Going Green in Conservation of Contemporary Art and Design Objects: Evaluation of Surface Cleaning with Biodegradable Agents

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With the aim of finding efficient cleaning methods and materials and addressing environmentally benign conservation treatments within museum practice, the study presented here introduces a series of selected environmentally friendly and biodegradable solvents and cleaning agents and assesses their potential use for the cleaning of contemporary art objects made of plastics. The cleaning processes were tested on samples of polymethyl methacrylate (PMMA) and biodegradable polylactic acid (PLA), which is widely used for prototypes and 3D printed art and design objects. Surface alteration effects were studied using optical microscopy (OM), scanning electron microscopy (SEM), and atomic force microscopy (AFM). µ-Raman spectroscopy and gloss measurements were also used to evaluate the cleaning efficacy. The results of the study show the dependency of the cleaning result on the type of plastic. However, they are very encouraging regarding the incorporation of environmentally friendly methods and materials in modern and contemporary art conservation practice.

INTRODUCTION

Contemporary art and design museums include in their collections a large number of objects comprised of synthetic polymers, the chemical and physical properties of which have been modified by the use of additives that ease their manipulation and shaping. The proportion of plastic materials in museum collections is constantly increasing as even more contemporary artists use them in their artworks but also as plastic objects enter the museums for their social, cultural, and historical significance in modern life. The drawback of plastics' instability has raised concerns regarding the longevity of such artworks and objects, and the lack of long-standing conservation experience in plastics has increased the research interest in establishing appropriate conservation treatments. The accumulated knowledge from the conservation of traditional materials has a limited contribution to the conservation of plastics, mainly due to compositional differences and also due to the vast variety of plastic types and methods of synthesis, manufacture, and processing, which are continuously evolving. Surface cleaning is a necessary conservation treatment in order to remove superficial dirt deposits and restore the aesthetic appearance and longevity of artworks. However, not all solvents used in the conservation field are appropriate for plastics, and some of them can cause irreversible damage (Shashoua 2008, 207–13).

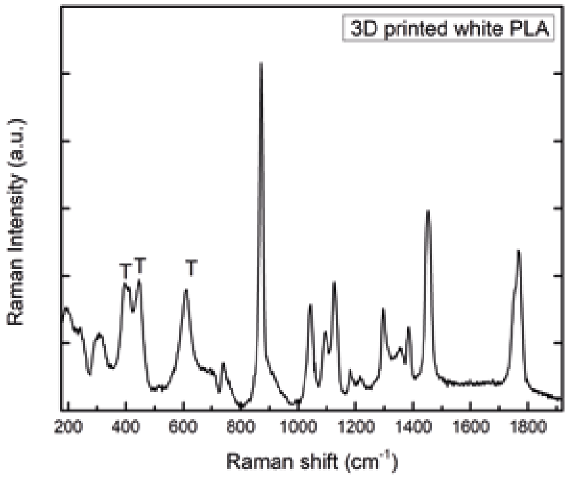
Museums as cultural institutions are encouraged to adopt environmentally sustainable practices and, thus, contribute to the achievement of the 17 Global Goals of the United Nations (McGhie 2020). Concerns about environmental and human health necessitate the replacement of substances that are environmentally questionable regarding toxicity with alternative ones that are considered to be safer to human health and the environment. In the literature, there are already numerous reports of environmentally friendly materials that have been tested for conservation purposes. These include essential oils, biogels, enzymes, ionic liquids, biodegradable surfactants, and solvents (Belluci et al. 1999; Macchia et al. 2019; Marco et al. 2020; Ormsby et al. 2013; Pacheco et al. 2013; Prati et al. 2018). Green materials and methods have also been applied in the conservation of plastics but in most cases in combination with conventional solvents (Hackett 2014; Kavda et al. 2017).

The present study aims to contribute to the development of environmentally friendly cleaning protocols that are safe to use in the conservation of plastic objects. Polymethyl methacrylate (PMMA), a plastic often found in museum collections, and the biodegradable polylactic acid (PLA), which is used for 3D printed design objects and artworks, were treated for evaluating aqueous surface cleaning as well as the use of natural agar gel, a biodegradable chelate, a biodegradable surfactant, and two green solvents as cleaning agents. The cleaning agents were evaluated regarding cleaning residues and induced surface alterations, and were tested for their cleaning efficacy.

