THE STUDY OF CYCLODODECANE AS A TEMPORARY BARRIER IN THE MOLDING OF STONE SCULPTURES AND ORNAMENTS

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ABSTRACT: *This paper describes the study of the hydrocarbon cyclododecane (C12H24) and its suitability as a temporary barrier in the moulding of petrous pieces of different porosities. For this purpose, two types of petrous materials were chosen, Bateig biocalcarenite from Novelda (Alicante, Spain) and marble from Estremoz (Evora, Portugal). The moulding process of a work of art is a very complex task entailing an exhaustive study of the piece and the selection of the most ideal barrier to prevent the presence of residues and unwanted chromatic changes over time. Therefore, the intervention methodology was based on both the preparation of petrous specimens with different dilutions of CDD and subsequent testing samples to study their accelerated ageing by irradiation with ultraviolet light. The results were obtained with colorimetric measurements and Fourier transform infrared spectroscopy (FT-IR). When using CDD as a temporary barrier on different types of stone, we observed some very important properties in relation to other more generally used mould-release agents; its sublimation capacity prevents the presence of residues and chromatic changes, thus making it one of the most promising substances in the field of Conservation and Restoration of Cultural Property.*

KEYWORDS: cyclododecane, stone sculptures and ornaments, reproduction, bateig biocalcarenite, estremoz marble, barrier film

INTRODUCTION

Nowadays, moulding techniques are still used to solve conservation problems. There are cases in which a work of art is greatly deteriorated due to its exposure to the elements. Therefore, the creation of a replica is necessary in order to conserve the original under controlled conditions for its preservation, that is, a museum (García 1994; Roig 1995; Fernández 1999; Mas 2006).

The biggest problem in making a mould over an original piece is protection in terms of the moulding materials (Matteini et al. 1990; Mas 2006; Pereira and López 2008). The selection of the best mould-release agent will guarantee the reversibility and respect of the work of art.

For this purpose, a good release agent should prevent the material used in the moulding process, generally RTV (room temperature vulcanising) silicones, from adhering to the sculpture's surface. It should also offer the ability to form a fine film which conserves the impression, thus helping to release the sculpture from the mould and its subsequent removal. Another very significant characteristic that defines a good release agent is its ability to be removed without having to subject the work to chemical or physical-mechanical means.

In 1995, Hans Michael Hangleiter, Elisabeth Jägers, and Erhard Jägers proposed the use of a material with interesting characteristics for different conservation and restoration specialities. They searched for a suitable material that would guarantee a temporary consolidation in both the transfer and treatment phases. The ideal agent would have to have good filmogenic characteristics, a low melting point, and be water insoluble, soluble in organic solvents, with a slight level of toxicity and reversibility. Of the set of cyclical hydrocarbons studied, cyclododecane (CDD) offered the nearest desired characteristics ($C_{12}H_{24}$ (P.M. 58-61°C)) (Bruckle et al. 1999). CDD is able to sublimate (directly change from a solid state to a gas state), thus making it a substance with the ideal properties to act as a release agent and to eliminate the worst problems in the reproduction works of pieces, for example, the subsequent phase of barrier removal (Stein et al. 2000; Mas et al. 2006).

One of the inconveniences in relation to the application of CDD as a mould-release agent worth highlighting is its affinity with silicone materials. Since it is a non-polar substance, CDD is soluble in silicone oils (non-polar). The solution to this problem would be the application of a polar interface film (Bruckle et al. 1999; Maish and Risser 2002), which would act as an insulation among the two non-polar layers, the CDD film and silicone.

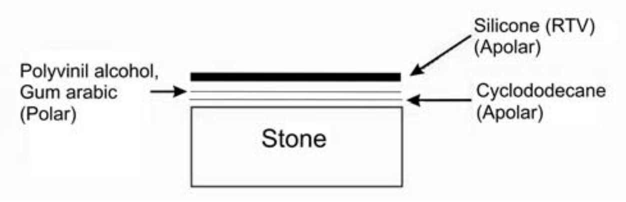

Figure 1. Diagram of the procedure for the application of the layers.

