ANALYSIS OF OPTICAL PROPERTIES OF LABORATORY PAPERS MADE FROM STRAW PULP AND COATED WITH TITANIUM DIOXIDE WHITE INK

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Considering that optical properties play a very important role in the printing industry, this research focused on analysing the optical properties (opacity, whiteness, brightness and yellowness) of laboratory made papers from recycled wood pulp, with the addition of different straw contents, coated with titanium dioxide white ink. Titanium dioxide white ink was applied on laboratory papers to achieve optical characteristics as those of commercial papers recommended by ISO 12647. All the coated laboratory papers presented whiteness within the recommendations of the standard. Almost all the coated papers made with the addition of straw pulp achieved satisfactory optical properties, especially those containing 10% and 20% straw pulp. Coated laboratory papers containing 10% and 20% wheat pulp were comparable to commercial paper and reached a similar value of brightness, while their opacity and yellowness values were even better than those of commercial paper. In order to preserve the natural appearance of papers made with the addition of straw pulp, it is possible to conduct selective white printing as background for high-quality printing.

Keywords: opacity, whiteness, brightness, yellowness, titanium dioxide, straw pulp

INTRODUCTION

In accordance with the EU's environmental directives, the production of paper and board is increasingly focused on alternative non-wood materials. Given that the paper industry is still using wood as a major source of fibre supply, it is wise to replace it as much as possible with other plant biomasses by utilizing non-wood fibre materials to produce cellulose pulps.¹ Straw as a by-product of crop farming could be used as an environmentally friendly and economical supplement to wood in terms of fibre supply, as it has numerous advantages, namely its abundance and renewability. Wheat, barley and triticale straws are a good fibre material, produced annually in huge quantities worldwide in a much shorter growing cycle than wood. After proper pulping and cleaning, the pulps from these straws can be used for manufacturing various paper products.¹

Naturally, any paper used for printing must have good optical properties, such as opacity, whiteness, brightness and yellowness. Opacity (the degree of non-transparency) is a fundamental property of paper as a whole, yet the measurement of opacity is determined by the ratio of reflectance. The opacity of a sheet is influenced by its thickness, the amount and kind of filler, degree of fibre bleaching, coating etc.³ For a quality print, it is necessary that the opacity exceeds 80%. The highest print quality is determined by high whiteness values, specifically, ISO whiteness values from 140 to 175, and it is also important to understand that opacity is related to other optical properties, such as brightness and Y-value.⁴ Brightness is the numerical value of the reflectance factor of a sample with respect to blue light of specific spectral and geometric characteristics. This method is used for the determination of the brightness of white, near white and naturally coloured pulp, paper and paperboard. Whiteness is a subjectively perceived property and most people consider that it increases when the material has a slightly blue tone. It is thus clear that the

whiteness is in some way an aspect of colour perception, and that any measurement of whiteness must be based on colour measurement. Whiteness is based on the reflectance data obtained over the full visible spectral range, in contrast to brightness measurement, which is limited to the nominal wavelength of 457 nm reflected from the measured surface, therefore whiteness could provide more reliable information on the observed whiteness of the paper.⁵ Yellowness is defined as a measure of the degree to which the colour of a surface is shifted from preferred white (or colourless) towards vellow. Yellowness was developed specifically for determining the yellowness of homogeneous, non-fluorescent, nearly colourless, transparent, nearly white translucent, or opaque plastics (as viewed under daylight lightning conditions), but can also be applied to materials other than plastic fitting this description.⁶

In general, printing paper goes through a bleaching process during production. However, if viewed from another perspective, especially for smaller production, the paper whitening process could be done differently, in terms of paper coating with white pigments. Pigments, together with dyestuffs, are probably the most important items in printing ink and each pigment is identified by names in common use, followed by the first colour index number. White pigments and extenders have been described separately from other pigments, as a special description of types and other properties is essential. Titanium dioxide (TiO₂) is the most important white pigment currently in use. It has replaced nearly all other whites, as it has exceptional resistance qualities, being a very lightfast and heat resistant inert material.⁷ Titanium dioxide as a white powder pigment is mainly used in products, such as paints, coatings, plastics, paper, inks, fibres, food and cosmetics, due to its brightness and high refractive index (>2.4). It occurs as an odourless, colourless to white crystalline powder in two forms: anatase and rutile. Anatase pigments are

used more in liquid printing inks due to their softer structure. Rutile pigments have a higher refractive index, higher opacity and a somewhat rougher structure.⁸ Commercial titanium dioxide pigment is produced either by the sulphate process or by the chloride process.⁹

