Chapter 23
Novel "Green" Polymers, Gels and Composites for Cultural Heritage Preservation

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Abstract Novel "green" materials are crucial in cultural heritage (CH) preservation since they allow time- and cost-effective interventions and avoid risks related to the use of toxic compounds. To this purpose, the physico-chemical properties of the new materials must be tailored to the tasks they must address. This chapter discusses some of the latest advanced materials for CH conservation, e.g. "green" polymers/olygomers and composites to formulate gels and coatings, tuning their physico-chemical properties to achieve optimal cleaning or protection of works of art. In particular, recent findings in the formulation of hydrogels and organogels for cleaning tasks, and coatings for the protection of metals against corrosion, are discussed, and new aspects are highlighted in the research of these materials. Gels with controlled hydrophilicity/hydrophobicity and tortuosity, and coatings with active response to corrosion, are illustrated to show the large applicative potential of sustainable materials in CH conservation.

Keywords Sustainable materials · Hydrogels · Organogels · Composites · Coatings · Cleaning · Protection · Cultural heritage preservation

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23.1 "Green" Oligomers as Gels/Coatings Precursors

Nowadays, the development of novel materials and tools in cultural heritage (CH) preservation represents a central requirement for enhancing intervention practices, reducing restorative time and avoiding risks related to the use of toxic compounds (Bandelli et al., 2024b, c). Despite the easiness of this concept, the material's design requires a strict translation from the type of restorative task required, to its desired physico-chemical properties. It follows that, given the multitude of materials, surface morphologies and degree of ageing found on CH objects, the research of "sustainable" and "smarter" advanced tools for counteracting degradation represent an ambitious, multidisciplinary field of study.

In the last decades, a vast library of systems based on polymers and oligomers, such as gels, hybrid materials, nanostructured fluids, protective coatings and polymeric films have been developed for CH preservation aims, enabling the cleaning, the consolidation and the protection (e.g. active coatings) of organic and inorganic (Casini et al., 2023; Bandelli et al., 2024b, c). If, on one hand, the use of polymers in, and beyond, CH goals rely on the high availability, low price and ease of application, on the other hand, most polymers employed in CH practices are obtained from non-renewable petrochemical resources. Given the growing significance of sustainability, renewability and zero-impact concepts in national and international policies, such as the European Green Deal, the research of alternatives to non-renewable materials is the main goal of CH scientists. Besides, the production of "green" polymeric systems should be carried out employing a diverse feedstock base, enabling optimum energy consumption, and employing benign solvents in all the steps of the material's production (Cheng et al., 2015; Casini et al., 2023).

However, the development and use of new materials represent a delicate task, since it is based on a careful evaluation of synthetic design, structure-property relationship of the materials, and the activity of the final systems. As a result, a vast library of features should be considered and tailored to access the desired effect, resulting in time-consuming practices if traditional "try and error" approaches are employed.

To this aim, the determination of a short list of desired features can be beneficial to speed up material's development. In particular, the knowledge of polymer hydrophilic lipophilic balance (HLB) is pivotal to tune parameters such as degradation and biodegradation, swelling capability, and processability. HLB calculation rely on counting single contributions connected to the compound's chemical identities, i.e. the methylene groups content and the types and contents of hydrophilic moieties, resulting in an optimum estimation for small molecules (Guo et al., 2006). Representing macromolecules able to rearrange in solution, polymers feature dynamics that are not only related to chemical composition but are strictly connected to the balance between polymer-solvent and polymer-polymer interactions, making HLB calculation a complex operation. In this regard, the octanol/water partition coefficient in its logarithm form (logP) is commonly employed to gain insights on the HLB of small molecules and amphiphilic polymers and oligomers.

Experimentally, logP can be obtained by means of different approaches such as the shake-flask method, chromatography, and nuclear magnetic resonance analyses, relying on the partition of the target compound in water or octanol phases (Harris & Logan, 2014). It follows that logP estimation depends on the solubility in water and octanol of the target compound, hindering such estimation for macromolecules that are not soluble in the two phases.

