



Fully functional absorbent pads for fresh meat packaging made of enzymatically pre-treated TMP fibers, CTMP core, lignin-based treatment and adhesive

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ABSTRACT

Absorbent pads for meat packaging are crucial for extended meat preservation and to keep meat appealing to the customers. Cellulose based packaging has been replacing more and more plastic packaging due to cellulose's sustainable sourcing, biodegradability, high recycling rates and absence of microplastics formation. Here an absorbent pad was fully made of mechanical pulps and wood derivatives. The film that maintains the core together was made of fibrillated thermomechanical pulp (TMP). The pulp was pre-treated with xylanase enzymes before refining to improve fiber fibrillation. The fibrillated enzymatic pretreated pulp (Enz-TMP) demonstrated good film-forming capabilities, and films with 25 and 50 g/m² could be manufactured with mechanical strength of 43 and 55 MPa, respectively. Their wet resistance to tension was comparable to a commercial paper reference. The films were modified with a lignin-based treatment to provide water barrier properties to the surface in contact with meat to avoid meat drying. Cobb60 values of 2.7 g/m² and 3.5 g/m² were obtained after lignin addition. The core of the pad was composed of fluff chemi-thermomechanical pulp (CTMP) to provide good absorption capacity. Finally, fully functional pads were manufactured and composed of: i) CTMP in the core, ii) fibrillated Enz-TMP as the basis for films that were surface modified and perforated on the top and iii) an adhesive of fibrillated Enz-TMP/lignin mixture. The absorption performance of the pads (core dimension 155 × 60 mm) was 23.8 mL/Pad, as assessed with the industrially established BfR (German Federal Institute for Risk Assessment)-method.

1. Introduction

Food packaging is crucial to protect food from spoilage and during transport. Packaging is essential to maintain the quality of food and protect the food from external factors (microbes, light, oxygen...) [1]. Plastic has been first choice for fresh food packaging due to its price, processability and properties. However, the end of life of plastic packaging is still a problem due to limited recycling facilities and high quantities still going to landfill or incineration [2,3]. Cellulose fibers are an alternative to plastic due to their sustainable source and high recycling rate. But challenges remain to make fully fiber-based packaging due to the demanding storage conditions and high standard to preserve fresh food. Fresh meat packaging is usually composed of three main components: i) the tray, ii) a pad to absorb liquids and iii) a transparent

film to cover the tray. Here we develop an absorbent pad for meat packaging that is fully fiber based.

Absorbent pads in meat packaging aim to remove fluids (water, blood, fat and others) from the packaging to avoid microbial growth and reduction in shelf life [4]. In addition, the presence of liquid in the packaging can be considered unappealing for the consumer. Work have been done to increase the shelf life of meat by studying parameters such as absorption capacity of the absorbent pad [5], gas composition in the modified atmosphere packaging (MAP) [6] and addition of antimicrobial and antioxidant compounds to the absorbent pads [7,8]. Moreover, there are constraints from the industry, the pads need to be thin and light with high absorption capacity. For example, Fontes-Candia et al. produced cellulose based aerogels with water absorption capacities between 10 and 50 g/g and densities between 10 and 25 mg/cm³ [8].

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Although, aerogels can have high water retention capacities, they usually have very low densities. Commercial absorbents have densities that are ten times higher than aerogels.

In many commercial products, synthetic superabsorbent polymers (SAP) are used to increase the absorption capacity of pads [9]. Cellulose pulps also have relatively high absorbency due to the hydrophilic nature of cellulose. Mechanical pulps (thermomechanical pulp (TMP) and chemi-thermomechanical pulp (CTMP)) have been used for their absorbency as they are more bulky and stiff than chemical pulps [10]. The presence of lignin helps to maintain a bulky structure when wet [11,12]. Pulps for fluff products are characterized by high amount of long fibers and stiff fibers that can give high bulk [13]. Similar fluff pulp has been used for absorbent pads for food and for hygiene products [14,15]. In both cases, high absorption under load is a key property. There are two different absorption mechanisms, it can be the absorption in the fibers structure leading to fiber swelling or in the cavities created by the fiber network by capillary forces. Surface chemistry could also influence the absorbency of the fibers as the presence of hydrophilic groups can be favorable for higher absorption [16].

A balance should be found between absorbing the extra fluids and not drying the meat [6]. To avoid drying a non-absorbent film is needed between the fibers and the meat. To compete with polyethylene (PE) film present on the market, focus on the thickness and mechanical properties is needed. Resistance to water is also important. Micro-fibrillated cellulose (MFC) fibers can form thin films with high mechanical properties [17]. MFC has been mostly produced from chemical pulps. Trials have been done to prepare them from mechanical pulps however, it has been shown that chemical or enzymatic pre-treatments of the fibers are necessary to fibrillate mechanical pulps into individual fibrils [18–21]. The presence of lignin makes the fibers brittle and prone to fiber breakage instead of fibrillation. Laccases and endoglucanases have been used successfully to ease the fibrillation of mechanical pulp [19]. Lignin was first modified by the laccases which facilitated accessibility for the endoglucanase to weaken the fibers.

The films present around the pads need to resist under wet conditions. Films made from refined cellulose pulps are water sensitive. To improve wet strength of paper, additives made of synthetic or natural polymers have been used [22,23]. Lignin is among the biobased solutions to increase wet strength of paper as it can act as a binder between the fibers [24]. Here, a lignin-based treatment was used to make the film nonabsorbent on the side in contact with the food, thus reducing the potential capillary forces from the film that may dry the fresh meat.