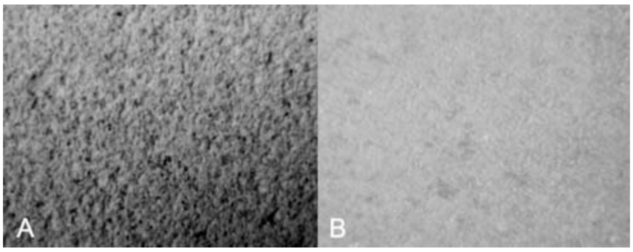

Figure 2. Types of petrous supports studied. A. Novelda Stone, Bateig Cream. B. Marble from Estremoz.

This paper presents the study conducted on the suitability of cyclical hydrocarbon CDD (non-polar) as a mould-release agent/barrier applied to the petrous biocalcarenite supports Bateig from Novelda (Alicante) and marble from Estremoz (Evora, Portugal). Likewise, the suitability of polyvinyl alcohol (PVOH) and gum arabic (G.A.) as polar separating substances (interface layers) between CDD and silicone was studied, as shown in Figure 1.

2. EXPERIMENTAL

2.1 Reagents

The following products were used in this project: CDD (non-polar barrier) is an unsaturated and chemically stable cyclical hydrocarbon ($C_{12}H_{24}$). It boasts the specific sublimation property at room temperature and is of low toxicity (Cagna and Riggiardi 2006). Its melting point is between 58° and 61°C which makes it a manageable substance. This product is supplied by CTS Spain. Isooctane is a branched-chain alkane (C_8H_{18}), octane isomer (2,2,4-trimethylpentane). In addition, it is colourless, water insoluble and has a density (20/4) of 0.690. This product is supplied by Panreac Química S.A. Xylene (non-polar solvents) is an aromatic hydrocarbon composed of a mixture of three isomers: para-xylene, meta-xylene and ortho-xylene (C_8H_{11}, dimethylbenzene). It is a colourless liquid with a density of 0.87 kg/l at 20°C. This product is supplied by CTS Spain. Polyvinyl alcohol is a resin obtained through the saponification of polyvinyl acetate. It is water-soluble, is soluble in ethanol and methanol up to certain limits, and is also insoluble in organic solvents. Films are stable and transparent. This product is supplied by CTS Spain as an ivory white powder with a pH of 5 at 4%. G.A. is a natural secretion of several acacia species, exuded into small pearls. In this study, G.A. from the Acacia senegal tree was used. It is water soluble, but insoluble in alcohol and in the majority of organic solvents. It is supplied by G.C.AGAR AGAR, SL Spain. Latex (polar barrier) is a natural rubber, a copolymer in aqueous dispersion. The film obtained is light, stable, transparent, flexible and elastic. It is presented as a milky white liquid <1% ammonia with a pH of 10.5 and a viscosity of 35 cps. This product is supplied by CTS Spain. Finally, RTV silicone is a SILASTIC 3483 RTV silicon elastomer manufactured by Dow Corning. It is a white silicone rubber with excellent mechanical properties, it is resistant and offers high fluidity. It is a bicomponent product composed of a fluid base and a hardener, SILASTIC 83 curing agent, which catalyses at room temperature, when mixed (22°-25°C), by a condensation reaction, thus obtaining optimal mechanical properties in relation to tearing and bending (shore hardness of 13 A, tensile strength of 3.5 MPa). This product is supplied by CTS Spain.

2.2 Stone materials

Bateig cream

Bateig cream is a carbonatic biocalcarenite rock extracted from the Vinalopó Medio area, Novelda (Alicante, Spain) (Roig 1995; Mas 2006). Shell remains and other benthic organisms can be observed in Bateig cream which present a structure that is reminiscent of heavily carbonate-cemented arsenic. This stone has a high porosity of 16% (Figure 2A).