Aiming to comply with “green” metrics, a new protocol of logP estimation was developed employing a library of degradable oligoesters obtained via the reaction of selected dicarboxylic acids, i.e. sebacic, adipic, and succinic acid, and diols, 1,6-hexanediol and 1,4-butanediol, without the addition of any solvent or catalyst (Bandelli et al., 2024b, c). Such reactants can be produced avoiding the use of petrochemical resources and, according to the European Chemical Agency, are employed in fertilizers, cleaning products and pharmaceuticals. The final oligoester species (Ox) were analysed by means of size exclusion chromatography (SEC) in tetrahydrofuran mobile phase with a system equipped with multi-angle light scattering, viscometer and refractive index detector to access reliable molar mass estimation by means of both light scattering analysis and universal calibration approaches. Overall, both methodologies resulted in a similar molar mass estimation of ~3 kg mol^{-1} for all the Ox, while moderate to high dispersity index, above 1.36, were detected. In principle, having similar molar mass, the oligoesters differed in the chemical composition impaired from the use of different diols/diacids reactants. As a result, an increase of the parameter α of Mark-Houwink, calculated from SEC data, corresponding to a transition from compact to elongated structures in tetrahydrofuran, was detected increasing the calculated logP (clogP) of the oligoesters’ repetition unit.

Aiming to gain insights on the dynamics of the Ox samples in a list of solvents with a broad range of logP, diffusion order spectroscopy nuclear magnetic resonance (DOSY NMR) analyses were performed in selected deuterated solvents, namely acetone, tetrahydrofuran, dichloromethane, chloroform, benzene and toluene at 25 °C. DOSY NMR analysis of the oligoesters enabled the estimation of their diffusion coefficient, that was translated to the hydrodynamic radius (R_h) by applying the Stokes-Einstein equation (Eq. 23.1).

$$R_h = \frac{k_b T}{6\pi\eta D} \tag{23.1}$$

where kb is the Boltzmann constant, T is the absolute temperature, η is the solvent viscosity at a given temperature and pressure, and D is the diffusion coefficient.

Independently on the sample analysed, the evolution of R_h over the solvent logP featured a pattern resembling a peak function, revealing that the Ox species featured a maximum R_h with solvents such as dichloromethane and chloroform (Fig. 23.1).

In principle, the capability of a solvent to dissolve a target macromolecule depends on the increase of polymer-solvent interactions, and the condition of highest solvation gives an indirect estimation of their logP. As expected, such estimation

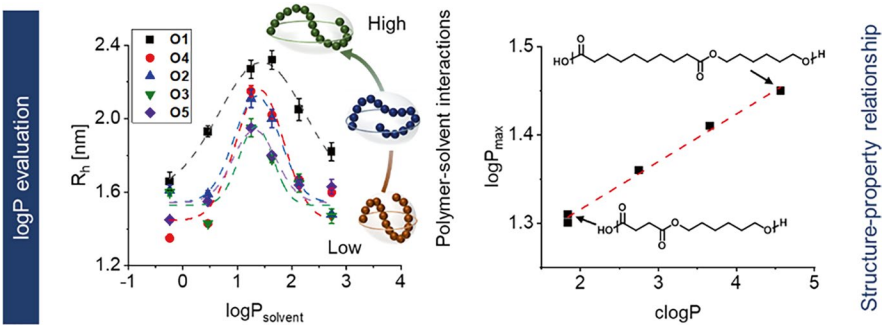

Fig. 23.1 Insights on the logP evaluation protocol. Left. Evolution of hydrodynamic radius over solvent's logP and fitting curves. Right. Evolution of the estimated logP from DOSY NMR analysis over repetition unit's logP. Reproduced with permissions from D. Bandelli et al., *Chem. Sci.* 2024, *15*, 2443–2455 (Bandelli et al., 2024b, c)

resulted in a linear logP increase over the clogP of the oligoesters' repetition unit, similarly to the trend depicted for the parameter *a* of Mark-Houwink (Fig. 23.1). Moreover, also bulk properties such as melting and crystallization temperature, and their degree of crystallinity linearly correlated with clogP increase, resulting in a strict structure-property relationship.

Overall, the library of "green" oligoesters featured similar molar mass, but different chemical composition, logP, and thermal properties, making them ideal candidates for the development of new materials such as coatings and gels for applications in cultural heritage practices and beyond.

In principle, the tailoring of polymeric features such as logP, degree of crystallinity, and glass temperature, are beneficial to tune its hydrolysis behaviour, a major parameter if degradable polymers are employed as coatings. In fact, the increase of such features reduces bulk permeability, due to a slower diffusion in the polymer bulk of water and hydrolysis reactants.