The goal in this research was to produce 100% biobased absorbent pads with absorption capacity comparable to commercial pads. Hence, various technologies (enzymatic pre-treatment, refining, film formation, protective treatment, pulp fluffing) were combined to make a new kind of absorbent pad that is fully biobased and has comparable properties to commercial products. We hypothesized that the enzymatic treatment of the TMP fibers could improve the refining to get good film formation at low grammage to replace PE film in the absorbent pads. First, the films for the absorbent pads were made from fibrillated TMP fibers, focus was put on wet strength and nonabsorbent properties. Then a comparison between TMP and CTMP fibers was performed regarding absorption capacity of the core. At the end the complete pad solution was brought together with an adhesive composed of highly fibrillated TMP fibers and lignin, and the full pads were tested for liquid absorption following an industrially established method.

2. Materials and methods

2.1. Materials

TMP reject fibers was provided by Norske Skog Saugbrugs (Norway). CTMP fibers, the commercial pads sample and the reference films (PE and cellulose/heat sealable polymer) were provided by McAirmaid's (Germany). The reference films were used for mechanical testing in wet

conditions and the cellulose-based film was used for the adhesion test as well. The commercial pads were composed of a 300 g/m² CTMP core that was held together by two polyethylene (PE) films (21 g/m² each). The commercial pads were used for comparison of full pad absorption in Table 5.

Lignin for the adhesive was provided by Fibenol (Estonia). METNIN™ SHIELD was produced by MetGen (Finland). It is a formulation containing mainly lignin that is fractionated using the METNIN™ fractionation process. The high molecular weight lignin is used in this case to make a biobased formulation that can be sprayed on the surface of paper and board products. METNIN™ SHIELD is used as an additive that penetrates the surface and protects the fiber material from moisture. The xylanases (XB), provided by MetGen, were fungal xylanase with optimal working temperature and pH of 50–70 °C and 6–7 respectively.

2.2. Enzymatic treatment and refining of TMP fibers

Enzymatic pre-treatment of the TMP was performed to ease the fibrillation of the pulp. For that purpose, approximately 8.6 kg of TMP was soaked overnight in Britton and Robinson buffer (pH 6). After soaking the pulp was diluted to 3.2 wt% and the temperature was set to 50 °C. Once the temperature was stable, the xylanases were added with a dosage of 5% of the dry pulp. After one hour of reaction the temperature was increased to 100 °C for 10 min to inactivate the enzymes. The pulp was then cooled to 10 °C before the next steps.

The enzymatically pre-treated TMP fibers were refined by Valmet (Finland), using a conical refiner. The target refining energies were 500, 1000 and 2000 kWh/t. A control sample of TMP pulp without enzymatic pre-treatment was also refined in the same conditions (Table 1).

2.3. Film of refined TMP and Enz-TMP

2.3.1. Film preparation

Preliminary work suggested that TMP 2000 was more adequate for film formation than TMP 500 and 1000. Films from the different TMP 2000 (untreated and enzymatically pre-treated) were prepared with a Rapid Köthen sheet former (Table 2). The suspensions were diluted to 9 g/L before sheet forming. Films of 25 and 50 g/m² were made by using a nylon filter with 1 µm opening size. After forming, the films were covered with another 1 µm nylon filter and pressed between blotting paper at 2 bar. The films that were not coated were dried in the dryers of the Rapid Köthen at 92 °C for 7 min with vacuum. After drying the films were pressed with 10 MPa for 3 min at 200 °C using a Fontijne laboratory press.

2.3.2. Treatment with METNIN™ SHIELD

The METNIN™ SHIELD was sprayed on the wet films after the pressing at 2 bar (to partly remove the water and optimize moisture content for further treatment). Since the METNIN™ SHIELD solution is sprayed on wet/moist substrate, water acts as a carrier to bring METNIN™ SHIELD deeper within the fiber network, thus providing

Table 1

Samples designation after refining TMP and xylanase pretreated TMP at different target specific energy consumptions.

Sample Name	Pulp	Target specific energy consumption under refining (kWh/t)
TMP 500	TMP	500
TMP 1000	TMP	1000
TMP 2000	TMP	2000
Enz-TMP 500	TMP pre-treated with xylanase	500
Enz-TMP 1000	TMP pre-treated with xylanase	1000
Enz-TMP 2000	TMP pre-treated with xylanase	2000

Table 2

Description of the films made from refined TMP and Enz-TMP with different grammage (25 or 50 g/m²) and in the presence or absence of METNIN™ SHIELD treatment.

Sample name	Pulp	Grammage (g/m ²)	METNIN™ SHIELD treatment
TMP 2000	TMP 2000	50	No
TMP 2000 + Shield	TMP 2000	50	Yes
Enz-TMP 2000	Enz-TMP 2000	50	No
Enz-TMP 2000 + Shield	Enz-TMP 2000	50	Yes
Enz-TMP 2000 (25)	Enz-TMP 2000	25	No
Enz-TMP 2000 (25) + Shield	Enz-TMP 2000	25	Yes

hydrophobicity once the material is dried. An Ultra Handheld Airless Sprayer from Graco was used to spray the METNIN™ SHIELD. The solution was sprayed as received with a concentration of 18 wt%. The targeted dry coat weight was 10 g/m². After treatment the films were handled in the same conditions as the non-sprayed films i.e. dried in the Rapid Köthen dryers and then hot-pressed.

2.4. Core formation

The TMP pulp was first hammer milled to fluff the pulps. A 5 mm hole size was used with a 4500 rpm rotation speed. The CTMP was used as received.

The fluff pulp was then formed into cores of 300 g/m² and diameter of 50 mm, with the help of a laboratory-scale air-laid former (see Fig. 1). The laboratory-scale air-laid former consisted of a fiber inlet that led to a formation chamber. A mesh was positioned at the bottom of the formation chamber and vacuum was applied at the bottom to form the core. After formation some of the cores were pressed with 10 tons for 30 s and others were analyzed without pressing. The cores were stored at 23 °C and 50%RH until further processing.

