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Nanocellulose-polypyrrole-coated paperboard for food packaging application

Benoit Bideau[1], Eric Loranger[1]*, Claude Daneault[2]

1. *Lignocellulosic Materials Research Center, Université du Québec à Trois-Rivières, 3351 boul. des Forges, C.P. 500, Trois-Rivières, QC, G9A 5H7, Canada*
2. *Department of Chemical Engineering, Université du Québec à Trois-Rivières, 3351 boul. des Forges, C.P. 500, Trois-Rivières, QC, G9A 5H7, Canada*

benoit.bideau@uqtr.ca
eric.loranger1@uqtr.ca
claude.daneault@uqtr.ca

ABSTRACT

Currently, studies on packaging that improves the shelf life of perishable food while reducing the waste that is produced upon disposal are encouraged. Thus, exploration of the property improvement of paperboard (Pb) packaging is of interest since this type of packaging is biodegradable and recyclable. This work emphasizes the added value of (2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO) oxidized cellulose nanofibres (TOCN) and polypyrrole (PPy) coating on such paperboard. The mechanical properties and reduced gas permeability of the coated paperboard (CPb) were significantly improved due to the dense network formed by TOCN and polypyrrole particles. These results suggest that surface coating by polypyrrole particles may be utilized for the manufacture of multilayer paperboard containers in industrial applications to reduce packaging waste generated by the often added conventional plastic.

Keywords: Paperboard; polypyrrole; nanofibrillated cellulose; coating; composite

1. INTRODUCTION

Food packaging must provide sufficient protection to ensure food quality and safety. Thus, the selection of packaging materials is of crucial importance in the food market. These materials need to withstand environmental factors, such as temperature and relative humidity, that may affect their properties such as their barrier or permeability characteristics [1]. Moreover, the food industry, which is one of the largest users of packaging, also wants to use the minimal amount of raw materials to reduce their production costs [2].

In response to the growing expectations of our society, works on nanotechnology applications in packaging have attracted significant attention in recent years [3,4,5]. These materials can offer better food quality preservation and product security by slowing and preventing microbial development [6,7]. In particular, the use of active packaging of antibacterial type aims to reduce or inhibit the growth of bacteria that could develop on food [1,7]. Synthetic polymer materials, such as polyethylene, have been widely used as food packaging because they are easy to process, present good gas or grease barrier properties and are adaptable. However, these packaging materials, which are almost always non-degradable, have generated great waste pollution [8]. Therefore, the increasing concerns about environmental impacts and fossil material availability have led to the need for new packaging materials. Currently, substantial efforts are ongoing to find alternative packaging that could still extend the food shelf life and quality while reducing packaging waste [9]. In this matter, the industry is seeking solutions with biodegradable packaging from renewable resources, as their use could solve the waste problem to some extent [1,10].

Thereby, cellulosic materials such as paper or paperboard are increasingly used as food-packaging materials. However, like their synthetic polymer counterpart, bio-based packaging must have the same functions, such as protection, quality preservation and food safety [7]. Unfortunately, the use of these materials is strongly

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*Corresponding author. Tel.: +1 819 376-5011 ext 4518
E-Mail address: Eric.loranger1@uqtr.ca (E.Loranger)

limited because of their susceptibility to humidity and weak mechanical properties [11]. Thus, numerous solutions have been explored to improve the properties of bio-based packaging, such as the blending of these materials with synthetic polymers, chemical modification or a simple coating. Great attention has recently emerged around the use of protective coatings on paperboards for food packaging [10]. This solution allows the product quality and freshness to be kept over time, as required for its commercialization until its consumption [5,12]. Ideally, this layer can also be used as a barrier against organic solvents (increase in the hydrophobicity) or to decrease the water vapour transmission rate and oxygen permeability of paper and paperboard [13], but some challenges would result from this approach. Moreover, the dispersion coating has the advantage of being an efficient and fast pace production process. This process allows the use of mono or multilayer structures of the coated paperboard, with acceptable properties for food packaging, and this process can lead to a more compostable product if it has the right kind of coating [14,15].

