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Effects of Non-Wood Fibres in Printed Paper Substrate on Barrier and Migration Properties

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Abstract: Nowadays, there is a strong initiative to use recycled or biodegradable materials in all aspects of production including the graphic industry. In this study, paper was used as a material fulfilling the two of mentioned properties. Under laboratory conditions, papers were made of 70% pulp from recycled wood fibres with an addition of 30% straw pulp (wheat, barley or triticale). Considering the importance of the possibility of printing such media based on their end use, the influence of fibre type on vapour barrier properties was studied and overall migration to hydrophilic and fatty food simulants was measured. Analyses were performed on digital, flexographic, and offset prints obtained by printing laboratory papers with UV-curable black ink. It was found that prints produced using the offset technique, in which the ink remains on the surface of the paper, had lower overall levels of migration compared to other printing techniques. The paper produced appears to have the potential to be used as a secondary food packaging material.

Keywords: non-wood fibres; overall migration; printing techniques, recycled fibres; water vapour permeability

1 INTRODUCTION

Over the past decade, there have been significant initiatives to reduce the negative environmental impact of synthetic materials and related products in the packaging industry. As a result, there is a strong demand for greener, sustainable, renewable and bio-based packaging materials. In this context, paper is considered as one of the environmentally friendly and sustainable packaging materials used for both food and non-food products [1]. The interest in paper-based packaging materials dates back centuries and accounts for 31% of the global packaging market segment in 2017 [2]. Its eco-friendly label makes paper the first choice for the food industry [3], where it is mainly used as primary or secondary packaging. While primary packaging implies direct contact with food, secondary packaging aims not to come into direct contact with food, but to serve for transportation and storage of the primary packaging. Worldwide, half of all paper produced is used as packaging material. About 420 million tonnes of paper-based packaging were produced, including nearly 90 million tonnes in Europe in 2018 [4, 5]. Paper is mostly made from a network of cellulose fibres derived from wood and non-wood plants, forming a compact material. As public interest in the conservation of natural resources has increased in recent years, emphasis has been placed on the use of alternative fibre sources by recycling used paper as a source of secondary fibres and using virgin non-wood fibres (including bamboo, bagasse, hemp, wheat straw, flax) in the paper industry [6,7]. The use of alternative sources of cellulose fibre reduces pressure on forest resources, which has recently become attractive not only because of limited wood supply but also because of environmental concerns in countries with acceptable wood sources. The reuse of agricultural waste for fibre production also contributes to sustainability. This raw material is interesting because it is available worldwide on an annual basis and its chemical composition makes it suitable for processing into pulp. Unlike wood materials, it is important to know that straw contains less lignin and more hemicellulose with an extremely high content of extractives at the same cellulose content [6]. The principle of pulping is the separation of the fibres from the plant tissue by chemical cooking to remove the lignin, or by mechanical separation combined with chemical softening, which is much easier and shorter for such raw materials than for wood raw materials [8]. However, it should be emphasised that straw cannot have an advantage over wood raw materials because the quality of straw is naturally very heterogeneous, and depends on the type of grain, the time of sowing and harvesting, and vegetation conditions. Therefore, this type of raw material provides fibres that are mixed with wood fibre pulp in varying proportions during paper production. The quality of prints on papers with pulp from agricultural residues has not been thoroughly investigated. In recent research [9], the possibility of using mixed pulp from wood and triticale straw up to 30% was shown on a laboratory scale. The use of paper with pulp from agricultural residues (wheat, barley and triticale straw) in the printing industry has been confirmed in several studies [10-13]. The main advantages of using wood-based papers for commercial packaging include: excellent print quality for most boards, very good mechanical protection for the products, relatively low production and processing costs, and easy recyclability [4]. The selection of a suitable substrate is very important, as the interaction of the substrate with the printing inks should not affect the packaged product and should not change the appearance of the packaging. Information on printed food packaging plays an important role in presentation, promotion and consumer information [14]. However, mixing wood fibre pulp with non-wood fibres can change the appearance of the packaging. In fact, the choice of raw material for pulp and paper production can affect the papermaking process and paper quality in terms of its physicochemical, barrier, mechanical, and printing properties [4].