23.2 Composite Coatings for the Protection of Artworks

Traditionally, polyolefins such as polymethacrylates and polyacrylates are employed in coatings formulation for CH goals, resulting in the preparation of protecting layers (Bandelli et al., 2024b, c). Despite their use, such polymers can undergo degradative processes, resulting in the formation of patinas that should be removed to retrieve the original surface appearance. As an alternative, sustainable polymeric coatings have been developed from renewable biopolymers such as cellulose, pectin and chitosan (Caruso et al., 2023). Despite their use in CH practices, only few polymers employed in coatings have been extensively characterize with respect to their features, making the evaluation of structure-property relationship of outmost importance for further material's improvement.

In addition to the polymeric matrix, the development of sustainable and smart composites that, used as coatings, protect metal surfaces from corrosion by combining stimuli-responsive as well as superhydrophobic nanoparticles offers a promising solution for artworks long-term protection. This multifunctional approach addresses not only corrosion protection but also provides anti-biofouling and self-cleaning benefits (Piscitelli et al., 2019). An alternative strategy to protective coatings realized by embedding free active agents, such as corrosion inhibitors, within the polymeric matrix consists in approaches based on the loading of active agents into nanocarriers followed by the effective dispersion of the nanocarrier/active agent system in composite coatings. This strategy offers several advantages in the design of effective smart coatings:

(a) Due to their porosity, nanocarriers are potentially able to load high amounts of different active agents (i.e. anticorrosive and antifouling agents), allowing to overcome their solubility limit when used in polymeric coatings (Tundisi et al., 2023);
(b) Embedding active agents in the coatings through nanocarriers, also active agents that are not miscible with the coatings matrices can be used;
(c) Engineered nanocarriers/active agents systems can be designed, able to tailoring their release in selected conditions (stimuli-responsive release) (Bakhshian Nik et al., 2020);
(d) Nanocarriers protect the active agents from environmental degradation, such as UV-induced corrosion inhibitors degradation (Salzano de Luna et al., 2018);
(e) Enhancing nanocarriers dispersion and interfacial interactions with the coating matrix, also functional and structural properties of the coatings (e.g. mechanical, adhesion, indentation) can be improved.

Amongst different nanocarriers, mesoporous silica nanoparticles (MSNs) stand out for the relevant properties, including high surface area, chemical stability, low reactivity and good compatibility with several matrices (Olivieri et al., 2021a, b). More in detail, when MSNs are employed as nanocontainers of active species in coatings, both the loading of active agents and possibly their stimuli-responsive release must be properly designed and optimized. The release of inhibitor from MSNs can be modulated by taking account the inhibitor dimension and surface chemistry of MSN porous structure (Castaldo et al., 2020). More effectively, suitable capping/release mechanisms have to be designed to promote the release of the actives in selected conditions, ideally in presence of aggressive species for the substrate to be protected or soon after degradation phenomena start: capping/release mechanisms can be based on the application of external polymers or oligomer layers able to prevent the release of the agents, on the formation of coordination complexes in the external layer of the nanocarrier/active ingredient system (Xie et al., 2021), or by creating grafting the actives on the nanocarriers by reversible covalent bonds (Olivieri et al., 2023).

Recently, a highly effective capping/release system for the controlled release of a widely used anticorrosion agent, namely 1,2,3-benzotriazole (BTA), has been developed (Fig. 23.2, left) (Olivieri et al., 2021a, b). The capping system is realized

by treating MSNs preloaded with BTA with a solution containing Ag(I) ions, that promotes the formation of a layer of BTA-Ag coordination complex around the loaded MSNs. This BTA-Ag complex has a pH-dependent stability, being soluble in acid (below 5) or basic conditions (above 8) while it is insoluble in neutral conditions. Thus, at neutral pH conditions, the capping system is stable and the release of BTA from the MSN nanocontainers is negligible. On the contrary, the BTA-Ag complex dissolves in acidic or basic conditions and BTA is released from MSN and diffuses into the coating matrix towards the metal substrate, creating a protective layer adsorbed on the metal surface to be protected.

However, when the BTA-Ag complex is solubilized and silver ions are released together with BTA, an active protective mechanism, due to the silver capability to sequestrate chloride ions and thus to delay the permeation of aggressive species toward the metal surface, is also effective (see Fig. 23.2, Left side).

The effectiveness of the MSN-BTA-Ag nanoparticles was validated in acrylic coatings applied on metals, demonstrating its superior protective effectiveness when the metal was aged by accelerated acid and basic corrosion tests. Activities are currently on going to develop further smart systems realized by loading onto MSN new bio-based anticorrosion agents, alternative to BTA, and suitable capping/release mechanisms and to exploit these systems in bio-derived and sustainable polymer coatings.

