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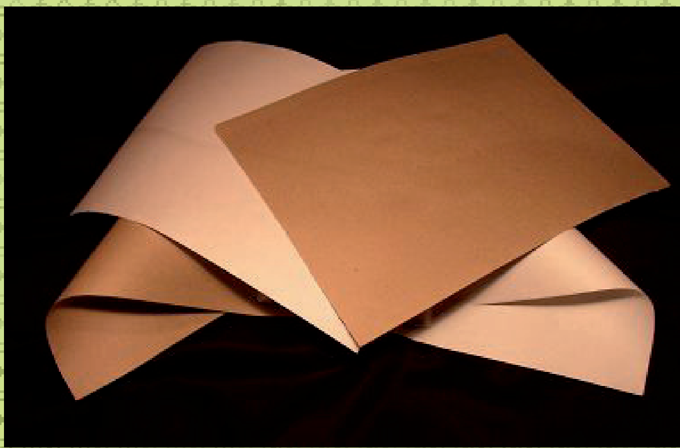

CONTENT

Papíripar

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CONTENT

Preface

It is a pleasure to present this year's third issue of the journal Papíripar in English to our readers.

Our editorial board is excited to observe continuous growth of the journal and its transition into a true multidisciplinary publication.

The journal is continuing to receive interesting and high-quality manuscripts from Hungary and from countries of the region. The articles in this issue cover a broad variety of topics from basic graphic communications, and technical science to industrial grasses and corrugated board.

At the end of this preface, I want to thank our readers and authors for their continuing interest in Papíripar, and each and every member of our editorial and scientific boards for their hard and dedication, which made it possible to bring another issue of Papíripar to our broad multidisciplinary international audience.

Laszlo Koltai Ph.D.
Editor in Cheaf

Budapest, 2012. December

New annual Hungarian plants (industrial grasses) as raw materials in the pulp and paper industry

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Abstract

Important task is, considering sustainable development, the better utilisation of the yearly renewable biomass. Large proportion of the Great Hungarian Plain can not be utilised for food production as earlier, consequently it is open for cultivation of plants for industrial utilisation. In one of the Hungarian Agricultural Centres in Szarvas appropriate researchers have developed, patented and produced in large scale a new yearly renewable source of biomass called "Szarvas – I Industrial Grass". Details related to agricultural production of the studied industrial grasses has also been discussed. The yearly production (metric tons/hectare) of biomass of industrial grass origin has been compared with those of conventional agricultural (e.g. straw, hemp, flax) and forest (e.g. Coniferous trees, Broad-leaved trees) plants. In cooperation with the Szarvas Research Group the Hungarian Paper Research Institute and with the a Research Group at the Department of Organic Chemistry and Technology of the Budapest University of Technology and Economics have started research activities for utilising this grasses in the paper industry.

Complex analytical studies in separating different components of industrial grass proved the possibility of utilisation of its components as follows: extracted by hexane 1.5 %, hot water extraction 1.0%, holocellulose 65.0%, lignin 29%, ash content 1.0 %.

Keywords: industrial grass, renewable raw material, pulp- and paper industry

1. Introduction

Three Hungarian groups of researchers cooperate in the project: the first one is Greenline Hungary Ltd. located in Szarvas the second one is the Paper Research Institute at the West-Hungarian University (Sopron) and the third one is the Department of Organic Chemistry and Technology of the Budapest University of Technology and Economics.

Dr. János Janowszky in cooperation with his son Zsolt Janowszky (Greenline Hungary Ltd.) has improved within several decades the industrial grass as one of yearly renewable cellulosic raw material.

The possible application of industrial grass as raw material of pulp and paper industry has been the subject of research for decades earlier at the direction of Dr. Éva Polyánszky and since more than five years by Associate Professor István Lele (Paper Research Institute at the West-Hungarian University).

Different investigations and analyses in correlation with the elaborated technologies and products have been performed at the Department of Organic Chemistry and Technology (Budapest University of Technology and Economics).

2. Results and discussions

2.1. The industrial grass plant

Dr. Janowszky and his group have developed since the middle of the eighties types of grasses of high solid content adaptable for energetic and industrial uses.

Such plants have to be selected which were not too sensitive to the quality of the soil of their cultivation. In success it might increase the chance of employment for inhabitants of such areas.

There is plenty of land (700-800 thousand hectares) in the Hungarian Great Plain not suitable for food production which however might be used for cultivation of plants of industrial purposes *(Figure 1)*.

The discussed industrial grass, a perennial shrubby plant, has been hybridized from grasses of saliferous area of lowland and of Middle-Asian arid areas. Up to 1.8-2.5 m deep in to the soil can be found the great mass of its root system. Its 180-220 cm high flat and hard, slate green stem is foliaged sparsely.
The numbers of nodes are on the stem 2-4. The leaves are stiff and their surface is uneven. Their 20-30 cm high yellow inflorescence is straight.
Flourishing ends at the beginning of July and the grains can be harvested generally at the beginning of August. The 0.8-1.2 cm long grains are of lance shaped. 6.0-6.5 g. is the mass of 10000 pieces of grains.

Figure 1: Industrial grass (number of nodus: 2-4)

2.2. Cultivation of industrial grass
The lifetime of industrial grass is long. It can be cultivated for up to 10-15 years at the same agricultural location. The cost of its plantation is by 90% less than that of the forests. It is yearly renewable for industrial application opposite to sources of wood origin which might be utilised once in 5-8 years.
Perennial grasses need only single preparation of the soil followed by only a few activities in plant protection. Their cultivation might prevent the soil erosion. It is an excellent biomeliorative plant (biological soil protection) *(Figure 2)*.
Up to 20-25 metric tons/hectare might be eroded under cultivation of one year plants whereas only less than 0.2-2 t/hectares under cultivation of perennial plants.
Industrial grass is resistant to vegetable-illnesses such as powdery mildew, brown-red rots.

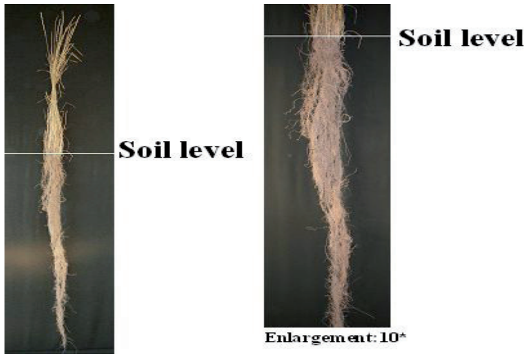

Figure 2: Industrial grass as biomeliorative plant

The yield of industrial grass reaches10-15 t/hectare in the first year after plantation, later, depending on the quality of the soil and on the yearly average participations, the yield might reach 10-25 t/hectare *(Figure 3)*.
No expensive and special machinery is needed for its growing and harvesting, because those of corn cultivation are easily adaptable.

Figure 3: Cultivation of industrial grass

The cultivation of industrial grass can be interrupted at any time not changing the quality of land use (plough land).

2.3 Significant characteristics of industrial grass
The Table 1 shows the comparison between significant characteristics of industrial grass with those of selected other plants. The ash content of industrial grass is 50 % of that of straw whereas it is the double of that of broad leaved trees.

Chemical composition	Coniferous trees	Broad-leaved trees	Cereal straw	Industrial grass
Extract content (n-hexane)	1.5	1.5	3.5	5.0
Hot water extract	1.0	3.0	16.0	15.0
Holocellulose	65.0	68.0	62.0	67.0
Lignin content	29.0	17.0	13.0	17.0
Ash content	1.0	2.0	8.0	3.5

Table 1: Chemical composition of traditional raw materials as well as that of industrial grass [%] [1]

The lignin content of industrial grass is approximately equal to that of broad leaved trees and about two third of that of coniferous trees.

The higher is the lignin content of a source the higher is their capacity of energy. This is due to the high carbon content (64%) of lignin.

The holocellulose content of the industrial grass is rather high (67%) comparable with those of the coniferous trees (65%) broad leaved trees (68%), consequently it is promising raw material for production of papers.

3. Fields of application

Industrial grass is able to substitute wood as industrial raw material in different fields of application.

The seed harvest of industrial grass is rather simple and economic. Local industrial raw material is being established by its cultivation enabling short and not expensive transportation.

Possibilities for making use of industrial grass:

– Source of energy (in solid, fluid and gaseous state),
– Raw material for pulp and paper industry,
– Fibrous raw material for different other industrial purposes,.
– Use in the chemical industry,
– Use in building industry,
– Animal feed,
– Biological soil conservation, soil reclamation.

3.1. Source of energy

From the second crop right after harvesting animal food might be produced (e. g. hay) or it might serve for the production of biogas [2].

The yearly productions of biomass/hectare of industrial grass compared with that of different plants are shown in *Table 2* [3], [4].

Raw material	Biomass suitable for industrial utilisation [t/year/hectare]
Coniferous	1.5 – 2.0
Broad-leaved trees	2.5 – 3.0
Grain straw	3.5 – 4.0
Flax	2.5 – 3.0
Hemp	6.0 – 8.0
Industrial grass	10.0 – 15.0

Table 2: Produced different biomass by one hectare of different plants [t/year]

The relative yearly production of biomass by industrial grass is twice up to ten times of that by other plants *(Figure 4).*

Figure 4: Energy content of industrial grass

3.2. Raw material for pulp- and paper industry

Bleached (elemental chlorine free (ECF)) and unbleached cellulose has been produced from industrial grass within alkaline conditions in the presence of anthraquinone under industrial and pilot scales *(Figure 5)* [5].

