

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration

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Keywords

methylcellulose, ether, foaming, conservation,
restoration, filling, bonding

Abstract

This paper explores the foaming of methylcellulose (MC) using straightforward foaming and drying methods for different applications in the field of conservation and restoration. The aim was to elaborate a low-tech, inexpensive and reproducible foaming method to achieve dimensionally stable, homogeneous, dried foams. Crucial influencing parameters of the foaming process such as viscosity grade, concentration, water temperature, mixing device, whipping time and drying method were investigated. Dried foams were produced with MC A4C 5–7 wt% and A4M 4–6 wt% and then characterized by comparing them with industrial polyethylene foams, as reference material, with respect to their cell size and indentation hardness. The tested samples consisted of heterogeneous cells between 0.3 and 2.3 mm in diameter, and the hardness was 1.2–15.1 N/cm². Possible areas of application of dried MC foam in the field of conservation-restoration, e.g. as a filling material, are explored within a case study.

INTRODUCTION

Methylcellulose (MC) ethers, specifically the viscosity grades A4M and A4C, have been increasingly used in conservation since the mid-20th century, due to their physicochemical properties, aging stability, and long-term reworkability (Feller and Wilt 1990, Steger et al. 2022). The typical applications of MCs are as adhesives, consolidation or retouching media, thickeners, and gelling agents for surface cleaning (Baker 1984, Haller 1995, Hoppmann and Schubert 2005, Horie 2011, Soppa 2018). Recently, MC as adhesive meshes has been researched as well (Konietzny et al. 2018). MC as a foamed material is relatively new in conservation and its utility is a promising field of study, in line with the inexhaustible and creative use of materials in art and cultural heritage, which requires the ongoing development of conservation materials.

The authors' first MC foaming experiments date back to 2012, when Bunz produced aqueous MC foams as part of a student project at the Modern Materials and Media Conservation workshop at the Bern Academy of the Arts (HKB). Basic tests were conducted with Methocel A15LV, A4C, and A4M (Bunz 2013). The initial idea was to use MC foam to compensate for losses in the joining parts of an expanded polystyrene relief artwork with an acrylic paint layer. The application of liquid 8–10 wt% Methocel A4C foam on a mockup caused undesired shrinkage and the drying time was excessive, ruling out its use as a viable material in this case. However, these preliminary tests were the starting point for further investigations into applications of dried MC foam as a filling material for artworks made of rigid polyurethane (PU) foam (Bründler et al. 2019). Other applications of light, fast reactivable, oven- or freeze-dried foams have recently been investigated by Soppa et al. (2022). Furthermore, the application of small-dose liquid MC A4M foam as an aqueous adhesive and filling material for flaking paint layers was explored by Ritler in her master's thesis (2023). The liquid foam is produced using the double-syringe technique described by Schad et al. (2021).

MC as a foaming agent has applications in several different areas and has been the subject of scientific study (Hu et al. 2016, Gordeyeva 2018).¹ However, the foams described in those reports often included additives, were prepared as blends or required extensive lab equipment. The main objective of the present study was to develop a low-tech, inexpensive foaming and drying method that could be easily reproduced by conservators in their

SCIENTIFIC RESEARCH

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration

studios. The foams should be dimensionally stable and homogeneous upon drying. Additionally, it should be possible to adjust the recipe, without the need for additives, in order to produce foams with properties ranging from soft and flexible to firm and rigid, thus facilitating a multitude of potential uses.

Over a period of one year, the authors conducted several foaming tests with MC A4C and A4M. To enable reproducibility, crucial influential parameters of the foaming process were investigated, including MC type, manufacturer and batch, two different mixing devices, solid content, whipping time, and water temperature as well as the drying support and drying method.

