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American Institute for Conservation
727 15th Street NW, Suite 500
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RACHEL MOCHON

Nanocellulose in Practice: Properties of Microfibrillated Cellulose and Cellulose Nanocrystals

INTRODUCTION

Nanocellulose is a term encompassing several highly refined cellulosic materials prepared by mechanical and/or chemical processes to isolate the smallest part of the cellulose fiber. Within the past decade, paper conservators have begun to study this material for its sustainability, strength, transparency, and chemical stability. It has also been tested and incorporated into many industries, including medical, food, cosmetics, and electronics. More familiar applications of nanocellulose outside of the conservation field include dermal poultices, the SCOBY or mother used in fermentation of kombucha, and Nata-de-coco, an authentic dish from the Philippines consisting of bacterial cellulose from the fermentation of coconut water.

There are three main types of nanocellulose: microfibrillated cellulose (MFC), cellulose nanocrystals (CNC), and bacterial cellulose (BC). Cellulose can be prepared from multiple sources, including wood, cotton, tunicate, algae, and bacteria (Dufresne 2012, 3). Both MFC and CNC, however, are primarily prepared from plant matter. The terminology is highly inconsistent for these three kinds of nanocellulose. The following terms listed in Table 1 are used to describe the same product by various sources. Distinguishing between the three types is essential, as they have very different chemical and physical characteristics.

Microfibrillated cellulose (MFC) is likely the most common nanocellulose currently being tested within the field of conservation. The diameter of the fibers can range from 5–100 nm (Völkel et al. 2017, 2), and lengths range from 1–10 μm (Dreyfuss-Deseigne, 2017a, 22). As such this form of nanocellulose is known as both “microfibrillated cellulose,” referring to the length of the fibrils on the micrometer scale, or synonymously “nanofibrillated cellulose,” referring to its width on the nanometer scale. To produce MFC, a wood or plant-based pulp may first undergo chemical, enzymatic, or mechanical treatments to purify the cellulose and remove impurities, such as hemicellulose and lignin (Dreyfuss-Deseigne 2017a, 21). Next, a heavy mechanical shearing process fibrillates the fibers, and the microfibrils are isolated (Dreyfuss-Deseigne 2017c, 110). The resulting gel or slurry contains both crystalline and amorphous cellulose.

The second type, cellulose nanocrystals (CNC), differs from the first type primarily in that it does not have the amorphous region of the cellulose, but only the crystalline portion. It is prepared by acid hydrolysis, using primarily solutions of hydrochloric or sulfuric acid, which break down the amorphous regions and leave behind the rod-like crystalline structure (Habibi, Lucia & Rojas 2010, 3483–4). The fiber width ranges from 3–25 nm, while the length is smaller than MFC, ranging from 0.1–2 μm (George 2015, 49).

Bacterial cellulose (BC) can be grown from many types of bacteria, but the *acetobacter* species is the only type to produce enough cellulose for commercial purposes (Dufresne 2012, 125). The bacteria is cultured in a medium, including sucrose and yeast-extract, in a lab over time. A pellicle forms on the surface of the medium and thickens, as the cellulose grows. The cellulose can then be rinsed and purified, removing the bacteria (Iguchi, Yamanaka, and Budhiono 2000, 263). The resulting fiber width ranges from 70–130 nm, and the length ranges from 1–20 μm (Dufresne 2012, 127). Of the three types of nanocellulose, BC has the highest degree of purity because there are no plant-based impurities (Dufresne 2012, 125).

CURRENT RESEARCH

In 2017, Rémy Dreyfuss-Deseigne published several articles presenting methods for casting MFC films, results of accelerated aging, strength testing, and color measurements, as well as applications of the films as a mending material for transparent papers (Dreyfuss-Deseigne 2017a, 2017b, 2017c). Several researchers have further explored the use of MFC as a mending and fill material for transparent papers (Knauf and Utter

Papers presented during the Book and Paper Group Session, AIC’s 50th Annual Meeting, May 13–18, 2022, Los Angeles, California

Microfibrillated Cellulose	Cellulose Nanocrystals	Bacterial Cellulose
Fiber width: 5–100 nm	Fiber width: 3–25 nm	Fiber width: 70–130 nm
Fiber length: 1–10 µm • Microfibrillated cellulose (MFC) • Nanofibrillated cellulose (NFC) • Cellulose microfibrils • Cellulose nanofibrils • Cellulose nanofibers • Nanocellulose (generally)	Fiber length: 100–2000 nm • Cellulose nanocrystals (CNC) • Nanocrystalline cellulose (NCC) • Whiskers • Microcrystallites • Cellulose nanowhiskers	Fiber length: 1–20 µm • Bacterial cellulose (BC)

