

Physicochemical Characterization of Plant Oil-based Coatings and the Resultant Coated Papers Intended for Food Packaging

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ABSTRACT

The trend of take-out food consumption continues to rise, raising concerns about ensuring that packaging is safe for consumers and the environment. Paper is a widely used packaging material; however, it must be coated to make it impermeable to water and oil. An option for a safe and biodegradable coating material is plant oil. In this study, various oils (candlenut, linseed, palm, olive, soybean, and walnut) were applied to papers and then heated to induce oxidative polymerization. The oil-coated papers were analyzed for their resistance to oil and water, coating mass and thickness, morphology, texture, Fourier transform infrared (FTIR) spectroscopy profile, and X-ray diffraction (XRD) profile. Candlenut oil-, linseed oil-, soybean oil-, and walnut oil-coated papers were impermeable to water and oil. Differences were observed in the oils' viscosities and the papers' dry coating mass, morphology, puncture force, puncture distance, tensile force, and elongation distance, as well as their FTIR and XRD profiles. Morphological data indicated that oils with higher polyunsaturated fatty acid content showed more cross-linking. The coated papers demonstrated mechanical properties comparable to or better than those of uncoated paper. These findings suggest that candlenut, linseed, soybean, and walnut oils are promising candidates as lining materials for paper used as take-out food packaging. *Prog Color Colorants Coat. 20 (2027), 299-315* © Institute for Color Science and Technology.

1. Introduction

The trend of ordering takeout and having meals delivered to homes continues to rise. These options reduce the time and energy required for food preparation and provide the convenience of enjoying meals outside of restaurants. The National Restaurant Association of the United States reported that 75 % of restaurant traffic consists of take out orders [1]. This trend has been increasing for some time, with the

COVID-19 pandemic contributing to its rise. During the pandemic, when public gatherings, including those at restaurants, were restricted, this trend provided an effective solution.

An essential aspect of this trend is the use of food containers. Various materials are used for take-out food packaging, including plastic, paper/cardboard, metal, and glass. Paper and cardboard are widely selected for their numerous advantages, including

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affordability, lightweightness, and foldability. Paper packaging is mostly coated to make it impermeable to water and oil, which are commonly present in foods. Many manufacturers use synthetic petroleum-based polymer coatings, such as polyethylene, which compromise paper biodegradability and pose health risks [2]. These health risks are linked to microplastic ingestion and plasticizer migration. The total annual uptake of microplastics, including inhalation and consumption, can reach up to 121,664 particles per person, with primary sources being air, bottled water, and seafood [3]. Although approximately 90 % of ingested microplastics may be excreted via feces, those that remain in the body are concerning. They may induce an inflammatory response, exhibit size-dependent toxicity, leach toxic monomers and additives, and disrupt the gut microbiome [4]. Microplastics contain various additives, including plasticizers, flame retardants, pigments, antimicrobial agents, heat stabilizers, ultraviolet stabilizers, and fillers. They may cause localized particle toxicity and cumulative chronic effects upon absorption into the human body [4]. In addition, microplastics can adsorb toxic compounds from their environments, including persistent organic pollutants and pesticides, and transport these toxins to humans and animals [5]. Given the harmful effects of plastic coatings on food packaging, it is crucial to identify alternative materials that are both safe and environmentally friendly.

Plant oils may serve as suitable alternatives to address this issue. They are fully degradable, safe,

renewable, and affordable. Their lipids, particularly polyunsaturated fatty acids (PUFAs), can form a polymer network that acts as a barrier to liquid penetration. Polymerization may proceed via several mechanisms, including the Diels-Alder reaction, the formation of ether and ester bonds linking triacylglycerol monomers [6], and epoxidation followed by acrylation. The epoxidation of unsaturated fatty acids is mediated by peroxides or peracids. The subsequent step, acrylation, involves ring opening with acrylic acid to generate a β -hydroxyl derivative [7]. Another study demonstrated that, in the presence of lipase and hydrogen peroxide, unsaturated and/or saturated fatty acids (SFAs) can form peroxyacids that act as epoxidizing agents [8]. The rate of the epoxidation reaction is influenced by temperature, with higher temperatures accelerating the accumulation of oxiranes [9].

Some plant oils with varying fatty acid compositions have been investigated for their ability to form layers impermeable to water and oil. The selected oils were extracted from candlenut (*Aleurites moluccanus*), linseed (*Linum usitatissimum*), soybean (*Glycine max*), and walnut (*Juglans regia*) seeds, as well as from olive (*Olea europaea*) and oil palm (*Elaeis guineensis*) mesocarp. The fatty acid compositions of these oils are presented in Table 1. Candlenut, linseed, soybean, and walnut oils contain a high percentage of PUFAs, while olive and palm oils have lower PUFA concentrations. Oils with high PUFA content are expected to successfully develop polymer layers.

Table 1: Fatty acid compositions (expressed as % peak area) of various oils used in the current study.

Oil source	Fatty acid (% peak area)						
	C14:0	C16:0	C18:0	C18:1	C18:2	C18:3	PUFA
Candlenut[10]	-	6.51	2.53	26.35	39.71	25.00	64.71
Linseed[11]	-	4-6	2-3	10-22	12-18	56-71	68-89
Olive[12]	-	16.33	2.88	56.70	17.10	0.89	17.99
Palm [13]	2.50	58.80	6.00	24.70	7.60	-	7.60
Soybean [14]	-	10.33	3.53	21.23	54.96	6.25	61.21
Walnut [15]	-	6.06	2.62	14.10	61.70	15.00	76.70

Previous studies on natural and compostable coatings for paper have used a combination of beeswax and linseed oil [16], a combination of carboxymethyl chitosan with carboxymethyl cellulose (CMCS/CMC) or sodium alginate (CMCS/SA) [17], a mixture of beeswax and carnauba wax [18], as well as beeswax, biowax, soywax, and emulsion wax [19]. Papers coated with beeswax, biowax, soywax, or emulsion wax exhibited high water contact angles (WCAs) of 105.28–112.82°, but only a slight improvement was noted compared to uncoated paper (98.48°). Conversely, these coatings produced a 21–35 % increase in oil contact angles relative to the control; however, the values remained relatively low (52.07–57.8°) [19]. The WCA of the paper coated with acrylated epoxidized linseed oil (AELO) without beeswax was approximately 87.3°, which increased to 111.3° upon the addition of beeswax to the AELO coating [16]. However, the effectiveness of this coating in preventing oil absorption into the base paper was not reported. Meanwhile, the maximum water and oil contact angles of CMCS/SA-coated paper were approximately 70° and 43°, respectively, while those of CMCS/CMC-coated paper were 72° and 47°, respectively [17]. Although these innovations are promising, further improvements are desirable. In addition, the use of beeswax may be discouraged for packaging intended for vegan foods, whereas the addition of emulsion wax, which contains paraffin, does not meet the requirements for natural and fully compostable products. In contrast, plant oils fully comply with natural, degradable, and vegan requirements. They are also affordable and can be extracted without harmful solvents, for instance, by using a screw press. While the application of plant oils as water barriers for paper coatings is not novel, the simpler material preparation technique in this study may facilitate greater industrial adoption. In this study, the plant oil is not pretreated (e.g., epoxidized or acrylated) [20], dissolved in solvents, added with cross-linking agents [21], or transvinylated [22], thereby potentially reducing production costs compared to previous methods. In addition, the absence of additives or processing aids, such as cross-linkers and solvents, contributes to a more sustainable and safer production process. Notably, previous studies using unmodified plant oils for paper coatings have rarely been systematically and thoroughly investigated. Unlike some earlier research, the present study employs multiple characterization techniques to evaluate the coated paper,

including assessments of water and oil penetration (leakage and contact angle), chemical analysis (Fourier transform infrared (FTIR) spectroscopy), morphology (scanning electron microscopy (SEM)), mechanical properties (tensile and puncture tests), and physical analysis (X-ray diffraction (XRD)).

