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Archeology

ENVIRONMENTAL RESEARCH FOR ART CONSERVATION (ERA)*

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Abstract

The basic concept of this project is to identify and then use the changes which occur in the chemical and physical properties of traditional paint media both to indicate and integrate the effects of environmental conditions on paintings. To achieve this aim, test paint films are being prepared in accordance with traditional artists' recipes. Changes in material properties are monitored using a combination of non-invasive spectroscopy (Bacci), microsensors, thermoanalytical techniques (Odlyha), and microscale analytical mass spectrometry for molecular structure analysis (Boon). The test strips are calibrated by exposure to controlled environments (light, temperature, relative humidity and noxious gases) and alterations in their properties are quantified. This provides information on the nature and rate of change at the molecular level and a data base for evaluating the molecular monitoring strips after their exposure in the field. Field sites have been selected and include various locations in the Tate Gallery (UK), Sandham Chapel (Burghclere, UK), the Uffizi Gallery (It) and the Rijksmuseum (NL). Environmental conditions of some of these locations are being evaluated at present using the glass sensors described in project EV5VCT92 0144. Small piezoelectric quartz crystal humidity sensors will be installed to determine localised variations in relative humidity and temperature on [1] Stanley Spencer paintings in Sandham Chapel and [2] Giotto's "Madonna di Ognissanti" in the Uffizi Gallery. In addition novel coatings using picture varnishes are being applied to similar piezoelectric quartz crystal sensors to evaluate the effects of environmental impact on the chemistry of varnishes on paintings. Data are also being collected on the nature of chemical and physical changes in varnishes and paint media in actual paintings at the molecular level.

Keywords: conservation, environmental research for art

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The rationale for the ERA project

The preservation of a work of art depends on its original condition, the type of materials used by the artist, and on the quality of the environment surrounding the object. The environment in a museum, historic building or display cabinet contributes a complex set of factors which can interact with the object and cause decay [1]. This can influence the stability and overall appearance of the work. Only very recently the importance of good display and storage conditions has been acknowledged. Changes in the colour of paintings, for example, can pose problems in the art historical interpretation of works of art, as discussed by Villers and Hedley [2]. The question remains, how fast do these changes occur? What is the link between environmental data, and the nature and rate of change?

Some work has been carried out at the Uffizi Gallery where monitoring air quality has occurred for SO_2, NO_x and relative humidity variations in selected rooms in the museum [3]. The situation is complex since the factors are likely to act in a synergistic manner [4]. In the case of cellulosic materials it has been shown that relative humidity plays an important role in the uptake of pollutants [5]. Recent work [6] has provided an indication of the extent of corrosivity of indoor air conditions at a number of museums using glass sensors of a specific composition.

The effect of lighting levels has been discussed and maximum illuminances have been recommended [1]. Studies of pigment stability and monitoring the rate of change of colour in selected pigments under controlled gallery lighting conditions has been undertaken at the National Gallery in London [7]. Other contributions in this area have been made by Robert Feller [8], who examined the rates of change of vermilion, and Ruth Johnston Feller with Catherine Bailie, who examined van Dyke Brown and Alizarin Glazes [9]. In the Uffizi Gallery in Florence selected regions of Luca Signorelli's "Predella della Trinità" have been monitored over a period of 66 months and noticeable changes in colour have been detected [10]. The variation in colour of selected pigments can be considered as a marker of damage.

There is also a need to consider the effect of fluctuations in relative humidity and temperature on the mechanical properties of paintings since there is a differential response of canvas, paint and size to fluctuations in relative humidity [11]. The resulting movement in the paint composite structure may then contribute to visible damage such as the appearance of cracks, structural deformations, and paint loss. In the Uffizi Gallery at present monitoring is in progress to determine the movements which may be occurring in the panel painting by Giotto of "Madonna di Ognissanti".

The objectives and concepts of the ERA Project: to study the fate of paintings with "test paintings" as dosimeters

The objectives of this project are part of the overall aim of the European Commission Environment Programme for the Conservation of Cultural Heritage where the emphasis is on the effect of indoor environments on cultural property and its preservation. The specific aim of the ERA project is to develop a molecular monitoring system for assessing the quality of the environment surrounding paintings.