To further improve the protective performance of coatings, incorporating tailored superhydrophobic particles like Janus particles can be crucial. These particles are integrated into the coating and shield the underneath surfaces of artworks from harmful water interactions—whether from the environment or vandalism—by preventing any contact. Since most of developed sustainable coatings are water-based, the Janus particles must be dispersible in water while retaining their hydrophobicity. This is achieved by engineering nanoparticles with amphiphilic properties to improve dispersion in water-based coatings and hydrophobic properties to enhance surface protection (Fig. 23.2, Right side). Amongst different Janus nanoparticles used in protective coatings, silica based nanoparticles are gaining an even increasing interest for their effectiveness (Milian et al., 2023).

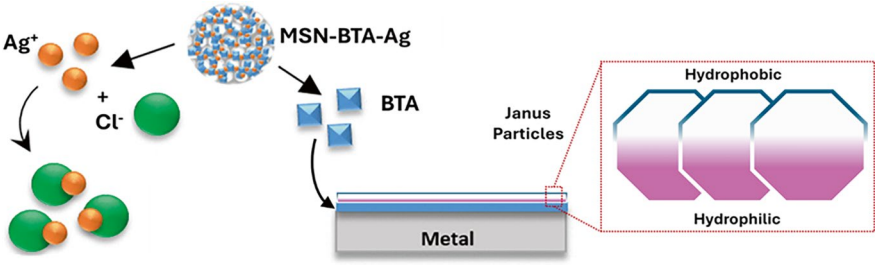

Fig. 23.2 (Left) Twofold anticorrosion mechanism of the developed MSN-BTA-Ag system for realization of active coatings; (Right) application of superhydrophobic nanoparticles for the realization of protective coatings

Combining the approaches above detailed, a very effective multifunctional coating can be designed, with a double layer structure: the inner layer in contact with the metal surface protects the metal surface with anticorrosion stimuli-responsive nanoparticles, while the outer superhydrophobic layer provides robust protection against water from environmental or intentional damage exploiting functional properties of Janus nanoparticles.

23.3 Gels for the Cleaning of Artworks

In addition to coatings, polymers and oligomers finds additional applications for CH practices as building blocks for gel development. Overall, polymeric gels represent some of the most advanced tools for cleaning practices, granting both optimum performances and controlled release of cleaning fluids on a surface of interest (Casini et al., 2023). Representing polymer-networks able of confining selected solvents or nanostructured fluids, gels can be obtained from polymers and monomers with different chemical identities and properties, resulting in hydrogels, i.e. capable of uptaking water solutions and dispersions, or organogels, i.e. capable of entrapping organic solvents (Bandelli et al., 2024b, c).

Traditionally, gels for CH practices are based on poly(acrylic acid) (PAA), polyacrylamide, poly(2-hydroxyethyl methacrylate) (pHEMA), polyvinylpyrrolidone (PVP), and poly(vinyl alcohol) (PVA), resulting in hydrogels and organogels with broad range of properties (Mastrangelo et al., 2020; Bandelli et al., 2024b, c). While such systems feature intriguing performance in CH practice there is still the need to design new gels capable to comply with “green” metrics, and, besides, to develop new protocol to estimate and to tune gel properties.

Representing gel systems capable of uptaking organic solvents, organogels enable the release of solvents that are traditionally employed in CH practices for the treatment of surfaces comprising pigments that are sensitive to water and, besides, to help the removal of unwanted organic layers such as coatings, and degraded patinas. Among the organogels employed in CH practices, systems based on the fully renewable and (bio)degradable castor oil (CO) are ideal candidates in CH practices, and can be obtained as polyurethanes (PU) without the use of solvents, employing reaction temperatures below 100 °C (Poggi et al., 2023). Such materials have been already employed for VOCs adsorption in the preventive conservation of museum enclosure but are in their infancy for remedial conservation aims and scope (Zuliani et al., 2022).

Representing a library of oligoesters with tailor made properties, such as logP, swelling behaviour in organic solvents and thermal properties, their use in CO organogels design can be beneficial to tune, and likely predict, the structure-property behaviour of the final materials. To this aim, a library of PU gels (Gx) was obtained from the reaction of CO, poly(hexamethylene diisocyanate) (pHDI), and Ox in a mass ratio of 72 to 18 to 10 in bulk at 80 °C (Bandelli et al., 2024b, c).