Packaging paper has been produced from the unbleached raw material (*Table 3*) whereas writing-printing one from the bleached resources (*Table 4, 5*). The properties of 44 % containing industrial grass based writing-printing paper have been evaluated under comparison with that of regular control paper (detailed data in *Table 4*).

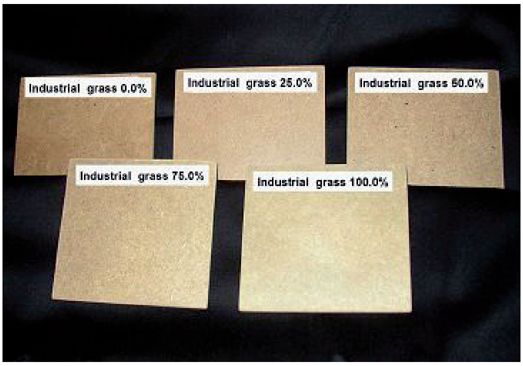

Figure 5: Industrial grass as raw material for pulp- and paper industry

Constituents	Control paper	Industrial cellulose containing paper
Fibres [%]		
Sulphate pine (Arhangelszki)	36	36
Sulphite pine (SÖDRA-BLUE)	20	20
Eucaliptus	44	0
Industrial grass pulp	0	44
Auxilary materials [%]		
Filler (PCC)	20	20
Size material (ASA)	0.3	0.3
Starch in masse	0.8	0.8

Table 4: Composition of writing-printing experimental and control samples

Properties	100 % OCC*	1/3 industrial grass + 2/3 OCC	2/3 industrial grass + 1/3 OCC	100 % industrial grass
Tensile index [Nm/g]				
md**	63.1	66.4	68.6	76.9
cd***	31.3	33.1	29.6	43.2
Elongation [%]				
md	1.48	1.61	1.65	1.83
cd	3.45	4.18	4.63	5.19
Burst index [$kPam^2/g$]	2.39	2.61	2.91	3.23
Tear index [mNm^2/g]				
md	7.09	7.21	7.37	7.69
cd	7.37	7.7	8.23	8.73
SCT [kN/m]				
md	1.75	2.66	3.06	3.27
cd	1.13	1.76	1.98	2.27
CMT_{30} [N]	108.0	127.4	142.2	163.1
RCT [N]				
md	60.7	87.4	94.6	110.3
cd	79.5	106.8	112.9	132.7

*OCC: Old Corrugated Containers
**md: machine direction
***cd: cross direction

Table 3: Properties of packaging paper produced from industrial grass cellulose, under industrial conditions

Properties	Control paper	Industrial cellulose containing paper
Grammage [g/m^2]	80.3	79.5
Thickness [mm]	0.136	0.132
Gravity [g/cm^3]	0.59	0.602
Tensile index [Nm/g]		
md*	64.6	66.1
cd**	21.4	26.0
Breaking length [m]		
md	6.581	6.740
cd	2.185	2.652
Elongation [%]		
md	1.0	1.1
cd	3.4	3.2
Tearing resistance [mN]		
md	544	523
cd	716	624
Tear index [mNm^2/g]		
md	6.3	5.7
cd	7.8	7.2
Burst pressure [kPa]	152.5	159.7
Burst index [$kPam^2/g$]	1.9	2.0
Brighteness [%]	82.4	85.2
Opacity [%]	93.1	91.3
Ash content (525°C) [%]	17.8	15.2

*md: machine direction
**cd: cross direction

Table 5: Properties of writing-printing papers

It could be concluded that no significant differences occurred between characteristics of indusrial grass containing and control papers.

Summary

Evaluating our experiments performed so far the industrial grass proved to be a hopeful yearly renewable raw material for energetic as well as for industrial application. It can be concluded from results of pilot and industrial scale production that the industrial grass cellulose was equivalent or even better in quality and value with papers produced from traditional resources.

Acknowledgement

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FT-IR and UV/VIS analysis of classic and recycled papers

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Abstract

The properties of classic and recycled papers with and without surface coatings were investigated by several methods. With the infrared attenuated total reflection spectroscopy, the molecular structure on sample surfaces was identified and analysed. This way the filler and coating pigment were detected and their amount on classic and on recycled papers. Ultraviolet/visible spectroscopy enables the reflectance and transmittance measurements. The most important result was detection of the optical brighteners and comparison of its amount in our paper samples. These results were compared with the calcium carbonate and china clay content in the bulk of examined papers, as calculated from the ash contents. Furthermore, differences between classic and recycled materials are discussed in more details.
Keywords: Recycled paper, FT-IR spectroscopy, UV/VIS spectroscopy, ash content.

1. Introduction

The infrared (IR) absorption spectroscopy is frequently used to identify the molecular structure of a sample qualitatively and quantitatively. The most useful for the analysis is the mid-IR spectra, i.e. the region from 4,000 to 400 cm^{-1}; in the upper wavenumber part (up to 1300 cm^{-1}) vibrations of functional groups in molecules can be detected, whereas the region with lower wavenumbers represent the so-called molecular fingerprint region of the analyte [1, 2]. The IR absorption is analysed with the help of absorbance spectra by the position, intensity (height), half-width and the shape of individual peaks.

The peak position is the basic characteristic of the corresponding vibration. It reveals an effective mass of vibrating group of atoms, the vibration geometry and coupling with the immediate surroundings, i.e. the vibrational species in the neighbourhood. A different position can indicate different effective mass of vibrating group and/or their different surroundings. The first effect produce different characteristic peaks, whereas differences the potential field in the surrounding of vibrating species can, in

general, shifts the corresponding peak which may indicate small differences in net material under investigation. The height of the peak presents strong, medium or weak vibrations, which depends on vibrating dipole moment, i.e. on the intrinsic nature of the matter. The peak width demonstrates several phenomena; crystalline matter, in general, reveal narrower peaks than their amorphous counterparts [3]. Basic characteristics of spectra show the chemical composition of the investigated sample; this is most frequently obtained by comparison to existing IR database. Small differences between spectra of samples with the same database match is then addressed to differences in molecular surrounding and/or degree of ordering in the structure.
The ultraviolet/visible (UV/VIS) spectroscopy enables the measuring of reflectance and transmittance of solid or liquid materials. The measurements are performed using collimated beam; when it is detected in collimated form, specular reflectance or directional transmittance is measured. The integrating sphere enables the collecting of the light emerging from the sample in arbitrary direction; thus, the diffuse reflectance or diffuse transmittance could be obtained. Such analysis enables the surface characterisation of glossy or rough solids or the photometric analyses of turbid, colloidal, transparent and translucent samples in solid or liquid forms.
The absorbance in this spectral region give information about electronic transitions within the investigated material, which is of great importance in many applications in the material science research. The UV/VIS spectroscopy is widely used in several applications such as characterisation of materials used in solar applications, printing inks, colour of samples, dyes, food composition etc [4].
The combination of IR and UV/VIS spectroscopy offers to give complete data about the material under investigation, i.e. their molecular structure, fingerprint, electronic transitions and surface properties. This is the reason why we applied this combination of analytical methods for our work.
The main goal of the research was to analyse the differences among classic and recycled papers from intrinsic material point of view as well as from their surface characteristics over wide spectral region, covering the entire optic part of electromagnetic spectrum.

2. Materials and methods

The analysis was performed on the upper (A) and bottom (B) side of the following paper samples:
– classic uncoated paper (CUP),
– classic matte coated paper (CCP),
– recycled uncoated paper (RUP) and
– recycled matte coated paper (RCP).

The samples were measured with a FT-IR spectrometer, using the diamond attenuated total reflectance (ATR) measuring cell which is suitable for surface measurement (measuring penetration depth up to about 2 μm). All spectra were examined with the resolution of 4 cm^{-1}, obtaining absorbance spectra of all samples, for side A and B.
– Hardware: Spectrum GX, FT-IR System, PerkinElmer,
– Software: Spectrum v 5.3.1,
– ATR measuring cell, Golden Gate (Harrick Scientific),
– Database: Nicodom IR/NIR Libraries for polymers, fibres, dyes, pigments.
The ash content was determined at two different temperatures [5, 6]. About 2.8 g of paper pieces, smaller than 1 cm^2 were weighted in a heat-resistance crucibles. After that the paper pieces were put into muffle furnace at 525 ± 25 °C and 900 ± 25 °C, cooled in a desiccators and weighted again. Ash content was calculated from both masses with consideration of moisture content. The procedure was carried out in duplicate and the average value was calculated [7]. We determined the ash content to compare results with FT-IR measurements.
Using the UV/VIS spectrometer with integrating sphere accessory, we measured the transmittance and reflectance values in the wavelength region 200–900 nm. The measurements of all samples were conducted in 10-nm steps, preparing 5 specimens for each sample, separately for side A and B, the average values of reflectance and transmittance of which was calculated with software.
– Hardware: Lambda 800 with PELA-1000 integrating sphere accessory, PerkinElmer,
– Software: UV WinLab 6.0.2.0723.