Estremoz Marble

It is a metamorphic white rock with an average grain-size distribution which may include pink shades and grey veins. This marble is extracted from the district of Evora conselho (Council) of Estremoz, Borba and Vila Viçosa (Portugal) (Carvalho et al. 2000). This stone has a very low porosity of 2% (Figure 2B).

2.3 Instrumentation

The analytical techniques used in this study were *cryogenic scanning electron microscopy* (cryo-SEM) to observe the morphology of the crystalline structure of the CDD, *visible spectrophotometry* which measured the specimens' chromatic coordinates before, during and after the accelerated ageing tests, *optical microscopy* (OM) which enabled the observation of the specimens' surface and the evolution of the CDD sublimation, *Fourier transform infrared spectroscopy* (FTIR) which determined the existence of residues on the specimens after testing, the *thermohygrometric ageing chamber* which simulated climatic conditions and effects, as well as the u*ltraviolet (UV) irradiation ageing chamber* which determined the effect of ultraviolet radiation on the specimens throughout testing.

2.4. Preparation of test specimens and conservation materials.

The study was divided into two parts. The first studied the sublimation property of pure and diluted CDD at different concentrations, 40-60-80%, in two solvents: isooctane and xylene. The selection of these two solvents was based on the differences between their boiling points (isooctane 99°C and xylene 136-139°C). The second part centred on the study of the PVOH and G.A. interface layers at different concentrations (10-25-50-75%).

The experimental methodology in the first part of the study was carried out by taking chromatic measurements, images of the surface using optical microscopy, cryogenic scanning electron microscopy (cryo-SEM) and the FTIR spectroscopic analysis of the samples extracted from the surface of the specimens of petrous material before and after applying CDD. In this case, the sublimation times of CDD on the petrous support were observed in terms of weight loss, and with controlled temperature (22°C) and humidity (60%) parameters. The second part of the study consisted in studying the response of the polar layers (PVOH and G.A.) applied on CDD. These layers were applied 60 min after most of the solvent had evaporated. Finally, 2h after drying the polar layers, the silicone elastomer, with thixotropic properties, was applied. Then 24h later once the elastomer had already vulcanised, this was removed and the polar layers were eliminated by mechanical means and water. Directly afterwards, specimens were subjected to accelerated ageing tests using ultraviolet irradiation (48h at 45°C). The study was completed with colorimetric measurements and an FTIR spectroscopy analysis of the samples extracted from the surface of all the specimens. The total number of 208 (6x5x3cm) specimens was used in both parts of the study.