Thus, the use of biopolymers, as dispersion coating, is increasing, especially the use of cellulosic fibres (nanocellulose or microfibres), which are biodegradable and recyclable [5,16]. Moreover, cellulose nanofibres have even demonstrated good resistance to oxygen in addition to their excellent mechanical properties due to the creation of a tight network [17,18,19]. Nonetheless, the high water affinity of cellulose is an important inconvenience for some packaging. Blends of biopolymers or synthetic polymers can solve this problem. Polypyrrole (PPy) coupled with cellulosic material has demonstrated an increase in the hydrophobic character and a decrease of the water vapour transmission rate (WVTR) [20]. The PPy coating on cellulosic materials was found as a very interesting alternative to preserve food quality and safety because it exhibits complementary attractive properties such as antibacterial and antioxidant properties [21,22,23].

The goal of this study is to prepare a coated paperboard with TOCN and polypyrrole as a food packaging medium. This coating should combine the biodegradability, the mechanical properties and low gas permeability of the nanocellulose with the physico-chemical properties of PPy. If successful, the multilayer composite appears to be an excellent candidate for active food packaging.

2. MATERIALS AND METHODS

2.1. Materials

Pyrrole (C_4H_5N) and iron (III) chloride ($FeCl_3$) were purchased from Sigma Aldrich and used as received. The TOCN gel was produced through the TEMPO oxidation and sonication treatments of bleached Kraft wood pulp [24]. This gel is composed of 30% long fibres (micro) and 70% short fibres (nanofibres) with an average width and length of approximately 3.5 ± 1.0 nm and 306 ± 112 nm, respectively [25]. Moreover, the carboxyl rate was evaluated according to [24] as 1480 ± 40 mmol/kg. A commercial unbleached flat paperboard (Pb) with a thickness of 50 μm and grammage of 45 g/m^2 was used.

2.2. Methods

2.2.1.Coating process

As the first step, 2 ml of a pyrrole solution (98%) was added to 80 ml of TOCN suspension (0.5% w/w). The mixture was then stirred for 10 minutes, and 10 ml of a 0.3 M oxidant solution ($FeCl_3$) was added to initiate the polymerization of pyrrole into polypyrrole (Figure 1). The mixture was kept under stirring for 30 minutes before turning black, showing signs of pyrrole polymerization. Afterwards, the TOCN-PPy mixture was rinsed with demineralized water before it was cast on the paperboard. Then, the coated paperboard (CPb) was dried under ambient air before it was calendered three times at room temperature and 200 Psi (1.3 MPa) to obtain a flat paperboard.

