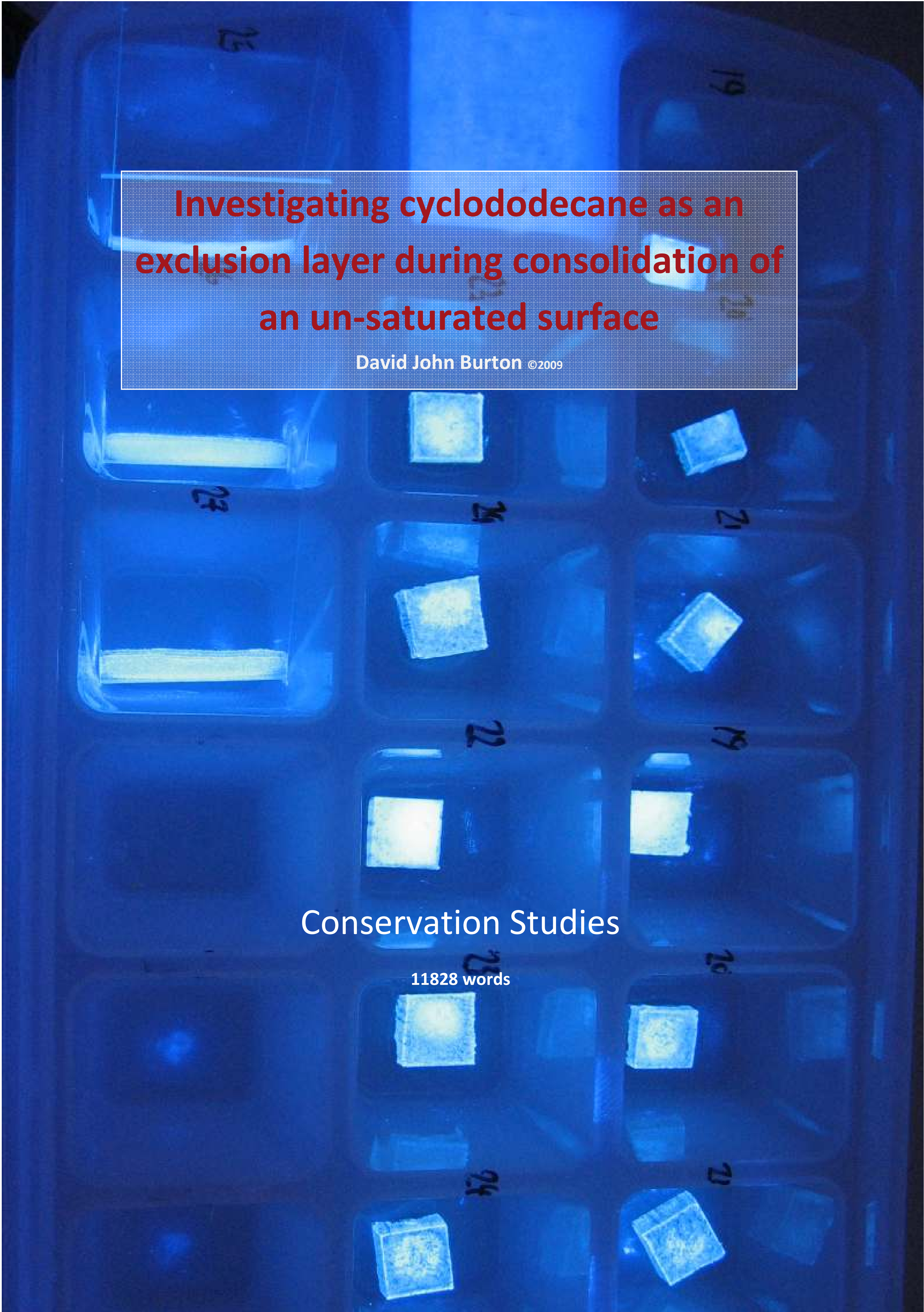
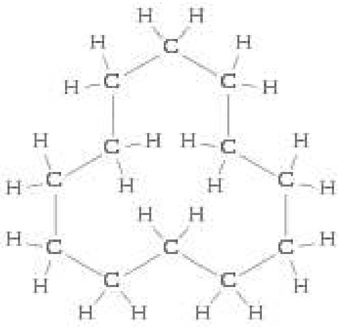
Investigating cyclododecane as an exclusion layer during consolidation of an un-saturated surface

David John Burton



Conservation Studies

11828 words



[1]

Contents

[1] http://www.wolframalpha.com/input/?i=cyclododecane

Abstract

At present conservators are forced to accept a certain level of surface colour alteration taking place when the substrate subordinate to a dry surface is consolidated using dissolved resins.

Research undertaken in other areas of conservation suggested that a temporary controlled isolation of this surface layer with a volatile material, such as cyclododecane would allow more controllable treatment of this underlying material to take place. The exclusion of consolidants provided by a temporary isolation would allow a conservator a time period in which to treat an object without this usually associated surface corruption.

An experimental regime has been developed to allow the testing of this hypothesis through visual comparison, natural light photography, UV photography and microscopy - the emerging trends have been recorded. There are obvious practical limitations with the experimental methods only modelling the problems of larger and more varied objects that may be encountered in real conservation scenarios.

The conclusions of this research provide a protocol for the controlled protection of an historic surface during a resin-based consolidation treatment. A relationship between the application method of the exclusion layer and the solution strength of a popular resinous consolidant has been established through experimentation. Further research will be necessary to eliminate minor practical modus operandi but the results provide strong evidence of a good practical solution to an historic problem in wooden objects conservation.

Introduction

The key quality of cyclododecane is that it sublimates at room temperature over time, offering an intriguing array of possible applications in objects conservation. Cyclododecane is also non-polar in nature and therefore is a highly suitable material for use as an exclusion coat to limit the passage to the historic surface of a polar consolidation solution applied to an underlying timber substrate.

An experimental model with accepted limitations was developed to allow the investigation of various application techniques for an exclusion layer of cyclododecane ($\mathbf{C_{12}H_{24}}$) using controlled procedures and techniques to provide data. Previous research suggested that cyclododecane may penetrate deeper into a porous surface if it were dissolved in a non-polar solvent; this was reflected in the testing process. In order to more accurately track the progress and integrity of the cyclododecane layer some samples were adulterated with Uvitex OB® (2,5-Thiophenediylbis 5-tert-butyl-1,3-benzoxazole). Uvitex OB® is an optical brightening agent used in the plastics industry that allowed the cyclododecane to be observed under Ultra Violet light for analytical purposes.

Butvar B98 (polyvinyl butyral resin) was chosen as a popular consolidant and tested in solution in 2 polar solvents; IMS (95% ethanol ($\mathbf{CH_3\ CH_2OH}$) – 5% methanol ($\mathbf{CH_3OH}$)) and Acetone ($\mathbf{CH_3\ COCH_3}$) at known practicable solution strengths. This consolidant was dyed using Sellaset H (1:2 di-sulphonated Co-complex dye) in order to allow visual discrimination for the purposes of this research.

After a range of consolidant solutions had been tested against a range of exclusion layer solutions the resulting data was visually assessed, cross referencing the results with those obtained from UV photography and UV microscopy. The resulting conclusions provide data substantiating the relationship between the non-polar exclusion coat and the polar consolidant solution.

The Problem for Conservators

Image 0-5

An unsaturated surface[2]

There is an historic problem in objects conservation in the ability to protect the visual appearance and integrity of an unsaturated surface.[3] The problem goes far beyond furniture; ethnographic material, polychrome sculpture, panel paintings, leather objects and wooden objects of all kinds suffer these same issues. In a study undertaken as long ago as 1980 at the Canadian conservation Institute it was stated that *"the two most commonly discussed features of resinous consolidants/impregnants for wood are whether they cause the treated surface to be shiny or to darken; both effects are considered to be most undesirable."*[4] This was a far-reaching study investigating 13 resinous consolidants, with this comment is being made boldly on page 2 as it was considered to be the most important practical compromise of this type of consolidation. In another article also published in 1980 it was stated "*darkening of the wood after impregnation is inevitable,"*[5] this study concluded that along with the improvement in the structural capabilities of the timber came a degradation of the historic surface.

In a comprehensive study undertaken in 1985 it was stated that *"treated wood may have an aesthetically unsatisfactory appearance."*[6] This research was undertaken to establish structural improvements provided by various consolidants, however the visual impact of these treatments was mentioned on more than one occasion.

[2] Base of 17th century pilgrims model of the Basilica of the Holy Sepulchre – untreated Olivewood
[3] Generic Images (image appendix) Image 0-5 –unsaturated surface – 17th century Olivewood pilgrims souvenir model of the Basilica of the Holy Sepulchre (shown disassembled)
[4] Grattan DW (1980) p2
[5] Grattan DW (1980) p2
[6] Wang Y & Schniewind AP (1985) p71

A more recent study published in 2006 investigating wood consolidation in objects damaged by insect pests[8] began with a statement;" *The consolidation of three-dimensional wooden objects - either deprived or coated with polychromy which are damaged by insects with the exception of structural and archaeological wooden objects, is still a critical operation. Many doubts remain on the modalities, on the most adequate material, as well as the effectiveness of the treatment*."[9]

image 0-4

Common furniture beetle damage[7]

These studies provide evidence that this type of resinous consolidation is still fraught with practical compromise regarding historic surface degradation. The question of consolidation involving saturation of a substrate usually arises at the point when an invasive treatment is necessary to prevent further structural deterioration. Consolidation is to "*stabilise a degraded or weakened structure by introducing within it or attaching to it materials capable of holding it together*,"[10] and usually limited options are available to the conservator at this point.

Any finished, polished, gilded, waxed or other surface treatments are by their very nature saturated. These surfaces do not change their visual appearance with the application of further treatments. These can include surface coatings, pollution by consolidants, adhesives and other substances that may saturate the surface. "*Saturation and gloss are inter-related optical properties of a surface. Saturation describes the degree of intensity or vividness of colour*"[11] the molecular weight, refractive index and gloss of a surface coating invariably affect the level of saturation of the surface to which it is applied. The ability of a coating to *wet* surface thoroughly (to displace air from a surface and to achieve an intimate contact with it at molecular level) will have a significant visual effect on the visual appearance of that surface.

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[7] Section of pine drawer side from 18th century bachelors chest
[8] Generic Images (image appendix) Image 0-4 – Common furniture beetle damage
[9] Maritato R & Snider D (2006) p149
[10] Mcgiffin R (1983) p222
[11] Rivers S & Umney R (2003) p587

Unsaturated surfaces may be the less decorative and unseen areas of an object, but not always; sometimes a decorative surface can become dry, friable and unsaturated in its nature and appearance due to degradation. A study undertaken in 1982 entitled: *“Friable ochre surfaces: Further research into the problems of colour changes associated with synthetic resin consolidation”* concentrated solely on the associated problems of colour alteration which inherent in this type of surface.[12] The study concluded that if *"the refractive index is below or above that of the ochres, problems with colour changes will be experienced unless there are no resin residues left on the surfaces of the consolidated materials."*[13]

When a conservator treats material that is adjoining a surface such as this, this potential colour change and therefore degradation of the dry surface is a significant factor in the decision-making process. As most of these changes are due to degradation often at a molecular level the introduction of other coatings or consolidants into this surface is fraught with ethical compromise. As the pollution of the historic surface is at present unavoidable, and a large proportion of the surfaces are unseen, factors regarding the overall structural integrity of the object often become the dominant factor in any decision made.

[12] I’ons A (1982) p13
[13] I’ons A (1982) p21

Cyclododecane

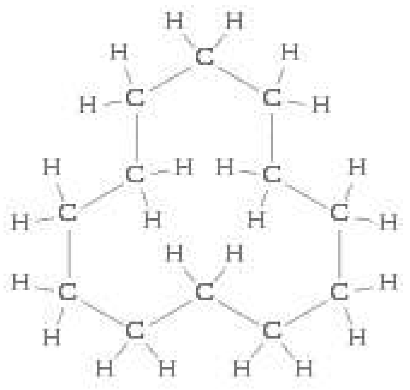

Fig 1

The cyclododecane molecule[14]

Cyclododecane is a saturated cyclic alkane; a hydrocarbon ring with the maximum amount of hydrogen atoms bonded to the carbon atoms. Its molecular structure takes the form of an undulating triad ring (Fig 1), *"it is neither a polymer nor a complex natural substance; rather it is a simple molecule."*[15] The triad ring not only provides volume, but also symmetry to the molecule allowing crystallisation to take place at room temperature.[16]

Cyclododecane was *"first synthesised in Zurich in 1926 by Leopold Ruzicka"*[17] as part of groundbreaking work on macro-cyclic compounds for the perfume industry. It is a white crystalline solid with a very slight odour at room temperature; it has a melting point of 58° to 61° Celsius depending on where data is obtained from. At room temperature this substance is very similar to other hard, non-polar, hydrocarbon waxes that the practicing conservator is used to handling.

There are minor health and safety issues to be considered; the MSDS recommends a well ventilated work area and the use of gloves and goggles - however this material is considered to be a similar health risk to other commonly used waxes. More worrying from a safety point of view is the variation in the quoted flashpoint - the flash point of a flammable liquid is the lowest temperature at which it can form an ignitable mixture in air, at this temperature the vapour may cease to burn when the source of ignition is removed. According to a study undertaken in 2008 this can be as low as 89°C or as high as 114°C.[18]

[14] http://www.wolframalpha.com/input/?i=cyclododecane
[15] Cagna M & Riggiardi D (2006) p89
[16] Billingham N 2009
[17] Rowe S Roziek C (2008) p18
[18] Rowe S Roziek C (2008) p18

Given its chemical formula of $C_{12}H_{24}$ one could be forgiven for thinking that this simple saturated hydrocarbon wax, given its obvious non-polar nature, would have little to offer the conservator. There are various forms of $C_{12}H_{24}$ however this particular form known as cyclododecane exhibits a very useful characteristic, that of sublimation - a transition from the solid to gas phase with no intermediate liquid stage. Carbon dioxide is a common example of a chemical that sublimates at room temperature and atmospheric pressure; a block of solid CO_2 (dry ice) at room temperature and at atmospheric pressure will turn into gas without passing through a liquid phase.

Sublimation is a universal Phenomenon exhibited by materials at temperatures below their triple points. In thermodynamics, the triple point of a substance is the temperature and pressure at which three phases; gas, liquid, and solid coexist in thermodynamic equilibrium. This phenomenon is due to the relatively high vapour pressure caused by the geometry of a given molecule. Vapour pressure is measured in millimetres of mercury or in bar; cyclododecane has a vapour pressure of 0.039 mBar at 25°C.[19]

The non-polar nature of cyclododecane was a crucial factor as a proposed exclusion layer within a larger polar consolidation regime. As polar and non-polar materials tend not to attract each other at molecular level the use of this material to stop the passage of a polar consolidant vehicle seemed an extremely viable prospect. Electrons in polar covalent bonds are unequally shared between the two bonded atoms, which results in partial positive and negative charges. The separation of the partial charges creates a dipole. The word dipole means two poles; separated partial positive and negative charges. A polar molecule results when a molecule contains polar bonds such as this in an asymmetrical arrangement.

"*Polarity results from the uneven charge distribution between various atoms in a compound; Atoms, such as nitrogen, oxygen, and halogens that are more electronegative have a tendency to have partial negative charges. Atoms, such as carbon and hydrogen, have a tendency to be more neutral or have partial positive charges*." [20]

[19] Cagna M & Riggiardi D (2006) p91

[20] http://www.elmhurst.edu/~chm/vchembook/210polarity.html

Non-polar molecules are of two types;

Molecules whose atoms have equal or nearly equal electro-negativities (as measured on the Pauling scale)[21] which have zero or very small dipole moments, cyclododecane falls into this category. The electro-negativities of hydrogen and carbon are 2.20 and 2.55[22] - the 0.35 difference in electro-negativity for the H-C bonds tells us that cyclododecane is essentially non-polar.

A second type of non-polar molecule has polar bonds, but the molecular geometry is symmetrical allowing the bond dipoles to cancel each other out.

