Cyclododecane:
A Closer Look at Practical Issues

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ABSTRACT

Cyclododecane ($C_{12}H_{24}$) is a cyclic hydrocarbon that sublimes from a waxy solid to a gas at room temperature. This makes it an appealing material for treatments requiring a temporary condsolidant, adhesive, or barrier layer. Use in the field during the 2006 excavation season at Kaman-Kalehöyük suggests that cyclododecane is useful, but has some practical limitations including a lengthy sublimation time. In order to gain a better understanding of this material, previous publications are discussed along with the physical properties of the cyclododecane. Alternatives to the current melted application method are explored in an effort to decrease sublimation time in order to decrease the wait between the time of facing and treatment. The disadvantages of using a solvent-borne application is noted, while possible advantages (and hazards) of adding a small amount of solvent to melted cyclododecane are discussed. The use of facing tissue impregnated with cyclododecane and reactivated with heat is purposed as a useful method for field consolidation.

Fig.1

1.1 CYCLODODECANE'S PHYSICAL PROPERTIES

Cyclododecane (abbreviated CDD) is a waxy 12-carbon cyclic hydrocarbon ($C_{12}H_{24}$) that is solid at room temperature. Due to its low vapor pressure, it undergoes sublimation (converts from a solid directly to a gas) at room temperature. The melting point of 58-60°C allows the material to be easily melted over a low flame, while the flash point and the fire point are high enough to make that endeavor reasonably safe. The working properties are similar to paraffin wax. While the toxicity has not been fully studied, it is generally considered inert and a low health risk. However, the potential off-gassing of volatile hydrocarbon gas and a lack of research are reasons to take all the standard precautions when working with CDD.

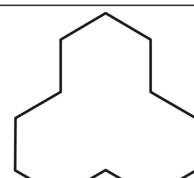
PHYSICAL PROPERTIES OF CYCLODODECANE		
Melting point	58-60 °C	
Boiling point	243 °C	
Flash point	98 °C	
Fire point	265 °C	
Vapor pressure	0.1 hPa	
Viscosity @65°C	2.2 mPa·s	
Toxicity	Not reported	Chemical structure of CDD

Fig.2

1.2 BACKGROUND

CDD was first suggested for conservation use in 1995 (Hangleiter, Jaegers and Jaegers 1995). Since then conservators have found a broad range of uses that take advantage of its sublimation ability. At Kaman-Kalehöyük, CDD is used as a temporary adhesive to attach facing tissue to an artifact when block lifting is required for safe transportation from the site back to the conservation lab. Choosing CDD for this treatment makes the procedure of removing the facing material simple and avoids additional stress on the artifact because the conservator needs only to wait for the CDD to sublime. Other alternative adhesives for this treatment, such as an acrylic or PVA resin, must be removed via solvent applied by swab, which can disturb fragile artifacts. Furthermore, while these polymers are often referred to as 'reversible adhesives' due to their favorable solubility, it is generally acknowledged that it is impossible to fully remove these resins once used on an object (Horie 1982). Perhaps of greater concern to the archaeological community is that these residues can alter the results of analytical techniques such as radiocarbon dating, DNA retrieval, isotope analysis, and scanning electron microscopy (Johnson 1994). The temporal nature of a CDD application offers an intriguing alternative to these other materials.

During the 2006 excavation, the conservation team used CDD to stabilize an iron artifact during a block lifting procedure by adhering loose-woven cotton gauze facing material to the surface of the artifact block. CDD was heated over a portable stove until molten and the CDD, which cooled nearly on contact, was applied with a brush through the facing material. CDD worked very effectively to secure the facing and had the added bonus of not requiring removal with solvents. However, it required approximately six weeks before it sublimed completely (apparently), leaving the cotton gauze unattached on the surface. Fortunately, the treatment was minimal, the object incurred no addition deterioration by remaining in the block assembly, and there were no deadlines for treatment save the end of the season. However, the lengthy sublimation time may in some situations be such a drawback that CDD is not considered an option. This paper aims to expand treatment options in situations with time constraints and to further understanding of working properties inherent in this material. Due to increasing instrumental sophistication at Kaman-Kalehöyük, CDD may have an important role to play in archaeological conservation.

