Barrier Coating of Fruit-packing Tissue Paper Using Nanofibrillated Cellulose and a Water-Repellent Agent

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An environmentally friendly barrier-coating strategy was studied for fruit-packing tissue paper intended for use in fruit protection bags under humid agricultural conditions. Because tissue paper has high porosity and hydrophilic characteristics, its practical use is limited by low mechanical strength, high air permeability, and poor water resistance. Three coating systems were evaluated using nanofibrillated cellulose (NFC) and a water-based acrylic water-repellent: tissue paper coated twice with NFC, tissue paper coated twice with the acrylic agent, and tissue paper sequentially coated with one NFC layer followed by one acrylic top coating. Double NFC coating increased tensile strength from 2.96 to 4.34 kN/m and improved Gurley air resistance from 34.3 to 114.6 s through pore filling and enhanced inter-fiber bonding. The double acrylic coating provided a water-repellency level of 8 but had limited effects on tensile strength and air resistance. The multilayer coating combined the advantages of both materials, maintaining improved tensile strength, reduced air permeability, and strong water repellency. FE-SEM observations confirmed reduced pore openness after NFC application and continuous film formation after acrylic top coating. These results indicate that sequential coating with NFC and an acrylic water-repellent provides an effective route for improving the barrier performance of tissue paper for fruit protection applications.

DOI: 10.15376/biores.21.3.7690-7702

Keywords: Barrier coating; Multi-layer; Nanofibrillated cellulose (NFC); Water-based acrylic repellent; Barrier performance

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INTRODUCTION

The growing demand for sustainable and environmentally friendly packaging materials has accelerated research aimed at replacing synthetic polymers and plastic-based coatings (Reichert *et al*. 2020; Perera *et al*. 2023; Khandeparkar *et al*. 2024). Fruit-packing tissue paper is as a promising candidate for eco-friendly packaging applications because of its biodegradability, recyclability, and functional versatility (Jasmani *et al*. 2021; Mathura and Maharaj 2025; Zhang *et al*. 2025). However, its intrinsically high porosity and hydrophilic nature result in poor water repellency, high air permeability, and limited barrier performance against moisture and gases (Wang *et al*. 2021; Abenghal *et al*. 2025). These limitations restrict its use in applications requiring controlled moisture resistance, air barrier properties, and sufficient mechanical durability. Therefore, advanced coating technologies that improve barrier performance while maintaining recyclability are needed (Silva *et al*. 2023; Lu *et al*. 2025).

Traditionally, the paper industry has employed synthetic polymers and chemical agents such as polyethylene, polyvinylidene chloride (PVDC), and fluorinated compounds to enhance barrier performance (Glenn *et al*. 2021; Spagnuolo *et al*. 2022). These coating materials effectively reduce oxygen and moisture transmission, thereby extending product shelf life and maintaining product quality (Maio *et al*. 2014; Smith *et al*. 2018; Norton 2020; Chen *et al*. 2024). However, such polymer-based coatings are non-biodegradable, difficult to separate from paper substrates during recycling, and may generate hazardous by-products during disposal or incineration (Bohlmann 2004; Shen *et al*. 2020), thereby reducing fiber recovery efficiency and contributing to environmental pollution. Consequently, they hinder recycling processes and raise concerns regarding long-term sustainability within a circular economy (Turner and Filella 2021; Sobhani *et al*. 2025).

Accordingly, eco-friendly alternatives such as nanofibrillated cellulose (NFC) and bio-based waxes have been investigated (Zhang *et al*. 2014; Nechita and Roman 2020). NFC offers excellent film-forming ability and gas barrier properties (Fotie *et al.* 2020; Jin *et al*. 2021), whereas bio-waxes provide strong moisture resistance (Krishnan *et al*. 2025; Jahangiri *et al*. 2024). A previous study demonstrated that multilayer coatings combining cationic and anionic NFC with paraffin-free biowax achieved low air permeability and reduced water vapor transmission, indicating potential for recyclable paper packaging applications (Jo *et al*. 2022). However, although NFC and biowax exhibit promising barrier performance, their relatively high material costs and process complexity remain limitations for application to low-value paper grades such as tissue paper, where coating costs are a critical factor (Nair *et al*. 2014; Koppolu *et al*. 2019).