MATERIAL AND METHODS

Following the results of our preliminary tests,² MC foam test samples were produced with 5–7 wt% or 4–6 wt% solid content in deionized water for MC A4C (Benecel A4C, Ashland: batch A and B; Methocel A4C, DuPont) and MC A4M (Methocel A4M, DuPont),³ respectively (Table 1). The MC powder was first stirred with 60 °C deionized water (total volume of 100 mL) for a few seconds until the powder was wetted, followed by mixing at 55 °C at full speed using either a Braun MultiQuick 7 mixer with the whisk-attachment (1080 rpm, 1000 W) or the cordless Norpro 2273 Mini Mixer with the stir-attachment (2000 rpm, two AA batteries). The revolutions per minute (rpm) obtained with the mixers were tested with a digital hand tachometer (Testo 470). The corresponding Braun plastic measuring cup (600 mL, ø 97 mm, H: 17 cm) served as a mixing container. The temperature during foaming was measured using an IR thermometer at intervals of 30 s (Votcraft IR 500-12S, emissivity of 0.95). Depending on the MC type and solid content, the mixture was stirred for 1–5 min. After the specific mixing time, the consistency of the foam was similar to that of whipped cream and was quickly scooped onto the drying support (Hollytex 3268 on a stretcher).⁴ The foam was air- or oven-dried in tablespoon sized heaps (for pretests) or lanes of 2 × 2.5 cm (w × h) to obtain the required sample material for use in compression tests. The foam was air-dried at 23 °C (RT) and 50% relative humidity (RH) for three to seven days depending on the sample size.

Table 1. Materials list

Trade name (Brand), Material	Type & grade	Viscosity at 2 wt% (RT)	Batch	Supplier
Benecel (Ashland), Methyl-cellulose	A4C	~400 cP	“A”	Unknown
	A4C	339 cP	“B”: 2559037	Chemical Marketing Concepts Europe, Waalwijk (NL)
	A4M	5222 cP	“B”: 2593954	
Methocel (DuPont), Methyl-cellulose	A4C industrial grade	~400 cP	512M411A11	Nutrition & Biosciences Switzerland GmbH, Lucerne (CH)
	A4M industrial grade	~4000 cP	unknown	Kremer Pigmente GmbH & Co.KG, Aichstetten (DE)
Ethafoam EF 220, Polyethylene foam				FoamPartner, Gontenschwil (CH)
Flexathen FT/PZ 29, Polyethylene foam				
Stratocell S, Polyethylene foam				
Hollytex 3265, Polyester fleece (81.4 g/m²)				Deffner & Johann, Rötthlein (DE)

SCIENTIFIC RESEARCH

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration

EXAMINATION OF PROPERTIES**Visual examinations**

The foams were photographed before and after drying to evaluate shrinkage. The cell structure of the dried foams, as seen in thin sections (cut with a razor blade), was examined using a Leitz DMRB microscope. Images were generated using blue-light excitation combined with the I3 filter set (BP 450-490 LP 515), a 2.5× objective, and microscope camera system (Jenoptik ProgRes Speed XT Core 3, software ProgRes Capture Pro 2.10). The structure of the dried foam samples (carbon evaporation coated) was also visualized by scanning electron microscopy (SEM, Zeiss Evo MA10, operated at 3.5 kV and 20 pA). The cell diameter and wall thickness of the foam were evaluated using Adobe Photoshop based on the light and electron microscopy images.⁵

Mechanical properties

Compression tests on selected test series were carried out on the Zwick 1120/TNS1 (software testXpert). The test parameters were selected according to ISO 2439:2008 Method A for the determination of the indentation hardness of flexible cellular materials.⁶

For each series, seven foam samples were cut ($6 \times 10 \times 10$ mm) and then pre-conditioned using saturated salt solutions at RHs of 33% (calcium chloride), 75% (sodium chloride), and ~50% (=room climate). The polyethylene (PE) foams Ethafoam EF 220, Flexathen FT/PZ 29, and Stratocell S were tested as reference materials. The results of the mechanical tests were recorded in N/cm² and are presented as the calculated averages and standard deviation.

RESULTS AND DISCUSSION**Hot foaming technique**

The foaming method applied in this study takes advantage of the insolubility of MCs in hot water (Dow 2002, 12). When MC powder is directly mixed with hot water, a lump-free dispersion is produced. Upon initial cooling during the foaming process, the MC hydrates and starts to dissolve, increasing the viscosity of the liquid (Ibid.). In this study the ideal water temperature when mixing MC with a 4–6 wt% solid content was 60–55 °C. The temperature of the ready-to-use MC foam when scooped onto the drying support and then measured with the IR thermometer was 35–42 °C. As the foam continues to cool below these temperatures, its increasing viscosity causes it to become lumpy and further processing is then difficult. The time window for the ideal processing of the ready-to-use foam decreases with increasing MC concentration.