2.1 THE RESIDUE QUESTION

Although no residue is visible after the CDD has sublimed from an artifact, the question of whether it actually leaves any residue is still debated in the literature. In 2000, Stein *et al*. conducted gas chromatography-mass spectroscopy (GC-MS) on solid crystals of CDD and extracts from ground, porous stone substrates after complete sublimation (gauged by weight) and noted "absence of detectable residue." These findings contrast a study by Caspi and Kaplan in 2001, when samples of "a white crystalline material" were sent to several labs for FTIR spectroscopy and GC-MS analysis. The residue was found to be composed of various hydrocarbons chemically similar to CDD, perhaps dimers that with an increased molecular weight could not sublime. However, no impurities were found when testing solid CDD crystals and it was determined that the residue found "does not present a threat to the substrate." This contradiction could also stem from differences in the commercial preparation of individual batches, which is why many conservators have recommended testing individual CDD stocks for impurities (Caspi and Kaplan 2001). Beyond the question of whether residues are a threat to the object, these residues might also bias analysis of the artifact. If present, the CDD residues are likely to be less than the residue left by conventional adhesive polymers after removal. However, since one of the advantages of CDD, as a material choice, is the that it will not bias artifact analysis, further testing of what effect CDD residue may have on various analytic techniques is recommended.

2.2 PUBLISHED USES OF CYCLODODECANE

While compared to other polymers, CDD is not a strong adhesive or consolidant, its ability to sublime often makes it a useful material in certain treatment situations. Past uses of CDD described in conservation literature generally fall into three main categories:

consolidant, adhesive, and barrier layer (*e.g.* Brückle *et al.* 1999; Caspi and Kaplan 2001; Maish and Risser 2002; Muros and Hirx 2004). It is reported to be useful as a temporary consolidant to protect fragile ceramics while moving, allowing easier handling and packing without damage to the objects (Caspi and Kaplan 2001). A similar treatment was reported with a deteriorated limestone column and a significant savings in labor was noted (Stein *et al*. 2000). CDD has also been reported to aid in the sampling of friable mortar, as a temporary consolidant. As a temporary adhesive it has been useful in instances where subsequent removal of an adhesive was impossible without damage to the object. It has been used to hold paper dams in place when selectively making a mold from a fragile fossil (Arenstein *et al*. 2004).

As a barrier layer CDD has been reported as particularly successful when used as protection against aqueous solutions. Examples of this include: applications on paper, ceramics, and textiles (Brückle *et al*. 1999; Muros and Hirx 2004; Larochette 2004). In each reported case, the treatment required the object to be immersed in an aqueous solution, yet contained small areas of water-sensitive media. After being applied molten, CDD served as an effective masking agent, allowing the objects to be immersed in an aqueous solution with no loss to the water-sensitive areas of the objects. Preparing an object for molding by filling its voids or cavities with CDD is another use as a barrier layer. When used in conjunction with other polymers, a thin coat of CDD applied first to an artifact prevents the isolating polymer layer from penetrating into the artifact's pores or voids, where it would be difficult to remove. The combination of CDD with a methylcellulose or gum Arabic isolating layer is noted as a particularly effective in porous ceramics (Brückle *et al*. 1999). This is a treatment that may be particularly useful at Kaman-Kalehöyük when taking impressions from cylinder seals. A similar idea uses CDD as a void-filling material for deep cracks or fissures when molding with latex or silicone rubber material. These molding materials have a low surface tension that allow them to flow into the smallest crevice yielding a high fidelity mold, but consequently can be impossible to remove. CDD is reported to be an excellent choice for this treatment because it will eventually sublime out of inaccessible cracks that might permanently retain other polymers (Brown 2006; Maish 2002).