Water-based acrylic water-repellent agents have recently attracted attention as environmentally compatible coating alternatives (Lee *et al*. 2023). Compared with conventional fluorochemical or wax-based treatments, acrylic dispersions exhibit good film-forming ability, compatibility with aqueous systems, and reduced volatile emissions (Sendra *et al*. 2025). Although they effectively impart hydrophobicity, they do not significantly improve air permeability or paper strength when applied alone. Therefore, combining NFC, which enhances mechanical properties and reduces pore openness (Mousavi *et al*. 2018), with water-based acrylic repellents may provide a complementary coating strategy for improving both strength and barrier performance (Hassan *et al*. 2018; Koppolu *et al*. 2019; Jin *et al*. 2021).

In this study, a multilayer coating system comprising NFC and a water-based acrylic repellent was evaluated for tissue paper used in fruit protection bags, with emphasis on improving moisture resistance, reducing air permeability, and enhancing mechanical strength under humid agricultural conditions.

EXPERIMENTAL

Materials

Fruit-packing tissue paper, manufactured by Namgang Paper Co., Ltd. (Jinju, Republic of Korea), was used as the substrate for barrier coating, as shown in Fig. 1. This paper was not a facial tissue or a creped tissue product, but a highly porous, lightweight base paper designed for agricultural fruit-protection bags. The base paper had a basis weight of 50 g/m² and was selected because its high porosity and low strength made it suitable for evaluating coating-induced improvements in barrier and mechanical properties.

Hardwood-bleached kraft pulp (Hw-BKP), supplied by Moorim Paper (Jinju, Republic of Korea), was used to produce nanofibrillated cellulose (NFC) in the laboratory. A water-based acrylic water-repellent agent (KRP2000, Dong Jin Industry Co., Ltd., Gyeongsan, Republic of Korea) was used as a coating component to impart water repellency to the paper. The specifications of the acrylic repellent are listed in Table 1.

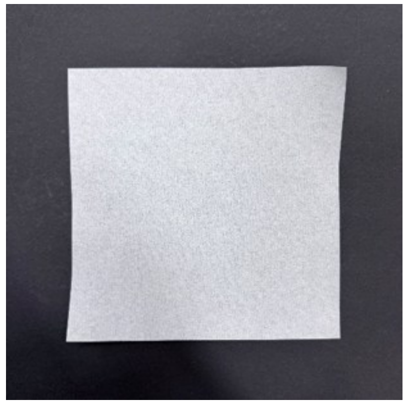

Fig. 1. Tissue paper with a basis weight of 50 g/m^2 used as a base paper for barrier coating

Table 1. Specifications of Water-Based Acrylic Repellent

Items	Property
Appearance	Light yellow liquid
Purity	35%
Viscosity	100 cPs
pH	8 to 9
Charge	Anionic

Preparation and Characterization of NFC

NFC was prepared by beating and micro-grinding Hw-BKP. Beating was performed at a consistency of 1.57% using a laboratory Hollander beater (FRANK-PTI GmbH, Birkenau, Germany). The Hw-BKP was beaten to a Canadian standard freeness of 450 ± 5 mL (Park *et al*. 2018; Lee *et al*. 2020). The beaten pulp was diluted to 1.0% consistency with distilled water and then fibrillated using a Super Mass Colloider (MKZA6-2, Masuko Sangyo Co., Ltd., Kawaguchi, Japan) at 1,500 rpm. The pulp slurry was continuously fed to the grinder, which consisted of two stacked stone grinding disks. The manufacturer's standard grinding stones were used, and no modifications to the stone profile were made during the fibrillation process. Because all samples were prepared under identical grinding conditions, the influence of stone geometry was kept constant throughout the experiment. The gap between the two disks was adjusted to a setting of approximately -150 μm, and the NFC sample was collected after nine passes under the same operating conditions.