Table 1. Terms for Three Kinds of Nanocellulose

2020; Canham 2022; Henniges et al. 2022), and recently Liquitex® Professional acrylic inks were introduced as a compatible colorant for toning MFC (Henniges et al. 2022). To date, various suspensions of BC, MFC, and CNC have been explored and tested to reinforce or consolidate deteriorated paper, in applications by brush or film applicator on a vacuum suction platen or table, by lining, and by spray (Santos et al. 2016a & 2016b; Gómez et al. 2017; Völkel et al. 2017; Völkel et al. 2020; Ma et al. 2021; Opermolla et al. 2021).

The aim of this paper is to assess the differences between the three types of nanocellulose, their various physical forms, and compatibility with additives and colorants. The use of toned MFC in the consolidation of Letraset dry transfer lettering on three works on paper by Mira Schendel will also be discussed.

POLARIZED LIGHT MICROSCOPY

The preparation of nanocellulose fibers requires highly specialized technologies, so three kinds of nanocellulose were purchased from different companies (fig. 1). Celova, a ~3% suspension of MFC in water, is sold from Weidmann Fiber Technology, and CNC from Celluforce was available in two forms, as a dry powder and as a 6.3% suspension. Lastly, Hydronan BC at a concentration of 0.3–0.5% was purchased from JeNaCell.[1]

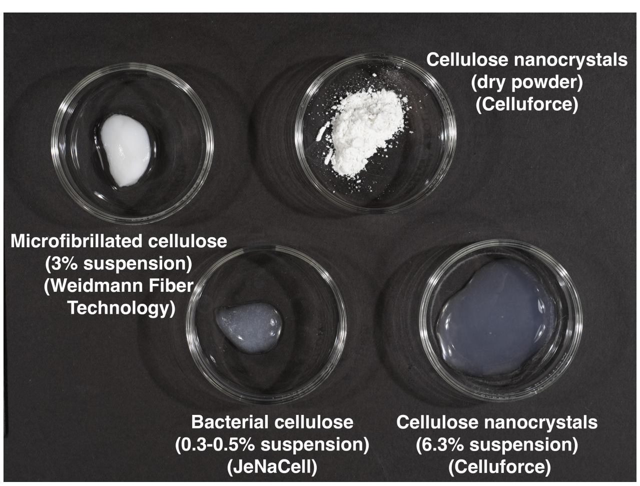

Fig. 1. Four different nanocellulose products as they arrived from three different manufacturers.

While transmission electron microscopy and atomic force microscopy are preferable for isolating nanocellulose fibers on such a small scale, polarized light microscopy provided information on the fibers' morphology and their scale.

Figure 2 compares five micrographs at 100x of the four forms of nanocellulose and typical unbleached softwood kraft fibers. Compared to the softwood kraft fibers (fig. 2a), the MFC sample (fig. 2b) is extremely fibrillated on a much smaller scale, and there are no intact fibers present. While the manufacturer identified the raw material as "softwood cellulose pulp," characteristic features of softwood, such as pitting, were not visible in the processed MFC pulp.

A C-stain was applied to the samples to further investigate the raw material sources. A C-stain is a solution that causes a color change in the fibers that can be indicative of the raw material source or specific pulping processes. The color change seen in the MFC sample was compared to known stain references in the literature (Graff 1940) and most closely matched softwood bleached kraft paper fibers or high alpha cellulose fibers.

Comparing the four forms of nanocellulose (fig. 2b–e), it becomes evident just how different these materials are, both in fiber size and morphology. Even the samples of CNC prepared at the same concentration from the slurry form (fig. 2c) and powdered form (fig. 2d) exhibit extremely different morphologies. Cellulose nanocrystals also have unusual rheological properties, as they exhibit liquid crystalline behavior (George 2015, 49). Both samples C-stained a violet-blue color with the naked eye. The C-stain did not narrow down the raw material beyond "wood pulp," as several known fiber species also C-stained to a similar color. While the source of the bacterial cellulose was known to be bacteria, the sample (fig. 2e) C-stained a bright orange, a similar color to jute or groundwood, but still much darker. While microscopy revealed information of scale and morphology, how the structures of these materials relates to their function remained an open question.