The project proceeds along two main directions: the first is the use painted test strips with a diverse sensitivity for internal processes occurring in paintings. The second is the use of direct monitoring of a surface sensitised to external environmental influences. The degree of change can be directly measured using piezoelectric effects.

The preparation and evaluation of the monitoring test strips

The first stage of our project has involved the preparation of the monitoring tests strips, after the decision was made as to which paint media would be used. Traditionally artists have worked with proteins, oils and natural resins. Based on data from a previous project [12], where we observed that it was possible to detect and measure differences between standard unaged and artificially aged samples of pigmented egg tempera, we decided to work with an egg tempera-resin mastic matrix. This allows the possibility of using marker compounds based on the proteinaceous network as well as the various natural additives (sterols, lipids and terpenoid compounds). Initially a range of pigments have been selected which include basic lead carbonate, basic copper carbonate or azurite, smalt (this is a cobalt containing silica glass), where the metallic ions are thought to have catalytic effects, and organic colorants such as alizarin, curcumin and indigo which are known to be unstable to light [7, 13]. The practical aspects in the preparation of the paint monitor required consideration of the following points: the optimum amount of pigment to be added, the thickness of the paint layer, and the nature of the substrate to be used. Studies have shown that the medium offers possibly some protection from colour change in the early stages of light exposure provided that the pigments are sufficiently well bound [7]. Different supports have been tested since the requirements of the various analytical approaches are not easily compatible. The paint strips are prepared in batches. The monitor will take the form of a number of paint strips and will be exposed for a prescribed time in selected sites in museums and public areas of art display. Special attention is being given to an aesthetic display module which is acceptable in such environments.

Preparation and calibration of first set of test strips

The first series of samples was prepared by a conservator at the Opificio delle Pietre Dure in Florence and circulated to the participants of the project for analysis. These included in the first instance only the organic colorants alizarin, curcumin, and indigo in a tempera medium (whole egg tempera mixed with resin mastic) on Melinex as substrate. A second series has been prepared more recently which includes additional inorganic pigments and a higher proportion of medium. For calibration the test strips were subjected to controlled light ageing procedures. The protocol for light ageing follows current ideas on lower light intensities and follows accepted theory and practice of conservation scientists [14]. Ageing was performed in the purpose built light ageing box at the Tate Gallery [15] for defined periods of

