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Improving Hydrophobicity and Oil Barrier Performance of Paper by Coating with PVA/Nanocellulose-Based Suspension

Peningkatan Hidrofobisitas dan Kemampuan Penghalang Minyak Kertas melalui Pelapisan dengan Suspensi Berbasis PVA/Nanoselulosa

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ABSTRAK

Kajian ini menyelidiki penggunaan nanoselulosa dan suspensi polivinil alkohol (PVA) sebagai formulasi pelapis pada substrat kertas dan pengaruhnya terhadap sifat-sifat penghalang minyak dan air pada kertas. Suspensi pelapis PVA/nanoselulosa dibuat melalui prosedur pencampuran nanoselulosa dengan PVA pada berbagai konsentrasi. Pelapisan dilakukan dengan mendepositkan bahan perekat dan beberapa lapisan suspensi nanoselulosa/PVA pada permukaan kertas dengan menggunakan pelapis batang. Hasil-hasil penelitian menunjukkan bahwa pelapisan hingga lapisan keempat menurunkan indeks opasitas dan tingkat permeabilitas udara terhadap kertas yang dilapisi, tetapi ada sedikit peningkatan pada permeabilitas udara pada lapisan keenam. Deposisi suspensi PVA/nanoselulosa pada permukaan kertas juga meningkatkan kinerja penghalang air dan minyak. Sudut kontak (CA) tertinggi sebesar 82° dan nilai oil kit sebesar 14 diperoleh pada lapisan keempat menggunakan suspensi dengan kandungan 2% PVA, dibandingkan dengan kertas yang tidak dilapisi (CA 43°). Menarik untuk dicatat bahwa pelapisan kertas menggunakan suspensi nanoselulosa/PVA dapat secara efektif meningkatkan hidrofobisitas dan kinerja penghalang minyak pada kertas. Hasil-hasil yang memuaskan pada sifat-sifat kertas setelah dilapisi dengan suspensi nanoselulosa/PVA dapat berpotensi menjadi bahan yang ramah lingkungan dalam penggunaan pengemasan makanan.

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ABSTRACT

This study investigates the use of nanocellulose and polyvinyl alcohol (PVA) suspension as a coating formulation on a paper substrate and its effects on the paper's oil and water barrier properties. The PVA/nanocellulose coating suspension was prepared via a simple nanocellulose mixing procedure with various concentrations of PVA. The coating was carried out by depositing an adhesive agent and multiple layers of nanocellulose/PVA suspension on the paper surface using a bar coater. The results showed that coating up to the fourth layer decreased the opacity index and air permeability rate of the coated papers, but there was a slight increase in the air permeability at the sixth layer. The deposition of PVA/nanocellulose suspension on the paper surface also increased the water and oil barrier performance. The highest contact angle (CA) of 82° and oil kit value of 14 was obtained at the fourth layer of coating using suspension with 2 wt% of PVA content, as compared to the uncoated paper (CA 43°). It is interesting to note that coating paper using nanocellulose/PVA suspension can effectively enhance the hydrophobicity and oil barrier performances of the paper. This satisfactory results in paper properties after being coated with nanocellulose/PVA suspension can be a potential environmentally friendly material in food packaging applications.

1. INTRODUCTION

1.1 Background

In the past decade, there has been a concerted global effort to replace non-biodegradable plastics with more environmentally friendly materials, particularly in the packaging industry (Francisco *et al*., 2020). This shift has been driven by both academic and industrial spheres, as the need to address the environmental impact of plastic waste has become increasingly urgent. The European Safety Gate data from 2022 highlights the severity of the issue, with 35% of recalled and banned products being due to environmental risks (European Commission, 2023). This has led to the implementation of more stringent regulations on chemicals and goods as governments and regulatory bodies attempt to mitigate the negative environmental consequences of certain materials. One such material of concern is paper-polyethylene laminates, which are commonly used in packaging applications that require a barrier layer, such as ethylene vinyl alcohol (EVOH). The challenge of delamination makes it difficult to recycle and compost these paper-based materials, although some advancements have been made in the development of peelable laminates in recent years (Sangroniz *et al*., 2019). Despite these efforts, the task of creating paper-based materials with barrier properties equivalent to those of plastic laminates remains a significant challenge for paper manufacturers and researchers (Shen *et al*., 2019). The need to develop sustainable, environmentally friendly packaging solutions that can match the performance of traditional plastic-based materials is a pressing concern that continues to drive innovation and research in this field (Yook *et al*., 2020).