Wet NFC test specimen pads were prepared using a vacuum filtration system for FE-SEM observation of fiber morphology. To prevent deformation during drying, the test specimens were dried using the solvent exchange method with ethyl alcohol, acetone, and n-hexane (Stelte and Sanadi 2009; Kim *et al*. 2019). After capturing field emission scanning electron microscopy (FE-SEM; JSM-7610F, JEOL, Tokyo, Japan) images of the pads, the fiber widths of 100 individual fibrils from each NFC specimen were measured using 3D image software (MP-45030TDI; JEOL, Osaka, Japan). The particle size of NFC was determined using laser diffraction (CILAS 1090 LD analyzer, Orléans, France). Because NFC has a high aspect ratio, laser scattering is not an ideal method for directly determining fiber dimensions (Gantenbein *et al*. 2011). However, particle size data were used to indirectly indicate differences in particle size among the NFC samples (Park *et al*. 2018). The zeta potential of a 0.01% NFC suspension was measured to assess the surface charge and colloidal stability of the NFC suspension using a zeta-potential analyzer (Nano ZS; Malvern Panalytical, Malvern, UK).

Evaluation of Individual Coating Systems Using NFC and Water-based Acrylic Repellent

In this study, a two-layer barrier-coating technology was developed using NFC and a water-based acrylic repellent. Prior to fabricating the two-layer coating, the performance of each coating agent was evaluated individually to identify its intrinsic contribution to coating behavior, surface coverage, barrier performance, and mechanical properties when applied independently to the tissue paper surface. For this purpose, an NFC slurry at 1.5% concentration and a water-based acrylic repellent at 35% solids were each applied twice to the same side of the base paper under identical coating and drying conditions to enable direct comparison of coating performance. Barrier coating was carried out using a laboratory rod coater (AUTO BAR COATER, HanTech Co., Ltd., Daejeon, Republic of Korea). The coating speed was set to 70 mm/s, and rod number 28 was used. The top side of the base paper was coated using the rod coater and dried in an air dryer at 105 °C for 150 s. The paper was then passed through a cylinder dryer at 120 °C to minimize curling after drying, and this procedure was applied identically to all samples without additional pressure. After the first coating and drying step, the second coating was applied to the same side of the paper using the same coating and drying conditions. Figure 2 illustrates the detailed conditions for the individual coating systems using NFC and water-based acrylic repellent.

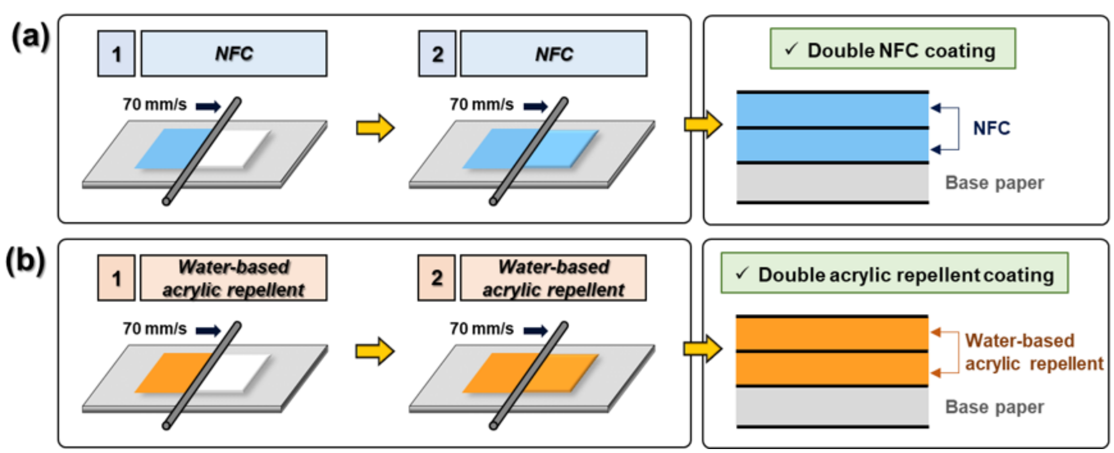

Fig. 2. Flow diagram of the individual coating systems using (a) NFC and (b) water-based acrylic repellent

The coated papers were conditioned at 23 °C and 50% relative humidity prior to testing, in accordance with TAPPI T402, under standard atmospheric conditions. Total dry coat weight (TAPPI T410 om-23 2023) and tensile strength (TAPPI T494 om-22 2022) were measured to evaluate the physical properties of the coated papers. Gurley air permeability (TAPPI T460 om-02) was measured using an air-permeability tester (4110N, Gurley Precision Instruments, USA), and water repellency was measured according to KS M 7057 (2025) to evaluate barrier properties. Surface coverage of the coated papers was examined using SEM images obtained with a field-emission scanning electron microscope (FE-SEM; JSM-7610F, JEOL, Tokyo, Japan).