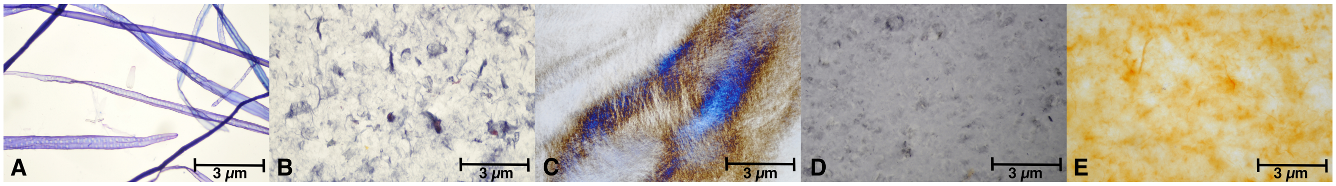

Fig. 2. Five micrographs at 100x magnification (left to right): (A) unbleached softwood kraft fibers, (B) 3% MFC from Weidmann Fiber Technology, (C) 3% CNC prepared from Celluforce pre-made suspension, (D) 3% CNC prepared from Celluforce dry powder, (E) 0.3–0.5% Hydronan BC from JeNaCell.

COMPARING CAST MFC AND CNC

MFC and CNC films were prepared with various additives and colorants to assess their effects on the working properties of the sheets. Dry sheets of MFC and CNC were both prepared using a cast-evaporation method, by which a suspension was prepared by mixing with a magnetic stir bar, poured into a petri dish, and allowed to air-dry (Dreyfuss-Deseigne 2017a, 22).

The MFC suspensions were prepared at 0.2–0.3% by weight, and 20 mL of the slurry was poured into a polystyrene dish. A square dish (9 × 9 cm) can be flexed to help release the film from the base after drying. Other conservators have used silicone petri dishes (Knauf and Utter 2020, 53; Knauf 2019) and Teflon trays (Robin Canham, e-mail to the author, November 2021). Casting a film at 0.1% by weight or lower can result in a film that is too thin and will stick to the bottom of the plastic petri dish, making it unusable. MFC at any concentration will stick to the bottom of a glass dish.

An internal sizing of methyl cellulose, added to the slurry prior to casting, improves the integrity and strength of the sheet, as well as its removal from the petri dish. Films prepared with MFC and methyl cellulose have an observed increase in transparency and mechanical strength compared to sheets without it (also observed by Henniges et al. 2022, 42). Nevertheless, both MFC prepared on its own or with methyl cellulose will wrinkle easily and exhibit strong electrical static charge (fig. 3). Because of their nanoscale, these fibers are extremely hygroscopic, making the dry films highly reactive to moisture and incompatible with many water-based conservation materials.

Additional additives, adhesives, and colorants were incorporated to assess how they would change the properties of the dry films. Several sheets were prepared with the addition of Ethulose, Klucel G, or Aquazol 500 to the slurries prior to casting. These adhesives contributed a subtle crisp quality to the films and made them more likely to tear along the edges. The sheets also responded well to toning with the addition of watercolor to the solution prior to casting.

Separate CNC slurries were prepared from the two manufacturer's forms, the dry powder and 6.3% pre-made suspension. The pre-made suspension was slightly yellow in

