AN INVESTIGATION OF CYCLODODECANE FOR MOLDING FOSSIL SPECIMENS

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What we thought we were doing.

Cyclododecane (a cyclic alkane hydrocarbon $C_{12}H_{24}$) is a wax-like substance that passes from a solid state directly to a vapor at room temperature, disappearing and leaving virtually no trace. The ability of this material to sublimate has led to its increasing use in the field of art and artifact conservation to temporarily stabilize objects for treatment and transport. This poster will provide a basic introduction to cyclododecane including its physical properties, health and safety issues and a synopsis of its use on museum artifacts.

Cyclododecane's properties also suggest great potential for use on vertebrate fossils as an alternative material for temporary backings and fills, consolidation and mold making. Conservators and Fossil Preparators together focused on the potential uses of cyclododecane for molding as a:

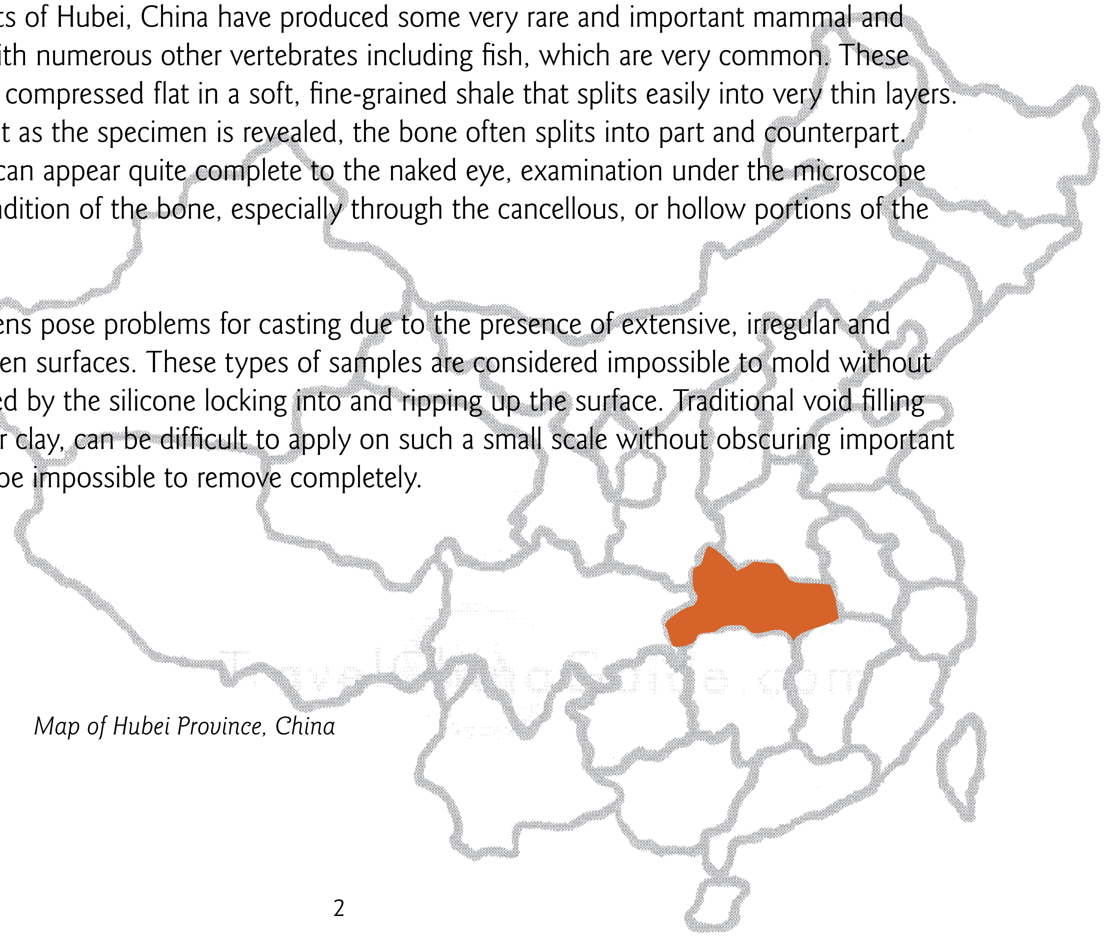
1. Thin barrier coating that functions as a separating layer
2. Fill to prevent silicone penetration into deep voids
3. Temporary consolidant for porous matrix
4. Dam to contain the silicone or isolate areas for spot molding

The rate of sublimation for samples mixed with several different bulking agents as well as in solvent was also investigated.