The objectives of this study were to identify plant oils that could form water- and oil-resistant coatings suitable for food packaging papers and to provide the physicochemical characteristics of the coated papers. A permeation test was conducted to assess water and oil resistance. A contact angle test was also performed to evaluate water resistance. The physicochemical characteristics analyzed included coating mass and thickness, morphology, strength, elasticity, as well as FTIR and XRD profiles. Additionally, the viscosity of the oils was examined to determine its relationship with the properties of the coated papers.

2. Experimental

2.1. Sample preparation

Linseed and olive oils were sourced from Ar-Rozy International (Indonesia) and Indus Chemika (Indonesia), respectively. Meanwhile, walnut, soybean, and candlenut oils were extracted from the seeds using an oil expeller (Shanyou Machinery, Guangzhou, China), and the resulting slurry was centrifuged at 4,000 rpm for 10 min. Red palm oil was extracted using a similar method, but from the mesocarp of the oil palm.

White paperboard, 0.22 ± 0.00 mm thick, was cut into 15×15 cm pieces. The paper was coated with walnut, linseed, soybean, candlenut, olive, or red palm oil using a brush until the entire surface was fully covered. Once it was fully coated, no additional layers were applied. The coating was applied only on one side of the paper. For each type of oil, three coated paper samples were prepared. The coated paper was then heated in an oven at 60°C until the coating was completely dry.

2.2. Viscosity of the oils

The oil viscosity was determined using an m-VROC viscometer (RheoSense, San Ramon, CA, USA). Approximately 100 μ L of each sample was pipetted into a syringe, which was then connected to a measurement chip. The chip temperature was set to 25°C. The samples were subjected to a shear stress of 21 Pa, and the

apparent viscosities were recorded. Each measurement was repeated five times. The chip was cleaned after each sample.

2.3. Mass and thickness of the coatings

The mass/thickness of the coating was determined by subtracting the mass/thickness of the uncoated paper from that of the coated paper. The coating mass was recorded both before and after drying using an analytical balance, while the thickness was measured only after drying using a micrometer screw gauge. The paper weight was recorded prior to oil application, and then weighed again after the oil was applied. The coated paper was reweighed after drying. All measurements were performed in triplicate.

2.4. Water and oil resistance of the papers

A 50 μL drop of water or oil was placed on the coated surface of the samples with dimensions of 3×3 cm. The bottom surface of the samples was monitored for signs of water or oil leakage for up to 60 min. The time at which leakage was first detected, if any, was recorded. This test was done in triplicate.

2.5. Water Contact Angle (WCA) measurement of the papers

The water permeability of the samples was evaluated by measuring the contact angle of a 50 μL water drop. The droplet was placed on the coated surface of each sample, and the contact angle was measured using a 3D digital microscope (VHX-5000, Keyence, Itasca, IL, USA). Observations were conducted for 125 s, and the WCAs were recorded at 0, 25, 50, 75, 100, and 125 s. This test was performed on three replicates for each treatment.

2.6. Morphology of the papers

The morphology of the paper was determined using three types of microscopes: an optical microscope, a 3D digital microscope (VHX-5000, Keyence, Itasca, IL, USA), and a scanning electron microscope with energy-dispersive X-ray spectroscopy. Prior to observation, the samples were cut into $3 \text{ cm} \times 3 \text{ cm}$ pieces. They were observed under the optical-microscope at magnifications of $40\times$, $100\times$, $200\times$, and $400\times$. For the digital 3D microscope, images were captured at $20\times$, $50\times$, $200\times$, $500\times$, and $1,000\times$ magnifications. The SEM images were taken at magnifications

of $500\times$, $2,000\times$, and $5,000\times$.

2.7. Mechanical properties of the papers

The analysis was performed using a TMS-Pro texture analyzer (Food Technology Corporation, Virginia, USA). For the tensile test, the samples were cut to dimensions of 1.5×15 cm. The test was conducted with an initial gap of 100 mm between the two tensile grips and a test speed of 25 mm/min. Load (N) and elongation (mm) at the start of sample deformation were reported. For the puncture test, the samples measured 5×5 cm. The puncture test was conducted at a speed of 10 mm/s, with a maximum load of 25 N applied. Force (N) and distance (mm) during deformation were recorded. All tests were performed in triplicate.

2.8. FTIR profile of the papers

The coated paper was cut into 3×3 cm pieces. Each piece was analyzed using a Tensor II ATR-FTIR spectrometer (Bruker Scientific LLC, Billerica, MA, USA). The transmittance values over the frequency range of $4,000$ to 500 cm^{-1} were recorded. The scan duration was 45 s, and the resolution was 4.0 cm^{-1} .

2.9. X-ray diffraction of the papers

The analysis was conducted on the coated papers and the control using an XRD instrument (SmartLab, Rigaku Corporation, Akishima-shi, Tokyo, Japan). The X-ray voltage and current were set to 40 kV and 30 mA, respectively, with a wavelength of $1.541862 \text{ \AA}$. The scan axis was $2\theta = \omega$, covering a scan range of 5.00 – 89.99° and a step size of 0.03° .

2.10. Statistical analysis

All the quantitative data were assessed for normality and equal variance. When the data were normally distributed and exhibited equal variance, they were analyzed using analysis of variance (ANOVA) and, if applicable, Tukey's HSD test. If the assumptions for ANOVA were not met, the data were analyzed using the Kruskal-Wallis test, followed by Dunn's test. Significant differences were determined based on p -values < 0.05 . The statistical analysis was performed using R version 4.5.0 and RStudio version 2025.05.0-496 (Posit Software, PBC, Boston, MA, USA).

3. Results and Discussion

3.1. Viscosity of the oils

Plant oils are predominantly Newtonian fluids [23-25]; thus, their viscosity remains constant regardless of the shear stress applied. The viscosities of the oils used in the current study varied. Olive oil exhibited the highest viscosity, while candlenut and walnut oils had the lowest. The viscosities of candlenut, walnut, linseed, and soybean oils did not differ significantly ($p < 0.05$). The viscosities of candlenut and walnut oils were significantly lower than those of palm and olive oils (Figure 1). The viscosities of olive, soybean, and walnut oils have been reported previously [24, 25], which are similar to or slightly lower than those reported in this study. These earlier studies also confirmed that olive oil had relatively high viscosity, while the viscosity of walnut oil was among the lowest.