3. Results and discussion

3.1. FT-IR database

The applied database detected calcium carbonate

($CaCO_3$) for classic as well as recycled uncoated paper (CUP and RUP). It detected $CaCO_3$ only on side A of the paper, which most likely indicates that side B contains a smaller share of $CaCO_3$. We were informed by the producers that they use $CaCO_3$ as the only fil-ler in the production of classic and recycled uncoated paper. Due to the similarity of $CaCO_3$ with our uncoated papers, we compared their spectra (*Figure 1*). The peaks which are located in the same positions as they are in the spectrum of $CaCO_3$ (at 1420, 870 and 710 cm^{-1}, spectrum from the applied IR database) indicated the $CaCO_3$ presence in uncoated papers. These peaks are assigned as asymmetric stretching (at about 1425 cm^{-1}) and out-of-plane bending (at 870 cm^{-1}) vibrations of CO_3 group and to the in-plane bending vibration of CO_2 group (at 710 cm^{-1}) [8].

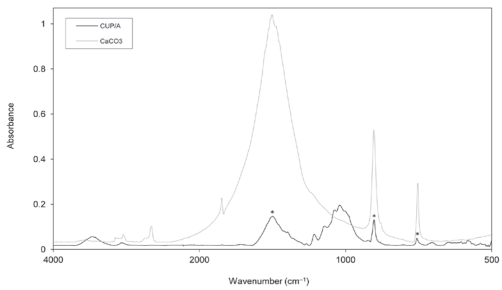

Figure 1: Spectra of CUP and $CaCO_3$ from the applied IR database. The peaks in CUP sample indicated by stars confirm the presence of $CaCO_3$.

For classic coated paper (CCP), we obtained information from the producer that they also use $CaCO_3$ as a filler in the paper production. Moreover, for the coating, they use apart from $CaCO_3$ also kaolin as a pigment. The database listed in the first place kaolin and in the fifth place $CaCO_3$ for side A and B of the paper. Recycled coated paper (RCP) contains $CaCO_3$ as a filler, while it contains apart from $CaCO_3$ for the coating also size and latex.
The database lists in the first place $CaCO_3$ and slightly lower kaolin, which was not listed by the producers; nevertheless, the coating most likely contains a smaller amount of kaolin on both paper sides. We compared the spectra of coated papers with those of $CaCO_3$ and kaolin (*Figure 2*).

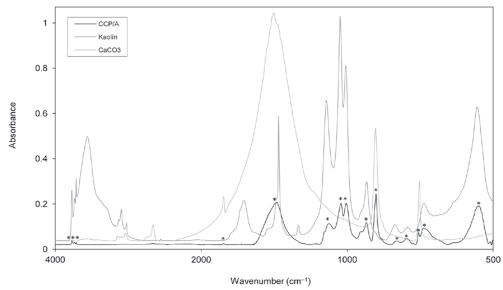

Figure 2: Spectra of CCP, kaolin and $CaCO_3$ from the applied IR database. The peaks in CCP sample indicated by stars confirm the presence of kaolin and $CaCO_3$.

Even here, the peaks are detected for the $CaCO_3$ content as at uncoated papers, while some of these peaks are less and some more distinctive. The peaks are located at 1800, 1400, 870 and 710 cm^{-1}. According to the literature [9], the $CaCO_3$ peaks which appear in the coated paper spectrum are to be located at around 2550, 1800, 1500–1300 and 890 cm^{-1}. The latter also proved correct in our case, not exposing the peak at 2550 cm^{-1} for being so small (in our case it appears at 2510 cm^{-1}). Slightly more distinct are the peaks located in the same places as kaolin. The first three very distinct peaks are at 3690, 3650 and 3620 cm^{-1}, followed by the peaks at the wavenumbers 1400, 1090, 1030, 1010, 910, 790, 750, 700 and 540 cm^{-1}.

3.2. FT-IR spectra

A substantially more precise analysis followed in the research, i.e. the analysis of our FT-IR spectra. We compared the recycled and non-recycled papers (side A and B), separately for uncoated and coated papers.
Using the program Spectrum, we defined the baseline for each spectrum individually and subtracted it from the spectrum. *Figure 3* shows the spectra of classic uncoated paper for side A and B, and of the recycled uncoated paper for side A and B.
Whereas *Figure 4* shows the spectra of classic and recycled coated paper, separately for each side. It can be instantly noticed that the peak positions separately for uncoated papers and coated papers are almost identical, while they differ in their peak height ratios.

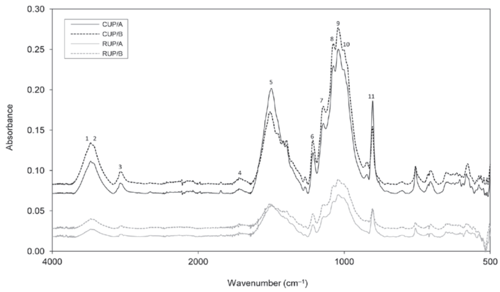

Figure 3: Spectra of classic (CUP) and recycled (RUP) uncoated papers for side A and B.

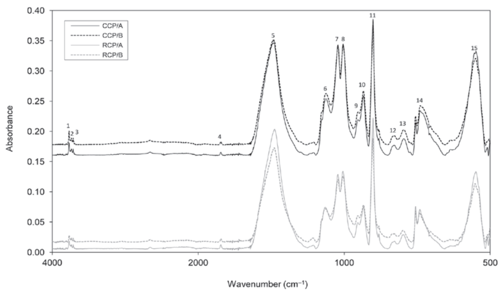

Figure 4: Spectra of classic (CCP) and recycled (RCP) coated papers for side A and B.

We defined the position v0 (cm^{-1}), height I0 and half-width hw (cm^{-1}) of individual peaks which are standing out the most. The data were inserted into Tables 1 and 2, separately for uncoated and coated papers. Moreover, each of these tables was further divided into two parts. While the first part contains the peaks within the region of functional groups (from 4,000 to 1,300 cm^{-1}), the second part contains the peaks within the fingerprint region (from 1,300 to 600 cm^{-1}).

With a more detailed overview of *Table 1*, it can be established that regarding the peak position, samples CUP and RUP differ slightly more only in the position of peak 3 (C–H vibrations [1, 10]). CUP has by 13 and 12 cm^{-1}, respectively, higher wavenumber value than RUP, which could be attributed only to different potential energy of atoms as a consequence of various surroundings.

Regarding the peak height, *Figure 3* and *Table 1* demonstrate that all peaks of CUP are higher than those of RUP, in some places more and in some less, which may indicate higher density of these molecular groups. However, the effect could also be a consequence of a different contact of the sample with the ATR crystal at the measurements due to unequal surface roughness, or different pressure applied when measuring.

Only at peak 4 (C=C, C=O, C=N vibrations [1, 10], water molecules vibrations [11]), it can be said that the heights are comparable. Comparing only side A and B, the differences are more substantial at peak 5 and 11 of both samples, i.e. CUP and RUP. Since these are the peaks typical of $CaCO_3$, it can be said that side A of samples CUP and RUP has a higher content of $CaCO_3$ than side B. The difference in the peak width is bigger only at peaks 1 and 2, where CUP on side B has by 10 cm^{-1} greater width than on side A. Furthermore, CUP on side B has by 13 cm^{-1} greater width than RUP. Larger width means differences in effective surroundings, which could represent less symmetry of the local environment and therefore less uniform potential for vibrating species.

A note is in place here, namely that a slightly higher difference in the width could also be a consequence of overlapping peaks. In the region around 1000 cm^{-1}, we register more vibrations (stretching and deformations) of more groups: C–O–C, C–O, C–C, C–N. These groups are located in cellulose fibres. As this is a mixture of more substances, more peaks appear; thus the overlapping into a wide absorption band.

This is a consequence of the vibration of more groups in variable molecular surroundings, which cause distinctive peaks shift and widen. The asymmetric stretching of the C–O–C groups is caused by the peak at 1160 cm^{-1}, whereas the stretching of C–O in cellulose/hemicellulose mole-cules is caused by the peaks at 1104 and 1028 cm^{-1}. The strongest peak at 1028 cm^{-1} is accompanied by two distinct peaks at 1051 and 1002 cm^{-1} [8].

At a more exact overview of *Table 2*, it can be instantly noticed that the differences in the position, height and half-width of peaks CCP and RCP are smaller than those for CUP and RUP.

At peak 5 between samples CCP and RCP, the difference in the position is slightly bigger (the corresponding peak shifts by 7 cm^{-1} for side A and B), as well as in the half-width (RCP has by 6 cm^{-1} bigger width on side A).

Table 1: Position (v0), height (I0) and half-width (hw) of individual peaks of classic (CUP) and recycled (RUP) uncoated paper for side A and B.