What was our problem?

The early Eocene deposits of Hubei, China have produced some very rare and important mammal and bird specimens, along with numerous other vertebrates including fish, which are very common. These specimens are preserved compressed flat in a soft, fine-grained shale that splits easily into very thin layers. This allows quarrying but as the specimen is revealed, the bone often splits into part and counterpart. While these specimens can appear quite complete to the naked eye, examination under the microscope reveals the shattered condition of the bone, especially through the cancellous, or hollow portions of the skeleton.

The Hubei fossil specimens pose problems for casting due to the presence of extensive, irregular and minute voids in the broken surfaces. These types of samples are considered impossible to mold without irreparable damage caused by the silicone locking into and ripping up the surface. Traditional void filling materials, such as wax or clay, can be difficult to apply on such a small scale without obscuring important morphology and would be impossible to remove completely.



Map of Hubei Province, China

What was our goal?

Our goal was to determine if cyclododecane (CDD) could be used to prepare delicate fossil specimens for molding and casting. We also hoped to gain experience in handling the material and knowledge about how it behaved in a fossil preparation context.

What did we use for testing?

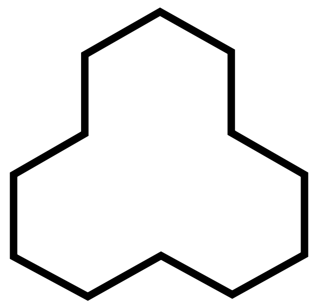
A common fish fossil from Hubei was selected for testing. This sample exhibited similar surface problems as other, rarer vertebrate specimens, which we have been unable to cast. Following standard preparation procedures, the fish specimen was coated lightly with Butvar B76 (poly-vinyl butyral) in ethanol and allowed to dry overnight. The melting point of CDD is below the glass transition temperature of Butvar making their use together compatible.

Fish specimen from Hubei, China

What is special about cyclododecane?

Cyclododecane is a cyclic hydrocarbon ($C_{12}H_{24}$), one of a class of volatile waxy solids. The extremely high vapor pressure of CDD leads it to sublime at room temperature, passing directly from a solid to vapor. The ability of CDD to reverse itself, leaving virtually no trace on the substrate, has made it increasingly popular with the art conservation community for use as a temporary consolidant, sealant, water repellant barrier, coating and separation layer. Increasing research on its potential uses been published since its introduction into the field in the late 1990's (Hangleiter).

Melting Point 58-61 C
Boiling Point 243 C
Flash Point 98 C
Fire Point 265 C
Vapor Pressure (20 C) 0.1 hPa



Structure of Cyclododecane

How do I apply cyclododecane?

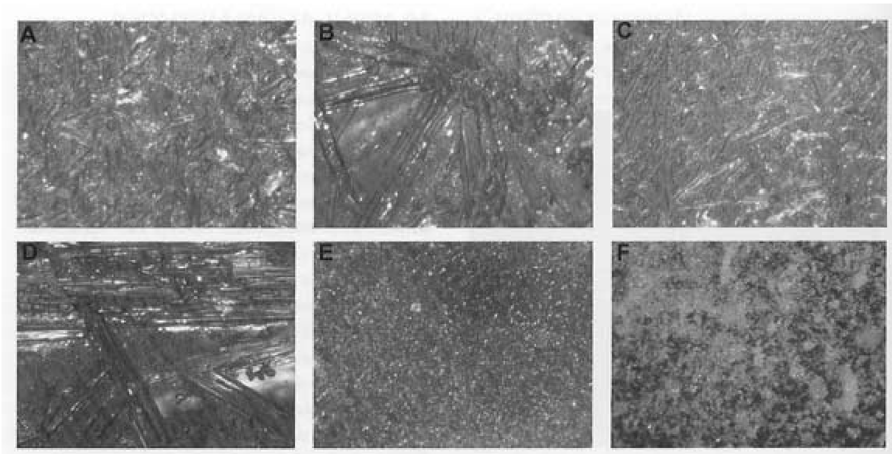
Cyclododecane can be applied in three forms: as a melted solid, in solution or as an aerosol spray. Each application technique results in a coating with different film characteristics, which have been described in several publications (Bruckle et al.; Hangleiter; Maish et al.; Muros et al.; Stein et al.). None of the films are homogeneous - rather they are described by the length and density of their network of crystals.

