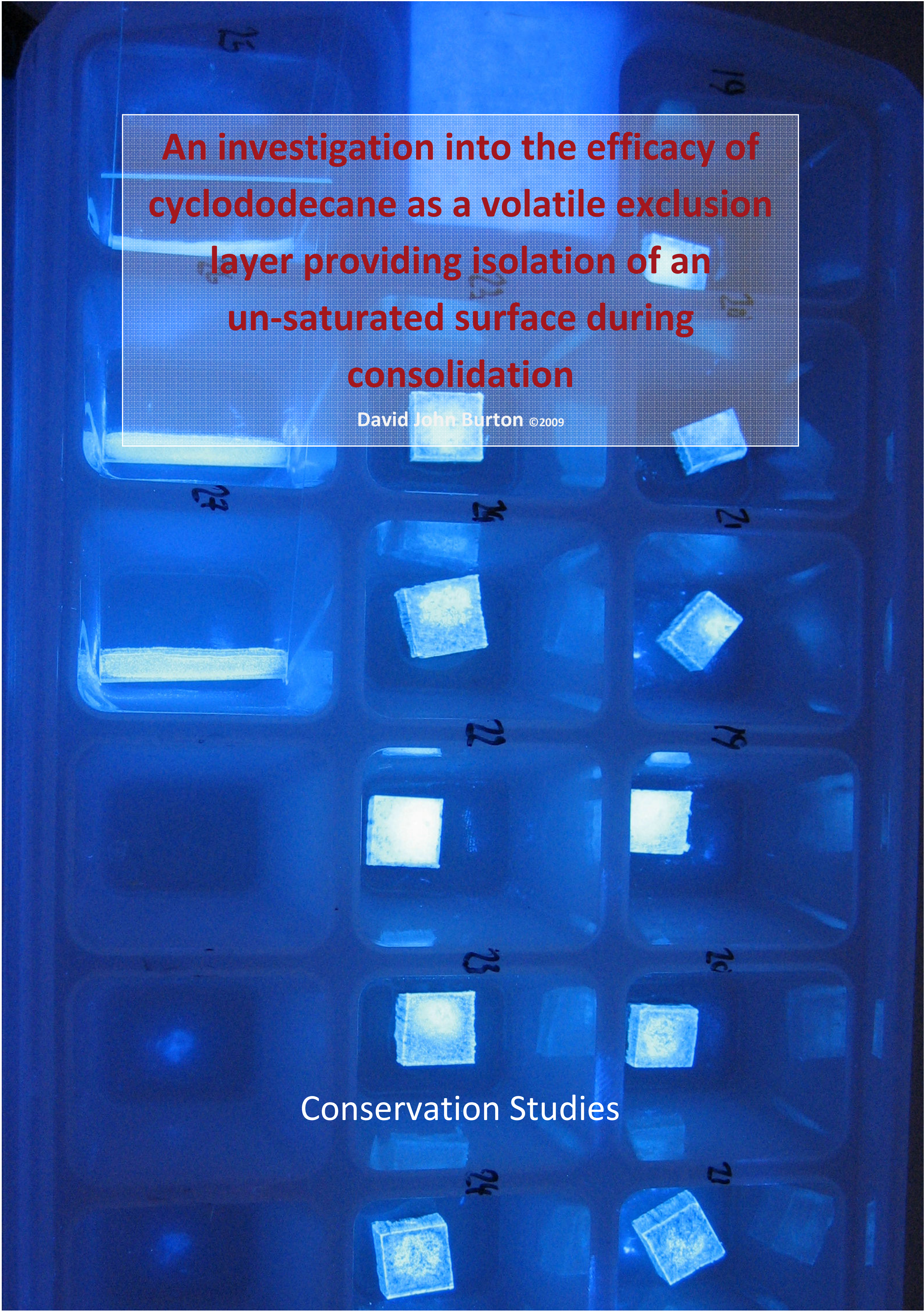

An investigation into the efficacy of cyclododecane as a volatile exclusion layer providing isolation of an un-saturated surface during consolidation

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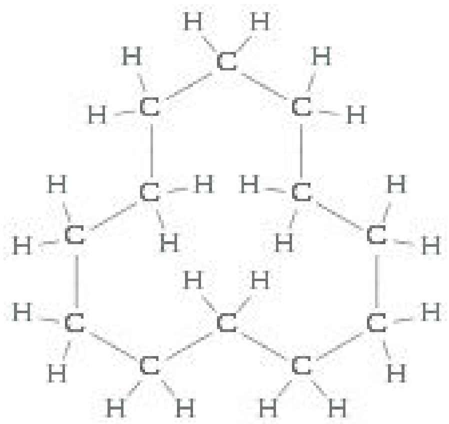
Conservation Studies

An investigation into the efficacy of cyclododecane as a volatile exclusion layer providing isolation of an un-saturated surface during consolidation - 9810 words



Contents

Abstract

Historically there has been an issue for conservators with unfavourable colour saturation of untreated timber surfaces and other decorative and historically important dry or friable surfaces when using dissolved resins for consolidation of underlying substrate material. At present conservators are forced to accept a certain level of surface colour saturation taking place when the substrate subordinate to a dry surface is consolidated; this limits the options available to the conservator undertaking such treatment.

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

An experimental regime has been developed to allow the testing of this hypothesis and using visual comparison through natural light photography, and UV photography and microscopy the emerging trends have been recorded.

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

Introduction

An experimental model was developed to allow the investigation of various application techniques for an exclusion layer of cyclododecane (**$C_{12}H_{24}$**) using controlled procedures and techniques to provide data. The key quality of cyclododecane is that it sublimates at room temperature, 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 stop the passage to the historic surface of a polar consolidation solution applied to an underlying timber substrate.

Previous research suggests that cyclododecane may penetrate deeper into a porous surface if it were dissolved in a non-polar solvent; this was reflected in the test regime. 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), an optical brightening agent used in the plastics industry that would allow the cyclododecane to be observed under Ultra Violet light.

A popular consolidant - Butvar B98 (polyvinyl butyral resin) was chosen and then tested in solution in 2 polar solvents; IMS (95% ethanol (**CH_3 CH_2OH**) – 5% methanol (**CH_3OH**)) and Acetone (**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

There is an historic problem in object conservation in relation to the protection of the visual appearance and integrity of the unsaturated surface.[1] The problem goes far beyond furniture; ethnographic material, polychrome sculpture, panel paintings, leather objects and wooden objects of all kinds. 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."*[2] 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 an article published as long ago as 1980 it was stated "*darkening of the wood after impregnation is inevitable,"*[3] this study also concluded that along with the improvement in the structural capabilities of the timber came this adulteration of the historic surface. A more recent study published in 2006 investigating wood consolidation in objects damaged by insect pests[4] 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*."[5]

These studies provide evidence that this type of resinous consolidation is still in the relatively early stages of development as far as the elimination of practical compromise is concerned. The proposed use of cyclododecane as a temporary barrier coat in order to eliminate this surface contamination is interestingly but not unusually new research based on problems produced by advances in technique developed by previous research. The question of consolidation of material involving saturation of a substrate usually arises at the point when an invasive treatment is necessary to prevent further

[1] Generic Images (image appendix) Image 0-5 – Example – unsaturated surface – 17th century Olivewood pilgrims souvenir model of the Basilica of the Holy Sepulchre (shown disassembled)
[2] Grattan DW (1980) p2
[3] Grattan DW (1980) p2
[4] Generic Images (image appendix) Image 0-4
[5] Maritato R & Snider D (2006) p149

structural deterioration, by very definition consolidation is to "*stabilise a degraded or weakened structure by introducing within it or attaching to it materials capable of holding it together*."[6]

The saturated surface e.g. any finished, polished, gilded, waxed or other surface treatments are by their very nature saturated, and therefore do not change their visual appearance with the application of further treatments such as surface coatings, adulteration by consolidants, adhesives and other substances that may be applied to the surface in order to facilitate a given conservation or restoration treatment, unless of course the surface coating, transparent or opaque is aged or otherwise degraded.

"*Saturation and gloss are inter-related optical properties of a surface. Saturation describes the degree of intensity or vividness of colour*"[7] the molecular weight, refractive index and gloss over surface coating invariably affects the level of saturation of the surface below. 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 a surface.

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 colour saturation inherent in this technique.[8] The study concluded that *"the refractive index is below or above that of the ochres, problems with colour changes will be experienced unless there are <u>no</u> resin residues left on the surfaces of the consolidated materials."*[9]

Most coatings used on historic objects are polymers dissolved in a solvent vehicle to allow application, with the evaporation of the solvent completing the process. Once the polymer has lost

6 Mcgiffin R (1983) p222
7 Rivers S & Umney R (2003) p587
8 I'ons A (1982) p13
9 I'ons A (1982) p21

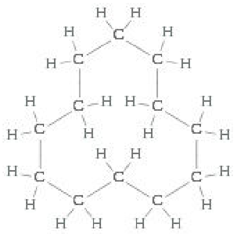
its solvent vehicle cleavage from the surface to which it has been applied, cross-linking of the polymer at a molecular level and conjugation and other molecular bond evolution can all have a significant effect on the visual appearance of a coating. As we can see the problem of unsaturated surface is not strictly demarcated by the presence of a finish or coating on that surface.

When a conservator treats material that is adjoining a surface such as this, this potential colour saturation 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. In a comprehensive study undertaken in 1985 it was noted that *"treated wood may have an aesthetically unsatisfactory appearance."*[10] 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.

As the saturation 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.

[10] Wang Y & Schniewind AP (1985) p71

Cyclododecane



Cyclododecane was *"first synthesised in Zurich in 1926 by Leopold Ruzicka"*[11] as part of groundbreaking work on macro-cyclic compounds for the perfume industry. Cyclododecane is a saturated cyclic alkane - a hydrocarbon ring with the maximum amount of hydrogen atoms bonded to the carbon atoms. Wts molecular structure takes the form of an undulating triad ring, *"it is neither a polymer nor a complex natural substance; rather it is a simple molecule."*[12] The triad ring not only provides volume, but also symmetry to the molecule allowing crystallisation to take place at room temperature.[13]

Cyclododecane is a white crystalline solid with a very slight odour; 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 and in fact has a very similar appearance to many of the acrylic and butyl resins previously mentioned as consolidants when in their un-dissolved form.

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.[14]

[11] Rowe S Roziek C (2008) p18
[12] Cagna M & Riggiardi D (2006) p89
[13] Billingham N 2009
[14] 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, given its obvious non-polar nature, would have little to offer the conservator. This particular form of $C_{12}H_{24}$ known as cyclododecane however exhibits a very interesting characteristic, that of sublimation. Carbon dioxide is a common example of a chemical compound that sublimates at atmospheric pressure—a block of solid CO_2 (dry ice) at room temperature and at atmospheric pressure will turn into gas without becoming a liquid. Sublimation is a transition from the solid to gas phase with no intermediate liquid stage.

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. The vapour pressure is measured in millimetres of mercury or in bar; cyclododecane has a vapour pressure of 0.039 mBar at 25°C.[15]

The non-polar nature is key to the choice of cyclododecane as an exclusion layer as part of 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. Polarity or non-polarity is the characteristic of a molecule defined in broad terms by the types of molecular bonds that exist between the atoms of that molecule. The unequal sharing of electrons within a covalent bond usually leads to localised polarity; whereas the sharing of electrons is equal the charges cancel each other out giving the molecule a non-polar nature.

"*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*." [16]

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

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

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: the separated partial positive and negative charges. A polar molecule results when a molecule contains polar bonds in an asymmetrical arrangement.

Non-polar molecules are of two types;

Molecules whose atoms have equal or nearly equal electro-negativities 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[17] - 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.

[17] 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.[18] 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.[19] This is a well documented example of the successful use of this material and the experiment was considered to be a considerable success.

The British Museum used cyclododecane as a surface consolidant during the transportation of a fragile Egyptian wall painting. [20] 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.[21]

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.[22] Studies undertaken in 2007 using Raman Spectra to investigate canvas samples impregnated with cyclododecane for their rate and quality of sublimation were very successful and confirmed *"no damage"*[23] had taken place to the surface when viewed at x200 magnification.

[18] Rowe S Roziek C (2008) p18
[19] Cleere DC(2005) p26
[20] Cleere(2005) p26
[21] Cleere(2005) p26
[22] Podmaniczky MS - conversation April 2009
[23] Kuvvetli F (2007) p31

As we can see this interesting 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 I am seeking to explore 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 of this material, successfully investigating application techniques, sublimation rates and a range of uses within conservation.[24]

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 found serious limitations in the technique several years previously.[25]

Yadin Larochette also found serious practical limitations when using cyclododecane as a barrier during aqueous textile bleaching conservation treatments.[26] 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.[27]

[24] Rowe S Roziek C (2008) p22
[25] Bruckle I, Thornton P, Nichols K, Strickler G (1999) p6
[26] Larochette (2004) p5
[27] 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 in 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 (silicone), which is itself a non-polar, cyclododecane, is often part of a much larger and more complex release layer regime. 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"*[28]

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.[29] 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. After all 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. [30], This technique has a long history and this new material was ideally suited to the challenges this process involved. [31] To give an example; one method of application would be to apply the cyclododecane to aluminium foil or Mylar, and apply face down to the surface of an object. With a gentle application of heat the conservator could melt the wax therefore saturating the surface and providing a mechanical support to the loose and vulnerable fragments. The Mylar 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.

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[28] Rowe S Roziek C (2008) p24
[29] Muros V (2004) p76
[30] Rivers & Umney (2003) p487
[31] Rivers & Umney (2003) p574

The British Museum famously used cyclododecane to secure unstable ceramic surfaces prior to transportation from their old basement store to the new storage area within the Department with *"promising results."*[32]

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 CDD or aerosol spray over delaminated or flaking areas worked better than CDD in hexane, which was more effective for consolidating a powdery areas with no flaking."*[33] 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.[34] Ceramic restorers have been known to use it in this way to fix shards of objects into place during restoration.

Application methods

Various application methods are available to the conservator for the application of this interesting 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 farther into samples than that applied using just the hot melt method. [35] The method of application has also been found to have a significant effect on the rate of sublimation, along with surrounding air temperature, and humidity and ventilation - with cyclododecane applied using the hot melt method taking two or three times as long to sublimate than that applied in solution.[36] 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.[37]

[32] Cleere(2005) p26
[33] Cleere DC (2005) p27
[34] Rowe S Roziek C (2008) p24
[35] Muros V (2004) p77
[36] Muros V (2004) p78
[37] Cagna M & Riggiardi D (2006) p90

Application as a spray

A heated spray gun generates a spray of melted cyclododecane, and an aerosol uses a solvent, 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. [38] 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.