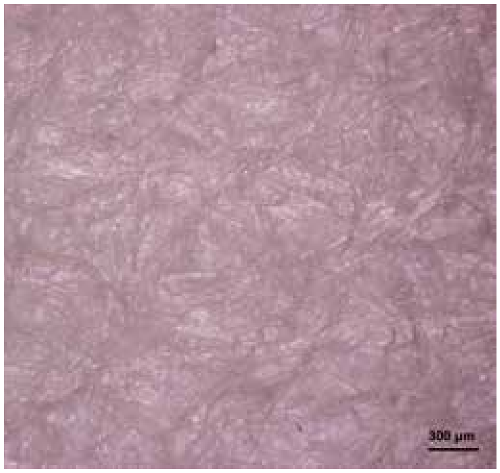
EXPERIMENTAL

Materials and Methods

Samples of commercially available colorless PMMA and 3D printed white PLA that were first analyzed by Raman spectroscopy regarding their composition, were used for the cleaning tests. The PLA was 3D printed by Planfab, a Greece-based printing service company, using PLA filament produced by the MCPP company (Mitsubishi Chemical Corporation). The white pigment in the PLA was identified as titanium dioxide (TiO_2) by Raman spectroscopy [F. 01]. The thickness of the PMMA and PLA samples was 3 mm and they were prepared in two different dimensions. A set of small samples with



[F. 01]



[F. 02]

dimensions of 2 x 2 cm was used for the evaluation of the cleaning tests under SEM and AFM. Larger samples of 6 x 20 cm were used for the gloss measurements and for the spectroscopic analysis.

Artificial soiling of the samples was done by applying solutions of 5% carbon black pigment of approximately 10 μm particle size (47200 IvoryblackJU from Kremer Pigmente GmbH& Co. KG) in mineral oil and 2% palmitic acid in 1-propanol imitating carbonaceous soil and oily substances soil and fingerprints (sebum) respectively. The carbonaceous soil was applied using a soft brush and the sebum soil by spraying the surface of the samples.

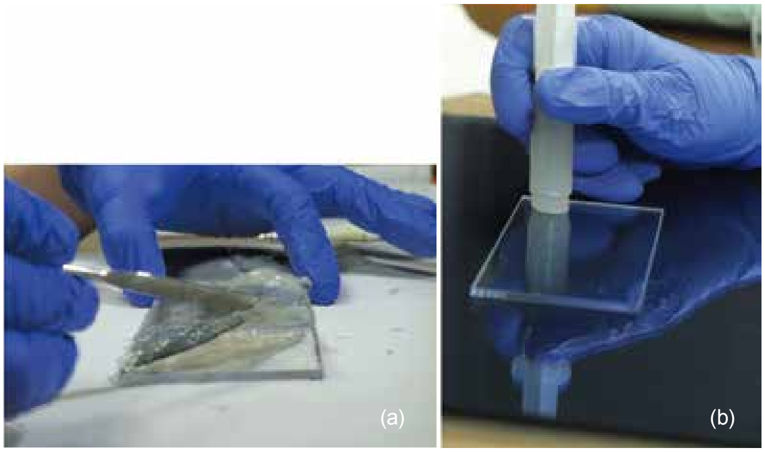
The cleaning agents tested were: a) Deionized (DI) water. b) Agar gel in DI water (4% w/v) prepared from agar powder (ACROS) and applied on the samples' surface as hot gel at approximately 40°C –50°C for the PMMA and 35°C for PLA, removed after 5-minute application time by simply peeling it off, and as water-containing eraser (cold application). In the latter case, cleaning action involved rubbing pressure. c) Trisodium salt of methylglycinediacetic acid (MGDA - Trilon® M by BASF) 2% in DI water, which is a biodegradable chelate that could be used instead of ethylenediaminetetracetic acid (EDTA), for which environmental concerns have recently been raised. d) Nonionic surfactant based on alkoxylated fatty alcohols (Plurafac® LF900 by BASF) 1% in DI water. It is a biodegradable surfactant, already used in household detergents and home care products, that exhibits low foaming, a desirable property for conservation applications. e) (R)-(+)- Limonene (Sigma-Aldrich). f) (-)-Ethyl L-lactate (Sigma-Aldrich). Limonene and ethyl lactate are considered environmentally friendly solvents that remove grease and oil (Cirimina et al. 2014; Pereira et al. 2011). Sterilized 100% pure cotton swabs and Evolon® CR (Freudenberg Performance Materials – Deffner&Johann), a nonwoven highly absorbent microfilament fabric of 70% polyester and 30% polyamide, specially manufactured for conservation, were used as cleaning materials. The clearance step for the removal of residues was carried out with DI water [F. 02].