Paper is an extremely porous medium that has small open spaces in the form of pores or voids that affect the interaction between inks and paper, as well as permeability properties [1]. The water vapour barrier properties of the substrate play an important role in mechanical resistance and maintaining the quality and safety of packaged food [15]. Paper contains cellulose or other fibres whose hydrophilic nature results in poor water vapour barrier properties [16]. Characterization of permeability is of great importance in predicting the barrier and printing properties of paper [17].

Another mass transfer, migration, is described as the transfer of low molecular weight compounds from a material to a packaged item. Paper and board are porous materials from which various compounds can migrate by diffusion from the packaging into the food. Other interactions include those from outside through the packaging, through a gas phase, or the set-off during material storage on rolls or in stacks. Excessive migration can occur due to various factors such as the duration of contact, the rising temperature, and expansion of the contact area, the composition of the ink and the composition of the packaged food rich in aggressive substances. When used as food packaging, the migration of ink can seriously affect food safety, consumer health, and consequently affect the acceptance (withdrawal) of such a product on the global market. Recycled paper must meet a number of basic safety criteria regarding possible migration. In the paper recycling process, many chemicals are used for bleaching and strengthening. It makes a big difference whether the material is used as primary or secondary packaging, with less impact and risk when used as secondary packaging. However, even if it is used as secondary packaging, which could be the case with papers used in this study, it is important to know the risks of the potential migration values. A recent review article [18] discussed approaches to reduce chemical migration from recycled paperboard. Inks have been shown to migrate through paper into dry and liquid food simulants [19]. The use of recycled materials such as fibres from recovered paper may also result in direct contact between ink components and food, or the pathway through the material could be shorter or altered if alternative fibres are used. Print stability is another extremely important parameter for reproduction quality, especially in the packaging industry. Castle has published a comprehensive review of potential contaminants in food contact materials made from recycled paper and cardboard [20] and there is a wide range of different ink and overprint varnish formulations available to meet the requirements of printing processes, substrate types and specifications for food packaging [14]. A number of specially formulated low-migration inks and overprint varnishes are available for the production of paper packaging for food. Variations can occur depending on the printing technique (offset, gravure, flexographic, digital) and ink type (solvent-based, water-based, cationic UV-curable, low-migration UV-curable, electron beam-cured) [14].

The aim of this study was to analyse the different properties of recycled paper enriched with non-wood fibres that can be used as secondary food packaging. Three printing techniques commonly used for packaging (digital, flexographic and offset) were selected for printing these innovative paper substrates with black UV-curable ink. All printing techniques used printing conditions that ensure high print quality at full tone. All printed substrates were tested for water vapour barrier, and print quality parameters (optical ink density and undertone) were determined. The stability of the prints was also tested by comparing the overall migration in two food simulants with unprinted paper samples.

2 MATERIALS AND METHODS

2.1 Papers with Straw Pulp

Laboratory papers weighing approximately 42.5 g/m^2, formed by Rapid-Köthen sheet former (FRANK-PTI) according to the standard EN ISO 5269-2:2004 [21], were made entirely from recycled wood pulp or from a blend of 70% recycled wood pulp and 30% unbleached wheat, barley, or triticale pulp (Tab. 1).

Table 1 Labeling of laboratory-produced papers used as printing substrates

Paper type	Composition	
	Straw pulp, %	Recycled pulp, %
N	0	100
NT	30	70
NB	30	70
NW	30	70
* straw type: N = only recycled wood; T = triticale; B = barley; W = wheat		

Semi-chemical straw pulp was obtained from crop residues left in the fields after harvest. These were collected, purified, manually cut and processed by the soda pulping method [22].

2.2 Printing Techniques

In this study, the three most common printing techniques were used: digital, flexographic and offset printing, in which black, UV-curable ink was applied to laboratory papers to achieve a full tone.