Application as a melt

According to recent studies[39] cyclododecane forms the densest film when applied in a molten state, probably due to the rapid cooling, minute closely packed crystals are able to form providing an extremely successful hydrophobic film. When cyclododecane is dissolved in a solvent, solidification is inhibited causing a larger crystals to form - this produces a less dense and effective film; 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.[40] Practical limitations such as the rapid rate of cooling associated with the difficulty in application that this form, coupled with the obvious heat-shock affecting the material to which the cyclododecane is applied provide obvious practical limitations to this method.

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 even white spirit.[41] 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 the sublime more quickly than a melted application"*[42]

[38] Rowe S Roziek C (2008) p20
[39] Rowe S Roziek C (2008) p18
[40] Bruckle I, Thornton P, Nichols K, Strickler G (1999) p4
[41] Rowe S Roziek C (2008) p19
[42] 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",*[43] 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.

The saturated (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 that tend to be out of sight can reveal the most evidence of the intangible cultural heritage of our given object; Tool marks, construction lines and other subtle visual clues are all evidence of construction techniques and sometimes more specifically the tools used in a piece's manufacture. Other evidence of the approach and methodology of the maker can frequently be seen here, often in an untouched form. Cyclododecane offers the possibility of protecting the visual and physical character of these dry porous surfaces and the historic evidence they convey during resinous consolidation treatment. Saturating a dry, friable or untreated surface in this way inadvertently through accidental pollution invariably involves a colour change, described as *"chromatically altering the treated zones"*[44] in a study undertaken in 2006 investigating consolidation of non-archaeological wood.

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."*[45] 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 to form saturated hydrocarbons that do not sublimate and therefore produce a residue that may remain in situ for a longer period of time.

[43] Caple (2000) p8
[44] Maritato R (2006) p150
[45] Jaegers E (2009) p1

Supporting data for this possibility has been found in several previous studies; the 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.[46] The results of other studies indicate the opposite; 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 is detectable with Raman spectroscopy and SEM."*[47] 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 anomalies 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."*[48] 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.

[46] Muros V (2004) p79
[47] Kuvvetli F (2007) p32
[48] 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"*[49] and decreased relative humidity. 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 environmental controllability.[50] As it would be counter-productive to evacuate the vapour continuously during the maintenance of a suitably high ambient temperature it was decided to set the fume cabinet on a timer extracting for only 15 minutes per hour.

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[51] the sublimation rate from porous surfaces is shown to be much slower than that from nonporous surfaces. It must be stressed however that the previously quoted 87 days was in an extremely cool environment with no aid to sublimation taking place. In some studies it has been noted that cyclododecane can sublimate away in as little as 24 hours when applied to historic paper objects.[52]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.[53]

Taking into account this 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 will be illuminated by a 60 W tungsten filament bulb present in the fume cabinet. Preliminary testing has established this will provide enough heat to simulate a possible conservation treatment environment established specifically for maximum sublimation rate.

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[49] Nichols K, Mustalish R (2002) P81
[50] Generic Images (image appendix) Image 0-6
[51] Muros V (2004) p79
[52]Szuhay B (2005) p105
[53] Stein R, Kimmel J, Marincola M, Klemm F (2000) p361

A *Tiny Tag*[54] environmental monitor will be present at all times in the fume cabinet capturing data. The temperature and relative humidity of the test environment will be monitored over the full period of the experiment. The rate of sublimation of cyclododecane is directly related to these factors, along with vapour saturation in the localised environment.

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

[55]

[54] Generic Images (image appendix) Image 0-7
[55] http://www.csgnetwork.com/specificgravwdtable.html

This material 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. 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."*[56] 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.[57] The preparation of the surfaces of these blocks was of crucial importance if reliable test data were to be extracted. Various different types of surface preparation were experimented with for the two faces of the cube that would be expected to 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.[58] In order to provide realistic data it was eventually decided to use the sanded surface, prepared with 240 grit abrasive as it was found that this finishing regime produced a smooth, realistic and repeatable surface texture that would provide comparable and reliable results.[59] As each series of blocks is prepared a duplicate set of blocks will be produced using adjoining balsa to provide control blocks to be used for comparison at the end of the test.[60] At every stage of the preparation visual discrimination will be used to make sure each control block is comparative to its relative test block, any natural anomalies in the timber will be dealt with at that stage.

The size of the balsawood blocks in relation to the solvent-based preparation (consolidant) that would be applied to them was also a crucial factor. If the blocks were too large capillary action would not carry the consolidant all the way through to the other surface, therefore 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 of course also relies on permeability and capillary action in order to gain penetration. Preliminary testing was used in order to finalise these factors.

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[56] Wang Y & Schniewind AP (1985) p3
[57] Test 3 (image appendix) Image 3A-25
[58] Generic Images (image appendix) Image 0-0, Image 0-1, Image 0-2, Image 0-3
[59] Generic Images (image appendix) Image 0-3
[60] Generic Images (image appendix) Image 3A-26 and Image 3A-27

The consolidant model

A resin-based consolidation technique generally relies on a solvent vehicle that is colourless, used to dissolve a colourless resin and carry it into damaged timber. Butvar B98 was chosen as a standard consolidant of choice, Schniewind & Kronkright found that polyvinyl butyral (Butvar) gave *"the most improvement in strength"*[61] in a 1984 study and remains a popular choice for conservators today.

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 phase of the development of the test regime. It was found that many proprietary spirit stains were more soluble in ethanol than in acetone. 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. 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 and a ratio of 1% was found to be the minimum necessary to provide clearly visible evidence of consolidant saturation.

Using the 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.

[61] Wang Y & Schniewind AP (1985) p79

The temporary exclusion layer

The use of cyclododecane as an exclusion layer provides similar visual limitations, as pure cyclododecane is transparent it does not allow visual discrimination for the purposes of ascertaining presence, penetration and sublimation. It was decided that the best method of tracking these factors in the exclusion layer was to adulterate the cyclododecane using a chemical that would fluoresce under UV microscopy.

After research Uvitex OB® (optical brightener) was chosen. Ciba® Uvitex OB® is a heat resistant, non polar-solvent soluble chemically stable fluorescent whitener that provides brighter looking colours for solvent based paints. It is used to offset the yellowness of coatings and to yield a whiter appearance. *"Uvitex OB® can also be used as a tracer in clear coatings."*[62] After preliminary testing it was found to dissolve readily in cyclododecane, fluorescing and clearly visible under UV microscopy at proportions as low as 0.01%. Uvitex OB® will 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 would be heated to 70°C in a laboratory crucible in order to provide a standardised application regime.[63]

UV microscopy

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 primarily 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. 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. It must always be remembered that due to the nature of fluorescence the light given off in the previously explained process of interaction with electrons is in the visible spectrum. This visible light can sometimes illuminate part of the sample producing confusing results.

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

Fluorescence

The electromagnetic energy of a photon is inversely proportional to its wavelength. Short wavelength 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 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 due to the types of atoms and how they bonded to one another. 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 decays to the ground state with the emission of a photon of visible light, this process is called fluorescence.

Photography

A Canon IXUS 60 6 mega Pixel camera mounted onto a fixed photographic Jig, with a standardised matt background will be used. [64] All photography will take place in a darkened room using a fixed 60w tungsten filament light or a fixed UV light source for illumination to provide comparable results. Photography of UV microscopy samples will capture with a Dinolite® digital camera inserted into the eyepiece of the microscope, all UV microscopy will be carried out in a darkened lab to provide standardised results. The surface of the sectioned blocks to be photographed under UV microscopy will be prepared using a standard Laboratory microtome to slice each section.[65]

[64] Generic Images (image appendix) Image 0-10
[65] Generic Images (image appendix) Image 0-11

Practical Testing

Preliminary test 1

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

With some studies quoting cyclododecane concentrations in solvent as low as 50%[66] and other studies using cyclododecane at 100% concentration (melt)[67] preliminary testing showed that concentrations below 75% were extremely ineffective in providing a contiguous 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 test blocks were treated with the following regime;

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 each solvent was a crucial factor in the choice of Sellaset H as a suitable dye.

66 Cagna M & Riggiari D (2006) p92
67 Cleere DC (2005) p27

The fully developed testing regimen;

1. Prepared cubes 10 mm x 10 mm x 5 mm of balsawood will be treated with cyclododecane at 100% (as a melt) and at 90% and 80% (as a heated solute dissolved in white spirit) to one face to provide an exclusion coat.

2. Each set of test blocks will have an identical (taken from the same section of balsa) control block prepared to be used 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 to allow identification will be prepared- 2 ml per block will be introduced from below using laboratory pipette.

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

5. Glass microscope slides will be 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 will be prepared to allow sectioning for UV microscopy.

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

8. After a full solvent evaporation and sublimation of the cyclododecane has taken place the cubes will be compared to the control blocks mentioned previously. Any visual difference created by the edition and subsequent sublimation of the cyclododecane will be noted.

Preliminary test 2

Having established the practical feasibility of the testing regime preliminary test 2 will endeavour to cover the full range of the proposed test consolidants and variations for the application of the exclusion layer. No Uvitex OB® will be used in this part of the testing process as UV photography and microscopy will not be necessary at this stage.

Visual examination and photography of the test blocks will establish whether or not trends are beginning to appear in the results. Preliminary test 2 will run 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 vehicle is necessary as only when the solvent has completely evaporated does the passage of the consolidant through the substrate stop. The sublimation and relative penetration of the cyclododecane will be investigated during the full controlled experiment in part 3. This is outside of the remit of this part of the test is at present we are trying to establish the success of the of the consolidant exclusion from the surface of the test samples.

(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

After analysing the results are preliminary test 2 full control test 3 will further establish trends in the relationship between the purity of the exclusion layer and the proportion of consolidant dissolved in the solvent. Test 3 will be divided into two parts; part A will consist of cyclododecane with an added 0.001% 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) will be prepared as a control. These blocks will be placed alongside the adulterated samples and will serve as a visual surface and texture comparison upon completion of the testing. The glass slide samples (25, 26, 27, 49, 50 and 51) will be viewed visually and photographed to provide evidence of residue left behind after sublimation is complete. These samples will **not** be viewed under UV light due to the fact that the Uvitex OB® will still be 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)

(Test 3 image appendix - Images 3-1 to 3-51)

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 or otherwise of the exclusion layer on the completed test blocks approximately 1 mm around the edge of each test block was considered inaccurate for the purposes of this experiment;

Preliminary test 1- results

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 as an exclusion coat.

Preliminary test 2- results

Test 2 is much broader ranging and has the benefit of full environmental monitoring. The test was run for 14 days, broadly considering *"11 days"*[68] taken in a 2007 study as a good average of published expected results. 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 visually all sublimation seem 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 ranges from 21.5°C to 28.5°C with the usual cyclic trend associated with day and night temperature fluctuations. The relative humidity remains fairly constant in the 40% to 45% range, not an ideal storage environment but unacceptable short-term conservation studio scenario.

[68] Kuvvetli F (2007) p32

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

The next three blocks (Images 2-4, 2-5 and 2-6) show 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) having 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 regimes of IMS-based consolidant used here.

The next three blocks in the series (Images 2-7, 2-8 and 2-9) seem 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 can 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 emerges.

At the conclusion of the test the 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 identical to the previously mentioned control blocks prepared for each test (example Image 3A-25) which had never been adulterated with cyclododecane.

Full control test 3 - results

Test 3 is much broader ranging still, repeating the range of cyclododecane regimes along with the consolidant regimes used in test 2. In addition to this full range these test blocks are 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 includes 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 ranges from 22°C to 28.5°C with the usual cyclic trend associated with day and night temperature fluctuations. The relative humidity remains fairly constant in the 41% to 46% range, not an ideal storage environment but again an unacceptable short-term conservation studio scenario. These 48 blocks were all treated in the fully developed test regime described previously. It is possible to see from comparison of these 48 blocks a significant confirmation of the trend seen emerging in test 2;

Blocks 1, 2 and 3 (Images 3-1, 3-2 and 3-3) show a clear difference in the amount of surface contamination. Block one (Image 3-1) has virtually no surface contamination whereas blocks two and three (Images 3-2 and 3-3) have 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.

The next three blocks (Images 3-4, 3-5 and 3-6) again show 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) having 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.

The next three blocks in the series (Images 3-7, 3-8 and 3-9) further repeat this trend with the first block (Image 3-7) (treated with 100% cyclododecane melt) seeming to resist the 10% Butvar B98 in IMS just 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 three blocks in sequence the same pattern emerges. Blocks 10 11 and 12 (Images 3-10, 3-11 and 3-12) continue the emerging pattern, blocks 13, 14 and15 (Images 3-13, 3-14 and 3-15) giving an even clearer example - at a very useful consolidant concentration, 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 three blocks.

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 pores present in the timber, continuing up to the surface (Image 3A-24). The crystalline nature of cyclododecane is likely to have its limitations regarding its ability to span relatively large voids. This natural characteristic of timber requiring treatment needs to be taken into consideration when considering the 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.

When viewed as a group, group A test blocks were photographed under visible light (Image 3A-4) and then under ultraviolet light (Image 3A-5). The addition of Uvitex OB® to group A sample blocks has provided significant data regarding the characteristics of the cyclododecane exclusion layer. UV photography was taken 24 hours from the start of the test allowing complete evaporation of any white spirit from the two adulterated cyclododecane regimes.

The results are striking to say the least; when we view the trend in sets of three (treated with 100%, 90% and 80% cyclododecane melt respectively) we can see 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 and again on each set of three for all 18 samples. Perhaps the most striking are blocks 10,11 and 12 which show this trend very clearly. The control samples, which are unadulterated 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.

The glass slides25, 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 can 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. This result however expected is not within the remit of the final analysis.

The three glass slides coated with the three different solutions of cyclododecane (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. [69] 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."* [70]

[69] Muros V (2004) p79
[70] Kuvvetli F (2007) p32

When the UV photographic data is juxtaposed with the cross sectional data provided by the UV microscopy the emerging trend is further established. The three cross sections from blocks 19, 20 and 21 (Image 3A-20, Image 3A-21 and Image 3A-22) seem 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 never been adulterated with cyclododecane.

Examples of this comparison can 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 saturation when compared to their counterpart control blocks that had been in an identical environment but left unadulterated.