Initially, both cleaning materials were tested dry on the untreated plastic samples, and any effects produced by the dry treatment were documented in order to choose the safer cleaning medium, which would then be used for applying the wet cleaning agents. This was done by gently rolling the cotton swab or rubbing the Evolon® CR cloth on the surface of the plastics, without any pressure. The cotton swabs were ready-made and had the same weight and dimensions; the Evolon® CR cloth—so as to safely make a comparative evaluation of the cleaning tests—was cut in pieces of the same dimensions, specifically 3 x 3 cm and 2 x 2 cm, for the big and small samples respectively.

[F. 01]
Raman spectrum of the 3D printed white PLA with the characteristic peaks of titanium dioxide (T) at 400, 445, and 610 cm^{-1}.

[F. 02]
Microphotograph of Evolon CR® cloth.

[F. 03]



[F. 04]

[F. 03]
Application of Evolon CR® cloth for the cleaning procedure.

[F. 04]
Cleaning treatment using agar in the form of hot film (a) and cold eraser (b).

The same applies for the wet cleaning tests, where the same amount of aqueous agent was used for each application, 0.40 mL for the big samples and 0.10 mL for the small samples. In the case of the big horizontal samples, more than one piece of Evolon® CR cloth was used in order to clean the whole surface of the plastic. The cleaning agents were applied by rubbing 15 them back and forth across the surface during the cleaning process for both plastics, in the case of the DI water, the agar eraser, the MGDA chelate, the Plurafac® surfactant, and the two solvents, namely limonene and ethyl lactate [F. 03]. Hot viscous agar film was applied by means of a soft brush and left on the surface for 5 minutes [F. 04a]. Agar in the form of an eraser was applied by gently rubbing the surface of the plastic [F. 04b]. After removing some of the soil, the top part of the eraser was cut off, in order to avoid redistributing the soil that has been attached to the agar gel. After the preliminary screening on the untreated plastics, the cleaning agents were then tested for their efficacy of soil removal. In this case, different application times were used for each sample, depending on feasible degree of soil removal, while a maximum of 30 passes across the surface was set for all cleaning agents, except for the hot viscous agar, which was left to dry on the surface for 5 minutes [F. 03 - 04].

The cleaning process was completed by applying a subsequent clearance step, aiming to remove the cleaning agents' residues from the plastic's surface and also to stop the cleaning action. A wet clearance with DI water was applied 10 times—in the case of the agar (both types), the chelate, and the surfactant—followed by a dry step, which was applied 5 times, using a dry piece of Evolon® CR cloth. The cleaning treatment with the two solvents and the DI water was completed by applying a single dry clearance step. Each cleaning agent was tested twice, using duplicates of each plastic type.

Observation of morphological changes and remaining residues on the surface of the samples was done by: a) Optical microscopy (OM): a Zeiss Axioplan 2 Imaging microscope equipped with a Nikon D700 camera was used for the visual observation of the samples at magnifications 50–200x. b) Scanning electron microscopy (SEM): a JEOL JSM-6390LV scanning microscope was used. Operating conditions were: accelerating voltage 5–20 kV, probe current 45nA, and counting time 60 seconds. The samples were coated with carbon, using a Jeol JEE-4X vacuum evaporator. c) Atomic force microscopy (AFM): a Bruker Innova AFM system operating in tapping mode with silicon probes was used. Images of 10 x 10 μm areas were collected in height, amplitude, and phase mode, using Bruker Nanodrive v.8.0 and analyzed with Bruker Nanoscope Analysis v.1.40 software. The tapping amplitude images gave the most comprehensive information, showing changes in the topography of the samples.

Raman spectra were collected using a LabRamHR (Horiba), single stage, micro-Raman spectrometer equipped with a Peltier-cooled CCD detector. For the excitation of the spectra, a Fandango (Cobolt) diode-pumped laser at 515 nm was used and the laser power was kept at ~1mW. The spectra resolution of the system was ~3.5 cm^{-1} and a standard 100x, N.A. 0.9 objective was employed.

A Rhopoint NOVO GLOSS 20/60/85 gloss meter was used for measuring the gloss of the plastic surfaces before and after the cleaning tests. Three readings were conducted on each sample, and the average was calculated and used, taking care to preserve the same relative positioning of the gloss meter with respect to the sample for the measurements taken both before and after the cleaning treatment. For the transparent PMMA, in order to avoid any reflections originating from the lower surface of the plastic, a matte black photographic foil was placed under the sample with a drop of water in between the two surfaces to optically bond the transparent plastic to the black foil. For the PMMA, that gave gloss values at the reflectance angle of 60° higher than 70 SGU (high gloss), the gloss was measured at 20°. For the PLA that gave values lower than 10 SGU (low gloss), the angle of 85° was used for improved resolution (Hanson 2006, 20–71).