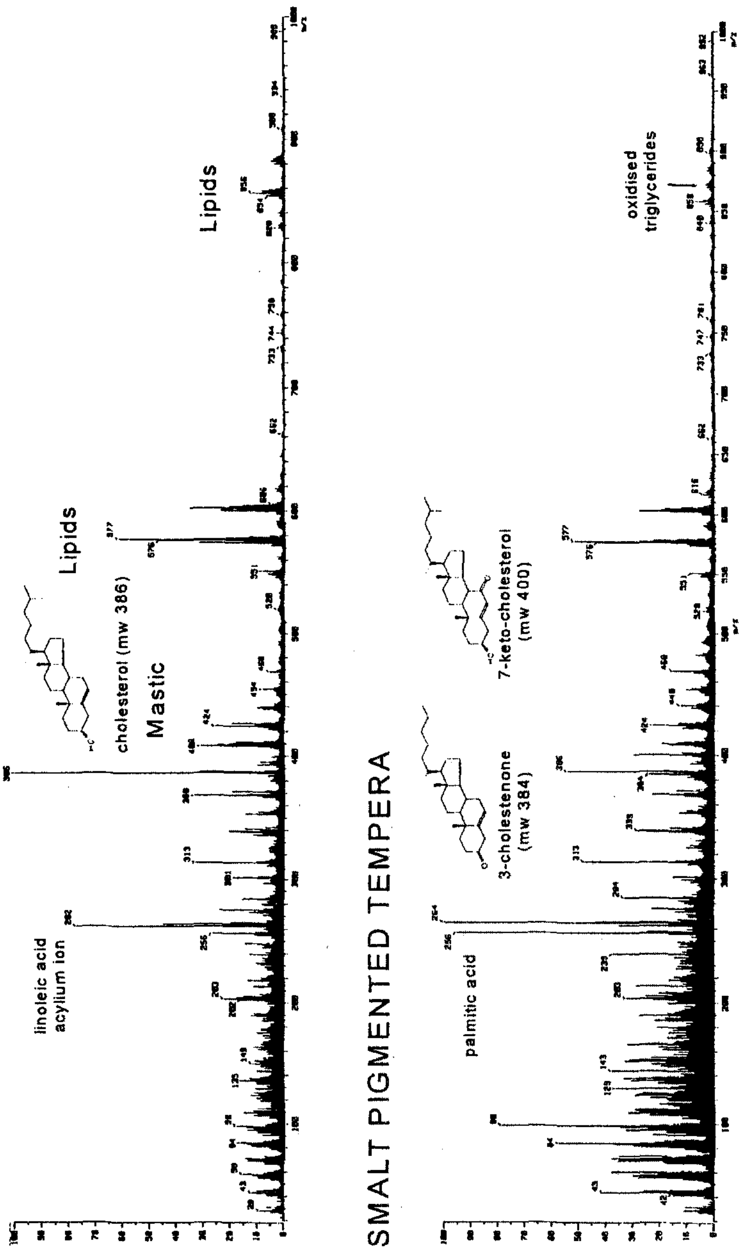

Fig. 1 DTMS summation spectra of unpigmented tempera (upper) and smalt pigmented tempera (lower)

time and under controlled conditions (light intensity: 20000 lux; temperature: 28°C; relative humidity: 40%). Light in itself is not the only factor but it is the motor for ageing mechanisms in experimental simulations. Relative humidity has been mentioned before and is obviously important where it is not completely controlled, such as in historic houses and chapels [16] , for its likely effect on chain hydrolytic reactions and resulting hydrolysis of the oil network of the tempera system.

Preliminary analytical data of light aged test strips

Mass spectrometric data

Comparative Direct Temperature resolved Mass Spectrometry (DTMS) of light aged and unaged tempera samples (Fig. 1) shows that this technique is capable of observing chemical changes in the mastic fraction, in the cholesterol and lipid fractions, in one analytical run. In the present paper we compare the DTMS summation spectrum of a smalt (a cobalt glass) pigmented tempera with that of unpigmented tempera stored in the dark for three weeks. It is clear that linoleic acid moieties (indicated by *m/z* 262) have been strongly affected by the presence of smalt due to the catalytic activity of cobalt. There is evidence for the oxygenation of the triglycerides and this can be deduced from a new cluster of peaks between *m/z* 856 and *m/z* 880. The smalt pigmented sample also shows peaks indicative of C8 and C9 dicarboxylic acids at *m/z* 84 and *m/z* 98. In the smalt pigmented tempera cholesterol (*m/z* 386) has been oxidised to 3-cholestenone (*m/z* 384) and 7-keto-cholesterol (*m/z* 400). Furthermore, a relatively intense peak at *m/z* 143 indicated that smalt had accelerated the oxidation of dammarane compounds mastic components [17]. Comparison of the DTMS spectra of tempera after 3 weeks' dark storage and after 3 months' storage shows that the lipid, cholesterol and mastic fractions of the paint have reached a stable point after 3 weeks' dark storage. This is the case for both the slowly oxidising unpigmented tempera and the rapidly oxidising smalt pigmented tempera. A curing period of 3 weeks seems sufficient.

Thermoanalytical data

Thermal degradation studies were performed to determine thermal stability indices. These are based on parameters measured from the thermal analysis curves (thermogravimetric TG, derivative thermogravimetric DTG (the rate of change of mass with time) and differential scanning calorimetric (DSC) curves recorded as a function of temperature). Previous studies of paint samples using Differential Scanning Calorimetry (DSC) have shown that the resulting curves are correlated with the age of the sample and its chemical composition, including the pigment type [18]. In the context of the ERA project both techniques were used to demonstrate the effect of pigment on the medium and whether there were any interactions between pigment and binder, and to assist in evaluating the pigment volume concentration in the preparation of the monitor.