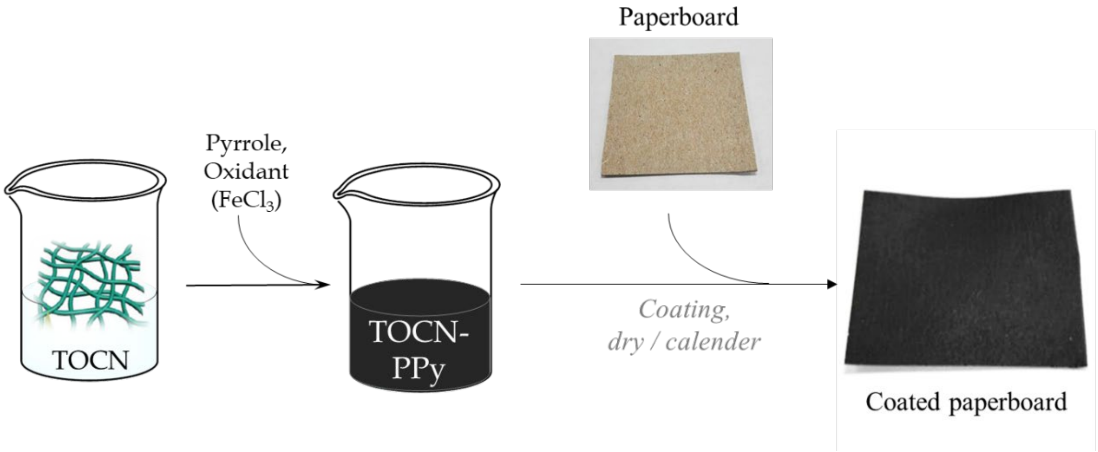

Figure 1 : Schematic illustration of the coating of paperboard by TOCN/PPy

2.2.2. Structural characterization of paperboards

Before characterizations, the samples were conditioned at room temperature and a relative humidity (RH) of 50% for 24 h. The weight was determined as an average value of the Pb and CPb by measuring at least six samples, with the precision of ± 0.001 g. According to the standard ISO 534:2011, the thickness was measured with a Lhomargy micrometre (± 0.01 mm) and was determined by six measurements from Pb and CPb. These values were also checked by cross-sectional (obtained by a microtome) scanning electron microscopy (SEM) images of our samples using a VP-SEM SU 1510 (Hitachi, Japan). Additionally, the morphology of the paperboards was obtained using this SEM equipped with energy-dispersive X-ray spectroscopy (EDX) analysis from Oxford Instruments (United Kingdom). The samples were gold-coated using an International Scientific Instrument PS-2 coating unit (India). Attenuated Total Reflectance - Fourier Transform Infrared Spectroscopy (ATR-FTIR) spectra were obtained at room temperature on a Nicolet IS10 FT-IR spectrometer (ThermoScientific, USA). Each spectrum was acquired in the range of 3600–600 cm^{-1} from 16 scans with a resolution of 4 cm^{-1}. Duplicates of each sample were realized at three different points.

2.2.3.Paperboards properties characterization

The air permeability of the samples was measured by a Parker Print Surf instrument, according to the standard ISO 5636:3. The airflow that passes through the sample for a known surface area, when clamped between two measuring rings at a tightening pressure of 1960 kPa, is expressed in ml/min. Tensile strength of the paperboards was measured at 10 mm/min extension speed on a universal testing machine (Instron 4201) at room temperature and controlled humidity. Young's modulus was determined from the stress–strain curves. The samples used in these measurements were cut from the coated paperboard and paperboard to length of 25 mm, width of 3 mm and thickness in the range of approximately 0.05 mm. The average value of five replicates for each paperboard was collected. Static contact angles were determined by a FTA4000 contact angle measuring system (First Ten Angstroms). At least 5 drops (approximately 3.10^{-2} µl in volume) of distilled water were deposited onto each paperboard and a total of 300 images were captured within 90 seconds for each drop.

2.2.4.Food packaging simulation

A simulation with cherry tomatoes was performed to determine if the CPb presents any advantage for use as food packaging. Small boxes from paperboard and coated paperboard were prepared. Since future box could need to be printed, which the final black colour prevent according to our previous work, an antibacterial agent should be added [20,22] by coating the interior faces of the boxes. Tomatoes were then placed inside the boxes, thus directly in contact with the coating, for 10 days at room temperature before being opened. The control was kept in open air at room temperature for 10 days. Both the boxes and the control were exposed to the same air movement or light exposition.

2.2.5.Recyclability evaluation

To recycle such paperboard, it would be necessary to separate the polypyrrole from the paperboard. To evaluate the recyclability of our coated paperboard, they were placed in water for ten minutes in order for the uncoated side to absorb water and promote the removal. Then, the coating was removed with a spatula, exposing the cardboard. While this process is far from a commercial pulper, this experiment is based on the same conditions that the cardboard would be exposed to. Real recycling trials will be conducted in the near future to access the exact conditions required for commercial pulp.

3. RESULTS AND DISCUSSION

3.1. Morphology and structure

Our recent studies [20,22] have shown excellent properties when using TOCN as a support material. However, in order to minimize the cost of such modified packaging, we aim to reduce the proportion of TOCN, which are expensive to produce, or even completely substitute them by a less expensive classic paperboard. The brown colour of the paperboard will nevertheless be replaced by the dark colour of the PPy coating. Table 1 shows that the coating increases the thickness of the paperboard by 2 µm on average. The density is also affected since the latter increases by 3.5 g/m^2 on average. Although this increase would be detrimental to the shipping cost as industries want to reduce their packaging weight, this additional thin coating will improve the paperboard characteristics.