The increasing environmental concerns and the need for sustainable solutions have led to a growing interest in non-toxic and biodegradable materials as alternatives to traditional plastics in paper packaging. This transition encompasses both the materials used and the production processes involved (Li *et al*., 2021). On the materials front, biobased and biodegradable macromolecules, such as cellulose, chitosan, and starch, have emerged as promising options (Salmah & Faisal, 2010; Idumah *et al*., 2022). These biomaterials exhibit excellent properties and are highly compatible with paper, making them suitable for various packaging applications. Their oil-repellent capabilities, for instance, can help reduce porosity, enhancing the overall performance of the packaging materials. However, it is important to note that the barrier performance of these biomaterials against water permeation is reportedly inferior to that of polyvinyl alcohol (PVA), a synthetic and biodegradable polymer that is frequently used as a surface coating material in packaging papers. This suggests that a combination of natural and synthetic materials or the development of novel biomaterials with enhanced water resistance may be necessary to achieve the desired performance characteristics in paper packaging.

Polyvinyl alcohol (PVA) is a biodegradable polymeric substance that exhibits strong adhesion, a smooth texture, and resistance to oil and solvents. This environmentally benign polymer can be broken down by microorganisms into water and carbon dioxide (Gottberg *et al*., 2019; Bolto *et al*., 2009). Consequently, PVA has been extensively utilized in paper production as an eco-friendly water barrier material. The application of PVA coating significantly enhances the oil resistance of paper; regrettably, the hydrophilic nature of PVA has an adverse impact on the paper's hydrophobicity (Tang & Alavi, 2011).

Nanocellulose is a cellulosic material with at least one dimension less than 100 nanometers derived from the chemical or mechanical processing of plant fibers (Govindan *et al*., 2014). Numerous studies have reported the use of nanocellulose as a sizing agent in coating formulations, enhancing the hydrophobicity, oil resistance, and surface strength of paper, resulting in a lower Cobb index and reduced roughness (Belbekhouche *et al*., 2011). The combination of nanocellulose and polyvinyl alcohol (PVA) may produce a coating agent with superior barrier performance on paper (Schmid *et al*., 2014). Previous investigations by Mazega *et al*. (2022) have revealed a strong affinity and excellent adhesion between cellulose nanofibrils (CNFs) and PVA molecules in composite films. Furthermore, Huang *et al*. (2022) demonstrated that increased CNFs loading can effectively enhance the lipophobicity, hydrophobicity, and tensile strength of PVA-coated paper.

To create a high-performance, multi-layered coated paper with excellent barrier properties, it is crucial to ensure strong adhesion between the coating formulation and the paper substrate. One potential approach to improving this adhesion is by depositing an adhesive agent between the coating mixture and the paper surface. It is well-established that poor adhesion can lead to detrimental effects on the barrier characteristics of the coated paper, as well as increase its vulnerability to environmental stresses and degradation (Tarrés *et al*., 2018). The application of a coating method that involves the deposition of alternating layers of adhesive agent and coating suspension can enhance the performance of biopolymer-based coatings, ultimately resulting in a final product with superior barrier properties.

1.2 Objectives

The purpose of this study is to develop a coating suspension composed of nanocellulose and polyvinyl alcohol (PVA). The barrier characteristics of the paper were assessed in relation to the PVA concentrations within the suspension, as well as the number of coating layers applied, in order to determine the optimal processing method and the ideal ratio of nanocellulose to PVA that yields the most effective barrier properties.

2. METHODS

2.1 Materials

Calendered uncoated base paper with an approximate grammage of 26 $g.m^{-2}$ was provided by PT Bukit Muria Jaya (Indonesia). Nanocellulose suspension (2 wt% of solid content) was supplied by Borregaard (Norway). Polyvinyl alcohol (PVA) with an Mw 30–70 kDa and an adhesive were purchased from Sigma-Aldrich (Schnelldorf, Germany).