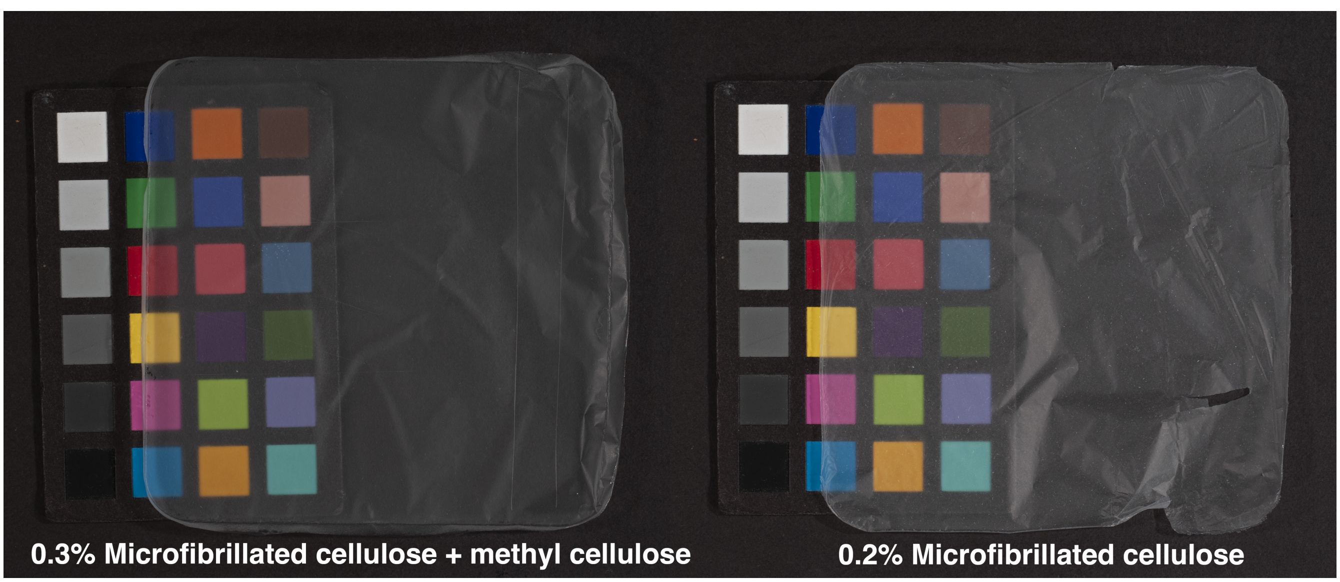

Fig. 3. A 0.3% MFC film prepared with methyl cellulose (left) exhibits slightly more transparency than a 0.2% MFC film (right).

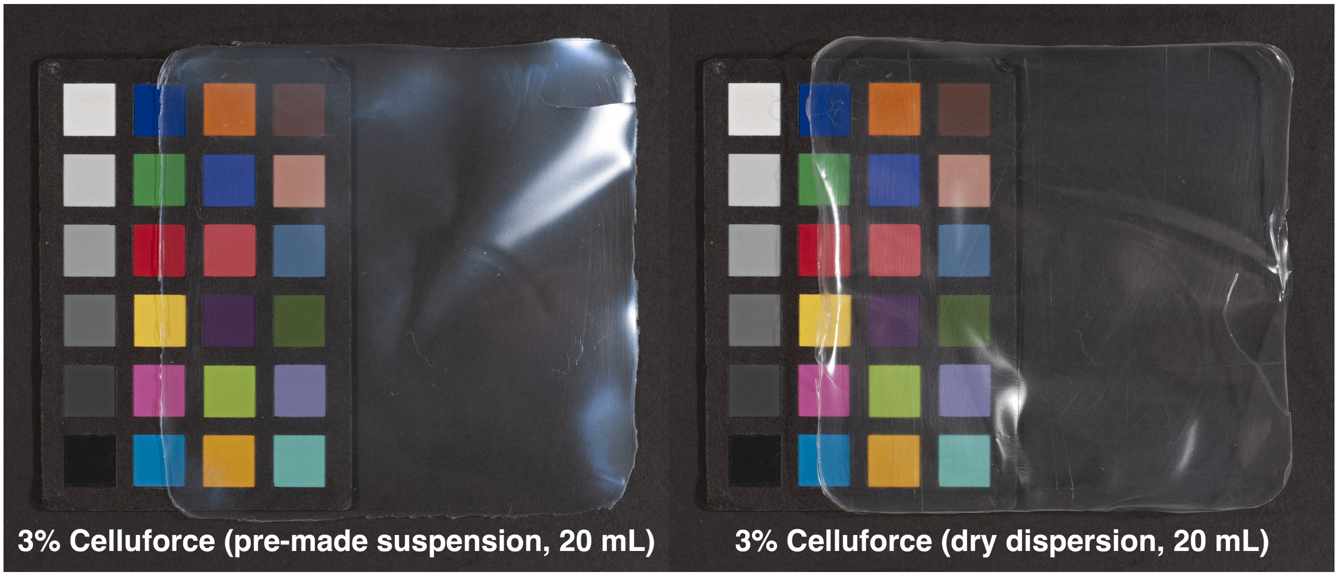

Fig. 4. While both 3% CNC films have about the same hardness, transparency, and flexibility, the sample prepared from the pre-made suspension (left) exhibits more iridescence than the sample prepared from the powder (right).

color and exhibited interference colors or iridescence. When the dry powder was dispersed in water, it formed a similar gel-like suspension but with less iridescence. Cellulose nanocrystals have different rheological properties than MFC, and the mixtures prepared are much more gel-like than MFC suspensions. CNC must be cast at a higher concentration, typically between 1.5 and 3%, compared to the MFC fibers cast at 0.3%.