With respect to the effect of pigment on the medium little attention has been paid to the modifying effect of typical artists' pigments on the stability of paint media to

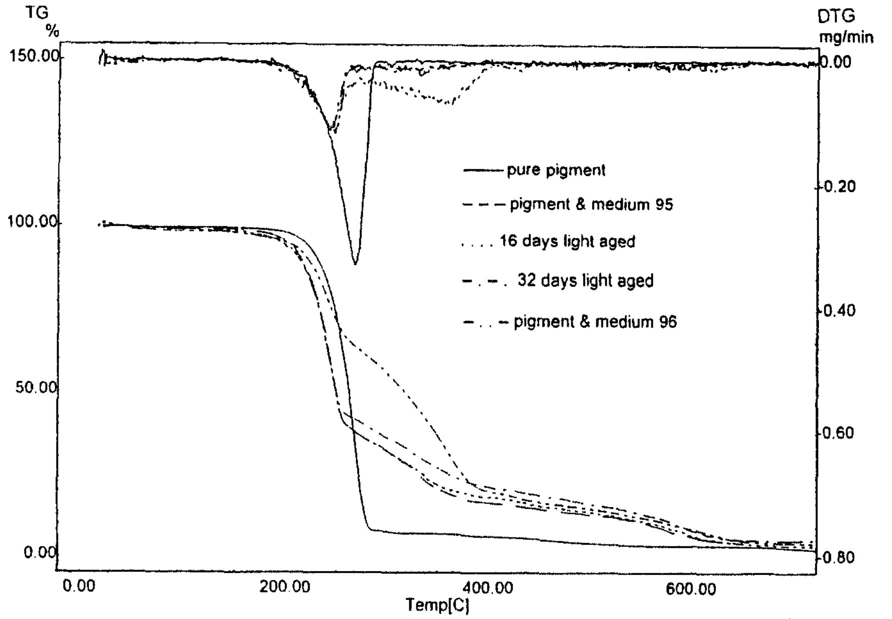

Fig. 2 Thermogravimetric curves (TG, DTG) showing the effect of increased medium content on the degradation curves for the alizarin tempera samples

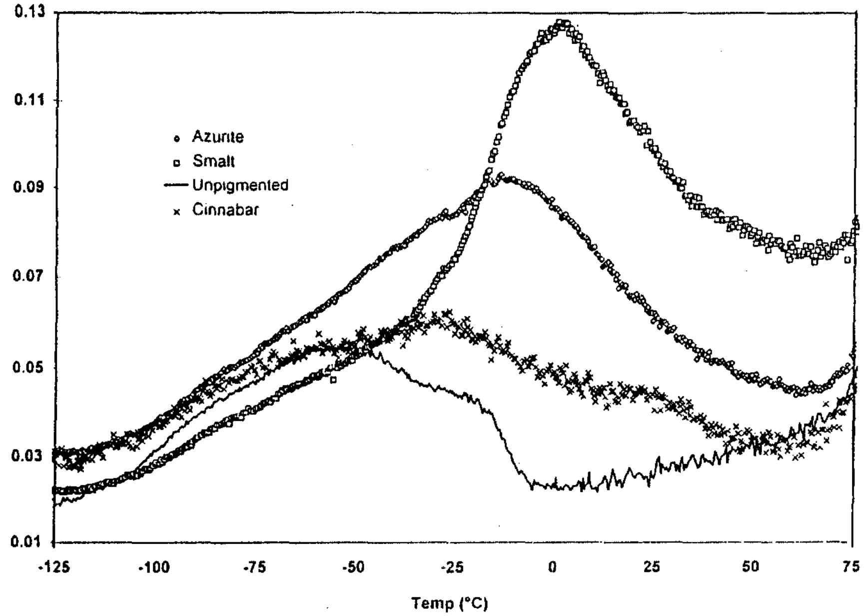

Fig. 3 Shows the variation in the observed peak (tanδ) for the differently pigmented samples