2.2 Preparation of Multilayered Coatings

2.2.1 PVA/Nanocellulose Suspension

PVA/nanocellulose-based suspension was prepared via a nanocellulose-mixing procedure with various concentrations of PVA. An aqueous solution of PVA was prepared by adding an amount of PVA (1, 2, 3% by weight) in 100 ml of aquadest. The mixture was heated at 90 °C while kept vigorously stirring for 1 h until a clear solution of PVA was obtained. Afterward, 1 wt% of nanocellulose was mixed into the PVA solution under agitation for 1 h, which then resulted in a PVA/nanocellulose suspension. In the meantime, an adhesive solution was prepared by diluting 1 wt% of the adhesive into 100 ml of aquadest at room temperature. Before use, all the coating suspensions and adhesive solution were stored overnight in a desiccator.

2.2.2 Coated Paper Preparation

The PVA/nanocellulose suspensions were applied to the paper surface using a bar coater. The adhesive solution was initially deposited on the paper, followed by the application of multiple layers of the PVA/nanocellulose suspension. Between each layer, the papers were maintained at a temperature of 40 °C for a minimum duration of 10 minutes to facilitate drying. The coated papers were prepared with 1, 2, 4, and 6 layers of the adhesion agent and PVA/nanocellulose suspension, with the single-layered coated paper consisting solely of the PVA/nanocellulose suspension. Figure 1 illustrates the samples with varying numbers of multiple layers.

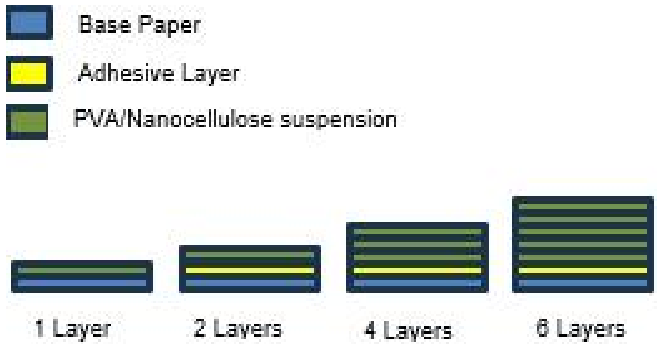

Figure 1. Sample structures at different numbers of multiple layers

2.3 Characterizations

Water contact angle measurements were conducted using the sessile drop technique (Kruss DSA 100 goniometer, France) under ambient conditions. Three to five water droplets of 0.8 μL were deposited on each film, and the average water contact angle was calculated.

Kit tests were carried out to evaluate the oil resistance of uncoated and coated papers, following the TAPPI procedure T559 (TAPPI, 2020). The highest number corresponding to the most aggressive mixture of castor oil, toluene, and heptane that the paper resisted is reported as the "kit rating".

Air resistance was estimated in accordance with the ISO standard 5636/5 (ISO TC/6, 2011). The time taken for 100 mL of air to pass through a 6.45 cm^2 cross-sectional area, driven by a pressure gradient of 1.22 kPa, was measured. The opacity (whiteness) of the uncoated and coated paper sheets was measured using an opacity meter (Technidyne).

The surface morphology and cross-sectional area of uncoated and coated papers were examined using a scanning electron microscope (SEM), specifically the JEOL model JSM 6260 LE. The specimens' surfaces and cross-sections were mounted on aluminum stubs and sputtered with palladium to prevent electrostatic charging during the analysis.

Fourier transform infrared spectroscopy (FTIR) was conducted on the uncoated and coated papers in ATR (attenuated total reflection) mode, utilizing the Perkin Elmer 1600 series instrument. The samples were scanned from 400 to 4000 cm^{-1} with a resolution of 4 cm^{-1}. Additionally, the weight, thickness, and grammage of all the sheets were measured using a digital micrometer.