Full-scale cores were formed using a dry forming apparatus that included: a forming head, forming table (300 mm × 300 mm) and vacuum box. A frame (300 mm × 300 mm) with 6 rectangular spaces of 155 mm × 60 mm was printed in an Ultimaker S5 3D printer (PLA filament, diameter 2.85 mm) and placed in the forming table. Six pads were manufactured simultaneously, with a grammage of 300 g/m² and lateral dimension of 155 × 60 mm. Three pads were compressed simultaneously with a Fontijne laboratory press, at ambient temperature

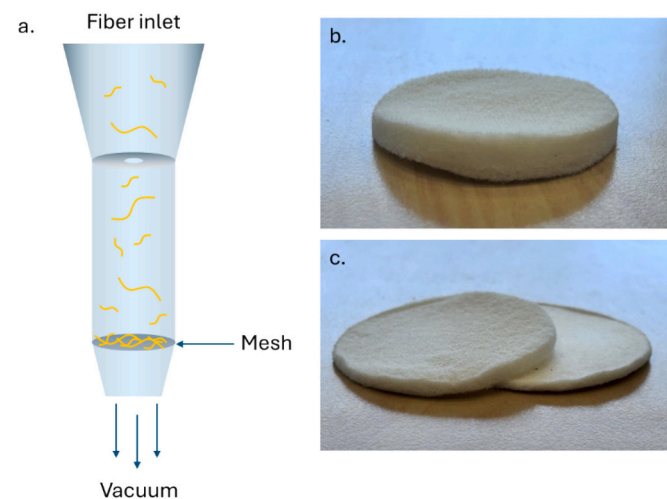


Fig. 1. a. Set up for core formation produced at 23 °C and 50%RH. b. fluff core after formation and c. cores compressed with 10 tons (pad diameter = 50 mm).

and 1372 kN for 30 s.

2.5. Assembling of the pads

Enz-TMP 2000 suspension (consistency of 4.1 wt%) with and without added lignin were applied as adhesive to seal the films around the core. The adhesive consisted of 50:50 mixture dry weight of lignin and Enz-TMP 2000.

Absorbent pads were made by placing a compressed core (300 g/m² and thickness ~ 1–2 mm) between two films and sealing the films with the adhesive. The films were made of Enz-TMP 2000 (25) + Shield, CTMP was used for the core and Enz-TMP 2000 mixture with lignin was used as the adhesive. The films were cut in circles of 7.5 cm in diameter. The adhesive was deposited on the bottom film and the core placed in the middle. The top film was added on top following Fig. 2. After assembling it was hot pressed with a circular frame to press only the area containing adhesive. Hot pressing was carried out at 200 °C for 30s surrounded by blotting papers and greaseproof papers. The top film was also perforated with needles to allow for liquid absorption. A new device was designed and 3D printed to facilitate the perforation of pads (upper and lower surface) to let the liquid come inside. The device was printed with PLA (filament diameter 2.85 mm) in an Ultimaker S5 3D printer. Needles were placed on the lock of the device. The distance between the needles was 6 mm.

2.6. Characterization

2.6.1. Fiber and film properties

The morphology of the fibers was characterized with a Fiber Tester Plus (Lorentzen & Wettre, ABB, Switzerland). For the analysis, 0.1 g of fiber is dispersed in 200 mL of water and injected into the system. The fiber length, width and perimeter were measured, and the fines content was determined. Fines are defined to be fibers with a length under 0.2 mm. Canadian Standard Freeness (CSF) was measured according to ISO 5267-2:2001. Two replicates were measured.

The apparent density of the films was calculated from their mass measured at 23 °C and 50%RH. The thickness was measured with a Lorentzen & Wettre micrometer.

The water absorption from the surface of the film was measured using a Cobb cup following ISO 535. The area of measurement was 25 cm² and the time of contact was 1 min. Two replicates were measured per sample.

The contact angle was measured with a Dynamic Absorption Tester (DAT 1100). At least 7 drops were analyzed and the results shown correspond to the average angle after one minute of contact.

The tensile properties of the films were measured with a universal material tester (Zmart.Pro) from Zwick (Germany). The test speed was 100 mm/min and the gap between the grips was set at 40 mm. The strips had a width of 1.5 cm. The dry tensile strength was measured at 23 °C and 50%RH while the wet tensile strength was measured after soaking the films for one hour in water. The wet thickness was measured before tensile testing. Three parallels were measured for each film in both conditions.

The fibers and films were imaged with a Hitachi SU 3500 scanning electron microscope (SEM) in secondary electron imaging mode. The samples were deposited on carbon tape, dried and sputtered with gold before imaging.

2.6.2. Film adhesion properties

TMP 2000 films were cut into squares of 2.54 cm × 2.54 cm and 0.25 g of one of the adhesives was spread on one square while another square was placed on top. The sandwich was then hot pressed at 200 °C for 30s surrounded by blotting papers and greaseproof papers. To compare the adhesives and tests their strength, testing was performed using a Zwick universal material tester and z-strength module following Tappi T541. In short, each sandwich was taped to one side of the

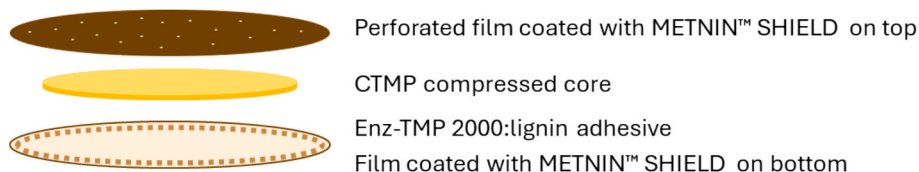


Fig. 2. Schematic representation of the assembly of the full pad including the different parts: the films under and over the core as well as the location of the adhesive.

module, a 900 N force was applied to tape the other side and then the sample was pulled until breakage at 66 mm/min. Five replicates were measured per adhesive.