Already the study of the gelation process during the formation of PU a revealed that the use of Ox enabled a faster gelation time in comparison to the blank CO-PU system. Interestingly, the comparison between the gelation time over the clogP of Ox revealed that matching of Ox and pHDI clogP resulted in faster gelation. Moreover, DOSY NMR analysis of selected samples revealed that, at the beginning of the gelation process, Ox react faster with isocyanate in comparison to CO. Overall, both rheology and NMR analysis revealed that the gelation process is strictly correlated to the isocyanate reactivity, that is maximised when an additive matching the isocyanate hydrophobicity is employed. As a result, PU gelation in the presence of Ox determines the formation of Ox-enriched aggregates that facilitates the reaction with CO.

The final Gx polyurethanes featured micro- and nanoscale solid-like regions containing Ox species as depicted from small- and wide-angle X-ray scattering analyses, confirming the outcome of the reaction studies. The presence of oligoester aggregates resulted in a controlled variation of mechanical features at 25 °C as depicted from rheological analysis, resulting in an increase of both storage and loss modulus increasing oligoester's logP and, and consequently, Ox solid-like fractions.

Having accessed a new library of polyurethanes with tuned mechanical properties and featuring solid-like domains with well-defined HLB, solvent entrapment experiments were performed. As expected, the use of different oligoester additives with well-known logP in the PU design resulted in a controlled variation of the maximum of swelling behaviour. When solvents with lower logP in comparison with the oligoester additives, i.e. acetone and ethanol, were selected for swelling experiments, a linear decrease of the gel's maximum swelling was detected increasing the oligoester's additive logP (Fig. 23.3). Vice versa, the use of solvents with higher logP than the Ox species resulted in the opposite trend.

Insights on the process of solvent uptake were obtained from the Peppas-Salhin approach (Eq. 23.2), (Peppas & Sahlin, 1989) resulting in the estimation of the ratio between Case II transport, or material swelling, over Fickian transport, i.e. purely diffusive solvent transport in the PU matrix, R/F (Eq. 23.3).

$$\frac{m_t}{m_\infty} = k_F t^m + k_R t^{2m} \tag{23.2}$$

$$\frac{R}{F} = \frac{k_R}{k_F} t^m \tag{23.3}$$

where m_t is the mass of the PU-gel at time t, m_∞ is the maximum mass of the PU-gel obtained at the plateaux of swelling, k_F is the fickian constant, k_R is the Case II transport constant, and m is the purely Fickian diffusion exponent.

The results indicate that R/F followed the same trend depicted for the maximum swelling behaviour for solvents such as ethanol, acetone, methylethyl ketone, diethyl carbonate and p-xylene.