environmental degradation [19]. Our first task has been to determine whether the influence of pigments was detectable. From DSC studies on the first series of test strips it was found that the thermal stability of tempera pigmented samples decreases in the order: indigo > curcumin > alizarin. In the case of the alizarin tempera the degradation processes of pigment and medium occurs almost as two individual processes which shows that the degree of interaction is small. Differences in thermal degradation profiles could be observed between the two series of alizarin temperas on the basis of medium content (Fig. 2) and on light ageing [20]. Other samples such the smalt tempera also showed changes on light ageing which were observed by DSC and infrared spectroscopy [21]. Hence observations have shown that light ageing produces changes in the degradation processes and this can be correlated with the mass spectral data.

Differences in the various pigmented tempera samples have also been demonstrated by the results obtained from dynamic mechanical thermal analysis [22]. This technique enables bulk properties to be related to molecular processes involving changes in cross-linking, degree of cystallinity and chemical structure. Figure 3 shows the variation in the observed peak (tanδ) for the differently pigmented samples. Since the value of this peak provides a measure of the viscoelastic properties of the tempera sample its magnitude is a marker for the effect of various metallic ions on the proteinaceous network and the lipid portion of the egg tempera. In varnishes it has been used to evaluate the changes which occur on ageing. Figure 4 shows the change in tanδ (which is also the glass transition temperature (T_g)) of resin mastic with age [23]. This is yet another marker that will be used for the evaluation of our test strips. A paint or varnish strip can be measured non-destructively so that the effect of ageing and environmental exposure can be monitored.

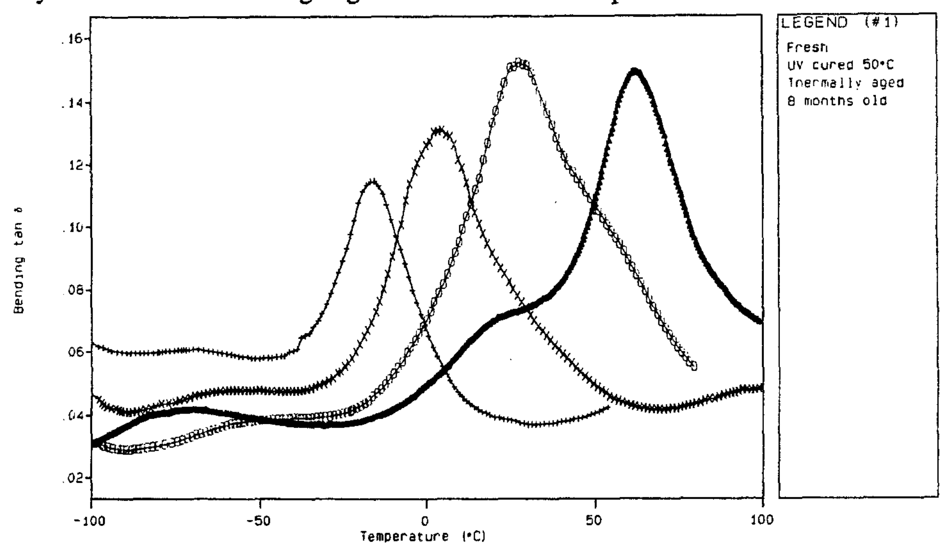

Fig. 4 Shows the change in temperature of tanδ (the glass transition temperature (T_g)) of resin mastic with age

The concept and objectives of the application of piezoelectric sensors in the ERA project