The high viscosity of OL oil may be attributed to its high proportion of triolein [23] and elevated levels of monounsaturated fatty acids (MUFAs), as suggested

by earlier studies [23, 25]. Another study found that, at the same temperature, the methyl ester of oleic acid exhibited higher viscosity than those of lauric, myristic, palmitic, stearic, linoleic, and linolenic acids [26]. The high viscosity of the oleic acid-predominant oil is likely due to the higher maximum molecular length of oleic acid (21.749 Å) than those of linoleic (18.013 Å), linolenic (16.056 Å), and palmitic (20.961 Å) acids. Red PA oil had the second-highest viscosity. This relatively high value can be attributed to the high proportion of palmitic acid, which has the second-longest maximum molecular length among the aforementioned fatty acids. In addition, palmitic acid is a medium-chain SFA that makes the oil semi-solid at room temperature [27]. Palmitic acid has a melting point between 61.0 and 62.5 °C [28]. Meanwhile, the low viscosity of CA and WA oils is likely due to their high PUFA content. High levels of PUFAs in oil correspond to lower viscosities [25]. The multiple double bonds in PUFAs are associated with shorter maximum molecular lengths and lower melting points.

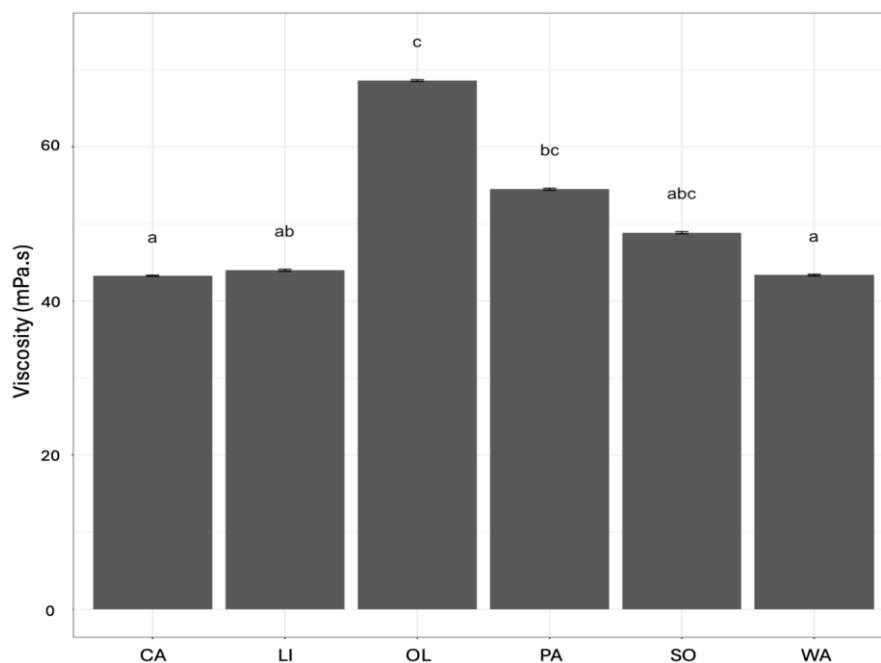


Figure 1: Viscosity (mPa·s) of candlenut (CA), linseed (LI), olive (OL), palm (PA), soybean (SO), and walnut (WA) oils.

*Different letters indicate significant differences ($p < 0.05$).

3.2. Mass and thickness of the coatings

The mass of the coating material applied to the paper surface did not differ significantly among the various types of oils ($p > 0.05$). This finding, along with low relative standard deviations for each treatment (0.51-10.60 %), suggests that the brushing technique used to apply the oils to paper produced a sufficiently homogeneous coating. In addition, the data indicated that variation in the viscosities of the oils did not affect the coating mass.

The coated paper was subsequently dried at 60 °C. This temperature was selected to minimize color changes and the release of volatiles during drying. Another consideration is that low-heat treatment poses minimal risk to workers if applied on a larger scale. LI oil-coated paper exhibited a significantly higher coating mass than PA oil-coated paper (Table 2). This finding corresponds to the longer drying time required for the PA oil coating than the LI oil coating. During heating, both oils can produce volatiles, which are secondary products of fatty acid or triacylglycerol degradation. Previous studies have shown that heating LI oil at 60 °C generated volatiles predominantly consisting of 2-butenal, 2,4-heptadienal, and 1-penten-3-one [29], whereas heating PA oil produced various volatiles, including heptanal, octanal, and nonanal [30]. The longer drying time likely results in more volatiles being released into the environment, thereby reducing the coating mass.

The coatings were relatively thin, with an average thickness ranging from 0.003 to 0.010 mm. The thickness did not vary significantly with oil type ($p >$

0.05), indicating that the physicochemical properties of the oil did not affect this parameter. Thickness measurements were taken using a micrometer screw gauge with a sensitivity of 0.01 mm. The control (uncoated paper) had a thickness of 0.22 mm. The thickness of each replicate of the coated papers, regardless of the oil source, was either 0.22 or 0.23 mm, corresponding to a coating thickness of either 0.00 or 0.01 mm, respectively. The average coating thickness for each treatment was either 0.003 mm (if one replicate measured 0.01 mm and the others 0.00 mm) or 0.010 mm (if each replicate measured 0.01 mm). The large standard deviations of the measurement were primarily due to the low sensitivity of the instrument rather than the homogeneity issue of the samples, as the coating mass (measured before drying) did not differ significantly, and the relative standard deviation of the coating mass for each treatment was low (0.51-10.60 %).

3.3. Water and oil resistance of the coatings

The control paper was highly permeable to water and oil. Water and oil leakage was observed shortly after the test began. Similarly, the OL and PA oil-coated papers were also not resistant to water and oil. This indicates that OL and PA oils are unsuitable as coatings because they do not provide adequate barriers against water and oil penetration. In contrast, CA, LI, SO, and WA oils formed barriers that limited water and oil absorption. Within 60-min observation, no water or oil permeated through these coated papers.

Table 2: Mass and thickness of coatings prepared from CA, LI, OL, PA, SO, and WA oils applied to paper samples measuring 15 × 15 cm.

Treatment	Coating mass (g)		Coating Thickness (mm)
	Before drying	After drying	
CA	2.28 ± 0.24a	1.77 ± 0.06ab	0.010 ± 0.000a
LI	2.17 ± 0.23a	1.89 ± 0.12b	0.010 ± 0.000a
OL	1.93 ± 0.04a	1.69 ± 0.04ab	0.003 ± 0.010a
PA	1.86 ± 0.13a	1.65 ± 0.02a	0.003 ± 0.010a
SO	1.95 ± 0.01a	1.73 ± 0.05ab	0.003 ± 0.010a
WA	2.08 ± 0.22a	1.74 ± 0.05ab	0.003 ± 0.010a

*Different letters indicate significant differences ($p < 0.05$).

Based on the leak time, the OL oil-coated paper was more permeable to water and oil than the PA oil-coated paper (Table 3). This difference may be related to the higher concentration of oleic acid in olive oil, which is predominantly liquid at room temperature. Although oleic acid contains a double bond, it is relatively stable against oxidative polymerization because the bis-allylic hydrogen adjacent to the double bond is not as reactive as that of PUFAs.