EXPERIMENTAL

Materials

Laboratory papers were formed from recycled wood pulp with the addition of different contents of straw pulp obtained from agricultural residues of three different annual crops: wheat, barley and triticale. According to the soda method, the straw cut to 1-3 cm length was converted into semi-chemical pulp.¹⁰ The conditions for the soda method are presented in Table 1.

The obtained unbleached straw pulp was mixed with recycled wood pulp in different weight ratios (10%, 20% and 30% for each straw). In this research, commercial paper for newsprint was used as the basic component, respectively as recycled wood pulp. Newsprint paper characteristics are listed in Table 2. Laboratory papers were formed on a Rapid Köthen sheet former (FRANK-PTI GmbH, Birkenau, Germany) according to EN ISO 526 9-2:2001 standard and weighed approximately 42.5 g⁻². In total, 10 types of laboratory papers were formed. Paper PS7 as per ISO 12647-2 standard¹¹ was used as reference paper for comparing the optical properties of papers containing straw pulp, such as whiteness and colorimetric difference values; and commercial paper cPS7 was used as reference paper for determining opacity, brightness and yellowness values. Abbreviations used in marking all samples are listed in Table 3.

Methods

Weight determination

All the samples were weighed before and after coating, to determine the amount of TiO_2 white ink on each paper in grams. Measurement was carried out with a Mettler Toledo KS205DU Dual Range Analytic Scale, which allows measurement in the range of 0-220 g, with a precision of 0.01 mg.

Table 1Operating conditions of straw pulping

Agricultural residues	Pulping method	Extraction conditions			
Wheat straw		Temperature	120 °C		
Barley straw	Soda pulping	Alkali level of 16%	60 min		
Triticale straw		Biomass ratio	10:1		

Basis weight (ISO 536), g	42.5	
Brightness D65 (ISO 247		58.0
Roughness Bendtsen (ISC) 8791-2), mL min ⁻¹	100.0 - 160.0
D (190 4297 1) (1	Machine direction (MD)	2.576 ± 0.319
R _a (ISO 4287-1), %	Cross machine direction (CD)	3.166 ± 0.254
Ash (ISO 2144), %		10.00 ± 0.04
CWSN		9A

Table 2 Properties of commercial UPM News C paper

Table 3 Abbreviations used in marking samples

PS7	Commercial paper (reference) as per standard ISO 12647-2
cPS7	Commercial paper (reference)
100N	Laboratory paper with 100% recycled wood pulp
90N10W	Laboratory paper with 10% wheat pulp
80N20W	Laboratory paper with 20% wheat pulp
70N30W	Laboratory paper with 30% wheat pulp
90N10B	Laboratory paper with 10% barley pulp
80N20B	Laboratory paper with 20% barley pulp
70N30B	Laboratory paper with 30% barley pulp
90N10T	Laboratory paper with 10% triticale pulp
80N20T	Laboratory paper with 20% triticale pulp
70N30T	Laboratory paper with 30% triticale pulp

Table 4 Components of used Ink jet ink

Components	Weight (%)
Acrylic esters	30-40
Titanium dioxide	10-20
Hexamethylene diacrylate	10-20
Tri (propylene glycol) diacrylate	10-20
Phosphine oxide derivative	5-15
Other photo-sensitive monomers	0-5
Synthetic resins	0.5-5

Table 5

Presentation of particle size, particle shape, refractive index and ISO-brightness for titanium dioxide pigments

Coating pigment	Particle size (µm)	Shape	Crystal structure	Refractive index	ISO Brightness (%)
Titanium dioxide (Anatase)	0.2-0.5	Rod-like		2.55	98-99
Titanium dioxide (Rutile)	0.2-0.5	Roundish		2.7	97-99