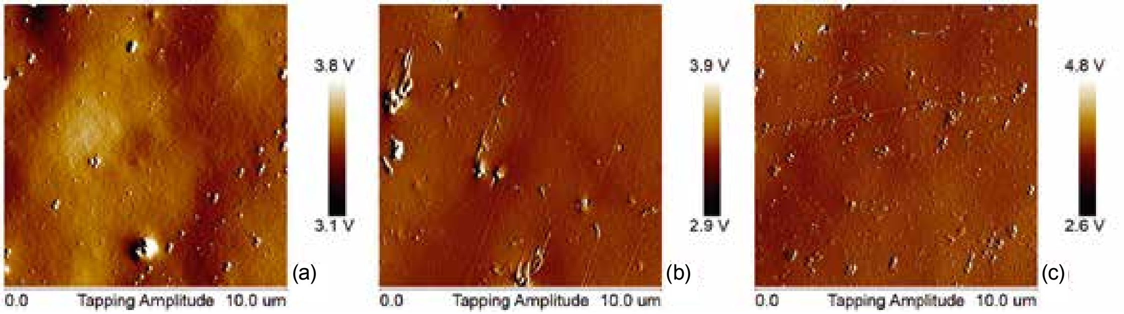
Results

Polylactic acid (PLA)

Testing the two cleaning materials, namely the cotton swab and the Evolon® CR cloth, on the PLA surface had as a result the observation of scratch marks caused by the swab. These marks were observed mainly by the AFM, while they were not noticed with naked eye or under the optical microscope [F. 05]. Despite the fact that this effect was recorded on the nano scale, it was alarming regarding the use of the cotton swab on the relatively soft surface of the PLA. Some residues from the mechanical action of both cleaning materials were left on the plastic surface. However, they were easily removed during the wet clearance step. The PLA surface was also characteristically distorted by the rubbing action of the agar eraser application, indicating that the specific plastic surface is rather sensitive to any form of pressure [F. 05–06].

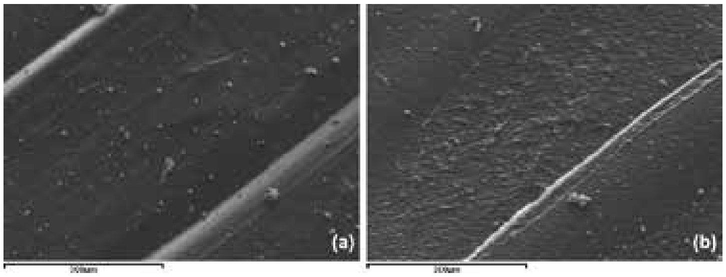
The treatment of the unsoiled PLA surface with DI water changed the topography of the surface. This was mainly observed using AFM microscopy, where roughness changes were recorded. In this case, significant changes in the roughness were interpreted as a result of swelling occurring from the adsorption of water on the PLA. Despite the hydrophobic nature of PLA (Baran and Erbil 2019), 3D printed PLA has a structure of higher porosity that is obviously affected by water, which is absorbed through the pores of the 3D printed structure (Ecker et al. 2019). For this reason, water was not tested for the soil removal. However, the decision was made to proceed with the rest of the cleaning agents that were prepared as aqueous solutions, so as to test if the degree of swelling decreased.

Indeed, the presence of the MGDA chelate reduced the swelling effect. The main drawback for the MGDA, observed with OM and SEM, was the amount of cleaning residues left on the plastic surface. The removal of the residues could be achieved by amending the clearance step and increasing the number of passes with the wet Evolon® CR cloth. Nevertheless, this prolonged water contact would not be appropriate for the PLA, since it would lead to increased surface distortion. As a result, the MGDA chelate was not tested for soil removal efficacy on the PLA surface. The Plurafac® solution also left some residues on the PLA surface that required additional clearance rubs with the wet cloth to be removed and consequently posed a risk for surface swelling. In this case, though, cleaning tests on the artificially soiled PLA samples showed an excellent cleaning efficacy of the Plurafac® solution for the carbonaceous soil [F. 07a]. Additionally, an immediate final