Evaluation of the Multi-layer Coating System Using NFC and Water-based Acrylic Repellent

In a previous study, a multilayer barrier coating comprising two or more layers applied on one side of the paper was developed to achieve the desired barrier properties. In the present study, a two-layer barrier coating was applied using NFC and a water-based acrylic repellent to obtain reduced air permeability and improved moisture resistance in barrier-coated tissue paper. NFC was first applied once to the same side of the base paper as the precoating layer to improve surface structure and reduce pore openness. After drying under the same conditions described above, a single layer of water-based acrylic repellent was subsequently applied to the same side as the top-coating layer to provide water repellency. Each coating step was followed by air drying at 105 °C for 150 s and cylinder drying at 120 °C under identical conditions without additional pressure. Figure 3 illustrates the detailed conditions for the multilayer barrier coating, including those for each layer.

The physical and barrier properties of the barrier-coated paper, including coat weight, tensile strength, air resistance, and water repellency, were evaluated using the same methods described in the preceding section.

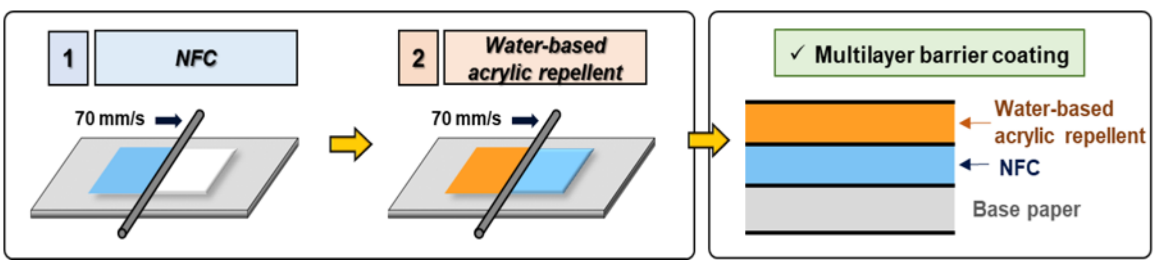

Fig. 3. Flow diagram of the multi-layer coating system using NFC and water-based acrylic repellent

RESULTS AND DISCUSSION

Effect of NFC and Water-based Acrylic Coating Systems

Table 2 summarizes the physicochemical properties of NFC prepared in this study. The material exhibited a fiber width of 38.6 ± 15.0 nm, an average particle size of 19.9 ± 1.9 µm, and a zeta potential of −17.8 ± 0.1 mV. The measured fiber width was well below the 100 nm threshold commonly used to distinguish nanofibers from microfibers, thereby confirming the nanoscale nature of the fibers. The apparent discrepancy between nanoscale fiber width and microscale particle size reflects the high aspect-ratio structure of NFC, in which individual nanofibrils form entangled fibril aggregates in aqueous suspension. The moderately negative zeta potential suggests limited colloidal stabilization, which may allow partial fibril aggregation during coating and thereby contribute to pore filling on the tissue paper surface. Collectively, these results confirm that the laboratory-prepared material was successfully produced as nanoscale NFC, making it suitable for subsequent application in tissue paper coating.

