overall response is insufficient to dominate the frequency dependence in the frequency range of the measurements.

The mid-frequency loss peaks ($C_1(\omega)$) revealed by subtracting $C_{1\infty}$, $C_{2\infty}$, G_1 , G_2 , and the q-dc response ($C_2(\omega)$) from the data are given in Figure 8 for clarity.

Table 1. Fit parameters for the dispersion processes in Aged alkyl ester-paper (EIP) at 20°C

Ageing Period (days)	DH1 Loss Peak parameters				DH2 q-dc parameters			
	n_1	m	ω_p (Hz)	C_{10} (pF)	n_2	p	ω_c (Hz)	C_{20} (pF)
0	0.4	0.77	0.5	200	0.9	0.8	0.054	640
28	0	0.18	96.5	520	0.94	0.8	0.988	810
56	0	0.30	49	2100	0.9	0.65	249	150
84	0	0.12	162	3800	0.9	0.81	126	450

Table 2. Fit parameters for G_1 , G_2 , $C_{1\infty}$, and $C_{2\infty}$ at 20°C

Ageing Period (days)	G_1 (pS)	$C_{1\infty}$ (pF)	G_2 (pS)	$C_{2\infty}$ (pF)
0	720	270	140	530
28	2000	220	390	280
56	1800	260	2500	280
84	4600	250	6100	240

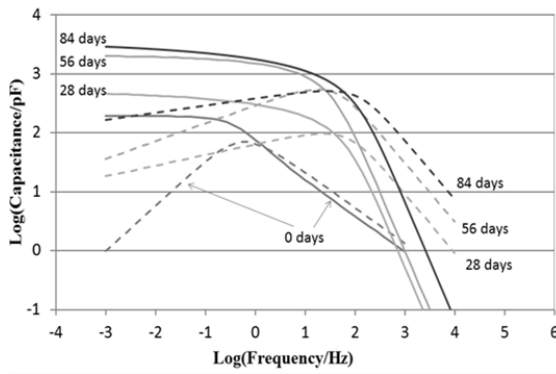


Figure 8. Loss peaks extracted from the dielectric response data of Figures 3-5 by subtracting the high frequency and the q-dc response at 20°C. The low frequency slopes are only approximate as the loss in this region is strongly influenced by the q-dc response. Real capacitance (solid lines), imaginary capacitance (dashed lines)

5. DISCUSSION

5.1 IONIC NATURE OF THE LOSS PEAK DISPERSION PROCESSES

PKOAE1 has a relative permittivity of ~ 2 in the frequency range $1\text{-}10^3$ Hz [14], and that of cellulose is ~ 5 . Therefore the C_∞ values obtained from the fitting are consistent with bulk oil impregnated paper of thickness 0.255 mm in a cell with 50mm diameter electrodes. Noting that the dc conductance circuit elements, G_1 and G_2 , are electrically in series (overall dc-conductance = $[(G_1)^{-1} + (G_2)^{-1}]^{-1}$) yields dc-conductivities at a test temperature of 20°C that range from $\sim 0.4 \times 10^{-10}$ S/m for the unaged material to 3.4×10^{-10} S/m after ageing for 84 days. These values are close to that reported for the unaged PKOAE1 oil fluid at 20°C (1.18×10^{-10} S/m) [14]. Furthermore the relative magnitudes of G_1 and G_2 , and $C_{1\infty}$ and $C_{2\infty}$ are not large enough for them to be regarded as due to the electrode-interface and bulk respectively. Instead all circuit elements must be assigned to origins in the body of the oil-paper sample.

