

A study on the food contact suitability of recycled paper and board

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Abstract

The suitability of recycled paper packaging materials for direct food contact applications is a major area of investigation. The evaluation of food contact suitability was conducted on two commercially produced recycled papers. The white top testliner and the fluting paper, commonly used as integral parts of corrugated container, were tested. Selected papers were also submitted to additional chemical cleaning, which was obtained by means of laboratory deinking flotation. The aim of this experiment was to evaluate a possible decrease in the amount of chemical contaminants in the deinked pulp after laboratory deinking flotation had been conducted. Two original paper samples as well as their corresponding deinked pulp handsheets were checked for the presence of potentially harmful substances that are usually found as residues of recycling procedures. Food contact analyses comprised determination of heavy metals (Cd, Pb, Hg), primary aromatic amines, diisopropyl naphthalenes (DIPN), phthalates and polychlorinated biphenyls (PCB) from aqueous or organic solvent extracts of paper samples. It was found that all amounts of measured contaminants were below the maximum limit of concentration proposed for these compounds by the European legislation. Furthermore, deinking flotation applied on the white testliner and fluting samples had a positive effect on the reduction of chemical contaminants from the deinked pulp.

Keywords: Food packaging, health safety, recycled fibres, chemical deinking flotation, reduction of contaminants.

1. Introduction

Recycled fibres have become an indispensable raw material for the paper manufacturing industry because of the favourable price of recycled fibres in comparison to the corresponding grades of virgin pulp and due to the promotion of recovered paper recycling by many European countries [1]. In addition,

a growing tendency of using recycled paper fibres is also present in the production of food packaging materials. Paper and board, partly or fully produced from recycled fibres, are being used both as primary and secondary packaging for wide range of foods in many European countries. However, in the last couple of years recycled fibres have been proved to be a source of contamination of various foods [2, 3]. Potentially harmful substances present in the recycled paper comprise the residues of printing inks, varnishes, adhesives and other substances, which are applied to the material in printing and converting processes undertaken in the previous use of the paper. Once present in the packaging material, they could migrate into food content under certain conditions, posing health concerns for the consumers [4]. In the scientific paper [5] published in 2002, Binderup et al. cited a list of chemicals that can be found in recycled paper. The list contains phthalates, solvents, azo colorants, diisopropyl naphthalenes, primary aromatic amines, polycyclic aromatic hydrocarbons, benzophenone and others. Considering that recycled fibres may be contaminated by many potentially harmful compounds, recycled grades must be used with a special caution in the production of food packaging.

In order to be classified as suitable for food contact, packaging paper has to meet many specific requirements proposed by the European legislation [6, 7]. However, the EU still has no harmonized legislation regarding food contact paper and board applications and the use of recycled paper fibres in the contact with foods. In addition, there is a lack of specific directives for paper and board materials intended to come into contact with food. The main rule for paper and board food contact applications comes from the EU Framework Regulation (EC) No 1935/2004 [6] and the Regulation on Good Manufacturing Practice (EC) No 2023/2006 [7]. Framework Regulation covers all groups of materials intended to come into contact with food and sets some general requirements that

all food contact materials must comply with. It states that substances that might endanger human health must not be transferred from packaging into the packed food in quantities which could endanger human health or bring about an unacceptable change in the composition of the food or deterioration in its organoleptic characteristics (essentially its taste and smell). In the absence of a specific directive, some European countries have developed their own national provisions specific to paper and board (e.g. Germany, France, Italy, and the Netherlands). Existing regulations define the chemicals that are allowed in the manufacture of paper and board and set limits for various contaminants (heavy metals, pentachlorophenol, polychlorinated biphenyls, etc.) in the finished products [8]. In addition, the Council of Europe (COE) has published non-legal binding Resolution AP (2002)1 on paper and board materials and articles intended to come into contact with foodstuffs [9], which can act as a reference for countries that have not yet established national regulations of their own.

A specific concern of the use of recycled fibres is also reflected in the Council of Europe Resolution AP (2002)1 on paper and board for food contact.

This paper deals with safety issues of materials based on recycled fibres and their suitability for direct food contact. The evaluation of food contact suitability was conducted on two brown paper grades made of predominantly recycled fibres: on the white top testliner and fluting paper, both obtained from industry. The majority of recovered papers used in the production of brown packaging grades are recycled with exclusively mechanical cleaning of the pulp i.e. without deinking.