Table 2. Physical Properties of NFC Prepared in this Study

Fiber width (nm)	Average particle size (μm)	Zeta-potential (mV)
38.6 ± 15.0	19.9 ± 1.9	-17.8 ± 0.1

Table 3 summarizes the physical and barrier properties of the coated tissue papers as a function of coating agent, whereas Figs. 4 and 5 show FE-SEM images of the paper surfaces coated with NFC and the acrylic water repellent, respectively. When the tissue paper was coated with NFC, the measured dry coat weight increased from 1.46 g/m² after a single coating pass to 2.39 g/m² after two coating passes. This increase confirms that additional NFC solids were retained on the paper surface or within the porous structure during the second coating step. However, the increase in coat weight was not directly proportional to the number of coating passes, suggesting that part of the NFC suspension penetrated the highly porous tissue paper during the first coating, while the second coating was applied to a partially filled, less-absorbent surface. With increasing coat weight, both tensile strength and Gurley air resistance increased. A pronounced improvement in tensile strength was observed after the first NFC coating, followed by a further increase after the second coating, indicating cumulative mechanical reinforcement. The marked improvement after the first NFC layer indicates that pore filling and inter-fiber reinforcement occurred predominantly during the initial coating stage, whereas the additional effect of the second coating layer was relatively smaller. In addition, the NFC layer formed a relatively uniform surface structure that effectively restricted air flow and reduced air permeability. Despite these improvements in mechanical and gas-barrier properties, NFC did not impart water repellency to the paper surface. This finding is tentatively attributed to the inherently hydrophilic nature of cellulose.

In contrast, the measured dry coat weight of the water-based acrylic repellent increased from 3.45 g/m² after a single coating pass to 5.11 g/m² after two coating passes. The higher coat weight compared with NFC indicates that the acrylic repellent was retained more effectively on or near the tissue paper surface. This behavior may be related to the higher solids content of the acrylic formulation and its lower tendency to penetrate deeply into the porous fiber network compared with the NFC suspension. Although the second acrylic coating increased the coat weight, the tensile strength and Gurley air resistance changed only slightly, indicating that the acrylic repellent contributed little to mechanical reinforcement or pore filling. However, even a single acrylic coating imparted strong water repellency, which was maintained after the second coating, suggesting the formation of a stable hydrophobic surface layer.

These results indicate that NFC primarily functioned as a reinforcing and pore-filling coating, improving mechanical strength and reducing air permeability without contributing to hydrophobicity. In contrast, the water-based acrylic repellent contributed little to mechanical reinforcement, but it was effective in imparting durable surface hydrophobicity to tissue paper.

Table 3. Effect of Coating Agent Types on the Physical and Barrier Properties of Coated Tissue Paper

Coating agent	Layer number	Coat weight (g/m^2)	Tensile strength (kN/m)	Gurley air permeability (s)	Water repellency
Control (uncoated)		-	2.96 ±0.07	34.3 ± 2.4	0
NFC	1	1.46 ± 0.39	4.16 ± 0.18	94.9 ± 16.0	0
	2	2.39 ± 0.23	4.34 ± 0.31	114.6 ± 16.0	0
Acrylic water repellent	1	3.45 ± 0.03	2.99 ± 0.07	36.2 ± 2.2	8
	2	5.11 ± 0.09	3.14 ± 0.07	40.7 ± 0.9	8

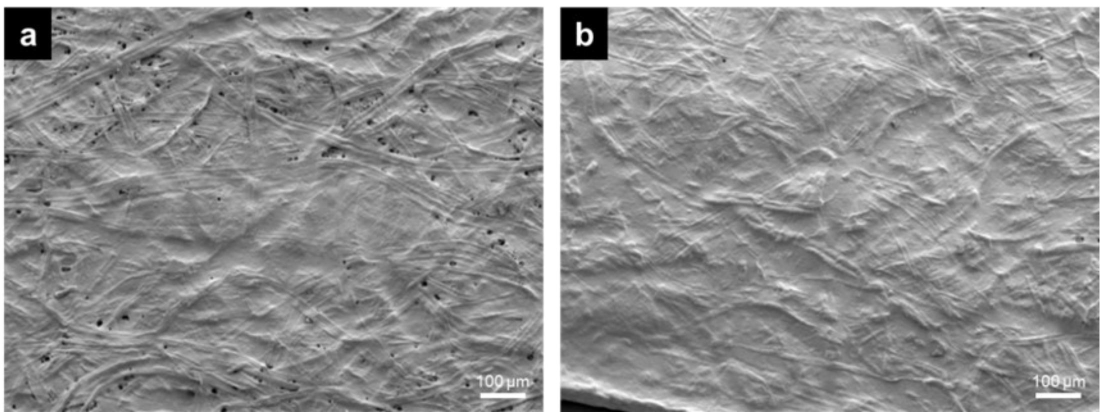

Fig. 4. SEM images of the surface of the coated tissue paper with NFC only. (a) Precoating (b) top-coating