Results Analysis

Preliminary test 1- analysis

The basic criteria for the further development of the testing modus operandi have been met. A standardised test block preparation has been arrived at; suitable volume of consolidant has been established for each block. The method in temperature for the application of the cyclododecane has been proved to be practicable. The test environment has been established and a standardised photography method has been satisfactorily achieved. From this simple preliminary test it seems 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 are likely to have an effect on later results of this test.

Preliminary test 2- analysis

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

A trend seems to be emerging that the less adulterated the cyclododecane -- the more protection it offers to the surface it is applied to, with 100% cyclododecane melt being the most effective in every case. This result is not altogether unexpected as studies undertaken as recently as 2004 show *"the melt was found to be a more effective barrier than the solutions."*[71] Another trend is also emerging, that of the three differing concentrations of consolidant seeming to have an effect on the results. It certainly appears that in these first 18 blocks the cyclododecane is not as effective at resisting the 0% Butvar B98 (100% solvent) preparation but seems much more effective at resisting the 5% Butvar B98 consolidant and even more effective at resisting the 10% Butvar B98 consolidant.

[71] Muros V (2004) p82

These early results from test 2 seem to imply that there are two factors involved in the relationship between the cyclododecane exclusion barrier and the alcohol-based consolidant. It seems 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 second generation of testing has proved successful; the expected trend has consistently continued in line with expectations. Examination under ultraviolet light using photography and microscopy has yielded further information regarding the behaviour of this material. New data provided by this ultraviolet imaging has significantly added to the body of the conclusion of this research.The trend seems to be 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.

This result is not altogether unexpected as studies undertaken as recently as 2004 show *"the melt was found to be a more effective barrier than the solutions."*[72]

The ultraviolet photography (Image 3A-5) taken of group A provides 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 contiguous surface film.
2. The decrease in fluorescence as the amount of solvent increases indicates more penetration leaving less cyclododecane at the surface.

Of course it is quite feasible that both of these scenarios could co-exist and be correct;

[72] Muros V (2004) p82

The data provided by the cross-sectional UV microscopy (Image 3A-20, Image 3A-21 and Image 3A-22) suggests that this is likely. As mentioned previously the addition of solvent to cyclododecane has been shown in previous studies to affect crystal size upon solidification.[73]

Finally -- but probably most importantly as with tests one and two, test three has shown consistent results confirming that cyclododecane has not affected the visual saturation or texture of any tested blocks.

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

Conclusions

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 has emerged; the proportion of solvent adulteration of the cyclododecane is 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.

The consolidant solution strength chosen to use with this exclusion layer is as significant 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 stopped by the exclusion layer. 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 saturation.

Using the above 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 potentially has serious practical merits. These 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 saturation.

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 needs to be taken into consideration when applying this technique - as should flight holes caused by common furniture beetles and other textural features liable to interfere with crystal formation; as these are likely to exhibit a similar disruption to the exclusion layer.

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. This may not be in the best interests of the historic surface of an object. Materials applied at these temperatures may well have a detrimental effect on the surface to which they are applied.[74] There are also practical limitations of applying and material such as this to a larger surface area in the molten form.

After a suitable, sustainable and repeatable application regime has been established for larger objects further testing can 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 treatment.

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. 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.

[74] Larochette Y (2004) p4

Image appendix

Generic images

Image 0-0

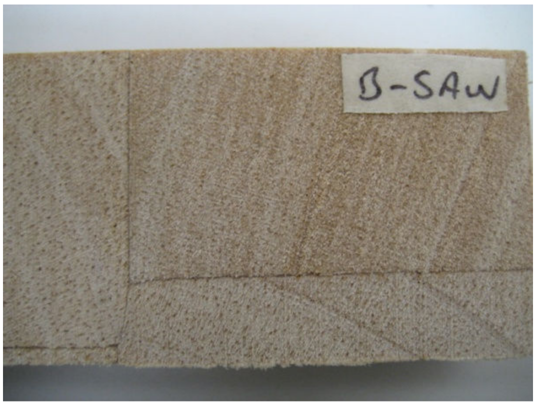


Image 0-1

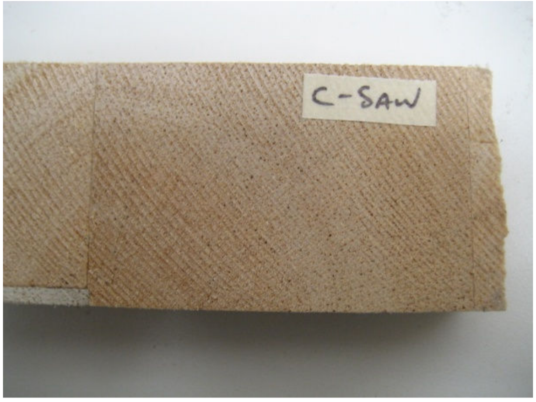


Image 0-2

Image 0-3

Image 0-4

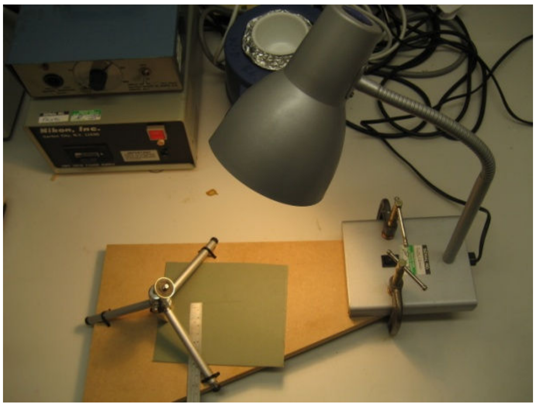
Image 0-5

Image 0-6

Image 0-7

Image 0-8

Image 0-9



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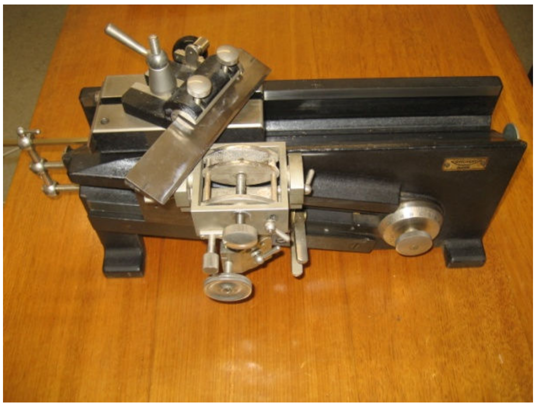
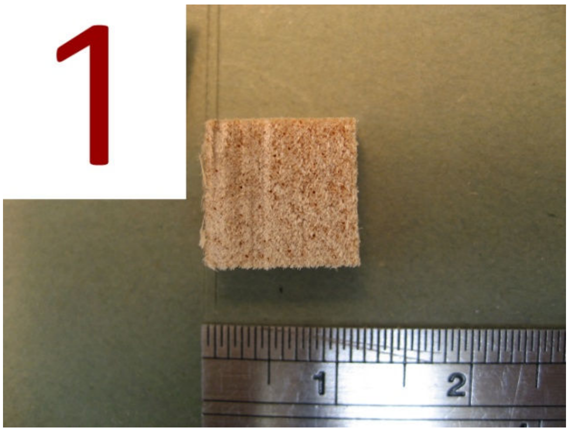


Image 0-11

Test 1



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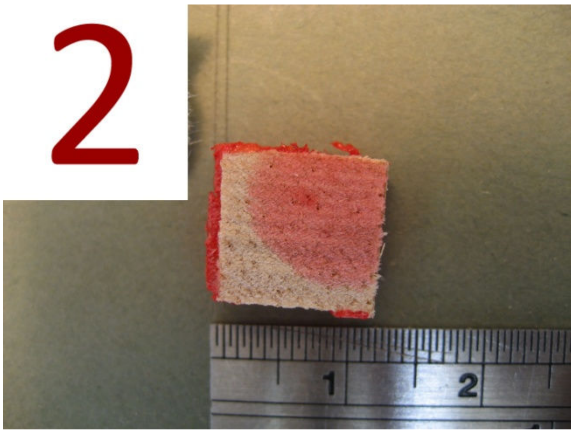

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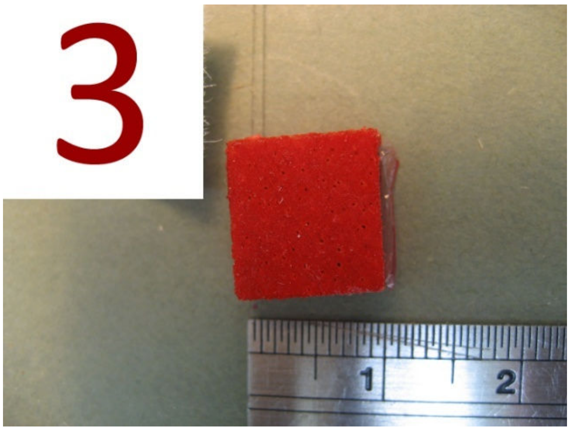

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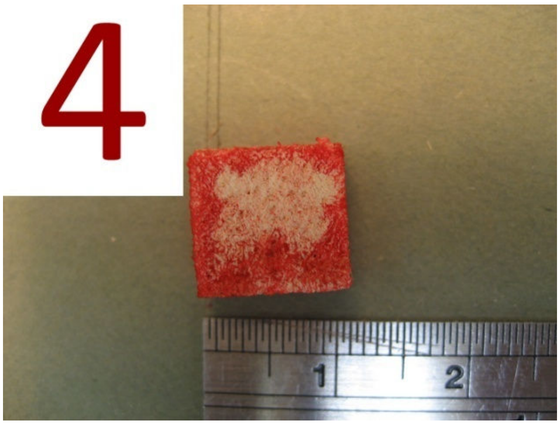

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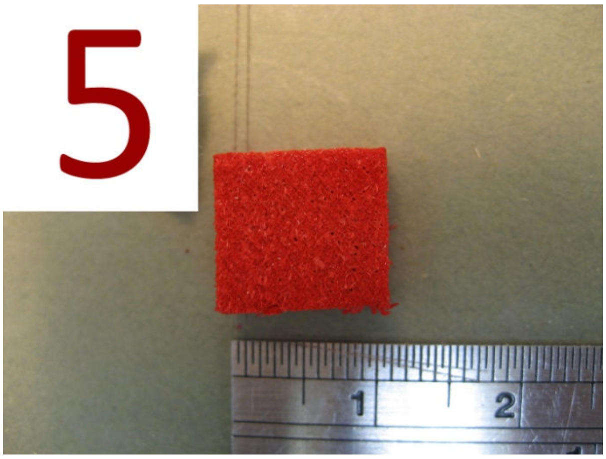

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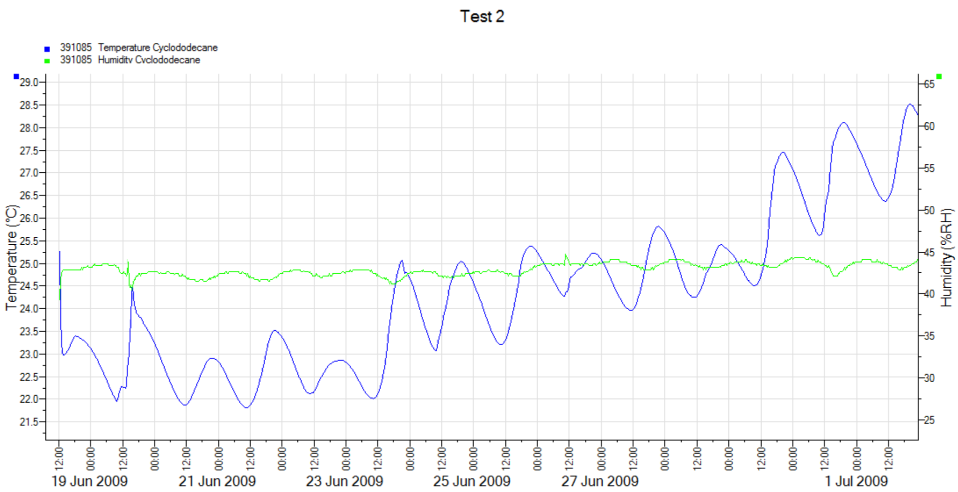
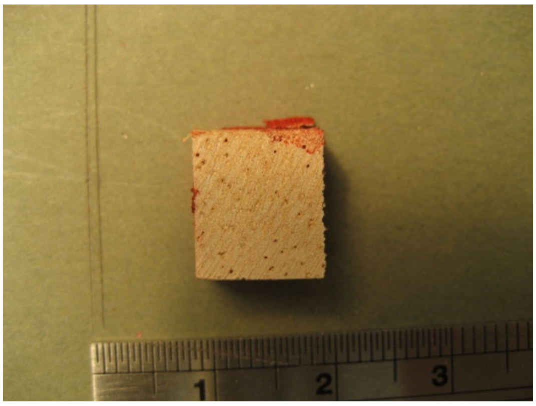


Image 2A-1

Image 2A-2



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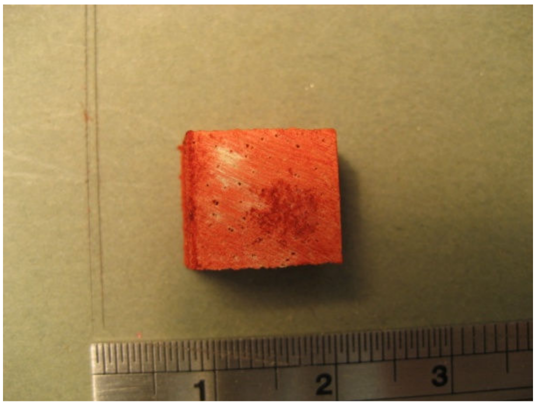

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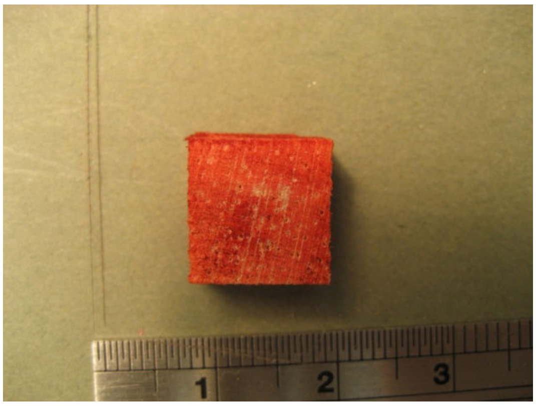