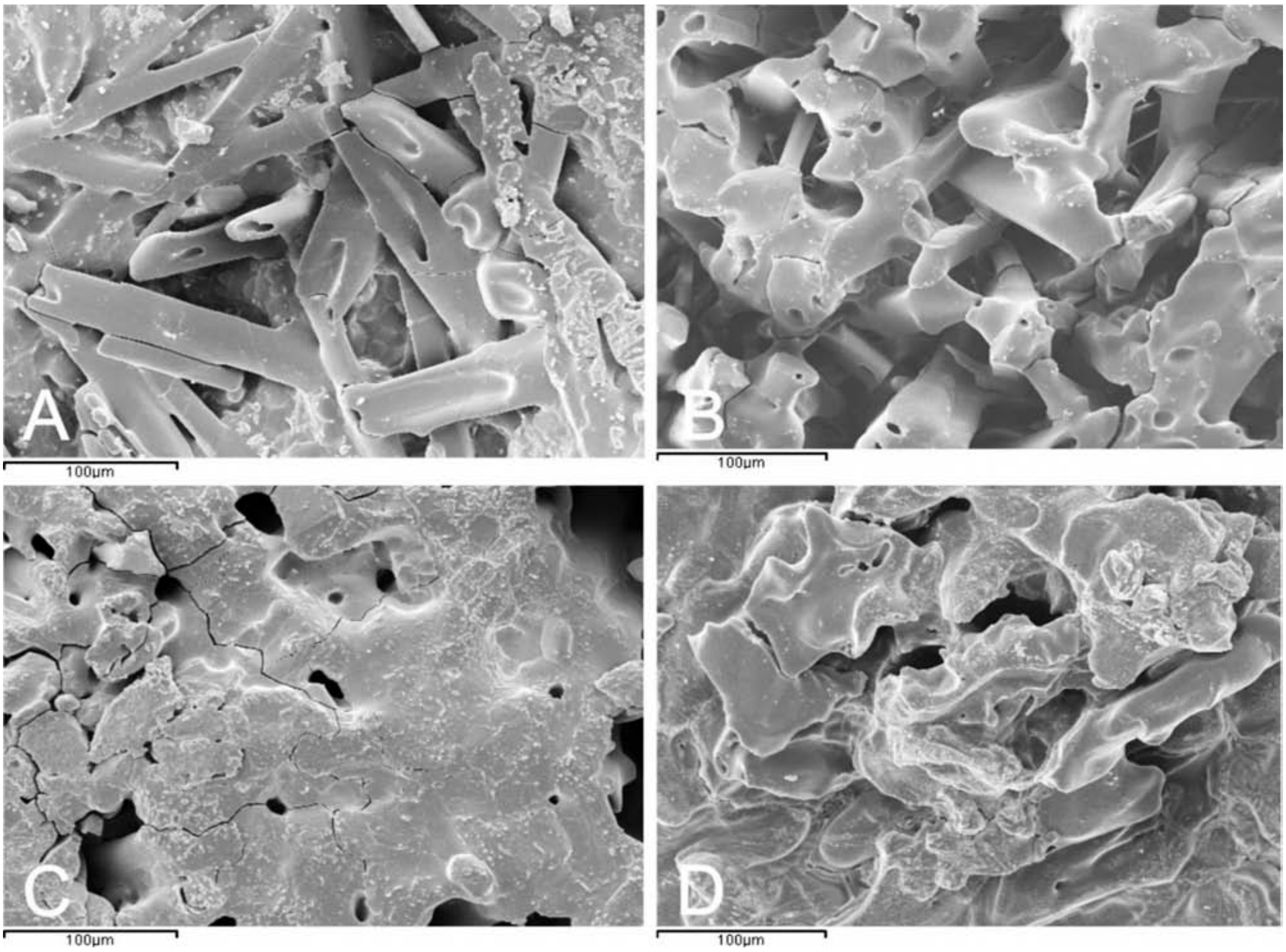

Figure 3. A. Structure of 40% CDD applied on Bateig cream. B. Structure of 60% CDD applied on Bateig cream. C. Structure of 80% CDD applied on Bateig cream. D. Structure of 60% CDD applied on Estremoz marble

3. RESULTS AND DISCUSSION

This section presents the results obtained throughout the experimental procedure. The chromatic changes observed in the specimens, the presence of visible residues seen with the naked eye, and the analyses via FTIR spectroscopy following the removal of the interface and elastomer layers, as well as the morphological changes to the CDD films using cryo-SEM, have been provided.

3.1. Pure CDD applied to the different petrous specimens

The application of pure melted CDD to the petrous specimens resulted in the loss of all the impression details due to fast solidification. This prevented us from obtaining an exact copy of the original, which is why we rejected the use of pure CDD.

3.2. CDD diluted to 40%, 60% and 80% in xylene and isooctane applied to the petrous materials.

Two solvents (isooctane and xylene) were chosen for the study (Stein et al 2000).

The results obtained in terms of the sublimation time of CDD showed that CDD in isooctane took longer to sublimate (irrespectively of concentration), and that diluting the CDD with xylene enabled better workability in terms of the use of isooctane. Indeed we observed that the faster the evaporation process of the solvent (generally with isooctane), the denser and more homogeneous the layer formed by CDD. Isooctane, therefore, presented an evaporation velocity which was too fast, thus making the correct application of CDD difficult.

