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PACKAGING MADE FROM RECYCLED PAPER AND BOARD: EVALUATION OF THE SUITABILITY FOR DIRECT FOOD CONTACT

Sonja JAMNICKI[1], Branka LOZO[1], Vera RUTAR[2]
[1]University of Zagreb, Faculty of Graphic Arts, Getaldiceva 2, 10000 Zagreb, Croatia
[2]Pulp and Paper Institute / Bogisiceva 8, 1000 Ljubljana, Slovenia

Abstract:

This paper deals with safety issues of the recycled-fibre based materials and their suitability for direct food contact. The evaluation of food contact suitability was conducted on two recycled paper samples commonly used as integral parts of corrugated container, such as pizza packaging containerboard. The white top testliner sample and the fluting sample were obtained from industry. Since the majority of fibres in the examined materials were produced by recycling processes in which only the mechanical cleaning methods were 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 pulp after chemical deinking flotation had been conducted. Food contact analyses were done on the original paper samples and the deinked pulp handsheets made after deinking flotation. Food contact analyses comprised determination of heavy metals (Cd, Pb, Hg), primary aromatic amines, diisopropylnaphthalenes (DIPN), phthalates and polychlorinated biphenyls (PCB) from aqueous or organic solvent extracts of paper samples. The fastness of the fluorescent whitening agents was determined as well. It was found that all of the investigated contaminants were below the maximum limit of concentration proposed for these compounds by the European legislation. Furthermore, applied deinking flotation on the white testliner and fluting samples had a positive impact on the reduction of chemical contaminants from the fibre suspension.

Keywords:

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

1 INTRODUCTION

Having in mind the environmental protection, trend of using recycled paper fibres in production of food packaging has been on rise. However, the suitability of using recycled fibres in food contact applications is still being largely debated among the scientific community since it is difficult to exclude exposure to traces of ink chemicals, varnishes, adhesives, and other potentially harmful substances in recycled fibres. New waste directive adopted by the EU (Directive 2008/98/EC) is for ecological reasons prioritizing paper recycling over incineration, however, the recycling of printed paper and board can lead to the production of food contact materials that may be harmful to consumers' health. In the scientific paper published in 2002, Binderup *et al.* cited a list of chemicals that can be found in recovered paper. The list contains phthalates, solvents, azocolorants, diisopropyl naphthalenes, primary aromatic amines, polycyclic aromatic hydrocarbons, benzophenone and others [1]. Moreover, in the recent past we have witnessed several food scandals caused by migration of mineral oils from recycled paper and board packages into foods [2, 3]. Considering that recycled fibers have a less certain history than virgin pulp, recycled grades must be used with special caution in the production of food packaging.

In order to be classified as suitable for food contact, packaging paper has to meet specific requirements proposed by the European legislation. However, in the EU there is still no harmonized legislation on food contact paper and board applications, and on the use of recycled paper fibers in the contact with foods. In addition, there is also a lack of the specific directive for paper and board materials intended to come into contact with foods. The main rule for paper and board food contact applications comes from the EU Framework Regulation (EC) No 1935/2004 [4] and the Regulation on Good Manufacturing Practice (EC) No 2023/2006 [5]. Framework Regulation covers all groups of materials intended to come into contact with foods 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 the specific directive, some European countries have developed their own national provisions specific to paper and board (e.g. Germany, France, Italy, and the Netherlands). 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 [6], which can act as a reference for countries that have not yet established national regulations of their own. However, the existing national laws, regulations and recommendations are not always well aligned. Moreover, the lack of the specific directive for paper and board food contact materials has created disadvantage in the market because paper and board materials appear to be "unregulated". In response to this situation, the European paper and board food packaging supply chain comprising CEPI[1], CITPA[2], CEFIC[3] and FPE[4], has developed an Industry guideline for the compliance of paper and board materials and articles for food contact [7]. The aim of this document is to provide a single text that can be used by all operators in the paper and board packaging supply chain in order to establish compliance with Framework Regulation 1935/2004. In addition, similar guideline is available – the Nordic report on paper and board food contact materials, developed by the Nordic Council of Ministers [8].