[21] http://www.hull.ac.uk/chemistry/electroneg.php

[22] http://preparatorychemistry.com/Bishop_molecular_polarity.htm

Historical Use of Cyclododecane in Conservation

Cyclododecane has only been in use for around a decade in conservation and was first proposed as a conservation material in Germany in 1995 by Hans Michel Hangleiter.[23] It has been documented in various applications since then, but gained significant prominence as a temporary consolidant an unstable ceramic surfaces with salt damage in a study undertaken by the National Museum of the American Indian - USA - 1997. Cyclododecane was used to consolidate archaeological vessels from South America which were highly contaminated with soluble salts, these objects needed to be transported from the museums research centre to another site, and due to time constraints it was not possible to desalinate and stabilise the ceramics prior to their move.[24] This is a well documented example of the successful use of this material and the experiment was considered to be a mixed success.

The British Museum used cyclododecane as a surface consolidant during the transportation of a fragile Egyptian wall painting. [25] Following this initial success it was used again in 2004 by collection of Egyptian ceramics is a temporary consolidant prior to transportation and subsequent treatment.[26]

In 2005, a canvas wall mural project, under the direction of lead conservator Dr. Joyce Hill Stoner included the temporary consolidation by spray application of cyclododecane in petroleum benzene (white spirit) of the friable painted surface during dismounting and transportation from the church to the conservation studio.[27]

A considerable number of studies endorse cyclododecane as a viable volatile binding media with many positive characteristics; studies undertaken in 2007 using Raman Spectra to investigate canvas samples impregnated with cyclododecane for the rate and quality of sublimation were very successful and confirmed *"no damage"*[28] had taken place to the surface.

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[23] Rowe S Roziek C (2008) p18
[24] Cleere DC(2005) p26
[25] Cleere(2005) p26
[26] Cleere(2005) p26
[27] Podmaniczky MS - conversation April 2009
[28] Kuvvetli F (2007) p31

As we can see this material has already found a wide number of applications within conservation, it has historically been used in conservation and a number of areas, to summarise;

As a Hydrophobic consolidant

This is probably the most common historical use for cyclododecane within conservation, and largely embodies the properties explored in this research. The use of cyclododecane as a hydrophobic mask to allow treatment of surrounding areas on the wall paintings and other fine Art objects is well documented. A comprehensive study undertaken as recently as 2008 and published in the IIC journal "Reviews in Conservation" investigated a wide range of applications for this material, successfully investigating application techniques, sublimation rates and a range of uses within conservation.[29]

A considerable amount of work has also been done in the field of textile and paper conservation using cyclododecane with varying degrees of success. Irene Bruckle's detailed study into the use of cyclododecane to protect water based inks during aqueous treatments of paper however found limitations in the technique several years previously.[30]

Yadin Larochette also found practical limitations when using cyclododecane as a barrier during aqueous textile bleaching conservation treatments in 2004.[31] Problems with colour change in archaeological objects that have been consolidated in this way and then desalinated have also been well documented, highlighting further limitations in the practical use of this material.[32]

[29] Rowe S Roziek C (2008) p22
[30] Bruckle I, Thornton P, Nichols K, Strickler G (1999) p6
[31] Larochette (2004) p5
[32] Rowe S Roziek C (2008) p25

As a Release Layer

Cyclododecane has been a popular choice as a separation or barrier layer for mould making and casting with mixed success; often the inherent crystalline nature of the material can interfere with the transfers of surface detail in such techniques. Due to the nature of many of the materials used in casting (often silicone), which is itself a non-polar, cyclododecane, is often part of a much larger and more complex release layer regime. This application has had mixed results; in a study investigating casting replacements for marble sculptures it was found that *"slight darkening of the stone was seen in some tests, the result overall compared favourably with alternative barrier systems"*[33]

As a temporary consolidant during intervention

This is probably the most well documented and earliest use for this material; as a temporary consolidant during the facing a fine Art objects, often for the purposes of transportation.[34] Fine Art conservation is fraught with problems of fragile and friable surfaces, and particularly in the field of paintings conservation one could argue that these objects were some of the most difficult objects to treat. The decorative surface of a painting is arguably everything; no one goes to the National portrait Gallery to see the quality of the canvases masterpieces are painted on, or to observe the gesso upon which the paint is applied. As these delicate surfaces degrade it is sometimes necessary to stabilise them in order for conservation to take place. Cyclododecane was seen as a possible solution to this problem and was used as a material for *facing* over a decade ago. [35]Facing has a long history and this new material was ideally suited to the challenges this process presented. [36] To give an example; one method of application would be to apply the cyclododecane to aluminium foil or Melinex and apply face down to the surface of an object. With a gentle application of heat the conservator would melt the wax saturating the surface and providing a mechanical support to the loose and vulnerable fragments. The Melinex film could then be removed allowing uninhibited sublimation to take place. This treatment provided enough mechanical advantage whilst the cyclododecane was present in solid form to allow treatment to take place without disrupting the fragile surface.

[33] Rowe S Roziek C (2008) p24
[34] Muros V (2004) p76
[35] Rivers & Umney (2003) p487
[36] Rivers & Umney (2003) p574

The British Museum famously used cyclododecane in this way to secure unstable ceramic surfaces prior to transportation from their old basement store to the new storage area within the Department with *"promising results."*[37]

Archaeologists were at the forefront of the use of this material as a temporary consolidant during excavation of objects. There are several well-documented examples of the use of cyclododecane to impart structural integrity during the removal of artefacts from burial sites. In a 2005 study the British Museum funded research that noted; *"hot melt cyclododecane or aerosol spray over delaminated or flaking areas worked better than cyclododecane in hexane, which was more effective for consolidating a powdery areas with no flaking."*[38]

Archaeologists have also used cyclododecane as an alternative to synthetic resins for the consolidation of archaeological metalwork before cleaning with air abrasion with mixed success.[39] Ceramic restorers have been known to use it in this way to fix shards of objects temporarily into place during restoration.

Application methods

Various application methods are available to the conservator for the application of this material. Tests have shown that the application method can affect how deeply cyclododecane penetrates into a porous substrate, in solution has been found to travel further into samples than when applied in pure form using the hot melt method. [40] In a 2006 research paper investigating cyclododecane as a temporary consolidant the rate of sublimation was cited as being an important part of any temporary consolidation regime.[41] The method of application has also been found to have a significant effect on the rate of sublimation along with surrounding air temperature, humidity and ventilation. Cyclododecane applied using the hot melt method can take two or three times as long to sublimate than the same material applied in solution.[42]

[37] Cleere(2005) p26
[38] Cleere DC (2005) p27
[39] Rowe S Roziek C (2008) p24
[40] Muros V (2004) p77
[41] Cagna M & Riggiardi D (2006) p90
[42] Muros V (2004) p78

Application as a spray

Commercially available aerosol preparations are available, using an extremely volatile solvent mixture of methane and butane which also acts as a propellant. The recommended application distance for these sprays is a mere 3 to 4 cm and it is generally accepted that the resultant film is thinner and less hydrophobic than that applied by as a pure melt. An aerosol uses a solvent and a heated spray gun generates a spray of melted cyclododecane - both methods produce a non-penetrating film. The film produced is amorphous rather than crystalline probably as a result of the product hitting the surface in very small droplets. [43]

Application as a melt

In a study undertaken in 1999 it was established that the film formed after the evaporation of a solvent was composed of large crystals, whereas cyclododecane applied in a 100% melt produced smaller crystals in the resulting film.[44] A more recent study undertaken in 2008 [45] stated that cyclododecane formed the densest film when applied in a molten state, probably due to the rapid cooling, closely packed crystals are able to form providing an extremely successful hydrophobic film. It seems that when cyclododecane is dissolved in a solvent, solidification is inhibited causing a larger crystals to form - this produces a less dense and effective film.

Application as a solution

Cyclododecane can be dissolved in a range of non-polar solvents including saturated, aromatic and halogenated hydrocarbons such as methylbenzene (toluene), dimethylbenzenes (xylene), cyclohexane, petroleum ether (benzene), pentane, octane, iso-octane, naptha, dichloromethane and white spirit.[46] A 2002 study published in "The Book and Paper Group Annual" noted that *"a film produced from a solvent solution is thought to produce a thinner and perhaps less dense film than that produced from a melt and therefore it sublimates more quickly than a melted application"*[47]

[43] Rowe S Roziek C (2008) p20
[44] Bruckle I, Thornton P, Nichols K, Strickler G (1999) p4
[45] Rowe S Roziek C (2008) p18
[46] Rowe S Roziek C (2008) p19
[47] Nichols K, Mustalish R (2002) P82

Ethical Considerations

The Conservator will usually construct a hierarchy of importance to aid the decision-making process, *"juxtaposing knowledge with another weighting factor often unconsciously",*[48] and always aiming for a maximum degree of *retreatability* in any given treatment regime. The preservation of the visual and structural integrity of the historic surface is always towards the top of this list of importance.

Fig 2

A saturated surface[49]

The saturated **(**Fig 2) (polished or finished) decorative surfaces of an object have often been refinished as part of a legitimate and necessary maintenance regime rendering these surfaces frequently less reliable as historic documents.

The dry surfaces **(**Fig 3) that tend to be out of sight can reveal the most evidence of the intangible cultural heritage of a given object; construction lines and other subtle visual clues are all evidence of construction techniques and sometimes more specifically the tool marks left behind betray the tools used in a piece's manufacture.

Fig 3

An un-saturated surface[50]

Saturating a dry, friable or untreated surface inadvertently through accidental pollution invariably involves a colour change and an increase in surface gloss. In a study undertaken in 2006 investigating consolidation of non-archaeological wood this was described as *"chromatically altering the treated zones."*[51]

[48] Caple (2000) p8
[49] A 17th century Italian chair applied and gilded decorative fleur
[50] A 17th century Italian chair applied and gilded decorative fleur
[51] Maritato R (2006) p150

Any residue left behind after sublimation has taken place is also a contentious issue; Elizabeth Jaegers stated that *"a fundamental precondition for the use of [cyclododecane] is the complete and absolutely residue-free evaporation, complete evaporation is guaranteed only when the materials are free from impurities."*[52] Although the sublimation of pure cyclododecane is theoretically absolute, minor impurities (caused by variations in molecular geometry) resulting from the manufacturing process have been found in some studies to form saturated hydrocarbons that do not sublimate. These saturated hydrocarbons can produce a residue that may remain in situ for a longer period of time.[53] Extensive research carried out by Vanessa Muros and John Hirx in 2004 alludes to this possibility, but concludes that any residues remaining after sublimation were "*extremely minimal and not harmful to an object*."[54]

The results of other studies however indicate the opposite; in a study carried out in 2007 using cyclododecane on canvas paintings as a temporary consolidant stated that *"the sublimation of CDD was complete in 11 days without leaving residue is detectable with Raman spectroscopy and SEM."*[55]

One can only conclude when juxtaposing a large range of previous studies that any residue remaining is likely to be minimal and relatively inconsequential.

In the context of the development of a new technique these practical limitations may well fall within the realms of acceptability as *"the notions of removability and retreatability are a subtle acknowledgement that reversibility is an unattainable goal."*[56] If surface pollution of the object is apparent this will obviously need to be weighed against the tangible structural benefits provided by the overall treatment.

[52] Jaegers E (2009) p1
[53] Muros V (2004) p79
[54] Muros V (2004) p79
[55] Kuvvetli F (2007) p32
[56] Munos-Vinas S (2005) p188

Test methodology

In a 2004 investigation into the use of cyclododecane in paper conservation it was noted that sublimation of cyclododecane *"is encouraged with increased air exchange over the surface and with increased temperature"*[58]. It was therefore necessary to control and monitor temperature and relative humidity along with containing any vapour released. It was decided to carry out testing in a laboratory fume cabinet; an Air-One FC-640 was chosen due to its compact size and subsequent controllability.[59] It would be counter-productive to evacuate the vapour continuously during the maintenance of a high ambient temperature therefore it was decided to set the fume cabinet on a timer extracting for only 15 minutes per hour.

Image 0-6

The fume cabinet[57]

With the far-reaching study undertaken by Vanessa Muros and John Hirx quoting unaided sublimation of cyclododecane taking up to 87 days when applied to terracotta[60] the sublimation rate from porous surfaces is expected to be much slower than that from nonporous surfaces. It must be stressed however that this 87 days was in an extremely cool environment with no aid to sublimation whatsoever. In some studies it has been noted that cyclododecane can sublimate away in as little as 24 hours when applied to historic paper objects.[61]A study undertaken in 2000 testing the sublimation rates of cyclododecane from limestone and sandstone suggest a much more palatable 6 to 9 days as a likely rate.[62]

[57] Air-One FC-640 laboratory fume cabinet (West Dean College)
[58] Nichols K, Mustalish R (2002) P81
[59] Generic Images (image appendix) Image 0-6
[60] Muros V (2004) p79
[61]Szuhay B (2005) p105
[62] Stein R, Kimmel J, Marincola M, Klemm F (2000) p361

Taking into account the full body of research it was decided to carry out the experiment at the highest reasonably acceptable temperature and the lowest relative humidity that the wooden artefact would be expected to exist in during a conservation treatment. In order to increase the rate of sublimation the test samples were illuminated by a 60 W tungsten filament bulb present in the fume cabinet throughout the test process.

Image 0-7

Environmental monitor[63]

Preliminary testing established this provided enough heat to simulate a possible conservation treatment environment established specifically for maximum sublimation rate.

A *Tiny Tag*[64] environmental monitor was present at all times in the fume cabinet capturing data. The temperature and relative humidity of the test environment was monitored over the full period of the experiment.

[63] Tiny Tag® environmental monitor
[64] Generic Images (image appendix) Image 0-7

The substrate model

In order to model a substrate similar to that generally in need of consolidation various timbers were considered. Balsawood was chosen due to its extremely low specific gravity, and therefore low proportion of timber per centimetre cubed;

Timber – (seasoned & dry)	Kg per cubic metre
Afromosia	705
Apple	660 - 830
Ash, black	540
Ash, white	670
Aspen	420
Balsa	**170**
Bamboo	300 - 400
Birch (British)	670
Cedar, red	380
Cypress	510
Douglas Fir	530
Ebony	960 - 1120
Elm (English)	600
Elm (Wych)	690
Elm (Rock)	815
Iroko	655
Larch	590
Lignum Vitae	1280 - 1370
Mahogany (Honduras)	545
Mahogany (African)	495 - 850
Maple	755
Oak	590 - 930
Pine (Oregon)	530
Pine (Parana)	560
Pine (Canadian)	350 - 560
Pine (Red)	370 - 660
Redwood (American)	450
Redwood (European)	510
Spruce (Canadian)	450
Spruce (Sitka)	450
Sycamore	590
Teak	630 - 720
Willow	420

[65]

[65] http://www.csgnetwork.com/specificgravwdtable.html

Balsa adequately models the type of substrate encountered after timber has suffered insect damage, often from the common furniture beetle (Anobium punctatum). When degraded in this way a large amount of material is lost in the process, leaving tunnels (galleries) similar to the large and open porous nature of balsawood.

image 3A-25

Balsawood test block[66]

The seminal article published by Wang & Schniewind in 1985 states that *"permeability is a basic characteristic of wood determining the flow of fluids during the impregnation process -- in general, permeability is higher in the longitudinal than in the transverse direction."*[67] Using this data the preparation of the balsawood test blocks was standardised, with the orientation of the grain running vertically through the block to allow maximum absorption on each face.[68] The preparation of the surfaces of these blocks was of crucial importance if realistic results were to be extracted. Various different types of surface preparation were compared for the two faces of the cube that would absorb cyclododecane and dyed consolidant preparation respectively. A planed surface, a band-sawn surface, a circular-sawn surface and finally a sanded surface were compared.[69]

To provide a realistic model it was decided to use the sanded surface, prepared with 240 grit abrasive as it was found that this finishing regime produced a smooth and repeatable surface texture that would provide results comparable with real object surfaces.[70] As each series of blocks was prepared duplicate blocks were produced using adjoining balsa to provide control blocks for comparison at the end of the test.[71] At every stage of the preparation visual discrimination was used to assure each control block was comparative to its relative test block, any natural anomalies in the timber were dealt with at that stage.