The dilution of CDD to 80% in isooctane formed a very similar layer to that formed by pure CDD. Xylene, on the other hand, underwent a gradual evaporation velocity which allowed a more appropriate application as well as the formation of a uniform layer.

3.3. Characterisation of the film formed by CDD in xylene at different concentrations: 40%, 60% and 80%.

The petrous specimens prepared with CDD at different concentrations with xylene were observed by cryo-SEM. The studies performed found differences in the crystalline structure of CDD, which varied according to the dilutions used and the different petrous supports prepared. Figure 3 (A-D) shows these significant modifications which confirm the formation of a thin layer of porous, heterogeneous and intertwined crystals as the concentration of CDD decreased.

The presence of a larger amount of solvent allowed the formation of a layer of CDD crystals. However, the crystals decreased as the concentration of CDD increased, in which case, a more homogeneous and dense layer was formed.

On the other hand, different behaviours were also observed when the same concentration of CDD was applied to petrous supports with different porosities. Thus, the most porous petrous support absorbed the solvent the fastest, and prevented the layer of CDD crystals from forming (Motta 2004). In this case, when 60% CDD was applied on both the Bateig cream stone and the Estremoz marble, significant differences were observed. On the most porous support, a dense and homogeneous layer formed (Figure 3B), while a more crystalline layer formed on the less porous support (Figure 3D)

3.4 Study of the interface layers (PVOH and G.A.) with 40%, 60% and 80% CDD and after applying silicone.

The petrous specimens were prepared with CDD applications at different concentrations. Next, interface layers were applied and, after drying, the silicon elastomer was applied.

Colorimetric measurements were taken of all the specimens to observe the most significant changes in total colour.

In general, the most satisfactory results, with a ΔE of between 2 and 3.5 points, were obtained with the specimens treated with 75% PVOH at any CDD concentration and for both petrous supports. This is a result of PVOH's ability to create a very homogeneous and flexible film on CDD, thus avoiding contact with silicone.

At the same time, specimens showed a decrease in ΔE after being subjected to the accelerated ageing test with ultraviolet light. The most satisfactory results decreased to values of between 0.39 and 1.36 points, and corresponded to the specimens treated with 75% PVOH at any CDD concentration and for both petrous supports (Tables 1-3).

Table 1. Values of total colour variation for the Estremoz Marble (EM and Bateig Cream (BC) treated with 40% CDD, PVOH and G.A. interface layers at different concentrations.

Specimen	ΔE* after removal	Final ΔE* after U.V	
EM8 (25% PVOH)	**4,37**	**3,69**	
EM9 (50% PVOH)	**3,99**	**3,45**	
EM10 (75% PVOH)	**2,12**	**0,72**	
EM11 (10% G.A.)	**3,68**	**3,11**	
EM12 (25% G.A.)	**3,58**	**3,68**	
EM13 (50% G.A.)	**3,43**	**1,39**	
BC8 (25% PVOH)	**6,72**	**4,89**	
BC9 (50% PVOH)	**4,83**	**3,64**	
BC10 (75% PVOH)	**3,35**	**0,87**	
BC11 (10% G.A.)	**3,43**	**3,61**	
BC12 (25% G.A.)	**2,01**	**0,96**	
BC13 (50% G.A.)	**0,73**	**0,74**	

Table 2. Values of total colour variation for the Estremoz Marble (EM) and Bateig Cream (BC) treated with 60% CDD, PVOH and G.A. interface layers at different concentrations. .