3.1 CYCLODODECANE SUBLIMATION RATES AND THEIR IMPLICATIONS

Though the conservation staff at Kaman-Kalehöyük had previous experience with CDD, the slow rate at which it sublimed in the 2006 season was still surprising. The layer was approximately 1-3 mm thick and it was set near a south-facing window in the lab, eventually taking over a full month to sublime. Studies suggest that an average rate of CDD sublimation from a non-porous substrate is 30 days/mm of melted film and longer for porous substrates (Arenstein 2004). However, the rate of sublimation depends on various factors: the thickness of the film, the temperature of the material, the porosity of the substrate, and airflow (Stein *et al*. 2000). Conservators often use these factors to either speed up or slow down sublimation times. To speed up sublimation the object can be placed in a warm place with constant air circulation. Fans or hair dryers are often employed, as well as an area receiving direct sun (if this does not adversely affect the object). If sublimation is undesirable then airflow around the treated object can be reduced using layers of aluminum foil and/or plastic bags. Though it is beyond the scope of this paper to quantify to what extent each of these factors affect the sublimation rate, Fig.3.1a illustrates how dramatically the thickness of the film affects this rate.

To look at sublimation rates in a controlled manner, four different containers with melted films of CDD were monitored for weight loss. In two identical sets of glassware containers, one set with a diameter of 26mm and the other with a diameter of 53mm, weights of 3g and 7g CDD were melted. The containers, set aside in the lab, were exposed to identical conditions of ambient airflow, temperature, etc. and periodically weighed. The 53mm/3g sample was the first to sublime completely, followed by the 53mm/7g sample. After over six months, both 26mm containers still had not sublimed completely. Fig.3.1a shows the percent weight loss while Fig. 3.1b