2.2.1 Digital Technique

All laboratory papers were printed using a digital EFI Rastek H652 UV-curable inkjet printer. Variable piezoelectric drop-on-demand printing technology ensures that full tone areas are printed with black ink on each laboratory-made paper at a resolution of 600 × 600 dpi (with high-quality mode in eight passes), at a printing speed of 12.10 m^2/h. In this technique, the printing process is performed based on ink droplets sprayed from the print head nozzles. The data from the digital print job is transferred directly to the inkjet system, which transfers the ink to the printing substrate via the nozzles [23].

2.2.2 Flexographic Technique

Printing on laboratory papers with black UV-curable ink Solarflex Integra (Sun Chemical) in fulltone pattern was performed with flexographic laboratory device F1-basic Printability Tester. It was performed at a speed of 0.5 m/s, a printing force of 300 N and an anilox roller force of 200 N. An anilox roller with 90 lines cm^{-1} (60° raster angle) and a cell volume of 18 cm^3/m^2 was used for printing at a temperature of 23 °C and a relative humidity of 50%. The prints were additionally dried using the Technigraf Aktiprint

L 10-1 UV dryer (UV-C tube, with a light source power of 120 W/cm and an intensity of 60%).

2.2.3 Offset Technique

All laboratory-produced papers were printed in full tone by laboratory device Prüfbau multipurpose printability testing machine with SunCure Starluxe low migration black ink (manufacturer Sun Chemicals) at a temperature of 23 °C and relative humidity of 50%. It was printed at a speed of 0.5 m/s and a pressure of 600 N. The prints were then additionally dried using a Technigraf Aktiprint L 10-1 UV dryer [24].

2.3 Analysis

2.3.1 Material Thickness

Material thickness (x) was measured using a digital gauge with an accuracy of 0.001 mm (Digimet, HP, Helios Preisser, Germany). For all calculations, the average value of ten thickness measurements at different positions per paper type was used.

2.3.2 Water Vapour Permeability

The water vapour permeability (WVP) of the paper and print samples was determined gravimetrically according to the modified ASTM E96-80 standard method [25, 26]. The surface area (A; dm^2) of the sample and the sample thickness (x; m) was measured before experiments. The relative humidity difference was set up to 100% > 30%, and the measurement temperature was 25±1 °C. The glass cells were filled with distilled water and placed in a ventilated climatic chamber (Memmert HPP110, Germany) and difference in pressure on both sides of the sample (Δp; Pa) and slope on the graph ($G\ t^{-1}$; g/s) were monitored. The water vapour permeability (WVP, g/(m·s·Pa)) and the water vapour transmission rate ($WVTR$, g/(m^2·s)) were calculated from the change in cell weight over time at steady state according to Eqs. (1) and (2), respectively.

$$WVP = \frac{WVTR \cdot x}{\Delta p} \tag{1}$$

$$WVTR = \frac{G}{t \cdot A} \tag{2}$$

2.3.3 Overall migration

Determination of the overall migration values (OM) was performed to verify the maximum amount of low molecular weight compounds that migrated from paper or printed samples into selected food simulants. Aqueous acetic acid (CH_3COOH) 3% [w/v] and ethanol (CH_3CH_2OH, EtOH) 95% [v/v, used as simulants for hydrophilic foods with pH < 4.5 like infusions, coffee, tea, beers (Commission Regulation EU No 10/2011 [27-29]) and were used as simulants for all measurements. Measurements were performed using the migration cell (MigraCell®; FABES Forschungs-GmbH, Munich, Germany) and the immersion method (EN 1186-1 Standard) [30]. The surface area (A; dm^2) of the sample was measured before experiments. The migration cells were stored at 40 °C for 10 days, corresponding to the peer case scenario or potentially prolonged contact. Subsequently, the food simulant solution was evaporated at high temperatures (> 300 °C) in a previously weighed glass cell (m_1; mg). After all the solution was evaporated, the glass cell was dried at 105 °C to constant weight (m_2; mg). All measurements were performed in triplicate. The overall migration (OM, (mg/dm^2)) was calculated using the Eq. (3).