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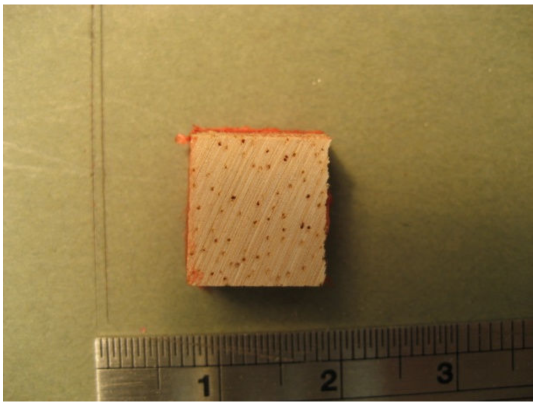

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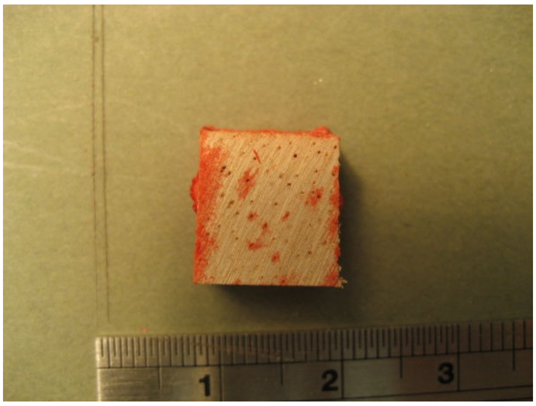

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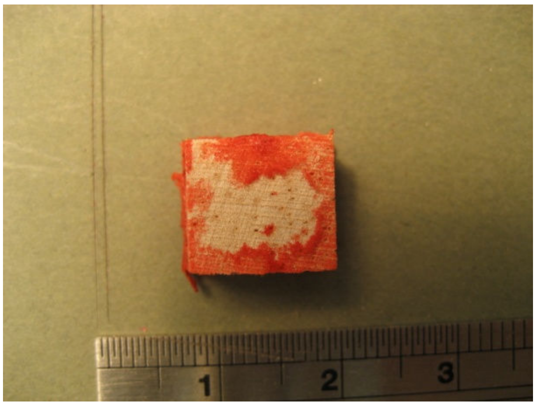

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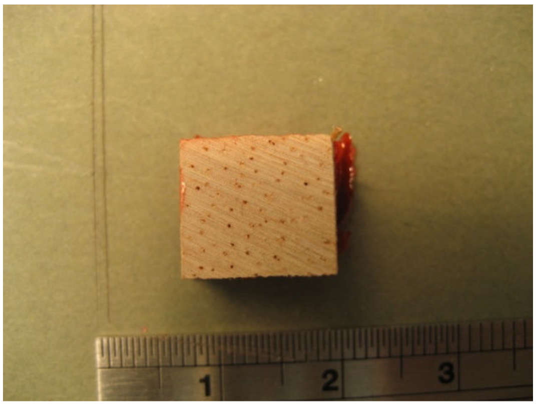

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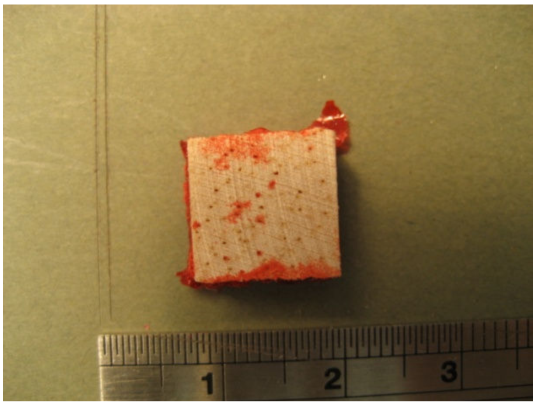

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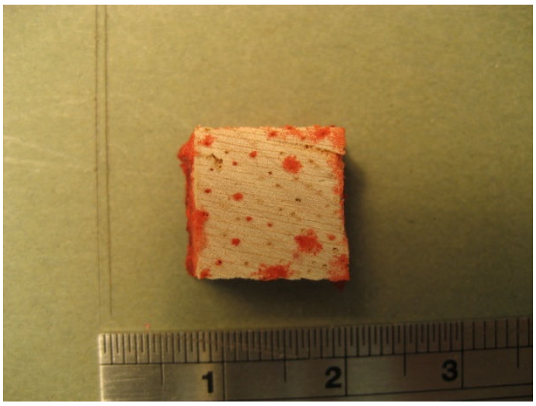

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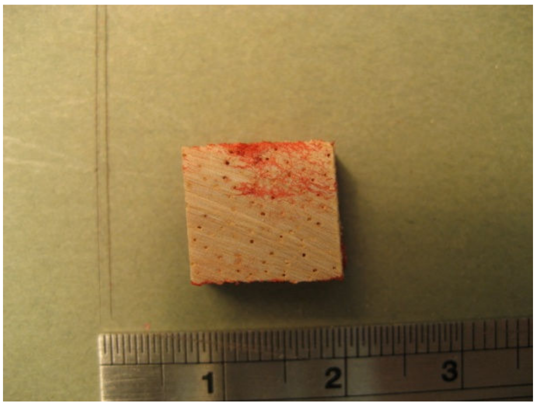

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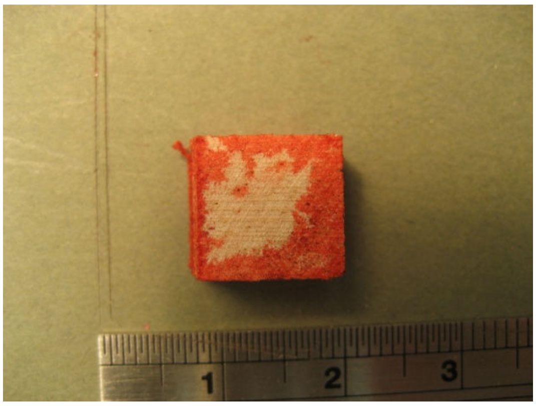

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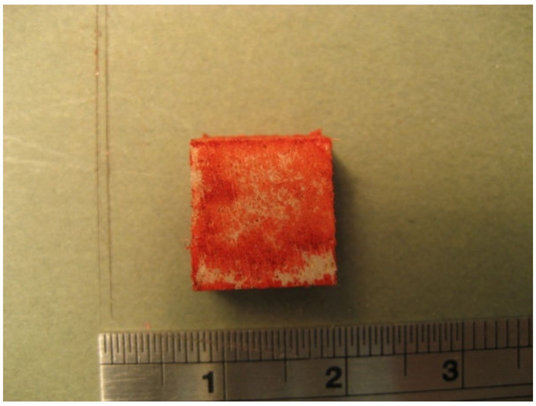

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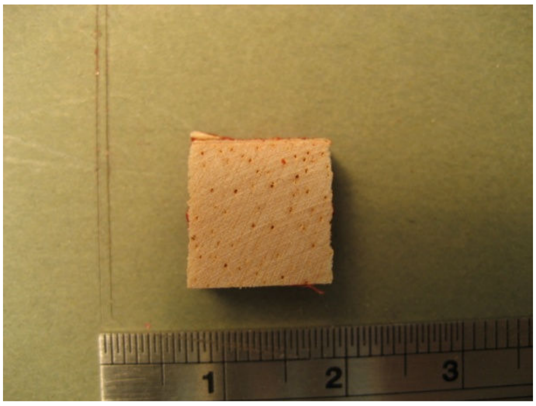

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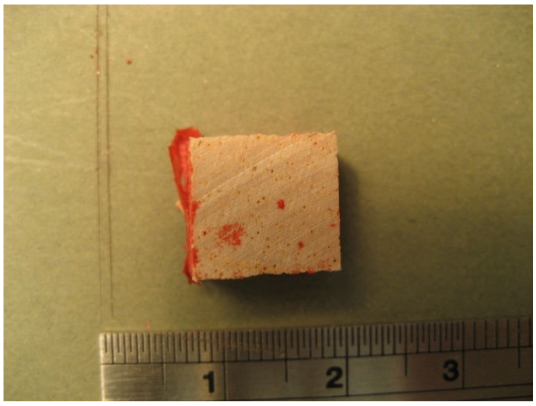

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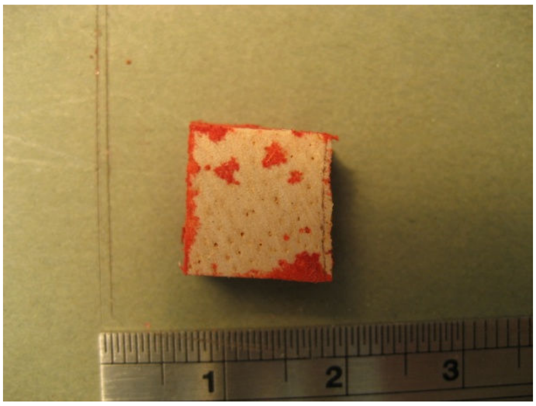

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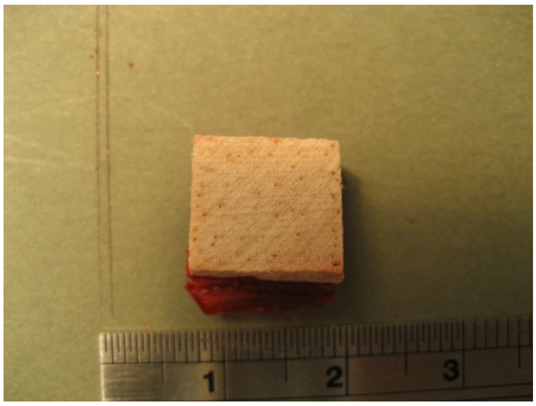

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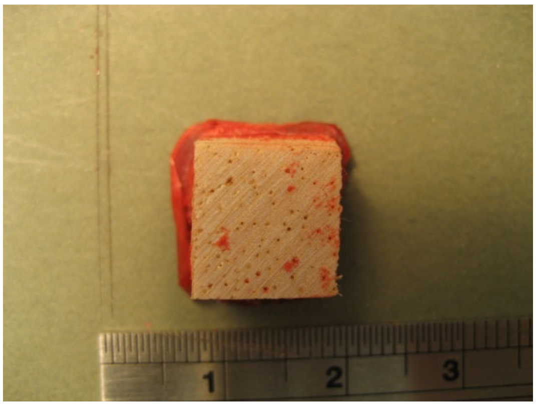

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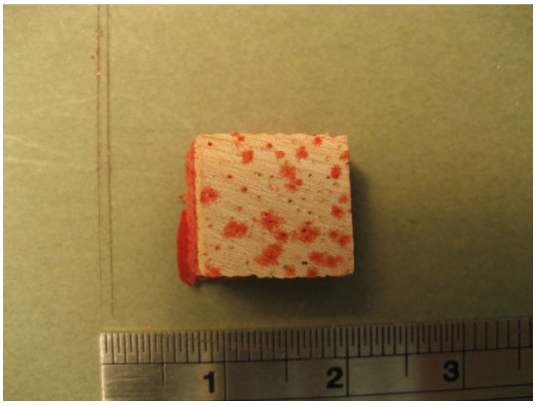
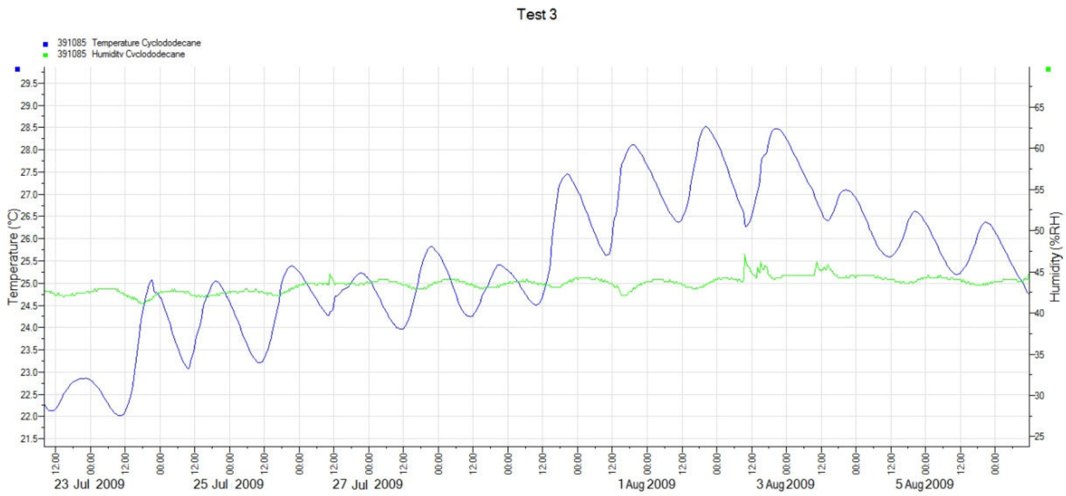


Image 2-18

Test 3



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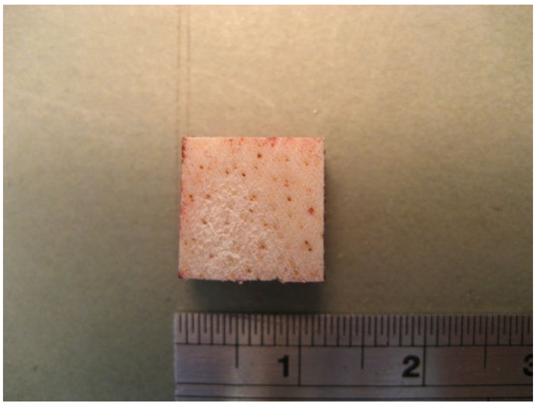

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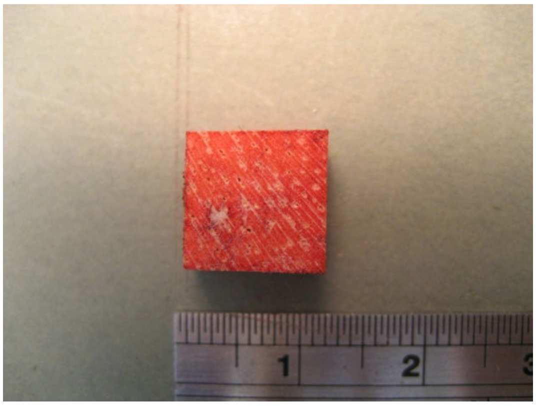