3. RESULTS AND DISCUSSION

3.1 Hydrophobic Properties

The concept of hydrophobicity generally refers to the ability of a material surface to maintain contact with water droplets. This characteristic is determined by measuring the contact angle between the solid surface and the water droplet. Based on the size of the contact angle, materials can be classified as super-hydrophilic, hydrophilic, hydrophobic, or super-hydrophobic (Jang *et al*., 2023). Figure 2 illustrates the relationship between the coating layers and the water contact angle of the coated papers. The increase in the number of coating layers effectively enhanced the water contact angle (CA) of the coated papers up to the fourth layer, after which it declined at the sixth coating layer. The deposition of PVA/nanocellulose suspensions significantly improved the water barrier performance of the coated papers, as evidenced by a higher contact angle than the uncoated paper (CA 43.2°). It is believed that the nanocellulose interacted with PVA through hydrogen bonding, which is often assumed to generate ester linkages. The hydroxyl oxygen of PVA can act as a hydrogen bond acceptor towards nanocellulose, resulting in fewer available hydroxyl groups for hydrogen bonding with water molecules, as they are partially attached to the paper surface. The highest contact angle was observed on the coated papers with four coating layers (CA 82.3°). However, the excessive application of coating layers had a detrimental effect on the water barrier properties, likely due to the formation of internal cracks within the paper structure caused by the multiple drying processes.

The existence of an adhesive agent resulted in the surface of the coated paper sheets being consistently more hydrophobic than the coated paper without an adhesive agent (one-layer coated paper). However, the increased polyvinyl alcohol (PVA) content in the suspension did not significantly increase the contact angle values of the coated papers with a similar number of layers. The interaction between nanocellulose and PVA in the suspension may have contributed to the increase in suspension viscosity due to the formation of intermolecular bonds between the hydrophilic part of PVA and the hydroxyl groups of the nanocellulose. The high amount of PVA in the suspension could have led to improved viscosity, which adversely affected the effectiveness of the coating uniformity. Consequently, the water barrier performance decreased in each coated paper with various layer numbers. Overall, the decrease in the contact angle may be attributed to the deposition of the PVA/nanocellulose suspension physically changing the topography of the paper surface and the hydrophilic character of the nanocellulose resulting in higher liquid-solid adhesive forces (Aulin & Ström, 2013).

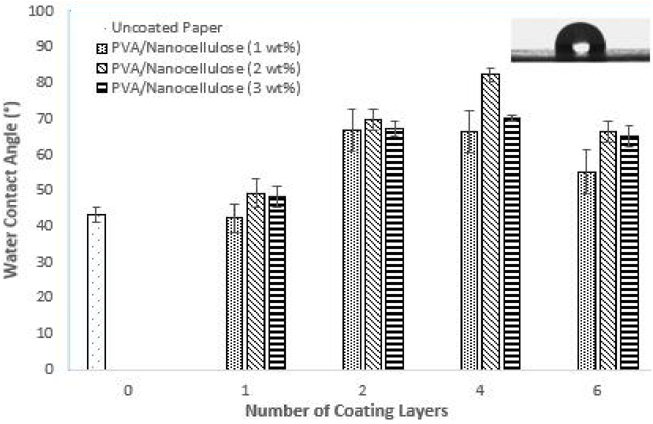

Figure 2. The water contact angle of coated papers as a function of the number of coating layers and PVA content in the nanocellulose suspension

3.2 Oil Resistance Performance

Oil barrier properties are defined as the ability of a material surface to resist an oil permeation. Figure 3 exhibits the oil kit rating of coated papers as a function of multiple layer numbers and PVA concentration in the nanocellulose suspension. It can be seen the increasing number of coating layers is imparted to the oil resistance performance of the coated papers. For one-layer coated paper, the kit rating was in the range of 6 to 8 for all PVA/nanocellulose suspension, which was considerably higher than the uncoated paper, which had a kit rating of 5. It seemed that the presence of nanocellulose suspension on the paper surface had changed the topography and surface chemistry of the paper, enhancing the oleophobic character. Upon the increase in layer numbers, the coated papers' kit rating increased to 14 at the fourth layer, then dramatically decreased at the sixth layer of coatings. This decreased kit rating at the sixth layer might be due to surface damage and internal cracks of the paper induced by multiple drying between each layer. It seemed the optimal kit rating was obtained at four-layer coatings.