• Melted CDD results in a generally thick coating with a dense, tight network of small crystals. The faster the melt cools, the denser the network.

• CDD is soluble in non-polar solvents (i.e. aliphatic or aromatic hydrocarbons such as hexane, heptane, toluene, xylene, dichloromethane, mineral spirits, naptha, petroleum benzine, petroleum ether). CDD is not soluble in polar solvents (i.e. acetone and alcohols), nor is it miscible with water. Application in solution will result in a thinner film than a melt but one with a porous structure due to the formation of long, needle-like crystals. The speed of solvent evaporation determines the shape of the crystal formation. More volatile solvents form somewhat shorter crystals and denser networks than when slower evaporating solvents are used.

• The aerosol spray supposedly results in a more even and thin film made of globular crystals, with a density between that of melt or solution applications (Muros et al.).

How do I apply cyclododecane? (cont.)



Crystalline structure of cyclododecane applied in various forms:
(A) in petroleum ether
(B) in mineral sprits
(C) in dichloromethane
(D) in toluene
(E) as a melt
(F) as a spray (magnification 40x)

"Note how the slower-evaporating solvents, such as mineral spirits and toluene, formed long needlelike crystals with spaces in between each of them. Faster-evaporating solvents formed smaller crystals, in a tighter, less porous crystal network. The melt appears to be made up of small crystals formed closely together creating a compact, dense film. The spray film seems to be comprised of small, globular crystals rather than acicular [needle shaped] ones like the other films."(Muros et al. 2004, p.78)

These observations were confirmed in working trials at AMNH. The melt application appeared to the naked eye as a fairly solid coating but tests with CDD applied in solutions of either mineral spirits (less volatile) or naptha (more volatile) formed long crystals in open lattices. This was deemed inappropriate for a use as a barrier layer, as the morphology of the crystals would obscure the microscopic morphological detail of the fossil.

Image of CDD in solvent showing the development of long needle-like crystals that climbed up the side of the sample tray during solvent sublimation.

HELPFUL TIP

Molten cyclododecane has an extremely short working time. Electric heat tools may be useful for delivering CDD onto a sample. Gentle heating of the specimen greatly improves flow. When possible, use disposable tools for easy cleanup. Non-disposable tools can be placed under a heat lamp to speed sublimation.

How fast does cyclododecane sublimate?

Cyclododecane's ability to sublimate makes it useful as a temporary barrier, sealant or consolidant, requiring that the user simply waits for it to disappear. Research by Hangleiter; Hiby; Muros et al.; and Stein et al. documents that the rate of sublimation will depend on several factors:

• The thickness and density of the CDD application - the thicker and denser the film, the slower the rate of sublimation.

• The porosity of the substrate - the greater the porosity of the substrate the slower the rate of sublimation.

• Temperature - an increase in heat will quickly speed sublimation with the most substantial increases seen with applied heat greater than 30 C (86 F)

• Ventilation - sublimation rates can be sped by an increase in airflow over the sample. Covering the sample with foil or polyethylene plastic will retard sublimation.

• Humidity - can potentially affect the speed of sublimation.

General guidelines indicate that on a non-porous surface a coating of melted CDD 1.0 mm thick will disappear in approximately 30 days. A 1.0 mm aerosol spray film or CDD in solvent solution might sublimate in a matter of three to eight days (Hangleiter, Muros et al.).

How fast does cyclododecane sublimate? (cont.)

Our previous experience using CDD in conjunction with a porous bulking agent seemed to confirm research that sublimation from a porous surface was slower than the guidelines above. A simple experiment was attempted to determine sublimation rates for CDD when soaked into several different bulking materials of potential use in fossil prep applications for reversible fills or temporary supports. The goals were to observe gross behavior and to gain an understanding of whether the addition of bulking materials to CDD affected the sublimation rate.