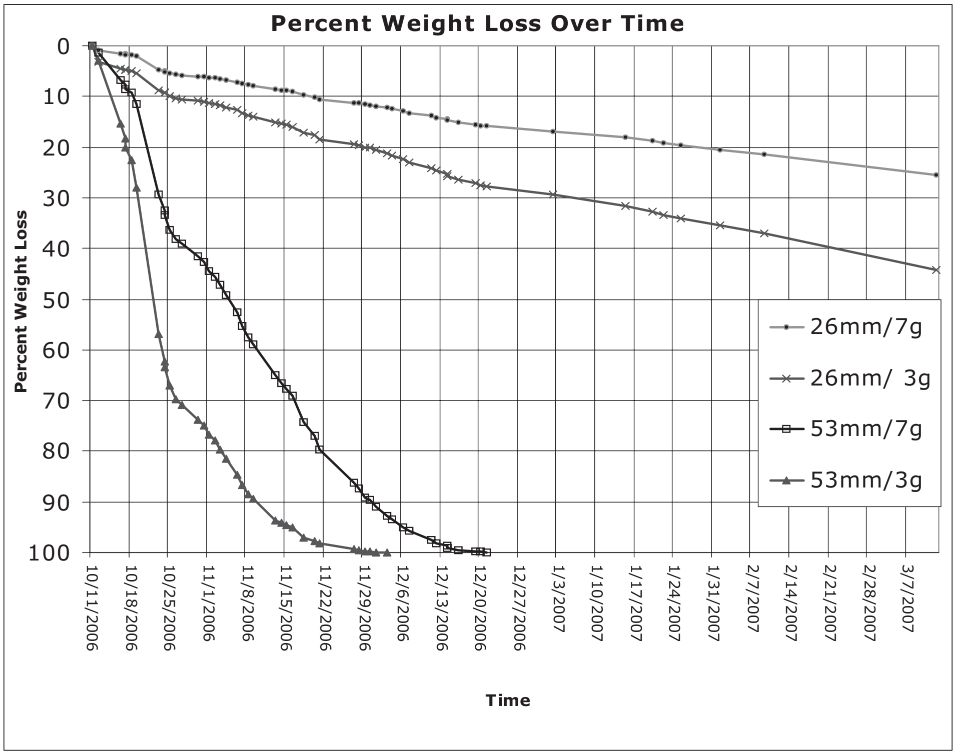


Fig.3.1a Percent weight loss of cyclododecane over time

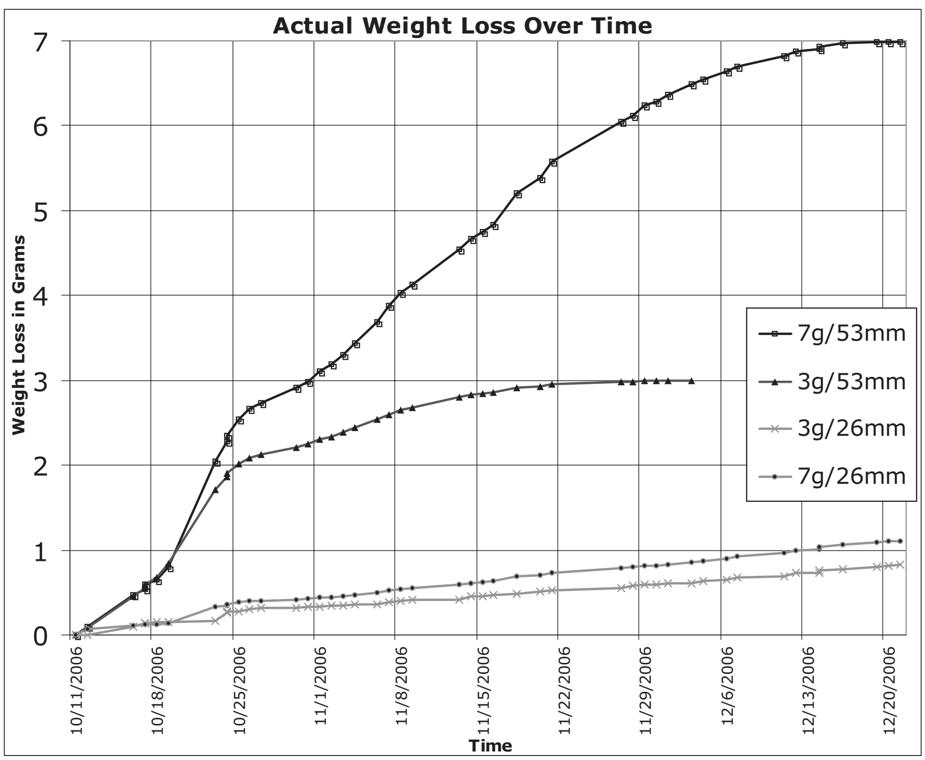


Fig.3.1b Actual weight loss of cyclododecane over time

shows the actual weight loss over time. In Fig.3.1a for both sets of samples (26mm and 53mm), the sample with the lesser mass and consequently thinner film, sublimed at a faster overall rate. These graphs demonstrate two factors that directly relate to the length of sublimation time: film thickness and surface area. Though this is rather a matter of common sense, but it is helpful to empirically confirm.

Sublimation of CDD appears to function solely according to the reaction rate of a thin surface layer of molecules. This implication is most obvious when comparing the 53mm samples in Fig.3.1a and Fig.3.1b. During the initial part of the experiment, the slope of the 53mm/3g line is much steeper than the 53mm/7g line, but during the same time period in Fig.3.1b the lines representing the two samples overlap for the beginning portion of the experiment. This is because they are subliming the same actual weight of material evident in Fig.3.1b, but since the 53mm/3g sample contains less mass the weight percentage loss is higher resulting in a steeper slope in Fig.3.1a. Though this requires more testing toConfirm, the lines probably only diverge when the bottom of the 53mm/3g container was reached, drastically altering the surface area. This same pattern is also evident in the 26mm sample set, though to a lesser degree (perhaps due to the slower overall rate of sublimation). It is reasonable to assume the fraction that is actively subliming is only the top layer as opposed to the entire mass actively diffusing into the air. This means that surface area and film thickness are crucial in determining sublimation time. Since surface area is usually not a controllable factor, if the goal is to reduce sublimation time it is apparent the focus must be on achieving an evenly thin layer.

