Design of Adhesive-free Bio-based Suspended Ceiling Tiles Using Nanocellulose

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An adhesive-free, bio-based suspended ceiling tile (nanocellulose ceiling tile (NCT)) with mildew and mold resistance, fire retardancy, and water repellence was developed and compared with commercial products (M257, M266, and M935). The NCT produced the greatest contact angle values: 145.0° (1.37% coefficient of variation (CV)) on the top surface, 137.8° (2.15% CV) on the bottom surface, and 142.2° (3.41% CV) on the edges. The thermal resistivity (R-value) of the NCT was also greater (3.14 °F·ft^2·h/BTU (1.32% CV)), so the thermal conductivity (k-value) was lower (0.046 W/(m·K)) than the compared commercial products (M257, M266, and M935). The NCT demonstrated resistance to mildew and mold growth, with zero defacement. Each product (NCT, M257, M266, and M935) evaluated was fire retardant, with zero horizontal flame propagation. The results showed that the NCT was an advanced ceiling tile prototype treated against fire, water, and mildew and mold growth throughout its thickness, with promising results compared to current ceiling tiles on the market.

Keywords: Ceiling tile; Nanocellulose; Forest bio-products; Freeze-drying; Adhesive free; Mildew/Mold resistant; Light reflectance value (LRV)

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INTRODUCTION

Suspended ceiling tiles are used most commonly in public and commercial buildings, including commercial offices, retail stores, hospitals, schools, and government buildings. These ceiling tiles offer flexibility and easy access to utilities throughout their lifetime. Suspended ceiling tiles vary considerably in composition, price, and quality. However, most are not durable, sag or stain when exposed to moisture; they may off-gas hazardous compounds, release airborne particles that are hazardous to health when broken or cut, and promote mildew and mold growth through moisture retention. With few exceptions, damaged tiles are not fully compostable and therefore must be landfilled for disposal.

Many tiles on the market contain materials that pose substantial human health risks through the release of airborne particles or chemicals. The global ceiling tile market is dominated by products containing mineral fibers such as slag wool, man-made vitreous (silicate) fibers and/or stone wool, expanded perlite clay (kaolin), or recycled paper in a starch binder. The second most widely used tiles contain gypsum. Some tiles contain fiberglass, which is intended to increase their insulation properties. Formaldehyde is also used as a binding agent in most of the tiles containing mineral fiber, fiberglass, medium density fiberboard (MDF), or non-wet formed perlite. Polystyrene is used in other tiles. The building block of polystyrene is styrene, a possible human carcinogen (Huff and

Infante 2011).

In addition to the human health risks summarized in Table 1, the commercial ceiling tiles (fiberglass, polystyrene, mineral fiber based) available on the market today pose potential threats to the environment. The same airborne particles that irritate skin, eyes, and the respiratory system pose ecological risks if released to the environment *via* improper disposal. Although some companies run recycling programs for some of their products (Armstrong Ceilings 2016), most ceiling tiles are not recyclable and contribute to landfills.

Table 1. Problems with Current Ceiling Tiles

Material Content	**Associated Health Hazards**
Mineral fiber, gypsum, fiberglass	Skin, eye, throat, and respiratory system irritant; lung disease and/or cancer if prolonged exposure; silicosis (OSHA 2005; Neghab *et al*. 2016; EPA 2016)
Formaldehyde	Skin, eye, nose, and throat irritant; possible carcinogen at high levels (EPA 2010)
Polystyrene	Possible human carcinogen; skin, eye, and upper respiratory track irritant (EPA 2016)
Property	**Associated Disadvantages**
Water absorption	Moisture retention resulting in possible mold/bacterial growth, staining, and sagging
Inertness	Not compostable, requiring landfill disposal; potential environmental hazard if improperly disposed

The overarching goal of this study was to assess the feasibility of developing a bio-based, eco-friendly ceiling tile using bio-polymers and applying novel technology to the production process. The additional sub-objectives were to investigate the performance properties of the ceiling tile developed in this study and to compare its performance properties with the traditional commercial products on the market.