Bulking materials were chosen that could be easily available to a fossil preparator including:

- Cotton Wool (CW)
- Kimwipe Tissue (KW)
- Japanese Tissue (JT)
- Loose sandy matrix (Ours was from Ukhaa Tolgod, Mongolia) (GM)
- Cellulose Powder (CP)
- Cellulose Pulp (P)

Each bulking material was impregnated with a melt in both 5.0 and 10.0g amounts and in saturated solutions of mineral spirits (less volatile) and naptha (more volatile). The choice of solvent was based on volatility, toxicity and what might be most commonly found in a standard lab. The tests were conducted during the summer at room temperature, in ambient relative humidity and normal ventilation. The rate of sublimation was determined by weight loss of the samples using a digital balance sensitive to 0.01 of a gram. Sublimation was considered complete when the weight of the sample equaled its weight before CDD application. Dust settling on the samples was not considered an issue due to the relative insensitivity of the analytical balance.

Sample dishes with cyclododecane and bulking materials
1) Sandy matrix
2) Cotton Wool
3) Cellulose Pulp
4) Cellulose Powder
5) Kimwipe Tissue
6) Japanese Tissue

How fast does cyclododecane sublimate? (cont.)

The accompanying graph plotting weight loss over time shows the following:

- An initial rapid decline in mass of the solution samples, due to solvent evaporation. This was also noted by others (Maish et al.; Muros et al.; Stein et al.).

- After solvent evaporation those samples showed a regular, slow rate of mass loss due to CDD sublimation

- The sublimation rate of the solvent samples matched that of the melt samples.

- All samples showed a loss of around 0.2 grams per week irrespective of the bulking material.

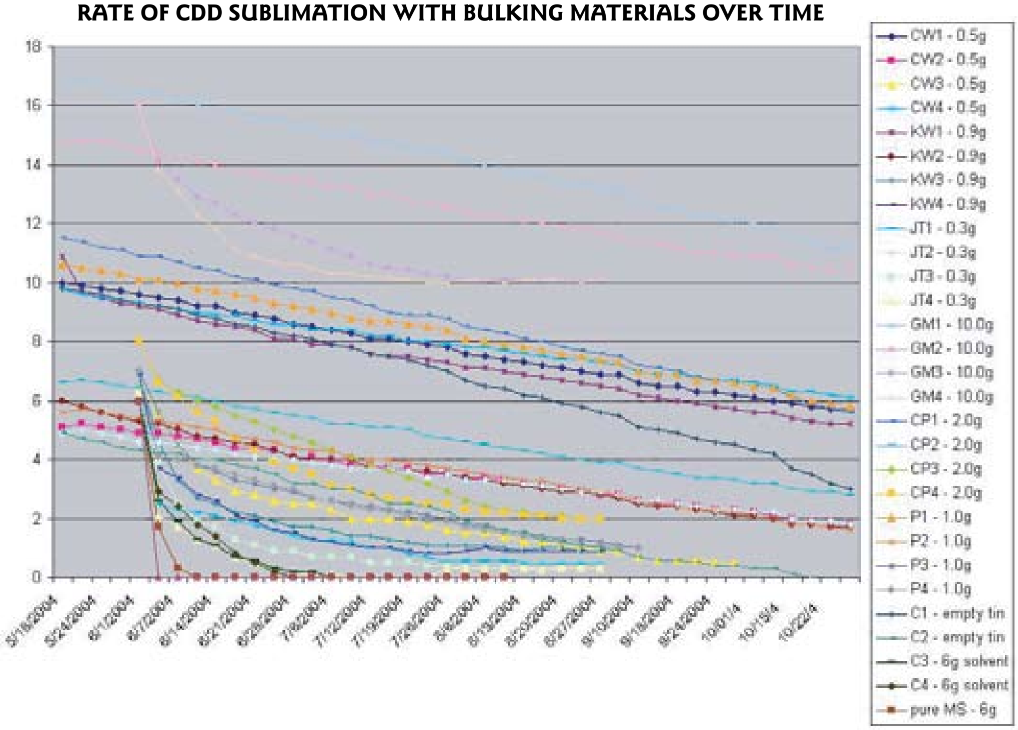
- The bulked melt samples did take longer to sublimate than the pure CDD control samples. But at this level of resolution it does not appear that there is any difference between the different bulking agents. However this experiment is still in progress.