Mixing MC directly with water at a temperature higher than its solubility temperature (hot foaming) allows for a higher binder concentration than is possible with mixing already-dissolved, highly viscous MC solutions (cold foaming). A ready-to-use MC solution above 4 wt% at RT could not be whipped using the tested household mixers and would require more elaborated equipment, while lower concentrations did not produce the hardness required for use in this study.

SCIENTIFIC RESEARCH

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration

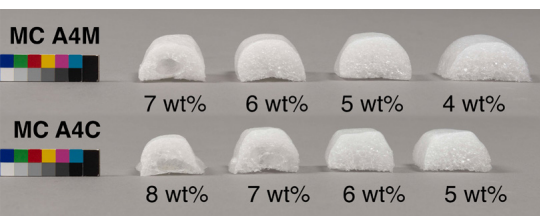


Figure 1. Air-dried Methocel A4C and A4M test samples (S. Bründler and S. Bunz)

Viscosity grade, concentration, and batch

Preliminary foaming tests with 1–12 wt% MC A4C (Benecel A4C, Batch A) and 1–10 wt% MC A4M (Methocel A4M) showed that concentrations below 3 wt% resulted in irregular, dimensionally unstable or flat foams. Foams above 7 wt% were more irregular and stiff and cavities formed in their centers. Air-dried foams prepared with 5 and 6 wt% MC A4C as well as A4M 4 and 5 wt% were more homogeneous, form-stable, and flexible (Figure 1). A significant difference was observed in the two tested batches of Ashland Benecel A4C after drying: While they behaved identically during foaming, batch B dried to a flat, irregular film. Also, the hardness achieved with MC products of the same viscosity grade obtained from two manufacturers, Ashland (batch A) and DuPont, differed significantly (see indentation hardness below). The reason for the discrepancy in the results has not yet been fully clarified and will require further research and material analysis.

Mixing device and stirring duration

Both tested mixing devices produced positive results. For concentrations of 5 and 6 wt% (both viscosity grades), the optimal stirring duration was 2 min and 30 s using the Braun mixer and 4 min and 30 s using the Norpro Mini Mixer. The whipping duration is strongly linked to the binder concentration, water temperature, and mixing device used. The stirring duration needed to achieve a form-stable foam was longer with lower than with higher MC concentrations. Over- or under-whipping the foam will cause it to be too liquid or too lumpy. Variables such as the whipping time, rpm of the device, the chosen whisk attachment, and the insulation capability of the mixing container can also impact the temperature of the mixture and the foams' properties.

Drying method

The average shrinkage of the samples after drying was around 40%. With MC concentrations above 7 wt%, cavities formed in the bottom center of the foams upon air-drying. The tendency to form cavities increased if the foams were piled higher than 2.5 cm. Cavity formation seemed to occur in areas that took longer to dry and thus favored the collapse of the foam structure (Figure 1). To reduce cavity formation and increase the homogeneity of the foam, the drying process should be accelerated. Tests with oven-drying (SalvisLab Thermocenter) for 12 h at 50, 70 and 85 °C without convection were thus carried out but did not produce the desired outcomes. Oven-drying was only successful when the foam had cooled for at least 1 h and the MC was more dissolved; otherwise, flocculation occurred. Further testing is needed to determine the parameters (e.g., ideal temperature and RH) enabling oven-drying. Other drying methods might also be promising. In fact, freeze-drying was tested successfully, resulting in homogeneous, fine-pored, soft foams produced using MC A4M solutions of 1 wt% (Soppa et al. 2022), but this drying technique is less sustainable and more expensive and was not further considered.

Cell structure

The cell size distribution of the samples was quite heterogeneous (Figure 2). In the dried test samples produced with the Braun mixer, the cell sizes

SCIENTIFIC RESEARCH

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration

varied between 0.3 and 2.3 mm. The cells obtained with Benecel A4C 6 wt% had a mean size of $915 \pm 386 \mu\text{m}$, with cell walls of $2.7 \pm 1.7 \mu\text{m}$, and were the most homogeneous in terms of their size distribution; Methocel A4C 6 wt% produced cells measuring $1089 \pm 221 \mu\text{m}$, with thinner cell walls ($0.8 \pm 0.19 \mu\text{m}$). The cell sizes obtained with Methocel A4M 5 wt% and 6 wt% were quite similar, $1059 \pm 213 \mu\text{m}$ and $1069 \pm 235 \mu\text{m}$, respectively, and Methocel A4M 4 wt% resulted in the largest cells, with an average size of $1120 \mu\text{m}$ and also the largest size deviation ($\pm 457 \mu\text{m}$).