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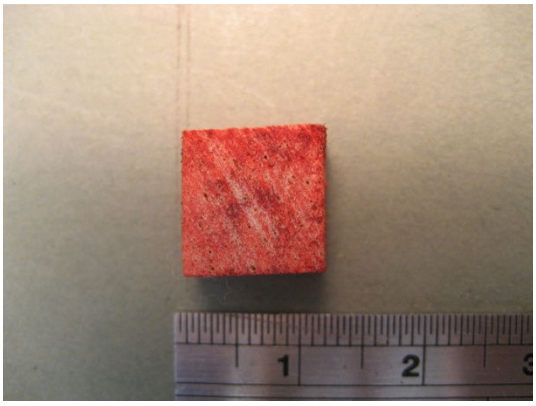

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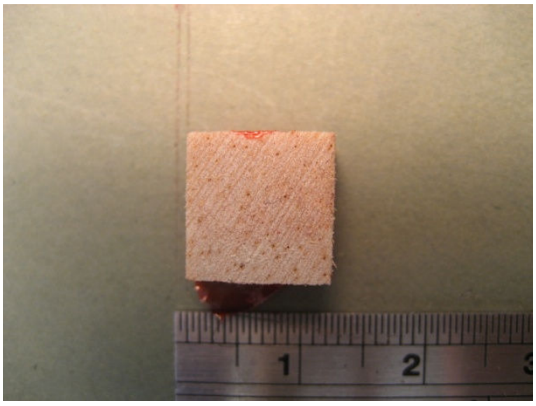

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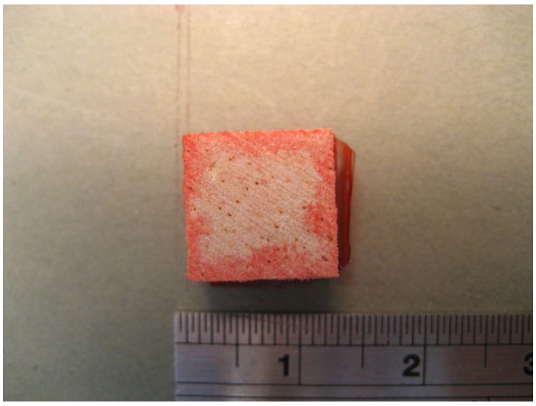

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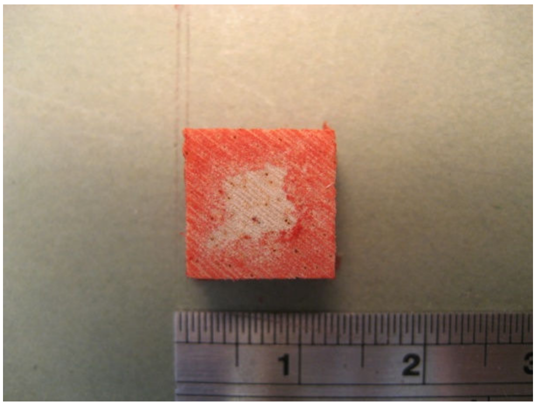

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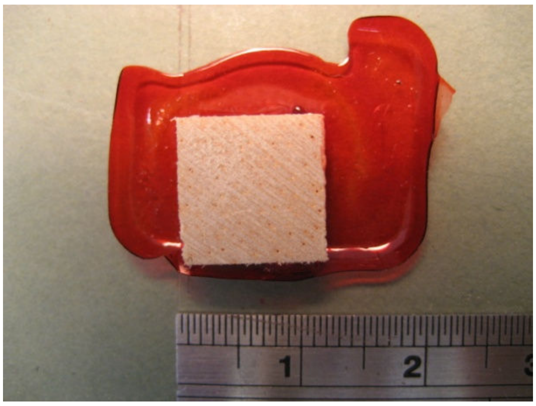

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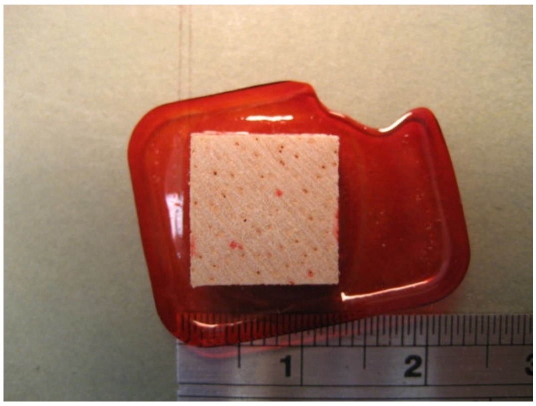

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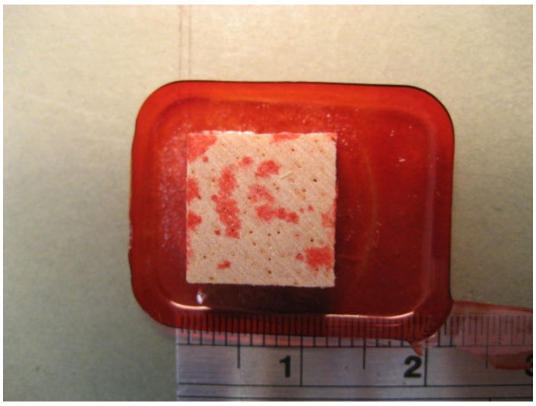

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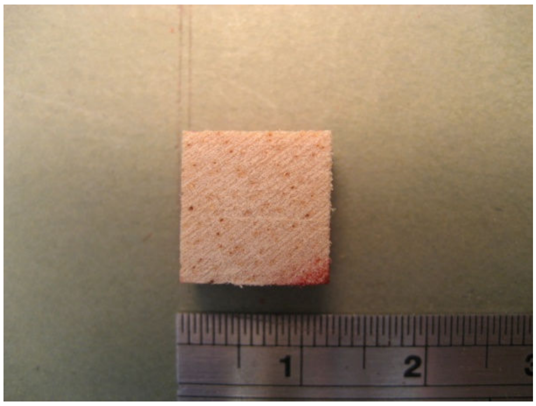

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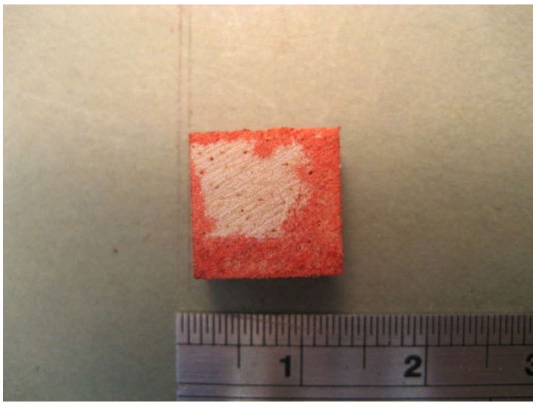

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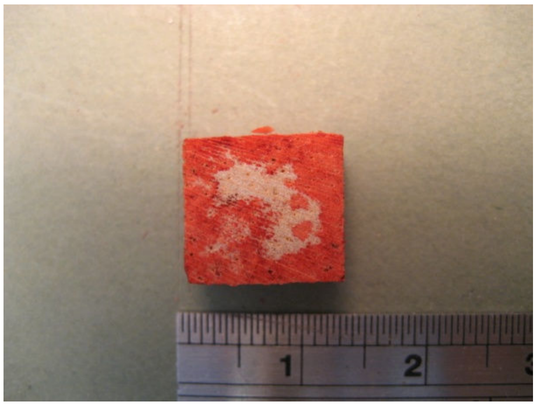

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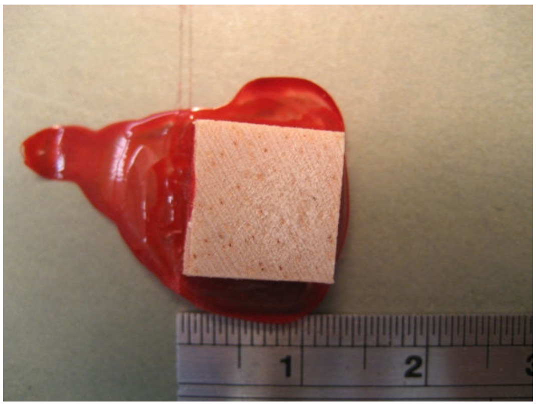

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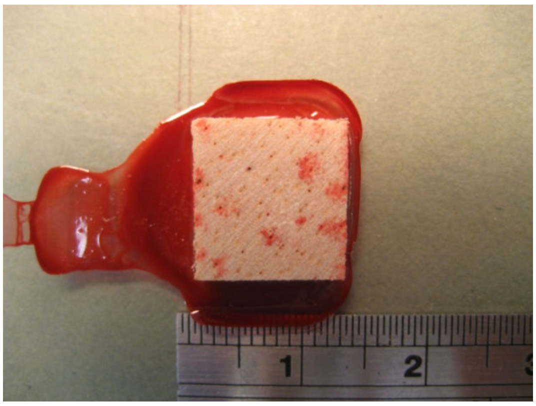

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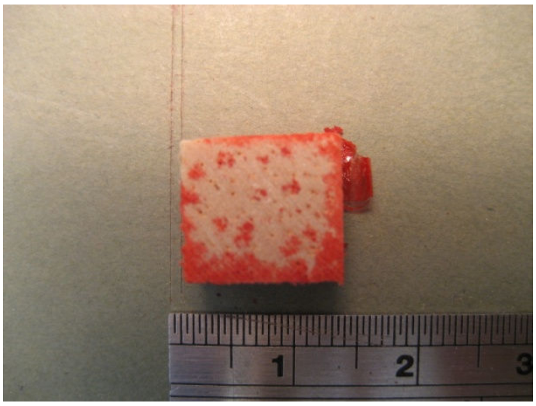

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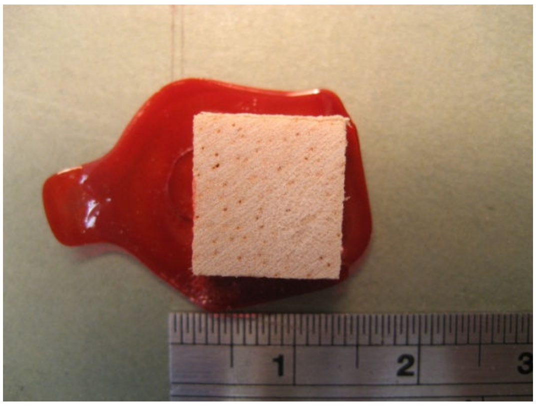

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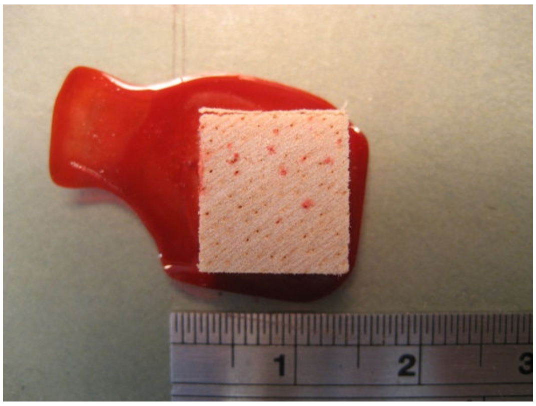

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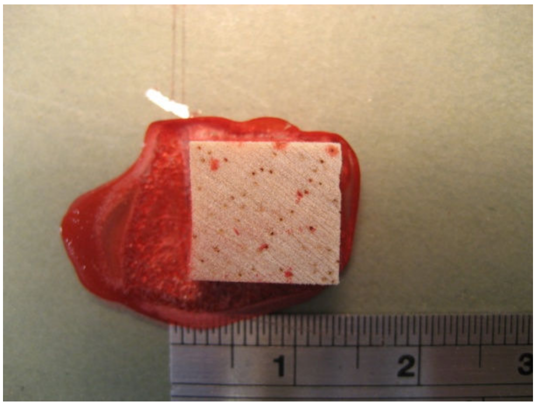

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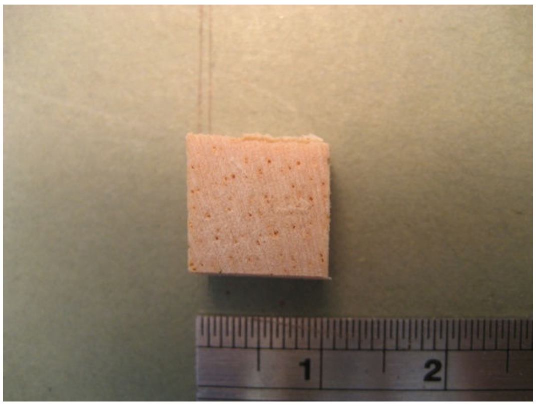

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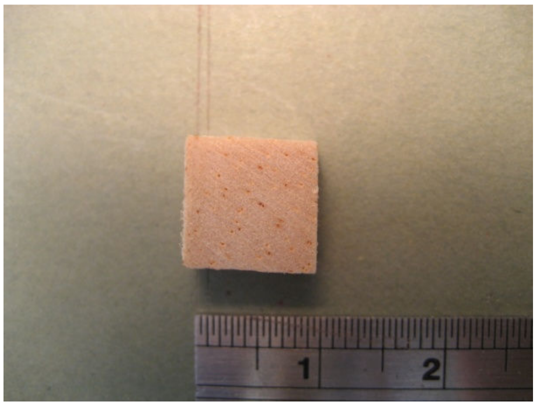

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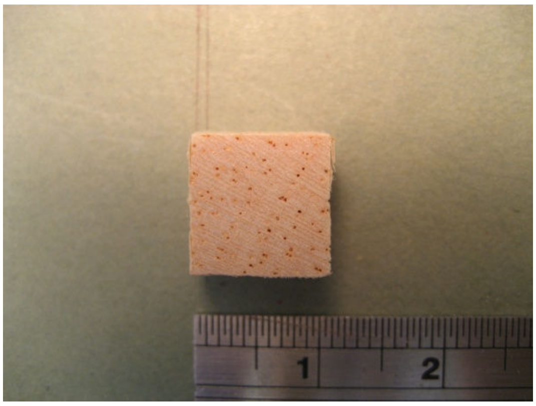

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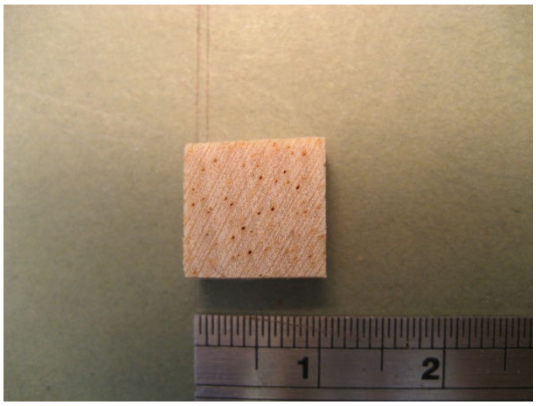