[66] Balsawood test block 5mm x 5mm x 10mm
[67] Wang Y & Schniewind AP (1985) p3
[68] Test 3 (image appendix) Image 3A-25
[69] Generic Images (image appendix) Image 0-0, Image 0-1, Image 0-2, Image 0-3
[70] Generic Images (image appendix) Image 0-3
[71] Generic Images (image appendix) Image 3A-26 and Image 3A-27

The size of the balsawood blocks in relation to the volume of consolidant was also a crucial factor, preliminary testing was used in order to finalise these factors. If the blocks were too large capillary action would not carry the consolidant all the way through to the other surface; the success or otherwise of the exclusion layer would be impossible to judge. Should the balsawood blocks be too small then the consolidant would be carried through in all samples without cyclododecane applied to the surface, and not carried through in all samples were cyclododecane was applied to the surface as the absorption of cyclododecane also relies on permeability and capillary action in order to gain penetration.

There was an acceptance that this model has some limitations regarding the practical problems likely to be encountered when conserving larger, and more varied object surfaces.

The consolidant model

Butvar B98 was chosen as a standard consolidant of choice, Schniewind & Kronkright found that polyvinyl butyral (Butvar) gave *"the most improvement in strength"*[73] in a 1984 study; it remains a popular choice for conservators today.

image 3A-25

Consolidant preparations[72]

A resin-based consolidation technique generally relies on a solvent vehicle that is colourless, used to dissolve an also colourless resin and carry it into damaged timber. In order to track the passage of a test consolidant through a substrate model it was necessary to investigate colouring the consolidant in order to allow visual identification. Various spirit dyes were compared for solubility and visibility in the preliminary testing phase. It was necessary to use a dye that was equally soluble in IMS and acetone in order print to provide consistent results upon visual examination; many proprietary spirit stains were more soluble in IMS than in acetone.

[72] Butvar B98 at 0% - 5% and 10% concentration
[73] Wang Y & Schniewind AP (1985) p79

Sellaset H was developed by the Ciba-Geigy organisation for the trichromatic dyeing of leather (the use of three primary colours in various proportions in order to produce a range of colours). This dye was chosen as it was found to be equally soluble in both solvents. Various proportions of Sellaset H were compared during preliminary testing and **a ratio of 1%** was found to be the minimum necessary to provide clearly visible evidence of consolidant presence.

Using the previously developed 5 mm thick end grained balsa woodblocks preliminary testing was carried out in order to evaluate the optimum amount of consolidant to be added to each test block. **A volume of 0.2 ml** was found to be a large enough volume of consolidant to saturate the block totally if uninhibited by an exclusion layer.

A range of consolidant strengths was chosen; 0% was included to show the passage of the consolidant vehicle without added consolidant, 5% and 10% solutions accurately model preparation strengths in common use in wooden objects conservation. Wang and Shniewind used a range of 5% to 20%[74] in their study in 1985, however they were using vacuum impregnation in order to drive the consolidant into the timber. Preliminary testing, further research[75] and practical experience suggested 10% to be a suitable maximum for atmospheric pressure impregnation.

The temporary exclusion layer

As previously discussed a large body of previous research suggested that 100% hot melt applied directly would provide the most effective exclusion layer. The use of cyclododecane as an exclusion layer provides similar visual limitations to those previously described for a resin-based consolidant; pure cyclododecane is transparent and does not allow visual discrimination for the purposes of ascertaining presence, penetration and sublimation rate.

image 0-9

Laboratory crucible[76]

[74] Wang & Schniewind (1985) p4
[75] Rivers & Umney (2003) p565
[76] Electromantle laboratory crucible set at 70°C

It was decided that the best method of tracking these factors in the exclusion layer was to treat the cyclododecane using a chemical that would fluoresce under UV microscopy. After research Uvitex OB® (optical brightener) was chosen as a heat resistant, non polar-solvent soluble, chemically stable chemical marker. This fluorescent whitener is used in the paint and plastics industry and provides brighter looking colours for solvent based paints. *"Uvitex OB® can also be used as a tracer in clear coatings."*[77]

After preliminary testing it was found to dissolve readily in cyclododecane, fluorescing under UV microscopy at proportions as low as 0.01%. Uvitex OB® will of course remain present in the timber after the cyclododecane has sublimated, therefore it is unlikely that this chemical marker would be used on a historic object due to this limitation. The cyclododecane at 100%, 90%, 80% concentrations in white spirit was be heated to 70°C in a laboratory crucible in order to provide a standardised application regime.[78]

Once **heated to 70°C each test block was gripped in tweezers and placed face down on the surface of the molten cyclododecane for 3 seconds** to allow full surface saturation to take place. 100%, 90%, and 80% concentrations were chosen as previous research confirmed by preliminary testing showed that lower concentrations of cyclododecane were practically ineffective at providing exclusion of consolidant.

[77] http://www.ciba.com/uvitex_ob_optical_brightener.htm
[78] Generic Images (image appendix) Image 0-9

Fluorescence

Fluorescence has been described as *"a luminescence phenomenon in which electron de-excitation occurs almost spontaneously."* [79]The electromagnetic energy of a photon (of light) is inversely proportional to its corresponding wavelength. Short wavelength *(less than 310 nm[80])* or blue light has a higher energy than red light. Due to these differences the light causes different effects when it interacts with molecules, and molecular bonds. In the ultraviolet-visible region, electronic transitions are mainly observed; caused when a photon of the proper energy is absorbed by a molecule, an electron is excited to higher energy level or shell. For a photon to be absorbed, the energy of the photon must correspond exactly[81] to the difference in energy between the ground state and the excited state to which the electron transfers.

The energy levels of the molecules are related to the types of atoms and how they bonded to one another.[82] The geometry of the molecule as well as its environment is also a factor in the structuring of these quantised energy levels. After the electron has jumped to the excited state, it then almost immediately decays to its ground state with the emission of a photon of visible light, this process is called fluorescence.

[79] http://scienceworld.wolfram.com/physics/Fluorescence.html
[80] http://www.biologie.uni-hamburg.de/b-online/e07/07c.htm
[81] http://www.kentchemistry.com/links/AtomicStructure/PlanckQuantized.htm
[82] http://www.ndt-ed.org/EducationResources/CommunityCollege/Materials/Structure/bonds.htm

UV microscopy

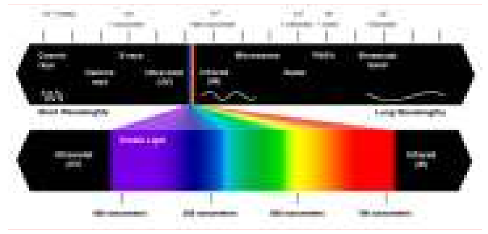

image 0-12

Electromagnetic spectrum[83]

UV microscopy relies on various wavelengths of ultraviolet light illuminating a sample placed on the stage of the microscope in order to visually differentiate often between inorganic and organic materials. At a more advanced level coloured dyes that fluoresce at certain wavelengths can be introduced onto the sample in order to produce quantifiable results and further visual discrimination. One of the great benefits of UV microscopy is that visible light may also be used in conjunction with ultraviolet light providing the researcher with a valuable insight into the material under scrutiny. Photography of UV microscopy samples was captured with a Dinolite® digital camera inserted into the eyepiece of the microscope; all UV microscopy was carried out in a darkened lab to provide standardised results. The surface of the sectioned blocks photographed under UV microscopy was prepared using a standard Laboratory microtome to slice each cross-section.[84]

Photography

A Canon IXUS 60 6-mega Pixel camera mounted onto a bespoke photographic Jig, with a matt background was used to record the results. [86] All photography took place in a darkened room using a fixed 60w tungsten filament light / fixed UV light source for illumination to provide comparable results.

image 0-10

Photographic jig[85]

[83] http://local.content.compendiumblog.com/uploads/user/2af9dc1d-8541-42e4-a91f-6aaf97caf33a/4844a17e-a4fb-4018-9d3a-31dc846044ee/Visible%20spectrum.jpg
[84] Generic Images (image appendix) Image 0-11
[85] Bespoke apparatus designed for standardised photography
[86] Generic Images (image appendix) Image 0-10

Practical Testing

Preliminary test 1

Before the controlled experiment began preliminary testing was carried out in order to ascertain the practicalities of the theoretically developed testing process.

Image 1A-1

Preliminary test 1

With some studies quoting cyclododecane concentrations in solvent as low as 50%[87] and other studies using cyclododecane at 100% concentration (melt)[88] preliminary testing showed that concentrations below 75% were extremely ineffective in providing a continuous film suitable for the exclusion of consolidant. It was decided at this stage to proceed with 80%, 90%, 100% concentrations of cyclododecane in white spirit based on these results.

In order to establish the feasibility of this proposal 5 test blocks were treated with the following combinations;[89]

1. 100% cyclododecane (Image 1-1)
2. 100% cyclododecane + (10% Butvar B98 in IMS(dyed)) (Image 1-2)
3. 00% cyclododecane +(10% Butvar B98 in IMS (dyed)) (Image 1-3)
4. 100% cyclododecane +(0% Butvar B98 in IMS (dyed)) (Image 1-4)
5. 00% cyclododecane +(0% Butvar B98 in IMS (dyed)) (Image1- 5)

   (Test 1 image appendix - Images 1-1 to 1-4)

Preliminary testing on various spirit dyes suitable for tracking the consolidant was carried out at this stage as discussed previously. The relative solubility in the two proposed solvents was a crucial factor in the choice of Sellaset H as a suitable dye, as previously mentioned a 1% concentration was chosen.

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87 Cagna M & Riggiari D (2006) p92
88 Cleere DC (2005) p27
89 Test 1 Images (image appendix) Image 1A-1

The fully developed testing process;

1. Prepared cubes **10 mm x 10 mm x 5 mm of balsawood** were placed face down for **3 seconds** in cyclododecane as a melt applied at **70°C at 100%, 90% and 80%** as a heated solute dissolved in white spirit. (A study undertaken in 2008[90] stated that cyclododecane formed the densest film when applied in a molten state).

2. For each set of test blocks an identical control block taken from the same section of balsa was prepared for comparison at the conclusion of the test.

3. Three viscosities of consolidant (**Butvar B98 at 0%, 5%, 10% in IMS and acetone**) dyed with **Sellaset H (at 1%)** was prepared, **with 0.2 ml per block** introduced from below using laboratory pipette.

4. Two complete duplicate sets of the above preparations were prepared; - **one containing Uvitex OB®** to allow UV identification - **one without Uvitex OB®** as a control.

5. Glass microscope slides were coated with each test preparation of cyclododecane and allowed to sublimate, any residue remaining after sublimation will then be visible - providing evidence of its purity.

6. Some repeat samples were prepared to allow sectioning for UV microscopy and to increase the result reliability.

7. After a full solvent evaporation has taken place the cubes will then photographed to establish the degree of surface contamination. Some were sectioned and the progress of the consolidants examined under UV microscopy to establish the success of the exclusion layer. The results were judged visually based on obvious consolidant presence on the surface. As the sublimation rate cannot be quantified using this experimental process surface examination relied on comparative photography.

8. After a full solvent evaporation and sublimation of the cyclododecane had taken place the cubes were compared to the control blocks mentioned previously. Any visual difference caused by the addition / sublimation of the cyclododecane was noted.

[90] Rowe S Roziek C (2008) p18

Preliminary test 2

Having established the practical feasibility of the testing regime in test 1, test 2 covered the full range of test consolidants and variations of the exclusion layer. Uvitex OB® was not used in this part of the testing process as UV photography and microscopy was not undertaken at this stage. Full environmental monitoring took place to record temperature and humidity within the test environment.

Image 2A-2

Preliminary test 2

Visual examination and photography of the test blocks established whether or not trends were appearing in the results. Preliminary test 2 ran for enough time for solvent vehicle containing the consolidant resin to evaporate providing a stable result suitable for photography. The complete evaporation of the solvent was necessary as only when the solvent had completely evaporated did the passage of the consolidant through the substrate stop.

(0% Uvitex OB®)	100% CDD - MELT	90% CDD + WHITE SPIRIT MELT	80% CDD + WHITE SPIRIT MELT
Butvar 98 +IMS@ **0%**	**1** (Image 2-1)	**2** (Image 2-2)	**3** (Image 2-3)
Butvar 98 +IMS@ **5%**	**4** (Image 2-4)	**5** (Image 2-5)	**6** (Image 2-6)
Butvar 98 +IMS@ **10%**	**7** (Image 2-7)	**8** (Image 2-8)	**9** (Image 2-9)
Butvar 98 +Acetone@ **0%**	**10** (Image 2-10)	**11** (Image 2-11)	**12** (Image 2-12)
Butvar 98 +Acetone@ **5%**	**13** (Image 2-13)	**14** (Image 2-14)	**15** (Image 2-15)
Butvar 98 +Acetone@ **10%**	**16** (Image 2-16)	**17** (Image 2-17)	**18** (Image 2-18)

(Test 2 image appendix - Images 2-1 to 2-18)

Full Control test 3

		
Image 3A-2	**Image 3A-5**	**Image 3A-21**
18 of the test blocks	**….Viewed under UV light**	**….and under UV microscopy**

After analysing the results are preliminary test 2, test 3 further established trends in the relationship between the purity of the exclusion layer and the proportion of consolidant dissolved in the solvent. Test 3 was divided into two parts; part A consisted of cyclododecane with an added 0.01% Uvitex OB® in order to allow the penetration of the cyclododecane to be observed under UV microscopy (samples 19, 20 and21).

A similar set of blocks (taken from the same balsa) were prepared as a control. These blocks were placed alongside the treated samples in the test environment and served as a visual surface and texture comparison upon completion of the testing. Full environmental monitoring took place to record temperature and humidity within the test environment.

A set of 6 blocks 52-57;(Image 3-52, Image 3-53, Image 3-54, Image 3-55, Image 3-56, Image 3-57) were treated with the full range of consolidants as a control for visual comparison at the completion of the test -- in order to ascertain the degree of surface pollution without the cyclododecane exclusion layer.

The glass slide samples (25, 26, 27, 49, 50 and 51) were viewed visually and photographed to provide evidence of residue left behind after sublimation was complete. These samples were **not** viewed under UV light due to the fact that the Uvitex OB® was still present after the cyclododecane has sublimated as previously explained.

A (+ 0.001% Uvitex OB®)	**100% CDD - MELT**	**90% CDD + WHITE SPIRIT MELT**	**80% CDD + WHITE SPIRIT MELT**
Butvar 98 +IMS@ **0%**	**1** (Image 3-1)	**2** (Image 3-2)	**3** (Image 3-3)
Butvar 98 +IMS@ **5%**	**4** (Image 3-4)	**5** (Image 3-5)	**6** (Image 3-6)
Butvar 98 +IMS@ **10%**	**7** (Image 3-7)	**8** (Image 3-8)	**9** (Image 3-9)
Butvar 98 +Acetone@ **0%**	**10** (Image 3-10)	**11** (Image 3-11)	**12** (Image 3-12)
Butvar 98 +Acetone@ **5%**	**13** (Image 3-13)	**14** (Image 3-14)	**15** (Image 3-15)
Butvar 98 +Acetone@ **10%**	**16** (Image 3-16)	**17** (Image 3-17)	**18** (Image 3-18)
Image 3A-2 ↑ Image 3A-6 ↓			
Sectioned and examined	**19** (Image 3-19)	**20** (Image 3-20)	**21** (Image 3-21)
NO consolidant	**22** (Image 3-22)	**23** (Image 3-23)	**24** (Image 3-24)
Glass slide	**25** (Image 3-25)	**26** (Image 3-26)	**27** (Image 3-27)
Image 3A-3 ↓			
B (NO Uvitex OB®)	**100% CDD - MELT**	**90% CDD + WHITE SPIRIT MELT**	**80% CDD + WHITE SPIRIT MELT**
Butvar 98 +Ethanol@ **0%**	**28** (Image 3-28)	**29** (Image 3-29)	**30** (Image 3-30)
Butvar 98 +Ethanol@ **5%**	**31** (Image 3-31)	**32** (Image 3-32)	**33** (Image 3-33)
Butvar 98 +Ethanol@ **10%**	**34** (Image 3-34)	**35** (Image 3-35)	**36** (Image 3-36)
Butvar 98 +Acetone@ **0%**	**37**(Image 3-37)	**38** (Image 3-38)	**39** (Image 3-39)
Butvar 98 +Acetone@ **5%**	**40** (Image 3-40)	**41** (Image 3-41)	**42** (Image 3-42)
Butvar 98 +Acetone@ **10%**	**43** (Image 3-43)	**44** (Image 3-44)	**45** (Image 3-45)
NO consolidant	**46** (Image 3-46)	**47** (Image 3-47)	**48** (Image 3-48)
Glass slide	**49** (Image 3-49)	**50** (Image 3-50)	**51** (Image 3-51)
C (Control blocks)	**0% CDD**		
Butvar 98 +IMS@ **0%**	**52** (Image 3-52)		
Butvar 98 +IMS@ **5%**	**53** (Image 3-53)		
Butvar 98 +IMS@ **10%**	**54** (Image 3-54)		
Butvar 98 +Acetone@ **0%**	**55** (Image 3-55)		
Butvar 98 +Acetone@ **5%**	**56** (Image 3-56)		
Butvar 98 +Acetone@ **10%**	**57** (Image 3-57)		

(Test 3 image appendix - Images 3-1 to 3-57)

Test results

It was noted that during the addition of the consolidant using the laboratory pipette due to pooling and splashing some contamination of the edges of the test blocks took place. Therefore when judging the success of the exclusion layer on the completed test blocks approximately 1mm around the edge of each test block was considered unrepresentative within the remit of this experiment;

Preliminary test 1- results

Image 1-2

100% cyclododecane + 10% Butvar (IMS)

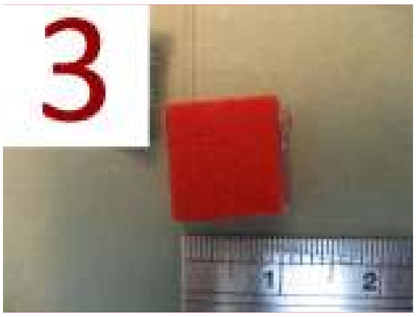


Image 1-3

0% cyclododecane + 10% Butvar (IMS)

It can be seen from these first test images that the application of cyclododecane to the face of block 2 (Image 1-2) when seen in comparison to block 3 (Image 1-3)seems to have had a significant effect on the amount of surface contamination on the block. A similar pattern can be seen when comparing blocks 4&5 (Image 1-4 and 1-5); with no consolidant added to the solvent vehicle it seems the cyclododecane may be less effective in inhibiting the passage of the solvent.