** Half-width listed at peak 2 is actually half-width of peaks 1 and 2.*

	Peak 1		Peak 2			Peak 3		Peak 4		Peak 5	
	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	hw (cm^{-1})	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0
CUP/A	3333	0.040	3296	0.038	256*	2898	0.012	1649	0.006	1412	0.130
CUP/B	3332	0.051	3295	0.049	266*	2899	0.016	1648	0.008	1421	0.090
RUP/A	3332	0.009	3292	0.009	258*	2885	0.003	1649	0.006	1417	0.039
RUP/B	3332	0.012	3293	0.011	253*	2887	0.003	1649	0.006	1423	0.031

	Peak 6			Peak 7		Peak 8		Peak 9		Peak 10		Peak 11		
	ν_0 (cm^{-1})	I_0	hw (cm^{-1})	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	hw (cm^{-1})
CUP/A	1160	0.051	30	1104	0.087	1052	0.158	1028	0.179	1003	0.154	873	0.115	14
CUP/B	1161	0.055	30	1104	0.097	1053	0.174	1029	0.194	1002	0.167	874	0.072	14
RUP/A	1160	0.015	25	1102	0.025	1051	0.044	1029	0.052	1003	0.048	873	0.034	15
RUP/B	1160	0.018	26	1103	0.030	1053	0.053	1029	0.061	1002	0.055	874	0.024	16

Table 2: Position (v0), height (I0) and half-width (hw) of individual peaks of classic (CCP) and recycled (RCP) coated paper for side A and B.

	Peak 1		Peak 2		Peak 3		Peak 4			Peak 5		
	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	hw (cm^{-1})	ν_0 (cm^{-1})	I_0	hw (cm^{-1})
CCP/A	3693	0.021	3649	0.011	3621	0.010	1797	0.005	11	1398	0.186	129
CCP/B	3692	0.023	3649	0.011	3621	0.011	1797	0.005	12	1400	0.174	128
RCP/A	3692	0.013	3649	0.007	3620	0.007	1797	0.007	12	1391	0.198	135
RCP/B	3692	0.011	3649	0.007	3620	0.006	1797	0.006	12	1393	0.156	131

	Peak 6		Peak 7		Peak 8		Peak 9		Peak 10		Peak 11		
	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	hw (cm^{-1})
CCP/A	1091	0.093	1030	0.182	1005	0.184	937	0.059	913	0.098	872	0.225	14
CCP/B	1090	0.085	1030	0.163	1005	0.164	937	0.054	913	0.089	872	0.198	14
RCP/A	1094	0.069	1030	0.123	1006	0.128	938	0.043	912	0.069	872	0.216	13
RCP/B	1094	0.055	1030	0.104	1006	0.109	937	0.044	912	0.061	872	0.172	14

	Peak 12		Peak 13		Peak 14		Peak 15		
	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	ν_0 (cm^{-1})	I_0	hw (cm^{-1})
CCP/A	789	0.016	753	0.027	697	0.073	535	0.172	34
CCP/B	789	0.015	753	0.025	697	0.065	536	0.143	34
RCP/A	788	0.012	756	0.023	698	0.065	535	0.128	31
RCP/B	789	0.014	756	0.019	698	0.049	537	0.097	31

Bigger differences in the peak height between side A and B of both samples (cf. *Figure 4* and *Table 2*) appear at peaks 5, 7, 8, 11 and 15 (sides A have higher values), and between samples CCP and RCP at the height of peaks 7, 8 and 15 (CCP has higher values). The results show that side A has in comparison with side B higher kaolin and $CaCO_3$ content, and CCP compared to RCP higher kaolin content.

3.3 Ash content

The results of residues on ignition at 525 °C and 900 °C on oven-dry basis, calcium carbonate and kaolin content for all paper samples are presented in *Table 3*.

The percentages of calcium carbonate and kaolin in the papers were calculated by the following equations [7]:

$$CaCO_3 = (Ash_{525} - Ash_{900}) \times \frac{100}{44}$$

$$Clay = (Ash_{525} - CaCO_3) \times 1.13$$

where Ash525 and Ash900 are the residues on ignition expressed as mass percentage of the oven-dry samples, $CaCO_3$ is the calcium carbonate content and Clay is the content of the china clay also known as kaolin. The $CaCO_3$ content and the content of the china clay were determined to examine the FT-IR results.

Table 3: Content of ash on ignition at 525 °C and 900 °C, calcium carbonate and china clay in all paper samples.

	Ash_{525} (%)	Ash_{900} (%)	$CaCO_3$ (%)	Clay (%)
CUP	23.4	13.4	22.8	0.7
CCP	39.7	28.3	26.0	15.5
RUP	31.2	19.3	27.2	4.6
RCP	39.6	25.5	32.1	8.5

The results from Table 3 show that both classic papers contain smaller amount of calcium carbonate than recycled papers. Classic uncoated paper (CUP) has 16% lower $CaCO_3$ content in comparison with recycled uncoated paper (RUP), while classic coated paper (CCP) has 19% lower $CaCO_3$ content compared to recycled coated paper (RCP).
Both uncoated papers contain very small amount of kaolin. Meanwhile at coated papers it is clearly seen that classic paper (CCP) contains much larger amount of kaolin than recycled one (RCP), 82% larger. The latter is also proved with FT-IR analysis, where it was recognised that CCP has in comparison with RCP higher kaolin content.

3.4 UV/VIS spectroscopy
Figures 5 and *6* show the reflectance of uncoated and coated papers in dependence of the wavelengths. Particularly interesting are the reflectance values of uncoated papers, the values of which exceed 100%. This is a typical phenomenon of papers which contain optical brighteners, due to which paper emits light even more in the UV spectral region. The phenomenon is the strongest at classic uncoated paper (CUP), where the maximum reflectance value equals 250%. The phenomenon is smaller, yet still noticeable at recycled uncoated paper (RUP) on side B, the reflectance value being 120%.
The coating on paper partially supresses the effect of brighteners, thus it is less distinct or not noticeable at all. In our case, the reflectance values of coated papers are lower than 100%, despite containing optical brighteners. Classic papers have in the UV spectral region higher reflectance values than recycled papers.
Apart from reflectance, we also measured the transmittance values of our samples (*cf. Figures 7 and 8*). The transmittance of recycled uncoated paper (RUP) is in the UV spectral region smaller, while it increases in the visible region and is higher than classic uncoated paper (CUP).

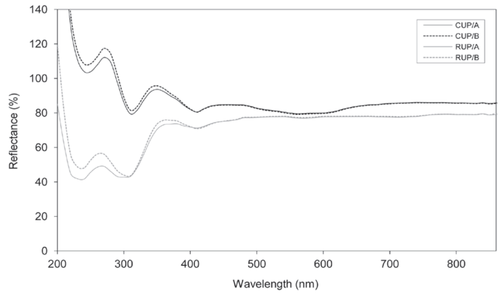

Figure 5: Reflectance spectra for uncoated papers.

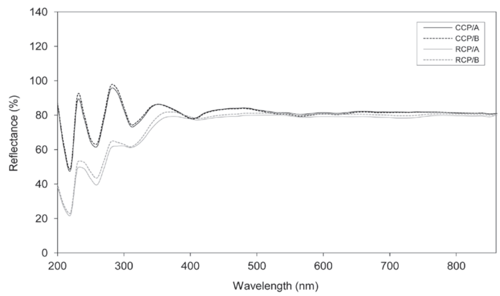

Figure 6: Reflectance spectra for coated papers.

At coated papers, by increasing the wavelength, transmittance increases as well. Classic coated paper (CCP) has higher transmittance than recycled coated paper (RCP) throughout the whole UV and visible spectrum. The differences between the reflectance and transmittance values for side A and B of uncoated and coated papers are extremely small.

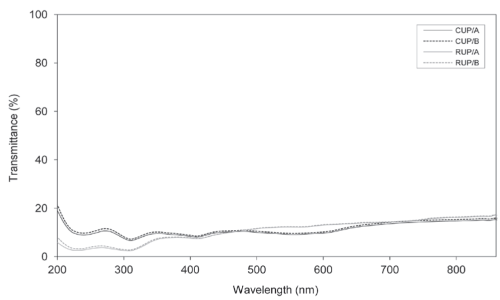

Figure 7: Transmittance spectra for uncoated papers.

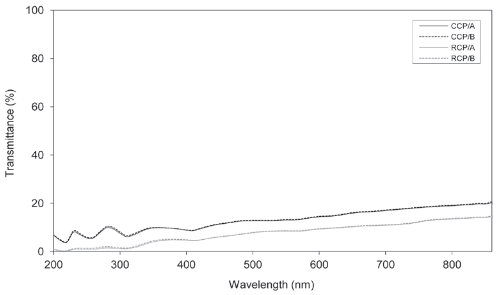

Figure 8: Transmittance spectra for coated papers.

4. Conclusions

With the help of FT-IR-ATR spectra, we detected the chemical structure on surfaces of the investigated classic and recycled papers.

By the help of IR database the calcium carbonate was shown in all papers, whereas on coated papers the additional presence of kaolin was obtained. Comparing IR spectra of samples, we established that the surface of classic coated paper contains more kaolin than the surface of recycled coated paper.

The ash content determination confirms the last finding and shows the percentages of calcium carbonate and kaolin in all paper samples.