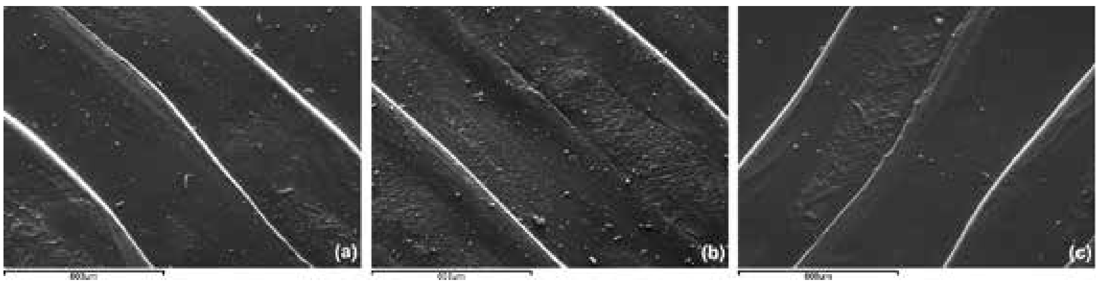
[F. 05]

[F. 05]
AFM images obtained from PLA untreated (a) and after the application of cotton swab (b) and Evolon CR® cloth (c).



[F. 06]

[F. 06]
SEM images obtained from samples of PLA, after the application of agar as hot film (a) and as cold eraser (b).

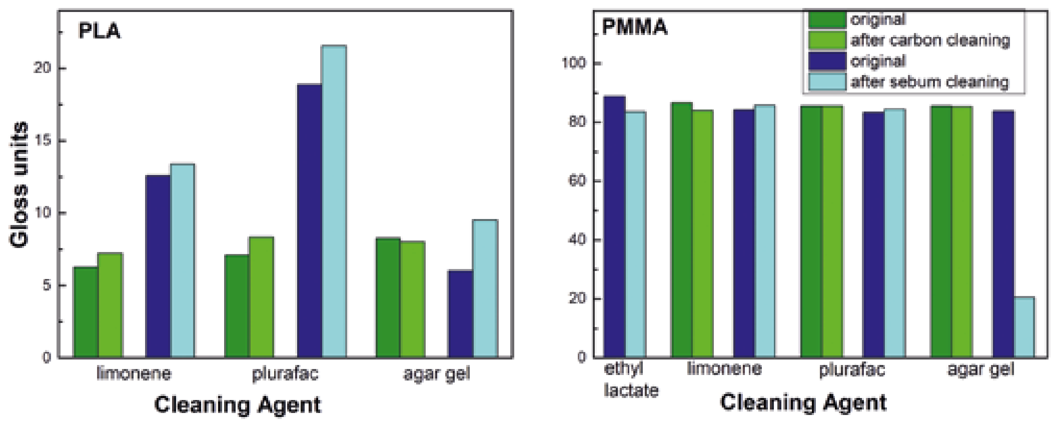


[F. 07]

[F. 07]
SEM images obtained from PLA carbon-soiled samples after the cleaning application with Plurafac® (a), agar as hot film (b), and limonene (c).

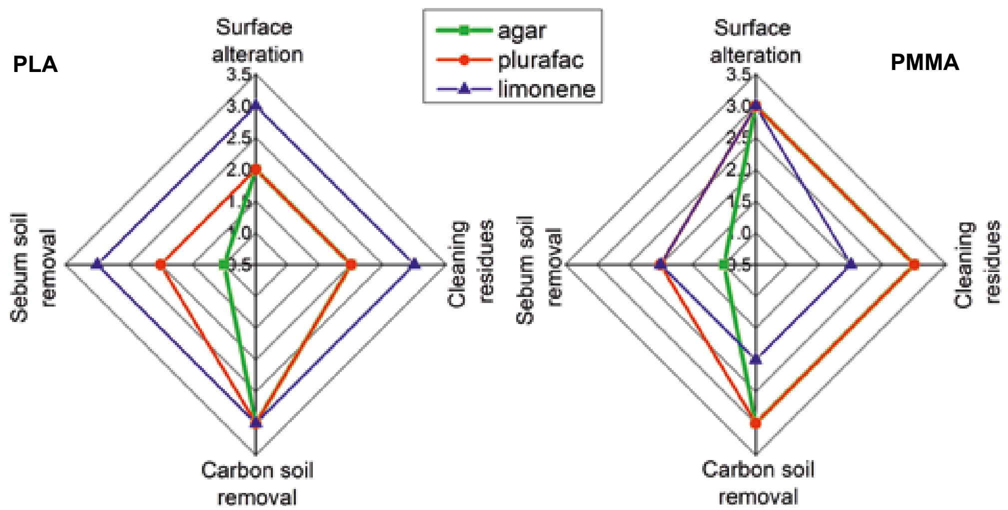
wiping with a dry piece of Evolon® CR cloth minimized the water absorption, and the final plastic surface looked clean with hardly any soil residues and with no remarkable topography variations from the original one. The cleaning efficacy for the sebum soil was not as satisfactory as for the carbonaceous soil, and in combination with the rough surface of the 3D printed PLA, it resulted in remaining soil residues.