The WCA results confirmed the water permeability test findings. The contact angle of a water droplet on the control decreased rapidly. The OL and PA oil-coated papers also exhibited hydrophilicity, although to a lesser degree than the control. The contact angles of the water droplets declined rapidly within 125 s of observation (Table 3 and S1). On the other hand, the reduction of the WCA over time was minimal in CA, LI, SO, and WA oil-coated papers, indicating that these papers were hydrophobic. This property is particularly important for papers intended for use as food packaging. The water resistance is likely linked to the high content of PUFAs in these oils, which facilitates sufficient polymer formation. A study demonstrated that, following thermooxidation treatment, SA oil, a PUFA-rich oil, produced almost twice as many

polymerized triacylglycerols as OL oil, which is high in MUFAs [31]. The polymerization of SO oil is mediated by ester bond formation, although other mechanisms may also occur and require further investigation [6].

Oils with high linolenic acid content, such as LI oil, tend to undergo more extensive polymerization than oils with low linolenic acid content. Linolenic acid contains more bis-allylic hydrogens, which provide radical initiation sites required for autooxidation. These hydrogens are readily abstracted by radical oxygen species. Subsequently, oxidation occurs, producing carboxylates with double bonds in the cis configuration, which can bind to each other, forming dimers. Further cross-linking may occur, generating high-molecular weight oligomers or polymers [32].

The oil layer is firmly attached to the paper surface, probably due to both physical and chemical interactions. Triacylglycerol molecules may fill the pores within the cellulose structure and form on its surface. Additionally, they may undergo transesterification with cellulose [33], which also explains the improved hydrophobicity of the layer. Despite the increased hydrophobicity, the layer remained impermeable to oil because the lipid layer hardened during drying.

Table 3: Water resistance, oil resistance, and contact angle of a water droplet on the surface of the control (CO) and the papers coated with CA, LI, OL, PA, SO, and WA oils*.

Treatment	Water resistance	Oil resistance	Contact angle (°) of the water drop					
			0 s	25 s	50 s	75 s	100 s	125 s
CO	No (leaked after 3.06 ± 0.05 min) ^a	No (leaked after 0.17 ± 0.02 min) ^a	97.00 ± 2.65 ^b	75.67 ± 6.66 ^a	60.33 ± 20.31 ^a	57.00 ± 2.83 ^a	40.00 ± 2.83 ^a	32.50 ± 7.78 ^a
CA	Yes ^{**}	Yes	96.00 ± 1.00 ^b	95.00 ± 1.00 ^b	94.00 ± 1.00 ^c	92.33 ± 2.31 ^b	92.00 ± 1.73 ^b	91.33 ± 2.31 ^c
LI	Yes	Yes	95.67 ± 3.79 ^b	90.67 ± 2.08 ^b	90.67 ± 1.15 ^c	88.00 ± 3.00 ^b	87.67 ± 4.04 ^b	88.00 ± 1.73 ^c
OL	No (leaked after 5.16 ± 0.03 min) ^b	No (leaked after 1.30 ± 0.13 min) ^b	88.00 ± 3.46 ^{ab}	65.33 ± 6.81 ^a	62.67 ± 12.86 ^a	57.00 ± 11.53 ^a	52.33 ± 13.01 ^a	49.67 ± 11.72 ^{ab}
PA	No (leaked after 5.32 ± 0.09 min) ^c	No (leaked after 5.41 ± 0.14 min) ^c	79.67 ± 11.59 ^a	67.00 ± 7.21 ^a	63.33 ± 8.08 ^{ab}	59.00 ± 8.72 ^a	57.33 ± 6.66 ^a	56.33 ± 5.77 ^b
SO	Yes	Yes	101.33 ± 6.66 ^b	97.67 ± 6.66 ^b	89.67 ± 0.58 ^{bc}	89.33 ± 0.58 ^b	86.33 ± 2.08 ^b	83.00 ± 1.00 ^c
WA	Yes	Yes	95.67 ± 3.06 ^b	94.00 ± 1.00 ^b	94.33 ± 2.52 ^c	91.33 ± 0.58 ^b	91.00 ± 1.00 ^b	90.00 ± 1.00 ^c

*Significant differences ($p < 0.05$) in leak time and contact angles across the treatments were noted as different letters.

**No leakage was observed during the 60-min observation period.

3.4. Appearance and morphology of the papers

All the coated papers developed spots or patches, with the LI oil-coated paper showing the largest patches (Figure 2). These patches likely indicate intense lipid polymerization, which can occur at relatively low temperatures, such as 60 °C, and is influenced by fatty acid composition [34]. The intense polymerization observed in the LI oil treatment is attributed to its high linolenic acid content, which enables it to function as a drying oil for many applications, including wood varnish and paint [35]. Additionally, the LI oil-coated paper appeared yellowish (Figure 2), indicating fatty acid oxidation. A study demonstrated that, under identical conditions, samples with high linolenic acid content became more yellow or brown compared to samples dominated by linoleic acid [36]. This yellowing results from the oxidation of triacylglycerols, diacylglycerols, and monoacylglycerols; Maillard browning of phospholipids; oxidation of tocopherols or phospholipids; and the production of polyenes [37].

The enlargement of the samples using the light microscope (Figure 3) confirmed that the LI oil-coated paper exhibited more patches and a more developed-polymer network than the other treatments. The WA oil-coated paper also displayed similar network patterns, but with lower intensity. The network, particularly in the LI oil-coated sample, may indicate hyperbranched or dendrimer structures. These structures are classified as the fourth generation of polymer architectures, following linear, branched, and cross-linked forms [38]. They have various applications, including as vesicles for drug delivery purposes [39,40].

Observations using the 3D microscope revealed that the LI and WA oil-coated papers exhibited relatively large lipid network patches, whereas the CA and SO oil-coated papers displayed smaller lipid network patches (Figure 4). Notably, the patches in the SO oil-coated paper were more evenly distributed. The OL and PA oil-coated papers also exhibited large patches; however, these likely formed through different mechanisms. The patches in the LI and WA oil-coated papers were likely formed due to the polymerization of PUFAs, while those in the OL and PA oil-coated samples were likely formed through the crystallization of SFAs.

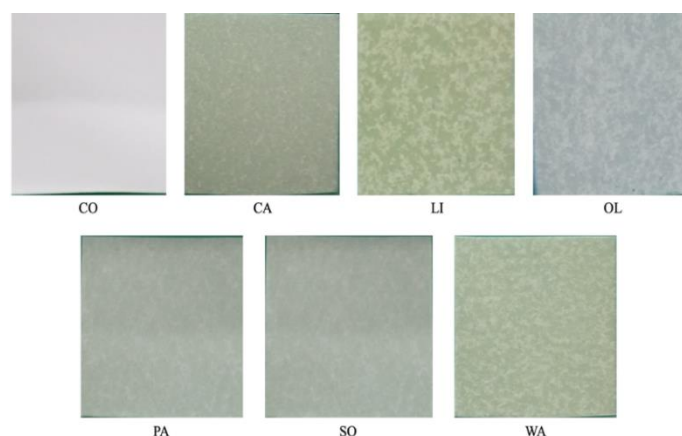


Figure 2: Control or uncoated paper (CO) and papers coated with CA, LI, OL, PA, SO, and WA oils.