This paper deals with the safety issues of recycled-fibre based materials and their suitability for direct food contact. The evaluation of food contact suitability was conducted on two brown grade papers made with high content of recycled fibres: on the white top testliner and fluting paper, both obtained from industry. In general, the majority of recovered papers used in the production of brown packaging grades are recycled without using the chemical deinking flotation which is applied only in individual cases and mainly in the production of the white top layer of board [9]. Since the majority of fibres in the examined materials were produced by recycling processes where only the mechanical cleaning methods were 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 brown grade papers, the white top testliner and fluting paper, were chosen to be tested for the 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

[1] CEPI: Confederation of European Paper Industries
[2] CITPA: International Confederation of Paper and Board Converters in Europe
[3] CEFIC: European Chemical Industry Council
[4] FPE: Flexible Packaging Europe

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 characteristics of the selected papers are shown in Table 1 and 2. The origin of the recycled fibre furnish is presented in accordance with the existing European standard EN 643, European List of Standard Grades of Recovered Paper and Board by giving the numerical code of the specific grade [10].

Table 1: White top testliner characteristics

White top testliner	
Basis weight	130 gm^{-2}
Ash content	15.1 %
Bulk	1.44 cm^3g^{-1}
Composition	***Top ply:*** 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).
	Base ply: 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).

Table 2: Fluting paper characteristics

Fluting	
Basis weight	170 gm^{-2}
Ash content	15.0 %
Bulk	1.56 cm^3g^{-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 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 by means of 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 ºC, the consistency of pulp was set to 3.75%. Afterwards, the deinking chemicals were added: 22.9 ml of sodium hydroxide ($NaOH$), 20 ml of hydrogen peroxide (H_2O_2), 17.3 ml of sodium silicate (Na_2SiO_3), 0.38 g of DTPA and 2.25 g of surfactant. The industrial deinking process was simulated with these amounts of added chemicals. The obtained pH was between 10.8-11.

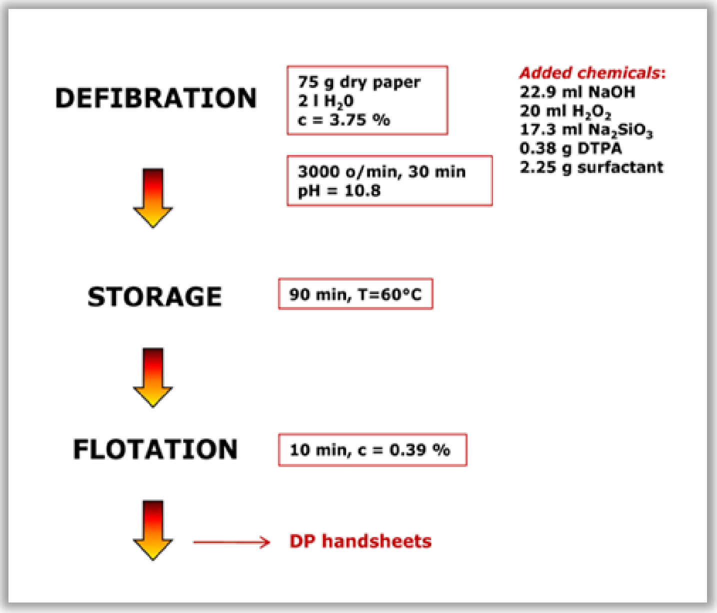

Figure 1: The deinking flotation procedure scheme

The pulp was disintegrated (beaten) in the pulper at 3000 rpm for 30 minutes and was subsequently stored for 90 minutes. Afterwards, the suspension was diluted with tap water up to the volume of 19 litres and was then transferred to the flotation cell and flotated for 10 minutes. During the flotation process, the flotation froth was collected manually and removed from the cell. After the flotation process had been completed, the deinked pulp handsheets were formed according to the TAPPI 205 standard method.

2.2 Food contact analyses

Food contact analyses were done on the original paper samples (the white top testliner and fluting paper) as well as on the deinked pulp handsheets made after conducted deinking flotation. Food contact analyses comprised determination of heavy metals (cadmium, lead and mercury), primary aromatic amines, diisopropylnaphthalene (DIPN), phthalates and polychlorinated biphenyls (PCB) from aqueous or organic solvent paper extracts. The fastness of the fluorescent whitening agents (FWA) was determined as well.

In order to determine heavy metals, cold-water extracts were prepared from all paper samples in accordance with the EN 645:1993 standard [11]. 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 standards [12, 13]. Detection of metals was conducted by means of the atomic absorption spectroscopy (AAS).