Unlike the MFC, the cellulose nanocrystal films readily separated from the petri dishes without the addition of a sizing agent. In fact, the CNC films pull away from the petri dish during drying and do not lie flat. Adequate volume of the suspension must be added to the petri dishes to avoid the formation of large central holes and cracks in the film, which speak to the strong internal forces pulling the slurry to the edges of the dish. In figure 4, the CNC sample prepared from the powder exhibits less iridescence than the CNC film prepared from pre-made suspension. However, both samples had about the same hardness, transparency, and flexibility when prepared at the same concentration. For the purpose of comparison, a CNC sample was also prepared with methyl cellulose and took over four days to dry, speaking to its extreme hygroscopic nature. The sheet exhibits about the same transparency as other CNC films, but it is slightly less shiny and a little softer. Most significantly, the methyl cellulose improved the issue of distortion, and the sheet dried completely flat.

While MFC and CNC films behave nothing like paper, they may have applications in the treatment of synthetic papers or plastics as they share many of the same visual and physical properties. Their thinness and transparency are comparable to other translucent papers and films used in conservation (fig. 5). However, MFC often only *appears* more transparent than Japanese tissue because it does not exhibit long individual fibers. The extremely short fibers of the MFC and CNC make it impossible to create a feathered edge. Nanocellulose films must be cut with a hard edge, which in many instances may make a nanocellulose mend more visible than one of Japanese paper. The suitability and visibility of nanocellulose in treatment applications varies greatly depending on the substrate and materials, but it may be a highly compatible material with some of artists' less common and most unusual materials.

TREATMENT APPLICATION: TONED MFC AND LETRASET DRY TRANSFER LETTERING

As part of a campus-wide conservation initiative at the Harry Ransom Center, the treatment of several works by Mira Schendel featuring cracking and delaminating Letraset letters (fig. 6) prompted further testing of colorants in MFC and CNC samples. Letraset is a proprietary dry transfer lettering system composed of layers of ink printed on a clear film with a layer of pressure-sensitive adhesive (Vinelott 2021). MFC's smooth surface, thinness, and lack of visibly distinguishable fibers strongly resemble the surface of the transfer lettering. As such, MFC could serve as a possible fill material for the open cracks that no longer realign in Schendel's lettering (fig. 7).

Three methods of introducing color to MFC and CNC were tested: (1) adding colorant to the slurry prior to casting, (2) dyeing the films with Orasol dye dissolved in solvent, and (3) applying solvent-based Gamblin Conservation Colors to