The SEM images of the products obtained with MC 6 wt% show mostly closed cell structures but also some open cells (Figure 3). Overall, the MC foam samples were more heterogeneous than the industrially manufactured

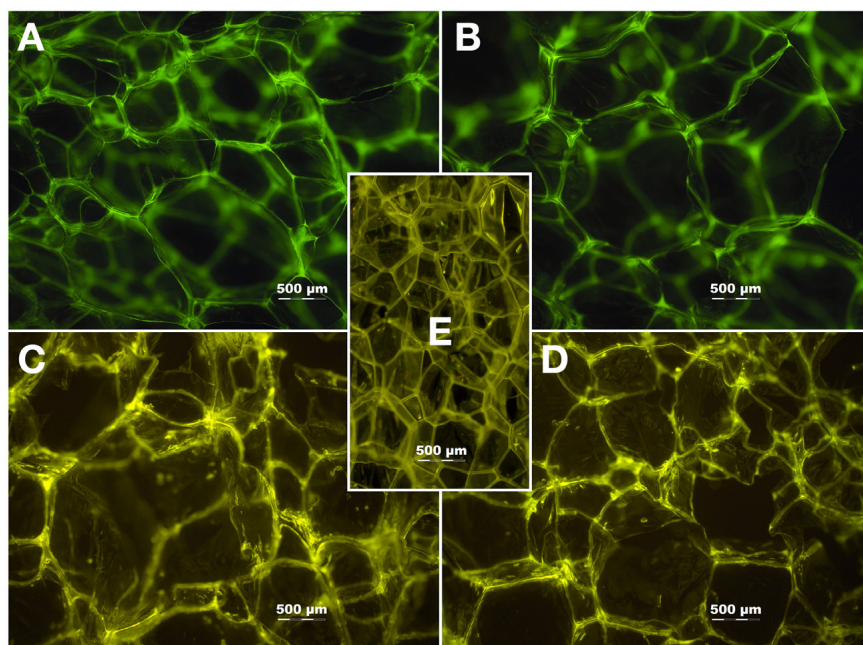


Figure 2. Microscope images (2.5× magnification) of air-dried MC foams: (A) Benecel A4C 6 wt%; (B) Benecel A4C 5 wt%; (C) Methocel A4M 4 wt%; (D) Methocel A4C 5 wt%; and (E) reference material Flexathen FT/PZ 29 (S. Bründler and S. Bunz)

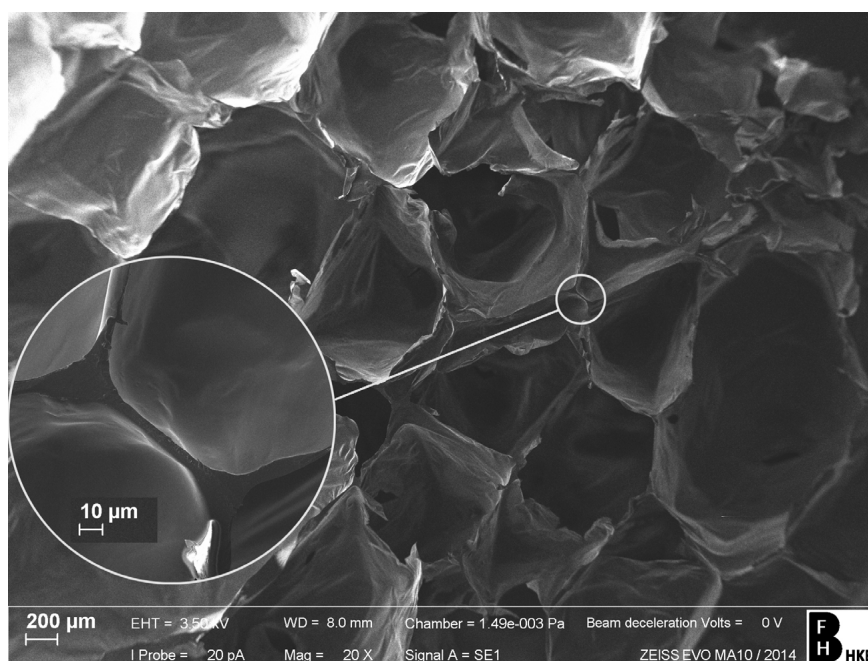


Figure 3. SEM macrograph (magnification 20x3) of air-dried Methocel A4C foam 6 wt% (N. Scherrer)