Following on from this we note that each master plot brings all the dispersive processes into coincidence with the same displacements along the frequency axis. This means that the same activation energy applies to the relaxation time for the loss peak as for the timescale for the charge transport in the q-dc response. This observation links the two processes together and suggests that the loss peak refers to negative and positive charge carriers that are bound together as a dipole, which at lower frequencies become the independent carriers of the q-dc process. The fitted parameters in Table 1 for a test temperature of 20°C show that the relaxation time ($(\omega_p)^{-1}$) for this dipole is substantially shorter than the onset time ($(\omega_c)^{-1}$) for the separation of the carriers as independently moving entities up to 56 days of ageing where they become the same order of magnitude. A similar result is found for the dielectric response measured at 50°C.

Since the oil is a source of ions from its water and acid content it is reasonable to assume that the separation of

ionic charge across the clusters of locally-connected oil-filled cavities in the form of a bound dipole would give a dielectric loss peak with a dipole relaxation time determined by the transport rate within the cluster and the blocking capacitances that define the boundary of the cluster. The onset time for the constant phase angle transport component ($\chi'(\omega) \propto \chi''(\omega) \propto \omega^{-p}$) of the q-dc corresponds to further charge separation in the structured matrix of paths formed by the cellulose fibres, in which ions of different polarity move independently of one another.

Since the activation energy is the same for the q-dc transport as for the loss peak relaxation time it seems that the separation of ions within the cluster of connected cavities occurs via the same motions as does the transport, i.e. via ion transport on the surfaces of the paper fibres and not through liquid oil droplets. At times less than $(\omega_p)^{-1}$ the cluster boundary is undergoing charging. At longer times (i.e. $t > (\omega_c)^{-1}$) charges of opposite polarity are able to move independently along the surfaces of fibres through the matrix of structured paths within the sample, where they may connect to other clusters and establish long transport paths in the convoluted space of the fibre surfaces. The scaling of the spatially restricted transport paths gives the scaling hierarchy of series resistor-capacitors required for the constant phase angle response [16]. The exponent p is dependent upon the geometry of the transport paths [17] although the random walk dynamics of charge motion on such paths may also be involved [18]. The build-up of polarization as the frequency reduces corresponds to the neutralization of the cavity dipoles by transport of ions between them and the consequent separation of opposite polarity charge over progressively larger distances.

A second feature of the master plots revealed by the locus of the displacements is that the amplitude of the response does not change with temperature, i.e. no displacement in the y-axis is required to bring the responses into coincidence at any given stage of ageing. This means that at any stage of the ageing process the dipoles formed by ion separation across a cluster have a magnitude independent of temperature and the q-dc amplitude characteristic is also independent of temperature. The measurement temperature therefore does not affect the concentration of ions, just their speed of movement and hence the characteristic timescale for relaxation across a cluster, and release and transport as independent ions.

5.2 INFLUENCE OF AGEING

A number of features of the dielectric response of oil-paper insulation are known to change as a result of ageing [19]. In our case the characteristic frequencies of the loss peak and the q-dc process initially move to higher values as ageing progresses followed by a levelling off at long ageing times, as is shown in Figures 9 and 10. It has previously been noted that adsorbed moisture can have such an effect upon q-dc transport in various materials (see for example [20, 21]) and specifically in oil impregnated pressboard and paper samples [22-24]. The experimental procedure that we have adopted here is intended to reduce the initial amount of absorbed moisture to a minimum and inhibit the adsorption