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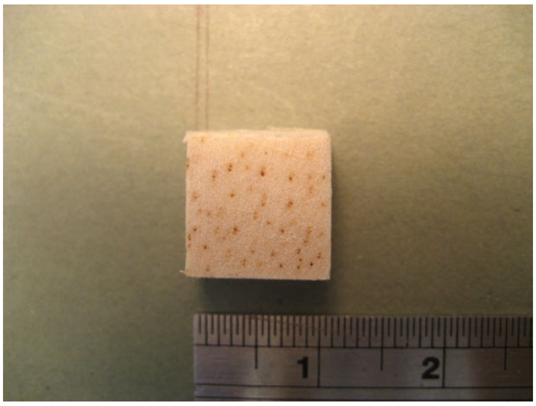

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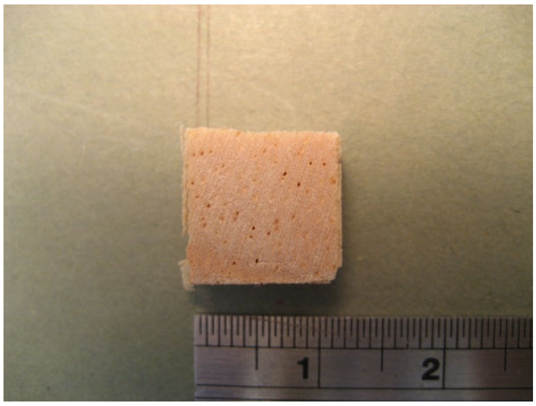

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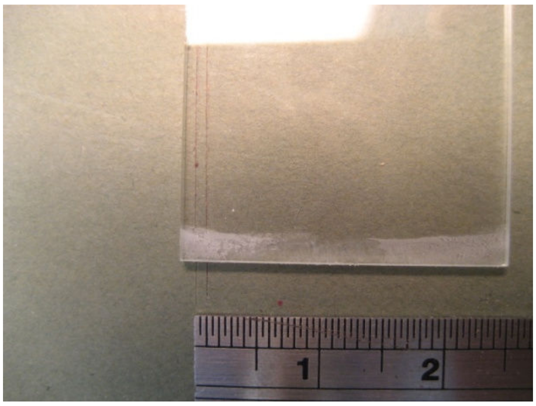

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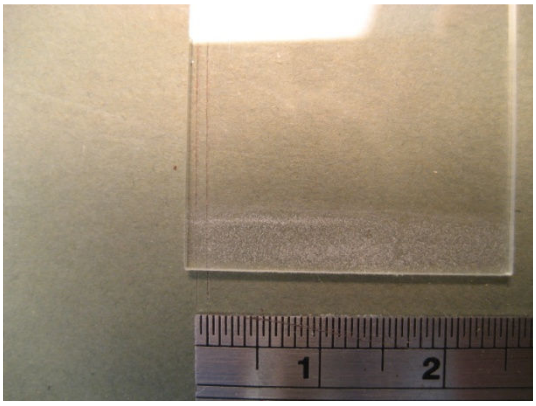

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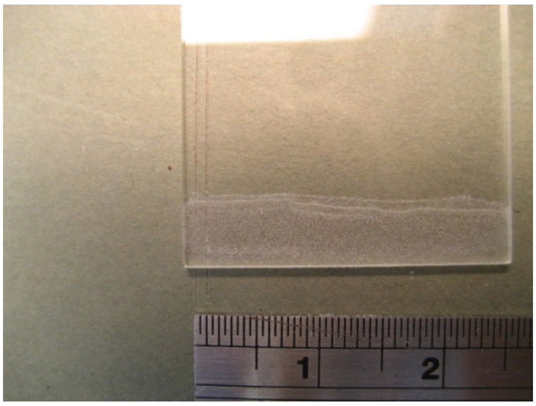

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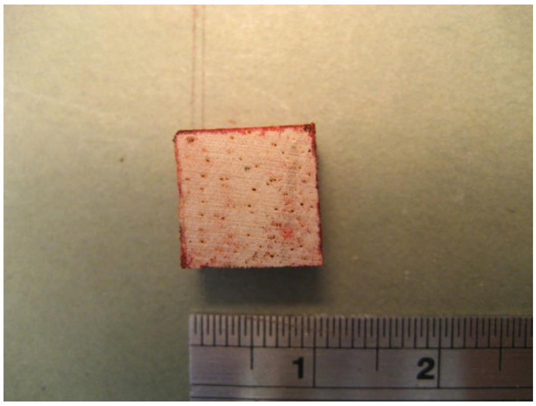

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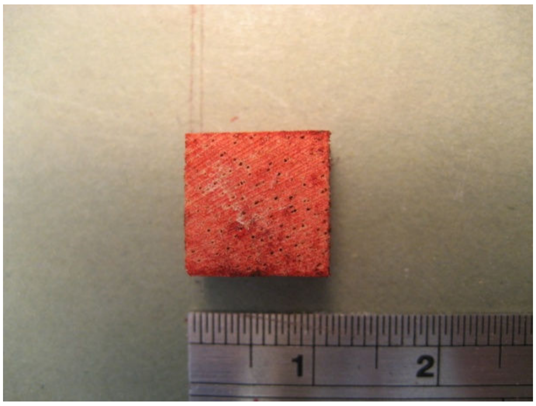

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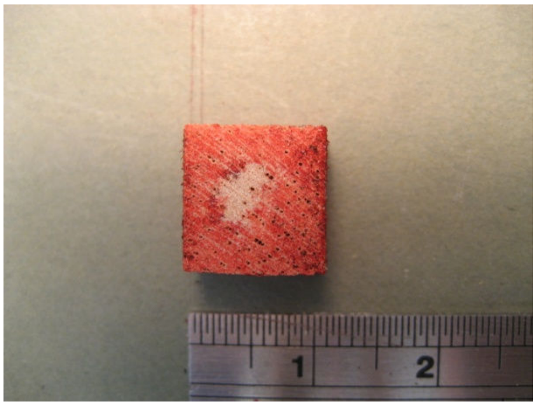

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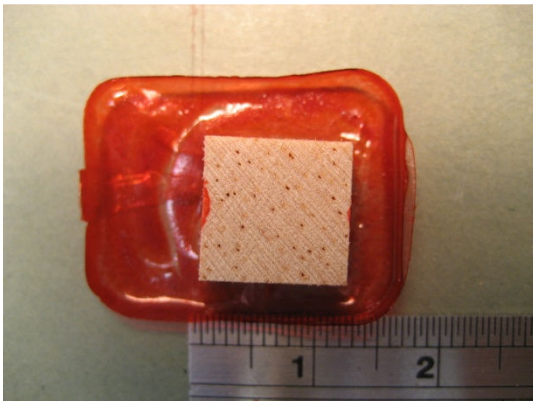

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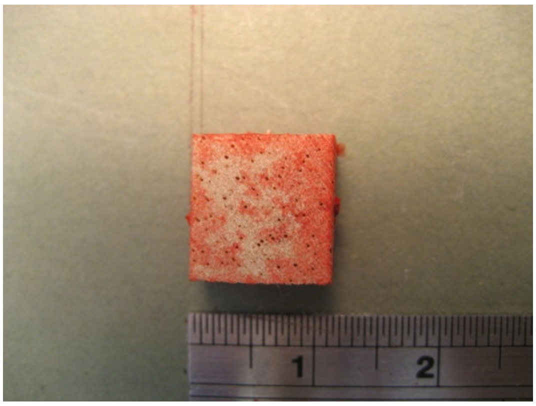

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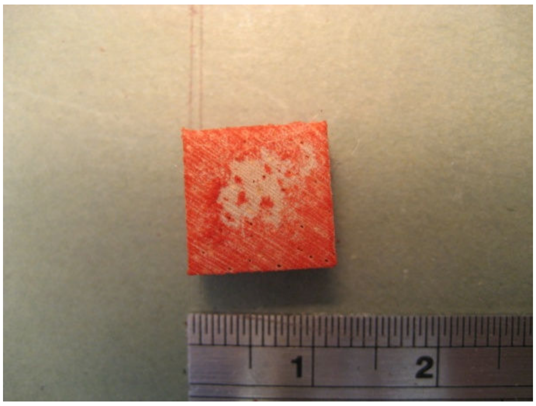

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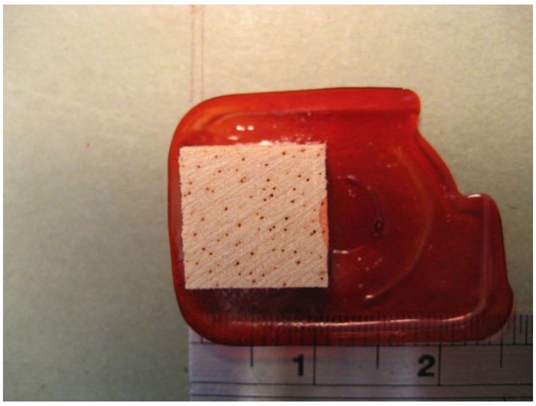

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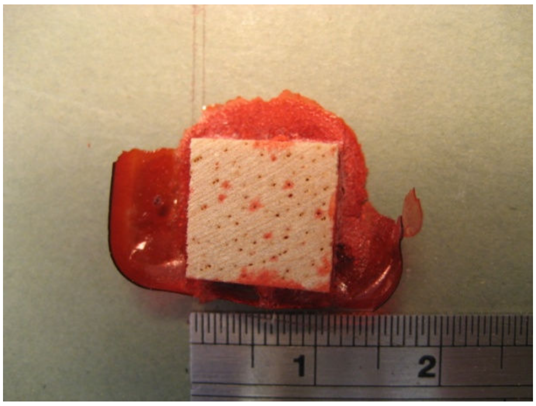

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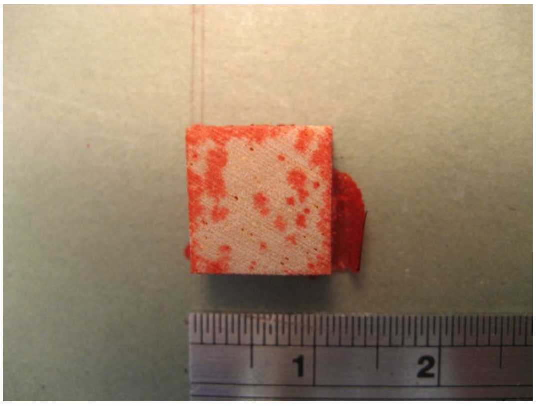

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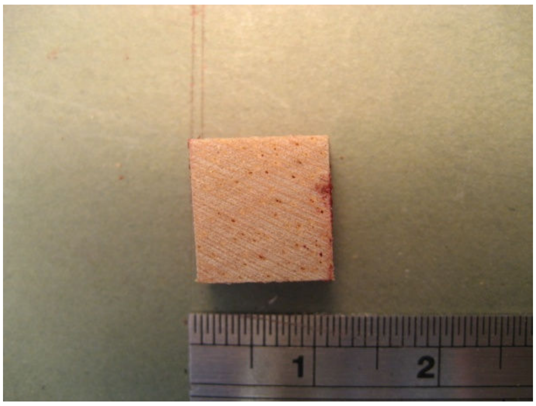

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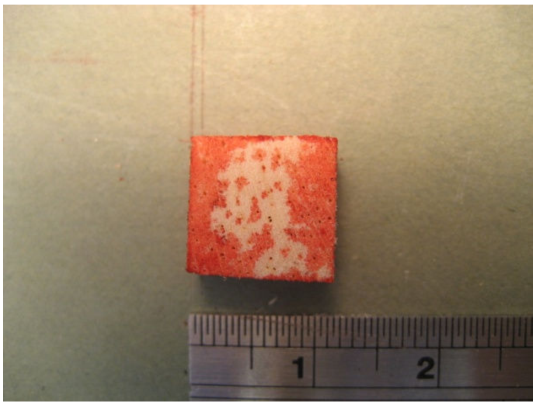

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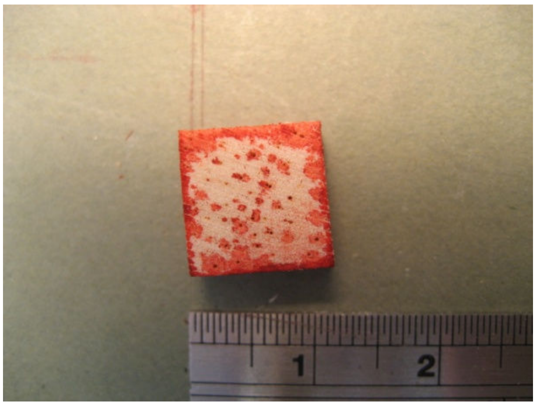

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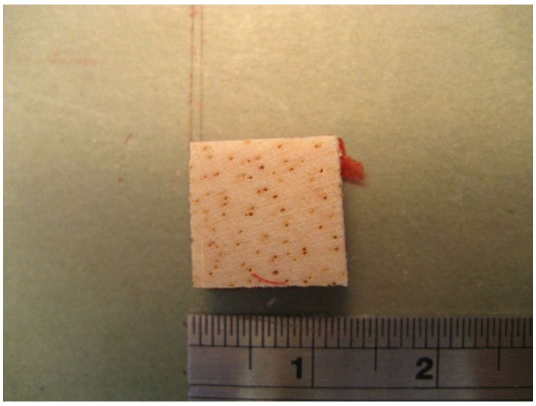

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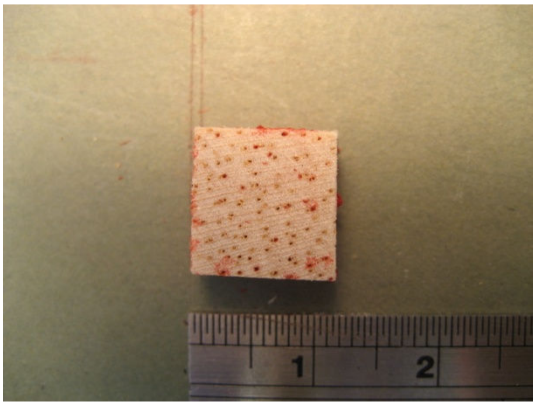