3.2 THE SOLUBILITY OF CDD

Application thickness is a factor that conservators can directly control. By evenly applying a thinner film of CDD, the sublimation time can be reduced. When applying CDD in the melted state, the material cools so quickly that achieving a thin, even layer is very difficult. Generally when applying a facing with conventional adhesives, the adhesive is brushed through the facing material in long, even strokes. This is impossible when working with melted CDD because it cools and hardens too quickly to be effectively brush applied. Applying CDD from a solution like a varnish is an appealing notion to effectively control film thickness. A number of different solvents including mineral spirits, petroleum ethers, dichloromethane, and toluene have been noted as good solvents for CDD (Brückle *et al*. 1999; Muros and Hirx 2004). However, a more thorough study of CDD's solubility was undertaken.

Solubility is a kinetic process and is dependent on several factors. Time, temperature, and concentration are individual factors that control the process and also relate to each other. For example, at a higher temperature the time to dissolution is decreased. Another variable is individual solvents, where the concentration of saturation differs. Stein *et al*. (2000) reported "a saturated solution in Shellsol OMS was found to be approximately 80% wt/wt, while a saturated solution in xylenes was approximately 120% wt/wt and in hexanes was 140% wt/wt". These findings are mostly consistent with what the author observed with the exception that aromatics were found to dissolve approximately the same weight as aliphatic solvents. An experiment was undertaken to more clearly define the solubility of CDD.

To observe solubility, three grams of solid CDD as received from the supplier were placed in flint glass containers at room temperature (approximately 20^{o}C) and ten milliliters of solvent was added and allowed to sit with occasional stirring. Fifty different solvents were tested in this manner. Solvents in which the CDD completely dissolved within 12 hours were considered good solvents, while those in which at least dissolved the majority of CDD in 48 hours were considered borderline solvents, and those solvents that had little effect at this concentration were considered non-solvents. The results of the tests are plotted on a Teas chart in Fig.3.2. The solubility of a substance can be graphically represented on a Teas chart, which is a triangulated plot graph based on the three attractive forces: dispersion forces, polarity, and hydrogen bonding.

Being a waxy hydrocarbon it is highly soluble in

	Solvent	fd	fp	fh
1	Hexane	100	0	0
2	Heptane	100	0	0
3	OMS	98	1	1
4	ShelSol 340HT	96	2	2
5	Mineral Spirits	90	4	6
6	Petroleum Benzine	94	3	3
7	Cyclohexane	94	2	4
8	Turpentine	77	18	5
9	Benzene	78	8	14
10	Toluene	80	7	13
11	Xylene	83	5	12
12	Dichloromethane	59	21	20
13	Chloroform	67	12	21
14	Carbon tetrachloride	85	2	13
15	1,2-Dichloroethane	67	19	14
16	Trichloroethylene	68	12	20
17	Tetrachloroethylene	67	23	10
18	Tetrahydrofuran (THF)	55	19	26
19	1,4-Dioxane	67	7	26
20	2-Ethoxyethanol	42	20	38
21	2-Methoxyethanol	39	22	39
22	2-Butoxyethanol	46	18	36
23	1-Methoxy-2-propanol (methyl proxitol)	47	19	34
24	2-Ethoxyethyl acetate	51	15	34
25	Methyl acetate	45	36	19
26	Ethyl acetate	51	18	31
27	i-Propyl acetate	54	16	30
28	n-Butyl acetate	60	13	27
29	i-Amyl acetate	60	12	28
30	Propylene carbonate	48	38	14
31	Acetone	47	32	21
32	Methyl ethyl ketone (MEK)	53	30	17
33	Methyl isobutyl ketone (MIK)	58	22	20
34	Ethylene glycol	30	18	52
35	Propylene glycol	34	16	50
36	Methanol	30	22	48
37	Ethanol	36	18	46
38	i-Propanol	38	17	45
39	n-Butanol	43	15	42
40	Diacetone alcohol	45	24	31
41	Nitromethane	40	47	13
42	Acetonitrile	39	45	16
43	N-Methyl-2-pyrrolidone	48	32	20
44	N,N-Dimethylformamide (DMF)	41	32	27
45	Pyridine	56	26	18
46	Carbon disulfide	88	8	4
47	Dimethyl sulfoxide (DMSO)	41	36	23
48	Ethanolamine	32	29	39
49	Triethanolamine (TEA)	27	36	37
50	Water	18	28	54