Preliminary test 2- results

Test 2 was much broader ranging with full environmental monitoring. The test ran for 14 days, broadly considering *"11 days"*[91] taken in a 2007 study as a good average of published expected sublimation rates. Through observation it was noted that evaporation of the consolidant solvent seemed to be complete within the first 2 to 4 days, the test was allowed to run for the full 14 days by which time all sublimation seemed to have taken place.

As we can see from the tiny tag data (Image 2A-1) the test environment was largely within the range of acceptability for a wooden artefact undergoing conservation treatment. The temperature ranged from 21.5°C to 28.5°C with the usual cyclic trend associated with day and night temperature fluctuations. The relative humidity remained fairly constant in the 40% to 45% range, not an ideal storage environment but unacceptable short-term conservation studio scenario.

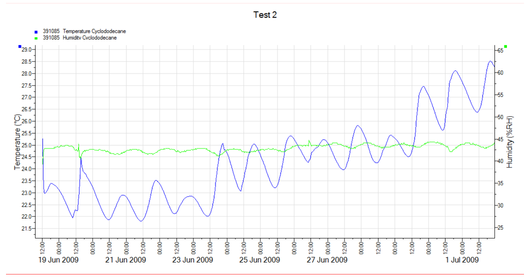
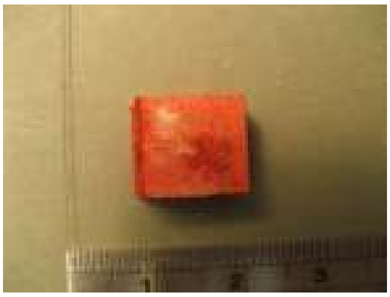
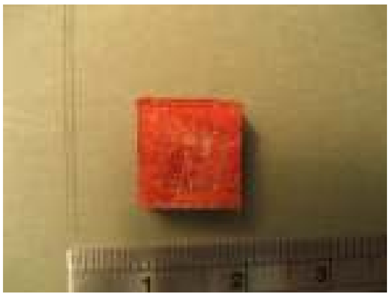


Image 2A-1

Environmental data

[91] Kuvvetli F (2007) p32

		
Image 2-1	**Image 2-2**	**Image 2-3**
100% CDD + 0% Butvar (IMS)	**90% CDD + 0% Butvar (IMS)**	**80% CDD + 0% Butvar (IMS)**

The 18 blocks were all treated using the fully developed test regime described previously. It was possible to see from comparison of this set of blocks a significant trend emerging. Blocks 1, 2 and 3 (Images 2-1, 2-2 and 2-3) showed a clear difference in the amount of surface contamination. Block 1 (Image 2-1) had virtually no surface contamination whereas blocks 2 and 3 (Images 2-2 and 2-3) had significant surface contamination. These blocks were treated with 0% Butvar B98 in IMS, the lowest viscosity of the three regimes of IMS-based consolidant.

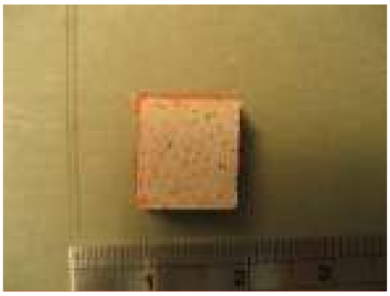
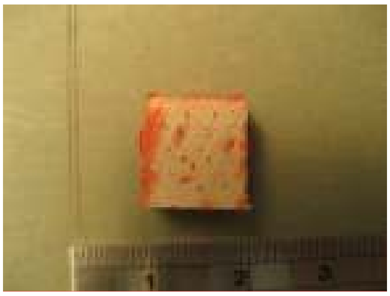
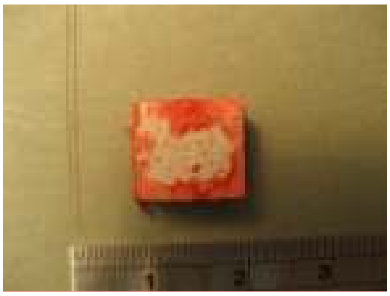
		

Image 2-4	**Image 2-5**	**Image 2-6**
100% CDD + 5% Butvar (IMS)	**90% CDD + 5% Butvar (IMS)**	**80% CDD + 5% Butvar (IMS)**

The next 3 blocks (Images 2-4, 2-5 and 2-6) showed a clearer trend with the first block (treated with 100% cyclododecane melt) having virtually no surface contamination, the second one in the series (treated with 90% cyclododecane melt) had slightly more surface contamination, and the third block (treated with 80% cyclododecane melt) even more surface contamination. These blocks were treated with 5% Butvar B98 in IMS, the medium viscosity of the three preparations of IMS-based consolidant used here.

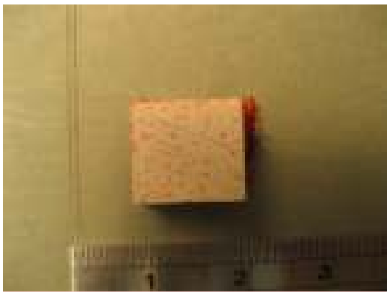
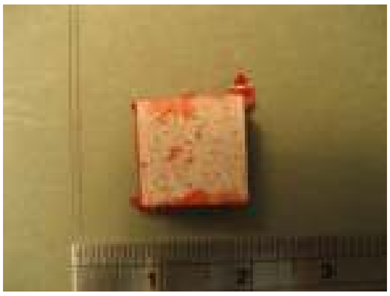
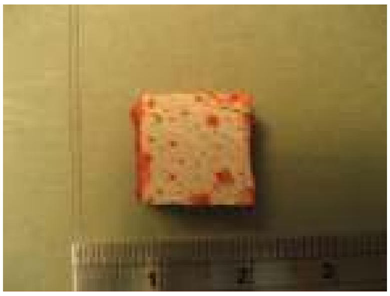
		

Image 2-7	**Image 2-8**	**Image 2-9**
100% CDD + 10% Butvar (IMS)	**90% CDD + 10% Butvar (IMS)**	**80% CDD + 10% Butvar (IMS)**

The next 3 blocks in the series (Images 2-7, 2-8 and 2-9) seemed to repeat this trend with the first block (Image 2-7) (treated with 100% cyclododecane melt) seeming to resist the 10% Butvar B98 in IMS more effectively than the second block (Image 2-8) (treated with 90% cyclododecane melt), and again more effectively than the third block (Image 2-9) (treated with 80% cyclododecane melt).The trend as described above could be seen even more clearly in the second set of test samples, those with Butvar B98 dissolved in acetone at 0%, 5% and 10% respectively. If we again look at each set of three blocks in sequence the same pattern emerged.

At the conclusion of the test blocks which had suffered little or no surface contamination (Image 2-1, Image 2-4, Image 2-7, Image 2-13 and Image 2-16) were visually indistinguishable from the previously mentioned control blocks prepared for each test (Image 3A-26 and Image 3A-27) which had not been treated with cyclododecane.

Full control test 3 - results

Test 3 was much broader ranging still, repeating the range of cyclododecane melts along with the consolidant preparations used in test 2. In addition to this a set of test blocks were duplicated with the addition of Uvitex OB® added at 0.01% to the cyclododecane to allow the characteristics of the exclusion layer to be much more fully investigated. Test 3 also included microscope slides coated with each of the 3 cyclododecane preparations to provide data showing the speed and integrity of the sublimation process.

We can see from can see from the tiny tag data (Image 3A-1) the test environment was still largely within the range of acceptability for a wooden artefact undergoing conservation treatment. The temperature ranged from 22°C to 28.5°C with the usual cyclic trend associated with day and night temperature fluctuations. The relative humidity remained fairly constant in the 41% to 46% range, not an ideal storage environment but again an acceptable short-term conservation studio scenario. These 48 blocks were all treated in the fully developed test regime described previously. It was possible to see from comparison of these 48 blocks a significant confirmation of the trend seen emerging in test 2;

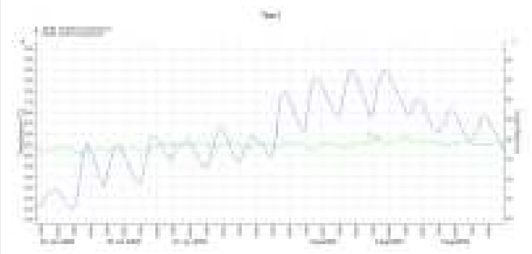


Image 3A-1

Environmental data

When compared with the unprotected control blocks 52 to 57 (Images 3-52, 3-53, 3-54, 3-55, 3-56, and 3-57) the results clearly show that the application of cyclododecane has had a significant effect on the surface contamination of the test blocks. All strengths of consolidant from 0% (pure vehicle) through to 10% (an acceptable maximum practical consolidant strength) showed considerable surface contamination without an applied isolation layer.

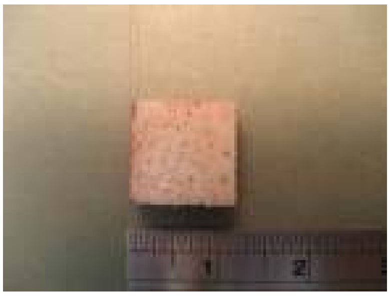


Image 3-1	**Image 3-2**	**Image 3-3**
100% CDD + 0% Butvar (IMS)	**90% CDD + 0% Butvar (IMS)**	**80% CDD + 0% Butvar (IMS)**

Blocks 1, 2 and 3 (Images 3-1, 3-2 and 3-3) showed a clear difference in the amount of surface contamination. Block 1 (Image 3-1) had virtually no surface contamination whereas blocks 2 and 3 (Images 3-2 and 3-3) had significant surface contamination. These blocks were treated with 0% Butvar B98 in IMS, the lowest viscosity of the three preparations of IMS-based consolidant solution.

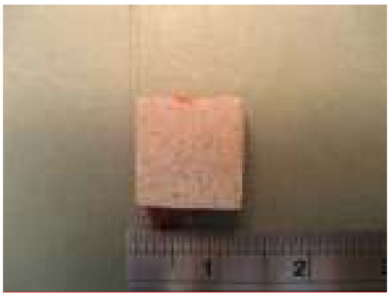


Image 3-4	**Image 3-5**	**Image 3-6**
100% CDD + 5% Butvar (IMS)	**90% CDD + 5% Butvar (IMS)**	**80% CDD + 5% Butvar (IMS)**

The next 3 blocks (Images 3-4, 3-5 and 3-6) again showed a clearer trend with the first block (treated with 100% cyclododecane melt) having virtually no surface contamination, the second one in the series (treated with 90% cyclododecane melt) had slightly more surface contamination, and the third block (treated with 80% cyclododecane melt) even more surface contamination. These blocks were treated with 5% Butvar B98 in IMS, the medium viscosity of the three preparations of IMS-based consolidant.

		
Image 3-7	**Image 3-8**	**Image 3-9**
100% CDD + 10% Butvar (IMS)	**90% CDD +10% Butvar (IMS)**	**80% CDD + 10% Butvar (IMS)**

The next 3 blocks in the series (Images 3-7, 3-8 and 3-9) further repeated this trend with the first block (Image 3-7) (treated with 100% cyclododecane melt) seeming to resist the 10% Butvar B98 in IMS almost as effectively as the second block (Image 3-8) (treated with 90% cyclododecane melt), with a significant increase in contamination for the third block (Image 3-9) (treated with 80% cyclododecane melt).

The trend can be seeing even more clearly in the second set of test samples, those with Butvar B98 dissolved in acetone at 0%, 5% and 10% respectively. If we again look at each set of 3 blocks in sequence the same pattern emerges.

		
Image 3-10	**Image 3-11**	**Image 3-12**
100% CDD + 0% Butvar (acetone)	**90% CDD + 0% Butvar (acetone)**	**80% CDD + 0% Butvar (acetone)**

Blocks 10, 11 and 12 (Images 3-10, 3-11 and 3-12) continue to demonstrate the emerging trend, blocks 13, 14 and 15 (Images 3-13, 3-14 and 3-15) giving an even clearer example - at a very useful consolidant concentration (5% to 10%). Blocks 16, 17 and 18 (Images 3-16, 3-17 and 3-18) showing the same pattern, however not as pronounced as in the previous set of 3 blocks.

The addition of Uvitex OB® to group A sample blocks has provided significant data regarding the characteristics of the cyclododecane exclusion layer.

Image 3A-4

Visible light

Image 3A-5

Ultraviolet light

UV photography was taken 24 hours from the start of the test allowing complete evaporation of any white spirit from the two Uvitex OB® treated cyclododecane preparations.Group A test blocks were photographed under visible light (Image 3A-4) and then under ultraviolet light (Image 3A-5).

The results were striking to say the least; when the trend is view in sets of 3 (treated with 100%, 90% and 80% cyclododecane melt respectively) we saw clearly from the ultraviolet photography (Image 3A-5) that the 100% cyclododecane fluorescence is significantly higher than the 90%, which is significantly higher than the 80% layer.

In this image the same trend is repeated for blocks 4, 5 and 6 (Images 3-4, 3-5 and 3-6) and again on each set of 3 for all 18 samples. Perhaps the most striking are blocks 10, 11 and 12 (Images 3-10, 3-11 and 3-12) which show this trend very clearly.

The control samples, which were not treated with any consolidant also repeat this trend (Image 3A-7). These control samples were duplicated to increase the reliability of the results, with blocks 19, 20, 21, 22, 23 and 24 clearly showing this relationship between cyclododecane purity and surface film volume.

Image 3A-7

Ultraviolet light

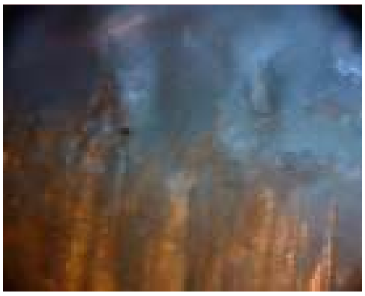
The glass slides 25, 26 and 27 (Image 3A-8, Image 3A-10 and Image 3A-12) when viewed under UV light (Image 3A-9, Image 3A-11 and Image 3A-13) provide a good sublimation rate benchmark that could be used when judging the above samples. These slides when viewed at the end of the test showed the results expected, the cyclododecane seems to have sublimated leaving behind the Uvitex OB® on the surface of the glass.