- The bulked samples with CDD in solution contained less CDD and therefore completed sublimation the quickest - taking an average of 91 days to fully sublimate. The CDD in bulked melt samples had not fully sublimated after 170 days.

- The samples with 5.0 grams of CDD finished sublimating sooner than the samples with 10.0 grams of CDD.

- What is not apparent in this graph is the large loss of cyclododecane while heating to its melting point. In almost all cases, cyclododecane sublimated during melting before mixing with the bulking material.

RATE OF CDD SUBLIMATION WITH BULKING MATERIALS OVER TIME

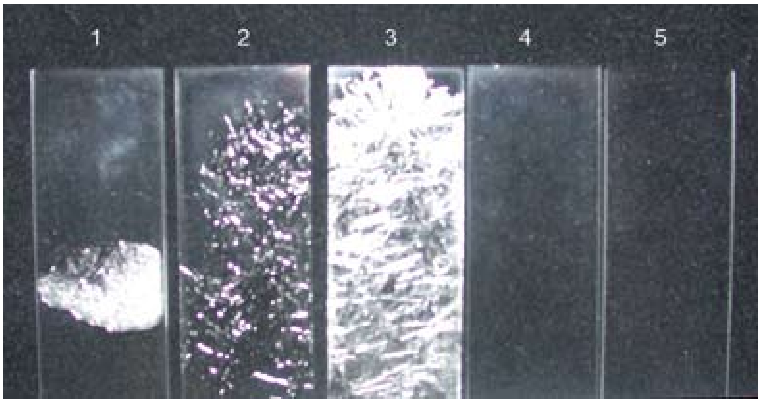


HELPFUL TIP

Sublimation speeds can be easily manipulated. We found that while melting CDD to apply to the bulking samples we lost a measurable amount (0.1 - 3.0g). Similarly, increasing the temperature using either a lamp or hot air blower can substantially speed deliberate sublimation on a specimen. Bagging a sample treated with CDD and reducing airflow can slow sublimation by a matter of days or weeks to allow for increased working time. We recommend testing cyclododecane on your particular substrate using various delivery methods to get the most accurate determination of sublimation rates.

Does it really disappear?

Recent research has examined whether CDD actually sublimates completely. Minute quantities of cyclododecane residues have been noted on treated ceramics and glass slides (Caspi, et al.). Analysis indicates these are remnant components from the CDD manufacturing process and that there is a variation in purity based on the manufacturer and batch. When found, these residues can be removed either mechanically or using solvents. We tested for residues by applying films of pure CDD melt, solutions in naptha and mineral spirits and also pure solvent to glass slides. Our visual examination of test slides after sublimation showed a slight residual smudge on the glass slide with the CDD melt.



Testing for residues by application to glass slides
1) Molten CDD
2) CDD In mineral spirits
3) CDD in naptha
4) Mineral spirits - control
5) Naptha - control

HELPFUL TIP

Each new batch of cyclododecane should be tested before use by applying a small amount to a glass slide. After complete sublimation, visual examination of the slide should be sufficient to determine whether impurities are present.

IS IT SAFE?

CDD contains no components considered to be health hazards by the Occupational Safety and Health Administration (OSHA) and there are no current recommended Permissible Exposure Limits (PELs). A review of several Material Safety Data Sheets (MSDS) for cyclododecane finds that there is no evidence that it is toxic. However, there has been little testing on which to base the claim that it is non-toxic (Goldberg, personal communication).

The Hazardous Materials Identification System (HMIS) 0 - 4 scale (with 0 being the lowest hazard) categorizes CDD as a 1, meaning slight hazard. In contrast, ethanol, commonly used in fossil preparation, is listed as a 1 or 2 (depending on the manufacturer) - 2 is a moderate hazard. CDD is considered a minor skin irritant and various MSDS's differ as to whether it is considered irritating to eyes and respiratory systems. This may be, in part, a function of the lack of regulation in drawing up material safety data sheets. When heated CDD emits a characteristic musty wax odor.