The application of PVA/nanocellulose suspension on the coated paper demonstrated a positive impact on the oil barrier performance. At the same coating layers, the papers coated with 2 wt% PVA containing nanocellulose suspension exhibited the highest kit rating, indicating superior oil resistance. Notably, adding an adhesive agent layer further improved the oil barrier performance of the coated papers compared to the uncoated and coated papers without the adhesive agent. This suggests that the synergistic combination of PVA/nanocellulose suspension and the adhesive agent contributed positively to the paper's ability to repel oils. In other words, these components collaborated to seal the pores of the paper. Importantly, they did not become solvated by oil or non-polar solvents, and their intermolecular interactions with oils, such as dispersive forces, did not supersede their strong hydrogen bonding (Tarrés *et al*., 2016).

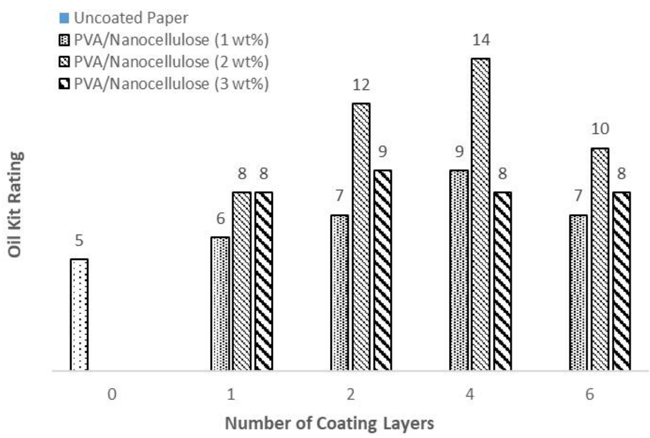

Figure 3. Oil kit rating of coated papers at different multiple layers

3.3 IR Spectra Analysis

Figure 4 shows an analysis of the IR spectra of uncoated and coated paper containing different PVA concentrations and nanocellulose. It can be seen the IR spectrum of uncoated paper exhibited a typical spectrum of cellulose where the wavelength peak that was located at 3330 cm^{-1} corresponded to a vibrational stretch of hydroxyl (-OH) functional groups of cellulose from the paper, absorption peak at 2895 cm^{-1} assigned to C-H stretching of cellulose from the paper, while the others absorption peaks located at 1160 cm^{-1}, 1016 cm^{-1}, and 869 cm^{-1} corresponded to stretch vibration of ether linkage, pyranose ring and glycosidic bonds of the cellulose from the paper, respectively (Hospodarova *et al*., 2018). The deposition of PVA suspension on the paper surface did not significantly affect the paper's IR spectrum change.

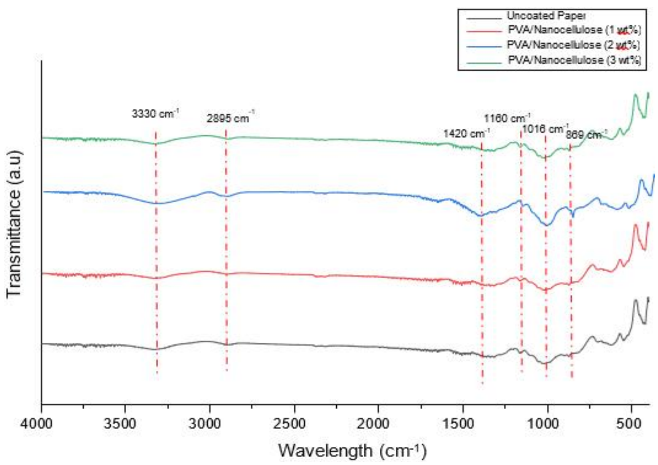

Figure 4. FTIR spectra of coated papers at varying content of PVA suspensions

All IR spectra of the coated papers exhibited almost a similar pattern, except for the coated paper containing PVA suspension of 2 wt%. There was a significant broad absorption at 3330 cm^{-1} that might be attributable to the existence of OH- groups of the PVA attached to the paper surface. Also, a strong absorption peak appeared at a wavelength peak of 1420 cm^{-1}, corresponding to the stretch vibration of -C-O- bonds (Lapuz *et al*., 2022). It is thought that the appearance of this absorption peak might be due to the presence of cellulosic structure from the nanocellulose on the paper surface, forming a hydrogen bridging. Furthermore, a narrow absorption peak at 869 cm^{-1} of the IR spectrum of the coated paper with PVA suspension of 2 wt% might indicate the existence of a higher content of glycosidic bonds. This finding supported the good performance of the coated paper with PVA suspension of 2 wt% against oil and water permeation.