2.6.3. Testing of liquid absorption

The core formation and testing were conducted at 23 °C and 50% relative humidity. The absorption under load was measured according to BfR (German Federal Institute for Risk Assessment). The absorption time was 20 min (for circular test pads, diameter 50 mm), and the solution was 0.2% NaCl. A load of 236 g was placed on top of the pad during the absorption test corresponding to 12 g/cm². The absorption under load (AuL) was calculated following Eq. 1.

$$AuL = \frac{m_w - m_d}{m_d}$$

The liquid absorption of the full-scale pads (155 mm × 60 mm) was measured as described above but with a load of 1116 g, in 0.9% saline and for 60 min (BfR method). Three parallel were measured for each pad.

3. Results and discussion

An absorption pad for meat is composed of several components, each

having a specific function. The core is the main material and contributes to the main function that is absorbing meat liquids during storage. The core is usually made of loose fibers that need to be confined by an external film. This film also limits the direct absorption of liquids from the meat by the core that could lead to meat drying. The films need to be sealed together to maintain the fibers inside, for thermoplastics (e.g. PE) heat sealing can be used but in the case of non-heat-sealable materials an adhesive is needed. In this paper, the film, the core and the adhesive have been optimized to form a 100% wood-based pad. Mechanical pulp fibers (TMP and CTMP) as well as lignin were used.

3.1. Refined enzymatically pretreated TMP fibers and properties of treated films

3.1.1. Morphological characteristics of refined fibers

Fig. 3 shows the evolution of the morphology of TMP fibers with increasing energy input during refining and the effect of enzymatic pre-treatment on the fibers. The length of the fibers decreased with increasing energy input. Fiber cutting can occur during refining of fibers containing large amounts of lignin. The fiber width also decreased with increasing refining energy. The fibril perimeter and fines content increased with refining energy which shows the development of the fibers towards shorter, thinner and more fibrillated fibers. The differences

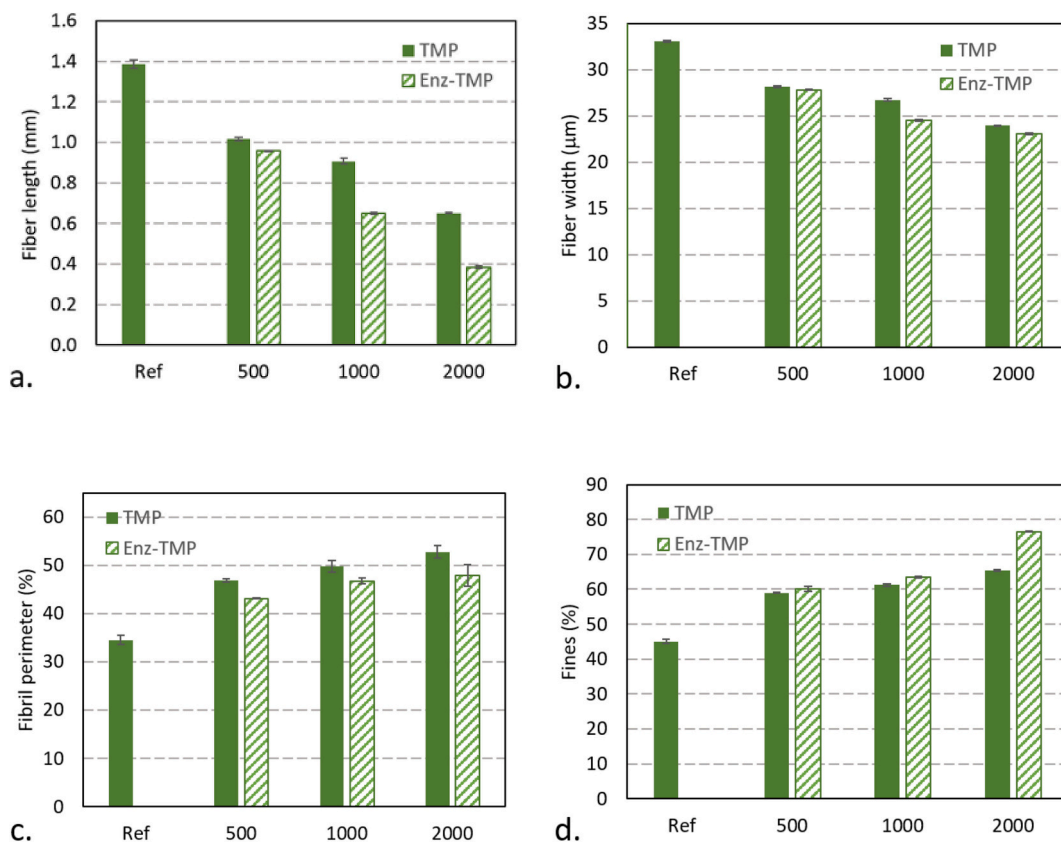


Fig. 3. Comparison of the morphology of the fibers (a. length, b. width, c. fibril perimeter and d. percentage of fines) with and without enzymatic pre-treatment at different refining energies. The error bars represent the standard deviations.

between the TMP and Enz-TMP were more pronounced as the refining energy increased. Xylanases target hemicelluloses and more precisely xylans in the fiber wall, this can lead to increase porosity and flexibility of the fibers [25,26]. Fig. 4 shows the images of fibers refined at 2000 SEC. Enz-TMP is composed of shorter fibrous objects ($<500\text{ }\mu\text{m}$) than the untreated TMP fibers (Fig. 4, upper panel). According to the SEM assessment, the Enz-TMP 2000 is fibrillated and contains shorter and less residual fibers, and micrometer sized fibrils (Fig. 4). This is considered an indication of a fibrillated pulp composed of lignocellulosic micro- and nanofibers.