Coating laboratory papers with titanium dioxide white ink

Laboratory papers were coated in one and two layers with titanium dioxide white ink to achieve the same or even better optical characteristics than those of commercial papers (PS7and c PS7). Layers of white ink in full tone were applied to all laboratory papers by a digital Roland Versa UV-LEC 300 Inkjet printing machine, which works on the piezo inkjet principle. The ink was a white liquid (producer Roland DG Corporation), with a viscosity of 6-8 mPa s, a characteristic odour and flash point of 66 °C. The components of the used inks, expressed in weight percentages, are presented in Table 4.¹² Table 5 shows some characteristics of two forms of titanium dioxide.⁶

Optical properties

To determine the optical properties of the laboratory papers containing straw pulp after coating with titanium dioxide white ink, in comparison with those of commercial papers cPS7 and PS7, several major optical characteristics were analysed. Opacity (TAPPI 425 om-06),³ whiteness (ASTM 313),¹³ brightness (TAPPI 452 om-08)¹⁴ and yellowness (ASTM 313)¹³ were measured with an X-Rite eXact spectrophotometer, with standard illuminate D65, 10° observer without polarization filter and M₀ (N₀) filter. Optical measurements were repeated 45 times on each sample.

Colour differences or Euclidean difference (ΔE_{00}^*)

The Euclidean difference (ΔE_{00}^*) of all the analysed laboratory papers, uncoated and coated with one or two layers of titanium dioxide white ink, was calculated based on measured colorimetric CIE $L^*a^*b^*$ values, which were determined by the X-Rite eXact spectrophotometer. In the CIE $L^*a^*b^*$ colour space, the value L^* represents the lightness of the colour, value $+a^*$ represents redness, value $-a^*$ represents greenness, value $+b^*$ represents yellowness and value $-b^*$ represents blueness.¹⁵ The colour differences of all the examined laboratory papers were calculated by the following equation using the characteristics of commercial paper PS7 as per ISO 12647-2 standard as reference:

$$\Delta E_{00}^{*} = \left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_c S_c}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T \frac{\Delta C'}{k_c S_c} \frac{\Delta H'}{k_c S_H}$$
(1)

where $\Delta L'$ is the transformed lightness difference between the laboratory paper and commercial paper PS7, $\Delta C'$ is the transformed Chroma difference between the laboratory paper and commercial paper PS7, $\Delta H'$ is the transformed hue difference between the laboratory paper and commercial paper PS7, R_T is the rotation function, k_L , k_C and k_H are the parametric factors for variation in the experimental conditions and S_L , S_C , S_H are the weighting functions.¹⁶ Tolerances of the colorimetric differences (ΔE^*_{00}) are presented in Table 6.¹⁷

The entire workflow in this research, from papermaking to final analysis is presented in Figure 1.

RESULTS AND DISCUSSION Weight determination

For defining the weight of white ink coating, the Inkjet nozzle distributed 2 pl of white ink on paper substrates, which were then dried by LED UV technology. Weight determination of the titanium dioxide white ink coating was conducted on all the analysed laboratory papers, one layer $\Delta w_1 = w_{1layer}$ $_{coated} - w_{uncoated(a)}$ and two layers $\Delta w_2 = w_{2layer coated}$ $- w_{uncoated(b)}$. The initial paper sample before coating with one layer of TiO₂ ink is marked as "a" and the paper sample before coating with two ink layers is marked as "b" in Table 7.

Opacity

Figures 2-4 present the opacity values of all the analysed papers, before and after coating with one or two layers of titanium dioxide white ink. The opacity values of the laboratory papers were compared to those of commercial paper cPS7. From the opacity values obtained for all the laboratory papers containing wheat pulp (Fig. 2), it is evident that the laboratory paper containing 10% wheat pulp (90N10W) has the highest opacity after coating with one and two layers of titanium dioxide white ink. The laboratory papers made from 100% recycled wood pulp (100N) have the lowest opacity of all, while the uncoated laboratory paper with 20% wheat pulp (80N20W) has the highest opacity value.