of water from the surroundings. However we have observed that 80 days of thermal ageing of the PKOAE1 oil on its own increases its water content from 201mg/kg to 452mg/kg and its acid content from 0.34 mg KOH/g to 1.68 mg KOH/g. This is consistent with previous investigations that show a general increase in the moisture content of natural ester during thermal ageing with a peak between 40 and 50 days [25-27]. An increase in acid content was also reported in [26]. Therefore it can be expected that both water and acidic entities will be generated in the oil component of the oil-impregnated paper during thermal ageing. Water is also generated, among other products, by the thermal degradation of the paper fibre. However PKOAE1 is a natural ester whose polar groups allow it to absorb more water than mineral oil and hence is capable of extracting some of the water from the paper fibres. In [25, 26, 28] it was found that the moisture content of the paper decreased steadily during ageing in natural ester in contrast to mineral oil where it increased. In [27] it was found to go through a peak at around 30 days and then decreased. Thus some of the water produced by thermal degradation of the paper component is able to cause chain scission in the cellulose by attacking the bridging oxygen, thereby reducing the degree of polymerization [29-31]. This will break up fibres and widen low density paths within the remaining fibre structure. These paths will also be extended as a result of the removal of inter-fibre contacts. As a result the ion transport, both within and between clusters will be facilitated and the process will move to higher frequencies. A similar frequency increase has been observed in the q-dc response of aged micro-particle filled epoxy resin [32] and glass fibre reinforced epoxy resin [33]. In these cases microscopic examination showed the increase in frequency to be associated respectively with de-binding of the micro-particle and the fibre from the resin, i.e. the creation of low density paths such as occurs here.

The temperature dependence of the frequency location of the complete dispersive response is also changed, defining a change in the activation energy common to both the loss peak and the q-dc process. An Arrhenius plot for the characteristic frequency of the complete response in the frequency window (taken from the frequency shifts that bring the data into coincidence in the master plot) of each of the samples is given in Figure 11, where it can be seen that the data yields a good fit to a thermally activated process. The dependence of the activation energy on ageing is shown in Figure 12, and it is clear that the activation energy diminishes as ageing progresses, though it goes through a plateau region at intermediate ageing times. This result shows that as ageing progresses the potential barriers for charge movement along and around the fibre surfaces, in the matrix of connecting paths that constitute the system of clusters, are reduced. This is consistent with the deduction from the shift in characteristic frequencies that the connecting amorphous paths are being widened, and the inter-fibre contacts are being broken as the degree of polymerization reduces.

The other changes that ageing brings to the dielectric response are mainly concerned with the loss peak. In the

first instance the value of n_1 becomes indistinguishable from zero on ageing. This implies that after ageing the loss peak is produced by an ideal dc-conductance in the connections within each cluster. Table 1 also shows that ageing produces a 2-order of magnitude increase in the frequency of the cluster relaxation. The ions therefore move faster within the cluster than before ageing. Figure 9 indicates that there is a peak in the relaxation frequency around 28 days of ageing, which may correspond to the peak in paper water content observed in [27], however the change is essentially completed between 28 and 56 days of ageing. Over the same period the characteristic frequency for the q-dc process increases by 3-orders of magnitude but thereafter remains relatively constant. It becomes of a comparable magnitude to that of the loss peak by 56 days, which indicates that after this period of ageing the onset of the q-dc transport component takes place as soon as the cluster boundary becomes fully charged. This tends to suggest that the breakup of the fibres during the first 56 days of ageing has made the clusters almost uniform regions of ion transport bounded by residual cellulose that can be penetrated on structured paths once the boundary becomes fully charged.

The magnitude of the loss peak capacitance (C_{10}) shows a monotonic increase by a factor of ~ 19 over the ageing period, though it should be noted that the largest change occurs after 28 days. One possible contribution to this increase is the fivefold increase in ion content of the PKOAE1 produced by thermal ageing [30]. It is most likely however that the largest part of this change is associated with a reduction in the thickness of the capacitive boundaries of the clusters as the paper fibres break up, equivalent to larger charge separations and effectively bigger cluster dipoles. In contrast to the behaviour of the loss peak the magnitude of the q-dc process (C_{20}) remains substantially unchanged during thermal ageing. This indicates that the amount of charges that can become independent remains unchanged although the rate at which they become independent and move on the inter-cluster matrix of paths increases strongly. This associates the q-dc process with charge transport in the cellulose matrix that remains intact albeit with the connecting paths widened and extended.