The demand for "green" building materials is growing, and many companies have been working on integrating green products into their projects. Therefore, there is a growing market opportunity for green ceiling tiles. The development of bio-based ceiling tiles was performed using bio-nanopolymers cellulose nanofibrils (CNFs). Cellulose nanofibrils are microscopic fibrils that can be obtained chemically or mechanically from wood, plants, and even sea animals (Moon *et al*. 2011) and have excellent mechanical and thermal properties (Lahiji *et al*. 2010; Wagner *et al*. 2011; Pakzad *et al*. 2012; Wu *et al*. 2013). They consist of D-glucopyranose ring units, each linked by β-1,4-glycosidic bonds (Krässig 1993). Each β-glucose molecule is rotated 180° (Hubbe *et al*. 2008) compared with the one next to it, and these rotations through the long chain and amorphous sections in the chemical structure provide flexibility to the final product (Klemm *et al*. 2011). Another biopolymer, cornstarch (CS), was used to produce rigidity by creating cross-linking.

The CNFs and CS are both hydrophilic polymers and adsorb water easily. The available free hydroxyl groups (OH-) found in CNFs and CS make final products hydrophilic (Song *et al*. 2014). This hydrophilicity produces easy interaction with water. Once the water molecules are adsorbed by the final product, the cells and cellular structures collapse and mechanical performance decreases (Topgaard and Söderman 2001; Hofstetter *et al*. 2006). Silanating agents are the common reactive molecules used to modify the surfaces of materials; these have diverse usages in applications such as polymer composites.

In this study, n-octadecyltriethoxysilane ($C_{24}H_{52}O_3Si$) was used to add water repellence to the final product. The cellulosic materials and corn starch-based materials (Yin *et al*. 2005) are also flammable due to the elements (C, H, and O) found in their chemical structure that help start the combustion. Furthermore, having highly available hydroxyl groups makes them easily flammable (Gaan and Sun 2007). To overcome this challenge, a treatment of boric acid (BA, $B(OH)_3$, 99.94% pure), which is one of the most frequently used fire retardants for starch-based and cellulosic materials (Horrocks 2011), was applied. The addition of BA reduces flame spread in cellulosic material (Zenat *et al*. 2011) by increasing char forming and releasing the water in the composite structure. Addition of BA also improves mechanical performance by crosslinking (Yin *et al*. 2005) and helps produce a rigid structure in the final product.

Potential mildew or mold growth is another challenge for products used in moist environments and is one of the most significant issues for the ceiling tile industry. Mildew and molds are forms of fungus and can be differentiated by their colors. Mildew appears white; mold can appear in shades of black, blue, red, or green. These simple microscopic organisms can thrive anywhere there is moisture. Molds are beneficial in nature, where they help break down organic matter, but they pose a significant problem indoors. They lower the esthetic quality of the material used indoors. Boric acid is also sold as a pesticide providing mildew and mold resistance. Consequently, the BA used for fire retardancy also provides mildew and mold resistance as a bonus value-added property.

In this study, a bio-based ceiling tile that eliminates the majority of the problems (flammability, hydrophilicity, and mildew and mold growth) posed by currently available products and that has equal or superior performance was developed. Its performance properties were investigated and compared with randomly purchased ceiling tiles available on the market: M257, M266, and M935.

EXPERIMENTAL

Materials and Methods

The raw cellulose nanofibrils (CNFs) were obtained from Revolution Research, Inc. (RRI, Orono, ME, USA). The cross-linker, Ethylex 2025 industrial corn starch (CS) was obtained from Tate & Lyle (Decatur, IL, United States). The n-octadecyltriethoxy-silane was purchased from Gelest, Inc. (Morrisville, PA, USA). The boric acid (BA) was purchased from Rose Mill Co. (West Hartford, CT, USA).