	
Image 3A-14	**Image 3A-15**
Before sublimation	**After sublimation**

The 3 glass slides coated with the three different solutions of cyclododecane without Uvitex OB® (Image 3A-14, Image 3A-16 and Image 3A-18) do not seem to show any evidence of residual material remaining after sublimation (Image 3A-15, Image 3A-17 and Image 3A-19). It seems that the sublimation is total; research undertaken in 2004 suggests this is a highly possible outcome. [92] In a study carried out in 2007 using cyclododecane on canvas painting as a temporary consolidant *"the sublimation of CDD was complete in 11 days without leaving residue [that was] detectable with Raman spectroscopy and SEM."* [93]

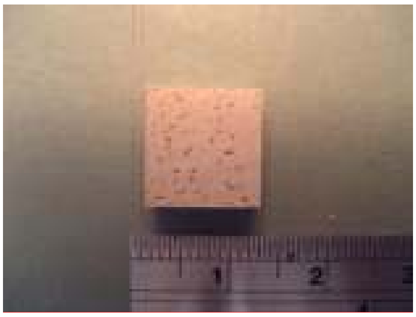
[92] Muros V (2004) p79
[93] Kuvvetli F (2007) p32

When the UV photographic data was juxtaposed with the cross sectional data provided by the UV microscopy the emerging trend was further established;

		
Image 3A-20	**Image 3A-21**	**Image 3A-22**
100% cyclododecane	**90% cyclododecane**	**80% cyclododecane**

The 3 cross sections from blocks 19, 20 and 21 (Image 3A-20, Image 3A-21 and Image 3A-22) seemed to show a clearly demarcated division between the balsa- wood and the exclusion layer for 100% cyclododecane (Image 3A-20), slightly more penetration and less film integrity for 90% cyclododecane (Image 3A-21) and even more penetration and film disruption for the 80% cyclododecane (Image 3A-22).

At the conclusion of the test the blocks which had suffered little or no surface contamination (Image 3-1, Image 3-4, Image 3-7, Image 3-10, Image 3-13, Image 3-16, Image 3-19, Image 3-20, Image 3-21, Image 3-22, Image 3-23, Image 3-24, Image 3-31, Image 3-34, Image 3-37, Image 3-40, Image 3-43, Image 3-44, Image 3-45, Image 3-46, Image 3-47 and Image 3-48) were visually identical to the previously mentioned control blocks prepared for each test (Image 3A-26 and Image 3A-27) which had not been treated with cyclododecane.

	
Image 3-1	**Image 3A-26**
100% cyclododecane	**0% cyclododecane**

Examples of this comparison could be seen in (Image 3A-29 compared to Image 3A-30 - Image 3A-31 compared to Image 3A-32 - Image 3A-33 compared to Image 3A-34). Observation of all surfaces that had been exposed to cyclododecane showed no difference in texture or colour when compared to their equivalent control blocks that had been in an identical environment but left untreated.

Results Analysis

Preliminary test 1- analysis

The basic criteria for the further development of the testing modus operandi were met. A standardised test block preparation was arrived at; a suitable volume of consolidant had been established for each block. The method and temperature for the application of the cyclododecane melt had been proved to be practicable. The test environment had been established and standardised photography methods had been satisfactorily achieved. From this simple preliminary test it seemed evident that not only the application and purity of the cyclododecane applied to the face of the block, but also the proportion of consolidant resin to its solvent vehicle were likely to have an effect on the outcome of this test.

Preliminary test 2- analysis

The second generation of testing proved equally successful; the test environment was successfully monitored and found to be within reasonable limits. The successful application of the consolidant and exclusion layer had shown consistent results in line with researched expectations.

A trend seemed to be emerging that the less adulterated the cyclododecane - the more protection it offered to the surface it is applied to, with 100% cyclododecane melt being the most effective in every case. This result was not altogether unexpected as studies undertaken as recently as 2004 show *"the melt was found to be a more effective barrier than the solutions."*[94] Another trend was also emerging, that of the three differing concentrations of consolidant seeming to have an effect on the results. It appeared that in these first 18 blocks the cyclododecane was not as effective at resisting the 0% Butvar B98 (100% solvent) preparation but seemed much more effective at resisting the 5% Butvar B98 consolidant and even more effective at resisting the 10% Butvar B98 consolidant solution.

[94] Muros V (2004) p82

These early results from test 2 seemed to imply that there were 2 factors involved in the relationship between the cyclododecane exclusion barrier and the alcohol-based consolidant. It seemed from these results that there may be an ideal balance between the purity of the exclusion coat and the viscosity of the consolidant solution.

Full control test 3 - analysis

The third generation of testing also proved successful; the expected trend consistently continued in line with expectations. Examination under ultraviolet light using photography and microscopy yielded further information regarding the behaviour of the cyclododecane. New data provided by this ultraviolet imaging significantly added to the body of evidence providing a conclusion to this research. The trend was further confirmed that the less adulterated the cyclododecane upon application - the more protection it offers to the surface it is applied to; with 100% cyclododecane melt again being the most effective in every case.

When compared with the control blocks 52 to 57 (Images 3-52, 3-53, 3-54, 3-55, 3-56, and 3-57) the results clearly showed a significant effect in line with expectations when cyclododecane is applied to a porous surface such as this.

	
Image 3-52	**Image 3-7**
0% cyclododecane	**100% cyclododecane**

The control blocks showed a 100% contamination (Image 3-52), in line with previous research that concluded; *"darkening of the wood after impregnation is inevitable"*[95] when consolidating without isolating the surface layer. These control blocks were duplicated to double the reliability of the results; (Image 3A-28).

[95] Grattan DW (1980) p2

The ultraviolet photography (Image 3A-5) of group A provided a strong indication that the physical characteristics of a cyclododecane exclusion coat is significantly affected by the addition of a non-polar solvent - in this case white spirit. This significant difference in fluorescence characteristics could be interpreted in two ways;

1. The decrease in fluorescence as the amount of solvent increases shows a less continuous surface film.

2. The decrease in fluorescence as the amount of solvent increases indicates more penetration leaving less cyclododecane present at the surface.

Of course it is quite likely in view of previous research that both of these hypotheses could co-exist and be correct;

The data provided by the cross-sectional UV microscopy (Image 3A-20, Image 3A-21 and Image 3A-22) suggested that this was likely. As mentioned previously the addition of solvent to cyclododecane had been shown in previous studies to affect crystal size upon solidification.[96]

[96] Bruckle I, Thornton P, Nichols K, Strickler G (1999) p4

The small anomalies visible on the surface of some blocks (the small spots where the exclusion layer seems to have failed) could possibly be due to the large visible pores present in the timber, continuing up to the surface (Image 3A-24). The crystalline nature of cyclododecane is likely to have its limitations in its ability to span relatively large voids. This natural characteristic of the timber requiring treatment will need to be taken into consideration when considering the future practical application of this technique. Should this be the case the flight holes caused by common furniture beetle damage to wooden artefacts are likely to exhibit a similar disruption to the exclusion layer.

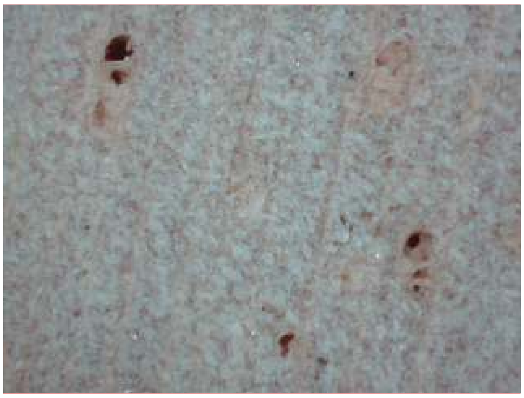


image 3A-24

Large pores[97]

Finally -- but probably most importantly as with tests 1 and 2, test 3 showed consistent results confirming that cyclododecane did not visually affect the appearance or texture of any tested blocks. When compared with the control blocks consolidated without an exclusion coat (Image 3A-25 and Image 3A-26) the effectiveness of the cyclododecane was clearly visible.

[97] Balsawood test block at 50x magnification

Additional Test 4

It was decided at the conclusion of test 3 to carry out some additional testing, taking the results of the previous testing and applying them to a more realistic conservation scenario. The model used to test the original hypothesis was adequate for its purpose; however the effect of a 100% cyclododecane melt applied at 70°C on a commonly occurring dry timber surfaces on historic objects is of interest in the future practical application of this technique. The fully seasoned surface was approximately 10 years old, with the historic surfaces over 100 years of age. In total 4 larger test blocks were prepared;

1. A fully seasoned dry pine surface
2. An historic dry pine surface
3. An historic dry oak surface
4. An historic dry mahogany surface

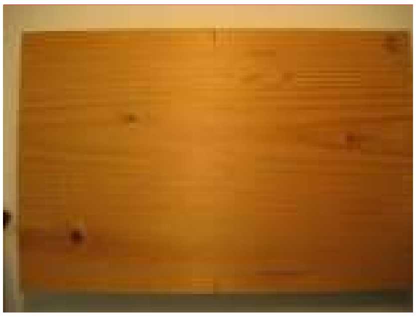


Image 4-1

dry pine surface

Image 4-2

historic dry pine surface

Image 4-3

historic dry oak surface

Image 4-4

historic dry mahogany surface

In order to provide results comparable with the experimentation previously undertaken each of the test samples had 100% cyclododecane melt added to the surface at 70°C. The 100% cyclododecane was then re-melted in situ using a hot air gun and kept molten for three seconds; as in full control test 3.

The test samples were then placed in the same test environment as was used for full control tests 2 and 3 and allowed to sublimate in the same way. The test samples were photographed at the beginning of the test before treatment with cyclododecane (Image 4-1, 4-2, 4-3 and Image 4-4). The test samples were again photographed immediately after application of cyclododecane (Image 4-5, 4-6, 4-7 and Image 4-8), and again after sublimation had taken place (Image 4-9, 4-10, 4-11 and Image 4-12).

Additional Test 4 Results

The test results from the additional testing show a clear trend. As we can see from the example below - the dry pine surface - the application and subsequent sublimation of cyclododecane to the dry surface has not had a detrimental effect on the visual appearance of the surface. Visually it seems that as with full control test 3 the cyclododecane has completely sublimated.

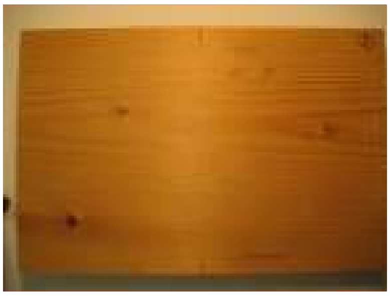
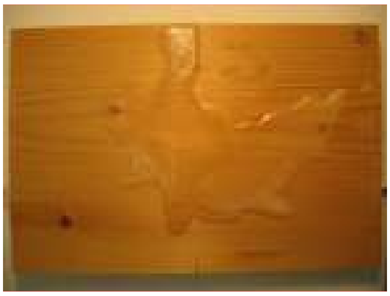
	

Image 4-1	**Image 4-5**	**Image 4-9**
Dry pine surface	**+100% cyclododecane**	**After sublimation**

The results are the same for all 4 timber samples, indicating that the application of pure cyclododecane as a melt at 70°C does not seem to have an effect on the visual appearance of a dry timber surface.

		
Image 4-4	**Image 4-8**	**Image 4-12**
historic dry mahogany surface	**+100% cyclododecane**	**After sublimation**

Conclusions

The testing process has verified the expected outcomes and added to the continuum of information regarding the use of cyclododecane within the conservation profession. Visual observations throughout the course of preliminary test 2 and full test 3 suggested that cyclododecane applied as a melt at 70°C produced a surface coating approximately 1 mm thick. This surface coating seemed to sublimate completely in 7 to 10 days. This suggests a practical working time comparable with this in which to complete the consolidation treatment. Sublimation will most likely take place from the surface downwards therefore the cyclododecane present underneath the surface will be the last to sublimate; thus working time may be slightly longer than the visible results suggest.

With the exception of 2 blocks (Images 3A- 29 and 3A-30) out of a total of 65 (excluding control blocks and duplicates) a consistent significant trend emerged from this testing process; the proportion of solvent present in the cyclododecane was a significant factor in its effectiveness as an exclusion coat. The experimental data seems to show that a 100% cyclododecane melt is capable of providing an effective exclusion layer that may have a significant effect on reducing surface contamination from resinous consolidants in practice. The testing also indicates that the consolidant solution strength chosen to use with this exclusion layer is as important as the composition of the exclusion layer itself. If the proportion of resin consolidant present in the solution is too low the consolidant may not be resisted by the exclusion layer as effectively. The control blocks (Image 3A-28) show the effectiveness of the exclusion layer compared to the results achieved when not using an exclusion layer, with 100% contamination of the surface.

It seems that the relationship between exclusion layer and consolidant is vital to the success of the overall treatment. Most importantly throughout the experimentation the results have been consistent with cyclododecane not having any impact on texture or surface colour. As previous research suggested *"no residual traces of cyclododecane or of any impurities or additives were visible on the treated glass slides after sublimation"*[98] - this research seems to show extremely limited ethical implications regarding the application of cyclododecane to an historic object surface.

[98] Stein R, Kimmel J, Marincola M, Klemm F (2000) p362

These results are shown in test 3 agree with the additional test 4 results carried out on larger dry timber surfaces. Visibly no detrimental colour alteration was evident, further endorsing the likely practical possibilities of this technique.

Using these experimental results it seems likely that the use of cyclododecane as a volatile exclusion layer to provide surface protection during a resin-based consolidation treatment has serious practical merits. The results indicate that a 100% exclusion layer of cyclododecane applied as a melt when coupled with a 5% to 10% Butvar B98 consolidation regime should have a significant effect on the surface contamination through consolidant pollution.

The small anomalies mentioned previously due to the pores present in the timber (Image 3A-24), when combined with the crystalline nature of cyclododecane are likely to provide some practical limitations, the natural characteristic of the surface requiring isolation will need to be taken into consideration when applying this technique to an historic object. These types of anomaly are likely to exhibit a disruption to the exclusion layer as are flight holes caused by common furniture beetle and other textural features liable to interfere with crystal formation.

FUTURE Research

The conclusions drawn by this research suggest that the most effective use of this material is demonstrated when it is applied at 70°C as a 100% melt. Specialised equipment may need to be developed in order to provide a practical modus operandi for larger objects. The practical limitations of direct application in this way when coupled with previous research suggest possibly spray application in a solvent-based solution would be much more pragmatic. This would of course need to be followed up by in-situ melting of this applied coat after full evaporation of the solvent had taken place. Due to the limitations shown when using a hot air gun to accomplish this (the disturbance to the cyclododecane caused by the movement of the air) - the use of heat lamps or other radiant heat sources may be a possible solution to this problem. It must nevertheless be borne in mind that materials applied at these temperatures may well have some detrimental effect on the surface to which they are applied.[99]

After a suitable, sustainable and repeatable application regime has been established for larger objects further testing could be carried out using alternative consolidants as it seems that the relationship between the exclusion coat and the consolidant is crucial to the success of the overall results. Polar consolidant vehicles are likely to be the only suitable substitute for the types chosen for this research.

Further studies could include purity testing for cyclododecane from different suppliers including comparisons of the behaviour when sublimating from porous and non-porous timber surfaces (saturated and unsaturated surfaces). A study of the effects of heat shock and temperature differentials within the range described would also provide data useful in the further development of this technique.