3.1.2. Films from fibrillated TMP fibers coated with METNIN™ SHIELD

Based on the morphological assessment, i.e., less residual fibers and higher fraction of fines (Fig. 3 and Fig. 4), Enz-TMP 2000 was selected to make films for the absorbent pads. Protective films form a barrier between the meat, and the absorbent core and are characterized by having low water absorption capacity and being water resistant (high wet strength). Its basis weight needs to be as low as possible to limit its contribution to the total weight of the pad, which implies a reduction of production and transportation costs. Additionally, a lignin-based solution was sprayed on one side of the film to decrease the water absorption from the outside surface of the pad.

Table 3 shows the characteristics of the films. Despite the films being hot-pressed the density was relatively low ($650\text{--}780\text{ kg/m}^3$) due to the presence of relatively large residual fibers ($23\text{--}27\text{ }\mu\text{m}$ width in average, Fig. 3) and relatively high air permeability. The air permeability of the films decreased considerably with the METNIN™ SHIELD treatment, reaching the maximum value measurable with the instrument. It demonstrates that METNIN™ SHIELD was present as a layer filling the porous surface of the films. For the 25 g/m^2 film the treatment reduced the air permeability to a lower extent, indicating the potential presence of thinner areas or inhomogeneity in the films. Smaller size of the residual fiber objects, higher fine content (Fig. 3) and apparently higher

Table 3

Characteristics of the films made from TMP and Enz-TMP with and without METNIN™ SHIELD. When the maximum value of the Gurley equipment was reached, it was not possible to calculate the average, hence the two measurements are shown.

	Grammage	Density	Gurley	Wet strength retention
	g/m^2	kg/m^3	s	%
TMP 2000	52	657	10350	24
TMP 2000 + Shield	60	720	28750, max	25
Enz-TMP 2000	53	725	6320, max	18
Enz-TMP 2000 + Shield	59	777	max, max	20
Enz-TMP 2000 (25)	27	680	11113	26
Enz-TMP 2000 (25) + Shield	35	699	14200	21

fibrillation degree (Fig. 4), yielded relatively dense films, causing higher resistance to air flow that was improved with the METNIN™ SHIELD.

The positive effect of the enzymatic pretreatment was also reflected in the mechanical properties of the films (see Fig. 5a) where enzymatically pretreated TMP fibers had higher strength than the reference sample. Increased amount of fines can also give high strength, Sanchez-Salvador showed that fines had a positive impact on the strength of paper from mechanical pulp [27]. The films of 25 g/m^2 were self-standing, however it was more difficult to make them homogeneous due to the low grammage and the relatively large size of the residual fiber objects. Therefore, the maximum dry strength of the 25 g/m^2 films was lower compared to the 50 g/m^2 films.

The pad product is aimed at being used in wet conditions, so the wet strength of the films was also measured. The films have a relatively high wet strength for TMP films thanks to the final hot pressing step [24]. Hot

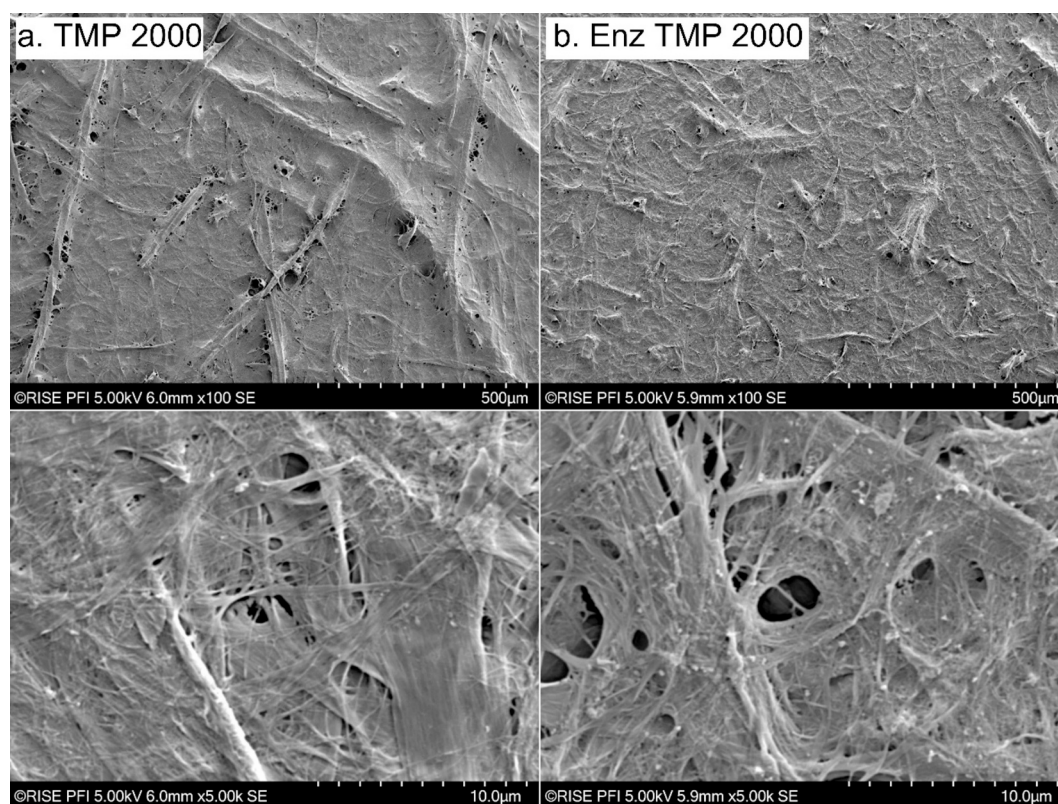


Fig. 4. SEM images of a. TMP 2000 (left column) and b. Enz-TMP 2000 (right column). Images acquired with $100\times$ magnification (upper panel) and $5000\times$ magnification (lower panel). The samples were air-dried before imaging.