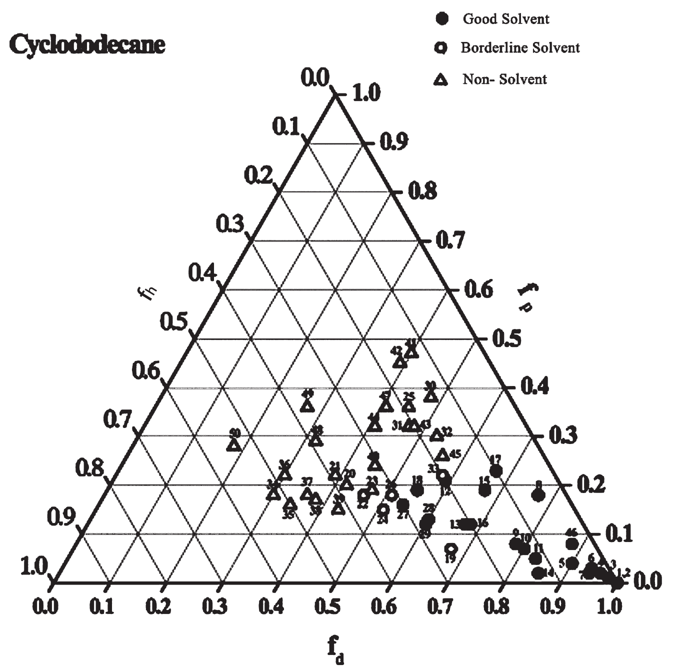


Fig.3.2 The solubility of CDD

aromatic and low polar solvents. What is more surprising is the wide range of solubility, which extends into acetate solvents. It should be noted that some solvents were faster than others, for example, mineral spirits was quite slow to dissolve the CDD, while n-butyl acetate was quick. It is tempting toConclude that CDD might be easily manipulated with solvent due to its wide solubility range; however, there are other factors at work.

3.3 PROBLEMS WITH SOLVENT-BORNE APPLICATIONS

Though CDD has a large solubility range, little use from solvent-borne applications has been reported. However, the difference in the crystal formation of CDD has been reported (Brückle *et al*. 1999; Stein *et al*. 2000; Muros and Hirx 2004). CDD, like other waxes, forms crystals from an amorphous mixture and the crystalline arrangement affects the physical properties of the wax (Horie 1987). Applying melted CDD results in small dense crystals, which make the film quite tough. Long needle–like crystals form when slower evaporating solvents are used and small powdery crystals form when a fast evaporating solvent is used. This is illustrated in Fig. 3.3a. Only the small dense crystals from melted applications seem to have the adhesive and film-forming properties that make CDD useful in conservation treatments. Solvent-borne films even when thoroughly dry wipe off the substrate easily. Melted films applied to glass slides adhere strongly to the substrate, while dried solvent-borne applications adhere poorly and have a powdery consistency. The facing tissue applied to a glass substrate in Fig.3.3 illustrates this problem. Fig.