[99] Larochette Y (2004) p4

Image appendix

Generic images

Image 0-0; Planed surface

Image 0-1; Band-sawn surface

Image 0-2; Circular-sawn surface

Image 0-3; Sanded surface

Image 0-4; Common furniture beetle damage

Image 0-5; Unsaturated surface

Image 0-6; Test environment

Image 0-7; Environmental monitor

Image 0-8; Prepared consolidants

Image 0-9; Laboratory crucible

Image 0-10; Photographic jig

Image 0-11; Microtome

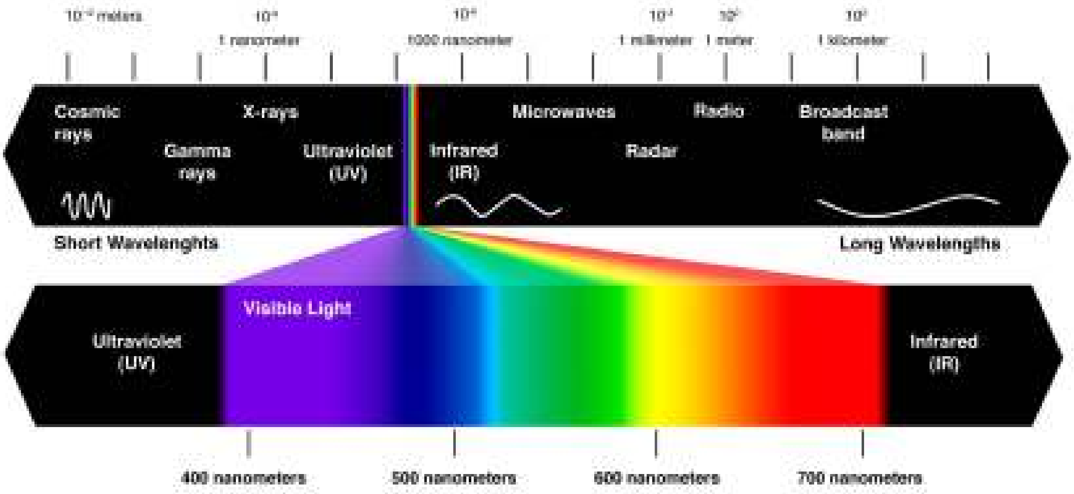


Image 0-12; Electromagnetic spectrum[100]

[100] http://local.content.compendiumblog.com/uploads/user/2af9dc1d-8541-42e4-a91f-6aaf97caf33a/4844a17e-a4fb-4018-9d3a-31dc846044ee/Visible%20spectrum.jpg