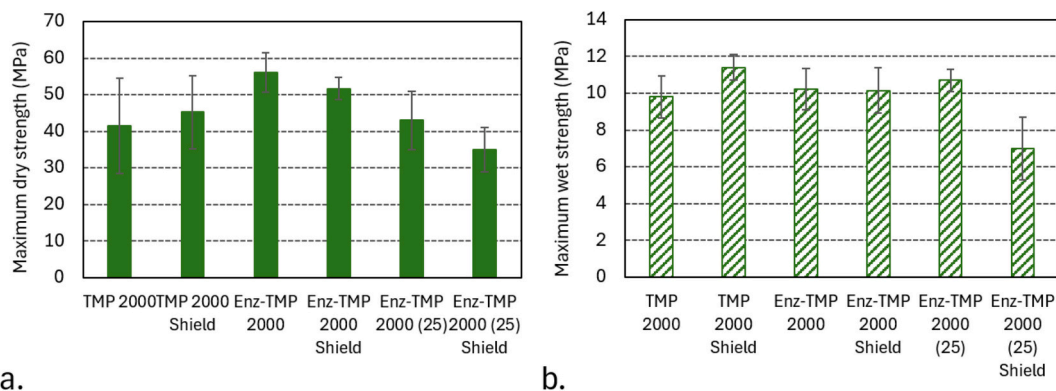


Fig. 5. a. Dry and b. wet tensile strength of the TMP 2000 and Enz-TMP 2000 films with and without METNIN™ SHIELD. The error bars represent the standard deviations.

pressing of paper and board have been shown to increase wet strength, and this is even more pronounced in the presence of lignin in the fibers [24,27,28]. The samples with lower grammage were weaker due to their relatively low density. Joellsson et al. showed that development of wet strength for high yield pulps happen when pressing between 150 and 200 °C [28]. All the films had a wet strength retention of over 15% (see Table 3) which is the limit to consider a paper stable in water [28].

Two commercial samples were used for comparison, i) a sample composed of cellulose fibers mixed with heat sealable polymers and ii) a plastic film (thickness 22 µm) made of PE. The wet strengths for the references were 5.7 ± 0.1 MPa and 18 ± 2 MPa for the commercial paper and commercial plastic, respectively. All the TMP films had maximum strength higher or similar than the commercial cellulose/heat sealable polymer sample indicating that the mechanical properties are adequate for the application as protective films.

Water surface interactions were measured to better understand the behavior of the films while in contact with water. Fig. 6 shows the results of the Cobb60 test and contact angle after one minute of contact. In the presence of METNIN™ SHIELD, the water absorption from the coated surface decreased by 87% and 93% for the 50 g/m² made from TMP 2000 and Enz-TMP 2000 respectively. For the 25 g/m² films the decrease was smaller due to lower thickness and inhomogeneities in the films. The presence of METNIN™ SHIELD led to an increase of contact angle for the films of 50 g/m². Importantly, the results show that in addition to protect the fiber core, the Enz-TMP 2000 film can also limit the direct absorption of liquid by the core thanks to the presence of METNIN™ SHIELD on the outside surface of the pads. This characteristic is expected to contribute to the absorption of drip loss while limiting the drying of the food by capillary forces.

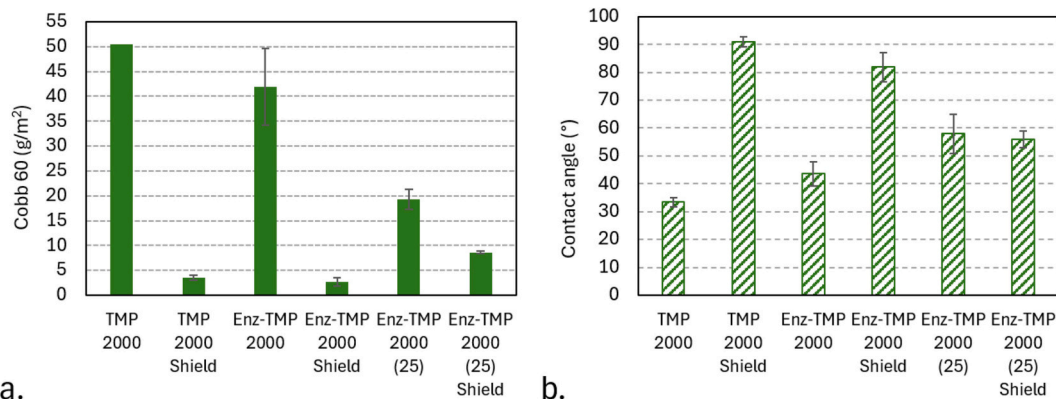


Fig. 6. Effect of the METNIN™ SHIELD on the a. surface water absorption (Cobb 60) and b. contact angle measured after 1 min of contact. Films of TMP 2000 and Enz-TMP 2000 with and without METNIN™ SHIELD were analyzed. For samples TMP 2000, Enz-TMP 2000 and Enz-TMP 2000 (25), the water went through the films during the COBB60 testing (a). The error bars represent the standard deviations.

3.2. Comparison of TMP and CTMP for the core

The main function of the core of the pad is to absorb liquids from the meat. CTMP and TMP were tested here. Their morphological characteristics are shown in Table 4. The CTMP are longer fibers that are less fibrillated than TMP. Freeness (CSF) can also be linked to absorption capacities of the fibers. Fibers with high freeness are good for absorption as they can maintain a bulky structure in wet conditions [13]. CTMP had a freeness of 670 ± 12 mL while TMP had a freeness of 421 ± 1 mL indicating that CTMP was more suitable for the absorbent pads.