SCIENTIFIC RESEARCH

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration

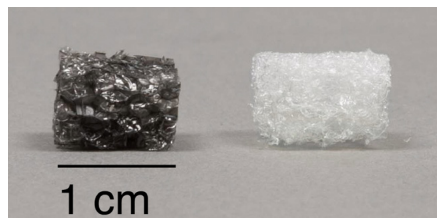


Figure 5. Stratocell S reference material and dried Benecel A4C 5 wt% foam test sample (produced with the Braun mixer) (S. Bründler and S. Bunz)

PE foam references, which were made up of either mostly closed (Flexathen FT/PZ 29) or mostly open (Ethaf foam EF 220) cell structures and cell sizes ranging from 0.3 to 2 mm.⁷ A comparison of the results obtained with the freeze-dried MC foams of 1 wt%, described by Soppa et al. (2022) (cell size: $209 \pm 47 \mu\text{m}$; cell wall $0.28 \pm 0.05 \mu\text{m}$), showed that the cell sizes achieved in the present test series were several times larger, with thicker cell walls, which might influence the foams' hardness, as described in the next section.

Indentation hardness

Unless otherwise noted, the following results were acquired at RT and 50% RH. The indicated indentation hardness always refers to the average of the tested seven samples per series (Figure 4).

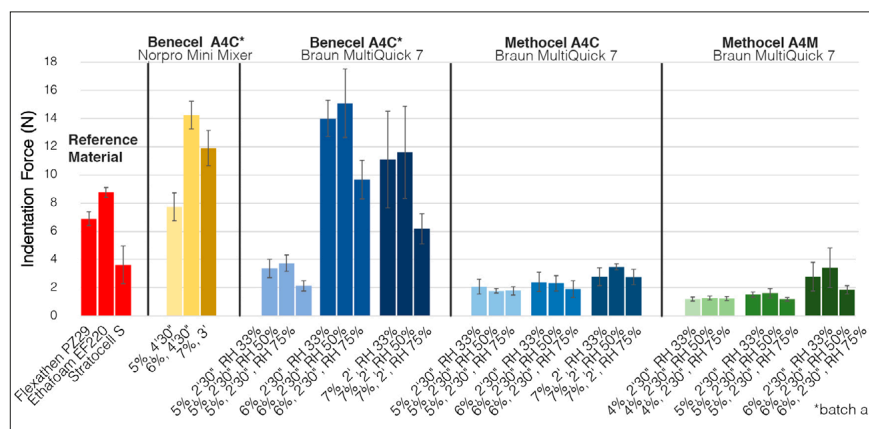


Figure 4. Overview of the indentation hardness values (S. Bründler and S. Bunz)

The indentation hardness of 5 wt% A4C Benecel (batch A) mixed for 2 min and 30 s ($3.75 \pm 0.58 \text{ N/cm}^2$) using a Braun mixer was similar to that of the tested reference material Stratocell S ($3.62 \pm 1.34 \text{ N/cm}^2$) (Figure 5). The same material at the same concentration but mixed with the Norpro Mini Mixer for 4 min and 30 s had an indentation hardness ($7.73 \pm 0.98 \text{ N/cm}^2$) between that of Flexathen FT/PZ 29 ($6.9 \pm 0.48 \text{ N/cm}^2$) and Ethaf foam EF 220 ($8.75 \pm 0.35 \text{ N/cm}^2$). The compressive strengths of samples with MC > 5 wt% were harder than those of Ethaf foam EF 220. Samples prepared with Benecel at a concentration > 6 wt% did not exhibit the expected exponential increase in compressive strength. Methocel A4C 5–7 wt% mixed with the Braun mixer produced foams that were much softer ($1.81\text{--}3.48 \text{ N/cm}^2$) than those of the corresponding Benecel A4C samples ($3.75\text{--}15.08 \text{ N/cm}^2$). At lower viscosities, the indentation hardness values achieved with A4M 4 wt% ($1.26 \pm 0.14 \text{ N/cm}^2$) and 5 wt% ($1.6 \pm 0.34 \text{ N/cm}^2$) were similar to the hardness achieved with Methocel A4C 5 wt% ($1.76 \pm 0.17 \text{ N/cm}^2$) while the indentation hardness produced by A4M 6 wt% ($3.42 \pm 1.4 \text{ N/cm}^2$) and A4C 7 wt% ($3.48 \pm 0.21 \text{ N/cm}^2$) was similar.