Specimen	ΔE* after removal	Final ΔE* after U.V
EM36 (10% G.A.)	**10,68**	**10,11**
EM26 (25% G.A.)	**11,27**	**10,23**
EM27 (50% G.A.)	**7,62**	**7,74**
EM8 (25% PVOH)	**7,37**	**7,69**
EM28 (50% PVOH)	**7,92**	**7,15**
EM29 (75% PVOH)	**3,21**	**1,36**
BC142 (10% G.A.)	**3,32**	**3,41**
BC26 (25 G.A.)	**1,26**	**1,02**
BC27 (50% G.A.)	**1,13**	**1,01**
BC37 (25% PVOH)	**5,71**	**4,52**
BC28 (50% PVOH)	**0,54**	**1,05**
BC29 (75% PVOH)	**1,05**	**0,39**

Table 3. Values of total colour variation for Estremoz Marble (EM) and Bateig Cream (BC) specimens treated with 80% CDD and PVOH and G.A. interface layers at different concentrations.

Specimen	ΔE* after removal	final ΔE* after U.V
EM42 (10% G.A.)	**9,68**	**9,11**
EM30 (25% G.A.)	**8,57**	**8,54**
EM31 (50% G.A.)	**8,16**	**6,41**
EM8 (25% PVOH)	**7,37**	**7,69**
EM32 (50% PVOH)	**5,71**	**3,63**
EM33 (75% PVOH)	**2,06**	**0,61**
BC39 (10% G.A.)	**4,43**	**4,19**
BC30 (25% G.A.)	**2,65**	**4,59**
BC31 (50% G.A.)	**7,13**	**5,09**
BC8 (25% PVOH)	**6,72**	**4,89**
BC32 (50% PVOH)	**1,48**	**1,12**
BC33 (75% PVOH)	**2,38**	**0,68**

On the other hand, the results obtained with the FTIR spectroscopy analysis of the superficial samples extracted from the specimens (before and after treatments) revealed that there was a lack of residues on the specimens treated with 75% PVOH at all the CDD concentrations and for both petrous supports, as shown in Figure 4 or, alternatively, values were lower than those levels detected by the equipment.

4. CONCLUSIONS

This paper presents a new moulding method for sculptures made with petrous supports which employs a bilayer formed by CDD and PVOH. This method consists in a layer system with different polarities which allowed the original object to be isolated and protected from silicone, this being one of the causes of irreversible stains on petrous substrates. The specific interest in the use of CDD as a temporary barrier is because of its ability to sublimate, thus preventing the original work from being subjected to subsequent removal actions from the barrier itself.

Firstly, this study confirms that applying melted CDD to petrous supports is not viable since it forms a very dense film that alters the impression due to its rapid solidification. Secondly, tests with

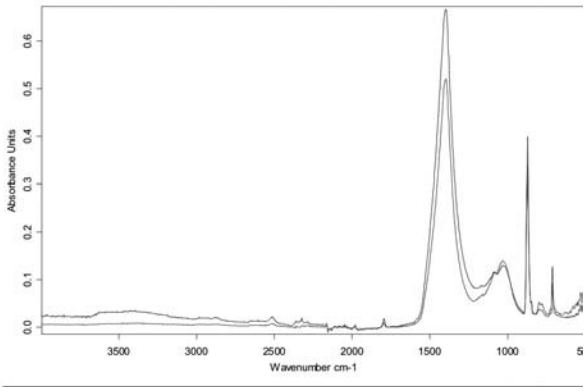

Figure 4. FTIR absorption spectra for the Estremoz Marble specimen before the tests (lower line)/FTIR absorption spectra of the Estremoz Marble specimen (with 75% PVOH, upper line) after the tests.

tested dilutions indicate that CDD diluted in xylene presents a more adequate evaporation speed than when isooctane is in use. The most suitable CDD concentration is 60% in xylene since it permits a correct application and film formation on stone. Thirdly, the CDD film formed on petrous materials depends on both the concentration used and the porosity of the petrous substrate. When a larger amount of solvent is used, CDD forms a slightly dense and heterogeneous crystalline film which becomes a denser and more homogeneous film as the CDD concentration increases. Finally, the polar substance which provides the most satisfactory results is 75% PVOH applied to any CDD concentration and on any petrous material. Therefore, the use of the CDD-PVOH bilayer system is a new technique to protect sculptural and ornamental stone pieces during moulding processes with silicon elastomers.