The absorption capacities are shown in Fig. 7. The absorption is measured under load to represent the use-case with meat on top of the pad. CTMP had higher absorption capacities than TMP due to the longer fibers and smaller fraction of fines as longer fibers make bulkier structures [13]. In addition, due to the presence of lignin, mechanical fibers are stiff which help to maintain high bulk in wet conditions [12]. Brodin et al. showed that CTMP had greater absorption and retention capacities than softwood kraft pulp [12]. After pressing the cores, the absorption capacity decreases due to higher density of the core after compression (Fig. 1b and c), indicating smaller cavities to absorb the water. Compressed cores of CTMP (thickness ~ 1–2 mm) had higher absorption capacities than TMP and were therefore selected to be used in the full pads.

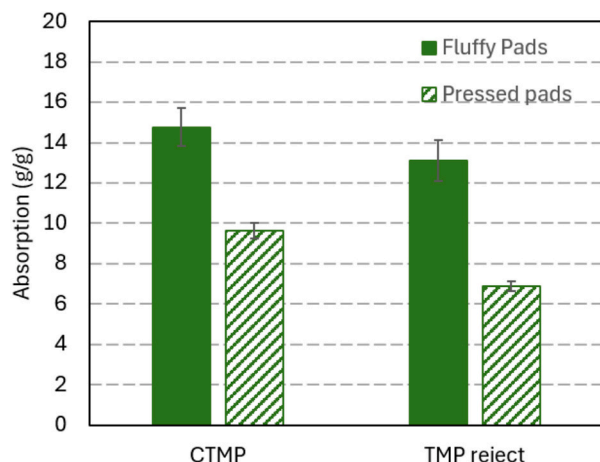
3.3. Optimization of the adhesive

The core of the pad is made of loose fibers that need to be contained. The absorbing fibers should also not be in direct contact with the meat as this could lead to drying of the meat. Here the fibers were contained in

Table 4

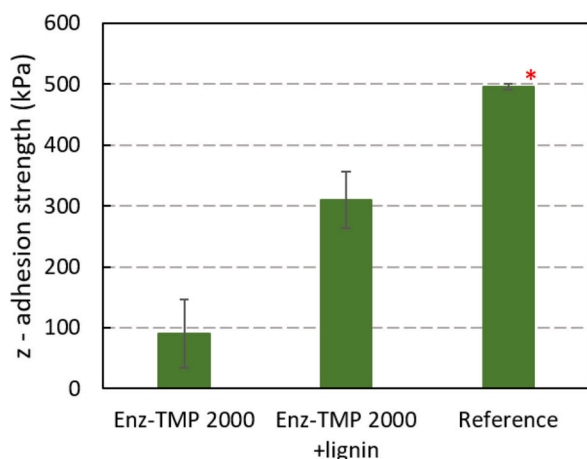
Characteristics of CTMP and TMP fibers tested for the core.

	Fiber length (mm)	Fiber width (mm)	Fibril perimeter (%)	Fines (%)	Freeness (mL)
CTMP	1.52 ± 0.00	34.80 ± 0.00	12.0 ± 0.85	51.8 ± 0.92	670 ± 12
TMP	1.22 ± 0.01	32.40 ± 0.14	21.2 ± 0.07	48.5 ± 0.07	421 ± 1

**Fig. 7.** Absorption under load of CTMP and TMP fibers with and without pressing the core. See also Fig. 1. The error bars represent the standard deviations.

between two films made from Enz-TMP 2000 (25) and coated with METNIN™ SHIELD. To seal the films together on the unmodified side of the film, a paste of Enz-TMP 2000 was used with hot pressing. A sample of Enz-TMP 2000 with lignin was also made to improve the adhesion. The films with lignin-based adhesive were hot-pressed in wet conditions to decrease the softening temperature of lignin, lignin softening is needed to create adhesion [29].

Fig. 8 shows the adhesion strength measured in the z direction (out of plane direction). The reference sample is composed of cellulose fibers containing synthetic fibers for hot sealing. The strength of the adhesion went from 91 ± 56 kPa to 310 ± 47 kPa with the addition of lignin. Lignin can soften at high temperatures and the transition temperature is reduced in the presence of water [10]. During hot pressing the adhesive was still wet, so the presence of water helped to soften the lignin. This helps the adhesive to impregnate the surface of the films. It was shown

**Fig. 8.** a. Adhesion strength of the films sealed with Enz-TMP 2000, Enz-TMP 2000 + lignin and the reference film. *the reference sample was stronger than the adhesive strength of the tape. The error bars represent the standard deviations.

that the softening temperature of lignin and hemicelluloses was linked to the adhesion. Lignin condensation can create covalent bonds that form a continuous matrix between the films. These interactions are resistant to water, in the same way lignin improved the wet strength of moulded cellulose products [24].

3.4. Assembling and testing of full pad

As proof of concept, full pads were made to test their absorption. The pads were assembled following Fig. 2 by using CTMP core, Enz-TMP 2000 (25) + SHIELD films and the Enz-TMP 2000 + lignin adhesive so the pads were fully made of mechanical pulp and lignin (see Fig. 9a). The absorption under load was measured in similar conditions to the core alone (Table 5). The full pads had an absorption under load that was 5.1 g/g, this was lower than when measuring the absorption of the core alone. This is due to the mass of the film that does not contribute to the absorption. To remove the bias, we looked at the actual quantity of liquid that was absorbed, in that case we obtained similar values. It needs to be noted that the quantity of CTMP fibers was the same in both cases. This shows that the absorption capacity of the core is not affected by its incorporation in between the films.

Compared with a commercial sample the absorption under load of our pads was lower. This was again due to the higher grammage of the protective films. It shows the significance of reducing the grammage of the film to maximize the absorption under load that is the measuring standard in the industry. Further, higher fibrillation of the Enz-TMP could provide films with lower grammage while maintaining their strength. Scaling up the production of the films with the barrier coating would be the next step to developing this proof of concept further.