The biggest part of the changes to the characteristic frequencies (Fig. 10), occur in the first 28-56 days of ageing as does the biggest reduction in activation energy, Fig. 12. After 56 days these changes slow down. It would therefore seem that the first stage of ageing involves the breaking of the fibres and inter-fibre contacts, by chain scission. Beyond 56 days the changes continue in most cases but much more slowly indicating that the breakup of the fibre matrix is being inhibited to some extent. There are a number of features that may be responsible for this. In the first place water may be extracted from the paper component into the PKOAE1 because its polar nature gives it a greater affinity for water [25-28]. Secondly high molecular weight fatty acids produced by thermal aging of the PKOAE1 cause the formation of waxy deposits, see Fig. 13, which may coat the fibres and reduce further degradation. Thirdly the water

dissolved in the PKOAE1 consumed by hydrolysis produces free low molecular weight fatty acids, which may react with the cellulose backbone via transesterification and as a result protect the cellulose from hydrolysis [26]. This will also have the further effect of allowing more water to be removed from the paper component to dissolve in the oil, i.e. it dries the paper component and reduces degradation by chain scission. This process may be responsible for the presence of a peak in the water content of the paper observed around 30 days in [27].

However the loss peak magnitude in Fig. 12 and conductances G_1 and G_2 (see Table 2) continue to increase steadily from 28 days. It therefore seems that ageing of the PKOAE1 impregnated Kraft paper involves two stages. The first stage is a breaking of the paper fibres. This process leads to deterioration of the paper matrix through a widening and extension of transport paths and a reduction of the activation energy for long range charge transport. A narrowing of the residual blocking regions between clusters of connected oil filled cavities also occurs giving an increase in the magnitude of the loss peak response due to the charging of the cluster boundaries. During this stage the degradation of the PKOAE1 oil produces fatty acids that penetrate into the degraded paper component and react with intact cellulose fibres thereby protecting them from further hydrolysis. A second stage then follows in which the magnitudes of the loss peak process and parallel conductances increase. Part of this change may be contributed by the increase in ion density through the continued generation of acid content by the thermal degradation of the PKOAE1. However the continued degradation of those regions of cellulose not protected from chain scission by reaction with fatty acids from the PKOAE1 will also contribute. A drop in the activation energy for the characteristic frequency of the q-dc process is also observed in this stage. This is probably caused by the breakup of barriers in the remaining matrix of transport paths. However there is no reason to believe that this reduction will continue indefinitely with further ageing as such breakup is likely to be accompanied by increased penetration of the PKOAE1 such that more of the cellulose fibres will be protected from scission and the ion transport will be governed by their movement in the oil. In unaged oil the activation energy for the oil conductivity is 0.27 eV [14], i.e. less than the activation energy for the oil-paper sample at times less than 84 days. Similar palm kernel oils [14, 30] with a higher acid content have lower activation energies. Therefore the increase in acid content on ageing can be expected to reduce the activation energy for ion transport in the oil to a value around that measured for the oil-paper system at 84 days.

5.3 MARKERS FOR THE STATE OF AGEING

The potential ageing markers from the analysis are: the activation energy of the characteristic frequency of the dielectric response, the frequency location (characteristic frequencies) of the response, and the amplitude of the response. Although the characteristic frequency of the q-dc

and the overall activation energy change monotonically with ageing they do not show a linear dependence upon ageing time (figures 9, 10 and 12), because the ageing effects appear to take place in two stages. This means that it is not easy to use any of them as an ageing marker. Furthermore life estimation needs parameter values that define the end of life. In the case of the activation energy a value of zero could be used and then an extrapolation of the decrease between 56 and 84 days would give a life of ~100 days under the accelerated thermal ageing regime. However we have no evidence that this decrease will continue with further ageing. It is noticeable that the characteristic frequency is effectively unchanged between 56 and 84 days (figure 10). This is what may be expected if the PKOAE1 oil has penetrated throughout the paper giving an activation energy and transport rate limited to that of the ion conductivity in the oil component. This activation energy depends upon the ion (acid content) of the oil and the measured content of 452mg/kg after 84 days would yield a value of about 0.2 eV, and certainly less than the unaged value of 0.27 eV [30]. This estimate is not too dissimilar to the value measured for the oil-paper sample at 84 days. Therefore the 100 day estimate for the lifetime must be treated with some caution.