Test 1

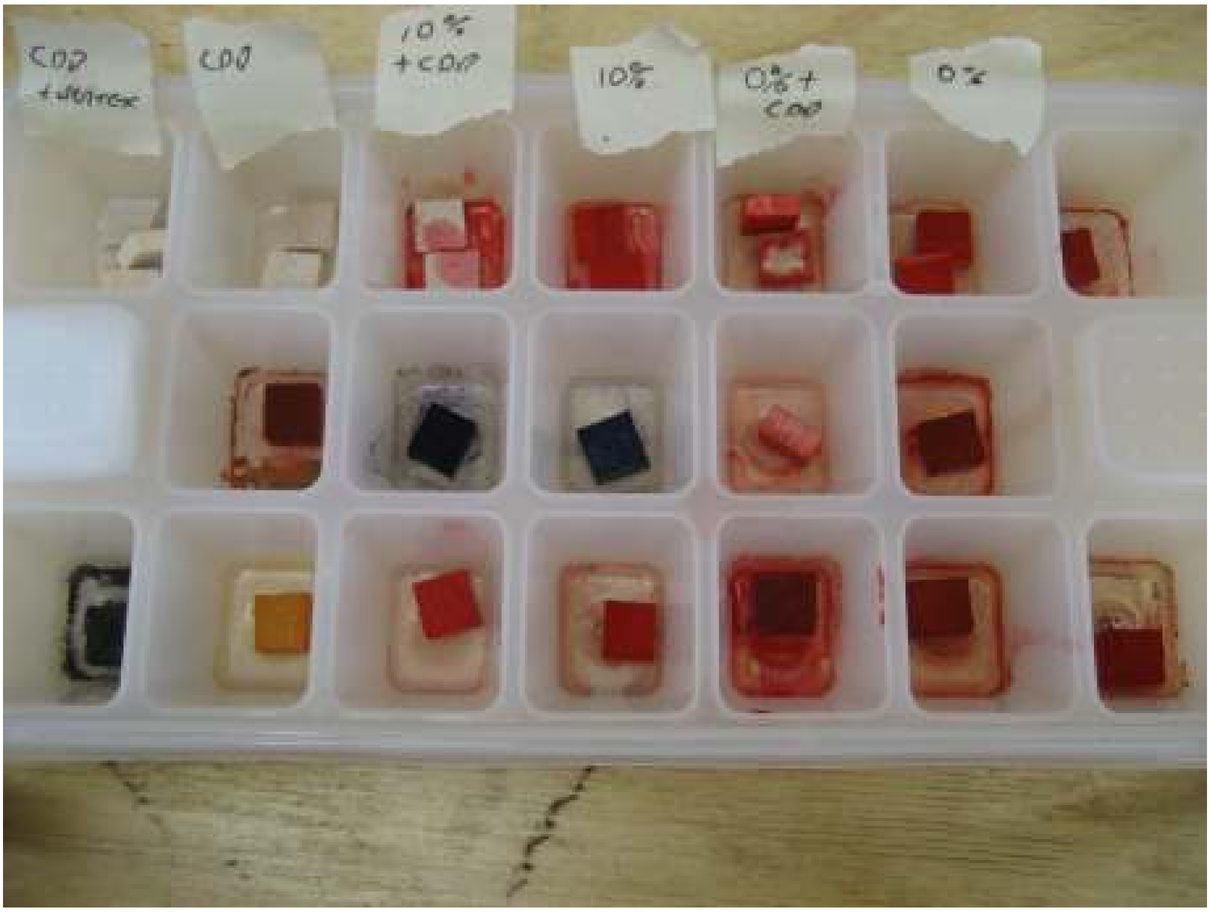


Image 1A-1; Test 1

Image 1-1

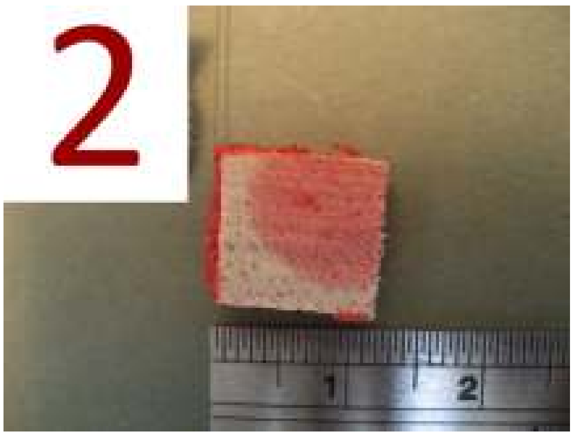


Image 1-2

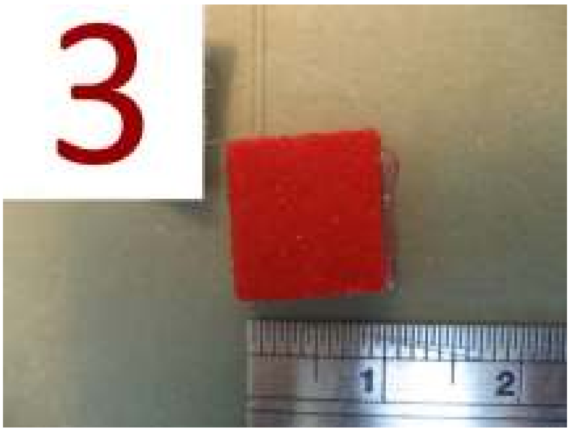


Image 1-3

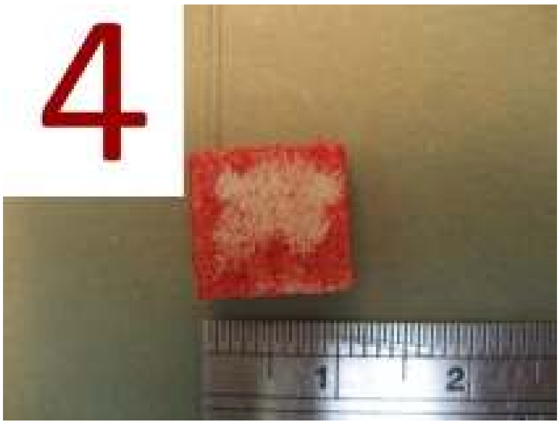


Image 1-4

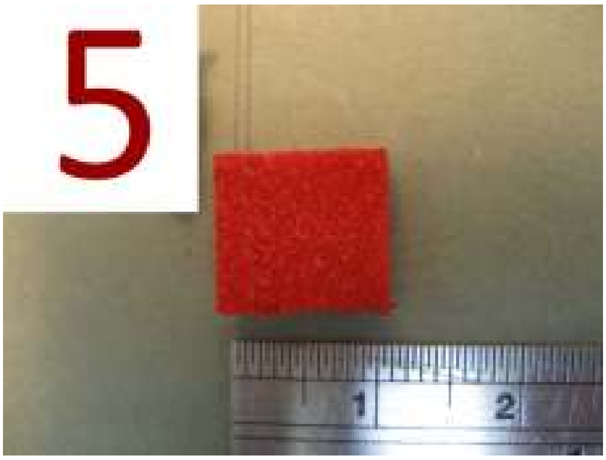


Image 1-5

Test 2

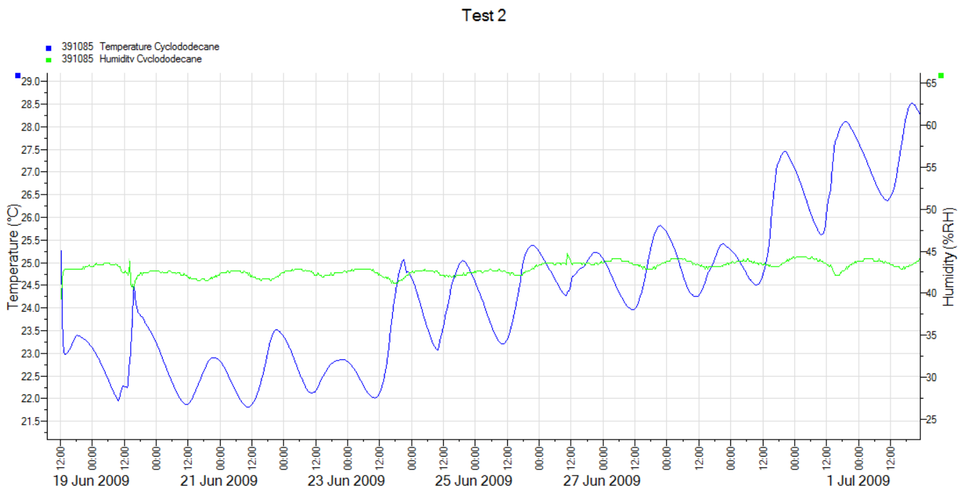


Image 2A-1; Test 2 environmental data

Image 2A-2; Test 2 blocks

Image 2-1

Image 2-2

Image 2-3

Image 2-4

Image 2-5

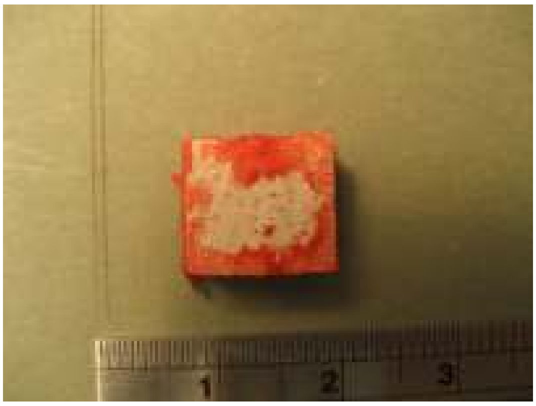


Image 2-6

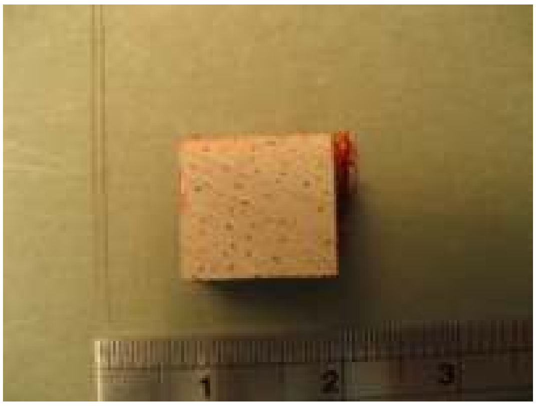


Image 2-7

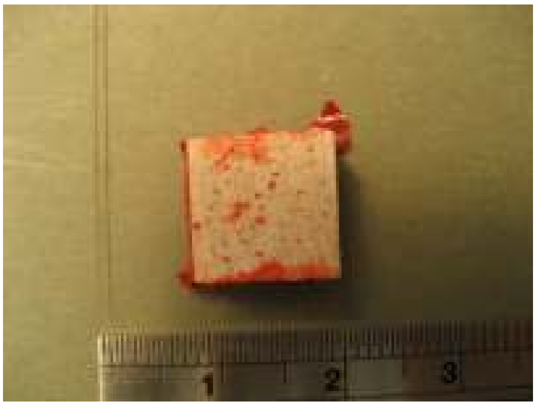


Image 2-8

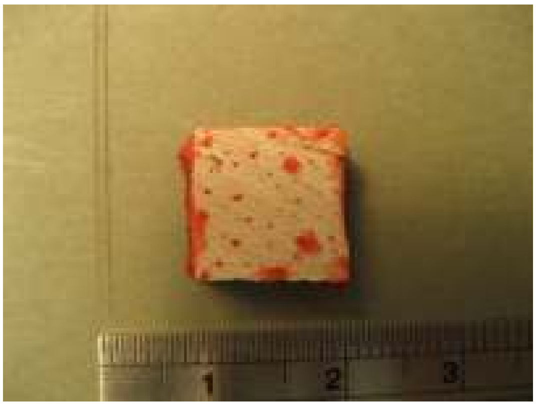


Image 2-9

Image 2-10

Image 2-11

Image 2-12

Image 2-13

Image 2-14

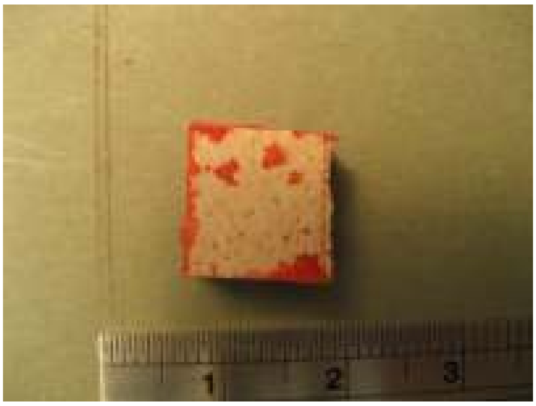


Image 2-15

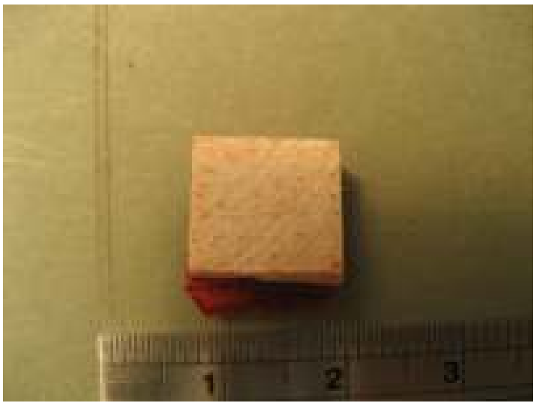


Image 2-16

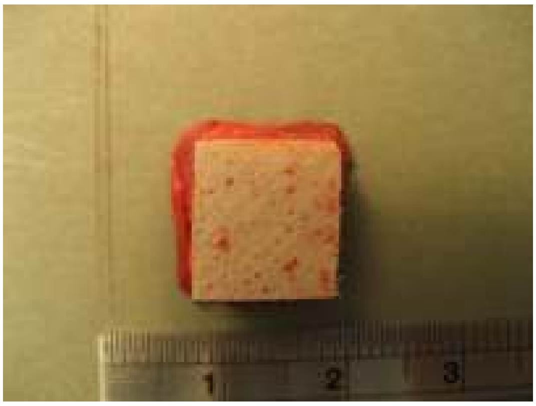


Image 2-17

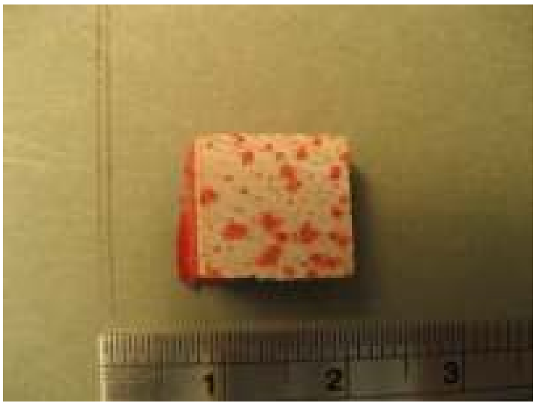


Image 2-18

Test 3

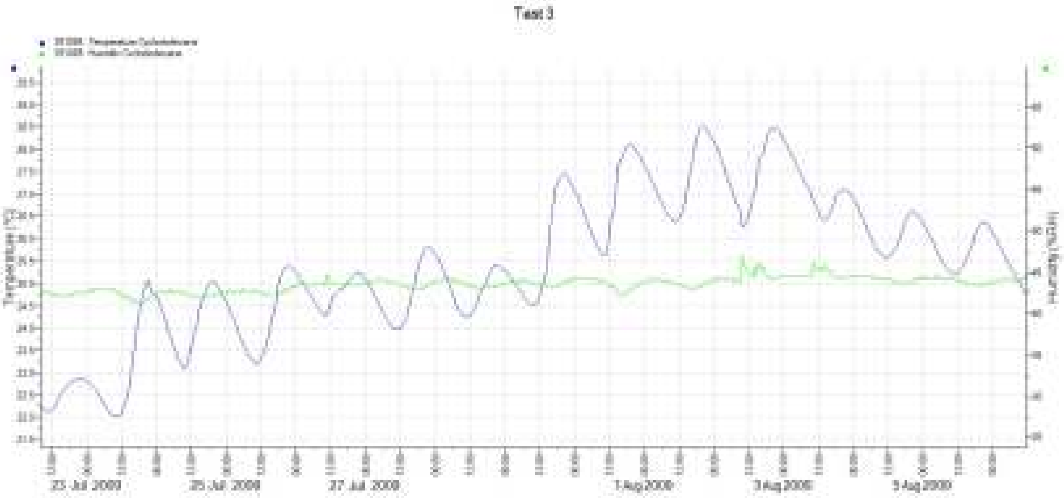


Image 3A-1; Test 3 environmental data

Image 3-1

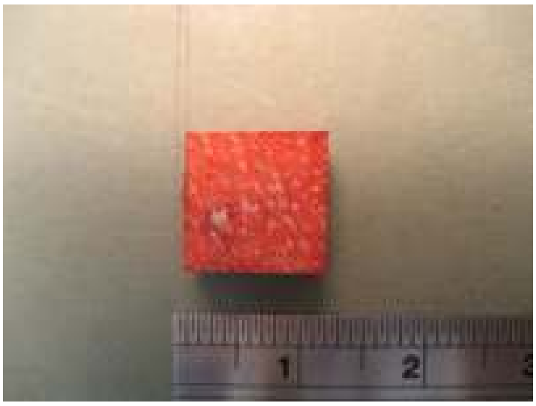


Image 3-2

Image 3-3

Image 3-4

Image 3-5

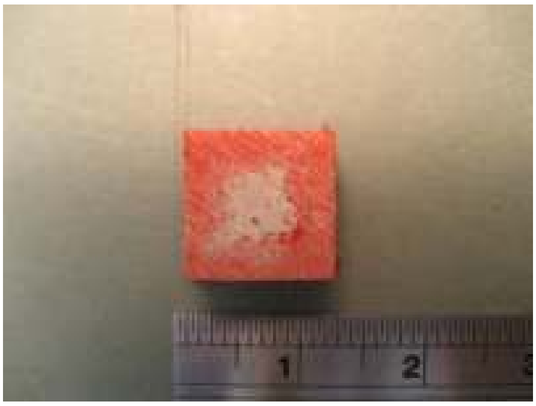


Image 3-6

Image 3-7

Image 3-8

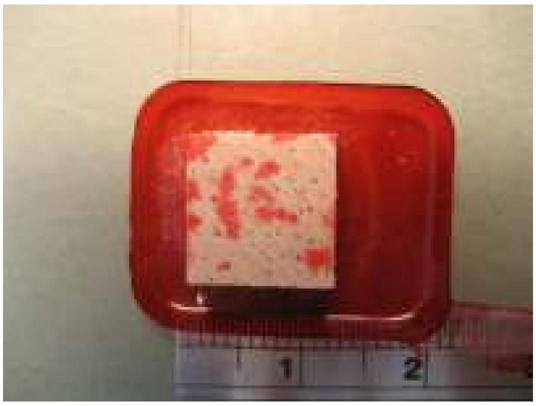


Image 3-9

Image 3-10

Image 3-11

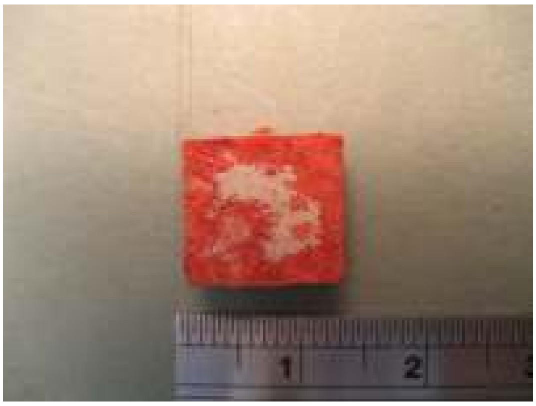


Image 3-12

Image 3-13

Image 3-14

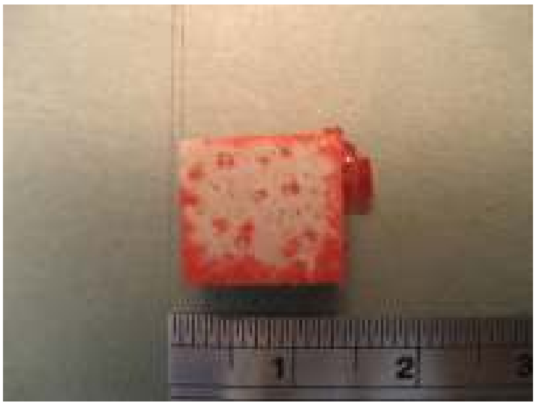


Image 3-15

Image 3-16

Image 3-17

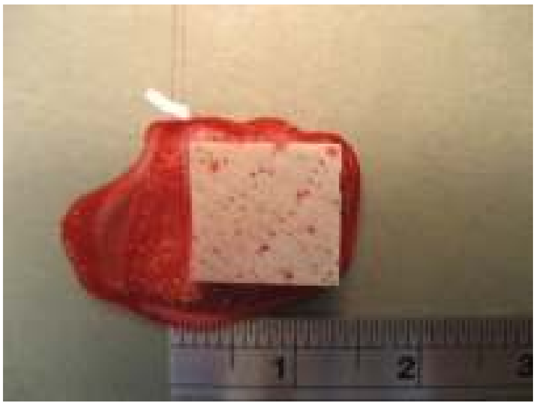


Image 3-18

Image 3-19

Image 3-20

Image 3-21

Image 3-22

Image 3-23

Image 3-24

Image 3-25

Image 3-26

Image 3-27

Image 3-28

Image 3-29

Image 3-30

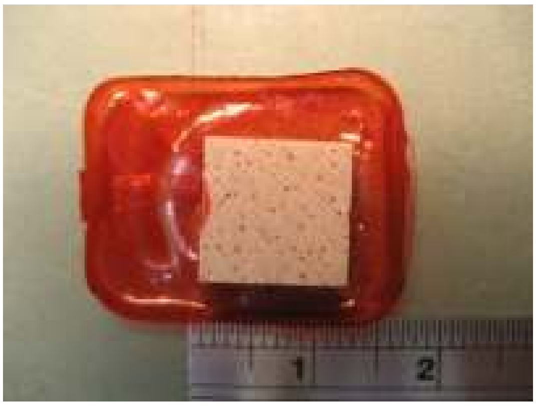
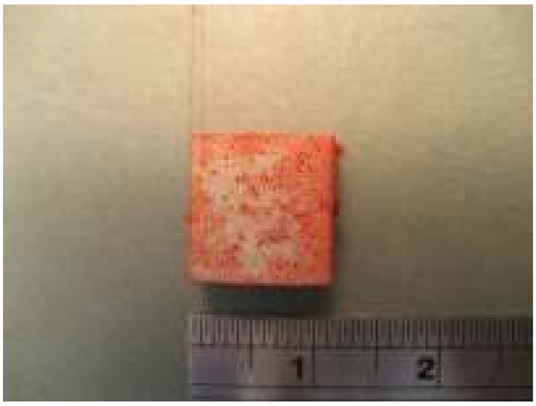


Image 3-31



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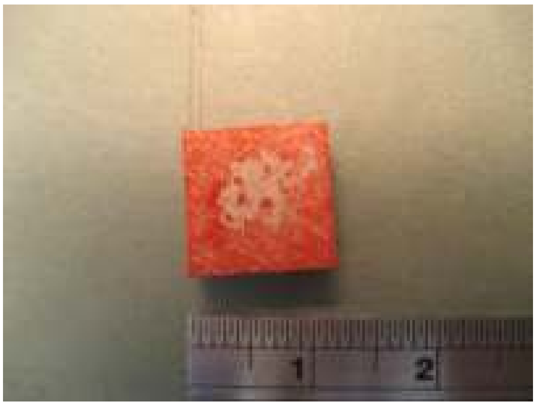
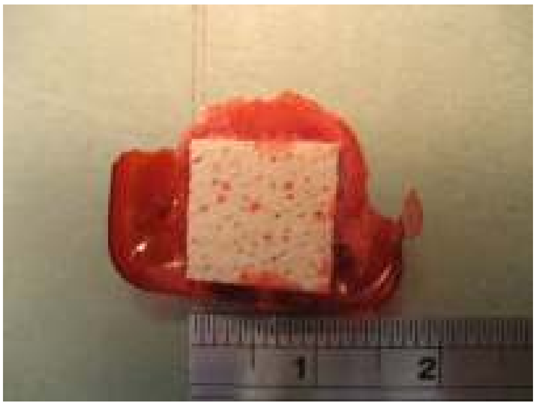


Image 3-33

Image 3-34



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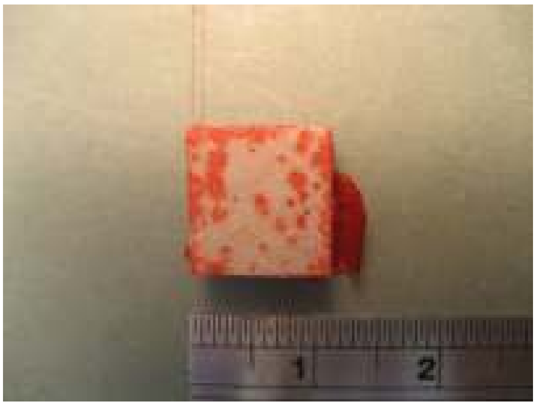


Image 3-36

Image 3-37

Image 3-38

Image 3-39

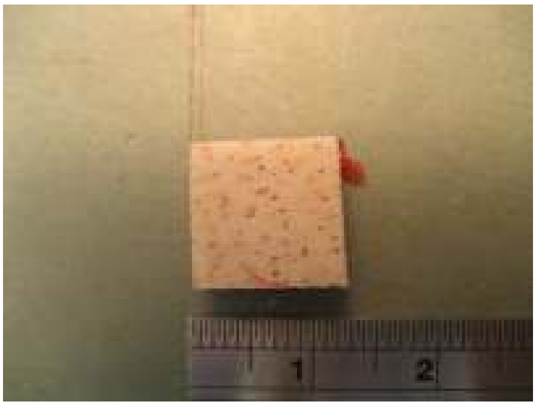


Image 3-40

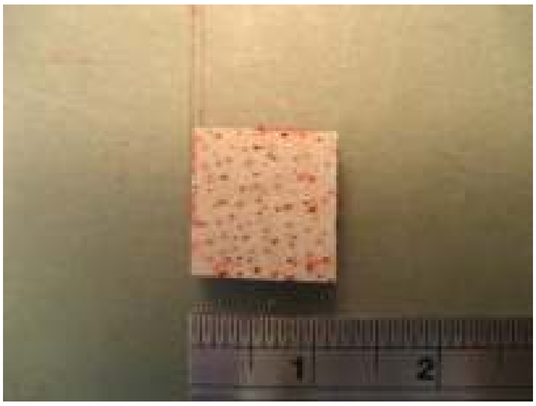


Image 3-41

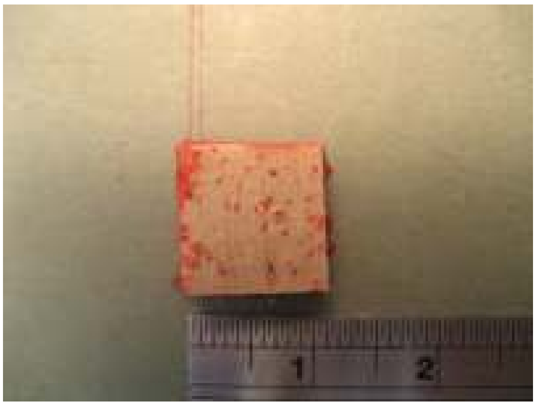
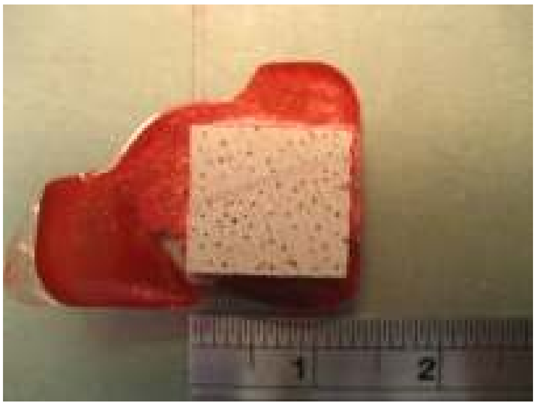


Image 3-42

Image 3-43



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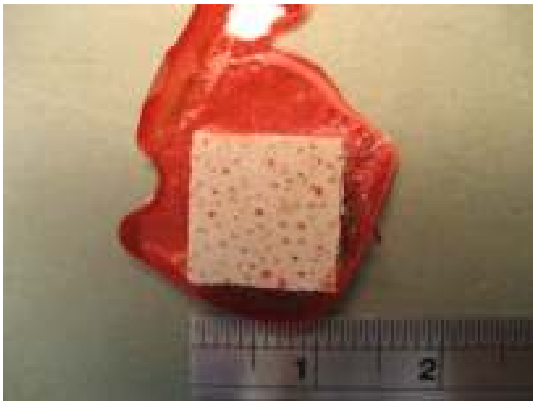

Image 3-45

Image 3-46

Image 3-47

Image 3-48

Image 3-49

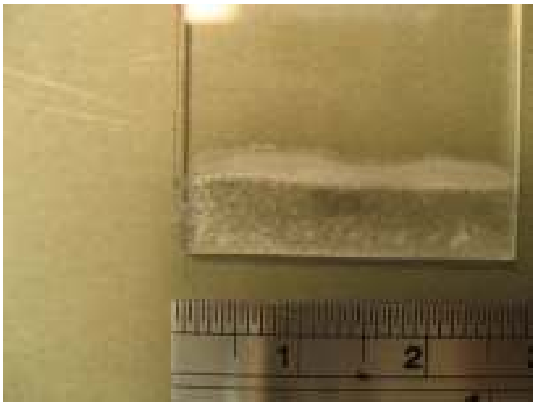

Image 3-50

Image 3-51


Image 3-52


Image 3-53


Image 3-54


Image 3-55

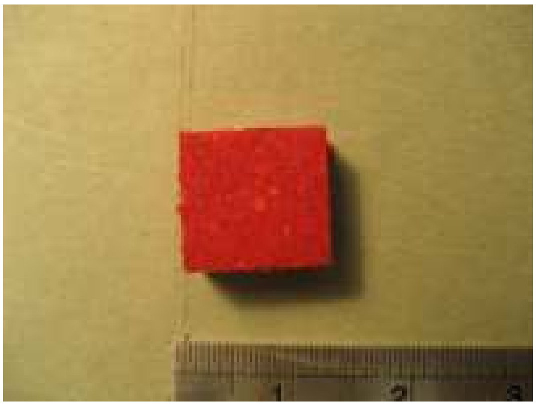
Image 3-56

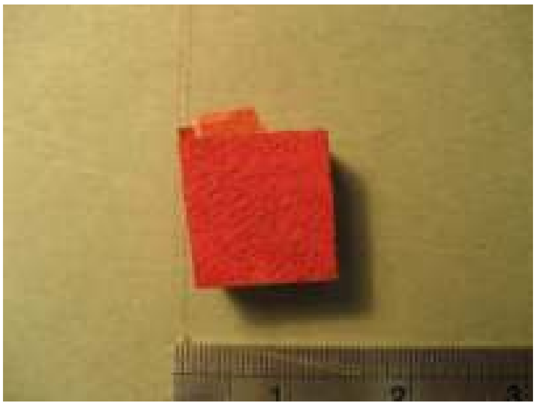
Image 3-57

Image 3A-2; Test 3 group A

Image 3A-3; Test 3 group B

Image 3A-4; Test 3 group A (Visible light)

