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OPTICAL PROPERTIES OF DIGITAL INKS ON STRAW-CONTAINING PAPERS WITH TiO_2-BASED COATING UPON AGEING

Ivana Plazonić [1], Valentina Radić Seleš [1], Irena Bates [1], Maja Rudolf [1], Katja Petric Maretić [1], Igor Majnarić [1]
[1]University of Zagreb, Faculty of Graphic Arts, Zagreb, Croatia

Abstract: *Compared to papers derived from virgin fibres, recycled papers possess somewhat limited optical, strength and printing properties. Strength properties are usually increased by blending recycled fibres with virgin fibres in pulp for paper production, while optical and printing properties are often improved by coating after the paper is being manufactured. In this study, the usability of virgin fibres derived from straw as the agricultural residue of wheat, barley and triticale crops for paper production was evaluated based on the stability of prints on laboratory-made paper. For that purpose, in laboratory conditions, the papers with the addition of straw pulp in the pulp of recycled fibres were formed and prior to printing they were coated with TiO_2-based coating. Process inks, in full-tone, were applied on coated straw-containing papers by digital printing process. Optical properties of digital prints upon accelerated ageing were evaluated based on Euclidean colour difference calculated from spectrophotometric values measured on black and magenta prints before and after 48 h and 96 h of ageing. The obtained results were compared with those provided by printing substrates made only from recycled fibres with and without TiO_2-based coating. It was observed that TiO_2-based coating has no influence on the stability of black digital prints, while each layer of TiO_2-based coating reduces deviations in magenta colour upon ageing.*

Key words: straw pulp, paper, TiO_2-based coating, ageing

1. INTRODUCTION

Waste reduction is a fundamental principle of sustainability, and recycling is an integral part of any waste management plan. The recovery of paper for recycling is significantly higher than glass, metal or plastic. Recycled papers are produced from secondary, already used, old paper fibres. However, fibres in the pulp used to make paper cannot be recycled indefinitely because they become damaged during each recycling cycle. Compared to papers derived from virgin fibres, recycled papers have somewhat limited optical, strength and printing properties (Grilj et al., 2011). The increasing use of recycled fibres as a substitute for virgin fibres has resulted with papers with poorer mechanical properties (Gulsoy & Erenturk, 2017; Obradovic & Mishra, 2020) due to reduced interfiber bonding and one way to restore bonding strength of recycled pulp is to blend it with virgin fibres (Czene & Koltai, 2020). To enhance the strength properties of the paper, virgin wood fibres are added to the recycled pulp in most paper mills (Minor et al., 1993; Rowell et al., 1992; Fioritti et al., 2021). Virgin fibres are needed to keep the global fiber cycle going, as recycled fibres degrade after several uses. Although some non-wood fibres are used by the mills, such as cotton, which is the purest form of natural cellulose and sugarcane bagasse, the possibilities of using other sources of non-wood fibres are being intensively studied (Ferdous et al., 2021). On the other hand, different types of coatings can be used to achieve the desired surface properties for high-performance printing on recycled paper. Coated papers restrict the amount of ink that is absorbed into the paper, allowing the ink to sit on top of the paper, in a crisp defined dot, while uncoated papers are more porous. Double coated papers are usually graded as high-quality papers, where the first layer of the coating, a precoat, serves to fill-in the surface pores while the topcoat, which will be printed on, is of higher quality (Mangin et al., 2012). Therefore, it was not surprising that a different number of TiO_2-based white ink layers printed on a paper substrate with the inkjet technology can influence the legibility of prints, where the best print legibility was achieved at two layers of white ink (Možina et al., 2016). However, paper as a printing substrate is subjected to numerous deterioration processes from the moment it is produced, which can lead to the irreversible degradation of text or image printed on it. Paper, bindings, printing inks, dyes and pigments are particularly sensitive to the light because they absorb light energy which can initiate many possible sequences of chemical reactions that damage the paper (weakening the cellulose fibres in the paper and yellowing or darkening the paper) and cause the printed text to fade or change colour (Afsharpour et al., 2011).