An alternative end of life could be when the capacitance of the cluster loss peak (C_{10}) is that of a layer of paper at the electrode interface, which assuming a thickness of 10 μ m would be ~ 9000 pF. Extrapolating C_{10} from 84 days under the assumption that it continues to increase at the same rate as it does between 56 and 84 days, gives a lifetime of ~ 270 days, under the accelerated ageing conditions of the test. This may be a better estimate of the lifetime, but again there is no guarantee that the trend between 56 and 84 days will continue at longer times.

The general features of the behaviour observed here is similar to those reported for changes in the degree of polymerization and tensile strength during the thermal ageing of natural ester- Kraft paper at 160 °C and 170 °C [28], i.e. a strong change over the first 3-4 days followed by a slow change thereafter, where for example the degree of polymerization is 39 % of its original value after 60 days, but thereafter reduces to only 31% at 126 days. Here in comparison the activation energy reduces to 45% of its original value at 56 days and reaches 25% at 84 days; $1/C_{10}$ (proportional to barrier capacitance thickness) decreases to ~10% of its initial value after 56 days and ~6% after 84 days. In [28] the natural ester-paper system still has a viable degree of polymerization at 126 days and this is consistent with our estimation of the end of life from C_{10} and suggests that the change in activation energy may slow down as we argue above.

In [28] it is also shown that the degree of polymerization of mineral oil paper is effectively reduced to zero by 126 days. Our results tend to confirm the implication that natural ester oil does indeed slow down the thermal degradation of oil impregnated Kraft paper. However given the different ways that dielectric and other properties show the effect of ageing and differences in their estimated life predictions we suggest

that the best way that a degree of degradation can be defined is via a basket of marker values [34], which together could be used as an ageing marker.

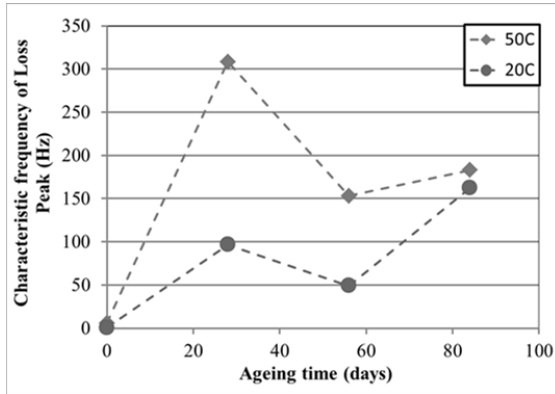


Figure 9. Relationship between the characteristic frequency of loss peak and ageing period at temperatures 20°C and 50°C.

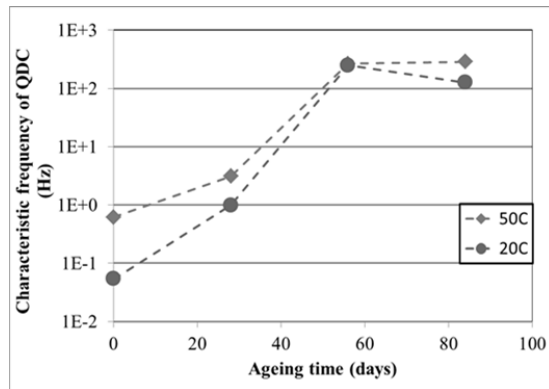


Figure 10. Relationship between the characteristic frequency of q-dc and ageing period at temperatures 20°C and 50°C.