3.4 SEM Micrograph Analysis

The surface characteristics of uncoated and one-layer coated paper samples using scanning electron microscopy (SEM) images are provided in Figure 5. The uncoated paper surface exhibited a rough texture, with cellulose fibers creating small voids along the surface, resulting in a highly porous structure (Figure 5A). In contrast, the coated paper surface showed a smoother appearance with fewer voids (Figure 5B), indicating that the coating formulation, containing a 2 wt% polyvinyl alcohol (PVA) suspension, effectively covered the paper surface and reduced its porosity. The cross-section of the coated paper (Figure 5C) revealed that the coating distribution was uneven along the surface. This suggests that the PVA/nanocellulose suspension was not homogeneously distributed on the paper, which could potentially impact the barrier properties of the coated paper against oil and water permeation. The analysis of the SEM images provides valuable insights into the surface morphology and coating characteristics of the paper samples. The smoother surface and reduced porosity observed in the coated paper suggest that the PVA/nanocellulose coating can potentially enhance the barrier performance of the paper. Still, the uneven coating distribution may pose a challenge in achieving consistent and effective barrier properties across the entire surface.

3.5 Other Properties

Incorporating nanocellulose and polyvinyl alcohol (PVA) as a coating agent, along with the increased number of coating layers, significantly enhanced the air resistance of paper sheets. As shown in Figure 6, the air resistance of the uncoated paper (3.35 $ml/cm^2.min$) was improved by approximately two to three orders of magnitude. The increase in the number of coating layers significantly enhanced the air resistance performance up to the fourth layer, after which it declined at the sixth layer. When the number of coating layers was similar, the coating suspension containing nanocellulose and 2 wt% of PVA exhibited the highest air resistance (0.43 $ml/cm^2.min$), suggesting the optimal coating formulation for air blocking on the paper sheets. The enhanced air-blocking mechanism on the paper sheets can be attributed to the synergistic intermolecular interaction between the crystalline part of the polymer and the highly hydrated nanocellulose, which impeded the intraparticle diffusion of air (N_2 and O_2) (Mo *et al*., 2022).

Table 1 tabulates the characterization of the physical properties of coated papers as a function of PVA content and the number of multiple layers. It can be seen that the deposition of PVA/nanocellulose coating suspension at different numbers of layers increased the thickness and opacity (whiteness) of paper sheets as compared with the uncoated paper. However, the paper sheets coated with multiple layers and PVA suspension content exhibited almost similar thickness. Surprisingly, the increased number of coating layers and PVA content apparently did not contribute to the increase in thickness, even though the coating grammage was enhanced considerably.

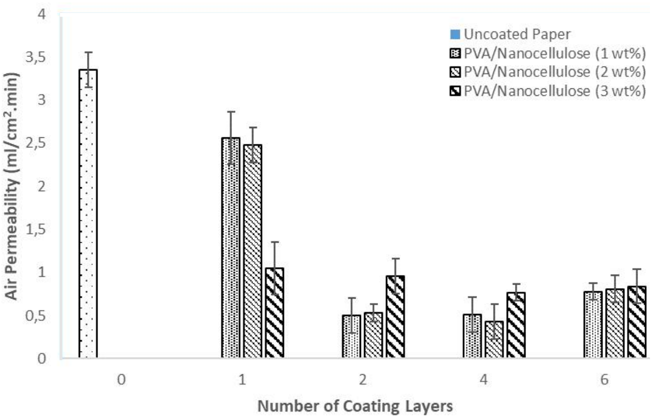

Figure 6. Effects of multiple coating layers and PVA content on air permeability of coated papers