$$OM = \frac{m_2 - m_1}{A} \tag{3}$$

2.3.4 Optical Ink Density

To observe the thickness of the black ink film on laboratory paper substrates (N, NT, NB, NW), the optical ink density parameter was used. The optical ink density (D_i) on all prints obtained with different printing techniques (digital, flexographic, offset) was determined using a densitometer eXact, X-Rite (D50/2º). Since the ink layer is opaque, the optical ink density was calculated from the values of the light intensity (I) reflected by the ink layer in relation to the light intensity (I_0) and the values of the light intensity (I_0) reflected by unprinted paper substrates according to Eq. (4) [31].

$$D_i = \log \frac{I_0}{I} \tag{4}$$

Thus, a higher optical ink density means a higher ink layer or a higher concentration of ink and a higher optical contrast [32].

2.3.5 Undertone

The undertone describes the colour of a thin layer of ink on paper that can be seen through its white background. The determination of undertone in this study was done using the Euclidean colour difference based on the colorimetric values of CIE L^*, a^*, b^* from a white background of the printed laboratory paper and a white background of the unprinted laboratory paper. The undertone values are calculated based on ΔE_{00}^*. The colorimetric values were measured by a spectrophotometer eXact, X-Rite under an illuminant D50 and 2° standard observer. The following Eq. (5) was used to calculate the Euclidean colour difference (ΔE_{00}^*) [33] from the measured values of the transformed lightness difference between samples ($\Delta L'$), the transformed chroma difference between samples ($\Delta C'$), the transformed hue difference between print samples ($\Delta H'$), the rotation function (RT), the parametric factors for the variation in the experimental conditions (k_L, k_C, k_H) and the weighting functions (S_L, S_C, S_H):

$$\Delta E_{00}^{*} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T\left(\frac{\Delta C'}{k_C S_C}\right)\left(\frac{\Delta H'}{k_H S_H}\right)} \quad (5)$$

2.3.6 Data Analysis

Statistical analysis was performed in XLStat using analysis of variance (ANOVA). All experiments were carried out at least in triplicate, and the results were reported as the means and the standard errors of differences of the means of these measurements.

3 RESULTS AND DISCUSSION

3.1 Water Vapour Permeability and Optical Ink Density of Prints

Like other paper properties, air permeability is an indicator of end-use performance and can be used to estimate how inks will penetrate and spread. One of the goals of this research was to measure the permeability to gases (O_2 and CO_2) to see if the presence of inks on papers with straw pulp would have a positive effect on lowering the gas permeability values. Unfortunately, all of the printed samples were extremely permeable which limited the ability to obtain accurate results before the test was finished, so they were not included in this study. The water barrier properties of the paper are important in extending the life cycle of the material. The comparison was made for UV-curable inks applied to a laboratory-produced paper substrate using three different printing techniques: digital (marked "d"), flexographic (marked "f") and offset (marked "off"). The measured *WVP* values were similar for all paper types and were within a range of 10^{-10} g/(m·s·Pa) (Tab. 2). This was attributed to the pore structure and hydrophilic nature of fibre-based paper samples. In [22], it was found that the water absorption of laboratory-produced papers with recycled fibre pulp increased by about 50% with the addition of 30% pulp from triticale, barley, or wheat pulp. Similar *WVP* values were also reported for uncoated commercial paper or for paper coated with a thin layer of biopolymers to reduce the sensitivity of the destructive paper matrix to water vapour [34]. Otherwise, the high permeability values were not surprising, since paper and cardboard are known for their relatively low resistance to moisture and gases [35]. Although the application of polymer ink coatings could improve barrier performance and compensate for the above disadvantage, this was not the case in the present study.