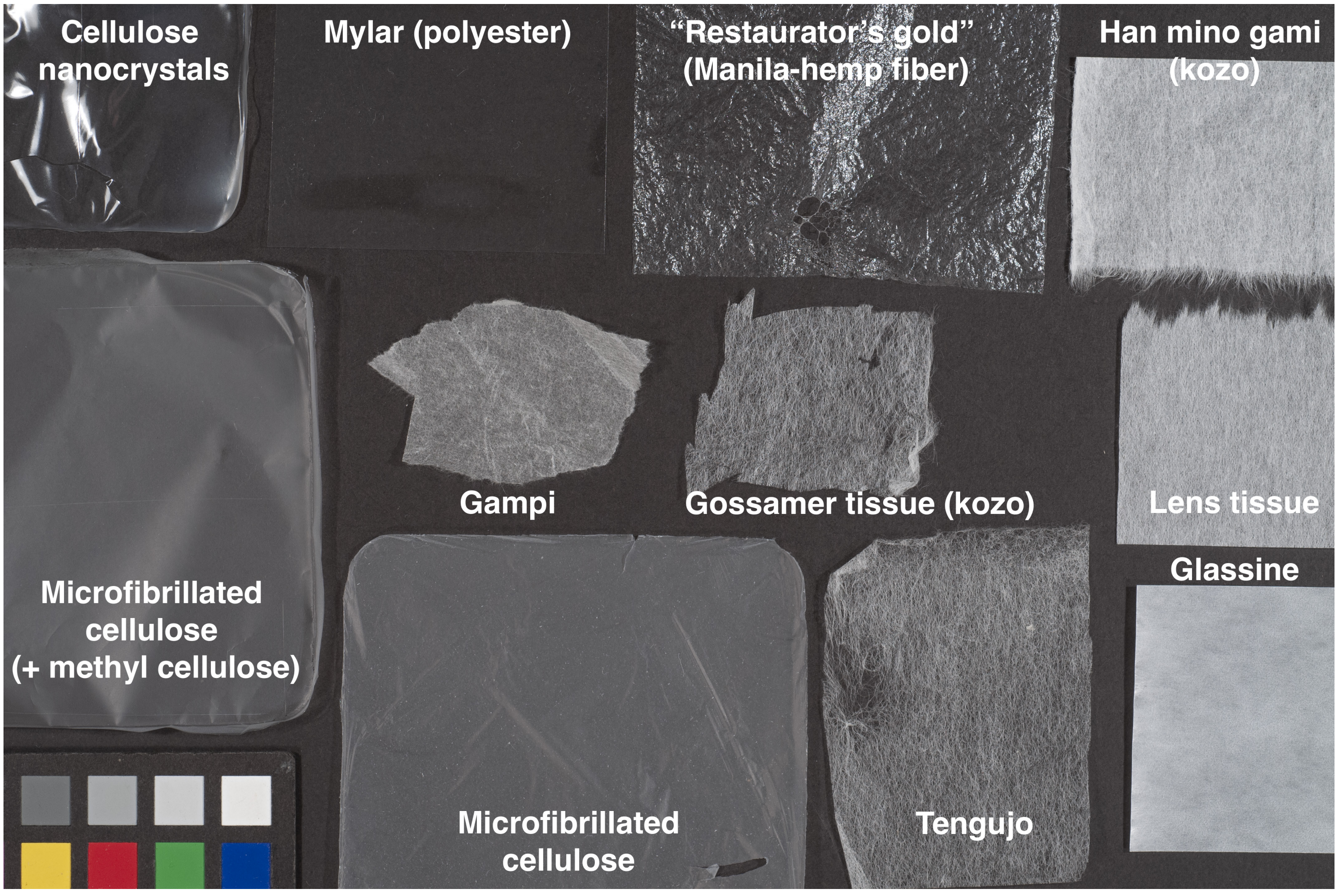

Fig. 5. MFC and CNC compared to a selection of thin, transparent papers and films used in conservation.

the dry films. These methods were selected due to the reactivity of nanocellulose to moisture when dry.

The various colorants added to the slurries included watercolors, gouache, dyes, and inks (fig. 8). While the unknown additives of proprietary formulations may raise questions of suitability for conservation, the interactions with MFC and CNC in terms of distribution, coloring power, and opacity were assessed. MFC films prepared with watercolor and gouache appeared to be the most successful in terms of distribution, opacity, and working properties, as most of the other films would not release easily from the petri dishes, possibly due to their additives.

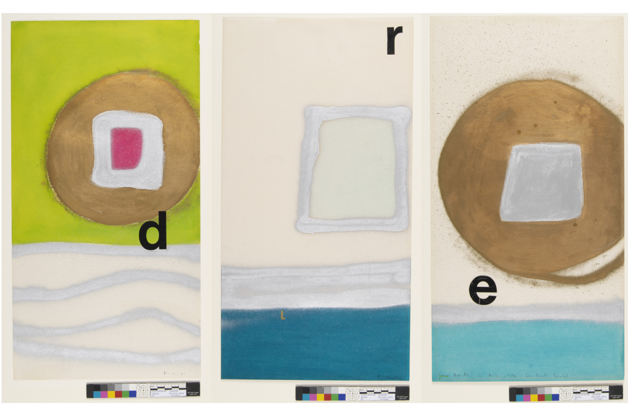

Fig. 6. Mira Schendel, Three *Untitled* works from the series *Spray*, 1970, Letraset transfer lettering, spray paint, and watercolor on paper, Blanton Museum of Art, The University of Texas at Austin, Archer M. Huntington Museum Fund, P1970.3.1, P1970.3.2, and P1970.3.5.

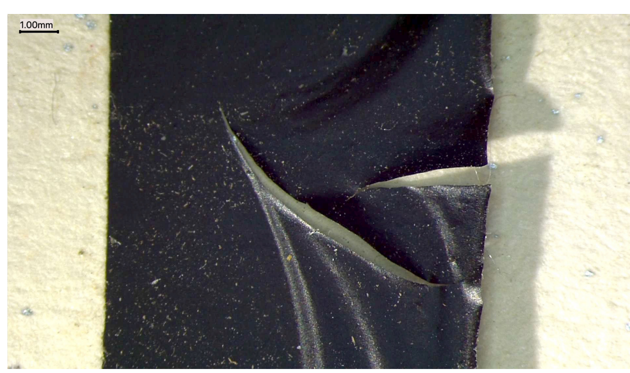

Fig. 7. Micrograph detail of the delamination and the open cracking in the Letraset “r” in one of Schendel’s *Untitled* works (P1970.3.2).