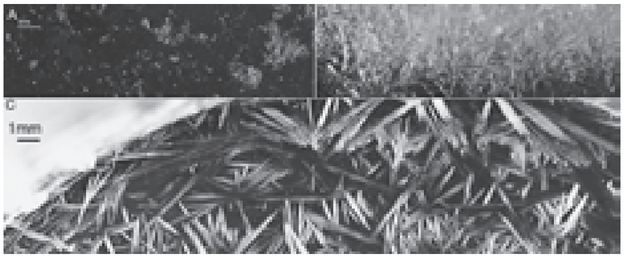


Fig.3.3a Crystal formation of cyclododecane in different solvents

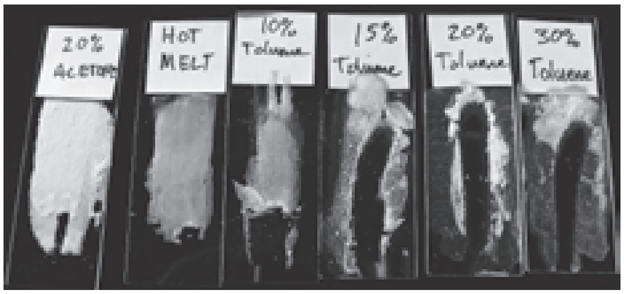


Fig.3.3b Scratch test with adding solvents to melted CDD

3.3b also shows that melted applications are difficult to remove when scratching with a fingernail, yet adding even as little as 15% solvent to molten CDD adhesion is lost. The reason for such a dramatic difference in adhesion is not fully understood, but has a definite correlation with crystal formation. This may also be because as the solvent evaporates, the CDD migrates with it and is deposited on the surface (Smith 2007). In some solvents, concentration at the point of saturation is too low to deliver enough CDD solids. As mentioned previously, different solvents reach saturation at different concentrations with the highest concentration at around 140% wt/wt in aliphatic hydrocarbons. With higher molecular weight polymers this concentration would not be possible or would result in a solution too viscous to be usable, yet at 140% wt/wt concentration with CDD the solution's viscosity is changed little from the pure solvent's viscosity. Even in this concentration, the solution cannot be used as an adhesive or consolidant. It is also obvious that used in this way CDD could not act as a barrier because the film is too porous. This porosity may account for its loss of adhesion. Another problem may be that the concentration of solvent-borne applications cannot deliver enough CDD through the facing material. However, from practical application it seems that solvent-borne applications are not a feasible technique. The solutions and solvents tested were not exhaustive, but were certainly extensive and from these results the possibilities of solvent-borne applications having relevance in any kind of conservation treatment are doubtful.

4.1 ADDING SOLVENTS TO MELTED CYCLODODECANE

SOLVENT	BP in ^{o}C
Xylenes	140
Hexane	69
Acetone	56
Toluene	111
Shellsol OMS	175

Mixing a solvent into hot CDD is a technique practiced, but not reported in the literature. This is perhaps because of the obvious fire hazard involved with adding flammable and volatile solvents to a hot liquid. While this can be done safely, it should be noted that even the most cautious conservators have reported incidents of the mixture catching fire, so it is crucial to take all appropriate fire hazard prevention practices. (NOTE: If solution catches fire, often the best procedure is to remove container from heat using **FIREPROOF** utensils and replace the lid.) This technique retains some of the film forming/adhesive properties of the melted application, while increasing working time and decreasing viscosity thus allowing the conservator to apply a thinner, more even coating. However, adding as little as 15% (solvent volume to CDD weight) can greatly affect its adhesion properties, as noted previously (refer to the 'scratch test' in Fig.3.3b to illustrate this). The concentration allowed before film-forming properties are gone varies with the solvent, from 10% to 25%. Some common solvents and their boiling points are listed and it reasonable to assume that a solvent with a boiling point over CDD's melting point at 60-61^{o}C would make a poor choice when combining solvents with melted CDD. Non-solvents tend to be a poor choice as well. When adding alcohols, the melted CDD cools and hardens on contact, eventually melting again after the alcohol evaporates. Though acetone is a non-solvent and has a boiling point lower than CDD's melting point, it appears to be miscible with melted CDD without boiling off. Tests also showed acetone affected film formation. It hardens with an increased opacity due to what appears to be many tiny air inclusions within the film, which is comparably tough and shown in Fig.3.3b. It remains to be seen whether there is actual solvency occurring because of the increased temperature or if this is a physical interaction caused by the solvent being lost through heating. Though not tested, perhaps the addition of other non-solvents is possible and could be useful. The addition of a solvent with an affinity for water may be useful when facing waterlogged artifacts that would probably repel CDD, which is hydrophobic. While it is difficult to recommend adding volatile, flammable solvents to melted CDD because of the potential danger, it may have some useful applications and allow for a smoother more even application.