Deinking flotation is applied only in individual cases and mainly in the production of the white top layer of board [10]. Since the majority of fibres in the tested materials were produced by recycling processes where only the mechanical cleaning was used (with slight exception of the white top ply of testliner sample for which the deinked pulp was used), these samples were subjected to additional chemical cleaning by means of laboratory deinking flotation. The aim of this experiment was to evaluate the possible decrease in the amount of chemical contaminants in the composition of the pulp after the chemical deinking flotation had been conducted.

2. Experimental

Two packaging papers, the white top testliner and fluting paper (*Table 1 and 2*) were chosen to be tested for direct food contacts suitability. Those two papers are commonly used as integral parts of the corrugated container (testliner being the flat liner part, and fluting being the corrugated medium of the container). Brown packaging grades, such as testliners and fluting papers, are usually made with high content of recycled fibre furnish that originates mostly from old corrugated containers (OCCs) and mixed paper and board grades.

The composition and the properties of selected papers are shown in *Tables 1 and 2*. The origin of the recycled fibre furnish is presented by giving the numerical code of the specific paper grade in accordance with the existing European standard EN 643, European List of Standard Grades of Recovered Paper and Board [11].

Table 1: White top testliner characteristics

| White top testliner | |
|---------------------|--|
| Basis weight | 130 gm ⁻² |
| Ash content | 15.1 % |
| Bulk | 1.44 cm ³ g ⁻¹ |
| Composition | <p><i>Top ply:</i> deinked pulp originating from printed white woodfree paper, woodfree books without hard covers, cuttings of lightly printed bleached sulphate board (EN 643: 2.07, 3.04, 3.09).</p> <p><i>Base ply:</i> recycled fibres originating from used paper and board packaging, containing a minimum of 70% of corrugated board, the rest being solid board and wrapping papers as well as the mixed papers and boards containing maximum of 40% of newspapers and magazines (EN 643: 1.02, 1.04).</p> |

Table 2: Fluting paper characteristics

| Fluting | |
|---------------------|--|
| Basis weight | 170 gm ⁻² |
| Ash content | 15.0 % |
| Bulk | 1.56 cm ³ g ⁻¹ |
| Composition | Semi-chemical pulp (60%), the rest being the mixed papers and boards containing maximum of 40% of newspapers and magazines (EN 643: 1.02). |

The white top testliner and fluting paper were unprinted, yet they contained a high amount of ink residues, fillers and other impurities since they had been produced from recycled fibres that did not undergo chemical flotation during recycling (with slight exception of the white ply of the testliner sample for which the deinked pulp was used). For that reason they were subjected to the additional

chemical cleaning which was obtained by performing the laboratory deinking flotation.

2.1. Deinking flotation

For the laboratory deinking flotation procedure (Figure 1) two recovered paper samples were prepared: the white top testliner and fluting sample. The samples were recycled separately but followed the same routine: 75 grams of absolutely dry paper was cut in 2 x 2 cm strips and put in the pulper. By adding two litres of deionised water at a temperature of 60 °C, the consistency of pulp was set to 3.75%. Afterwards, the deinking chemicals were added: 5% NaOH (22.90 ml), 1.5% H₂O₂ (20 ml), 6% Na₂SiO₃ (17.30 ml), 0.5% DTPA (0.38 g) and 3% surfactant (2.25 g). The industrial deinking process was simulated with these amounts of added chemicals. The obtained pH was between 10.8-11.

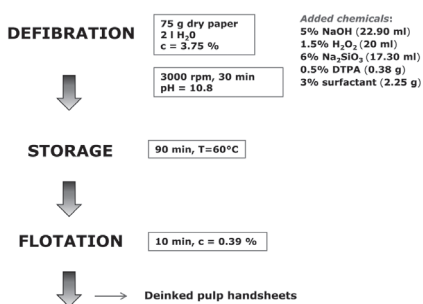


Figure 1: The deinking flotation procedure scheme

The pulp was disintegrated in the pulper at 3000 rpm for 30 minutes and was subsequently stored for 90 minutes at a temperature of 60 °C. The suspension was afterwards diluted with tap water up to the volume of 19 litres and transferred to the flotation cell, where it was floated for 10 minutes. During the flotation process, the flotation froth was collected manually and removed from the cell. After the flotation, the deinked pulp handsheets were formed according to the TAPPI 205 standard method [12].

2.2. Food contact analyses

Food contact analyses were conducted on the original paper samples (the white top testliner and fluting paper), as well as on the deinked pulp handsheets

formed after conducted deinking flotation. Food contact analyses comprised the determination of heavy metal contents (cadmium, lead and mercury), primary aromatic amines, diisopropylnaphthalene (DIPN), phthalates and polychlorinated biphenyls (PCB) from aqueous or organic solvent extracts of paper.