The indentation hardness at different RHs was compared and showed that the Benecel foams were the most stable at 50% RH, with a decrease in the maximum of 1 N/cm² at 33% and 5.4 N/cm² at 75% RH. The differences observed within the Methocel samples were minor.

SCIENTIFIC RESEARCH

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration

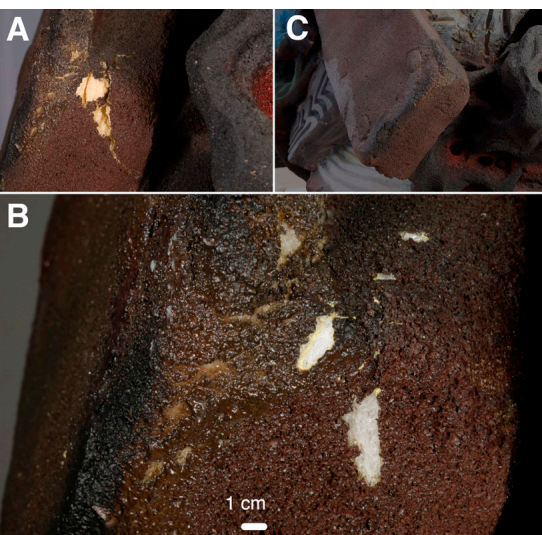


Figure 6. (A) Losses and cracks in the rigid PU foam sculpture, before treatment; (B) dried MC foam inlays before filling and retouching; (C) after treatment. Kunsthhaus Zürich

CASE STUDY AND FURTHER APPLICATIONS

Dried MC foam as a filling material was investigated and applied to stabilize deep cracks and losses in the large-scale, PU rigid foam sculpture *Falsche Götzen* (1983), by the artist duo Fischli/Weiss, at the conservation studio of the Kunsthhaus Zürich (Bründler et al. 2019). The two-component PU-ether foam shows a broad range of cell sizes and densities throughout the sculpture, as the material was not industrially manufactured. Dried MC foam was considered a promising filling material, as its hardness could be customized during production, but also because of its interesting properties for finishing (e.g., cutting, shaping, and reactivation). The MC foam that was finally used⁸ was produced by dispersing 8–10 wt% MC A4C in hot water at 75 °C using a magnetic stirrer until the temperature of the mixture reached 55 °C. Foaming was achieved using the Norpro Mini Mixer with the stir-attachment. When the desired consistency was reached, the foam was air-dried in small heaps before it was cut into suitable shapes with a scalpel. Due to the flexibility of the MC foam, the inlays could be well fitted into the cavity (Figure 6). As the sculpture was painted with acrylic dispersion paint, a thin layer of a MC-based filler was applied and structured using a syringe, with retouching accomplished using pigments bound with gum arabic. Both the filler and the retouching were applied rather dry in order to not affect the underlying MC foam.

Further applications of MC foams in the field of conservation and restoration can be envisioned due to their versatile properties: Liquid or dried MC foams can serve as an adhesive and simultaneously as a light-weight filling material; dried foams can be used as a gap filler or interim support; thin slices of dried foam could be reactivated, as described by Konietzny et al. (2018) for adhesive meshes. Additionally, dry MC foam could be combined for application with small amounts of liquid MC foam.