Table 1: Grammage (g/m^2) and thickness (µm) of each paperboard

	Grammage (g/m^2)	*Thickness (µm)*
Paperboard	45.5 ± 0.9	501.0 ± 2.0
Coated Paperboard	49.0 ± 0.7	520.0 ± 7.0

Figure 2 shows the absorbance spectra of paperboard (Pb) and coated paperboard (cPb). It is possible to distinguish the characteristic cellulose bands of C-O-C, C-H and O-H at 1028, 2916 and 3331 cm^{-1} respectively [20]. In the case of CPb, characteristic peaks at 786, 894, 1203 and 1542 cm^{-1}, which are assigned respectively to C=C and C-C stretching vibrations, C-N stretching vibration, C-H in-plane vibration and pyrrole ring vibrations, can be found. These bands indicate that PPy can be indeed found on the surface of the paperboard [25]. The presence of TOCN cannot be precisely determined because the nanofibres are entirely covered by PPy during polymerization, and if any TOCN were not coated, their vibrations are almost the same as the cellulose vibration.

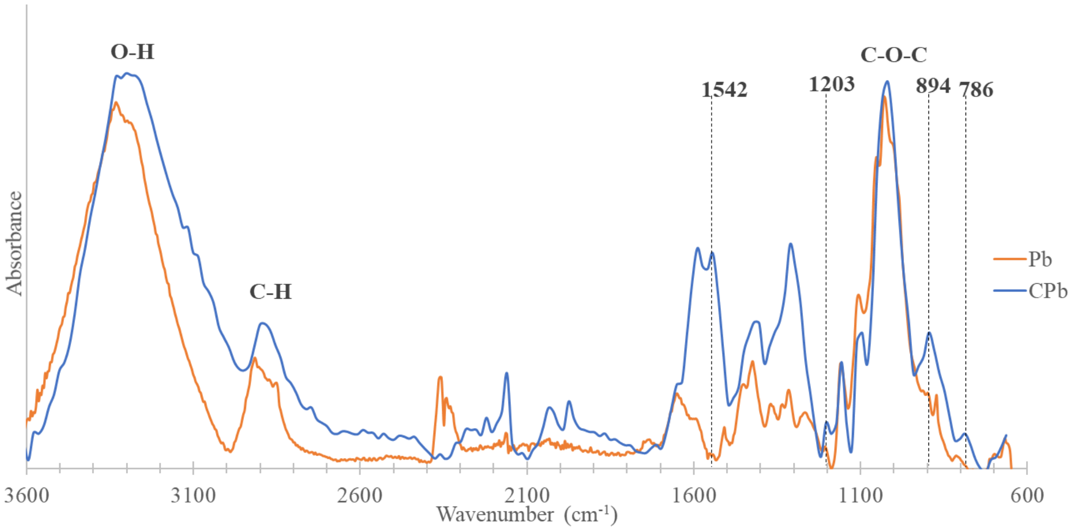

Figure 2 : ATR-FT-IR spectra of paperboard (Pb) and coated paperboard (CPb)

The SEM images show the surface of our paperboards and the cross section of the coated paperboard (Figure 3c). The paperboard (Figure 3a) structure is arranged in sheets and fibres, which creates a disorderly entanglement network. The surface appears very rough with many pores and some aggregates. When cross-referenced with Figure 3b, it can be deduced that the paperboard is completely covered by the coating of TOCN/PPy. During the pyrrole polymerization, PPy nanoparticles coat the TOCN, which explains that the cellulose structure disappears under the conjugated polymer as shown in Table 2 by the variation of the carbon and oxygen contents. The surface appears more planar and the pores present on the surface of the paperboard are closed by the coating. In Figure 3c, a cross section of the coated paperboard shows the coating on top of the paperboard. The layer of PPy determined by the ImageJ® software was estimated to be 20 µm, which is similar to the value deducted from Table 1. The structure of the TOCN/PPy coating is relatively close and compact. As polypyrrole has already started its polymerization before being applied to the paperboard, it seems that the coating could not diffuse deeply in the paperboard surface layer. In the middle, the paperboard presents a more open and disorderly structure than in the top coating. In this part, no trace of nitrogen has been detected by EDX, which confirms that the polypyrrole did not diffuse. Indeed, quantitative analysis of the elements C, O, N, and others (Al, Si and Ca) was performed on the coated paperboard (top layer and central layer) and paperboard by EDX analysis and reported in Table 2. Analysis performed on the cross-section and surface of CPb confirmed the presence of nitrogen only on the surface, and therefore the presence of PPy since paperboard is only composed of carbon, hydrogen and oxygen. The proportion of nitrogen (7%) is in agreement with the literature [20]. The aggregates in Figure 3a are mostly composed of Si, Al and Ca and are likely traces of calcium carbonate or clay often found in the whitewater used in the papermaking process. While not used in this kind of cardboard, the mills are not always exclusively dedicated to such product and often share a great deal of whitewater.