In this study, we investigate the role of TiO_2-based coating applied to the surface of the straw-containing paper in the protection of colour fading of the prints against the damaging effect of ultraviolet radiation and visible light degradation.

2. METHODS

This research is divided into the following steps: conversion of straw into pulp; production of straw-containing papers; single and double layer coating of laboratory-made papers; UV inkjet printing; accelerated ageing and evaluation of the stability of prints upon ageing.

Papers for this analysis were produced under laboratory conditions where the pulp of recycled wood fibres (N) was replaced, to a certain extent, by the straw pulp from three agricultural crops (Table 1).

Table 1: Abbreviations used for marking laboratory-made papers

Abbreviation	Pulp composition
N	100% recycled wood pulp
1NW, 1NB, 1NTR	10% straw pulp + 90% recycled wood pulp
2NW, 2NB, 2NTR	20% straw pulp + 80% recycled wood pulp
3NW, 3NB, 3NTR	30% straw pulp + 70% recycled wood pulp
* W = wheat pulp; B = barley pulp; TR = triticale pulp	

Straw collected after harvesting wheat, barley and triticale was purified, hand-cut and converted into pulp by the soda method under conditions summarized in Table 2 (Plazonic et al., 2016).

Table 2: Pulping conditions

Crop straw	Method	Pulping conditions
wheat	Soda pulping	Temperature of 120 °C, alkali level of 16% for 60 min, and a 10:1 liquid to biomass ratio
barley		
triticale		

This obtained unbleached straw pulp was added into the pulp of recycled wood fibres in the laboratory production of paper at the Rapid Köthen sheet former (FRANK-PTI GmbH, Birkenau, Germany) according to EN ISO 526 9-2:2004 standard. The papers produced in the laboratory have a diameter of 20 cm and a weight of about 42.5 g/m^2.

Since the paper substrates (Table 1) were made with the addition of unbleached straw pulp under laboratory conditions and their surface was not pure white, they were coated with one or two layers of TiO_2-based coating before printing to improve the quality of prints. Layers of TiO_2-based white coating were applied to the entire surface of all laboratory-made papers using a digital UV LED inkjet printing machine, Roland VersaUV LEC-300, which works on the piezo inkjet principle. Data on the composition of the TiO_2-based coating used are available in our previous research (Radić Seleš et al., 2020).

Using the same printing machine (Roland VersaUV LEC-300) a test full-tone pattern was printed with black and magenta inks recommended from Roland DG Corporation (Figure 1) on all prepared uncoated and coated substrates (Table 3).

Figure 1: Test pattern of black and magenta ink printed by inkjet printing machine

Table 3: Marks used for substrates prepared for printing

Marks	**Definition**
PS7	commercial paper used as a target reference sample
N_0, $1NW_0$, $2NW_0$, $3NW_0$, $1NB_0$, $2NB_0$, $3NB_0$, $1NTR_0$, $2NTR_0$, $3NTR_0$	laboratory-made papers without coating
N_1, $1NW_1$, $2NW_1$, $3NW_1$, $1NB_1$, $2NB_1$, $3NB_1$, $1NTR_1$, $2NTR_1$, $3NTR_1$	laboratory-made papers coated in one layer
N_2, $1NW_2$, $2NW_2$, $3NW_2$, $1NB_2$, $2NB_2$, $3NB_2$, $1NTR_2$, $2NTR_2$, $3NTR_2$	laboratory-made papers coated in two layers
* W = wheat pulp; B = barley pulp; TR = triticale pulp	

To determine the magenta and black ink layer thickness on the papers with straw pulp without and with coating, the optical ink density (D_i) was determined on all prints by a densitometer eXact, X-Rite (D50/2°). The optical ink density was calculated according to equation 1.

$$D_i = \log\frac{I_0}{I} \tag{1}$$

Where: I – intensity of the light emitted by the ink film in relation to the I_0 intensity of light; I_0 – intensity of the light emitted by unprinted paper substrates.