However, it should be pointed out here, that this method yields for the presence of calcium carbonate in the bulk of samples which is therefore not directly comparable to the presence on the surface. With a UV/VIS spectrometer, we detected in the measured reflectance the content of optical brighteners in the paper samples, due to which paper additionally emits light in the UV spectral region which gives higher reflectance in the blue region and therefore a whiter surface appearance.

The reflectance values in the UV and visible spectral region were higher at classic papers in comparison with the recycled ones.

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A study on the food contact suitablity 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, diisopropylnaphthalenes (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, azocolorants, 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^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 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% H_2O_2 (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.

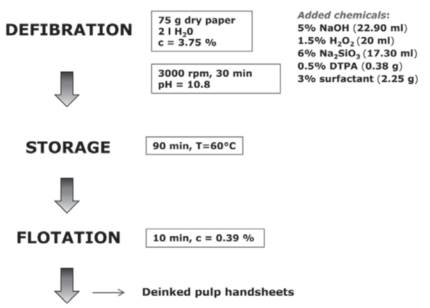

Figure 1: The deinking flotation procedure scheme

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 ^{0}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 flotated 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 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: 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 abd 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 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)	Grammage (g/m^2)	DIPN (mg/dm^2)
			Limit **1.33 mg/dm^2**
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*).

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

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 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/dm^2). 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)
			***Limit* 0.25 mg/dm^2**
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/dm^2). 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.

5. Acknowledgements

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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 Cathuolic 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].

2. Newspaper design

A daily newspaper is an actual paper with most current news – at least that was the case before the new, digital media. Nevertheless, it still brings news, information, analysis and comments about the political, social, economic, cultural, global and national events.

The newspaper head is most commonly placed at the top of the first page. The newspaper layout is divided into a few columns. For a different type of content (i.e. subtitles, titles, body text, headings for figures and tables, pagination etc), specific typography (typeface, type size, leading – i.e. space between lines) is defined [4, 7–9].

Based on their typical design, typefaces can be grouped according to the variations in stroke width and serif design into old-style, transitional and modern; or into a group without (or with a minor) variety in stroke width, i.e. slab serif and sans-serif (lineal). As it is evident from the designation of this typeface style, sans-serif typefaces do not have serifs. These type styles are the most useful for body sized text [4, 9, 10].

Old-style typeface: The principal features of old-style typefaces are a slight contrast between the thick and thin character strokes and an oblique emphasis. An early version (known as Humanist) of these typefaces has steeply sloping serifs and an angled crossbar in the lowercase e.

A later version (known as Garalde) of the old-style typeface has a more pronounced contrast between the thick and thin strokes. The crossbar in e has become resolutely horizontal, the serifs are finer and more horizontal [4, 10–13]. Generally, old style typefaces are legible as body text in printed media.

Transitional typeface: The contrast between thick and thin character strokes is much more marked. The emphasis of curved letters is vertical. Serifs have become even more horizontal. The crossbar in e is horizontal. [4, 10–13] In general, transitional typefaces are still very popular for book, journal and magazine print work, due to their good legibility of the body text.

Modern (didone) typeface: The contrast between the thick and thin strokes of characters has been reduced to hairlines. Serifs have become extremely thin lines – hairlines, which are unbracketed. Right angles predominate among strokes and serifs. The crossbar in e is horizontal [4, 10–13]. Modern typefaces are generally used nowadays as display typefaces and newspaper headlines etc; however, due to the lack of legibility, they are not used as body text.

Slab serif typeface: The contrast between character strokes is limited, slab serif typefaces having little or no difference between the thick and thin strokes. The crossbar in the letter e is horizontal [4, 10–13]. Slab serif typefaces are more preferably used for headlines than for the body text.

Sans-serif (lineal) typeface: As it is evident from the designation of this typeface category, sans-serif typefaces do not have serifs. The contrast between character strokes is limited, sans-serif typefaces having little or no difference between the thick and thin strokes. The crossbar in the letter e is horizontal [4, 10–13]. Sans-serif typefaces can be subdivided into four groups, namely into grotesque, neo-grotesque, geometric and humanist [4, 12].

For titles and bigger sized text, also a decorative, script and similar typestyles can be used [4, 9, 10].

3. Experimental

In the research, the typographic changes in the "old" Slovenec (1873–1945) and "new" newspaper (1991–1996) were analysed. The focus was put on the issues which were actually changed.

The changes in the head size and newspaper format, the size of layout, the number of columns, typestyle, type size and leading of titles, individual text, columns, chronicle etc were compared. The measurements were performed in the Didot sized points [4]. All the published issues of the newspaper are available in the Slovenian National and university library, where the analysis was conducted.

4. Results and discussion

4.1. "Old" newspaper (1873–1945)

The data about the newspaper design are in *Tables 1 and 2*. A part of the first page of the newspaper from its first year of publishing (1873), second (1894) and third (1905) redesign of it, and from when the last change occurred (1926) in this period of the publishing can be seen in *Figures 1–4*.

Table 1: Sizes of measured typographic elements in year of changes

Size (pt)	Year of newspaper design changes					
	1873	1885	1894	1904	1905	1926
Format height	1001	1194	1194	1269	1293	1301
Format width	674	797	792	868	890	876
Layout height	906	1085	1153	1217	1185	1229
Layout width	591	672	674	807	834	810
Head height	95	113	113	132	126	66
Head width	354	636	636	807	559	626
Column width	194	217	217	217	204	198
Size of body text	10	10	10	10	10	10
Leading	11	12	12	12	12	10
Size of titles	14	14	14	22	30	20

Table 2: Difference of typographic elements in year of changes

Page design	Year of newspaper design changes					
	1873	1885	1894	1904	1905	1926
No. of columns	3	3	3	3	4	4
Typestyle of body text	modern	modern	modern	modern	modern, transitional	modern, old-style
Typestyle of title	modern	modern	modern	decorative	decorative	lineal, decorative

The first substantial change occurred in 1905 when the modern typeface was in some types of text replaced with a transitional one and then in 1926, when an old-style typeface was used for the body text in some types of text. The only change in the number of columns appeared in 1905, while the newspaper format and layout was changing constantly. There was no change in the size of body text, whereas the leading altered twice.

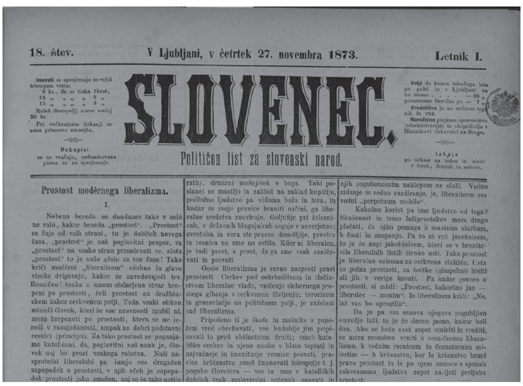

Figure 1: Part of first newspaper page in 1873

Figure 2: Part of first newspaper page in 1894

Figure 3: Part of first newspaper page in 1905

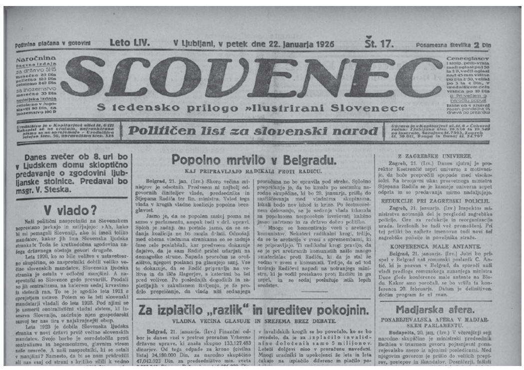

Figure 4: Part of first newspaper page in 1926

At each redesign, the head of the newspaper differed in the type style and almost every time in the size. Very often, the change of the editor resulted in a redesign of the newspaper.

4.2.“New” newspaper (1991–1996)

The data about the “new” newspaper design are in *Tables 3 and 4*. A part of the first page of the newspaper from the first year of revived publishing (1991) and from the year 1994 can be seen in *Figures 5 and 6*. The most widely used typestyle in the “new” newspaper was transitional and was used for most titles and subtitles, sometimes being replaced by lineal and seldom by slab serif typestyles (e.g. in the culture section).

Table 3: Sizes of measured typographic elements in year of changes

Size (pt)	Year of newspaper design changes			
	1991	1992	1993	1994
Format height	1172	1182	1195	1188
Format width	792	810	796	799
Layout height	1084	1100	1116	1121
Layout width	740	740	735	745
Head height	60	62	72	76
Head width	580	555	492	486
Column width	112	113	112	114
Size of body text	9	10	9	9
Leading	10	10	9.33	9.33
Size of titles	28	36	36	36

The size of the body text changed twice, while the size of titles only once.

The newspaper format and layout were constantly changing, whereas the number of columns did not change at all.

t can be seen in the “new” newspaper that the design of the head followed the last head redesign in 1926 and stayed unchanged until the end of the publishing of the newspaper.

There was only a minor difference in size, the letters got a shadow from 1992 onwards and became three-dimensional, and since 1993, the letters were slightly condensed.