ACKNOWLEDGEMENTS

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ANNEXE Instrumentation

Cyrogenic scanning electron microscopy (cryo-SEM)
A Jeol JSM 3600 scanning electron microscope with a Link-Oxford-Isis X-ray microanalysis system was used which operating at a voltage of 20 kV, a beam current of 2.10-9 A and a working distance of 15mm. Samples were frozen to prevent CDD from sublimating and were subjected to gold shadowing to obtain images.

Spectrophotometry--Colorimetry
Chromatic coordinates were obtained using a Minolta CM-2600d spectrophotometer and standard CIE D65 lighting conditions (daylight, colour temperature of 6500ºK) and the standard 10º observer, with a

measurement area of 8-mm diameter for Estremoz marble, Bateig cream and Moleanos limestone, and one of 4mm-diamter for the Tosca de Rocafort stone. Data were collected with the specular component included (SCI), with the specular component excluded (SCE) and with 100% UV. The wavelength range was between 400 and 700 nm, while the photometric range oscillated from 0 to 175% reflectance with 0.01% resolution. Repeatability presented a standard deviation of 0.1%. CIELAB colour space was used.

Optical microscopy (LM)

A Leica MZ APO Binocular Loupe was used with magnifications ranging from 8x to 80x, coupled with a digital photographic system and bilateral fibre optic illumination.

Fourier transform infrared spectroscopy (FTIR)

A VERTEX 70 spectrometer operating with an ATR (Attenuated Total Reflection) reflection system and equipped with a FR-DTGS temperature stabilised-coated detector was used. The operating conditions were the following: 32 scans; a resolution of 4 cm-1. Data were processed with the OPUS software, version 5.0.

Thermal and moisture ageing chamber

A Dycometal DI-100 climatic chamber was used with a temperature range of between 25ºC-150ºC and an RH of between 15-98%, with refrigeration and heating gradients of 1 and 2ºC/minute, respectively. The test conditions in the chamber throughout the study were 30ºC and 65% relative humidity.

Ultraviolet irradiation aging chamber

A QUV/Basic chamber was used with 8 UVB 313EL lamps (radiance 0.77W/m/nm), which are the equivalent to 40 watt fluorescent lamps. The spectral region of the UV lamps was between 280 and 315nm, with an emission peak at 13nm.

Versión española

TITULO: *Estudio del ciclodecano como barrera temporal en el moldeado de esculturas pétreas y piezas ornamentales.*

RESUMEN: *En esta comunicación se describe el estudio del hidrocarburo cyclododecane (C12H24) y su idoneidad como temporary barrier en el moldeado de piezas pétreas con diferente porosidad. Para ello, se han elegido dos tipos de materiales pétreos, la biocalcarenite Bateig de Novelda (Alicante, España) y el mármol de Estremoz (Évora, Portugal). El proceso de moldeado de una obra de arte es una tarea muy compleja que requiere de un estudio exhaustivo de la pieza y de la elección del barrier más idóneo que impida, con el tiempo, la presencia de residuos y cambios cromáticos indeseables. En este caso, la metodología de actuación se basó en preparar probetas de ambos materiales pétreos con diversas disoluciones de CDD y someterlas a ensayos de envejecimiento acelerado por irradiación con luz ultravioleta. Los resultados se obtuvieron a través de medidas de colorimetría y espectroscopía infrarroja por transformada de Fourier (FT-IR). Se observó que el CDD, empleado como temporary barrier en piedras de diversa naturaleza, presenta unas propiedades muy destacables con respecto a otros desmoldeantes de uso más generalizado y, la capacidad de sublimación que posee impide la presencia de cualquier residuo y cambio cromático, convirtiéndolo en una de las sustancias más prometedoras en el campo de la Conservación y Restauración de Bienes Culturales.*

PALABRAS CLAVES: *Ciclodecano, esculturas pétreas y ornamentos, reproducciones, Bateig, biocalcarenita, mármol Estremoz, película de barrido*