Although CDD is found in minute quantities in a myriad of everyday products from creams to printing applications, its uses in the field of conservation or fossil preparation involve substantially larger quantities (Goldberg, personal communication). As a result, whether used as a melt or in solution, good basic health and safety procedures should be followed when using CDD. Personal protective equipment should include gloves and safety glasses with side shields, especially if wearing contact lenses. As CDD sublimates it may no longer be visible but its molecules are dispersed into the atmosphere. As a result, heating and use of CDD should, whenever possible, occur in a fume hood or well-ventilated room. This is particularly true if using CDD in solution where the toxicity of the solvent must also be considered.

HELPFUL TIP

Heat larger amounts of cyclododecane in the fume hood but decant a small amount for immediate use.

How can I use cyclododecane to prepare a specimen for molding?

The possibility of using CDD in solution was discarded early on due to our observation of the formation of large, spiky, freestanding crystals. Time did not allow for testing of the aerosol spray at this date. Future work will examine the appropriateness of the spray.

Our current investigations concentrated on the manipulation of molten CDD while experimenting with different tools and application techniques.

1. Can I use it as a separating layer?

Attempts to apply CDD as a thin, molten coating revealed the difficulty of obtaining an even layer. Molten CDD was applied with a brush. The liquid CDD chilled and solidified very rapidly and, although it was discovered that gentle warming of the specimen (on a hot plate or under a heat lamp) promoted much better flow, it was still impossible to avoid over-coating some areas, resulting in obscured morphological detail. These overly thick areas also formed large crystals on the surface that would show up in a mold. At the same time, other areas of the coating became too thin or disappeared entirely with the CDD sublimating too quickly under the applied heat. Trying to control the use of heat to promote better flow over the sample proved impossible, resulting in uneven sublimation leaving some areas uncoated.

Images of the untreated sample on the left and the sample coated with an uneven layer of molten CDD on the right seen as the darker surface areas. Too much CDD was delivered to the bottom of the sample while the top was not fully coated. The detail shows areas of crystal formation making it unsuitable for molding.

2. Can I use it to fill and seal voids in a specimen?

We had mixed results with CDD as a fill on the fish. We achieved the best results in applying minute amounts of CDD using a Kistka, an inexpensive electric stylus (commonly used for wax egg decorating) fitted with an extra-fine tip. We also found it best to keep the specimen slightly warm under the microscope. This could be accomplished with lights or with a hot plate (on a very low setting). This gentle warming prevented the CDD from chilling into convex drops and allowed it to flow into a concave meniscus shape, which is more desirable for a tiny fill. A battery-operated wax pen (Super Max Wax Pen) was also useful for re-melting and manipulating CDD after initial application. The radiant heat from this tool can even be used to sublimate or "erase" tiny areas of CDD. In this manner we could fill many of the multitude of small voids in about one hour. This may have been adequate to seal a less problematic surface, but the Hubei specimens have a remarkably complex configuration of tiny voids within the broken bone. Despite extensive filling and sealing of the voids with CDD, the specimen was still deemed too porous to safely mold. This technique might well be useful for less complex specimens.

Kistka electric stylus (left) was found to be useful for delivery of minute amounts of CDD. The Super Max Wax Pen (right) was good for re-heating areas coated with CDD to improve flow.

3. Can I use it to consolidate porous matrix?

MAYBE. An untreated sample of extremely porous, loosely cemented sandstone from Ukhaa Tolgod, Mongolia was used to attempt consolidation for molding with CDD. Results were very similar to the coating experiment above, which was not encouraging. The sample was warmed and molten CDD was applied by brush. The CDD soaked into the sample readily, but very unevenly, and attempts to apply more in the underconsolidated area only resulted in an excessive build-up in others. The consolidation was deemed unsuccessful, and the sample was not molded. Future attempts will investigate alternative tools for this purpose. We have high hopes for the Batik Ball used in conjunction with the Englebrecht WzII Control Unit and Heat Spatula Arm.

We were unable to successfully consolidate porous sandstone matrix using CDD. The uncoated sample is seen on the left and the partially coated sample on the right.

The Batik Ball arm used in conjunction with a heat spatula arm and thermostat control unit may be helpful for future applications.