Table 4: Difference of typographic elements in year of changes

Page design	Year of newspaper design changes			
	1991	1992	1993	1994
No. of columns	6	6	6	6
Typestyle of body text	transitional	transitional	transitional	transitional
Typestyle of title	lineal	transitional	transitional	lineal


SLOVENEC
Leto LXXV/št. 49 Ustanovljen leta 1865 Četrtek, 22. avgust 1991
Puč propadel
Jelcin ruski Dubček - in zmagovalec

Figure 5: Part of first newspaper page in 1991


SLOVENEC
SREDA, 28. SEPTEMBRA 1994
Slovenija v GATT
Srečanje Clinton-Jelcin
Rusija in ZDA skupaj za mir in

Figure 6: Part of first newspaper page in 1996

5. Conclusion

The design changes in the newspaper Slovenec were made in 1885, 1894, 1904, 1905, 1926, and later in 1992, 1993 and 1994.

The most substantial change in the design can be noticed on the first pages of the newspaper, especially in the newspaper head of the “old” editions. The changes in the newspaper format and layout were typical of both editions of Slovenec. Between the “old” and the “new” newspaper, the differences in the used type size for body text are not significant, while the sizes of titles are very different. The “new” newspaper had many more columns (six) than the “old” one (three or four). For the body text, the transitional typestyle was used in the revival of the newspaper, as it was found in the first biggest redesign of the newspaper in 1905. The modern typestyle, which was mostly used in the “old” newspaper, was probably not used in the revival of the newspaper, as it was established in the 20th century that this typestyle is less legible than the transitional or the old-style one. The design of the "old" newspaper somehow followed the common European newspaper design, especially in the 19th and at the beginning of the 20th century, while in the "new", modern newspaper, the design could not be detected; it seems as if the newspaper wanted to show its historical connection with the "old" newspaper. Therefore, the respect and cultural heritage of the "old" newspaper was lost.

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Flexographic ink composition and its wetting influence on flexo printing plate and printed substrate (PE FOIL)

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Abstract

Surface topography of the printing plate and substrate and the printing ink composition are factors which highly influence ink transfer during flexographic printing process. Consequently, their influence on final imprint quality is certainly not in question.The research presented in this paper was aimed on determination of wetting characteristics of the printing plate and printing substrate, depending on printing ink composition, as well as their surface topology. Samples of printing ink were made with varying solvent and varnish concentration. Evaluation of the printing ink was made by measuring viscosity and the characterisation of surface topography of printing plate and PE foil was made by measuring roughness parameters. To determine wetting characteristics of the prepared ink samples on the used solids measurements of contact angle were performed. Obtained results showed that the ink composition has significant influence on the printing ink viscosity and on the wetting of the printing plate but on the printing substrate as well. The contact angle

between printing ink and both investigated surfaces is decreasing by increasing the solvent and varnish concentration thereby resulting in worse wetting.
The conducted research showed the importance of printing ink composition on the wetting of printing plate and printing substrate, consequently on the ink transfer from ink tray to imprint. Furthermore, one must monitor all observed parameters (surface topography, ink viscosity, wetting characteristics) in order to achieve best printing results.

Keywords: flexography, printing ink, polyethylene, contact angle, surface topography

1. Introduction

Flexography has a large scale growing rate in graphic industry turnaround as it has ability to produce relatively high quality imprints on a various substrates. Flexible printing plate and usage of various printing inks provide ability to print on coated and uncoated paper and board, non-porous substrates including metallised and paper foils and polymer films, used especially in the packaging industry. In order to achieve imprint with satisfactory quality demands one must ensure defined ink transfer from ink tank to the printing substrate. The inks used can be overlaid to achieve brilliant colours and special effects. Among the fluid inks used in flexography are aniline inks (aniline dyes dissolved in alcohol or some other volatile solvent), polyamide inks, acrylic inks and water-based inks. These are superior to oil-based printing inks because they adhere to the surface of the material, while oil-based inks must be absorbed into the material. Many parameters could influence ink transfer, among others printing pressure, surface characteristic of printing plate and printing substrate but also the ink composition. Surface roughness and chemical heterogeneities showed their importance as well as critical influence on contact angle values.
Numerous authors have proposed models to describe the relationship between wettability and roughness, especially on structured surfaces with low surface energy as are polymers. The detailed survay of these methods can be found in [1].
Characterisation of surface topography materials was made by measuring significant amplitude surface roughness parameters (Ra, Rp, Rv, Rq and Rz). Printing inks used in this research were determined by measuring viscosity. This study is aimed to determine the wetting characteristic of the flexographic printing plate and printing substrate (PE - polyethylene) depending on ink composition by measuring static contact angle.

2. Theory

Physical phenomenon described as the tendency for a liquid to spread on a solid substrate depending on the solid surface properties (surface chemistry and surface roughness) and the type of used liquid is wetting [2-7]. It is conditioned by surface tension decrease in solid - liquid system (reduction of surface free energy value occurs when a liquid is wetting a solid surface) [5-7]. The liquid on the solid surface is spreading until the balance between cohesion (internal forces) of liquid, capillary (surface tension) forces and gravity is reached [5-7]. The achieved state of equilibrium corresponds to the minimal energy state among the three phases and thus equilibrium (static) contact angle or Young angle [2,5,8]. The determination of the Young angle is important for the characterization of solid–liquid interfacial systems [5,9]. Relation between Young angle and interfacial energies of materials is given by the Young equation [1, 9-13] (*Figure 1*):

$$\gamma_{sg} = \gamma_{sl} + \gamma_{lg} \cos \theta, \quad (1)$$

where γ are the surface tension coefficients of solid–gas (sg), solid–liquid (sl) and liquid–gas (lg) interfaces.
The equation (1) stands only for ideally smooth and homogeneous solid surfaces. The Wenzel and Cassie–Baxter models of wetting are more appropriate for rough and homogenous surfaces, since they get into account the effect of surface roughness on the static contact angle [1,5,10-13].

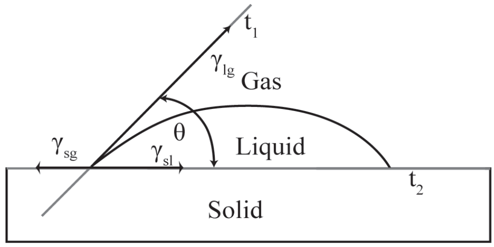


Figure 1: Contact angle between liquid and solid surface

Contact angle (CA) is defined as the angle between tangent on the liquid drop (t1) and the tangent on the solid surface (t2) in the point where all three phases (solid, liquid and gas) meet (*Figure 1*) [6,7]. It can be said that a liquid will spread on the surface with high surface free energy and would not on the surface with low surface free energy [8].
As mentioned before, spreading of the liquid on the solid surface is dynamic process influenced by gravity and surface tension of solid and liquid and therefore one must take into account time from liquid-solid contact in which measurement of the CA will be conducted [6,7]. In *Figure 2a* and b one can observe the significant phases of contact angle measurement.

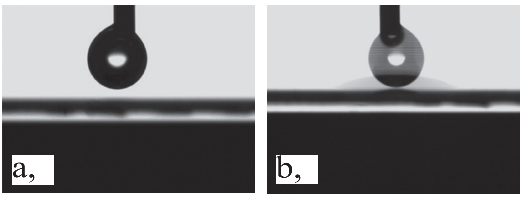


Figure 2: a) Drop forming and b) first contact between liquid and solid phase

On the other hand, if measuring a liquid which evaporates at measuring temperature, measurements of CA should be made at short time (before reaching equilibrium state).
Determination of surface roughness parameters of a material is used in many engineering industries as surface texture of the material often defines its functionality. Substrate and printing plate properties in relation to the pressure and printing speed have a major role in the ink transfer. Surface roughness of the printing plate is more significant than the surface energy when considering print quality [14].
Surface roughness parameters are depending on instrument settings, instrument characteristics, the post processing of obtained data and the nature of the surface texture [12,15]. Taking into account roughness, Young's contact angle and the Wetzel's equation it can be seen that differences in roughness significantly influence on measured values of Young's contact angle or solid–vapour interfacial energy (up to 20%) [12].
Surface roughness could be estimated by usage of various imaging methods such as SEM-scanning electron microscopy or AFM-atomic force microscopy, as well as profilometric methods, like MSP-mechanical stylus profilometry or non–contact laser profilometry [15].
Profilometric analysis is used in material science to quantify the morphology of material surfaces. In contact profilometry peaks and valleys are directly measured.
The measuring unit is equipped with sharp diamond tip which is moving along line of the investigated surface and measures displacements induced by surface irregularities. In this way method provides two dimensional data of the surface. Several test lines need to be recorded to get precise determination of surface texture [15,16].
There are many roughness parameters which can be used for the surface characterization, but most commonly used are amplitude ISO roughness parameters (ISO 4287:1997 and ISO 12218:1997): Ra, Rq, Rz-DIN, Rp and Rv [15,17-20].
The measured surface roughness parameters used in this study are compliant to the geometric product specification standards [15,19,20] and listed below:

– Ra is average surface roughness - the arithmetic mean of the absolute values of profile deviation of mean within sampling length, defined as:

$$R_a = \frac{1}{l}\int_0^l \left| y(x) \right| dx \qquad (2)$$

– Rq (Rms) - root-mean-square deviation – the square root of the arithmetic mean of the squares of profile deviation from mean within sampling length, mathematically described as:

$$R_q = \sqrt{\frac{1}{l}\int_0^l y^2(x)dx} \qquad (3)$$

– RzDIN - average maximum height of the profile (average of all vertical distances between the highest and the lowest point for a sampling length), defined by:

$$R_{zDIN} = \frac{1}{n}(Z_1 + Z_2 + \ldots + Z_n) \qquad (4)$$

– Rp – levelling depth, distance between highest peak and the reference line, and
– Rv – maximum depth of profile valley (see *Figure 3*).