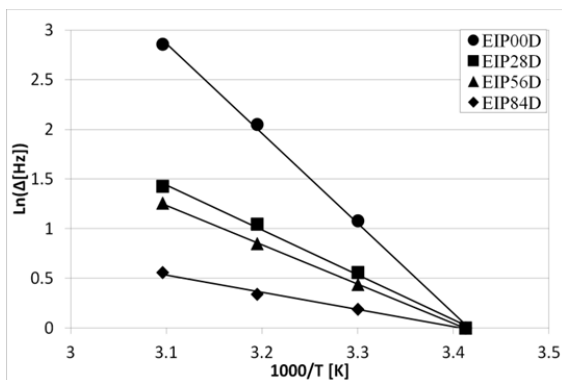


Figure 11. Arrhenius plot of the characteristic frequency shift Δ of the dielectric response given by the frequency displacements necessary to bring the data at different temperatures into co-incidence. The straight lines are the best fit (correlation coefficient $R^2 = 0.99$) to the Arrhenius expression with values of 0.78 eV (EIP00D), 0.39 eV (EIP28D), 0.34 eV (EIP56D) and 0.15 eV (EIP84D).

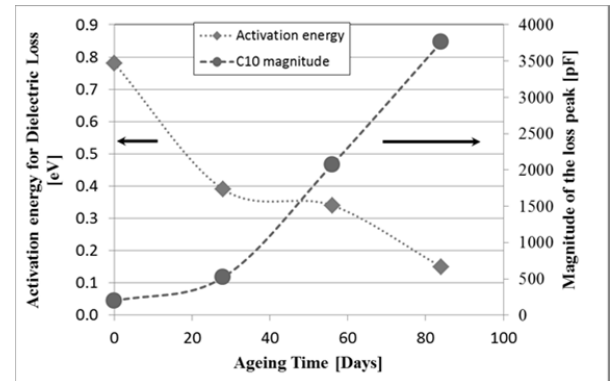


Figure 12. Relationship between activation energy of the characteristic frequency (taken from figure 11) and the ageing period and relationship between magnitude of the loss peak C_{10} (taken from Table 1) and the ageing period.

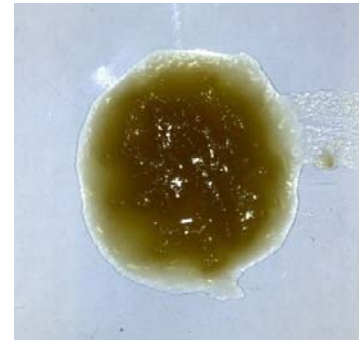


Figure 13. Photograph of polymerized ageing product of the oil after 84 days at 150 °C

6. CONCLUSIONS

The dielectric response of a composite vegetable oil-paper insulating system has been shown to exhibit a q-dc (low frequency dispersion) and a non-Debye loss peak rather than the previously proposed extended Debye like behaviour. The loss peaks and q-dc behaviour fit to the Dissado-Hill cluster model expression. A microscopic mechanism has been proposed for the origin of these responses in which clusters of connected oil-filled cavities in the cellulose paper create regions of high ionic charge concentration. Separation of ions across the cavity gives a dipole and the loss peak behaviour. At low frequencies the charged surfaces of the cavities feed ionic charge for transport through the material along the restricted paths of the fibre surfaces yielding the q-dc behaviour. The characteristic frequencies of both loss peak and q-dc processes have a common activation energy showing a common origin in ion-transport motion. Ageing was shown to reduce the activation energy for the ion motions identically in both dielectric dispersions, with a corresponding increase in the characteristic frequency of both processes at a given temperature. The amplitude of the loss-peak processes also continually increased. These changes were correlated with an ageing process comprised of two stages in which a) paper fibres and inter-fibre contacts were broken allowing the penetration of natural oil degradation products between the fibres and an increased

concentration of ionic carriers from the oil degradation, and b) reaction of the low molecular weight fatty acid degradation products of the oil with the intact fibres producing a protective coating and inhibiting or slowing down further degradation together with removal of moisture from the cellulose fibres by increased oil solvation.

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