As mentioned above, the rubbing action of the agar gel eraser caused surface alterations on the PLA. Thus, agar gel was tested for its cleaning effectiveness only in the form of a viscous film. The temperature of the gel for the treatment of PLA was 3°C, which is lower than the one usually used (40 °C–45°C) (Cremonesi 2016). The Tg of PLA is 57°C (Garlotta 2001), rendering it sensitive at temperatures around 50°C, as we noticed in a previous study (Kampasakali et al. 2019). Agar gel allowed limited and controllable contact between the water and the PLA surface, reducing in this way the swelling effect caused by water. Some cleaning residues that were left on the surface were easily removed during the clearance step. It must be also noted that in the case of agar gel eraser, the amount of the residues was higher, and this was also attributed to the rubbing friction applied on the plastic surface. Sebum soil could not be adequately removed by the agar gel. This cleaning agent was most effective against carbonaceous soil, leaving soil residues mainly in the indentations that exist on the PLA surface as result of the printing process [F. 07b].



[F. 08]

[F. 08]
Gloss values of untreated PLA and PMMA surfaces and after the cleaning treatment with biodegradable agents.



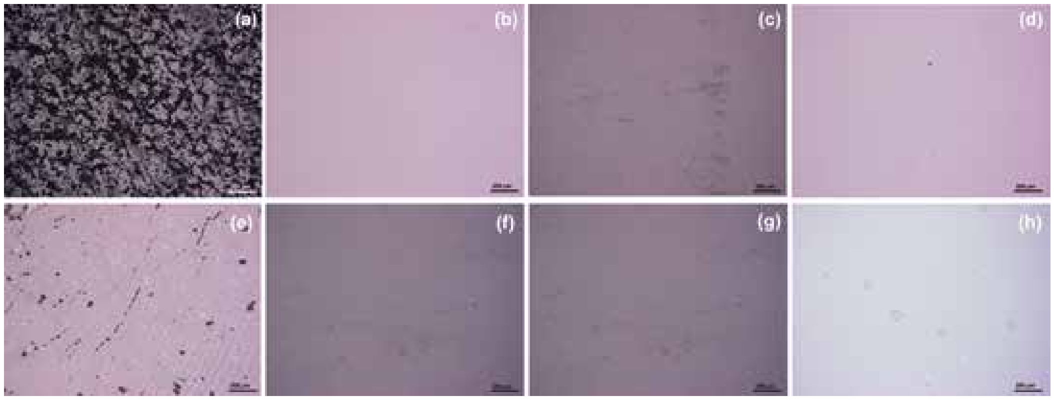
[F. 09]

[F. 09]
Radar charts for PLA and PMMA comparing agar, Plurafac®, and limonene regarding surface alterations, cleaning residues, and cleaning efficacy.

Regarding the two organic solvents, ethyl lactate and limonene, these had diametrically opposite effects on the PLA. Ethyl lactate proved detrimental for the PLA surface. According to the literature, PLA film is not soluble in polar protic solvents, such as ethyl lactate, but intense swelling can take place after immersion in the solvent (Sato et al. 2013). In this study, it was shown that it is possible for PLA, in the form of a 3D printed object, to swell and deform irreversibly, even after short contact with ethyl lactate. Therefore, ethyl lactate was not tested for soil removal from the PLA surface. On the other hand, the treatment of unsoiled PLA with limonene did not cause any swelling, deformations, or residues on the PLA. When tested against the two types of soil, namely carbonaceous and sebum, limonene was very effective in both cases, leaving a negligible amount of soil residues [F. 07c].

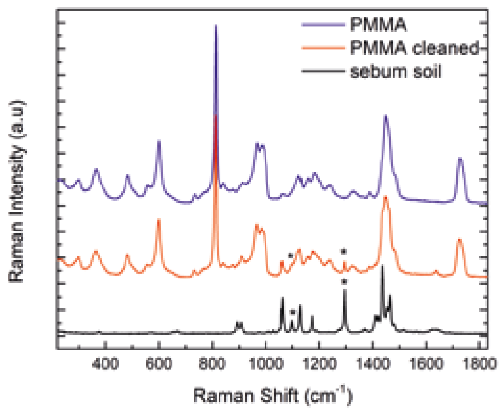
As regards the effect on the gloss of the PLA surface, only the use of ethyl lactate affected the gloss value, resulting in a decrease of almost 50% from the original value. Gloss values of the final PLA samples' surface, after the soiling and cleaning, were similar to the original ones, and the more noticeable increase observed after the cleaning of sebum soil with agar gel and Plurafac® solution was attributed to the residual soil that is glossier than the PLA surface [F. 08].