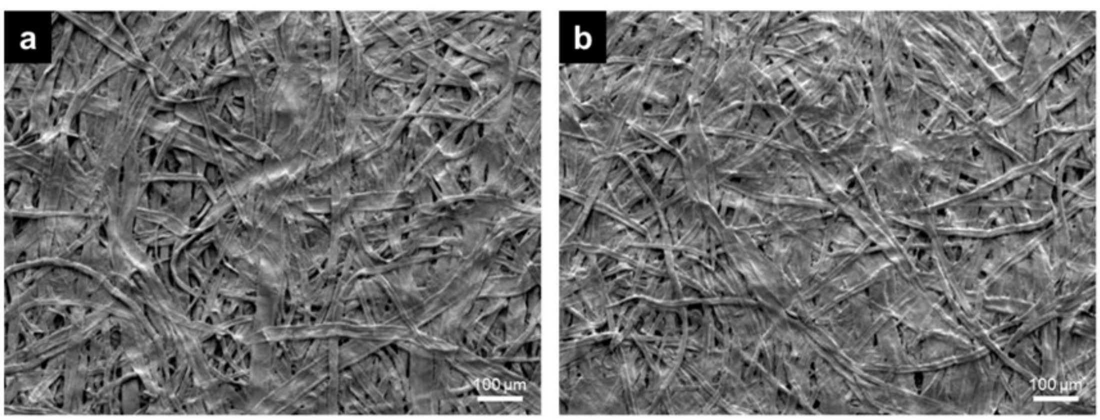

Fig. 5. SEM images of the surface of the coated tissue paper with acrylic repellent only. (a) Precoating (b) top-coating

Performance of the NFC/Acrylic Multi-Layer Coating System

The barrier performance of a two-layer coating system comprising NFC and a water-based acrylic repellent was investigated. Figures 6 through 9 present the coat weight, tensile strength, air permeability, and water repellency of tissue paper sequentially coated with NFC and the acrylic repellent.

Precoating with NFC yielded a coat weight of approximately 1.5 g/m^2, which was comparable to the values obtained for the NFC barrier coating discussed in the preceding section. This NFC layer increased tensile strength by nearly 40% relative to the uncoated base paper. This improvement can be attributed to the partial penetration of NFC into surface pores and inter-fiber voids, thereby increasing the effective bonding area between fibers and enhancing inter-fiber bonding. In addition, the NFC layer formed a relatively uniform film on the paper surface, resulting in a substantial reduction in air permeability. However, owing to the inherent hydrophilicity of NFC, no measurable water repellency was observed after NFC application. Subsequent top-coating with the acrylic repellent increased the total coat weight to approximately 4.2 g/m^2, which was slightly lower than that obtained when the acrylic repellent was applied twice consecutively in the absence of NFC. This difference suggests that the presence of the NFC underlayer influenced the deposition behavior of the polymer film.

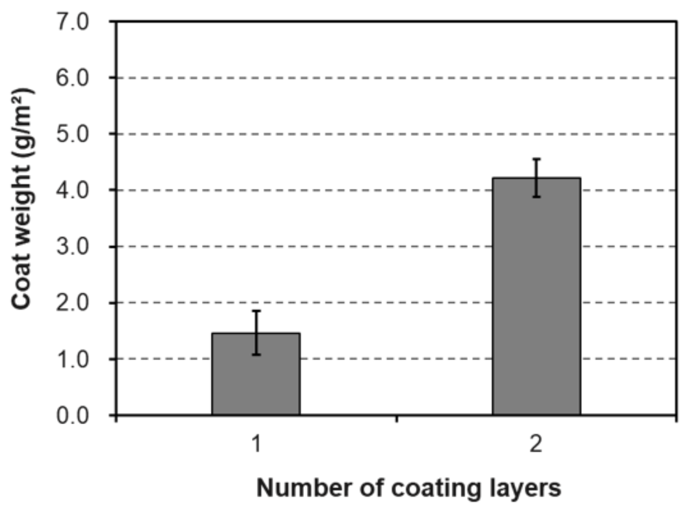

Fig. 6. Coat weight of the two-layer barrier-coated paper using NFC (1; first layer) and acrylic repellent (2; second layer on the first layer)