Although CNFs are being investigated for many applications, the production method used in this study, freeze-drying, has not been used for ceiling tile design and manufacturing. This is the first study to use freeze-drying to design and produce ceiling tiles. In the production process, no hazardous blowing agents or adhesives were used. Instead of starting from solid powders or fibrils, the production was started from a water-based suspension. The starting water suspension included, by weight, 7.5% CS, 2.5% CNF, 1% BA, and 1 % n-octadecyltriethoxysilane. An illustration of the phase changes through the production process is given in Fig. 1. The production started from the water suspension (water slurry), and then the suspension was frozen. Finally, sublimation was performed to convert ice molecules to gases without cellular collapse.

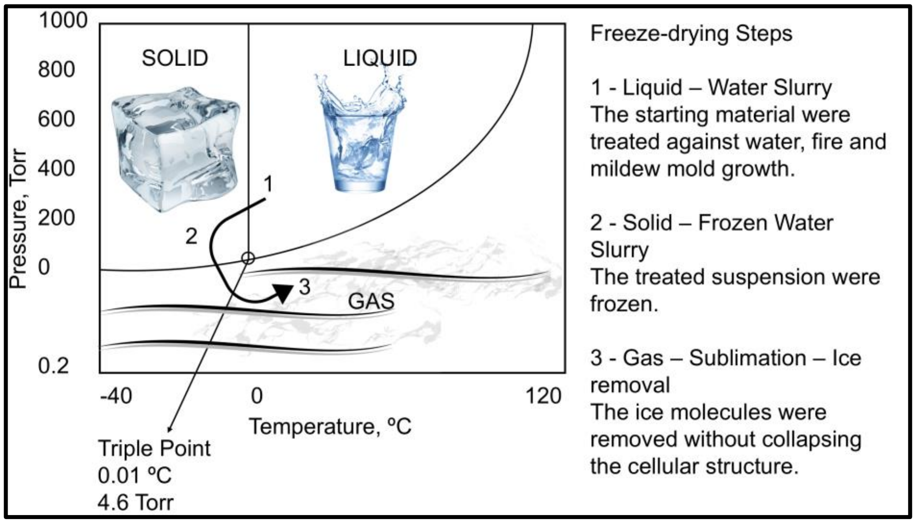

Fig. 1. Illustration of freeze-drying flow: (1) Liquid, water slurry – The starting materials were treated against flammability, hydrophilicity, and mildew and mold growth. (2) Solid, frozen slurry – The treated suspension was frozen. (3) Gas, ice removal *via* sublimation – The ice molecules were removed without collapsing the cellular structure.

Treatment materials to make the final product water repellent, mildew and mold resistant, and fire retardant were added to the suspension before freeze-drying (Fig. 2), resulting in a product with those desired characteristics throughout its thickness, not just on the surface as with other ceiling tiles.