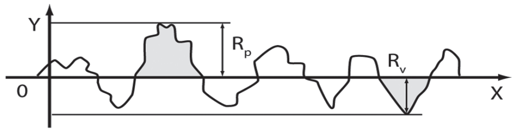


Figure 3: Levelling depth and maximum depth of profile valley

4. Materials and methods

The flexographic printing plate used in this study was a 1.14 mm thick thermal processing digital printing plate. As a printing substrate, a 0.045 mm thick polyethylene corona treated foil was used.

The ink samples were made of commercial black ink with different amount of containing components. The ink components were: base black slow, base technology varnish, base varnish and solvent.

The base black slow is a concentrated liquid black inorganic pigment dissolved in polyurethane binder and organic solvent (chemical mixture of nitrocellulose, ethyl acetate, ethyl alcohol, 1 – methoxy – 2 propanol).

The base technology varnish is a technology varnish based on nitrocellulose / polyurethane binder and organic solvents (chemical mixture of nitrocellulose, ethyl acetate, ethyl alcohol, 1 – methoxy – 2 propanol, 1 – propoxy – 2 propanol , 3 – methoxy – 1 – butanol).

Base varnish is a varnish made of nitrocellulose binder dissolved in organic solvents (chemical mixture of nitrocellulose, ethyl acetate, ethyl alcohol, 1 – methoxy – 2 propanol, n – propanol).

Solvent used is organic solvent used for nitrocellulose based dies.

Six samples of the printing ink were prepared, where amount of added base varnish and solvent was varied. They are named as Specimen 1 up to Specimen 6. Their composition is listed in *Table 1*. Viscosity of the ink samples was determined using Brookfild DV/II+ Pro programmable viscometer at temperature of 23^{0}C.

Table 1: Ink specimen composition specification

Ink specimen	Base black slow (g)	Base technology varnish (g)	Base varnish (g)	Solvent (g)
Specimen 1	500	300	100	100
Specimen 2	500	300	120	110
Specimen 3	500	300	110	110
Specimen 4	500	300	105	110
Specimen 5	500	300	100	110
Specimen 6	500	300	100	120

The contact angle (CA) analysis was performed in order to evaluate the wetting characteristics of used solids (flexo printing plate and PE foil) applying inks with different composition.

Measurements of static contact angle were performed by Dataphysics' OCA30 computer controlled measuring unit using Sessile drop method. Measurements were conducted at 24^{0}C with drop volume of 1.5 µl. CA computations were made by using Laplace-Young fitting method. *Figure 4* shows formation of drop and point of the measurement.

Figure 4. Measurement of the contact angle in OCA software

Profilometric roughness parameters - Ra, Rp, Rv, Rq and Rz were measured by the Portable Surface Roughness Tester TR 200 provided with a diamond tip with 2 µm radius. The unit is compatible with ISO 4287, DIN 4768, ANSI B 46.1 and JIS B601 standards. The relevant parameters of the device used in measurements are presented in *Table 2*.

Table 2. The measurement parameters of the TR200

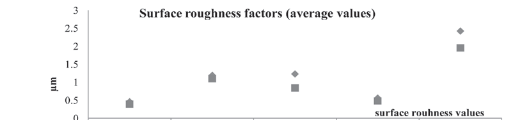
Sampling length	Traversing speed	Measuring range	Resolution
0.80 mm	0.5 mm/s	+/- 20 µm	0.01 µm

5. Results and discussion

Measurements of the roughness parameters of the investigated solids were repeated ten times longitudinal and ten times across to avoid possible variations in the corona processing of the PE foil or surface texture of the developing fabric in printing plate processing.

On the graph presented in *Figure 5* the average values of the measured roughness factors are given.

It can be seen that printing plate has higher values of the roughness parameters than PE foil.



	Ra	Rp	Rv	Rq	Rz
◆ Printing plate	0.46	1.19	1.23	0.56	2.42
St.dev.	0.059	0.19	0.19	0.07	0.37
■ PE foil	0.39	1.1	0.84	0.48	1.95
St.dev.	0.07	0.29	0.17	0.08	0.42

Figure 5. Surface roughness factors (average values)

Surface roughness of the printing plate is certainly influenced by thermal developing where the surface of the printing plate comes in contact with relatively rough fabric used in order to remove unexposed parts of photopolymer. Rather rough surface of the PE foil is made by corona treatment used to improve printing ink adsorption during the printing process.
Table 3 presents results of the viscosity measurement of investigated ink compositions. Results show that increasing concentration of the solvent and base varnish causes decrease of the printing ink's viscosity. Knowing the variations in amount of added base varnish and solvent (*Table 1*), it can be seen that solvent has a greater influence on ink viscosity meaning that the same amount of solvent added, causes lower ink viscosity than the same amount of added base varnish.

Table 3: Ink viscosity values

Ink specimen	Specimen 1	Specimen 2	Specimen 3	Specimen 4	Specimen 5	Specimen 6
Viscosity value (mPas)	39.2	29.2	26.6	24.2	20.8	15.7

CA measurements were repeated 12 times and results presented in *Figure 6* show the average values. Lines in the graphic presented in *Figure 5* are trend lines of the measured results.
Results show that the CA value measured on printing plate, as well as on the PE foil, decreases by increasing solvent and varnish concentration in printing ink. The differences between minimal and maximal value of CA are on both solids round 12o. The CA for PE foil shows constant decrease achieving minimal value at the last investigated printing ink sample. On the other hand, CA on the printing plate is increasing to reach maximum for specimen 4 after which it decreases.
These results indicate smaller influence of the roughness on the wetting of investigated solids with printing ink sample as printing plate has rougher surface then PE foil but in the same time higher CA value with all printing ink samples.
Although, one must keep in mind that diamond tip of the contact profilometer has diameter of 2 µm which makes it unable of detecting smaller peaks or valleys.

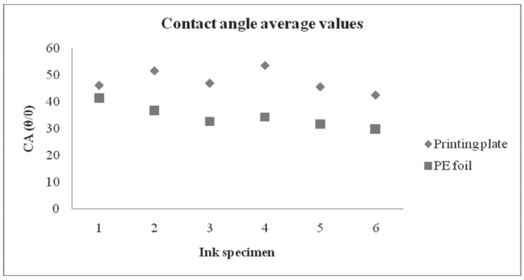


Figure 6. Contact angle average values of ink specimens on printing plate and PE foil

6. Conclusion

The objective of this paper was to define wetting characteristic, in dependence of ink composition and surface topology, of two different polymer solid materials: the flexographic printing plate and printing substrate PE – polyethylene foil. Wetting properties were determined by measuring static contact angle between prepared printing compositions and investigated solids. Characterisation of surface topography was made by measuring five amplitude roughness parameters - Ra, Rp, Rv, Rq and Rz using contact profilometry method. Used inks were characterised by viscosity measurement.
The investigation was made assuming that ink transfer is highly influenced by the surface characteristic of the printing plate and printing substrate as well as the ink composition.
Gained results showed that concentration of the solvent and varnish in printing ink has high impact on the ink viscosity meaning possible problems in the ink transfer from the ink tray to the printing plate. Contact angle values on the PE foil were smaller than on printing plate regardless of printing ink composition implying better adsorption of printing ink on the PE foil, i.e. good transfer of the printing ink from printing plate to the printing substrate. The lowest solvent and varnish concentration (the highest ink viscosity) causes smallest difference in contact angle

value, consequently smaller ink transfer from printing plate on the printing substrate. On the other hand, pressure in the printing press and higher viscosity of the printing ink with lower solvent concentration could positively influence ink transfer.
In addition, flexography is influenced by many parameters which all must be optimised to achieve desired level of the imprint quality. Obtained results have showed the influence of the ink composition on its viscosity and wetting of printing plate and PE foil. One should keep these results in mind and include them in future research of other parameters in order to optimize printing process to gain needed level of imprint quality.

7. Acknowledement

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Containerboard paper types and their properties

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Containerboard paper is produced in Hungary in the paper mill operated by Hamburger Hungaria Ltd. On the two paper machines located there, about 670.000 tonnes of paper are produced a year, exclusively utilising waste paper, accounting for 85% of the total paper production in Hungary.
The portfolio matches the recommendations by CEPI Containerboard.
Without proper professional experience, choosing from the containerboard grades available on the European market can be quite a headache for anyone wanting to use them for making corrugated products.
Contrary to several other industrial products, there are no official standards for paper grades in the paper industry; whereas, in case of a connector element, for example, an international standard regulates the minimum requirements concerning the element in question.