Thus, a higher optical ink density will mean a higher ink layer or concentration of inkjet ink and higher optical contrast.

All uncoated and coated paper substrates and prints were cut into strips of 60 mm x 90 mm and placed side by side in the Suntest XLS+ test chamber, which is equipped with a daylight filter that emits UV and visible radiation in the wavelength range of 300 nm - 800 nm. The artificial ageing procedure was carried out according to ASTM D 6789-02 standard. The procedure parameters are summarized in Table 4. UV irradiation was performed in two cycles over 48 hours. Approximately one hour of treatment under a xenon lamp corresponds to one day in nature (Debeljak & Gregor-Svetec, 2010; Izdebska et al., 2013).

Table 4: Standard ASTM D 6789-02 procedure in Suntest XLS+ test chamber

Accelerated ageing procedure		Samples in test chamber
Equipment	Suntest XLS+ test chamber	
Standard	ASTM D 6789-02	
Wavelength (nm)	300 - 800	
Irradiance (W/m^2)	765 ± 50	
Filter	daylight	
Relative humidity (%)	49	
Temperature of ambient (°C)	22.3	
Total duration of process (h)	96 (2 cycles of 48 h)	

The impact of a single and double layer TiO_2-based coating on the improvement of print stability upon accelerated ageing of papers containing straw pulp was evaluated based on the Euclidean colour difference calculated according to equation 2. The colorimetric values of papers as printing substrates and prints were determined using an X-Rite SpectroEye spectrophotometer. Colour data were measured under illuminant D50, 2° standard observer. Based on those measurements, the colorimetric difference (ΔE_{00}*), that occurred after ageing, was calculated using the following equation (Luo et al., 2001), using the corresponding unaged print as a reference:

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

Where: ΔE_{00}^* – total colour difference, the Euclidean colour difference; $\Delta L'$ – the transformed lightness difference between prints before and after accelerated ageing; $\Delta C'$ – the transformed chroma difference between prints before and after accelerated ageing; $\Delta H'$ – the transformed hue difference between prints before and after accelerated ageing; R_T – the rotation function; k_L, k_C, k_H – the parametric factors for variation in the experimental conditions; S_L, S_C, S_H – the weighting functions.

3. RESULTS AND DISCUSSION

Table 5 summarizes the ink densities of printed substrates before and after two cycles of the ageing process, where the result values represent the average of 10 measurements of the same sample with the corresponding standard deviation. It is important to emphasize that the thickness of the ink layer on the surface of the printing substrate is highly decisive in light resistance. Titanium dioxide (TiO_2) is a white powder, and coating based on TiO_2 increases the reflection of the light from the surface of coated printing substrates. A thicker layer of such a coating provides prints with higher contrast and better quality (Puhalo et al.,2013). As the thickness of the white TiO_2 film increases, the light-fastness will be higher as the number of pigments affected by the light in a particular region will increase. It is therefore important to maintain a continuous ink thickness during printing (Aydemir & Yenidoğan, 2018). From the obtained ink density values (Table 5), it is evident that each layer of TiO_2-based coating applied to the printing substrate slightly increases the ink density of black and magenta prints regardless of the type of printing substrate used. It is also noticed that the ink density of black and magenta prints on uncoated printing substrates (N_0, $1NW_0$, $2NW_0$, $3NW_0$, $1NB_0$, $2NB_0$, $3NB_0$, $1NTR_0$, $2NTR_0$, $3NTR_0$) decreases with each 48-hour ageing cycle.