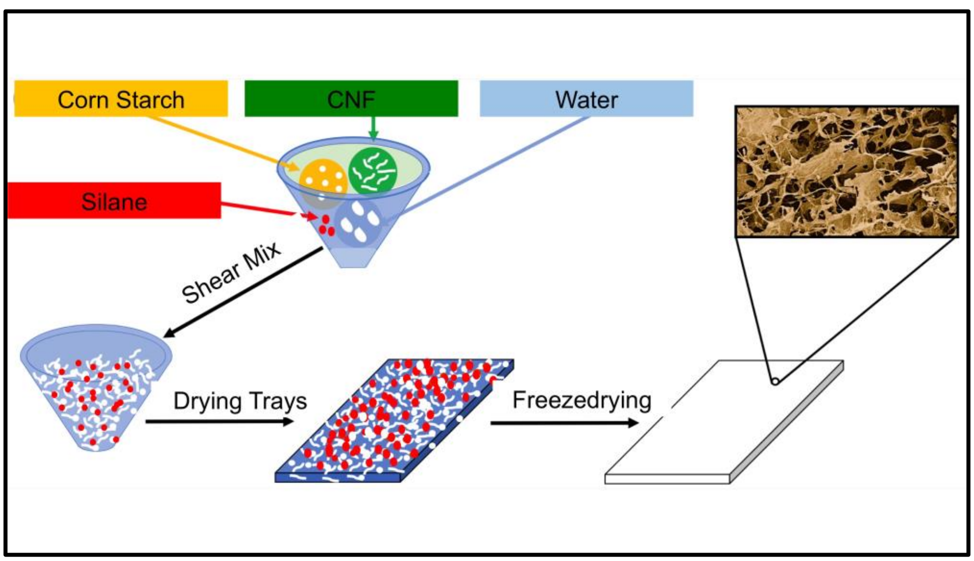

Fig. 2. Illustration of the manufacturing process

Performance Evaluation Methods

In this study, all data were collected and evaluated according to ASTM E1264 - 14 (2014). Data were compared with the randomly purchased commercial products (M257, M266, and M935) on the market. Table 2 lists the tests that were performed in this study.

Table 2. ASTM Standards Used in this Research

Standard	Description
ASTM C303 – 10 (2016)	Standard Test Method for Dimensions and Density of Preformed Block and Board-Type Thermal Insulation
ASTM D4442 – 16 (2016)	Standard Test Methods for Direct Moisture Content Measurement of Wood and Wood-Based Materials
ASTM E96 / E96M – 16 (2016)	Standard Test Methods for Water Vapor Transmission of Materials
ASTM C165 – 07 (2017)	Standard Test Method for Measuring Compressive Properties of Thermal Insulations
ASTM E1477 – 98A (2017)	Standard Test Method for Luminous Reflectance Factor of Acoustical Materials by Use of Integrating-Sphere Reflectometers
ASTM D7334 – 08 (2013)	Standard Practice for Surface Wettability of Coatings, Substrates and Pigments by Advancing Contact Angle Measurement
ASTM C518 – 17 (2017)	Standard Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus
ASTM D635 – 14 (2014)	Standard Test Method for Rate of Burning and/or Extent and Time of Burning of Plastics in Horizontal Position
ASTM D3273 – 16 (2016)	Standard Test Method for Resistance to Growth of Mold on the Surface of Interior Coatings in an Environmental Chamber

Statistical Analysis

Density, compression properties, moisture content, water vapor transmission, contact angle, and thermal conductivity were analyzed and compared *via* a one-way Means/ANOVA to check if there was a significant overall difference (alpha = 0.01) due to the difference in the types of evaluated ceiling tiles (nanocellulose ceiling tile (NCT), M257, M266, and M935). Significant differences between groups were evaluated using a Tukey-Kramer honestly significant difference (HSD) test (alpha = 0.05).

RESULTS AND DISCUSSION

The material performance evaluations were performed according to relevant ASTM standards. The density values were determined by dividing the measured sample mass to the measured sample volume ASTM C303 – 10 (2016). The moisture content values were determined according to ASTM D4442 – 16 (2016), and the water vapor transmission (WVT) values were determined using ASTM E96/E96M – 16 (2016). The mechanical performance, durability, is also a critical property for the ceiling tiles. The behavior of the material under compressive load was determined according to ASTM C165 - 07 (2017). The results for the density, moisture content, WVT, compression strength, and compression modulus are shown in Table 3.

As shown in Table 3, densities showed statistical differences between the groups. The NCT had a significantly lower density than the other groups. Notably, the density of NCT was one half (1/2) that of the M257. Because lower density yields lower weight, this is a clear advantage for building materials. Lighter products decrease the overall building weight, offer more economic transportation, and are more easily installed. The NCT was designed as a breathable material, making the product capable of holding and releasing moisture according to the environment. This moisture-holding capability yielded a greater WVT, a property related to the chemical components of the final product, the physical and morphological properties of the components, the available paths for the moisture, and the tortuosity, for the NCT. For WVT, no statistical differences were observed among the M257, the M266, and the M935, nor were they found among the NCT, the M266, and M935. The M257 showed statistically lower WVT values than the NCT.