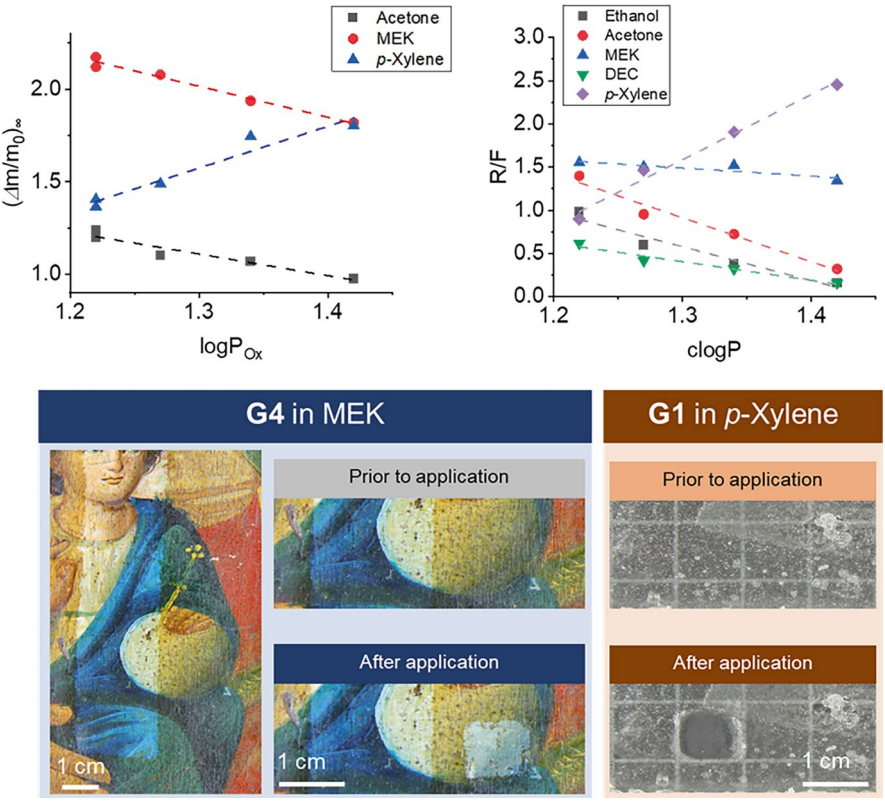

Fig. 23.3 Swelling behaviour and application of gels. Top left. Evolution of the maximum swelling capability over oligoester's logP ($logP_{Ox}$). Top right. Evolution of R/F ratio obtained from Peppas Salhin equation (Eqs. 23.2 and 23.3) over $logP_{Ox}$. Bottom. Cleaning tests using the CO-oligoester gels to remove varnish/coating from surfaces. The G4 gel was swollen in MEK and used to remove a polyhydroxy acid–based varnish from a painted wood panel. The G1 gel was swollen in p-xylene and used to remove beeswax from glass. The coated glass is lying on a slab with a grill that serves as spatial reference. Reproduced with permissions from D. Bandelli et al., *Chem. Sci.* **2024**, *15*, 2443–2455 and D. Bandelli et al., *Coll. Surf. A.* **2024**, *698, 134528* (Bandelli et al., 2024b, c)

The evaluation of the cleaning performances of PU-organogels was performed on ideal as well as real case study. In first instance, model mockups covered with wax (paraffin) or Paraloid B72 were treated with the Ox-based organogels that were previously swollen, up to doubling their mass, in p-xylene or acetone, respectively. The cleaning interventions resulted in a fast removal of both coatings in a fast, two applications of 10 min each, and controlled manner with minimum mechanical actions. At last, a byzantine icon case study covered with a degraded poly(hydroxy acids) varnish was treated with a selected gel swollen in methy lethyl ketone resulting in a prompt and effective varnish removal.

Overall, the development of new oligomeric species from renewable sources enabled the development of "green" castor oil polyurethanes with well-defined features, from structure, mechanical properties, and HLB behaviour. The tuning of such properties enabled the selective variation of solvent uptake mechanism and capability, making the new CO-PU materials ideal candidate for applications on surfaces exhibiting surface layers of hydrophobic varnish or organic degraded material.

Given the vast landscape of materials found in CH collections, cleaning practices based on gel do not solely aim for the removal of hydrophobic layers, but also on hydrophilic contaminants. In fact, already the accumulation of dust on CH objects results in the loss of the original surface appearance, requiring a prompt intervention. Nowadays, the use of PVA hydrogels represent the state-of-the-art materials for the removal of hydrophilic layers (Mastrangelo et al., 2020; Bandelli et al., 2024a). Such hydrogels exhibit optimal interfacial features, together with optimal micro- and nanostructural properties to carry out their function. To grant a uniform and fast cleaning action the porous PVA hydrogels must be able to embed cleaning fluids, allowing their diffusion at the interface and, at the same time, provide optimal roughness and adhesion. As a result, the structure-property of PVA hydrogels allow the applications of nanostructured fluids such as microemulsion, resulting in the removal of hydrophobic layers in substrates that are poorly sensible to water (Bandelli et al., 2024a). Recently, the role of gel's tortuosity, representing an evaluation of molecular diffusion in the gel matrix, has been introduced in the field of gels for cultural heritage preservation (Mastrangelo et al., 2024b). The tuning of such parameter has been obtained by means of varying the type of gel porogen, i.e. the additive that enables the formation of sponge-like interconnected cavity in the PVA cryogels, by varying its hydrophobicity (Mastrangelo et al., 2024a) Already the variation of the porogen structure, from linear to crosslinked, enabled the variation of mechanical properties, nanostructural features, and tortuosity of the final gels. Counterintuitively, the use of gel materials with high adaptivity in the removal of hydrophilic soil resulted in optimum performances when tortuosity increased, i.e. when molecular motions in the gel matrix were minimized, while gel adaptability on surfaces decreased. Overall, the study revealed that molecular motions of the solvent in the gel structure strongly impact the final applications, opening for additional development in the field of remedial conservation and beyond.

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