Table 5: Ink density of black and magenta prints due accelerated ageing

Substrate	Black ink density			Magenta ink density		
	unaged	aged 48 h	aged 96 h	unaged	aged 48 h	aged 96 h
PS7	1.45 ± 0.01	1.41 ± 0.01	1.39 ± 0.01	1.33 ± 0.02	1.28 ± 0.01	1.26 ± 0.01
N_o	1.16 ± 0.03	1.12 ± 0.02	1.13 ± 0.03	1.00 ± 0.03	0.96 ± 0.03	0.94 ± 0.03
N_1	1.17 ± 0.06	1.30 ± 0.02	1.28 ± 0.04	1.01 ± 0.03	1.07 ± 0.03	1.06 ± 0.03
N_2	1.25 ± 0.03	1.26 ± 0.03	1.25 ± 0.02	1.05 ± 0.02	1.13 ± 0.03	1.12 ± 0.02
$1NW_o$	1.15 ± 0.03	1.11 ± 0.04	1.09 ± 0.03	0.99 ± 0.02	0.94 ± 0.03	0.93 ± 0.03
$1NW_1$	1.17 ± 0.07	1.19 ± 0.04	1.19 ± 0.04	1.03 ± 0.05	1.01 ± 0.03	1.00 ± 0.03
$1NW_2$	1.26 ± 0.04	1.20 ± 0.11	1.16 ± 0.10	1.10 ± 0.03	1.08 ± 0.03	1.08 ± 0.02
$2NW_o$	1.28 ± 0.02	1.22 ± 0.04	1.23 ± 0.02	1.10 ± 0.02	1.04 ± 0.02	1.05 ± 0.02
$2NW_1$	1.17 ± 0.05	1.16 ± 0.06	1.15 ± 0.07	1.01 ± 0.03	1.02 ± 0.03	0.99 ± 0.03
$2NW_2$	1.17 ± 0.08	1.24 ± 0.05	1.24 ± 0.04	1.03 ± 0.04	1.06 ± 0.03	1.03 ± 0.02
$3NW_o$	1.11 ± 0.04	1.14 ± 0.03	1.08 ± 0.03	0.97 ± 0.02	0.98 ± 0.03	0.93 ± 0.04
$3NW_1$	1.15 ± 0.07	1.12 ± 0.08	1.12 ± 0.08	1.03 ± 0.04	0.99 ± 0.02	0.97 ± 0.04
$3NW_2$	1.26 ± 0.02	1.21 ± 0.03	1.15 ± 0.03	1.08 ± 0.05	1.07 ± 0.03	0.95 ± 0.03
$1NB_o$	1.10 ± 0.04	1.06 ± 0.05	1.05 ± 0.05	0.97 ± 0.01	0.93 ± 0.02	0.91 ± 0.02
$1NB_1$	1.13 ± 0.04	1.12 ± 0.04	1.11 ± 0.07	0.99 ± 0.06	0.97 ± 0.04	0.96 ± 0.04
$1NB_2$	1.23 ± 0.02	1.05 ± 0.05	1.10 ± 0.07	1.04 ± 0.04	0.82 ± 0.06	0.94 ± 0.05
$2NB_o$	1.07 ± 0.03	1.06 ± 0.04	1.05 ± 0.04	0.97 ± 0.04	0.95 ± 0.02	0.92 ± 0.04
$2NB_1$	1.16 ± 0.02	1.13 ± 0.03	1.10 ± 0.03	0.99 ± 0.04	0.98 ± 0.02	0.96 ± 0.03
$2NB_2$	1.23 ± 0.03	1.20 ± 0.05	1.21 ± 0.03	1.05 ± 0.03	1.04 ± 0.02	1.03 ± 0.02
$3NB_o$	1.09 ± 0.03	1.05 ± 0.03	1.03 ± 0.02	0.94 ± 0.03	0.93 ± 0.04	0.89 ± 0.04
$3NB_1$	1.13 ± 0.02	1.13 ± 0.05	1.16 ± 0.02	0.99 ± 0.04	0.97 ± 0.03	0.97 ± 0.04
$3NB_2$	1.21 ± 0.02	1.16 ± 0.05	1.17 ± 0.07	1.07 ± 0.04	1.07 ± 0.03	1.06 ± 0.04
$1NTR_o$	1.12 ± 0.03	1.09 ± 0.03	1.08 ± 0.03	1.02 ± 0.02	0.98 ± 0.02	0.95 ± 0.02
$1NTR_1$	1.19 ± 0.03	1.14 ± 0.04	1.13 ± 0.05	1.03 ± 0.05	0.98 ± 0.03	0.99 ± 0.02
$1NTR_2$	1.29 ± 0.03	1.27 ± 0.05	1.27 ± 0.04	1.09 ± 0.02	1.05 ± 0.03	1.07 ± 0.03
$2NTR_o$	1.07 ± 0.04	1.05 ± 0.03	0.10 ± 0.03	0.95 ± 0.02	0.92 ± 0.01	0.88 ± 0.01
$2NTR_1$	1.15 ± 0.03	1.09 ± 0.05	1.09 ± 0.06	0.99 ± 0.04	0.93 ± 0.03	0.95 ± 0.03
$2NTR_2$	1.23 ± 0.02	1.21 ± 0.03	1.16 ± 0.02	1.02 ± 0.05	0.93 ± 0.08	0.88 ± 0.10
$3NTR_o$	1.21 ± 0.05	1.15 ± 0.04	1.15 ± 0.05	1.07 ± 0.03	1.03 ± 0.03	1.00 ± 0.03
$3NTR_1$	1.13 ± 0.08	1.11 ± 0.04	1.09 ± 0.07	0.97 ± 0.06	0.95 ± 0.03	0.91 ± 0.05
$3NTR_2$	1.18 ± 0.06	1.19 ± 0.05	1.20 ± 0.05	1.00 ± 0.05	0.98 ± 0.04	0.98 ± 0.03

