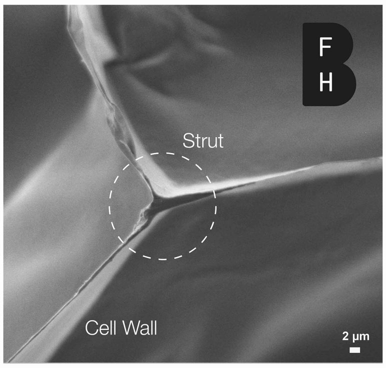
Adhesive Foams – A First Insight

Karolina Soppa, Magdalena Ritler and Nadim C. Scherrer

Bern University of Applied Sciences, Berner Fachhochschule (BFH)
Bern University of the Arts, Hochschule der Künste Bern (HKB)
Fellerstrasse 11, 3027 Bern, Switzerland
Contact: karolina.soppa@bfh.ch



Introduction

Many contemporary or antique (for example, Egyptian) paint flakes on canvas are completely loose and the canvas is very absorbent. Adhesives with low viscosity penetrate porous materials in an uncontrolled manner and form an inadequate and insufficient adhesive layer **[1]**. Even when using a highly viscous adhesive like methylcellulose A4M 4% (~100'000 mPas), penetration of an absorbent canvas will be substantial **[Fig. 1A]**. Current solutions to overcome these problems are the application of dry and solid adhesive films or grids that can be reactivated **[2]**. A new approach to achieve a thin adhesive layer on absorbent substrates is the reactivation of dry foams from pure, ageing-resistant adhesives often used in conservation, like methylcellulose. Two foam production methods are presented: freeze-drying (FD) and whipping with subsequent oven-drying (W-Foam).
The requirements for the foam are as follows: uniform, dimensionally stable, light, open-cell and thus fast reactivatable, so that adhesion can take place without uncontrolled penetration of the adhesive as well as the reactant into absorbent substrates with the lowest possible material input.

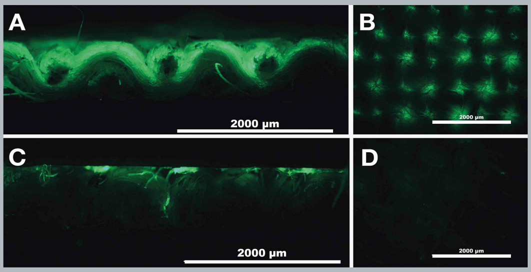


Fig. 1 Bonding of non-absorbent alkyd paint to a very absorbent, unsized canvas. **A:** Cross section: A4M 4% in H_2O with fluorescein sodium was used as adhesive. The canvas is clearly visible due to the fluorescein, which A4M could not hold back. **B:** Back: It is visible that the methylcellulose is not only between the threads, but also slightly in the threads around the cavities. **C:** Cross section: The reactivated W-foam, also mixed with fluorescein-sodium, is exactly where it should be: between the canvas and the paint layer. The canvas is black because no dye has penetrated. **D:** No fluorescence is visible at the back. Thus, the desired bond was achieved without penetration of the canvas.

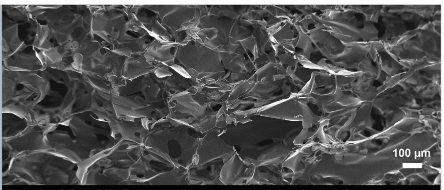


Fig. 2 SEM-SE: Freeze-dried foam. A4M 1% in H_2O. Mostly closed-cell with a cell wall of 1.0±0.05µm between the struts (~100µm²) as well as some open-cell sections are visible.

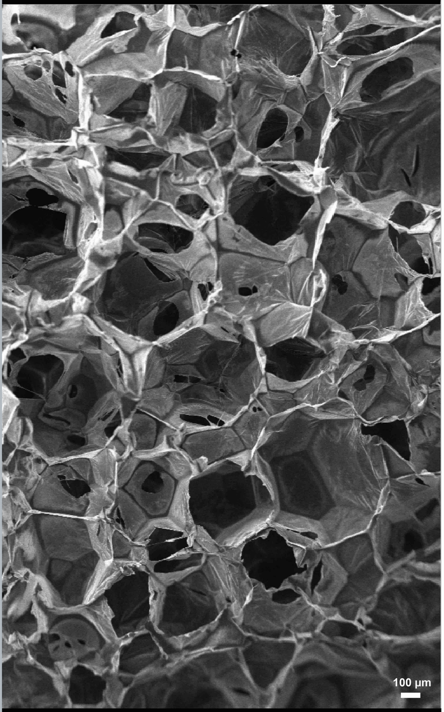


Fig. 3 SEM-SE: Cold whipped, heated and oven-dryed foam. Mostly closed-cell with a fine cell wall of 0.28±0.05µm between the struts (~40µm²) as well as windows are visible.