Paper type	Commercial name	Grammage range
Testliner	Austroliner 2	120 – 150 g/m^2
	Austroliner 3	120 – 175 g/m^2
Brown light weight recycled liner	Austroliner 2 Light	100 – 115 g/m^2
	Austroliner 3 Light	80 – 115 g/m^2
Other brown recycled liner	Austroliner 4	100 – 150 g/m^2
	Austroschrenz	100 – 140 g/m^2
	Austroschrenz Light	70 – 95 g/m^2
Recycled medium	Austrofluting-R	112 – 175 g/m^2
	Austrowelle	100 – 150 g/m^2
	Austrowelle 2	110 – 175 g/m^2
Light weight recycled medium	Austrowelle Light	70 – 95 g/m^2

Purchasing such an element would also give us exact information regarding what load it can withstand; however, in the paper industry, there may be significant differences between the same products made by different paper-makers.

The predecessor of CEPI Containerboard (the containerboard branch of the Confederation of European Paper Industries), Groupement Ondulé, compiled a list of European corrugated base paper types back in 1992.
Due to a lack of normative regulation, the year 2005 version of this list is currently used to give some basic assistance in categorising the containerboard grades.
It is important to mention that the list, although it was put together by paper-makers, was made not for paper-makers, but for manufacturers of corrugated products in order to make orientation easier; and although it cannot be regarded as a standard, most European paper-makers still structured and specified their product range accordingly.
The change in market demand, the endeavour on the side of corrugated product-makers to achieve cost-efficiency and efforts to minimise packaging encourage paper-makers to develop their products, resulting in several products in the market that are worth mentioning in the individual categories, beyond what is defined and listed by CEPI. These developments may result in a change in the containerboard papers list; therefore, the list is currently revised in order to implement the changes that have occurred since 2005.
CEPI Containerboard examines and classifies containerboard papers primarily according to their strength properties.
The requirements concerning strength are typically expressed as indices, that is, as specific values, which makes it possible to classify paper grades independently of grammage.
The requirements for individual paper grades determined by CEPI are minimum values, referring to the average value of the lowest reel in a minimum 20-tonne, homogenous shipment.
The parameters defined are measured in accordance with the relevant standards, while sampling is carried out according to standard ISO186; the climate conditions of measurements are defined by standard ISO187.
The CEPI regulations differentiate between the following containerboard paper types according to their strength properties:
CEPI categorises containerboard paper types according to their function, into liner and corrugating medium paper types. Liners are containerboard papers that are used to make the outer and inner, non-corrugated parts of the corrugated board; while corrugating medium (also called medium or fluting) is containerboard paper that gets corrugated during board production.

1. Liners

They can be classified in two groups, basically: kraftliners, which are liners produced mainly of primary fibre; and testliners, which are primarily made of recovered fibre.
Within these categories, a further distinction is made between brown and white, as well as coated and uncoated (in case of white) liners. The most important strength indices of top liners – based on which CEPI classifies them – are burst, or cross-direction SCT (short-span compression test), in case of white liners, this is complemented by certain surface characteristics, such as brightness and roughness.

1.1. Brown kraftliner

Paper made out of primary, or virgin fibre, whose most important characteristic is burst.

	Burst index [ISO 2759; kP*m²/g]	
	<250 g/m²	≥ 250 g/m²
Long fibre, brown	> 3.5	> 3.0
Short fibre, brown		
Wet strength		

1.2. White kraftliner

Also made mainly out of primary fibre, its most important characteristic is burst, coupled with brightness as a classification criterion.

	Burst index [ISO 2759; kP*m²/g]	Whiteness [ISO 2470-1; %]
Fully white	> 3.5	> 80
White top		> 70
White mottled		–
Coloured		–
Fully white, coated	> 3.5	> 85
White top, coated		> 75

1.3. Testliner

Basically made out of recovered fibre, non-white, non-low grammage paper, the most important strength characteristic of which is burst or cross-direction SCT. Based on these parameters, there are three grade categories.

		Burst index [ISO 2759; kP*m²/g]	SCT-KI [ISO 9895; N*m/g]
Testliner 1	125 – 200 g/m²	> 3.0	> 17.5
Testliner 1	≥ 200 g/m²	> 2.9	
Testliner 2	125 – 200 g/m²	> 2.5	> 15.5
Testliner 2	≥ 200 g/m²	> 2.4	
Testliner 3	125 – 200 g/m²	> 2.0	> 13.5
Testliner 3	≥ 200 g/m²	> 1.8	

1.4. Brown light weight recycled liner

Paper made basically out of recovered fibre, with a grammage strictly below 125 g/m².

Burst or cross-direction SCT is its main characteristic. In this particular case, SCT is defined as an absolute minimum value, not as a specific one.

	Burst index [ISO 2759; kP*m²/g]	SCT-KI [ISO 9895; N*m/g]
120 g/m²	> 2.0	1.60
115 g/m²		1.50
110 g/m²		1.40
100 g/m²		1.30
95 g/m²		1.25

1.5. Other brown recycled liners

These paper types are also made out of recovered fibre, whose strength properties must meet certain burst requirements, or – in case of Schrenz paper types –, there are no defined requirements concerning strength.

	Burst index [ISO 2759; kP*m²/g]
Brown bicolor / Brown duplex	> 1.6
Schrenz	without guarantees

1.6. White recycled liner, uncoated

Paper made basically out of recovered fibre, with guaranteed brightness, roughness and burst values.

	Brightness [ISO 2470-1; %]	Roughness [ISO 8791-2; ml/min]	Burst index [ISO 2759; kP*m²/g]
Grade A	≥ 76	≤ 600	≥ 1.9
Grade B	≥ 70	≤ 1000	≥ 1.7
Grade C	< 70	> 1000	< 1.7

In terms of surface water-absorbing capacity, grades A and B are sized, their typical Cobb60 [ISO 535] values are 25 - 45 g/m².

1.7. Mottled recycled liner

	Burst index [ISO 2759; kP*m/g]
Mottled 1	≥ 2.2
Mottled 2	< 2.2

1.8. White recycled liner, coated.

There are no strength criteria specified by CEPI in case of coated, white top liners.

2. Corrugating mediums

Taking into consideration the function of flutings, there are only strength criteria for them, which are: CMT30 (Concora Medium Test) or cross-direction SCT (short-span compression test).

2.1. SC Fluting

Paper made primarily out of semi chemical primary fibre, whose classification criteria are CMT30 and cross-direction SCT.

	CMT$_{30}$ index [ISO 7263; N*m²/g]	SCT-KI [ISO 9895; N*m/g]
SC fluting	≥ 1.9	≥ 17.0

2.2. Recycled fluting/medium

Paper made basically out of recovered fibre, with CMT$_{30}$ or cross-direction SCT regulation, grammage over 100 g/m².

	CMT$_{30}$ index [ISO 7263; N*m²/g]	SCT-KI [ISO 9895; N/m]
High-performance medium	≥ 1.8	≥ 18.0
Medium	≥ 1.6	≥ 16.0
Medium 2	≥ 1.3	≥ 13.5

Paper made basically out of recovered fibre, with a grammage strictly below 100 g/m². Either CMT30 or cross-direction SCT is important, with both parameters expressed in absolute minimum value, not specific value, in this particular case.

	CMT$_{30}$ [ISO 7263; N]	SCT-KI [ISO 9895; N/m]
100 g/m²	145	1.50
95 g/m²	135	1.40
90 g/m²	125	1.30
80 g/m²	95	1.10

2.4. Other flutings

Fluting made out of straw-pulp with CMT30 regulation, or Schrenz paper without strength guarantee.

	CMT_{30} index [ISO 7263; N*m²/g]
Straw fluting	> 1.4
Schrenz	without guarantees

As a final word, let me comment that as we can see in some cases, the now 7-year-old classification system is rather sketchy here and there, not to mention the fact that practical requirements concerning containerboard papers have also changed in the meantime. Therefore, CEPI Containerboard is working on revising the list, which is expected to yield more accurate data, and in some cases, the introduction of further classification parameters or inspection standards.

Rejtő Sándor Faculty of Light Indrustry and Environmental Engineering

The Faculty offers three BSc programs: Light Industry Engineering, Industrial Design Engineering, Environmental Engineering, and a MSc program in Light Industry Engineering.

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Engineers, with a BSc degree in Industrial Design Engineering will be able to initiate, compile and implement projects, to carry out analyses using relevant design methods and to professionally justify the implemented work procedures. They will have competence in comprehensive product design, taking into consideration aesthetic, usability, market, safety, and implementation aspects, as well as historic, cultural, social, economic, industrial and natural environmental factors related to industrial design and product development. Specialisations: Product Design and Product Management.

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Those who earned the BSc degree can continue their studies on the Light Industry Engineering MSc program. The training is organised in cooperation with the Faculty of Wood Sciences of University of West Hungary. Graduates of the BSc program may also continue their studies in the Engineering Teacher Master program in cooperation with Trefort Ágoston Centre for Engineering Education.

Our special Light Industry Engineering Assistant program offers a more practice-oriented training, the knowledge acquired can be converted to vocational expertise easily. In case of an appropriate performance the graduates of this program may continue their studies on BSc level, where most of their earned credits are taken into account.