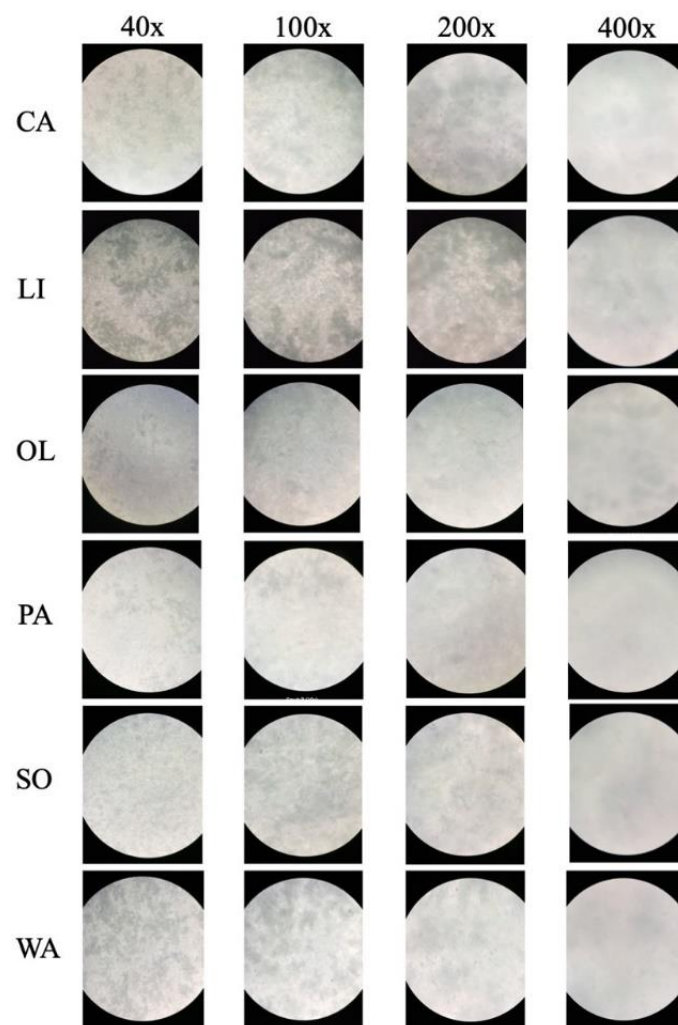


Figure 3: Enlargement (40×, 100×, 200×, and 400×) of the papers coated with CA, LI, OL, PA, SO, and WA oils, observed using a microscope (Eurekam 12 Plus).

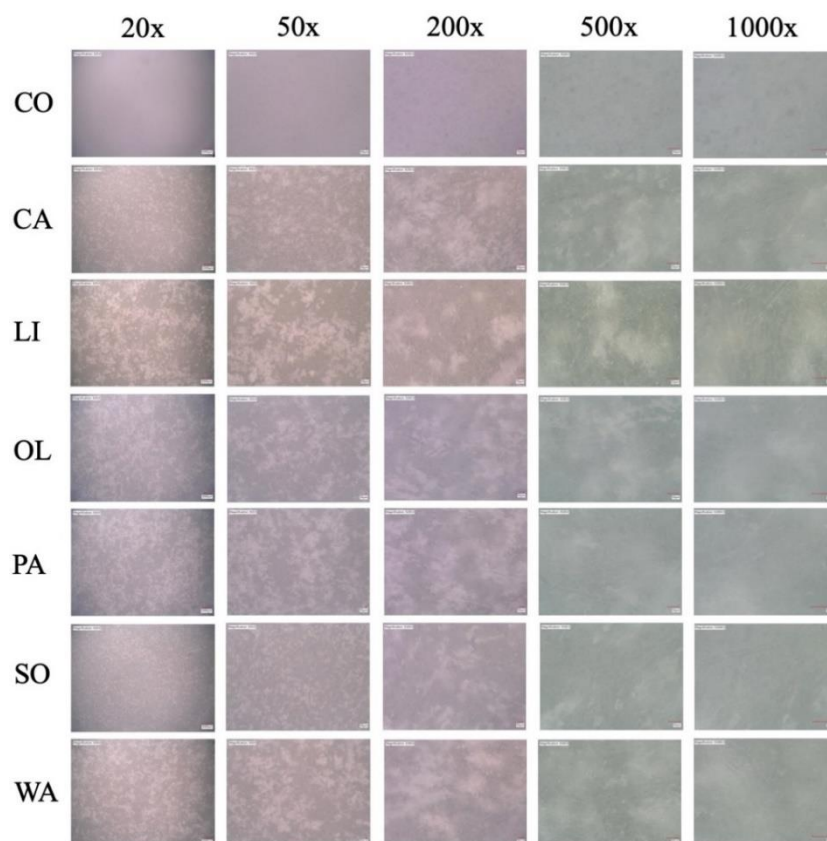


Figure 4: Enlargement (20 $\times$, 50 $\times$, 200 $\times$, 500 $\times$, and 1,000 $\times$) of the CO and the papers coated with CA, LI, OL, PA, SO, and WA oils, observed using a 3D digital microscope (VHX-5000).

The SEM results demonstrated that the samples coated with CA, LI, OL, PA, SO, and WA oils developed complex layers on the paper (Figure 5). The structures produced by LI, WA, and CA oils, which are rich in PUFAs, appeared smoother and contained fewer small globular structures than those produced by the other samples, particularly the OL oil and PA oil treatments. These small globular structures likely corresponded to unpolymerized triacylglycerols. The abundance of these structures was associated with increased water and oil permeability. Meanwhile, the SO oil-coated sample exhibited a smooth structure but contained more holes than the WA, LI, and CA oil treatments. However, hole size may not directly correlate with water and oil permeability, as the lipid coating appears to consist of multiple layers.

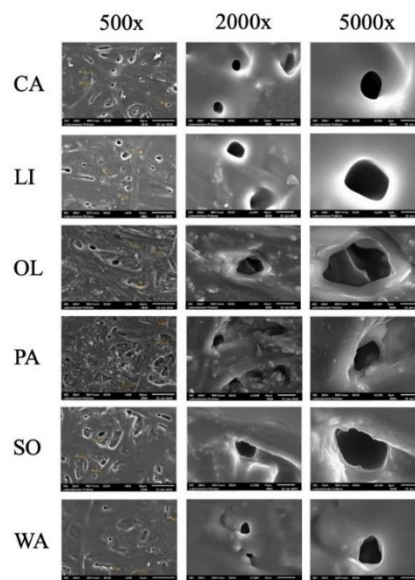


Figure 5: Enlargement (500 $\times$, 2,000 $\times$, and 5,000 $\times$) of the papers coated with CA, LI, OL, PA, SO, and WA oils, observed using a scanning electron microscope.