WVTR values ranged from 1.08 to 11.45×10^{-3} g/(m^2·s), with significantly lower values for offset prints (Tab. 2). It is possible that the ink fills the pores, which reduces the permeation of water vapour molecules and decreases the size of voids between fibres [36]. This is attributed to the composition of the ink. The offset inks used in this study were low migration-inks, which have lower optical ink density values [24]. The results were somewhat lower than the values found in the literature (33.29×10^{-3} g/(m^2·s) for the copy paper grade [37].

Table 2 Thickness (x), water vapour permeability (*WVP*) and water vapour transmission rate (*WVTR*) values of unprinted and printed laboratory-produced papers by different printing techniques

Paper type	x (μm)	*WVP* (× 10^{-10} g/(m·s·Pa))	*WVTR* (× 10^{-3} g/(m^2·s))
N unp	90.10±2.13^{b}	3.39±0.05^{c,d}	8.28±0.12^{d,e}
N d	87.20±2.57^{b}	4.53±0.13^{b}	11.45±0.33^{a,b}
N f	85.60±2.23^{b}	3.33±0.16^{c,d}	8.55±0.41^{d,e}
N off	92.14±13.83^{a,b}	5.49±2.30^{b}	1.31±0.55^{a,b}
NT unp	101.67±14.72^{a,b}	3.84±0.23^{b,c,d}	8.06±0.47^{d,e}
NT d	100.01±1.05^{a,b}	3.18±0.14^{d}	7.25±0.33^{e}
NT f	94.80±9.65^{a,b}	3.96±0.15^{b,c,d}	9.21±0.34^{c,d,e}
NT off	96.86±20.64^{a,b}	4.33±1.11^{b,c}	0.99±0.25^{a,b,c,d}
NB unp	91.67±4.08^{a,b}	3.41±0.12^{c,d}	8.34±0.29^{d,e}
NB d	91.70±3.75^{a,b}	3.19±0.17^{d}	7.65±0.41^{d,e}
NB f	92.60±8.58^{a,b}	3.93±0.12^{b,c,d}	9.34±0.29^{b,c,d}
NB off	97.71±12.98^{a,b}	4.77±0.17^{a,b}	1.08±0.04^{a,b,c}
NW unp	101.67±17.22^{a,b}	3.33±0.15^{c,d}	8.30±0.38^{d,e}
NW d	101.05±2.27^{a,b}	3.04±0.12^{d}	7.30±0.29^{e}
NW f	91.43±5.26^{a,b}	3.72±0.13^{b,c,d}	8.96±0.30^{c,d,e}
NW off	107.43±9.68^{a}	5.73±0.55^{a}	1.17±0.11^{a}

* N = laboratory-produced paper from pulp of 100% recycled wood fibres; NT = laboratory-produced paper with addition of 30% triticale pulp; NB = laboratory-produced paper with addition of 30% barley pulp; NW = laboratory-produced paper with addition of 30% wheat pulp; unp = unprinted; d = digital printing technique; f = flexographic printing technique; off =offset printing technique.
$^{a-e}$ Different superscripts within the column indicate significant differences between samples at $p < 0.05$.

The thickness of laboratory-made papers with straw pulp was significantly higher than that of the control sample (N), with no significant differences depending on the type of straw fibre. There were also no significant differences between prints on the same type of paper with respect to the printing technique used.

Table 3 Optical ink density of printed black ink with variuos printing techniques

Paper type	Optical ink density		
	digital prints	flexographic prints	offset prints
N	0.96 ± 0.01$^{a\,B}$	1.15 ± 0.01$^{a\,A}$	0.96 ± 0.01$^{b\,B}$
NT	0.91 ± 0.02$^{c\,C}$	1.10 ± 0.01$^{c\,A}$	1.01 ± 0.02$^{a\,B}$
NB	0.90 ± 0.01$^{c\,C}$	1.13 ± 0.01$^{b\,A}$	1.02 ± 0.02$^{a\,B}$
NW	0.93 ± 0.02$^{b\,C}$	1.13 ± 0.01$^{b\,A}$	0.98 ± 0.03$^{b\,B}$

Different superscripts within the column ($^{a-c}$) and in the row ($^{A-C}$) indicate significant differences between samples at $p < 0.05$.