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

The determination of diisopropylnaphthalene content (DIPN) was carried out in accordance with the standard EN 14719:2005 [14]. The content of total diisopropylnaphthalene (DIPN) was determined by solvent 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 tested 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 [15]. The paper samples were extracted with boiling ethanolic potassium hydroxide 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 fastness of fluorescent whitened papers (bleed fastness) was determined on paper samples showing the highest fluorescence that had been previously detected by measuring CIE fluorescence according to the ISO 11475 [16]. Bleed fastness analysis is conducted according to EN 648 [17]. whereby a value of 4 or 5 on the evaluation scale must be reached. In this test procedure the sample is brought into contact with glass fibre papers previously saturated with a test fluid (food simulant) and placed under a load (1 kg) for a given time period (24 h). The staining of the glass fibre paper was evaluated by comparison with a series of fluorescent whitened comparison papers. The test fluids used were deionised water, diluted acetic acid, soda and olive oil.

The results of conducted analyses were compared to the quantitative restrictions laid down in the German 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 (Nordic report on paper and board food contact materials).

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 μg per gram of paper (Cd); 3 μg per gram of paper (Pb); 0.3 μg per gram of paper (Hg). Testing is not necessary for paper and board intended to come into contact with dry, non-fatty foodstuffs.

The results of the detected amounts of metal ions show that detected concentrations in all recycled paper extracts are well below the maximum limit allowed, therefore, there is no danger of an eventual migration of these compounds into the food.

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 polychlorinated biphenyls per kg of paper. However, German BfR Recommendations do not require the testing of PCB amounts in finished paper, therefore, 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 extract of the finished product. However, the detection limit still has to be defined. In addition, Croatian Ordinace imposes 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 the detected amounts of polychlorinated biphenyls (PCB) and primary aromatic amines in solvent extracts, presented in Table 4, show that all detected concentrations are well below the maximum limits proposed by the Croatian and German legislation. With regard to these two food contact suitability parameters, all tested papers are considered suitable to be used in direct contact with foods.

The results of the total diisopropylnaphthalene (DIPN) content determination are presented in Table 5.

Table 5. Diisopropylnaphthalene (DIPN) content in solvent extracts of papers (DP-deinked pulp)

Sample	**DIPN** (mg/kg paper)
White testliner	14
White testliner DP handsheet	13.70
Fluting	15
Fluting DP handsheet	9.20

The presented results show that the highest concentrations of DIPN are found in the samples of fluting paper and white testliner (15 and 14 mg/kg respectively). The conducted deinking flotation reduced the content of DIPN by 2.1% in the white testliner deinked pulp handsheets and by 38.7% in the fluting paper deinked pulp handsheets.

German BfR Recommendations, as well as Croatian Ordinance on sanitary safety of materials and articles intended to come into direct contact with foodstuffs, impose that the 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 for permitted levels of DIPN in finished paper material. Therefore, the results obtained by chemical analyses were compared to the allowed maximum limit set in the Nordic report on paper and board food contact materials. Nordic guideline prescribes that the level of DIPN should not exceed the limit of 1.33 mg of DIPNs per dm^2 of paper. Since these limits in the material are expressed as weight/area units, whereas the results obtained by an analytical measurment provided the weight/weight results, a conversion to weight/area units had to be done. The conversion was performed by taking into account the actual grammage of analysed paper (Equation 1).

$$\boldsymbol{Qa} = (\boldsymbol{Qm} \times \boldsymbol{G})/\mathbf{10^5} \qquad (1)$$

where:

Qa is concentration of substance in paper expressed as mg/dm^2,
Qm is concentration of substance in paper expressed as mg/kg,
G is grammage of paper as expressed as g/m^2.

The results of DIPN content in the analysed paper samples expressed as mg/dm^2 are presented in Table 6.

Table 6. Diisopropylnaphthalene (DIPN) content in analysed papers expressed as mg/dm^2 of material (DP-deinked pulp)

Sample	**Grammage g/m^2**	**DIPN (mg/dm^2)**
		Limit **1.33 mg/dm^2**
White testliner	130	0.0182
White testliner DP handsheet	100	0.0137
Fluting	170	0.0255
Fluting DP handsheet	100	0.0092

The results presented in Table 6 indicate that all detected concentrations of DIPN in analysed samples are much lower than the maximum limit allowed (<1.33 mg/dm^2). Since all detected levels of DIPN, (according to the Nordic guideline established limit) do not impose a risk to human health as far as this food contact suitability parameter is concerned, all analysed papers are suitable to be used in direct contact with food.