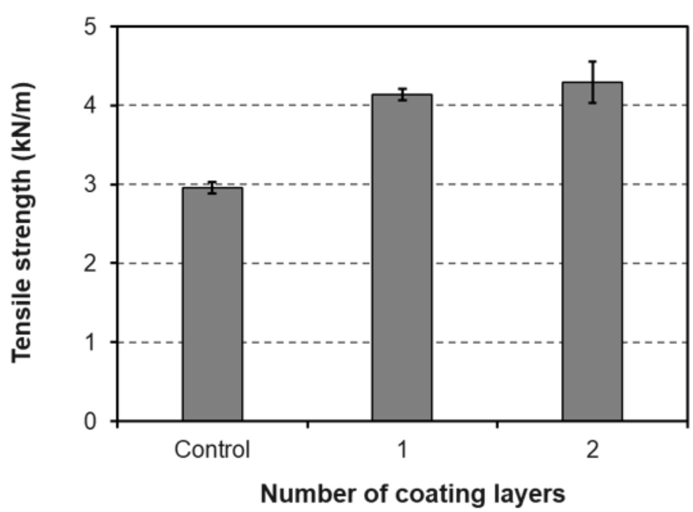

Fig. 7. Tensile strength of the two-layer barrier-coated paper using NFC (1; first layer) and acrylic repellent (2; second layer on the first layer)

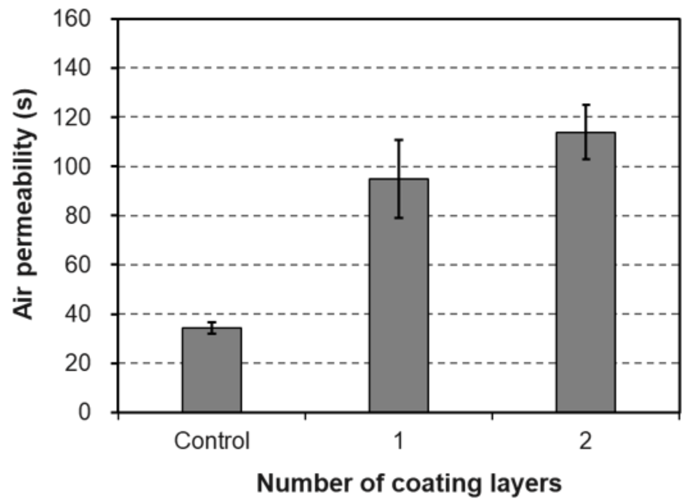

Fig. 8. Air permeability of the two-layer barrier-coated paper using NFC (1; first layer) and acrylic repellent (2; second layer on the first layer)

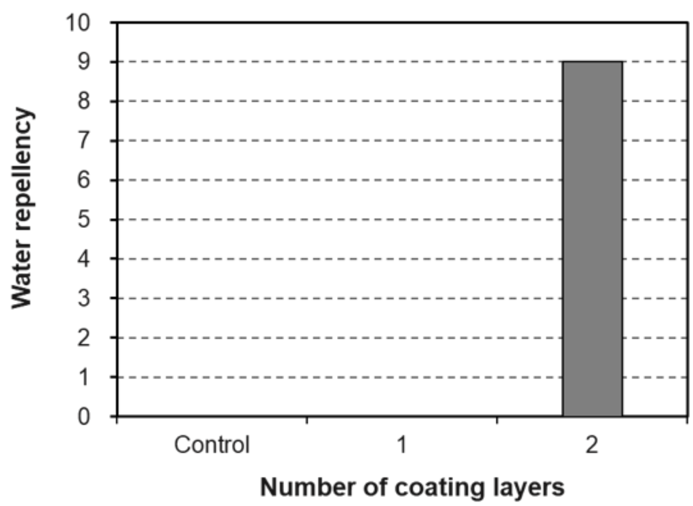

Fig. 9. Water repellent of the two-layer barrier-coated paper using NFC (1; first layer) and acrylic repellent (2; second layer on the first layer)

Despite the increased coat weight, no further improvement in tensile strength was observed, indicating that the acrylic repellent layer primarily contributed to surface sealing and hydrophobization rather than mechanical reinforcement. Nevertheless, the acrylic repellent top-coating imparted strong water repellency to the paper while preserving the enhanced tensile strength and reduced air permeability achieved by the NFC layer.