In Figure 09, the radar chart for PLA summarizes the effects of the cleaning agents tested for soil removal. The larger the area, the more promising the cleaning agent is (scale 1 to 3, 1: poor effect, 2: good effect, 3: very good effect), i.e., the limonene solvent is most promising for the PLA [F. 09].



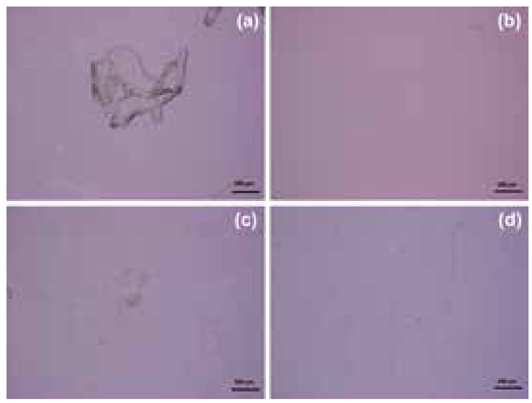
[F. 10]

[F. 10]
Optical microscopy images ontained from samples of PMMA soiled with carbon (a) and sebum soil (e), and after cleaning with Plurafac® (b and f), agar hot film (c and g), and limonene (d and h).



[F. 11]

[F. 11]
Raman spectrum of PMMA sample untreated and cleaned with Plurafac®, showing peaks of sebum soil (*).



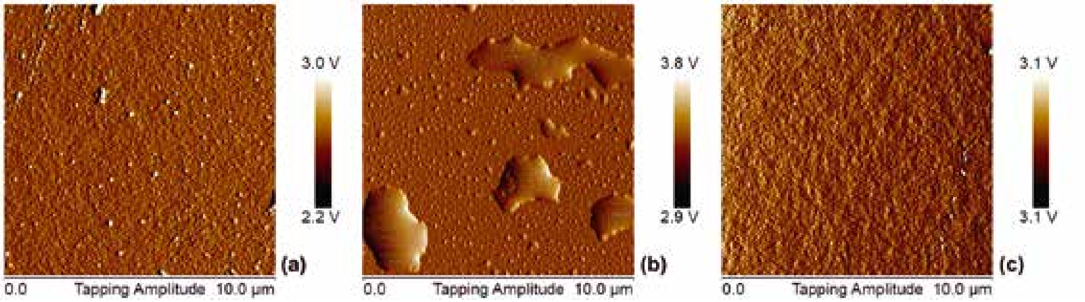
[F. 12]

[F. 12]
Optical microscopy images of untreated PMMA, after the application of hot agar film (a), agar in cold eraser form (c), and after the subsequent clearance using DI water (b and d).

Polymethyl methacrylate (PMMA)

Although the effect of the cotton swab regarding scratch marks was milder for the PMMA, all the cleaning tests on PMMA samples were conducted with Evolon® CR cloth as well. DI water did not cause any surface changes. It was tested only for carbonaceous soil removal because of the hydrophobic nature of the sebum soil. However, the cleaning result was not sufficient for the carbon soil either, and a considerable amount of residue was observed. The MGDA chelate left cleaning residues on the PMMA surface as it did on the PLA surface, despite the fact that the PMMA surface is much smoother. Consequently, the decision was made not to test it at this stage of the research as an additive that would improve the cleaning action of DI water. On the contrary, Plurafac® residues were very easily removed during the clearance step, leaving the final surface intact. Similarly, Plurafac® exhibited the best cleaning action against carbonaceous soil on PMMA with almost no residual soil observed with OM [F. 10b] and SEM. It was less effective against sebum soil where some residues were left either in the form of stains or of drag marks from the cleaning process [F. 10f]. In Figure 11, in the Raman spectrum of PMMA cleaned with Plurafac®, the presence of sebum soil residues is evidenced by the appearance of the respective sebum peaks [F. 10–11].