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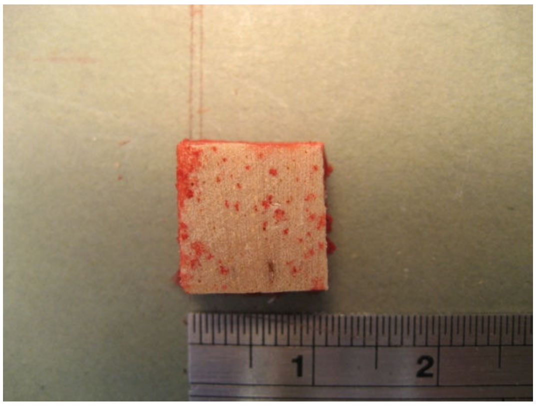

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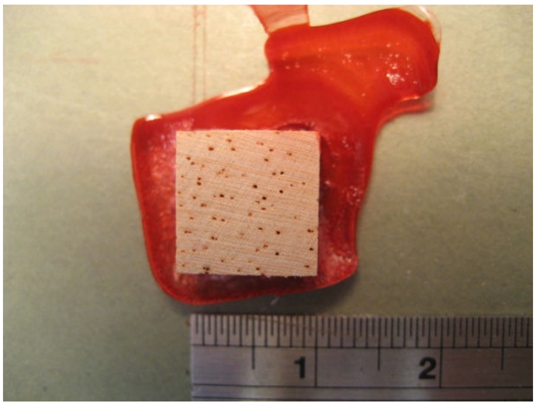

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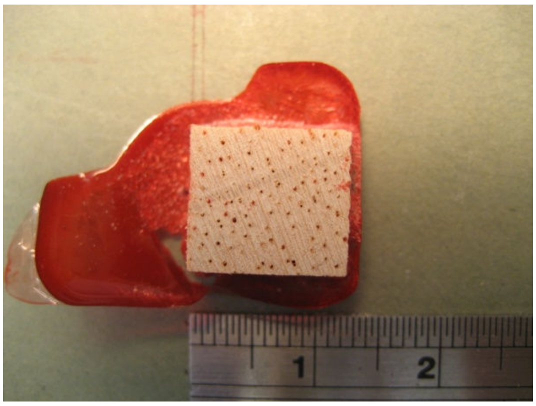

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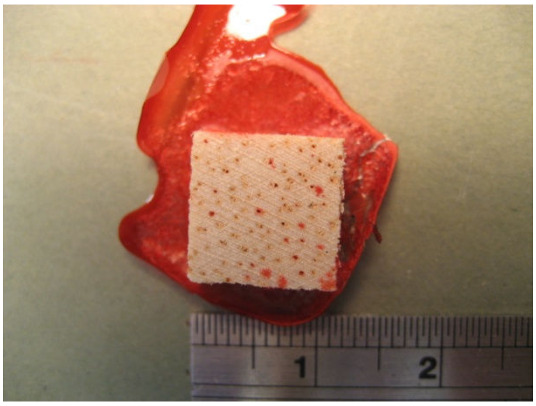

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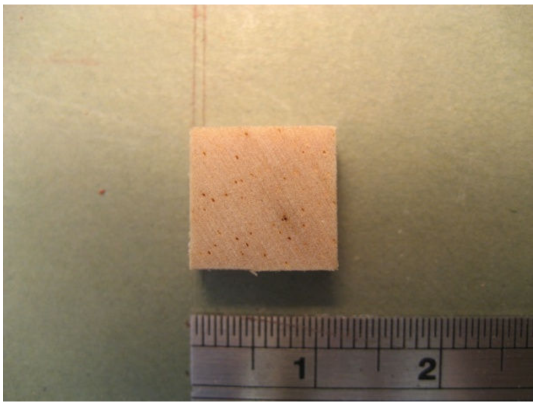

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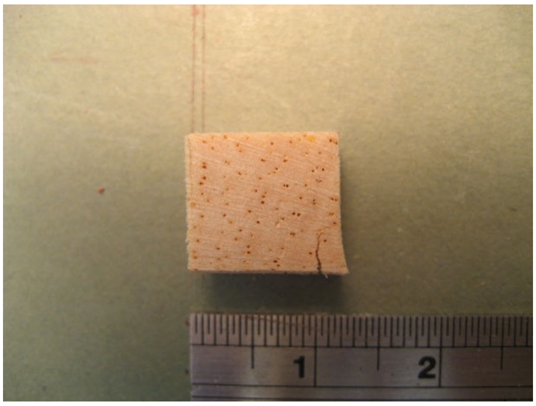

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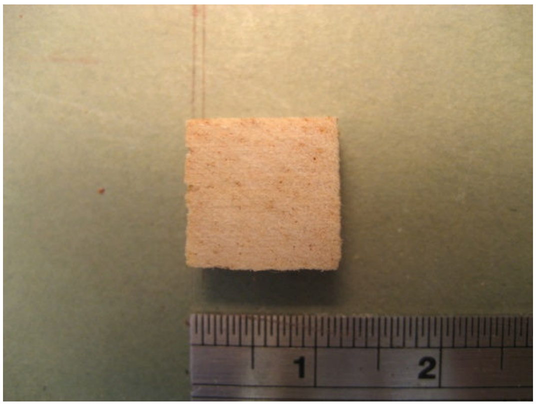

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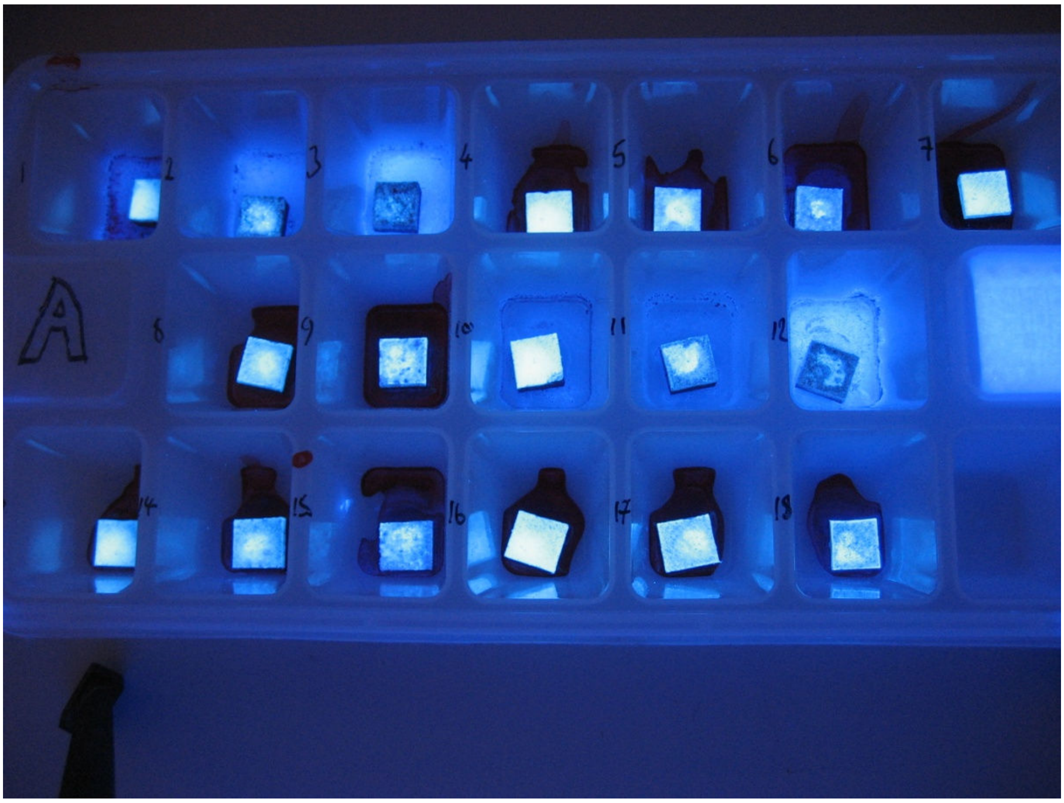
Image 3A-2

Image 3A-3

Image 3A-4



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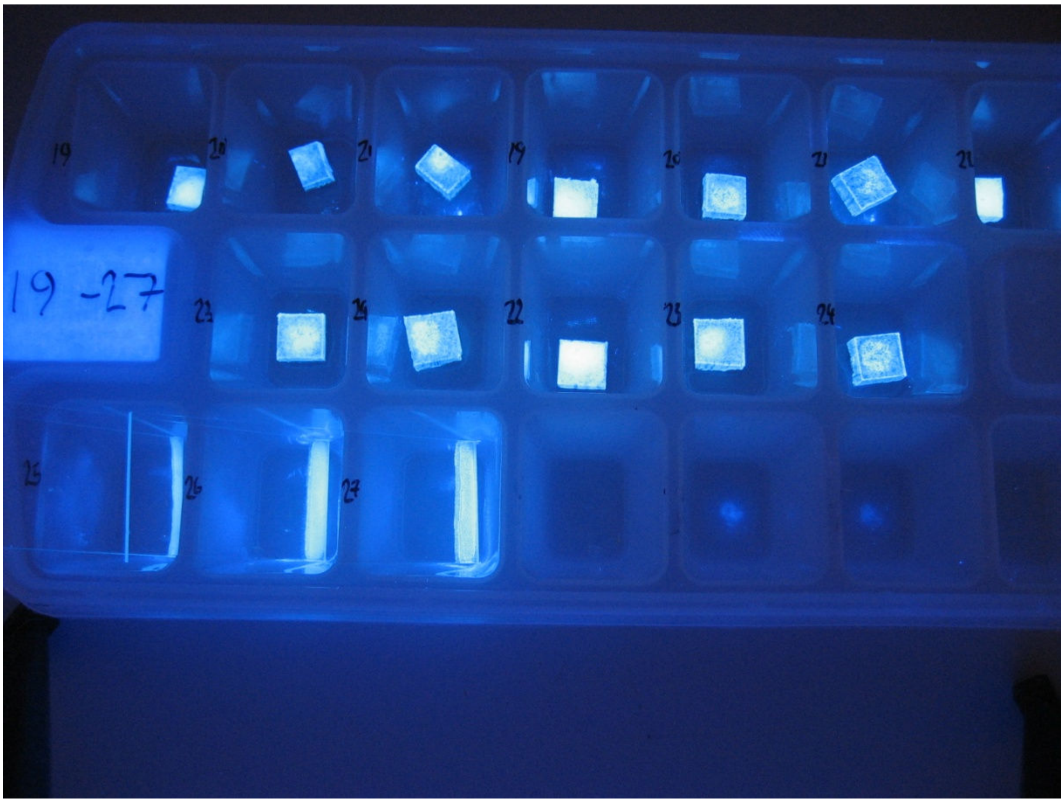

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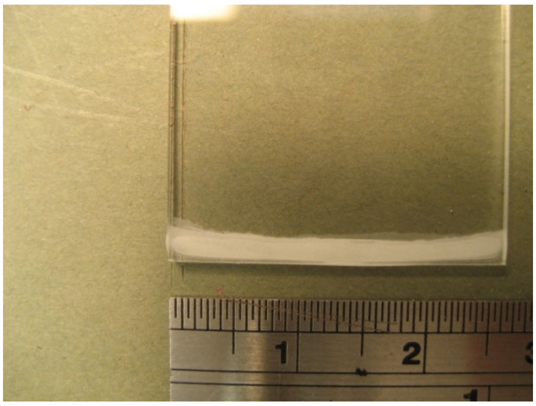

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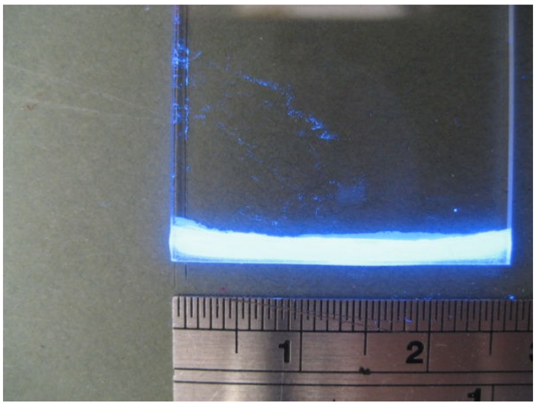

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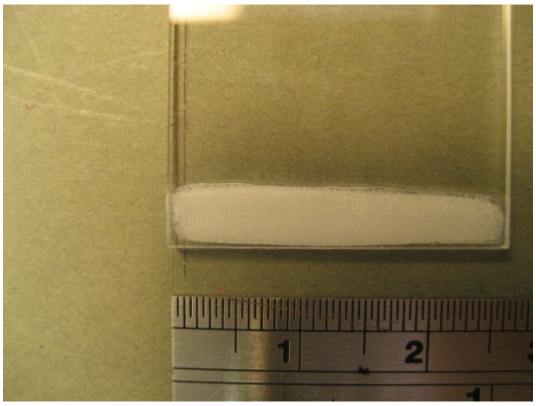

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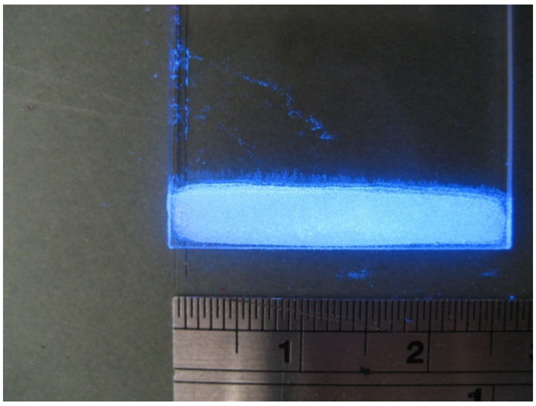

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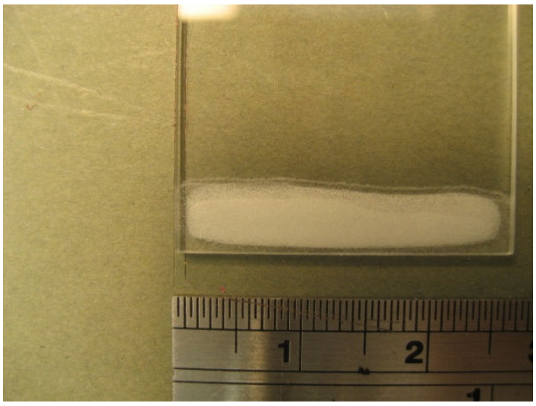