In order to determine heavy metals, cold-water extracts were prepared from all paper samples in accordance with the EN 645:1993 [13]. The determination of metal ions (cadmium, lead and mercury) in the cold-water extracts was carried out in accordance with the EN 12497 and EN 12498 [14, 15]. Detection of metals was conducted by atomic absorption spectroscopy (AAS).

For determination of primary aromatic amines, the paper samples were extracted in dichloromethane. The concentrations of primary aromatic amines (expressed as aniline) in solvent extracts were determined by liquid chromatography–mass spectrometry (LC-MS).

The determination of diisopropylnaphthalene content (DIPN) was carried out in accordance with the standard EN 14719:2005 [16]. The content of total diisopropylnaphthalene (DIPN) was determined by solvent (dichloromethane) extraction of the paper sample and analysed by gas chromatography with mass selective detection (GC-MS), using diethylnaphthalene as an internal standard.

For the determination of phthalates, the paper samples were extracted in dichloromethane. The total phthalate content in solvent extract was determined by gas chromatography with mass selective detection (GC-MS).

The determination of polychlorinated biphenyls (PCB) was carried out in accordance with the ISO 15318:1999 standard [17]. The paper samples were extracted with boiling ethanolic sodium hydroxide solution which was prepared by dissolving 30 g of NaOH (2% w/v) in a 19:1 v/v ethanol/water (1500 ml) solution. An aliquot of the extract was mixed with water and afterwards subjected to liquid-solid partitioning on a disposable C18 solid phase extraction cartridge followed by elution with hexane. The present PCBs were quantified by means of gas chromatography with electron-capture detection (GC-EDC). The results of analyses conducted were compared to the quantitative restrictions laid down in the Ger-

man BfR Recommendations (chapter XXXVI) [18] and/or Croatian Ordinance on sanitary safety of materials and articles intended to come into direct contact with foodstuffs [19] that they had to comply with. BfR Recommendation XXXVI is the most widely recognized existing standard within the EU. However, in case when the German or Croatian regulations did not specify clear limits for tested compounds, the results obtained by chemical analyses were compared to proposed restrictions laid down in the available Nordic guideline: the Nordic report on paper and board food contact materials [20], developed by the Nordic Council of Ministers. The basis for the Nordic report is the Council of Europe Resolution AP (2002)1.

3. Results and discussion

The results of metal ions determination (Cd, Pb and Hg) in the cold-water extracts are presented in Table 3.

Table 3: Amounts of metal ions determined in cold-water extracts (DP-deinked pulp)

| Amount in water extract | Hg | Cd | Pb |
|------------------------------|-------------|------------|----------|
| | mg/kg paper | | |
| Limit | 0.3 | 0.5 | 3 |
| White testliner | <0.0001 | <0.0002 | <0.002 |
| White testliner DP handsheet | <0.0001 | <0.0002 | <0.002 |
| Fluting | <0.0001 | <0.0002 | <0.002 |
| Fluting DP handsheet | <0.0001 | <0.0002 | <0.002 |

According to the German BfR Recommendations and the Croatian Ordinance on sanitary safety of materials and articles intended to come into direct contact with foodstuffs, the transfer of metal ions into foodstuffs must not exceed 0.5 mg per kg of paper (Cd); 3 mg per kg of paper (Pb); 0.3 mg per kg of paper (Hg). Testing is not necessary for paper and board intended to come into contact with dry, non-fatty foodstuffs.

The amounts of polychlorinated biphenyls (PCB) and primary aromatic amines determined in solvent extracts are presented in Table 4. According to the Croatian Ordinance on sanitary safety of materials and articles intended to come into direct contact with foodstuffs, finished products must not contain more than 2 mg of PCB per kg paper. On the other hand, German BfR Recommendations do not impose the testing of PCBs in finished paper, so no limits for PCBs are set within the existing German regulation.

Furthermore, according to the German BfR Recommendations, primary aromatic amines must not be detectable in the extract of the finished product. However, the detection limit still has to be defined. In addition, the Croatian Ordinance requires that the content of these substances must be below the limit of detection which is set to 0.1 mg of primary aromatic amines per kg of paper. Testing is not required for paper and board intended to come into contact with dry, non-fatty foodstuffs.

