

## X-ray Methods For Assessing Desalination Efficiency and Reusability of Microcellulose Foams and Nanocellulose Aerogels in Mural Paintings

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One of the primary challenges in the conservation of mural paintings is salt crystallization. Traditionally, cellulose poultices have served as the main option for salt removal in mural paintings. However, in recent decades, there have not been excessive efforts to provide new advanced cellulose-based materials for desalination of mural paintings.

Cellulose-based foams and aerogels have attracted significant attention recently, thanks to their high adsorption efficiency, eco-friendly prospects and cost effectiveness. While they have been extensively employed for pollutant removal [1], there is no existing literature reporting their potential application in the desalination of mural paintings. Foams and aerogels present significant advantages over traditional cellulose poultices in terms of water retention and reusability due to their chemically crosslinked micro- and nano-structure, respectively. Therefore, their application can minimize prolonged water exposure to the porous substrate while enhancing the capacity for salt removal. Besides formulating novel desalination solutions for mural paintings, an essential aspect involves establishing an appropriate analytical methodology to evaluate their efficacy. Usually, ion chromatography is the preferred technique for quantitatively evaluating the salt removal capacity. However, being a destructive technique, it is not possible to use it to monitor in the same painting the removal of salts before and after the desalination treatment application. Furthermore, this technique cannot offer insights into changes in the spatial distribution of salts within the painting stratigraphy. An alternative to monitor salt removal non-invasively could be micro-energy dispersive X-ray fluorescence ( $\mu$ -EDXRF) imaging. However, using this technique, obtaining

information on the entire volume of a mural painting is not possible. Being able to directly monitor the removal of salts, and the evolution of their spatial distribution, without manipulating the sample would be essential to design-controlled protocols that avoid partial desalinization, the formation of salt fronts in the painting or even back diffusion problems..

For all the mentioned above, this work focuses on the validation of a new methodology based on  $\mu$ -EDXRF and X-ray micro-tomography ( $\mu$ -CT) to monitor, in the whole volume of a mural painting, the desalination ability of novel microcellulose foams and nanocellulose aerogels developed in the context of ENCLOSURE project [2]. To achieve this, replicas, mocking up ancient Roman wall paintings, were vacuum-impregnated with chlorides and sulfates, two of the main types of salts affecting mural paintings, to simulate salt attack. An experiment including both salts was also considered.

To monitor the desalination capacity of the foam and aerogel, each cellulose-based material was applied on a mock-up several times over a determined period. An additional experiment was conducted using traditional cellulose poultices to demonstrate the added value of the new materials proposed. Each cellulose-base material was applied several times to evaluate the progressive desalination. After each application, desalination rate was determined by  $\mu$ -EDXRF imaging and the salt removal on the whole volume of the mock-ups was evaluated using  $\mu$ -CT. For the successive applications of foams and aerogels on each mock-up, the same cellulose-based material subjected to a manual squeezing was employed to demonstrate their reusability.

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### **References**

- [1] Y. Ji, Y. Wen, Z. Wang, S. Zhang, M. Guo. Eco-friendly fabrication of a cost-effective cellulose nanofiber-based aerogel for multifunctional applications in Cu(II) and organic pollutants removal, *J. Clean. Prod.* 2020, 255, 120276; doi: 10.1016/j.clepro.2020.120276
- [2] ENCLOSURE project: <https://enclosure-project.eu/>