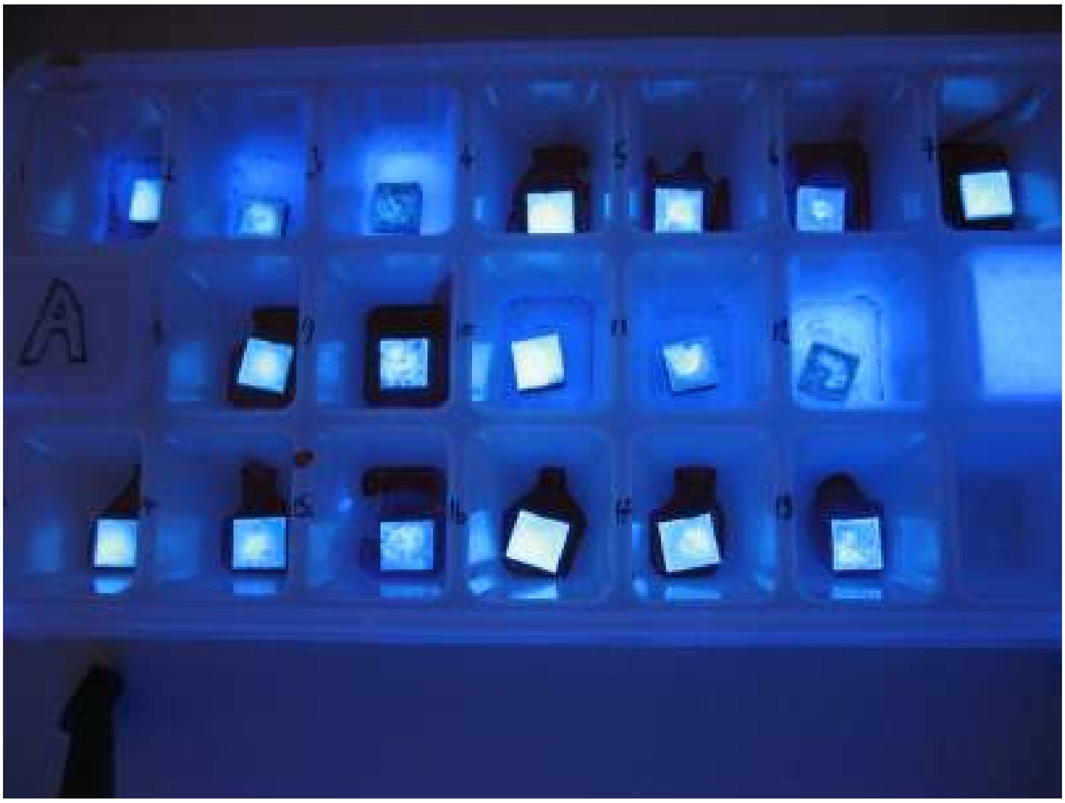

Image 3A-5 Test 3 group A (Ultraviolet light)

Image 3A-6; Test 3 19-27 (Visible light)

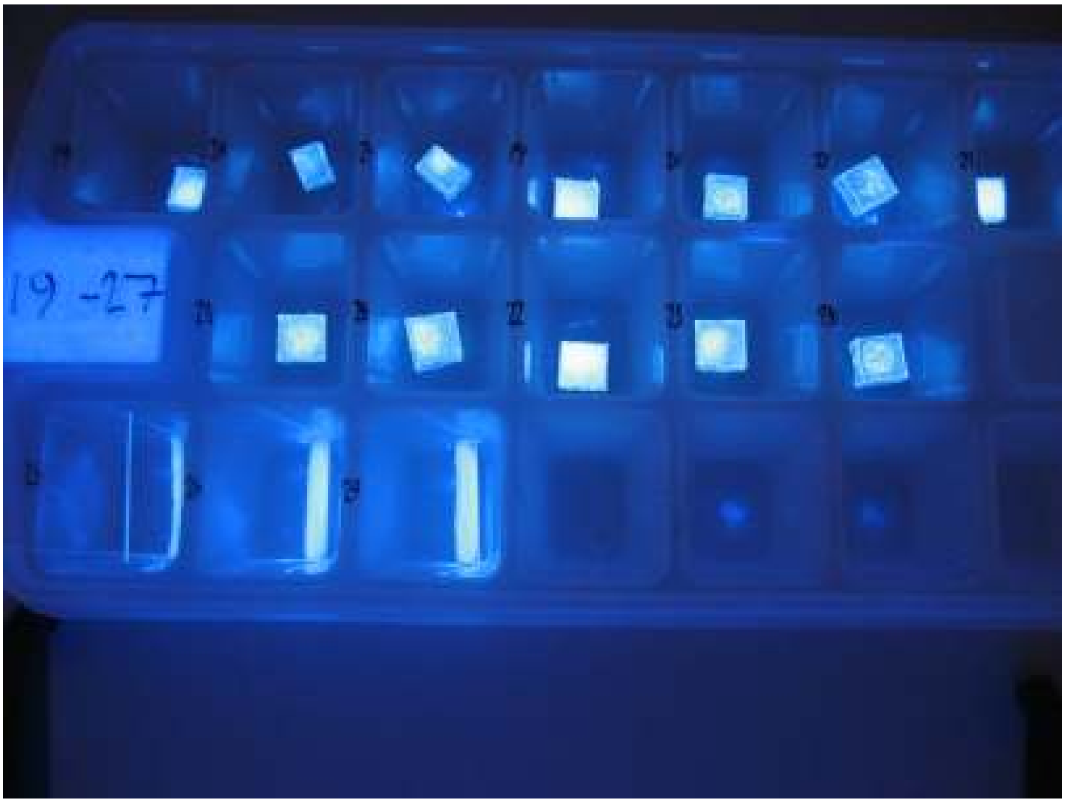

Image 3A-7; Test 3 19-27 (Ultraviolet light)

Image 3A-8

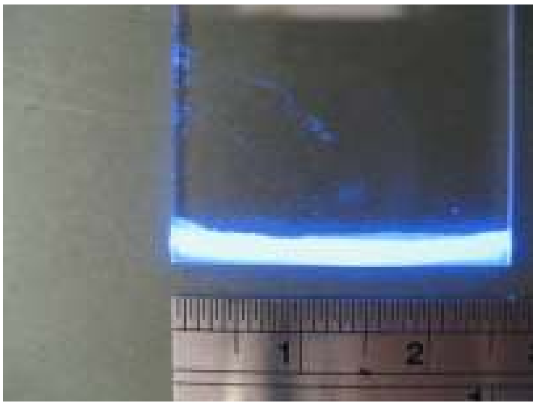

Image 3A-9

Image 3A-10

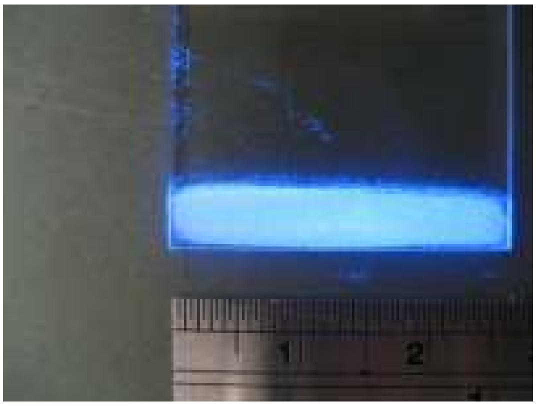

Image 3A-11

Image 3A-12

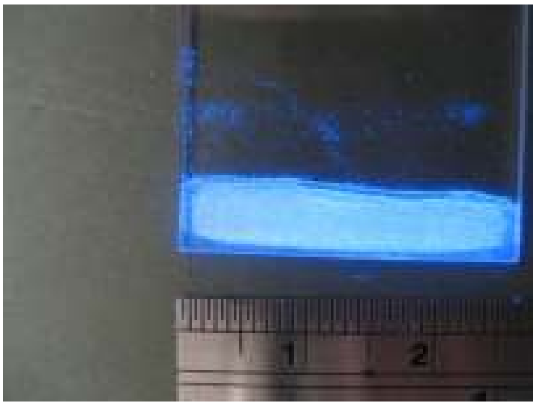

Image 3A-13

Image 3A-14

Image 3A-15

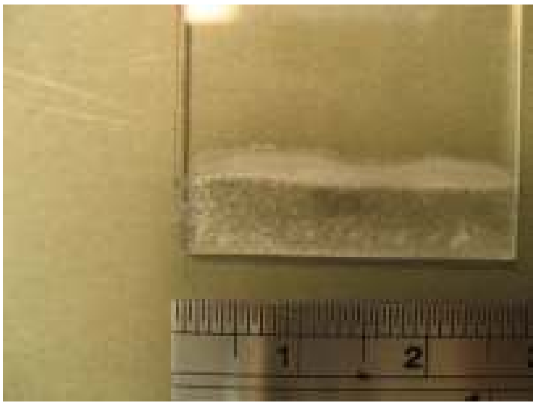

Image 3A-16

Image 3A-17

Image 3A-18

Image 3A-19

Image 3A-20; 100% cyclododecane

Image 3A-21; 90% cyclododecane

Image 3A-22; 80% cyclododecane

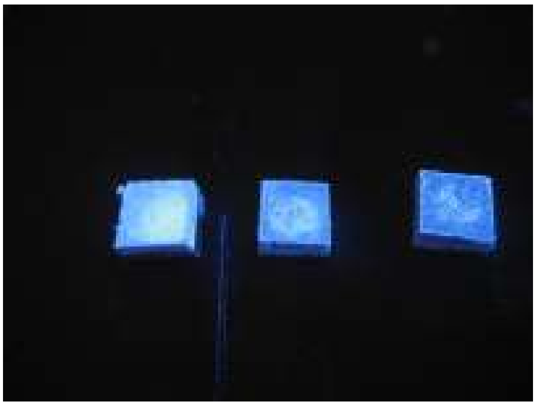

Image 3A-23; 100%, 90%, 80% cyclododecane

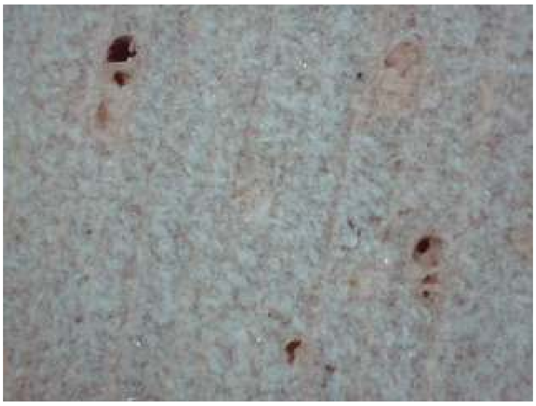

Image 3A-24; Large pores

Image 3A-25; 10mm x 10mm x 5mm test block

Image 3A-26; Control blocks – (0% cyclododecane + 0% consolidant)

Image 3A-27; Control blocks – (0% cyclododecane + 0% consolidant)

Image 3A-28; Control blocks – (0% cyclododecane + 0%, 5%, 10% consolidant)

Image 3A-29

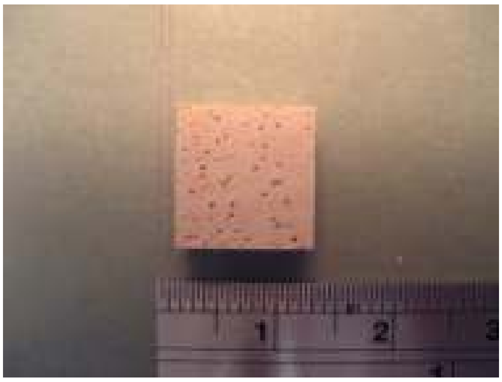

Image 3A-30

Image 3A-31

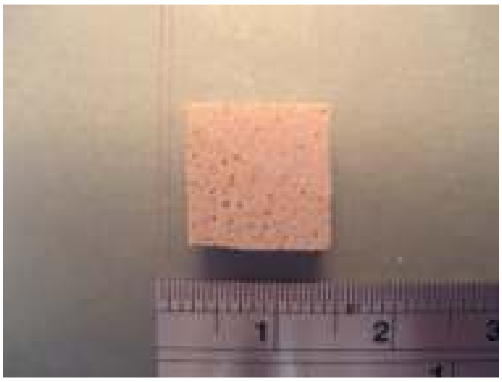

Image 3A-32

Image 3A-33

Image 3A-34

Additional Test 4

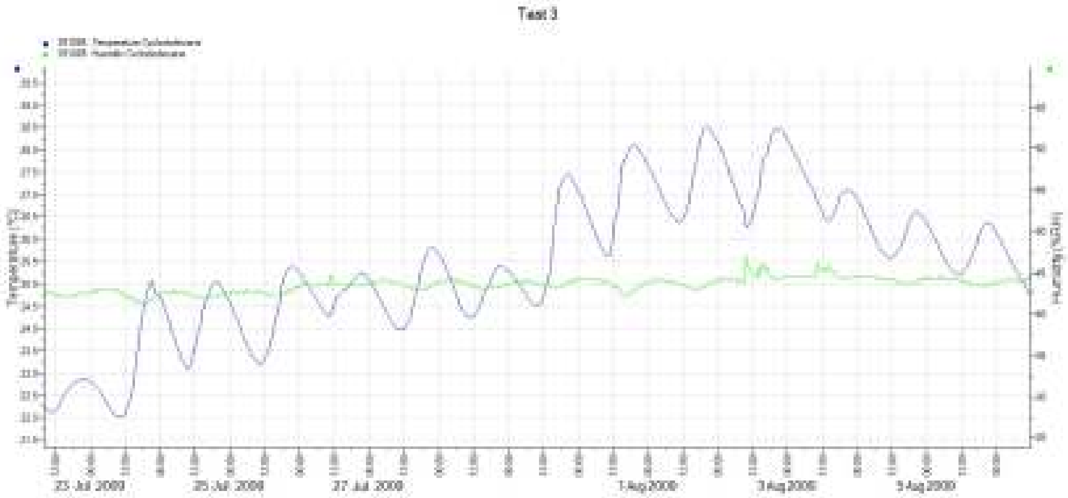

Image 4A-1; Test 4 environmental data

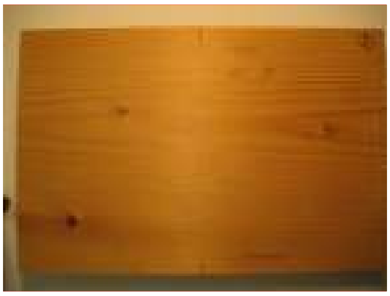

Image 4-1

Dry pine surface

Image 4-2

Historic pine surface

Image 4-3

Dry oak surface

Image 4-4

Dry pine surface

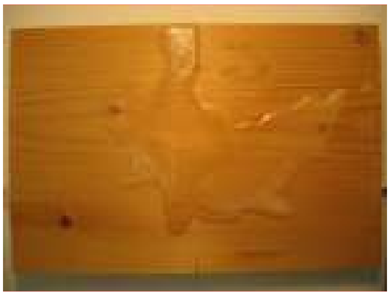

Image 4-5

Dry pine surface+100% CDD

Image 4-6

Historic pine surface+100% CDD

Image 4-7

Dry oak surface+100% CDD

Image 4-8

Dry pine surface + 100% CDD

Image 4-9

After sublimation

Image 4-10

After sublimation

Image 4-11

After sublimation

Image 4-12

After sublimation

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Appendices


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B-98 msds
CDD tech data sheet
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Cyclododecane msds
cyclododecane thermodynamics
IMS msds
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Uvitex msds