The basic idea is to investigate the potential of piezoelectric quartz crystal resonators as chemical sensors for the assessment of environmental risk factors. Preliminary results will be reported in another paper of measurement of sensor frequencies before and after controlled light ageing experiments. This paper describes how piezoelectric sensors have been coated with triterpenoid varnish, cured, calibrated in controlled environments, and subjected to field studies. The chemical changes in the varnish after exposure are to be identified by non-invasive and destructive microanalytical techniques. The principle of operation of the piezoelectric sensor can be described as follows: when an alternating potential is applied to an untreated sensor it vibrates with a fundamental frequency, when it is applied to a coated sensor there is a shift in the value of the fundamental frequency. This shift in frequency gives an accurate measure of the mass of material deposited on the active area of the device. Any subsequent change in the mass of the coating, due for instance to oxidation, would result in a further shift in the recorded frequency. The risk assessment factor in terms of air quality in this case would be the shift in the frequency. In the course of calibration this could be further related to the chemical changes which have occurred which will be evaluated at the molecular level by mass spectrometric techniques.

Piezoelectric humidity sensors

Piezoelectric humidity sensors have been developed for this project and will be used for the first time to determine localised values of relative humidity and temperature around paintings. A polymeric coating, which has been selected on the basis that it adsorbs and desorbs moisture in a reversible manner [24] is applied to the piezoelectric quartz crystal. The sensors will be used together with Pt-resistance thermometers to provide a measure of gradients of RH and temperature in paintings by Stanley Spencer in Sandham Chapel (UK). If we can define localised regions of gradients in RH and temperature which may develop during the exhibition of a painting, this may assist in identifying areas of stress and potential damage in paintings with environmental fluctuations (this site represents an uncontrolled environment where paintings are set in alcoves and where air movements at the rear side are restricted). It was also chosen since two of us (Boon and Odlyha) in collaboration with the UK National Trust (C. Sitwell and L. Bullock) and the Courtauld Institute of Art (A. Burnstock and M. Calwell) have been involved previously in the characterisation of the materials used by Stanley Spencer in an attempt to understand the mechanism of recurrent bloom formation on two of the paintings (Tea in Hospital Ward and Moving Kitbags) [25] .

The calibration of piezoelectric varnish sensors

In the case of the varnish coated sensors initially light ageing under controlled conditions was used. Monitoring of change was performed by measurement of the frequency shifts after exposure and then chemical analysis using mass spectrometric techniques. Data on the resin mastic coatings from the exposed piezoelectric sensors show that products are formed which also occur in naturally aged varnish. Therefore, after measurement of the frequencies, knowledge already available from previous and parallel work [17], could be used to interpret the chemical changes. Some initial field testing of the suitability of exposure of these devices has been carried out. Varnish coated piezoelectric sensors were given to Dr. Leissner for exposure at sites which had already been tested using the glass sensors and where the overall corrosivity had been evaluated, for example in new showcases containing ruby glasses in the Glass Gallery in Coburg. The preparation, evaluation of curing time for these sensors, and the results of light ageing experiments and field exposure will be described in another paper.

Field studies at selected sites

Glass sensors

Within the framework of the ERA project, sites selected for exposure of our tempera paint test strips were tested using glass sensors provided by Dr. Leissner. Table 1 lists the exposure sites at the Tate and at the Uffizi Galleries.

In addition glass sensors have been exposed in the Clore Gallery at the Tate Gallery in the proximity of Turner's painting "Opening of the Walhalla" This painting has been recently restored (1991). During the restoration work two of us were involved in the sampling and subsequent analysis by thermoanalytical and mass spectrometric techniques [26, 27]. So in this case there is a good understanding of the materials present in the painting: polymerised oils, oxidised triterpenoid acids (markers for the oxidation state of a painting), waxes and protein. It is appropriate that we are concerned with the quality of the environment surrounding the work and the changes it may produce. Resampling of this painting has been discussed with Stephen Hackney of the Conservation Department of the Tate Gallery who has also

Table 1 Overview of exposure sites

Tate Gallery	Uffizi Gallery
Within Gabo packing case with no carbon filters	Botticelli's room Polittico Portinari (Van der Goes)
The air intake to the air conditioning unit	Botticelli's room Primavera (Botticelli)
Within the painting frame of "The Black Brook" (J. Singer Sargent No. 4783)	Giotto's room Madonna d'Ognissanti (Giotto)
The air conditioned store	Giotto's room Maestà (Cimabue)
Gallery 38 which is a non conditioned public gallery	Giotto's room Maestà (Giotto)
The entrance octagon which is a large gallery with poor conditions of control	Leonardo's room Madonna e Santi (Perugino)

supplied the storage and display record of the painting including the period prior to its location in the Clore Gallery.