Table 3. Density, Moisture Content, WVT, Compression Strength, and Compression Modulus Results

Sample	Density (g/cm^3)	Moisture Content (%)	WVT (metric perm)	Compressive Strength (kPa)	Compression Modulus (kPa)
NCT	0.13 (1.53) D	9.02 (1.16) A	30.55 (12.14) A	90.42 (17.86) C	2625.09 (12.14) C
M257	0.26 (0.57) A	1.41 (1.21) B	23.44 (1.49) B	246.72 (20.61) B	5295.63 (1.49) B
M266	0.25 (0.60) B	1.18 (7.22) C	25.62 (3.81) AB	152.55 (24.15) C	4377.23 (3.81) BC
M935	0.20 (0.47) C	1.36 (3.34) B	27.11 (0.51) AB	372.32 (18.61) A	8313.56 (0.51) A

Parentheses indicate the coefficients of variation (COV, %). A, B, and C indicate the significant differences among the treatments.

The components of the final product played critical roles in the overall performance properties. The composite made from the polymers with high performance properties would potentially behave well against external loads. However, this is not the only parameter that affects the performance properties of the final product. Density and moisture content are also critical factors that affect the mechanical properties of the final product. In earlier studies, researchers showed that lower density values produced lower mechanical performance properties for composites developed using the same ingredients (components) (Yildirim *et al*. 2014). Additionally, moisture content is a critical factor for any type of hygroscopic material. An increase in moisture content can affect thermal properties by creating potential cell collapses and can decrease mechanical performance by collapsing the beams, columns, or walls that bond the cells together. It has been shown that increased moisture content decreases the mechanical performance of wood-based materials (Sombatsompop and Chaochanchaikul 2004). Therefore, the lower performance values of the NCT compared to M257 and M935 can be related to lower density and higher moisture content. Despite its lower density and higher moisture content, the NCT had statistically no difference in compression performance compared to the M266.

Table 4. Light Reflectance Value (LRV) Results for the Materials

Sample	LRV - D65/10 (%)	LRV - F2/10 (%)	LRV - A/10 (%)	LRV - C/2 (%)
NCT - top	82.13 (2.20) C	82.38 (2.19) C	82.46 (2.14) C	82.21 (2.28) C
NCT - bottom	85.97 (1.11) A	86.17 (1.10) A	86.24 (1.09) AB	86.11 (1.10) A
M257	84.12 (0.73) B	84.62 (0.72) B	84.89 (0.80) B	84.43 (0.72) B
M266	87.02 (1.25) A	87.05 (1.00) A	87.11 (1.00) A	86.97 (1.01) A
M935	86.53 (1.10) A	86.61 (1.08) A	86.63 (1.07) A	86.56 (1.09) A

Parentheses indicate the coefficients of variation (COV, %). A, B, and C indicate the significant differences among the treatments. "D65/10" Average daylight (including ultraviolet wavelength region). "F2/10" Cool white (fluorescent). "A/10" Incandescent light. "C/2" Average daylight (not including ultraviolet wavelength region)

Light reflectance value (LRV) is another important property for ceiling tiles. The brightness of the environment is affected by the color and reflectance of the used ceiling tiles. Mostly white ceiling tiles with high LRVs are preferred for commercial and residential buildings. The LRVs were determined according to ASMT E1477 and are summarized in Table 4.The NCT offered a unique feature: It could be suitably used with either surface exposed, unlike traditional ceiling tiles. Compared to other products, the mean LRVs of each surface were very similar, though the bottom surface provided a greater LRV than the top surface of the NCT. This result was due to the production process, freeze-drying, in which some of the molecules randomly were pushed to the top surface. Consequently, the top of the NCT had a fibrillary surface, while the bottom surface was flat (Fig. 3).