4. Can I use it to isolate areas for spot molding?

YES! CDD was found to be ideal for forming a temporary dam to contain silicone to isolate an area for spot molding. Rather than try to build up walls with CDD, we made walls out of paper soaked in cyanoacrylate, to impart the desired stiffness to the paper. This was formed into a ring or collar, which was then adhered using CDD onto the skull end of the specimen. This allowed for spot molding this particular area, isolating the rest of the specimen from the silicone. The CDD was applied using the kistka outfitted with the largest tip. Beads of solid CDD were fed into the kistka receptacle and the collar was quickly and easily adhered over the fragile bone. Larger treatments would be easier to accomplish with the batik ball, but this is a more expensive tool. Sublimation was delayed for three days over the weekend by double bagging at normal room temperature and there was no need to reapply CDD after this period of time. To remove the collar after molding, the CDD could be allowed to sublimate normally, or speeded by warming under a heat lamp and then the collar can be safely removed. No adverse effects to the specimen were observed when this was tried.

A paper collar stiffened with cyanoacrylate was adhered to the surface of the sample using a Kistka to melt the CDD.

The fish sample on the left has been placed under a heat lamp to speed CDD sublimation. On the right the sample has been placed in a polyethylene bag to retard sublimation and extend working time.

IN CONCLUSION: Is cyclododecane the magic solution for molding problem specimens?

The short answer is yes, and no. The grosser applications worked well. The more controlled delivery techniques we tried were generally not successful for the small scale of the morphological detail and voids on the Hubei fish sample. However, some of these techniques would probably be more successful on larger or less problematic specimens.

· CDD, applied in solutions using either mineral spirits or naptha, forms a thin, even film but with undesirable crystals, making it inappropriate for use on specimens.

· The cyclododecane spray is worthy of future investigation.

· We were not able to use CDD successfully as a melt to produce an even coating that would function well as a separating layer for this fish sample with microscopic morphological detail. Using a heat lamp or heat tools only sped the sublimation in thin areas exacerbating the uneven application. For creating a thin separating layer over bone morphology, CDD did not perform as well as a traditional adhesive coating, such as Butvar B76.

· We found that it is possible to achieve tiny concave fills using the kistka with an extra-fine tip on a warm specimen. It is also possible to manipulate small areas with the wax pen. Grosser applications of CDD in the form of blobs or pools would be very useful as a temporary fill for larger voids or to protect areas that should not be molded.

· Applying molten CDD using a brush is not a particularly good method for delivering an even coating. We have high hopes that conducting further investigation on tools for consolidating the porous sandy matrix will be successful.

· The ability of CDD to sublimate makes it superior to traditional adhesives for isolating areas in preparation for molding. Using CDD to adhere a paper collar to our sample to create a temporary dam on top of fragile bone was successful and easily reversible.

WHERE CAN I ORDER THOSE TOOLS & MATERIALS?

Cyclododecane - Item # 87100 500g / $25.00 or 1 kg / $40.00 available at Kremer Pigmente, 228 Elizabeth Street, New York, NY 10012 USA Tel. 212.219-2394 or 1-800 995 5501, Fax. 212.219-2395, e-mail: kremerinc@aol.com,
http://www.kremer-pigmente.de/englisch/catalog.htm

Super Max Wax Pen - Item #WX520, $27.95 available at Jewelry Supply
Phone 916.780.9610, Fax, 916.780.9617, e-mail - info@jewelrysupply.com, http://www.jewelbay.com

Kistka Multi-Point Electric Stylus - $55 with three points available at SURMA The Ukranian Shop, 11 East 7th St. New York, NY 10003, Phone 212-477-0729, Fax 212-473-0439, http://www.surmastore.com/

Batik Ball For use with the Engelbrecht WZII Unit and heat spatula arm. $40 each from Kolner L.L.C., 23 Grant Avenue, New Providence, N.J. 07974; tel: 718-802-1659

Englebrecht WzII Control Unit And Heat Spatula Arm. $310 for unit, $44 for heat spatula arm. Available from Kolner L.L.C., 23 Grant Avenue, New Providence, N.J. 07974; tel: 718-802-1659

Cellulose SS122 Filter Pulp, 500cc for $10.23, Manufactured by Schleicher and
Schnell Microscience, Inc. Available from Fisher Scientific, Catalog no. 9940820
www.fishersci.com

Fibrous Cellulose Powder CF11, Catalog no. 05-713-004. 500g for $65.60. Manufactured by Whatman. Available from Fisher Scientific, tel: 1-800-766-7000, www.fishersci.com

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