The values of optical ink density in Tab. 3 show that the highest values are contained on all laboratory papers produced by the flexographic printing technique, while the values on offset and digital prints were very close to each other.

3.2 Overall Migration

The overall migration was measured to verify the migration of all substances from the paper and print samples in 3% aqueous acetic acid and 95% ethanol [38, 39]. Although these papers are not primarily intended for primary food packaging, it was interesting to investigate how papers with a novel type of incorporated fibres would behave if they came into contact with food. Potentially, they could be used as disposable takeaway food materials or as secondary packaging.

The results of overall migration values (*OM*) for unprinted paper samples are shown in Fig. 1, and for printed samples in Fig. 2, where the red line indicates the legal overall migration limit of 10 mg/dm^2 [29].

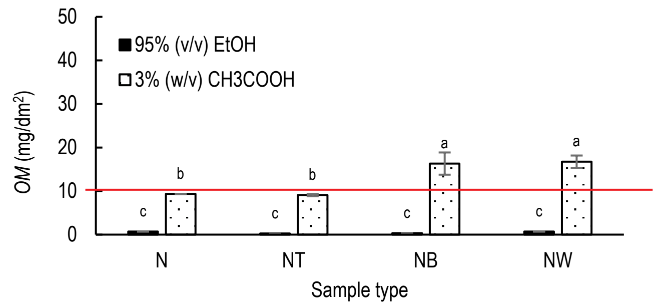

Figure 1 Overall migration values (*OM*) from unprinted paper sample types. [a-c] Different superscripts indicate significant differences between samples at $p < 0.05$.

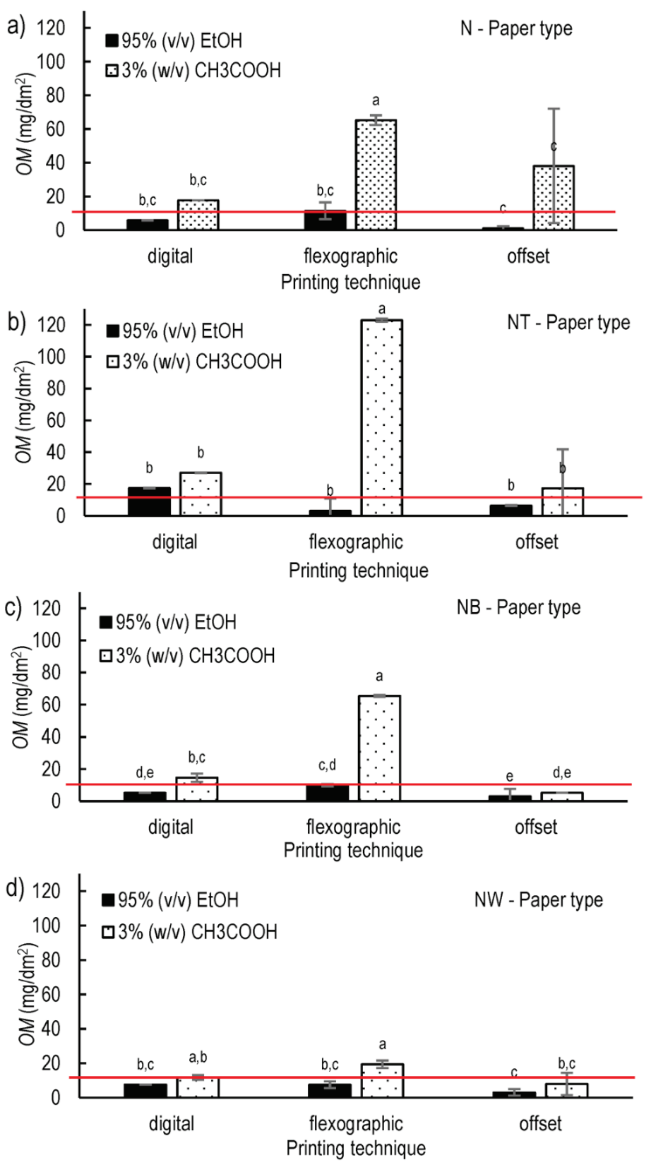

Figure 2 Overall migration values from prints on: a) N - paper type, b) NT - paper type, c) NB - paper type and d) NW - paper type. [a-e] Different superscripts indicate significant differences between samples at $p < 0.05$.