Fig. 8. Dry samples of 0.3% microfibrillated cellulose prepared with the addition of colorants to the slurries prior to casting in a petri dish. Colorants included Winsor & Newton (W&N) watercolors and gouache, Golden Fluid Acrylics, various inks, and dye.

Next, MFC and CNC films were dip-dyed or brushed with black Orasol dye RLI, which is a 1:2 chrome metal complex prepared by dissolution in ethanol (Ciba Specialty Chemicals 2001, 3) (fig. 9). The use of 99.5% ethanol allowed for submersion of the hygroscopic films but still caused very minor distortions to the sheets. Uniform application also proved challenging, as tidelines formed in the sheets, even when held vertically with tweezers during drying. The CNC collapsed at the point held by the tweezers. Although the samples dip-dyed in 2.5% and 5% were relatively opaque, the 5% dye continues to transfer and offset after the films are dry. Application by brush on top of blotter was more controllable in terms of saturation but also led to tidelines. Furthermore, the way these films may be used has yet to be explored as the dye remains soluble in ethanol, complicating applications using solvent-based adhesives.

Finally, Gamblin Conservation Colors were applied to nanocellulose films as an inpainting material. Gamblin Artist's Colors are paints made from pigments, resins, and mineral spirits that are soluble in solvents such as ethanol. In several samples, the paint was brush-applied after the film had been adhered to the support paper using 5% Klucel G in ethanol or Lascaux 498 HV in acetone. This was a more successful approach than painting and then adhering the film, as the solvent-based adhesive could re-solubilize the paint and cause offsetting on the substrate. In these applications, the MFC and CNC films would serve as a barrier layer between the support and the Gamblin paint.

Development of the most appropriate technique in the treatment of the Mira Schendel works on paper is ongoing (fig. 10). Samples of MFC, untoned and toned with watercolor, are being tested alongside 1972 Letraset samples. Testing has indicated Letraset is sensitive to solvents, including ethanol and acetone, as well as heat, posing challenges for the attachment of the MFC below the open cracks. However, application techniques using MFC films as a remoistenable tissue have proved promising (Canham 2022).

CONCLUSION

Nanocellulose is a complex material. It does not behave like Japanese paper and other more traditional materials used in conservation. The properties of nanocellulose go far beyond its extreme transparency. While MFC films are thin and wrinkle easily, CNC films are hard and vary in flexibility