With reference to laboratory papers with barley pulp, Figure 3 shows that the highest opacity value is achieved on the laboratory paper with 10% barley pulp (90N10B) coated with two layers of titanium dioxide white ink and the laboratory paper containing 20% barley pulp (80N20B) coated with one layer of titanium dioxide white ink. Among uncoated papers, 80N20B has the highest opacity value. In Figure 4, it is visible that the laboratory paper made from 100% wood pulp and the laboratory paper containing 20% triticale pulp (80N20T) coated with two layers of titanium dioxide white ink have the highest opacity values. The papers with the highest opacity values after applying one layer of titanium dioxide white ink are laboratory papers 100N, 90N10T and 80N20T. For uncoated papers, the highest opacity value is noticeable on the laboratory paper containing 20% triticale pulp.

Whiteness

Figures 5-7 present the whiteness values of all the analysed papers, before and after coating with one or two layers of titanium dioxide white ink. The grey area – whiteness from 40 to 80 – represents the range of values within the ISO 12647-2 standard for whiteness of commercial paper PS7. The measured whiteness value of commercial paper cPS7 is at the upper limit of the values recommended by ISO 12647-2 standard. The whiteness values of all the laboratory papers containing wheat pulp presented in Figure 5 indicate that the laboratory papers containing 10% wheat pulp achieve the highest whiteness value after coating with one and two layers of titanium dioxide white ink.

 Table 6

 Tolerances of Euclidean differences

Euclidean difference value	Euclidean difference tolerance
< 1	Average human eye does not see the difference
1 - 2	Very small difference – optimal $\Delta E^*_{00} = 2$
2 - 3	Moderate difference
3.5 - 5	Difference
> 5	Great difference



Figure 1: Workflow

Laboratory	· / .	ncoated papers g)	Mass (m) of white ink based on titanium dioxide (g)				
papers	a	b	Δw_1	Δw_2	$\Delta w_2 - \Delta w_1$		
100N	0.028	0.028	0.007	0.014	0.007		
90N10W	0.029	0.030	0.007	0.014	0.007		
80N20W	0.030	0.030	0.007	0.014	0.007		
70N30W	0.030	0.031	0.007	0.013	0.006		
90N10B	0.032	0.031	0.007	0.013	0.006		
80N20B	0.032	0.026	0.007	0.018	0.011		
70N30B	0.029	0.029	0.006	0.013	0.007		
90N10T	0.028	0.028	0.007	0.013	0.006		
80N20T	0.029	0.030	0.006	0.013	0.007		
70N30T	0.027	0.027	0.006	0.013	0.007		

Table 7 Titanium dioxide weight in grams on all laboratory papers







- a_Uncoated paper a_1 layer white 100 ⊙- b Uncoated paper b_2 layer white 98 96 Paper opacity (%) 94 92 90 88 86 100N 90N10B 80N20B 70N30B cPS7 Papers

Figure 3: Influence of coating with titanium dioxide white ink on opacity of laboratory papers with barley pulp compared to commercial paper cPS7



Figure 4: Influence of coating with titanium dioxide white ink on opacity of laboratory papers with triticale pulp compared to commercial paper cPS7

Figure 5: Influence of coating with titanium dioxide white ink on whiteness of laboratory papers with wheat pulp compared to commercial papers PS7¹¹ and cPS7



Figure 6: Influence of coating with titanium dioxide white ink on whiteness of laboratory papers with barley pulp compared to commercial papers PS7¹¹ and cPS7

Coated laboratory papers with 30% wheat pulp have the lowest whiteness values of all. Of all uncoated laboratory papers, the one made from 100% recycled wood pulp (100N) has the highest whiteness value, which was expected, as the added straw pulp was not subjected to the bleaching process. Figure 6 shows that the highest whiteness values are recorded for laboratory paper 100N coated with one and two layers of titanium dioxide white ink. Among uncoated papers, again laboratory paper 100N has the highest whiteness value. Of all papers containing barley pulp, coated and uncoated, the highest whiteness values are observed for the laboratory papers with 10% barley pulp. In Figure 7, a similar pattern is visible as in Figure 6, where laboratory papers made from 100% recycled wood pulp (coated and uncoated) have the highest whiteness values. Among the papers containing triticale pulp, coated and uncoated, the highest whiteness values are noted for the laboratory papers with 10% triticale pulp.