Table 4: Amounts of polychlorinated biphenyls (PCB) and primary aromatic amines in solvent extracts (DP-deinked pulp)

| Amount in solvent extract | PCB | Primary aromatic amines |
|------------------------------|-------------|-------------------------|
| | mg/kg paper | |
| Limit | 2 | 0.1 |
| White testliner | <0.02 | <0.05 |
| White testliner DP handsheet | <0.02 | <0.05 |
| Fluting | <0.02 | <0.05 |
| Fluting DP handsheet | <0.02 | <0.05 |

The results of conducted analyses indicate that all measured contaminants were found in extremely low concentrations. As shown in Table 3, all detected amounts of metal ions were actually below the quantification limit of the instrument, which leads to the conclusion that there is no danger whatsoever of a migration of these compounds from the paper samples into the food. In addition, the results of the detected amounts of polychlorinated biphenyls (PCB) and primary aromatic amines in solvent extracts (Table 4) show that all detected concentrations of analysed compounds were also below the quantification limit of the instrument. With regard to these three food contact suitability parameters, all tested papers are thus considered suitable to be used in direct contact with foods. The results of the total diisopropyl-naphthalene (DIPN) content determination are presented in Table 5.

Table 5: Diisopropyl-naphthalene (DIPN) content in solvent extracts of papers (DP-deinked pulp)

| Sample | DIPN (mg/kg) | Grammage (g/m ²) | DIPN (mg/dm ²) |
|------------------------------|-------------------------------------|------------------------------|----------------------------|
| | Limit 1.33 mg/dm² | | |
| White testliner | 14.00 | 130 | 0.0182 |
| White testliner DP handsheet | 13.70 | 100 | 0.0137 |
| Fluting | 15.00 | 170 | 0.0255 |
| Fluting DP handsheet | 9.20 | 100 | 0.0092 |

centrations of DIPNs were found in the samples of fluting paper and white testliner (15 and 14 mg/kg respectively). For comparison, in the survey that was carried out by the UK Ministry of Agriculture, Fisheries, and Food in 1998 [21], DIPNs were detected in 51 sample of recycled board obtained from paper mills at up to 33 mg/kg and in most samples of retail food packaging at up to 44 mg/kg.

The results presented in *Table 5* also indicate that the conducted deinking flotation reduced the DIPN content by 38.7% in the fluting deinked pulp handsheet, while in the case of the white testliner sample, deinking flotation had a negligible influence on the reduction of DIPNs.

German BfR Recommendations, as well as the Croatian Ordinance on sanitary safety of materials and articles intended to come into direct contact with foodstuffs, require that the content of DIPN in finished paper should be as low as technically possible. It is obvious that neither the Croatian nor the German regulations specify a clear limit of permitted levels of DIPN in finished paper material. The results obtained by chemical analyses were therefore compared to the maximum limit set in the Nordic report on paper and board food contact materials. The Nordic guideline proposes that the level of DIPN should not exceed the limit of 1.33 mg of DIPNs per dm² of paper. Since in this case the maximum limit is expressed as weight/area unit, whereas the results obtained by an analytical measurement provided the weight/weight results, a conversion to weight/area units had to be done. The conversion was done by taking into account the actual grammage of analysed paper (*Equation 1*).

$$Qa = (Qm \times G)/10^5$$

where:

Qa is concentration of substance in paper expressed as mg/dm²,

Qm is concentration of substance in paper expressed as mg/kg,

G is grammage of paper as expressed as g/m².

The results of detected DIPNs in analysed paper samples expressed as mg/dm² of material (*Table 5*, column 4) indicate that all detected concentrations of DIPNs are much lower than the maximum amount allowed (<1.33 mg/dm²). The latter means that all of

these paper samples, as far as this food contact suitability parameter is concerned, can be considered suitable to be used in direct contact with food.

The results of the total phthalate content determination are presented in *Table 6*. Presented results indicate that the highest concentrations of phthalates are found in samples of fluting paper and white testliner (15 and 5.4 mg/kg respectively). The conducted deinking flotation reduced the phthalate content by 70.1% in the fluting paper deinked pulp handsheets and by 21.3% in the white testliner deinked pulp handsheets.

Table 6: Total phthalate content in solvent extracts of papers (DP-deinked pulp)

| Sample | Total phthalate content (mg/kg) | Grammage (g/m ²) | Total phthalate content (mg/dm ²) |
|-------------------------------------|---------------------------------|------------------------------|---|
| <i>Limit 0.25 mg/dm²</i> | | | |
| White testliner | 5.40 | 130 | 0.0070 |
| White testliner DP handsheet | 4.25 | 100 | 0.0043 |
| Fluting | 15.00 | 170 | 0.0255 |
| Fluting DP handsheet | 4.49 | 100 | 0.0045 |

The maximum limit of the total phthalate content in paper material, expressed as a group restriction, was found in the Nordic guideline, whereas the German and Croatian regulations restrict the maximum limit only for individual phthalates.