Full pads (300 g/m², core target dimension 155 × 60 mm), with Enz-TMP 2000 films and modified with METNIN™ SHIELD are presented in Fig. 10. The pads dimensions are similar to commercial pads used in fresh meat packaging.

The absorption performance of the pads was 23.8 mL/Pad, as assessed with the BfR-method. The performance of the pads was found to be equivalent to current commercial pads. This is a most positive result as the pads manufactured in this study do not contain plastic, neither polyethylene nor polylactic acid that is usually used in films to cover the cores of pads. The pads (Fig. 10) are composed completely of wood components, i.e., i) CTMP in the core, ii) Enz-TMP 2000 and lignin-based treatment (METNIN™ SHIELD) in the films and iii) Enz-TMP 2000 and lignin to seal the films around the core.

4. Conclusion

Novel pads fully made of TMP, CTMP and lignin have been developed, considering three aspects, i.e., i) comparison of TMP with CTMP as absorbing core, ii) development of fibrillated Enz-TMP for protective films with liquid barrier properties and iii) highly fibrillated Enz-TMP and lignin as adhesive for sealing the films. The results showed that CTMP fibers, that are longer, wider and more intact than TMP, offer higher liquid absorption. On the other hand, based on the more developed morphology of TMP fibers, i.e. more occurrence of fibrils and disrupted fibers, TMP fibers were chosen for additional enzymatic pre-treatment and refining trials to produce an enzymatically pretreated fibrillated TMP grade (Enz-TMP 2000). The Enz-TMP 2000 demonstrated good film-forming capabilities, and films with 25 and 50 g/m² could be manufactured with mechanical strength of 35 and 55 MPa, respectively. The corresponding wet strength was approx. 10 MPa, and

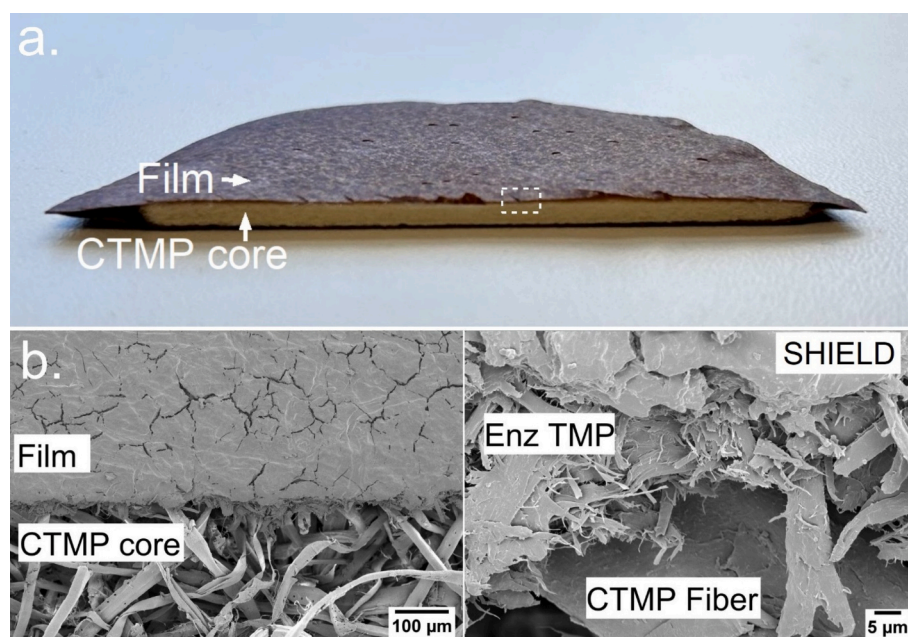


Fig. 9. a. Picture of a full absorbent pad (core, 2 films and adhesive). b. SEM images of the pad observed from the top, from the area indicated with a dashed rectangle in a. The METNIN™ SHIELD layer is visible on the upper part of the picture, and the CTMP fibers are visible on the lower part.

Table 5

Absorption capacities (20 min) of the full pad compared to the core and commercial sample.

Sample	Quantity of absorbed liquid (g)	Absorption under load (g/g)	Film grammage (g/m ²)
CTMP core	5.7 ± 0.2	9.6 ± 0.4	No film
Full pad	5.2 ± 0.4	5.1 ± 0.4	2 × 35*
Commercial sample	5.0 ± 0.3	7.2 ± 0.6	2 × 21

* Corresponds to the film (25 g/m²) and the METNIN™ SHIELD treatment (~10 g/m²).



Fig. 10. Five perforated pads with external dimensions of approx. 170 × 75 mm, composed of a CTMP core (thickness approx. 1–2 mm) and covered by two METNIN™ SHIELD-treated films based on Enz-TMP 2000.

comparable to commercial samples. Such films were surface modified with METNIN™ SHIELD, a lignin-based treatment, and provided good liquid barrier, i.e., Cobb60 values of 2.7 g/m² and 3.5 g/m². The films were comparable to commercial films presently used in absorbent pads for fresh meat packaging. Finally, fully functional pads were manufactured and composed of: i) CTMP in the core, ii) fibrillated Enz-TMP 2000 as the basis for films that were surface modified with METNIN™ SHIELD and iii) an adhesive of fibrillated Enz-TMP/lignin mixture. The absorption performance of the pads was 23.8 mL/Pad, as assessed with the BfR-method.

CRediT authorship contribution statement

Eva Pasquier: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis. **Pauliina Saloranta:** Writing – review & editing, Supervision, Methodology, Investigation. **Erik Andersin:** Writing – review & editing, Supervision, Methodology. **Gary Chingacarrasco:** Writing – review & editing, Visualization, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Co-author Pauliina Saloranta is employed by MetGen Oy that delivered the METNIN SHIELD technology used in this research.

Co-author Erik Andersin is employed by McAiraid's that delivered the CTMP and commercial pads tested in this research.

If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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