Some applications to actual cases

Identification of materials used in paintings

Pigments in the panel paintings, mentioned above, "Madonna di Ognissanti " by Giotto and the Predella della Trinità by Luca Signorelli have been identified by Dr. M. Bacci [10, 28]. The pigments include azurite, smalt and cinnabar, and these have also been used in the preparation of our paint monitors. Data on the medium of early Italian panel paintings e.g Coppo di Marcovaldo's painting in the Capelle Brancacci [18] and from later periods [29] are also available from previous studies. Data are also available on the chemical changes which occur in naturally aged dammar varnishes and the identification of a marker, ocotillone, as an indicator for oxidised dammar varnish [17]. Comparison can be made between the aged test monitors and actual samples to attempt to understand the degradation processes which have occurred. Some parallel studies include identification of materials used in frescoes: Zuccari's frescoes in the dome of S. Maria del Fiore (Florence) [30] and Alessandro Allori's frescoes (in the proximity of the Santa Maria Novella Church, Florence). The latter is an example where serious damage to colour had occurred in certain regions through probable interventions in the 18th century. For some of these samples medium analysis has been performed.

Assessment of damage with reflection spectroscopy

Dr. M. Bacci has monitored the colour change in Luca Signorelli's "Predella della Trinità" in the Leonardo room in the Uffizi Gallery over a period of 66 months. It was found that in spite of the environmental control in the room detectable changes in colour have occurred [10]. He has also identified the presence of smalt in Alessandro Allori's frescoes. In certain regions serious damage to the colour has occurred through probable interventions in the 18th century.

RH and temperature gradients as markers for potential damage

Preliminary evaluation of climatic conditions within Sandham chapel has been made, and the appropriate sensor housings and supports for the sensors have been designed [31]. Meanwhile the testing of these sensors has taken place in the laboratory. Figure 5 shows the 2 sensors with the housings on either side of a test painting under an imposed RH gradient of ca. 24% (above) and ca. 70% (below). Figure 6 shows the effect of RH variation on the modulus of a test piece of primed canvas using dynamic mechanical thermal analysis (DMTA). At Birkbeck College evaluations continue to provide information on the effect of variations in relative humidity and temperature on the mechanical properties of painting canvases, and to evaluate the effects of preventive conservation treatment (deacidification). Novel

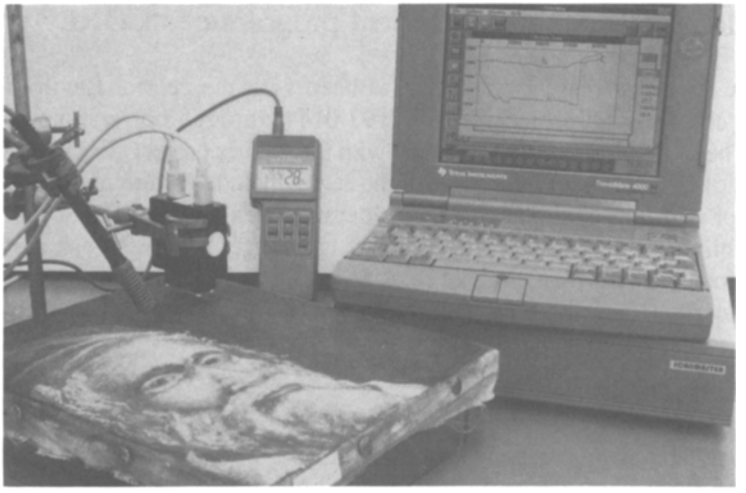

Fig. 5 This shows the two piezoelectric humidity sensors with the housings on either side of a test painting under an imposed RH gradient of ca. 24% (above) and ca. 70% (below)