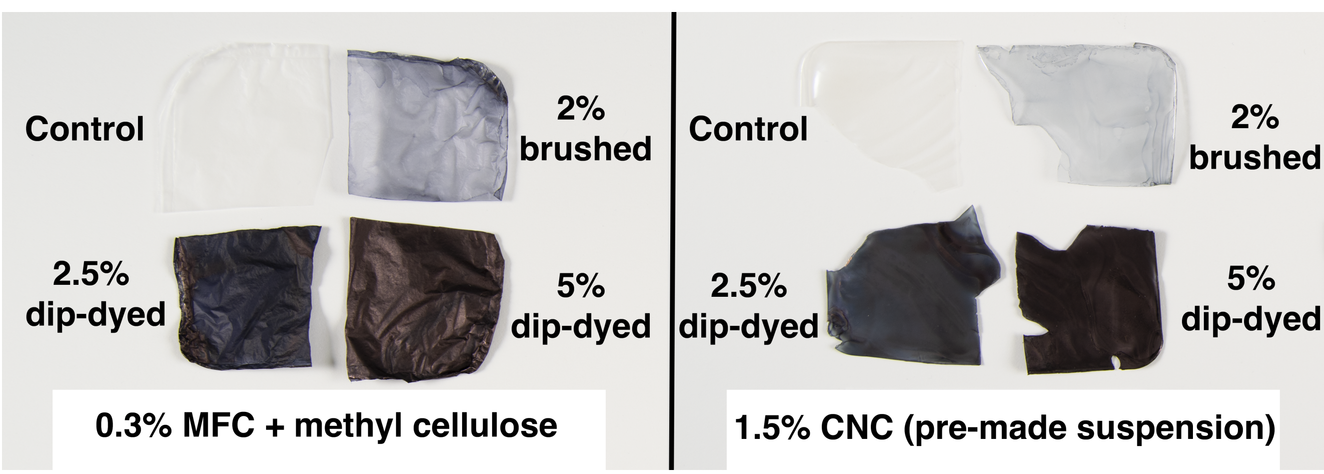

Fig. 9. Samples of 0.3% MFC prepared with methyl cellulose (left) and 1.5% CNC prepared from the pre-made suspension (right) were cut into quarters, and Orasol-dye was applied to three pieces of each sample. Samples in the upper right were toned by brush-applying 2% Orasol dye. Samples in the lower left and lower right were dip-dyed with Orasol dye at 2.5% and 5%, respectively.

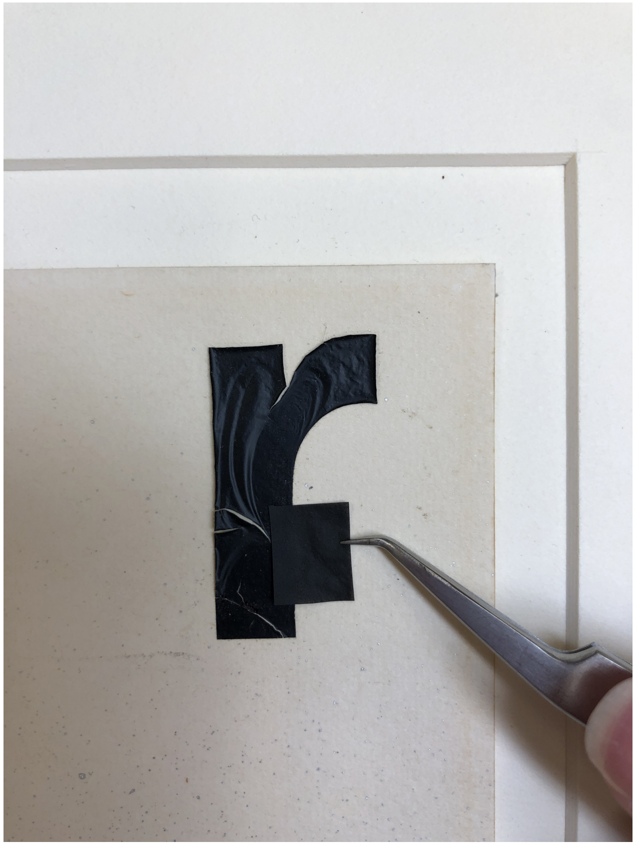

Fig. 10. Detail of Mira Schendel's Letraset alongside a sample of 0.3% MFC toned with watercolor.

and sheen depending on their preparation. The addition of methyl cellulose as a sizing agent may be used in both MFC and CNC films. When added to a MFC slurry prior to casting, methyl cellulose increases the transparency and strength of the film when dry, and in CNC, methyl cellulose appears to improve issues of distortion upon drying. Both MFC and CNC respond well to toning through the addition of watercolor to the suspensions before cast-evaporation. As such, nanocellulose films provide a range of surface textures, hardness, thicknesses, and opacities that may be compatible with translucent or synthetic papers, plastics, and some of artists' most unusual materials.

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NOTE

1. When purchasing any material for use in conservation, there are always questions of the safety of these materials for collection objects, and much more research is still needed. Preliminary results of analytical testing suggested that some of these materials from specific manufacturers may contain more than just cellulose, so the safety of some nanocellulose materials may vary depending on the proprietary preparations of different manufacturers.

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SOURCES OF MATERIALS

Cellulose Nanocrystals
CelluForce Inc.
2000 McGill College Avenue, 6th Floor
Montreal, Quebec, H3A 3H3
Canada

Celova for Art Conservation
Weidmann Fiber Technology
Neue Jonastrasse 60
8640 Rapperswil SG
Switzerland

Hydronan, Bacterial Cellulose
JeNaCell—An Evonik company
Göschwitzer Str. 22
07745 Jena
Germany

Gamblin Conservation Colors and Orasol Dye
Museum Services Corporation
385 Bridgepoint Way
South St. Paul, MN 55075
United States

AUTHOR INFORMATION

RACHEL MOCHON
Paper Conservator, Campus Conservation Initiative
Harry Ransom Center, University of Texas at Austin
Austin, TX
rachel.mochon@austin.utexas.edu