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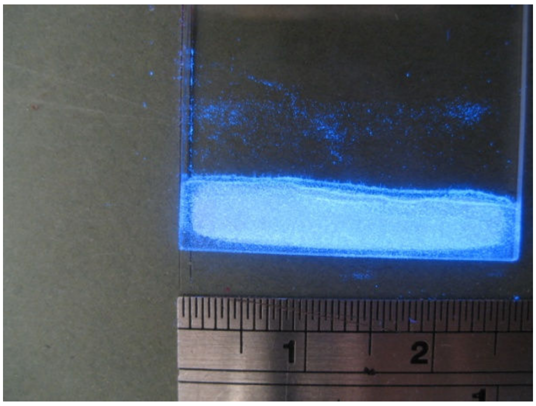

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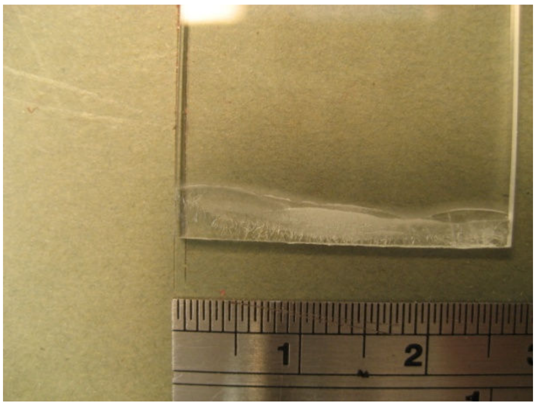

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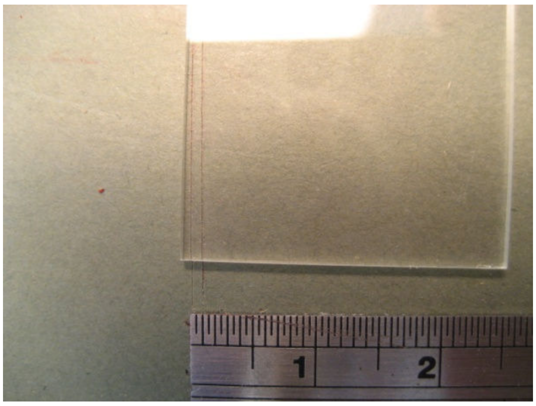

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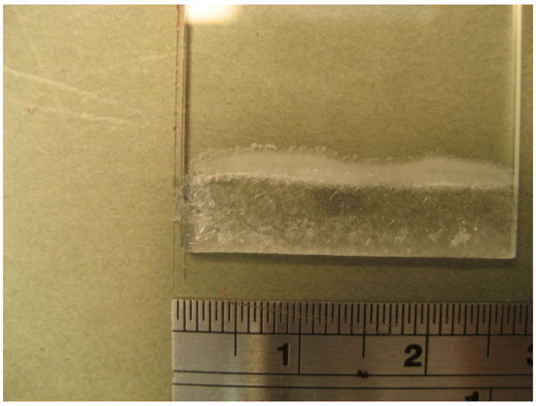

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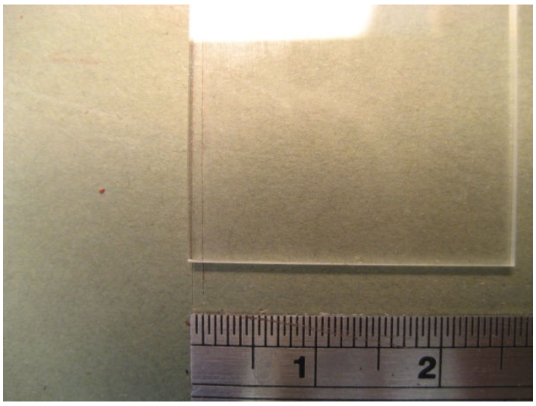

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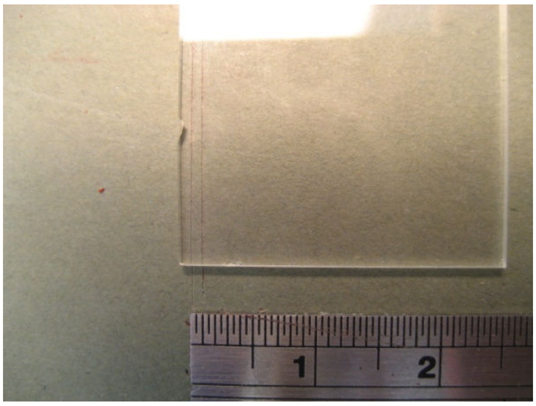

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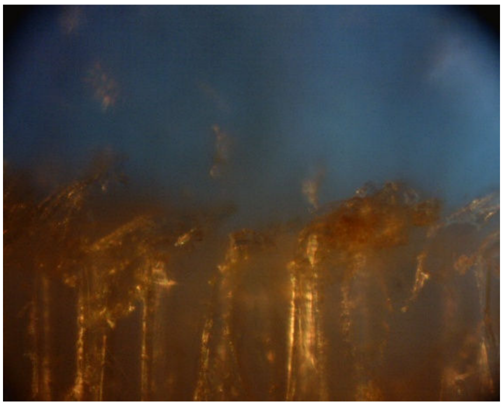

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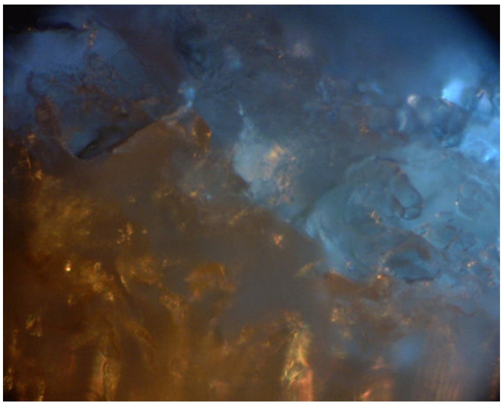
Image 3A-21



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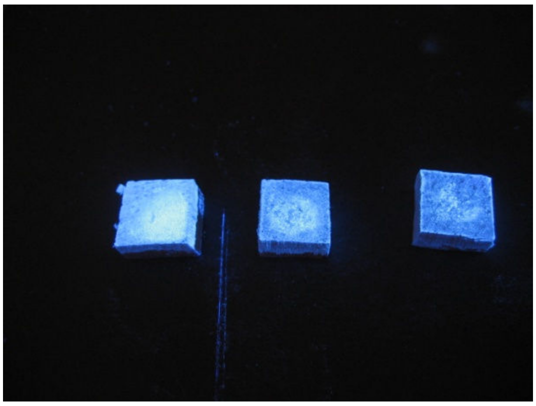

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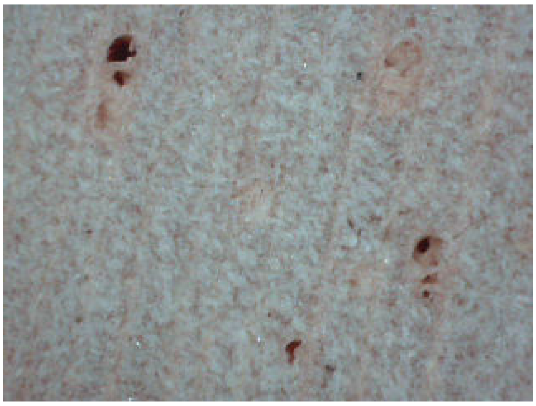

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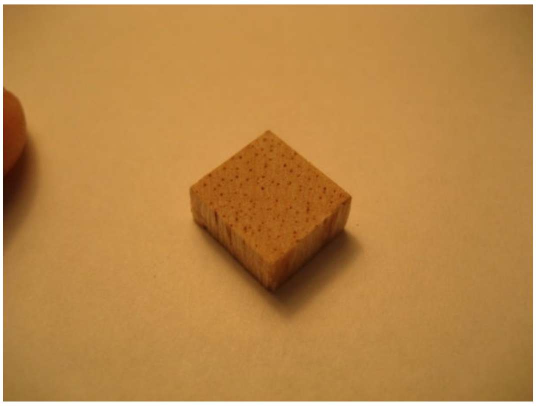

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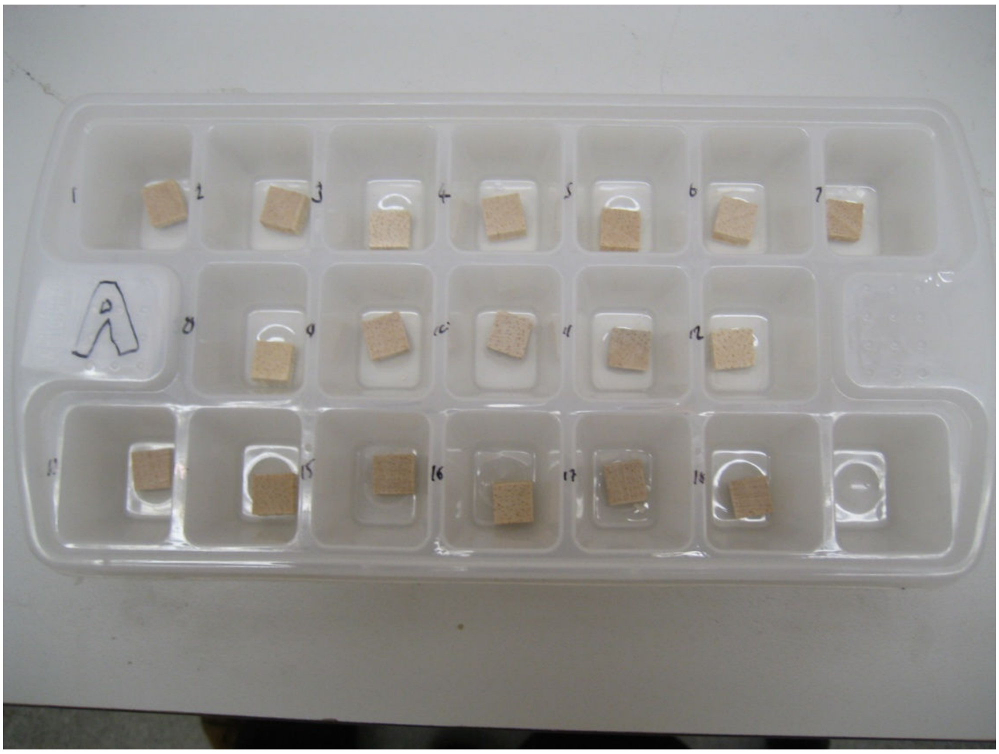

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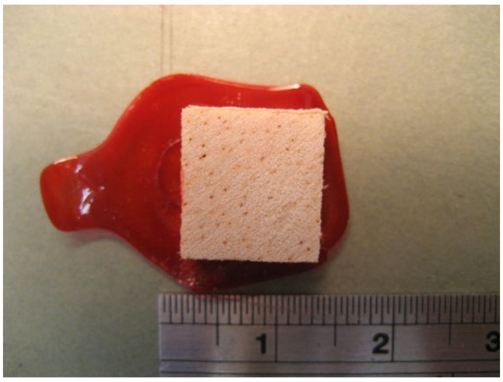

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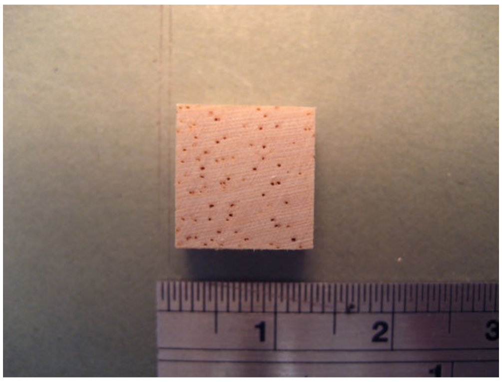

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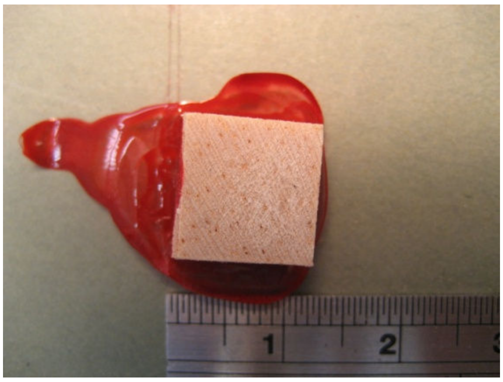

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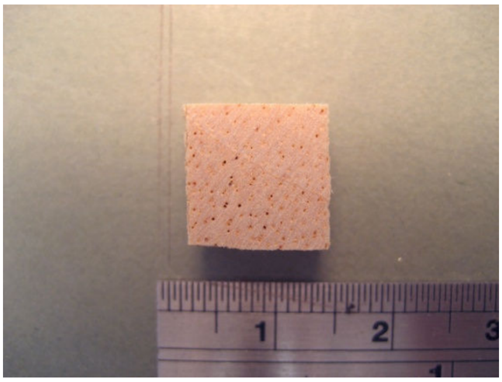

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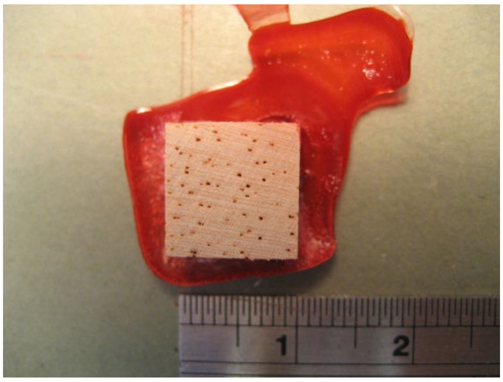

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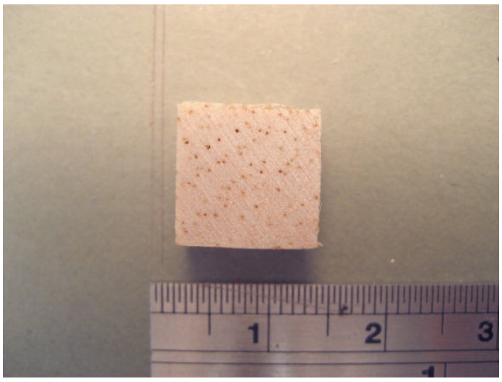

Image 3A-33

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Appendices


Acetone msds
B-98 msds
CDD tech data sheet
cubane sublimation
Cyclododecane msds
cyclododecane thermodynamics
IMS msds
Sellaset msds
Uvitex msds