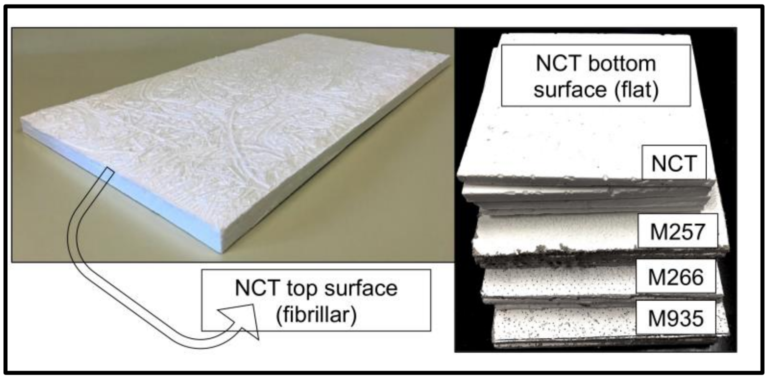

Fig. 3. The flat and fibrillary surfaces of the NCT and the evaluated samples

As mentioned earlier, the treatment materials were added to the suspension before freeze-drying, resulting in a product with those desired characteristics throughout its thickness, not just on the surface as with other ceiling tiles. The contact angle was one of the properties in need of enhancement due to the hydrophilicity of the used materials. As shown in Table 5, the contact angles were measured not only from the top surface but also from the bottom surface and the edges. The NCTs showed statistically greater contact angles (145.0° (top), 137.8° (bottom), and 142.2° (edge)) than all the other products evaluated.

Table 5. Contact Angles of the Materials

Sample	Contact Angle (°), Top	Contact Angle (°), Bottom	Contact Angle (°), Edge
NCT	145.03 (1.37) A	137.75 (2.15) A	142.25 (3.41) A
M257	51.35 (7.95) B	63.50 (9.68) B	120.55 (3.99) B
M266	61.68 (12.97) B	23.85 (5.82) C	126.28 (3.06) B
M935	29.35 (15.87) C	63.45 (4.09) C	129.15 (2.77) B

Parentheses indicate the coefficients of variation (COV, %). A, B, and C indicate the significant differences among the treatments.

Thermal insulation properties (thermal resistivity, R-Value) are also important parameters for the ceiling tiles. The R-values (thermal resistivity) and k-values (thermal conductivity) are shown in Table 6. The thermal resistivity (R-value) of the NCT was greater (3.14 ºF $\cdot ft^2 \cdot h$/BTU – 1.32% COV) than those of the M257, M266 and M935, statistically. Consequently, the thermal conductivity (k-value = 0.046 W/(m·K)) was lower than those of the compared commercial products (M257, M266, and M935), statistically. The decreased density corresponds with greater porosity, indicating a higher amount of air in the final product structure, which decreases thermal conductivity.

Table 6. The R-value and the k-value of the Materials

Sample	Thermal Resistivity, R-value (ºF·ft²·h/BTU)	Thermal Conductivity, k (W/(m·K))
NCT	3.14 (1.32) A	0.046 (1.32) D
M257	2.29 (1.01) D	0.063 (1.01) A
M266	2.35 (0.81) C	0.061 (0.81) B
M935	2.62 (0.58) B	0.055 (0.58) C

Parentheses indicate the coefficients of variation (COV, %). A, B, C, and D indicate the significant differences among the treatments.

The NCT and commercial products were tested for fire retardancy according to the relevant standard. The horizontal burning rates (V, mm/sec) were investigated (Fig. 4). All groups (NCT, M257, M266, and M935) studied were found fire retardant, and none of the samples were able to reach the 25 mm mark.