The results of the total phthalate content determination are presented in Table 7. 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 21.3% in the white testliner deinked pulp handsheets and by 70.1% in the fluting paper deinked pulp handsheets.

Table 7. Total phthalate content in solvent extracts of papers
(DP-deinked pulp)

Sample	Total phthalate content (mg/kg paper)
White testliner	5.4
White testliner DP handsheet	4.25
Fluting	15
Fluting DP handsheet	4.49

The Nordic guideline proposes the total phthalate content maxiumum limit (expressed as a group restriction), whereas the German and Croatian regulations restrict the maximum limit only for individual phthalates. Therefore, the Nordic reference was used in the interpretation of the obtained results. Nordic guideline imposes that the level total phthalate content should not exceed the limit of 0.25 mg of phthalates per dm^2 of paper. The results of total phtalate content in analysed paper samples expressed as mg/dm^2 of paper material are presented in Table 8.

Table 8.Total phthalate content in analysed papers expressed as $mg/dm^2$of material
(DP-deinked pulp)

Sample	Grammage g/m^2	Total phthalate content (mg/dm^2)
		Limit **0.25 mg/dm^2**
White testliner	130	0.0070
White testliner DP handsheet	100	0.0043
Fluting	170	0.0255
Fluting DP handsheet	100	0.0045

Presented results show that all detected levels of phthalates in the analysed samples are lower than the maximum limit allowed (<0.25 mg/dm^2). Therefore, according to the Nordic guideline established limit, all detected levels of phthalates in these materials do not impose a risk to human health. As far as this food contact suitability parameter is concerned, all the analysed papers are suitable to be used in direct contact with food.

The results of the fluorescent whitening agents (FWA) bleed fastness analysis are presented in Table 9. The bleed fastness analysis was conducted on paper samples that showed the highest fluorescence, which turned out to be the white testliner sample and the white testliner deinked pulp handsheet.

BfR Recommendations and Croatian Ordinance prescribe that fluorescent whitening agents (FWAs) must not migrate into food. Testing is not necessary for paper and board intended to come into contact with dry, non-fatty foodstuffs. In this test procedure the sample is brought into contact with glass fibre papers previously saturated with a test fluid and placed under a load of 1 kg for 24 hours. The staining of the glass fibre paper is evaluated by comparison with a series of fluorescent whitened comparison papers, whereby a value of 4 or 5 on the evaluation scale must be reached.

Table 9. The results of fluorescent whitening agents (FWA) bleed fastness analysis (DP-deinked pulp)

White testliner	SIDE A	SIDE B	**White testliner DP handsheet**	SIDE A	SIDE B
Test fluid	GRADE		*Test fluid*	GRADE	
H_2O	4	4	**H_2O**	4-5	4-5
CH_3COOH	4	4	**CH_3COOH**	4-5	4-5
Na_2CO_3	4	4	**Na_2CO_3**	4-5	4-5
Olive oil	5	5	**Olive oil**	5	5

The presented results indicate that no bleeding has been detected since in all cases the values of 4 or 5 on the evaluation scale were reached. The result of the analysis show that there is no danger of an eventual migration of FWAs into the food. Moreover, all analysed paper samples are found suitable to be used in direct contact with foods.

4 CONCLUSION

Reserach on the direct food contact suitability that was conducted on two comercialy available packaging papers (the white top testliner and fluting paper) and on their related deinked pulp handsheets showed that the most common contaminants present in the packaging paper grades are diisopropylnaphthalenes (DIPN) and phthalates. In these materials, phthalates and DIPNs were detected at concentrations 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), and fluorescent whitening agents (FWA), 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 2.1% in the white testliner deinked pulp and by 38.7% in the fluting paper deinked pulp. In addition, the deinking flotation reduced the phthalate content in deinked pulp handsheets by 21.3% in the case of white testliner sample and by 70.1% in the case of fluting paper sample.

While comparing the detected amounts of DIPNs and phthalates in tested 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 established limits. Therefore, it can be concluded, that all the tested papers regarding the analyses done in this research, are found suitable to be used in direct contact with foods. Nevertheless, additional analyses, such as migration of mineral oils from recycled fiber materials, must be conducted to further confirm their suitability for direct food contact.

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Corresponding author:

Sonja JAMNICKI
Faculty of Graphic Arts, University of Zagreb
Getaldiceva 2, 10000 Zagreb, Croatia
phone: +38512371080, fax: +3852371077, e-mail: sjamnick@grf.hr