The Nordic reference was therefore used in the interpretation of the obtained results. Nordic guideline imposes that the level of total phthalate content should not exceed the limit of 0.25 mg of phthalates per dm² of paper.

The results of total phthalate content in analysed papers expressed as mg/dm² of material (*Table 6*, column 4) indicate that all detected levels of phthalates in the analysed samples are much lower than the maximum limit (<0.25 mg/dm²). Thus, according to the Nordic guideline established limit, all detected levels of phthalates in these materials do not present a risk to human health. As far as this food contact suitability parameter is concerned, all analysed papers are suitable for direct contact with food.

4. Conclusion

Research on the direct food contact suitability that was conducted on two commercially available packaging papers (the white top testliner and fluting

paper) and on their corresponding deinked pulp handsheets showed that the most common contaminants present in the packaging paper grades are diisopropylnaphthalenes (DIPNs) and phthalates. In these materials, phthalates and DIPNs were detected at concentrations at up to 15 mg/kg. On the other hand, other evaluated contaminants such as heavy metals (Cd, Pb and Hg), primary aromatic amines, polychlorinated biphenyls (PCB) were found in extremely low concentrations.

The conducted deinking flotation on the white testliner and fluting sample had a positive impact on the reduction of DIPNs and phthalates from the deinked pulp. The deinking flotation reduced the DIPN content by 38.7% in the fluting paper deinked pulp, while in the case of the white testliner it showed no significant effect on the reduction of DIPNs.

In addition, the deinking flotation reduced the phthalate content in deinked pulp handsheets by 70.1% in the case of fluting paper sample and by 21.3% in the case of white testliner sample.

While comparing the detected amounts of DIPNs and phthalates in the analyzed paper samples to the quantitative restrictions laid down in the German or Croatian regulations, it was impossible to estimate whether those levels of chemicals impose a risk to human health due to the incomplete and imprecise regulations.

However, when compared to the maximum limits proposed within the Nordic report on paper and board food contact materials, all the concentrations found were much below the Nordic guideline proposed limits. It can therefore be concluded that all tested papers regarding the analyses done within this research are found suitable to be used in direct contact with foods.

Nevertheless, additional analyses, such as the migration of mineral oils from recycled fibre materials, must be conducted to further confirm their suitability for direct food contact.

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Typography and graphic design in newspaper Slovenec

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Abstract

The aim of the research was to analyse the typographic changes in the newspaper Slovenec from its inception in 1873 – first it was published three times per week and after ten years, it became a daily newspaper – until its decline in 1945, as well as during its revival from 1991 until 1996.

All the issues of the newspaper were analysed; however, the focus was put on those which were actually changed.

By analysing the typeface style, it was established that for the content, the most widely used typestyle in the “old” newspaper (1873–1945) was modern, which was not always the case with titles and subtitles where decorative or lineal typestyles were used. On the other hand, the most widely used typestyle in the “new” newspaper (1991–1996) was transitional and was used for titles and subtitles, sometimes replaced by slab serif and lineal typestyles. The design of the “old” newspaper somehow followed the common European newspaper design, while in the “new”, modern newspaper, the design could not be detected.

Therefore, the cultural heritage of the “old” newspaper was lost.

Keywords: cultural heritage, graphic design, newspaper, Slovenec, typography

1. Introduction

At the beginning of the 19th century, the printing art expansion was slowing down, since it already became established in the political, social and cultural life. At the end of the 19th century, Ljubljana got four new printing houses. One of the most important printers was Jožef Blaznik [1–4].

He was the supporter of young Slovenian writers and researchers, which is why the works published in his printing house were mostly works of literature and natural science written in the Slovenian language [5]. His printing house was one of the most up-to-date printing houses and the first one where Slovenec, a catholic newspaper in the Slovenian language, was printed between 1873 and 1883. During that time, it was published three times per week. Later, Slovenec became a daily newspaper and was printed in Catholic printing house, which was after the First World War in 1919 renamed into Yugoslav printing house. During and after the Second World War, the printing house worked under the name Printing house of national justice. Due to the catholic content of the newspaper Slovenec, its publishing stopped after the Second World War until its revival in 1991, when a new printing house was established for its printing and was closed down when the newspaper stopped being published in 1996 [1–6].