3.5. Mechanical properties of the papers

The oil-coated papers were assessed for their mechanical properties, particularly puncture force, puncture distance, tensile force, and elongation distance. The puncture force of the coated papers did not differ significantly from that of the control. The only significant difference in puncture force was noted between the WA oil- and PA oil-coated papers, in which the puncture force of the former was higher than that of the latter (Figure 6). This suggests that more cross-linking occurred during the polymerization of WA oil compared to PA oil, resulting in increased puncture resistance.

In addition to exhibiting the lowest puncture force, the PA oil-coated paper also demonstrated the lowest puncture distance. This value was significantly lower than those of the CA, LI, SO, and WA-coated papers. These results indicate that oils with high concentrations of PUFAs form polymer networks that provide greater elasticity compared to those formed by predominantly SFAs. The puncture distances of the CA and LI oil-coated papers were significantly higher than those of the control, as well as the OL and PA oil-coated papers. Meanwhile, the puncture distances of the SO and WA oil-coated papers did not differ significantly from that of the control (Figure 6).

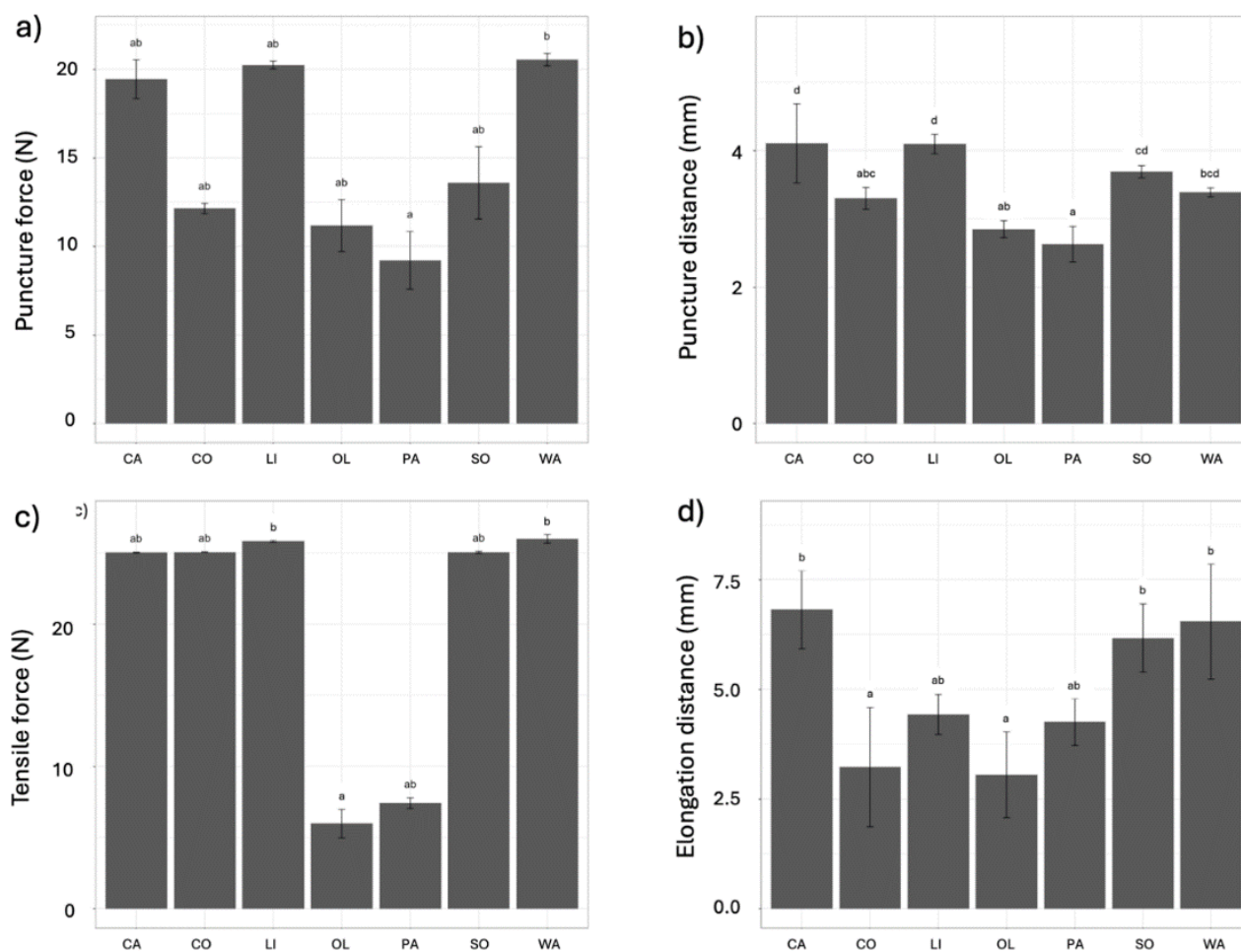


Figure 6: Puncture force (a), puncture distance (b), tensile force (c), and elongation distance (d) of the CO and the papers coated with CA, LI, OL, PA, SO, and WA oils.

*The measurements were conducted on 25 cm² paper for the puncture test and 22.5 cm² paper for the tensile test.

**Treatments with different letters were significantly different ($p < 0.05$).

The tensile forces of the LI and WA oil-coated papers were not significantly different from those of the control and the CA, PA, and SO oil-coated papers, but were considerably higher than those of the OL oil-coated paper ($p < 0.05$). The elongation distances of the CA, SO, and WA oil-coated papers were substantially greater than those of the control and the OL oil-coated paper ($p < 0.05$), although they did not differ significantly from those of the LI and PA oil-coated papers. Considering the data from both the puncture and tensile tests, the hardness and elasticity of the PA and OL oil-coated papers were relatively low, suggesting minimal cross-linking due to their low relative concentrations of PUFAs. It should be noted that the elongation distance exhibited a high standard deviation, as this parameter is highly sensitive to even small irregularities in the samples, instrument setup, and sample deformation.

The water- and oil-resistant papers in the current study demonstrated strength and elasticity comparable to or greater than the uncoated paper. This presents an advantage over grease-resistant protein-laminated

paper, which has lower strength than uncoated paper [41]. Improved paper strength and elasticity contribute to increased resistance to deformation, including bending and tearing, making it desirable for food packaging applications.

3.6. FTIR profile of the papers

The FTIR transmittance as a function of wavenumber is displayed in Figure 7. The peak positions, along with their corresponding functional groups and compound classes, are shown in Table 4. The coated papers exhibited lower transmittance at 2,917, 2,850-2,857 and 1,600-1,750 cm^{-1} than the control. The lower transmittance values at 2,917 and 2,850 cm^{-1} of the coated samples likely correspond to asymmetric and symmetric C-H modes in aliphatic compounds, respectively [42], both of which may be present in oils. The lowest transmittance values were observed in the OL and PA samples, as they contain more SFAs than the other oils, and increased saturation is associated with greater C-H stretching.

Table 4: Wavenumbers (cm^{-1}) of FTIR peaks and their associated functional groups and classes* for the CO and the papers coated with CA, LI, OL, PA, SO, and WA oils.