4.2 IMPREGNATED FACING TISSUE

Another option, which shows promise, is impregnating a facing tissue with CDD before application and reactivating it using heat. This application technique allows for a uniformly thin application and eliminates the brush as the carrier for the CDD. The technique allows for a strong facing with minimal applied pressure. For the facing of delicate objects, such as burned skeletal material, this seems to be a feasible method of delivery. Much of the methods delivery, however, depends on the type of facing tissue used. Various types of facing tissue were tested including: several types of Japanese tissue paper, several types of spun polyester cloth, a cotton "trace" cloth, cotton "cheesecloth", loose woven cotton gauze, polyester/cotton blend gauze, and toilet paper. The toilet paper and the loose woven cotton gauze were the most successful due the extremely loose drape of the fabric. The other kinds of fabric felt like wax paper when impregnated with CDD and did not conform to the surface of the object being faced. Another factor in the success of this technique is how the heat required for reactivation is delivered. Several heating devices were used including a heat gun, variable speed hair dryer, a small benzo-torch, and a radiant heat tool with a small resistance element. The hair dryer and the torch were difficult to Control, while the heat gun worked reasonably well. However, the radiant heat tool was by far the most effective.

There are a few practical considerations when attempting this technique. The first step is to prepare the facing tissue by dipping it into melted CDD and allowing it to harden. A good approach for this procedure is to use a Petri dish with a diameter at least as wide as the tissue to melt the CDD, then holding the tissue length at either end so the bottom dip of the tissue is submerged and slowly pass the rest of the tissue through the CDD in one fluid motion. Working in one direction and in a small area, gently heat the prepared impregnated tissue until it goes clear (Note: the CDD can easily be driven off in this step by over-heating the tissue), and then gently set the melted area down with a brush. This technique is a very sensitive facing system and could be safely applied to very fragile artifacts.

Alternatively, another option for application is to dip the tissue in molten CDD and immediately lay it across the artifact, so the CDD hardens while in contact with the artifact. While this method seems to produce greater adhesion, due to the fast hand movements required it is riskier to the object and would require some practice before being useful.

Impregnated tissue can be made up beforehand, then wrapped in foil and bagged so that a constant supply is at hand. Results show that these facing tissue release in 2-5 days in a typical lab environment (approximately 20^{o}C with moderate airflow), depending on the number of layers. If immediate release is required a hair dryer can be used, provided the artifact can withstand the increased air current and heat.

5. CONCLUSION

CDD is a useful material, but before using it in a treatment conservators should be well aware of its working properties and practical limitations. Under certain circumstances, it can save the conservator much effort and reduce potential stress to the object. However, heavy or uncontrolled application can pose an undesired obstacle in the artifact's treatment. Since sublimation of CDD takes place on the surface of a film, an increase in surface area or film thickness leads to an increase in sublimation time. Manipulation of heat, airflow, and film thickness can be used to increase or decrease the sublimation time of a CDD layer. A thin, even film thickness of CDD that sublimes within several days is optimal for facing of archaeological objects during block lifting procedures. This can be achieved by impregnating facing tissue with CDD before use and reactivating the impregnated facing tissue with heat. The nature of the facing tissue used has a strong influence on the success of this procedure. Adding solvents to molten CDD improves its working properties, but cannot be recommended due to the inherent danger in this procedure. Though CDD has a wide solubility range, solvent-borne solutions do not have the same adhesive properties are not useful in conservation treatments.

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