In our previous research, we noticed that the TiO_2-based coating did not affect the colorimetric values of black prints to such an extent as it affected the colorimetric values of the magenta prints (Plazonić et al., 2022). As can be seen from Figures 2a-f TiO_2-based coating provides significantly greater protection against degrading magenta than the black colour of prints under UV light exposure. Namely, black prints are more stable to UV radiation (ΔE_{00}^* max. = 4) than magenta prints (ΔE_{00}^* max. = 9), regardless of the type of used printing substrate (reference paper PS7, uncoated or coated control paper N and uncoated and coated straw-containing papers). Also, the duration of the exposure to UV light irradiation has stronger effect on colour changes of the magenta prints, while increasing the radiation time from 48 h to 96 h has no significant effect on the degradation of black prints. Printed reference paper (PS7) showed a significant difference in magenta prints after extending the UV light exposure time in the Solarbox from 48h to 96h as evidenced by an 80% increase in ΔE_{00}^* value, while black prints did not degrade further under the same test conditions. The 36% increase in of ΔE_{00}^* value after the second 48h ageing cycle of magenta prints on the unprinted control sample N_0 (laboratory-made paper with 100% recycled fibres) was reduced by 12% with one layer of TiO_2-based coating i.e., by 20% with two layers of TiO_2-based coating. The same trend of reduction in colour deviation was observed for printing substrates containing any type of straw pulp by applying TiO_2-based coating to the paper surface. In addition, a significant effect of the coating applied in two layers on the stability of the magenta prints was observed regardless of the composition of the printing substrate.