In general, all samples had higher migrations in acetic acid than in ethanol. This can be explained by the composition of paper. Indeed, paper can be defined as a sheet-like material consisting mainly of cellulose fibres and other organic and inorganic components. In terms of quantities used, fillers are the second most important constituent of paper after the fibres themselves [40]. Fillers are water-insoluble substances in the form of particles with a size of about 0.1 μm to 10 μm, which are added to the pulp before paper formation. Calcium carbonate ($CaCO_3$) is the most commonly used in the paper industry. In addition to the numerous advantages of this type of filler, which fills voids in the cellulose fiber network, it is also characterized by its solubility when the pH of medium drops below 6.5 (acidic medium). Therefore, the papers showed significant migration to acetic acid, as this food simulant lowers the pH value of the paper, affecting the solubility of the filler between the fibers and making it more permeable. For the unprinted samples, the overall migration values of N and NT were below the *OM* limit, while the overall migration values for NB and NW papers in acetic acid were slightly above the limit (Fig. 1). When comparing printing techniques, it appears that the lowest *OM* values were measured for offset printing, probably due to the use of low-migration inks, followed by digital prints and papers printed with flexographic inks with the highest *OM* values, especially for the acetic acid. However, no trends could be derived from the results and the samples studied. Recently, it was found in [23] that the ink penetrates less in paper samples containing non-wood fibers than in papers made exclusively from recycled wood fibres, which shows taht the surface ink from paper migrates more easily.

Table 4 Undertone of printed black ink with variuos printing techniques (digital, flexographic and offset) on laboratory-made papers

Paper type	Undertone of prints		
	Digital prints	Flexographic prints	Offset prints
N	$8.37 \pm 0.92^{c\,B}$	$19.89 \pm 2.64^{a\,A}$	$5.13 \pm 0.44^{c\,C}$
NT	$18.68 \pm 2.44^{a\,A}$	$19.45 \pm 2.26^{a\,A}$	$6.37 \pm 0.54^{a,b\,B}$
NB	$13.01 \pm 1.26^{b\,B}$	$19.34 \pm 3.15^{a\,A}$	$6.61 \pm 0.68^{a\,C}$
NW	$11.77 \pm 0.59^{b\,B}$	$16.22 \pm 1.73^{b\,A}$	$5.93 \pm 0.32^{b\,C}$

Different superscripts within the column ($^{a-c}$) and in the row ($^{A-C}$) indicate significant differences between samples at $p < 0.05$.

Tab. 4 shows that the undertone values are extremely low in prints produced by the offset printing technique, while the highest values were obtained in flexographic prints, which means that the ink was retained on the printed surface in offset prints. From the different ink properties, it can be concluded that the ink that did not penetrate deeply into the printing substrate had a positive effect on migration, i.e. lower migration in 3% aqueous acetic acid and 95% ethanol.

4 CONCLUSION

This study defined that the addition of non-wood fibers derived from cereal straw to recycled fiber pulp results in paper substrates that have approximately the same water vapor permeability as 100% recycled paper. The study also confirms that substrates with non-wood fibers can be used for packaging, which is preferably printed by offset printing due to the extremely low *WVTR* value. It is also concluded that in addition to the different ink properties, the ink printed by offset printing remains on the surface of the paper, resulting in lower overall migration values. Water vapour permeability values for digital prints were similar to flexographic one, while the overall migration values were higher than that measured in offset prints and lower than flexographic prints. The main limitation of this study was not uniformity of laboratory-scale produced paper as well as sample sizing. Further work will be focused on scaling-up to commercial production line and, if possible, case-study on real products.

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