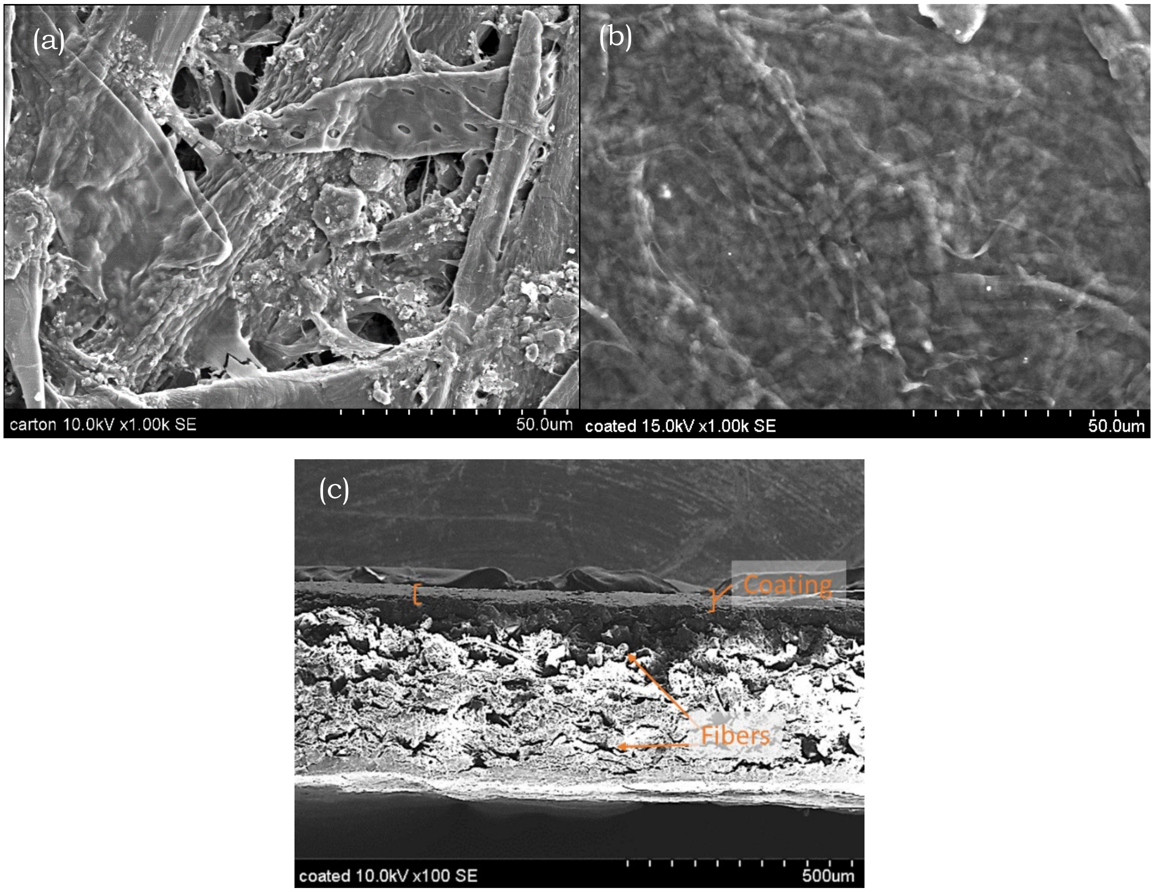

Figure 3: SEM micrographs of the paperboard (a) and coated paperboard (b: surface; c: cross-section)

Table 2: Atomic percent of elements for different positions on the film

	Paperboard	*Coated Paperboard (top layer coating)*	*Coated Paperboard (central layer)*
C (At%)	51.0	67.0	59.2
O (At%)	48.8	26.0	40.4
N (At%)	0.0	7.0	0.0
Si, Al, Ca (At%)	0.2	0.0	0.4