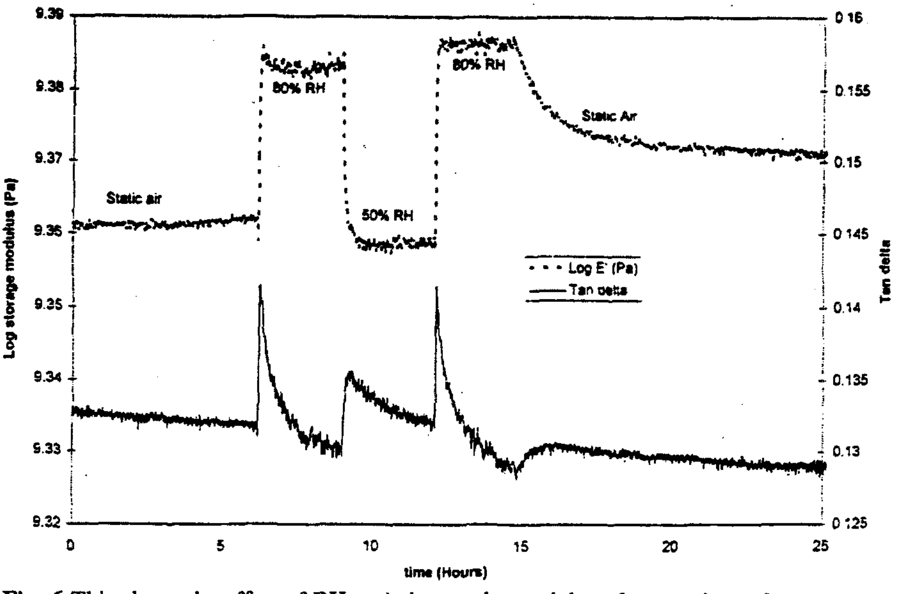

Fig. 6 This shows the effect of RH variation on the modulus of a test piece of primed canvas using dynamic mechanical thermal analysis (DMTA)

methodology is also being developed at Birkbeck College for monitoring the effects of different loads on the drying profiles of treated samples [30]. Mechanical and dielectric techniques are being used to obtain markers of change for canvases with changes in moisture content [32].

Interactions within the environment programme "CORE" group

At this stage our main interaction has been with the central Environment programme "core" group project (EV5VCT92 0144) through our common interest in sensors. There are also some interactions with the leather project (EV5VCT94 0514). Examples of our common interest with the latter include (a) the non invasive determination of moisture content and its consideration as a marker of ageing and the potential stability of the material, (b) monitoring changes in moisture content for materials exposed to different levels of relative humidity [33]. Also we share the problem of detecting changes in a complex organic matrix.

Conclusions

In conclusion at this stage of the ERA project we have succeeded in preparing two series of test paint films. They are based on an egg tempera-resin matrix and contain (1) organic and (2) inorganic colorants. Both series have undergone controlled light ageing and subsequent analysis by the three groups. The preparation of the paint strips provided an insight into the variety of traditional recipes used for egg tempera painting, and the problem of preparing reproducible samples suited to the measuring techniques of the three groups. In the preparation of the samples various pigments and various concentrations of pigment were used. The results are beginning to show the interesting and varied effects of the different metallic ions. This has not been previously studied and provides valuable data for conservation scientists in terms of understanding the mechanisms of ageing and degradation of tempera paint under various environmental conditions. Setting the ageing conditions for testing the sensors is also influenced by the conditions of the sites which have been selected such as those in Sandham Chapel (variation in RH and temperature) and the Uffizi Gallery where air monitoring has given some indication of levels of NO_x and SO_2 which can be found. In the design of the multistrip system special attention is being given to an aesthetic display module which is acceptable in the museum environment.

Parallel studies using varnish-coated piezoelectric sensors have demonstrated that controlled ageing shows measurable shifts in frequency and that mass spectra show changes which involve the light sensitive components of the material. Suitable housings for these sensors are being designed for their exposure together with the paint monitors at field sites previously evaluated by the glass sensors.

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