Brightness

Figures 8-10 present the brightness values of all the analysed papers, before and after coating with one or two layers of titanium dioxide white ink. The brightness values of the laboratory papers were compared to those of commercial paper cPS7, whose measured brightness was 75.5. From the brightness values of all the laboratory papers containing wheat pulp (Fig. 8), it may be



Figure 7: Influence of coating with titanium dioxide white ink on whiteness of laboratory papers with triticale pulp compared to commercial papers PS7¹¹ and cPS7

noted that the laboratory paper containing 10% wheat pulp reaches the highest brightness value after coating with two layers of titanium dioxide white ink. Laboratory papers 100N and 90N10W coated with one layer of titanium dioxide achieve approximately the same value of brightness, which is the highest measured value on all the papers coated with one layer. Coated and uncoated laboratory papers with 30% wheat pulp have the lowest brightness values of all. Of all uncoated laboratory papers, the one made from 100% recycled wood pulp has the highest brightness value. Figure 9 shows that the highest brightness values were measured on laboratory paper 100N coated with one or two layers of titanium dioxide white ink. Among uncoated papers, again laboratory paper 100N has the highest brightness value. Of all the papers containing barley pulp, coated and uncoated, the highest brightness values were noted for the laboratory papers with 10% barley pulp. Figure 10 shows that the laboratory papers made from 100% wood pulp (coated and uncoated) also have the highest brightness values. Among papers containing triticale pulp, coated and uncoated, the highest whiteness values were recorded for the laboratory papers with 10% triticale pulp. These brightness results obtained for uncoated papers were expected since unbleached straw pulp (wheat, barley and triticale) was used to form laboratory papers.

Yellowness

Figures 11-13 present the yellowness values of all the analysed papers, before and after coating with one or two layers of titanium dioxide white ink. The yellowness values of all the laboratory papers were compared to those of commercial paper cPS7. Namely, the yellowness of cPS7 paper was by far the highest from all the analysed laboratory papers (uncoated and coated ones, regardless of their composition).

From Figure 11, it is clearly visible that the laboratory papers containing 10% wheat pulp have the lowest yellowness values after coating with one and two layers of titanium dioxide white ink. Among uncoated papers, laboratory paper 100N has the lowest measured yellowness value.



Figure 8: Influence of coating with titanium dioxide white ink on brightness of laboratory papers with wheat pulp compared to commercial papers cPS7



Figure 10: Influence of coating with titanium dioxide white ink on brightness of laboratory papers with triticale pulp compared to commercial papers cPS7

Coated and uncoated laboratory papers with 30% wheat pulp have the highest yellowness values of all. The highest yellowness values were measured on all the laboratory papers made from 30% barley pulp, both uncoated and coated with one and two layers of titanium dioxide white ink. The lowest yellowness values were measured on the laboratory papers made from 100% recycled wood pulp, both coated and uncoated (Fig. 12). Figure 13 shows that, again, laboratory paper 100N (coated and uncoated) has the lowest vellowness values. Among the papers containing triticale pulp, coated and uncoated, the highest yellowness values are noted for the laboratory papers with 30% triticale pulp, and the lowest for the papers with 10% triticale pulp.



Figure 9: Influence of coating with titanium dioxide white ink on brightness of laboratory papers with barley pulp compared to commercial papers cPS7



Figure 11: Influence of coating with titanium dioxide white ink on yellowness of laboratory papers with wheat pulp compared to commercial papers cPS7





Figure 12: Influence of coating with titanium dioxide white ink on yellowness of laboratory papers with barley pulp compared to commercial papers cPS7

Figure 13: Influence of coating with titanium dioxide white ink on yellowness of laboratory papers with triticale pulp compared to commercial papers cPS7