3.2. Mechanical characterization

The results for the mechanical properties of each paperboard are given in Table 3 and Figure 4. Figure 4 shows clearly the advantage of using TOCN-PPy coating on the paperboard mechanical properties. Indeed, TOCN are known to confer high mechanical properties and are used, in this case, as a reinforcement agent in composite materials [16]. The strength of TOCN can be attributed to the stronger interactions due to the carboxyl group presence and inter- and intra-molecular hydrogen bonding occurring in the entangled structure of the cellulose nanofibres. For PPy, it is known to have very poor mechanical properties due to the chemical polymerization which results in the formation of a PPy powder and thus, no film can be obtained. In fact, the aliphatic chains cannot create strong interactions like hydrogen bonding to confer good cohesion between PPy particles.

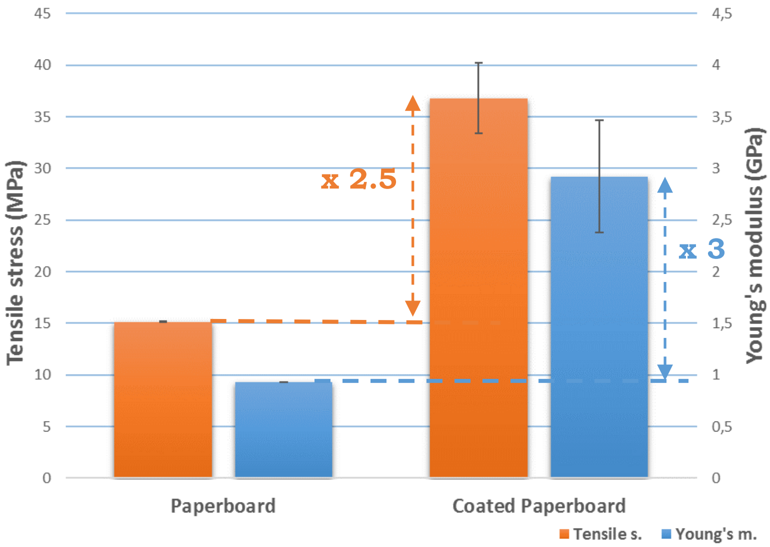

Figure 4: Mechanical properties (tensile stress and Young's modulus) of each paperboard

In this context, the tensile stress and the Young's Modulus increased by 2.5 and 3 times, respectively, in the presence of the coating. The improvement of the strength of the coated cardboard can be associated mainly to the presence of TOCN chains, which are a strong reinforcing material. However, the results indicate that the elongation at break (%) is highly decreased in the presence of TOCN-PPy (Table 3), which is also confirmed by the increased Young's modulus, indicating a higher stiffness. It is possible to think that the PPy presence limits the interactions between TOCN chains because of the PPy aliphatic chains which break the intermolecular hydrogen bonding. Nonetheless, the dense coating, visible in Figure 3c, acts as an interesting reinforcing agent.

Table 3: Mechanical properties, air permeability and contact angle of each paperboard

	Paperboard	***Coated Paperboard***
Tensile stress (MPa)	15.16 ± 0.01	36.80 ± 3.38
Young's modulus (GPa)	0.93 ± 0.01	2.92 ± 0.54
Elongation at the break (%)	7.66 ± 0.03	2.36 ± 0.34
Air permeability (ml/min)	12.5 ± 1.0	> 1.0
Contact angle (°)	74.3 ± 1.3	79.8 ± 1.2

In addition to increasing the mechanical properties, the presence of the coating increases slightly the hydrophobic character. The contact angle values are shown in Table 3. Though the paperboards are mainly constituted by cellulosic material, its apparent that the contact angle is high (74°) compared to pure cellulosic film (25°) [26]. It is possible to think that this behaviour is due to the presence of lignin since we used unbleached paperboard. The coating gave the expected increase in hydrophobic character (80°), which is explained by the presence of the PPy aliphatic chain [22]. Moreover, the thin PPy coating affects the surface morphology, thus influencing the paperboard absorption behaviour, as it is possible to see in Figure 5.