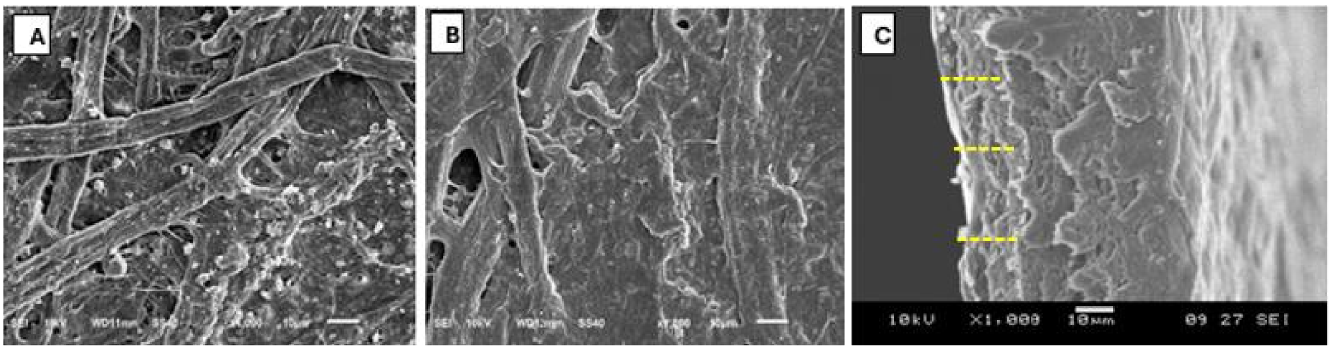

Figure 5. SEM micrographs: (A) uncoated paper, (B) one-layer coated paper with PVA suspension of 2 wt%, (C) cross-section coated paper containing PVA suspension of 2 wt%

Table 1. Some physical properties of coated papers as a function of PVA content and the number of multiple layers

	Physical Properties		
	Thickness (μm)	Opacity (%)	Coating Grammage (g/m^2)
Uncoated Paper	40.1	47	-
Coated Papers with PVA/Nanocellulose Suspension (1 wt%)			
1 Layer Coating	49.7±0.1	52.7±0,4	0.18±0.6
2 Layer Coating	50.3±0.2	52.4±0.3	0.24±0.2
4 Layer Coating	50.3±0.1	51.7±0.2	0.51±0.3
6 Layer Coating	51.6±0.3	50.9±0.1	1.22±0.4
Coated Papers with PVA/Nanocellulose Suspension (2 wt%)			
1 Layer Coating	48.7±0.3	53.8±0.3	0.27±0.5
2 Layer Coating	49.1±0.2	52.6±0.2	0.40±0.4
4 Layer Coating	49.6±0.2	52.5±0.3	0.68±0.2
6 Layer Coating	52.2±0.4	51.6±0.1	1.48±0.3
Coated Papers with PVA/Nanocellulose Suspension (3 wt%)			
1 Layer Coating	48.4±0.7	53.5±0.3	0.28±0.4
2 Layer Coating	50.5±0.5	51.6±0.3	0.92±0.3
4 Layer Coating	50.8±0.7	50.2±0.2	1.22±0.5
6 Layer Coating	51.4±0.3	49.6±0.5	1.79±0.2

4. CONCLUSION

The multiple coating layers comprised of polyvinyl alcohol (PVA)/nanocellulose suspension and an adhesive agent had been applied on paper sheets to investigate their water barrier performance and oil resistance properties. The results showed that the deposition of PVA/nanocellulose suspension on the paper surface increased the water and oil barrier performance. The highest contact angle (CA) of 82° and oil kit value of 14 was obtained at the fourth layer of coating using suspension with 2 wt% of PVA content, compared to the uncoated paper (CA 43°). This indicated that coating paper using nanocellulose/PVA suspension could effectively enhance the hydrophobicity and oil barrier performances of the paper. On the other hand, the coating up to the fourth layer decreased the opacity index and air permeability rate of the coated papers. The paper sheets coated with multiple layers and PVA suspension content exhibited almost similar thickness. The increased number of coating layers and PVA content apparently did not contribute to the increase in thickness, even though the coating grammage was enhanced considerably. Conclusively, the results on paper properties after being coated with nanocellulose/PVA suspension could be a potential environmentally friendly material in food packaging applications.

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