Materials and Methods

Foam production: Foams were produced from the following adhesives: Hydroxypropylcellulose (HPC) Klucel E and M (Ashland), Hydroxypropyl-methylcellulose (HPMC): E3 and E5 (Dupont), methylcellulose (MC): A15 (Dupont), A4C, A4M and A40M (Ashland) and gelatin type A, bloomgrade 180 and 240 (Carl Roth GmbH).
Freeze-drying parameter: -40°C, 0.02mbar.
Refrigerator: -20°C. Logger during freezing: Testo 177-H1 with external sensor (measuring interval: every minute).
Fluorescein sodium in H_2O was added to the solutions (10µl of a 10% solution per 100ml).
Whipping was performed with two-piece whisk foam attachment with 3140±94 revolutions per minute. A convection oven was used for drying (Memmert: 85°C and 20% air circulation).
Characterization of the foam: Sections of the foams with a disposable razor blade were characterised first using fluorescence microscopy (filter set: BP 475–495, LP 510, BP 512–542) and second with electron microscopy. SEM-SE: Zeiss Evo Ma10 (W-cathode). Images were acquired at 3.5kV and 20pA using an detector in high vacuum. Samples were coated at multiple angles with a carbon evaporation coater.
Characterization of penetration and adhesive strength: Test samples for penetration and tensile strength testing were prepared and conducted as described in Soppa et al. (2014).
Before bonding, the paint flakes were cleaned with isooctane. Tensile strength testing was carried out according to Soppa et al. (2014) with a Zwick 1120 (Zwick GmbH & Co. – testing speed was 1 mm/min). For each test series 10 samples were tested. All results were recorded in N/cm² and presented with calculated averages and confidence intervals of 95%.

Results and Discussion

Almost all requirements were met by the A4M solution (1%), which was foamed cold (RT), then heated to 55°C and dried in an oven (85°C) with circulating air (20%) - called (whipped) W-foam.
However, the freeze-dried foam of A4M 1% was superior to the W-foam in terms of regularity of the bubble size (209±47µm versus 361±82µm) **[Figs. 2 and 3]**. Thus, the W-foam has a slightly higher polydispersity (PDI: 0.35 versus 0.3 for the freeze-dried foam). Both foams exhibit closed and open pores, although the W-foam appears to be more open.
The W-foams have a four times thinner cell wall (1.0±0.1µm) and half the strut (~100µm)² compared to the freeze-dried foams - cell wall (0.28±0.05µm) and strut (~40µm², figure in the header). In addition, the W-foams with the larger bubble size and the open pore structure are less dense permitting faster reactivation and resulting in high suitability for bonding.
Fast **reactivation**, good wetting and fast drying properties have been achieved with a 1:1 mixture of H_2O and EtOH.

The **tensile strength** of the reactivated A4M W-foam on an unsized canvas is 12.4±2.2 N/cm², which is almost the same as the adhesive strength of an A4C 5% on an sized (!) canvas with an alkyd paint layer 13.7±2.3 N/cm² **[1]**. This is quite astonishing, considering that a gelatin does not stick at all under the same conditions as the foam. Moreover, the reactivated foam does not penetrate into the canvas **[Figs. 1C and D]**.
However, it should not be neglected that an A4M with 4% has a higher tensile strength (22.5±1.4 N/cm²) due to a larger, more effective adhesive area. Nevertheless, the compromise of the higher bond strength is the partial penetration of the absorbent material. This impedes later removal, while the adhesive foam forms a well removable, fleece-like film.
A note on the production efficiency: While the production via freeze-drying of a uniform MC-foam requieres 17h, the production of W-MC-foam (depending on the foam height) took only approx. 1-4 hours.

Conclusion and Outlook

A reactivatable adhesive foam was successfully produced. It was shown that each manufacturing method produces a different quality of foam. In addition, each adhesive has different manufacturing and reactivation requirements, which in turn control the adhesive strength. All results will be published in the upcoming paper.
Together with one of our future research partners *docusave* we want to produce reproducible and commercially available foams for bonding but also e.g. for filling as well as strip lining **[3]**.
We are also working on an application of wet methylcellulose foams and their drying on original works of art. A publication on this topic and a workshop can be expected in early 2023.

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Literature: **[1]** Soppa, K., T. Laaser, C. Krekel, M. Genton, and T. Seidel. 2014. Adhesion and penetration of sturgeon glue and gelatines with different Bloom grades. In ICOM-CC 17th Triennial Conference Preprints, Melbourne, 15–19 September 2014, ed. J. Bridgland, art. 1313, 9 pp. Paris: ICOM. **[2]** Konietzny, M, K. Soppa and U. Haller. 2018. Canvas Bonding with Adhesive Meshes. Poster. In: Consolidation and Communication. Hildesheim, 25.–27. January 2018. https://www.hornemann-institut.de/de/epubl_poster49.php, 20.06.2022. **[3]** Bründler, S., K. Mürer and S. Weber. 2019. Conservation and restoration of "Falsche Götzen" (False idols) of Fischli/Weiss at the Kunsthaus Zürich, CH. https://www.kunsthaus.ch/sammlung/restaurierung/fischli-weiss/, 20.06.2022.