Figure 10 shows FE-SEM images of the coated paper surfaces. The FE-SEM observations support the measured physical and barrier properties of the barrier-coated tissue papers. The NFC-coated surface exhibited a nanofiber network distributed across the paper matrix, filling pores and binding fibers. After application of the acrylic repellent, the surface became denser and smoother, with the polymer film covering the NFC network and forming a continuous barrier. This appearance suggests that the morphological transformation accounts for the improved hydrophobicity and the stabilization of barrier properties.

In summary, NFC was observed to function as an effective reinforcing and pore-filling base layer, whereas the acrylic repellent provided hydrophobicity and additional surface sealing. Compared with previously reported NFC-based multilayer coatings, the present system achieved similar mechanical reinforcement while providing water repellency through a simplified two-layer coating sequence. The two-layer coating strategy, therefore, indicates potential to improve barrier performance in tissue paper used for fruit protection applications.

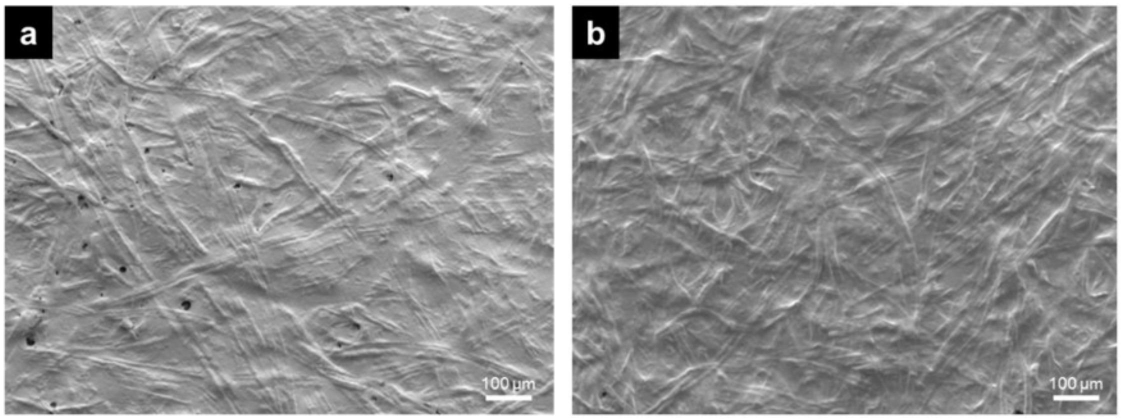

Fig. 10. SEM images of the surface of the two-layer barrier-coated paper using NFC and acrylic repellent (a: NFC layer, b: acrylic repellent on the NFC layer)

CONCLUSIONS

1. Double application of nanofibrillated cellulose (NFC) improved tensile strength and air resistance through pore filling and reinforcement of inter-fiber bonding. However, it did not contribute to water repellency because of its hydrophilic nature.
2. Double application of the water-based acrylic repellent formed a stable hydrophobic surface layer and maintained strong water repellency, while showing only limited influence on tensile strength and air resistance.
3. Sequential coating with NFC followed by an acrylic repellent combined the functions of both materials, maintaining improved mechanical strength, reduced air permeability, and strong water repellency, indicating its potential as a barrier treatment for tissue paper used in fruit protection applications.

ACKNOWLEDGMENTS

This work was supported by the Technology Development Program (Project No. RS-2023-00268756) funded by the Ministry of SMEs and Startups (MSS, Korea).

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Article submitted: January 30, 2026; Peer review completed: April 14, 2026; Revised version received: April 21, 2026; Accepted: June 22, 2026; Published: July 2, 2026.
DOI: 10.15376/biores.21.3.7690-7702