Wavenumber (cm^{-1})	Treatment							Functional group	Class
	CO	CA	LI	OL	PA	SO	WA		
708-719	X	X	X	X	X	X	X	C=C bending*	Alkene
									Benzene derivative
872	X	X	X	X	X	X	X	C-H bending*	Substituted benzene
1025-1027	X	X	X	X	X	X	X	C-N stretching*	Amine
1053	-	-	-	X	X	-	-	C-O stretching**	Primary alcohol
1100-1105	X	X	X	X	X	X	X	C-O stretching*	Secondary alcohol
1158-1160	X	X	X	-	-	X	X	C-O stretching*	Tertiary alcohol
1171	-	-	-	X	X	-	-	C-O stretching**	Ketone
1241-1250	-	X	X	X	X	X	X	C-O stretching*	Alkyl aryl ether
1311-1318	X	X	X	X	X	X	X	O-H bending*	Phenol
1371-1373	X	X	X	-	-	X	X	O-H bending*	Alcohol
								O-H bending*	Phenol
1384	-	-	-	X	X	-	-	O-H stretching***	Oxygen-containing group
1416-1421	X	X	X	X	X	X	X	O-H bending*	Alcohol
								O-H bending*	Carboxylic acid

Table 4: Continue.

Wavenumber (cm ⁻¹)	Treatment							Functional group	Class
	CO	CA	LI	OL	PA	SO	WA		
1456/1463	-	-	X	X	X	-	-	C-H bending*	Alkane
1539	-	-	-	-	X	-	-	N-O stretching*	Nitro compound
1576	-	-	-	-	X	-	-	C=C stretching*	Cyclic alkene
1630-1653	X	X	X	X	X	X	X	C=C stretching*	Alkene
1736-1742	X	X	X	X	X	X	X	C=O stretching*	Esters
2850-2857	X	X	X	X	X	X	X	C-H stretching*	Alkane
2916-2924	X	X	X	X	X	X	X	C-H stretching*	Alkane
3330-3335	X	X	X	X	X	X	X	O-H stretching (hydrogen-bonded)*	Alcohol
								C-H stretching*	Alkyne
3731-3741	X	X	X	X	X	X	X	O-H stretching (free)*	Phenol, alcohol

*The group and class associated with the peak position were obtained from [41].

**The group and class associated with the peak position were obtained from [47].

***The group and class associated with the peak position were obtained from [46].

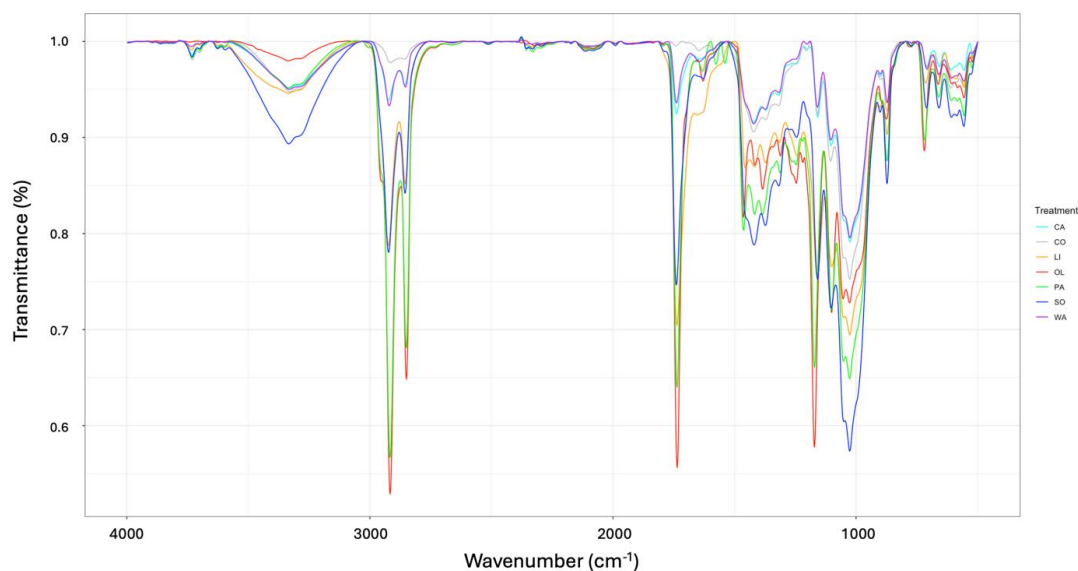


Figure 7: FTIR profiles of the uncoated paper (CO) and the papers coated with CA, LI, OL, PA, SO, and WA oils.

The lower transmittance values observed in the 1,600 and 1,750 cm⁻¹ bands of the coated papers, compared to the control, indicate lipid oxidation in the coated samples, particularly the formation of carbonyl

compounds, such as saturated aldehydes (1,732 cm⁻¹), saturated ketones (1,710 cm⁻¹), and unsaturated aldehydes (1,690 cm⁻¹) [43]. The lower transmittance at this wavelength also suggests cross-linking of tria-

cylglycerols or free fatty acids through ester linkages. A study demonstrated that upon open-air heating, approximately half of the carboxylate and olefinic carbons of oleic acid convert into ester, $-CHO$, and methylene carbons. Ester formation was confirmed by the presence of $C=O$ stretching bands at $1,736\text{ cm}^{-1}$ and $C-O$ bands near $1,180\text{ cm}^{-1}$ [44].

Besides ester linkages, lipid molecules can dimerize or polymerize via ether linkages [45]. This linkage was observed at $1,241\text{--}1,250\text{ cm}^{-1}$ in LI, SO, OL, and PA oil-coated papers, but was absent in the CO, CA, and WA oil treatments. The absence of this peak in the CA and WA oil-coated papers indicates that polymerization may occur through other mechanisms, including ester linkages, $-C-C-$, or $-C-O-O-C-$ bonds [45].

In some peak positions, the FTIR transmittance values of WA and CA oil-coated papers were similar, despite differences in their fatty acid profiles. Typically, the fatty acids of WA oil are palmitic acid (6.06 %), stearic acid (2.62 %), oleic acid (14.10 %), linoleic acid (61.70 %), and linolenic acid (15.00 %) [15]. In contrast, CA oil contains palmitic acid (6.51 %), stearic acid (2.53 %), oleic acid (26.35 %), linoleic acid (39.71 %), and linolenic acid (25.00 %) [10]. The similarity between these two oils is attributed to their viscosity and dry coating mass (Table 1), which likely results in similar intensities of certain functional groups.

The OL and PA oil-coated papers exhibited a peak at $1,384\text{ cm}^{-1}$, whereas the other treatments did not. This absorption is likely due to the stretching and deformation vibrations of the $-OH$ group in water [46]. This finding suggests that these samples absorbed moisture due to their water permeability.