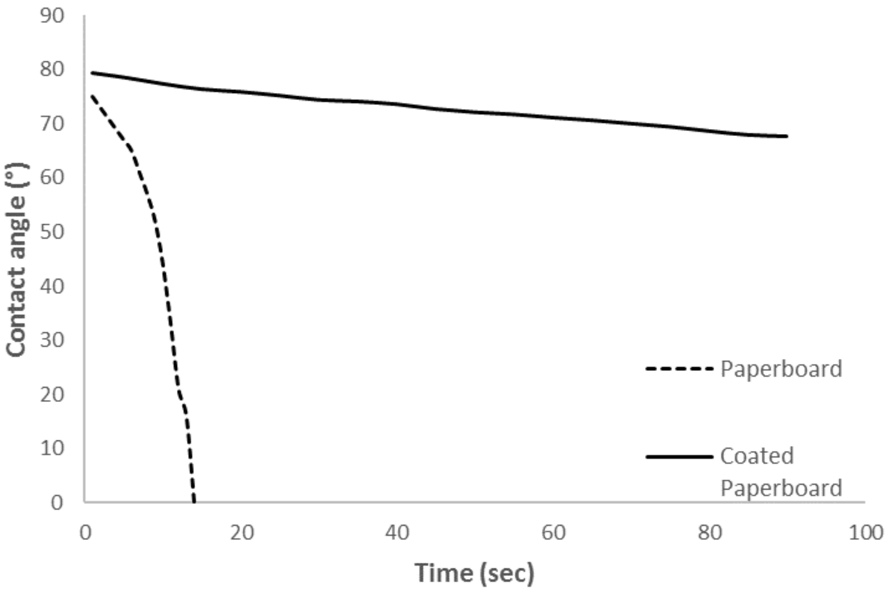

Figure 5: Water absorption of each paperboard

In Figure 5, without the coating, the water drop was absorbed in 14 seconds. The contact angle decreases quickly due to the high-water affinity of the cellulose fibres. In comparison, we can observe a weak absorption of water for CPb. This result can be explained by the coating structure, which is very dense, with the PPy layer on the surface that completely covers the cellulose fibres, thus concealing any presence of the highly hydrophilic material. This explains the slowest decrease in contact angles for CPb (80° to 67° after 90 seconds) than for Pb. Therefore, the coating decreases significantly the paperboard wettability which is very interesting for food packaging applications since the wettability reduces the material mechanical properties.

3.3. Food packaging simulation

Food quality has become very important for consumers who not only want good products but also products with a good-looking appearance. However, if the food is not consumed quickly, some factors (micro-organisms, oxygen, moisture and light) can accelerate its degradation and degrade its look. This phenomenon is visible especially in fruits and vegetables with advanced maturation (loss of firmness, wrinkles presence, colour changes) before rotting. These two stages generally lead to the waste of food as it is believed to be no longer good for consumption by 21st century consumers. Thus, following our previous work, we wanted to adapt PPy properties to paperboard food packaging [20,22]. These studies have demonstrated very good antibacterial and antioxidant properties for this field in order to improve the food preservation.

As a qualitative test, cherry tomatoes were placed for 10 days in Pb and CPb boxes. Several tomatoes were left in the open air as a control. Figure 6 shows that after 10 days, the tomatoes from the control are all in an advanced maturation state and very wrinkled. They do not appear to be consumable according to many consumers. Most of the tomatoes placed in the Pb box are in the same condition; however, some are still firm after 10 days. Finally, in the coated paperboard boxes, all tomatoes have kept a firm texture and an attractive look for consumption. This can be explained by the fact that the polypyrrole exhibits antioxidant properties which delay the tomato maturation and because there is less exchange with the external environment (Table 3). The air permeability is considerably reduced compared to paperboard (> 1.0 versus 12.5 ml/min). Since oxygen is one of the main factors of food degradation [27], a reduction of exchanges with the outside makes it possible to reduce the degradation phenomenon. While this testing is mostly qualitative, these results show the notable effect of the preservation of food with our coated paperboard.