Table 8 Colorimetric values

	U	ncoated pa	aper	Coated paper					
Papers		-	-	1	layer wh	ite	2	layer wh	ite
	L*	a*	b*	L*	a*	b*	L	a*	b*
100N	87.94	-0.10	7.64	90.13	0.60	5.16	92.46	-0.05	2.91
90N10W	86.47	0.01	11.88	90.20	0.71	4.90	93.01	-0.19	2.42
80N20W	85.30	0.32	14.17	89.44	1.07	5.85	92.56	0.01	2.75
70N30W	83.85	0.61	16.81	88.25	1.54	6.88	91.48	0.45	3.14
90N10B	86.92	0.10	11.53	88.95	1.11	6.21	91.40	0.26	3.20
80N20B	85.65	0.72	14.34	87.35	1.63	7.14	91.02	0.51	3.37
70N30B	84.39	0.90	14.94	86.86	2.14	7.83	90.41	0.83	3.53
90N10T	86.92	0.01	11.69	88.92	1.03	6.82	91.72	0.31	3.62
80N20T	86.57	-0.03	12.99	87.95	1.69	8.48	90.99	0.70	3.99
70N30T	85.77	0.03	14.04	87.70	1.95	9.88	90.52	1.03	4.50
PS7 (ISO 12647-2) ¹¹	89.00	0.00	3.00						
cPS7	89.61	0.26	-0.09						

Colorimetric values

CIE L*a*b* colorimetric values for all the analysed laboratory and commercial papers are listed in Table 8.

Euclidean difference

Figure 14 (a-c) presents the calculated colorimetric difference values (ΔE_{00}^*) between the CIE L*a*b* colorimetric values (Table 8) measured for the uncoated and coated laboratory papers, with and without straw pulp, and those of commercial paper PS7 as per ISO 12647-2 standard used as reference sample.

Figure 4 (a) shows that the lowest value of colour difference is perceived on the laboratory

paper made from 100% wood pulp ($\Delta E_{00}^* = 3.8$), while the laboratory papers with 30% wheat pulp ($\Delta E_{00}^* = 10.1$) and 30% barley pulp ($\Delta E_{00}^* = 9.1$) show the highest values in colour difference. These results obtained on uncoated papers were expected since unbleached straw pulp (wheat, barley or triticale) was used to form laboratory papers. The colorimetric difference values reduced significantly for all the laboratory papers after coating with one layer of titanium dioxide white ink (Fig. 14 b). The colorimetric difference values are the lowest on the laboratory paper containing 10% wheat pulp ($\Delta E_{00}^* = 2.03$), and the highest on the laboratory paper with 30% triticale pulp ($\Delta E_{00}^* = 5.88$).





Figure 14: Colorimetric difference values (ΔE^*_{00}) between (a) uncoated laboratory papers, (b) laboratory papers coated with one layer of titanium dioxide white ink, (c) laboratory papers coated with two layers of titanium dioxide white ink, and commercial paper PS7¹¹ used as reference sample

It is interesting to note that the second coating of titanium dioxide white ink gave laboratory papers with straw pulp a lower value of colour difference than that of paper 100N (Fig. 15 (c)). Namely, ΔE^*_{00} calculated for the paper with 100% recycled wood pulp was 2.15. Of all the papers containing straw, the lowest colour difference value is perceived on the laboratory paper containing 20% barley pulp ($\Delta E^*_{00} = 1.49$). The laboratory paper with 10% wheat pulp shows the highest colorimetric difference values ($\Delta E^*_{00} = 2.56$).

CONCLUSION

This study evaluated the possibility to improve the optical properties of laboratory papers containing unbleached straw pulp by coating with titanium dioxide white ink and allowed drawing the following conclusions. Laboratory made papers, coated with one and two layers of titanium dioxide white ink achieved even better opacity values than that of commercial cPS7 paper. Whiteness values were within the range of ISO 12647 standard for all laboratory papers coated with one and two layers of titanium dioxide white ink, except the laboratory paper containing 30% triticale pulp coated with one layer of titanium dioxide white ink. Compared to commercial paper cPS7, similar brightness values were observed for laboratory papers containing 10% and 20% wheat

pulp after coating with two layers of titanium dioxide white ink. The yellowness values of all the analysed laboratory papers, regardless of their composition, are excellent, in relation to commercial paper cPS7. The best colorimetric difference values ($\Delta E^*_{00} \leq 2$) were observed for the laboratory paper with 10% wheat pulp after coating with one layer of titanium dioxide white ink and for all laboratory papers coated with two layers of titanium dioxide white ink.

The conclusion of this study is that almost all laboratory papers with the addition of straw pulp showed satisfying optical properties after coating with titanium dioxide white ink, especially laboratory papers containing 10% and 20% straw pulp. In order to preserve the natural appearance of papers with the addition of straw, it is possible to conduct selective white printing as background for high-quality printing.

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