In the case where agar gel was applied as hot film (40°C–45°C) on the surface of PMMA, a few big pieces of residue were observed, which after the clearance step were totally removed [F. 12a and c]. In the case of the agar eraser, the residues were smaller in size but scattered over the whole surface, obviously because of the rubbing action. After the clearance step, the scattered



[F. 13]
AFM images obtained from PMMA samples: untreated (a), cleaned with limonene, showing solvent residues (b), and cleaned with limonene and wiped with dry cloth (c).

particles from the eraser were dispersed when they mixed with the water and left intense drag marks [F. 12b and d]. A prolonged clearance step was not undertaken because of the possible risk of scratching the PMMA surface. Because of the eraser cleaning residue issue, the cleaning tests were conducted with the agar film. This proved to be insufficient for the removal of sebum soil, but its efficacy against carbonaceous soil was good and improved compared to DI water, leaving a smaller amount of remaining soil on the PMMA surface [F. 10c and g] [F. 12].

Both of the organic solvents, limonene and ethyl lactate, left cleaning residues on the PMMA surface that required a dry wiping of the surface with Evolon® CR cloth after the cleaning treatment, for them to be removed [F. 13]. Limonene cleaned the PMMA sufficiently both from carbonaceous and sebum soil. When viewed macroscopically, the surface looked thoroughly clean, and a small amount of residual soil was visible with the OM and SEM [F. 10d and h]. Therefore, limonene was considered an efficient cleaning agent for the PMMA surface, perhaps with some modifications to the application method regarding the number of passes over the surface with the cleaning cloth. Ethyl lactate was tested against the sebum soil for the PMMA. The cleaned surface had stains of soil mixed with the solvent visible under the optical microscope, which with SEM resolution looked like a residual layer [F. 13].

Finally, the gloss values of the original PMMA samples' surface were not affected by the application of the cleaning agents in the case of the nonsoiled surfaces. The surfaces that were artificially soiled and then cleaned also had values similar to the original ones. The only exception to this was the one cleaned with agar gel after sebum soiling, where the cleaning action was very limited [F. 08].

The respective radar chart for PMMA is shown in Figure 09, and the area for Plurafac® is the largest one.

CONCLUSIONS

The use of biodegradable agents for the surface cleaning of PMMA and PLA was evaluated. Deionized water, agar gel, the MGDA chelate, the nonionic surfactant Plurafac® LF900, and the organic solvents limonene and ethyl lactate were tested. The assessment of the results obtained using OM, SEM, and AFM were encouraging, showing that the cleaning agents used did not cause any significant morphological changes, except in the case of ethyl lactate, which caused significant damage to the PLA surface. The minor changes in the gloss values between the original surfaces and the cleaned ones demonstrated the negligible effect of the cleaning agents on the aesthetic appearance of the plastics. The exception to this was again the effect of ethyl lactate on PLA, as well as the gloss change caused by the inadequate sebum removal by agar gel on PMMA. The cleaning efficiency of the agents depended on the type of plastic and the topography of the surface. In general, the Plurafac®LF900 solution and the limonene solvent showed good cleaning results for both types of soil. The agar gel film sufficiently cleaned the carbonaceous soil but not the sebum soil. The use of agar gel was more efficient when used as a film rather than in eraser form, due to surface alterations and higher amount of cleaning residues caused by the rubbing action. The application of the MGDA chelate resulted in a considerable amount of cleaning residues, while for the Plurafac®LF900 and the agar gel, the removal of any residues present was possible with the applied aqueous clearance step. However, aqueous treatment of PLA needs to be further explored and adjusted accordingly, in order to avoid water permeability and swelling effects. The use of Evolon® CR cloth proved to be safe for the plastic surfaces examined, especially for the softer PLA, causing fewer scratch marks compared to the cotton swab. The results of this study indicate that efficient surface cleaning of plastics is possible using exclusively green materials, and further research conducted on a broader range of plastic surfaces and biodegradable agents will certainly contribute to the adoption of sustainable conservation practices.

ACKNOWLEDGEMENTS

The authors acknowledge the support of this work by the project "Design and Development of Conservation Treatments with Biodegradable Products for Contemporary Art Museum Objects" (MIS 5047946) funded by the Human Resources Development – Education and Life Long Learning Operational Program (NSRF 2014-2020) and cofinanced by Greece and the European Union (European Regional Development Fund). They also acknowledge the Center of Interdisciplinary Research and Innovation of Aristotle University of Thessaloniki, Greece, for access to the Raman instrumentation (Faculty of Engineering) and Professor V. Zaspalis (Department of Chemical Engineering) for access to the optical microscope. They are grateful to BASF for kindly providing samples of Trilon® M and Plurafac® LF900.

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