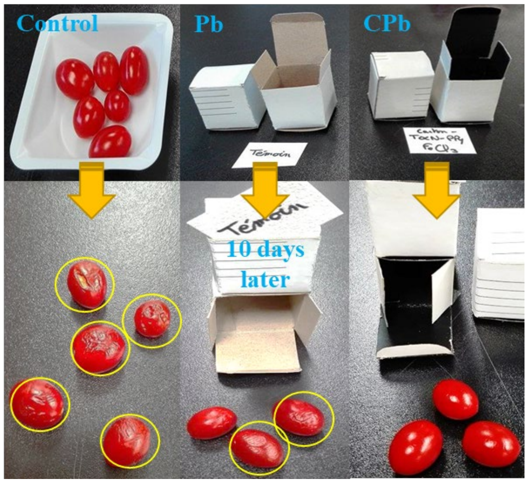

Figure 6: Food packaging simulation after 10 days with paperboard (Pb) and coated paperboard (CPb) boxes

Moreover, previous work in our laboratory [28] demonstrated that this new type of active packaging is safe with regard to the possible particle leaching problems. Indeed, it has been shown that after submersion for 48 h, only 0.2×10^{-3} ($\pm 5.2 \times 10^{-6}$) mol/L of PPy was released, thus making it a potential candidate for direct contact with food. Further investigations are required to assess that all requirements such as food contact authorization, sensory problems, long-term exposition, etc., are indeed achieved for a food packaging product according to food law compliance regulations.

3.4. Recyclability evaluation

The increased use of plastic packaging has become a problem for nature [29]. Therefore, the use of biodegradable or recyclable packaging is an interesting solution. In this respect, paperboard is a material that is easily recyclable and reusable, due to the bio-based material presence. In contrast, polypyrrole is not recyclable. Thus, it is important to be able to separate the PPy coating from the paperboard in order for our coated paperboard to be recyclable. After ten minutes in water, the coating can easily be removed from the paperboard, with some paperboard fibres (Figure 7). A loss of 10 to 15 wt% of the paperboard is noticed, but this indicates that almost 80 wt% of Pb can be reused. The increased shear rate of a commercial pulper will increase the PPy coating removal, but further experiments are needed to formally access the phenomenon. However, these preliminary testing show that the coating can be easily removed and that this product could be recyclable in part.

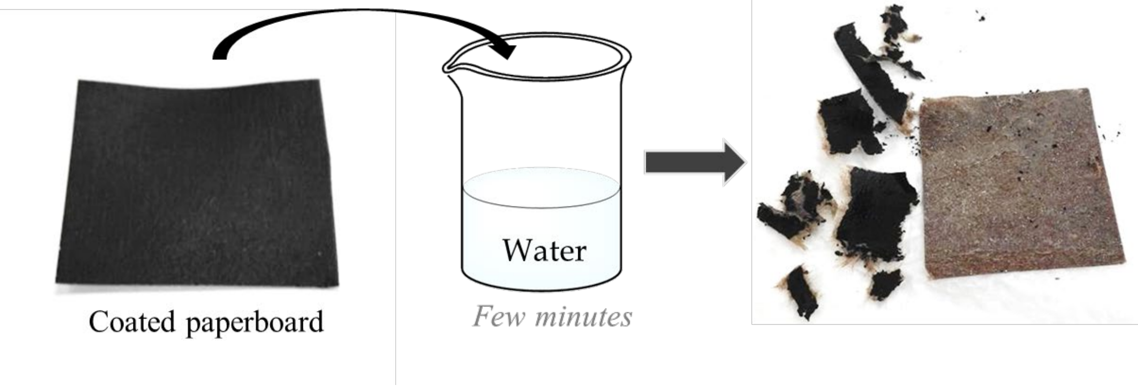

Figure 7: Separation process of coated paperboard to recycle paperboard

4. CONCLUSION

In this study, we reported the synthesis of a TOCN-PPy paperboard coating by chemical polymerization of pyrrole. With this coating, we improved the mechanical properties and greatly reduce the gas permeability of the paperboard. The improvement was attributed to the dense network formed by TOCN and polypyrrole particles on the paperboard surface. In addition, PPy aliphatic chains closed the paperboard structure and reduced the air permeability and the water absorption. Moreover, this method demonstrated that it was possible to produce a paperboard-based food packaging that was more efficient than usual paperboard packaging, allowing an improvement of the shelf life of perishable foods. Furthermore, the biodegradable character could also reduce packaging waste generated by plastics, since it is possible to separate the paperboard from the coating for recycling. Although the properties of plastic packaging remain better than any paperboard, this new TOCN-PPy coated packaging appears as an interesting alternative to the classic polymers used.

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