The interaction between hydrophobic substances, such as vegetable oil, and cellulose on paper surfaces primarily occurs through non-covalent forces, including hydrogen bonding and hydrophobic interactions. Hydroxyl groups on cellulose and the fatty acids in vegetable oil are critical determinants of these interactions. Initial binding between cellulose and vegetable oil results from the adsorption of fatty acid molecules onto binding sites on the surface of cellulose ribbons. The fatty acid molecules may subsequently diffuse into the ribbon shell and adsorb onto accessible microfibril surfaces. Non-covalent binding of fatty acids to cellulose is primarily driven by hydrogen bonding and hydrophobic interactions, with the

presence of hydroxy groups reported to favor adsorption. Tsompou et al. [48] suggested that OL oil can adhere to cellulose microfibrils, a process influenced by cellulose hydrophilicity and chemical properties. The band at $2,850\text{--}2,920\text{ cm}^{-1}$ corresponds to symmetric and asymmetric $-CH$ stretching in the fatty acid chains of vegetable oil. In untreated paper, the intensity was low; however, after coating, the intensity increased, indicating the incorporation of oil into the cellulosic chain. The $-OH$ bond vibration bands are located between $2,800$ and $3,800\text{ cm}^{-1}$. Higher intensity in the $-OH$ bond region on coated paper indicates enhanced intramolecular and intermolecular hydrogen bonding, characterized by the presence of a peak at $3336\text{--}3340\text{ cm}^{-1}$ [49]. Hydrogen bonding is also characterized by the red shift in the $-OH$ stretching band, where the wavenumber shifts to a lower value [50]. It was observed that the vibration at 3314 cm^{-1} (CO) shifted to a lower wavenumber to $3300\text{--}3291\text{ cm}^{-1}$ in the treated paper.

These FTIR data provide insights into the chemical properties of the coated papers. They reveal evidence and mechanisms of polymerization in papers coated with oils containing high amounts of PUFAs, the moisture permeability of OL and PA oil-coated papers, and the interactions between the oils and cellulose. However, due to the absence of replication in this analysis, these findings should be confirmed in a further study.

3.7. X-ray diffraction of the papers

The XRD profiles of the control and the coated papers are displayed in Figure 8. The control and all treated samples exhibited dominant peaks at $2\theta = 16.1^\circ$, 20.78° , and 29.51° , while only the OL and PA oil treatments showed peaks at $2\theta = 5.8^\circ$. The peaks at $2\theta = 16.1^\circ$, 20.78° , and 29.51° correspond to the paper samples, given their similarity to the control. The increased area of the amorphous region between $2\theta = 16.1^\circ$ and 20.78° in the treated samples, relative to the control, may indicate lipid polymerization and oxidation. The polymers did not form crystals, likely due to the large variation in fatty acids, triacylglycerols, and minor components such as tocopherols, as well as the non-linear structure of PUFAs. The presence of double bonds introduces bending in the molecular structure, increasing the number of amorphous regions.

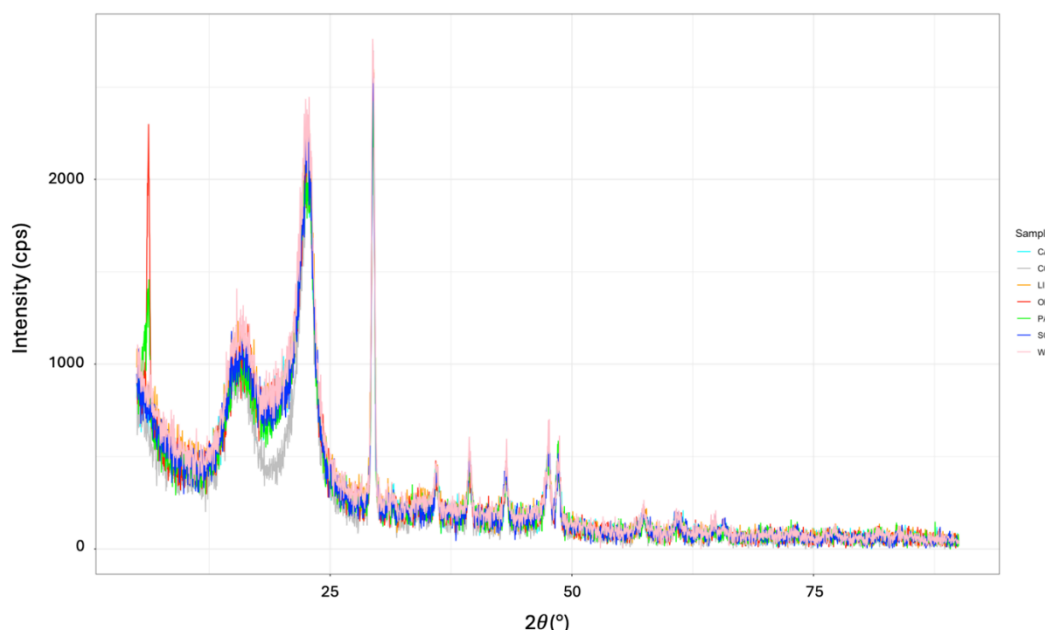


Figure 8: XRD profiles of the uncoated paper (CO) and the papers coated with CA, LI, OL, PA, SO, and WA oils.

On the other hand, the peak at $2\theta = 5.8^\circ$ observed in the OL and PA oil coatings was unique to the coatings and absent in the paper. Because the peak was singular, it likely indicates a crystal in the α form, which can be attributed to SFAs or triacylglycerols containing SFAs, comprising significant portions of both oils. Saturated fatty acids account for 67 and 17 % of the total fatty acids in PA oil [13] and OL oil [51], respectively. Saturated fatty acids lack kinked structures caused by cis double bonds, allowing closer molecular packing and, consequently, crystal formation. The intensity of the peak at $2\theta = 5.8^\circ$ was greater in the OL oil-coated paper than in the PA oil-coated paper, likely due to the slightly higher dry coating mass (Table 2), which results from the higher viscosity of OL oil (Figure 1).

Fatty acids and triacylglycerols can exist in various crystal forms, including α , β' , and β . The subcells of these three forms are hexagonal, orthorhombic perpendicular, and triclinic parallel, respectively [52]. Generally, lipids initially form α and β' crystals, which then transform into the more stable β form. The crystal types of lipids are influenced by multiple factors, including triacylglycerol variability, cooling rate, heat of crystallization, and agitation level. Formulations with high homogeneity of triacylglycerols can rapidly produce the β form, whereas those with greater variability of triacylglycerols are usually present in the β' form [53]. Although XRD provides new insights into this topic, these findings should be confirmed through

additional investigations due to the lack of replication.

4. Conclusions

Water- and oil-impermeable papers were successfully produced by heating papers coated with CA, LI, SO, and WA oils. In contrast, PA and OL oils did not produce water- and oil-resistant coatings. The formation of the water- and oil-proof barrier was correlated with the high concentrations of PUFAs in CA, LI, SO, and WA oils. Different oil types produced significant variations in oil viscosity, dry coating mass, puncture force, puncture distance, tensile force, elongation distance, morphology, as well as FTIR and XRD profiles of the coated papers. CA and WA oils exhibited the lowest viscosities, whereas OL oil had the highest. The water- and oil-resistant coated papers demonstrated strengths and elasticities comparable to or greater than those of the uncoated paper. Morphological analysis revealed extensive lipid polymerization, particularly in the LI and WA oil treatments. The FTIR analysis confirmed that oxidation occurred in the oil-coated papers, suggesting that polymerization of PUFAs may have occurred via ester and ether linkages. The XRD results demonstrated that the polymers did not form any crystals. Conversely, crystals observed in the OL and PA oil treatments likely originated from SFAs.

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