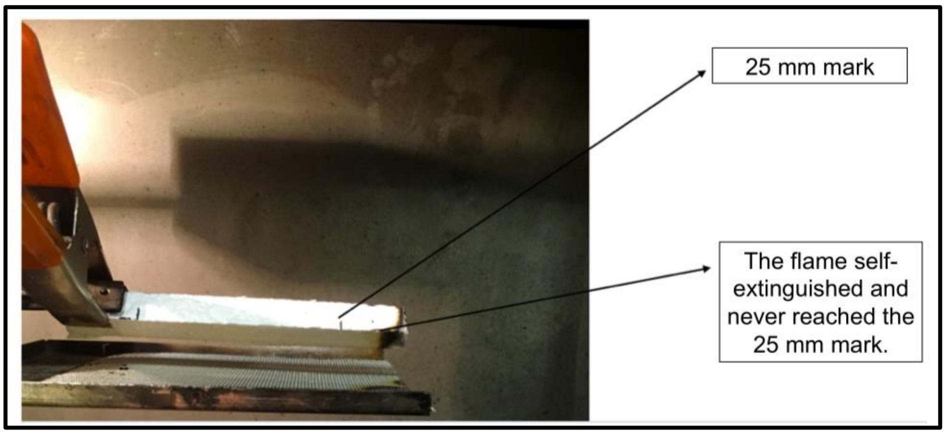

Fig. 4. Photograph of fire retardancy testing

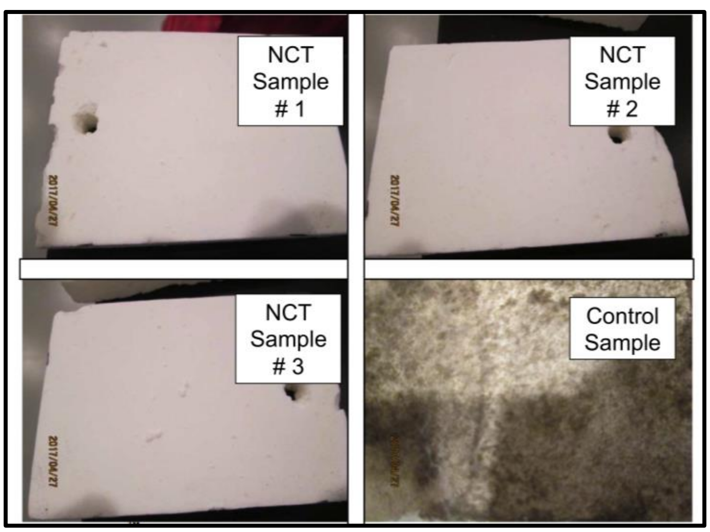

Fig. 5. Post-incubation photos of NCT *vs*. control sample

The BA treatment was successful in providing fire retardancy to the NCT. The overall horizontal burning test showed that the flame self-extinguished and never reached the 25 mm mark ASTM D635 – 14 (2014).

The mildew and mold growth tests were performed using *Aureobasidium pullulans*, *Aspergillus niger*, and *Penicillium citrinum* organism species. The NCT samples received a rating of 10, signifying zero defacement on the test specimens at the completion of the mildew/mold resistance evaluation (Fig. 5).

CONCLUSIONS

1. Nanocellulose ceiling tiles (NCT) could be produced using the freeze-drying technique.
2. Starting from the cellulose nanofibrils (CNF) suspension instead of dried CNF would eliminate drying costs, provide cost reduction, and save time.
3. Freeze-drying provided a micro/nano cellular structured final product.
4. The boric acid (BA) treatment provided fire retardancy and mildew/mold resistance.
5. The silane treatment provided water repellence to the final product, with contact angles of 145.0° (1.37 % coefficient of variation (CV)) on the top surface, 137.8° (2.15% CV) on the bottom surface, and 142.2° (3.41% CV) on the edges.
6. The ceiling tile manufactured using biopolymers provided high light reflectance values (LRV), between 82% and 87%.
7. The use of CNF produced reduced weight ceiling tiles with comparable performance properties, which is an important feature to decrease the total building load.

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Conflicts of Interest: The author declares no conflicts of interest.

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