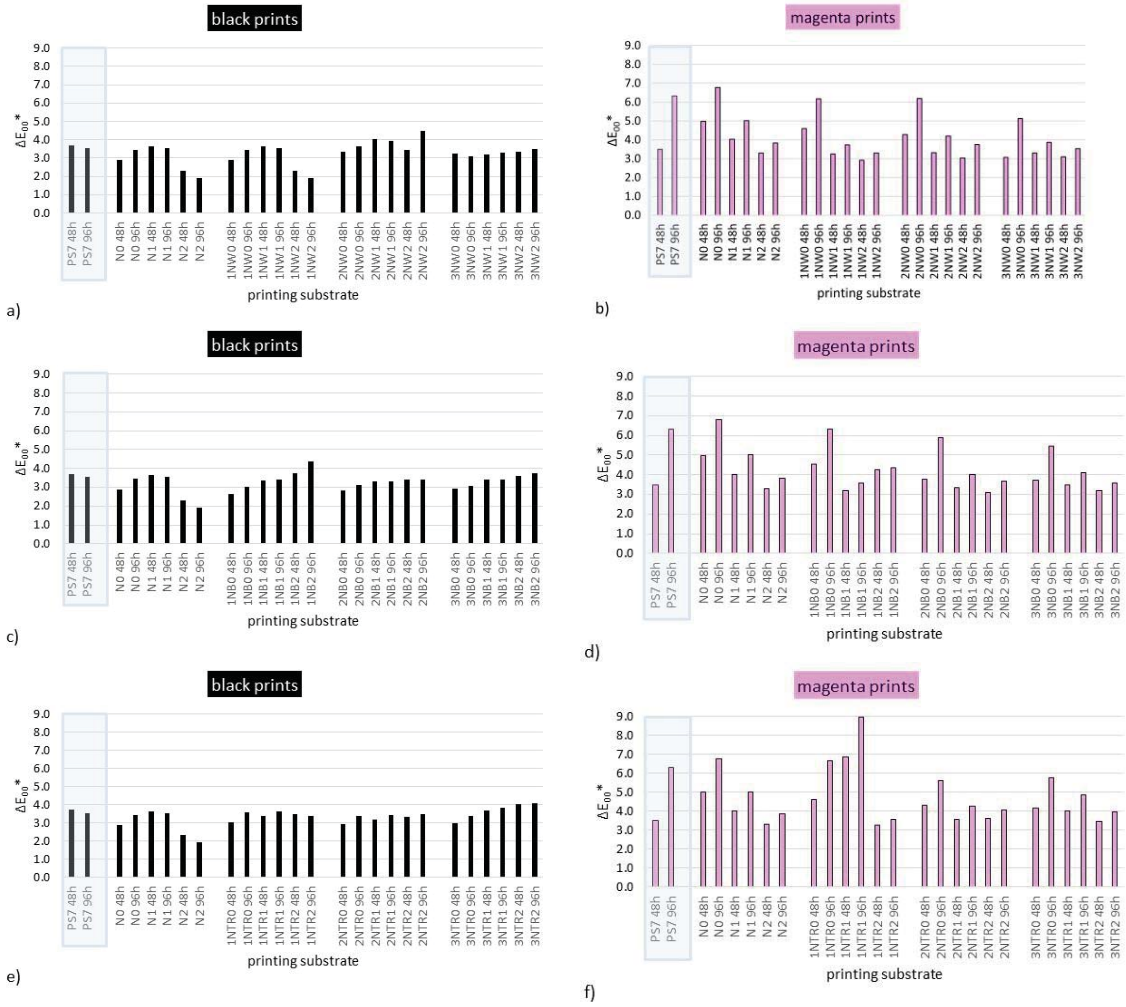

Figure 2: Stability of black and magenta prints made on all analyzed substrates after accelerated ageing expressed through Euclidean colour difference (ΔE_{00})*

4. CONCLUSIONS

Black prints show equal stability to accelerated aging regardless of the type of used printing substrate. Neither the change in the composition of the pulp for the production of the printing substrate nor the coating based on TiO_2 has any significant impact on the stability of the black ink over time. On the other hand, it was noticed that TiO_2-based coating has a dual positive impact on magenta prints exposed to UV radiation. Namely, coating with TiO_2 enables higher stability of magenta prints already with one and especially with two coating layers. After an ageing interval of 96 hours, a reduction in colour degradation was obtained for the coated magenta prints compared to the PS7 reference sample used for commercial purposes. In addition, it was noticed that a TiO_2-based coating has a positive influence on ink stability when exposure time is extended.

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