CONCLUSION

The MC foams produced with our low-tech hot foaming method hold great promise as they are a highly customizable material with great versatility. However, variables such as the MC concentration, water temperature, mixing device, whisk attachment, mixture volume, or container could influence the properties of the foams, including their hardness, the homogeneity of the cell size distribution, and the dimensional stability. The drying support and conditions as well as the heap size will also influence the results. The largest differences in the hardness and mechanical properties were not only between foams derived from MCs from different manufacturers but also between the MC batches tested. Therefore, each new batch of material should be assessed with a small test series to estimate its foaming properties. In addition to a systematic test series of the MC concentrations, the optimal stirring time should be determined starting with a MC concentration of 5 wt%, scooping out a spoon of foam every 30 s while mixing (Figure 7). We encourage experiments focused on other parameters in order to gain tactile knowledge. Similar to cooking, practice is needed to identify when the foam is ready and firm enough for the desired application.

SCIENTIFIC RESEARCH

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration



Figure 7. Undried Methocel A4M foams. Pretest series with different concentrations (from left to right: 1–7 wt%) (S. Bründler and S. Bunz)

FURTHER RESEARCH

Continuing studies are needed, aimed at further improving the homogeneity and hardness of the MC foam, especially at higher concentrations. This could be achieved by modifying the drying process and by adding fillers such as cellulose fibers or other cellulose material like CNC nanocellulose, as examined by Hu et al. (2016). Besides other MC types, blends of different MC grades could be promising. With the use of more elaborate techniques, the mixing of high-viscosity ready-to-use solutions using more powerful mixing devices, as well as extrusion foaming and hot molding, studied by Karlsson et al. (2016 and 2015) with hydroxypropyl MC, merit consideration.

In some specific cases, it might be desirable to add pigments or dyes to the foam, but this should be preceded by systematically studying the impact of additives on the foaming properties. As mentioned, further applications of liquid and dried MC foams in conservation restoration should also be pursued.

ACKNOWLEDGMENTS

The authors are grateful to Ashland and DuPont for providing sample material. We further thank Dr. Jamie Waterman for his invaluable help with translation and proofreading. Thanks also go to Dr. Zuzana Sediva from Groam Tech as well as Agathe Jarczyk and Marc Egger from Atelier Konserve for their support.

NOTES

- ¹ Among other applications, MC is used in the food and cosmetics industry not only as a binder, thickener, and stabilizer, but also as a foaming agent (Dow 2002, Dow 2005).
- ² In pretests of a wider range of MC A4C and A4M concentrations (up to 12 wt%), both cold foaming of ready MC solutions and hot foaming techniques were tested.
- ³ Commercial products are specifically named throughout this paper. This does not imply that other products are not suitable for the same purpose.
- ⁴ The drying support was developed in pretests. The tested materials were: a glass plate, disposable paper baking cups, a silkscreen frame, Hollytex 3257, and Hollytex 3268.
- ⁵ Foam cell and cell size were defined according to Oosten (2011, 118). Measurements were made using two-dimensional images and are therefore only points of reference and not the exact values of the diameters or three-dimensional geometries of the cells.
- ⁶ Preload 0.5 N and testing speed 100 mm/min. The main parameter deviating from the standard was the small sample size. The ISO standard and parameters were chosen during our first test series and later retained to allow comparisons with our following test series.
- ⁷ The cell sizes measured in the reference material Stratocell S ($1600 \pm 185 \mu\text{m}$, cell wall $4.64 \pm 2.33 \mu\text{m}$) were almost three times larger than those measured in Flexathen FT/PZ 29: (cell size $671 \pm 172 \mu\text{m}$) or Ethafoam EF 220 ($1523 \pm 148 \mu\text{m}$).

SCIENTIFIC RESEARCH

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration

⁸ The hardness of the dried MC foam was presumed to be close to that of the Benecel (batch A) A4C 6 wt% foam, produced using the Mini Mixer for 4 min and 30 s ($14.2 \pm 0.97 \text{ N/cm}^2$), but was not analyzed.

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SCIENTIFIC RESEARCH

Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration

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To cite this article:

Bründler, S., S. Bunz, M. Ritler, N.C. Scherrer, and K. Soppa. 2023. Dry methylcellulose foams: Investigation of simple foaming and drying methods for applications in the field of conservation and restoration. In *Working Towards a Sustainable Past. ICOM-CC 20th Triennial Conference Preprints, Valencia, 18–22 September 2023*, ed. J. Bridgland. Paris: International Council of Museums.