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Optical deterioration of samples printed with basic Pantone inks

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Abstract:

This study focuses on the research of changes in the optical characteristics of selected offset inks (Yellow, Red 032, Process Blue and Black) printed as a part of Pantone colour book. These changes that occur as a result of natural and accelerated ageing (caused by thermal and UV/VIS radiation) were observed through reflectance spectra in the visible region. A Pantone colour book is a system for identifying, matching and controlling ink colours in the graphic industry. This colour book is the international reference for accurate colour communication. The durability of colours and its unchanged quality play an important role in ensuring accurate colour communication and control. In order to examine optical modifications, the samples were exposed to elevated temperatures and UV/VIS lamp radiation. The FTIR spectroscopy was additionally used for the characterization of printed inks before and after ageing.

Keywords:

Accelerated Ageing; Colour Measurement; FTIR Spectroscopy

1. Introduction

The natural process of deterioration starts as soon as a colour print is obtained. Heat is one of the most important environmental influences on the stability of colour prints. High temperature and humidity adversely affect all colour print materials, although not to the same degree. Such elevated conditions cause the colours to deteriorate quite rapidly. Exposure of printed documents to hostile environment, such as the exposure to UV/VIS radiation and elevated temperatures, may provide information concerning natural changes that are likely to happen in the material over a period of time. The analyses of such processes represent a complex problem since during accelerated ageing procedure, chemical and mechanical properties of paper elements (cellulose, additives) and ink components are simultaneously changed (*Havlinova et al., 2002*).

Since the fifties a great variety of artificial ageing methods has been developed for paper, and the field of application of these methods in the practice of conservation of archival and library materials has broadened enormously. Nevertheless, the fundamental and experimental research into the reliability of artificial ageing analyses is only performed on a limited scale, i.e. the essential questions have not been resolved sufficiently and the findings have not resulted in the use of a generally accepted standard method (*Porck, 2000*). Meaningful application of accelerated tests is on the role of the various reactions that contribute to the deterioration of paper.

Offset lithographic inks are complex pastes in which a coloured organic or anorganic pigment is suspended in an oil-based solvent. Besides pigment and solvent, offset inks contain varnish (long oil alkyd, phennolic or urethane litho varnish), drier (cobalt and manganese salts) and modifier (wax compound for rub resistance). Pigments are particular coloured organic or inorganic solids, usually insoluble in the vehicle or substrate in which they are incoporated. Organic pigments are highly coloured inert syntetic compounds that are usually brither, purer and richer in colour than inorganic pigments. In general, however, they are less resistant to sunlight, chemicals and to high processing temperatures.

The Pantone colour book was created by the offset lithography printing process and contains 1.114 fulltone colours made of 14 basic pantone inks.

Black and white pigments, when compared to other pigments, bear different properties. Black pigment (CI Pigment Black 7) is a pigment with very fine particle size. This pigment is chemically inert and extremely fast to heat, ligth, acids, alkali and solvents. Typical formulation of black offset ink contains 10-15% higher amount of pigments in comparison to other offset inks (*Kipphan, 2001*). Black ink is chemically stable and does not affect the strength of the paper (*Field, 1999*).

2. Material and Methods

This research covers spectrophotometric and FTIR testing of samples obtained with fulltone yellow, red (Red 032), blue (Process Blue) and black inks printed on coated paper (named as C) and uncoated paper (named as U) using the same ink. Coated paper generally produces sharper, brighter images and has higher reflectivity than uncoated paper. During the production process it is filled and coated with a layer of ultra-fine mineral particles bound together with film-forming adhesive. Kaolin (*kaolinte*) is the most frequently used particulate mineral. The theoretical structural formula of kaolin is $Al_2O_3{\cdot}2SiO_2{\cdot}2H_2O$. It is a natural-mined mineral and is classified as coming from either primary or secondary deposits. Primary deposits are found in close association with parent rocks, and therefore have a high proportion of other minerals and impurities, which must be removed before the kaolin is ready for coating applications. Significant properties of kaolin are whitness, low viscosity and non-abrasiveness. All these properties have a very good effect on gloss, smoothness, brightness and opacity of coated paper (*Bund, Ishley, 1991; Preston et al., 2002*).

This study focuses primarily on optical changes of printed inks on offset papers (coated - 216g/m^2, uncoated - 133g/m^2). Two kinds of processes were carried out, natural ageing (3 years) and two methods of accelerated ageing: 12 hours of UV/VIS lamp radiation and exposure to elevated temperature in the oven at T=160°C and at T=130°C. The periods of thermal ageing treatment lasted one, three and six days. Naturally aged samples were stored during three years in a bookcase in a dark and dry place.

Spectrophotometric measurements in the visible part of electromagnetic spectrum were carried out by means of SpectroEye device. Spectrophotometric measurements provided data on the optical properties of analyzed samples that were observed using relative reflectance spectra and colourimetric characteristics by CIE $L^*a^*b^*$ values. Relative reflectance was measured using wavelenghts within an interval between 400 nm and 700 nm, every 10 nm.

Standard illuminant D65 and 10 degree observer were used as measurement conditions (Field, 1999). The colourimetric values $L^*a^*b^*$ were used for colour differences calculation (ΔE^*). Colour difference or Euclidean difference (ΔE^*) is the numerical value for describing difference between two colours (Eq 1.).

$$\Delta E^*_{ab} = \sqrt{\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}} \qquad (1)$$
$$\Delta L^* = L_1^* - L_2^*$$
$$\Delta a^* = a_1^* - a_2^*$$
$$\Delta b^* = b_1^* - b_2^*$$

The colorimetric values of the treated sample are L_1^*, a_1^*, b_1^* while the values of non-treated sample are L_2^*, a_2^*, b_2^* . In the *CIE* $L^*a^*b^*$ colour space the value L^* represents the lightness of the colour and the value $+a^*$ represents redness or the value $-a^*$ represents greenness, and the $+b^*$ value represents yellowness or the value $-b^*$ represents blueness (*Kipphan, 2001; Field, 1999*).

Polymers are difficult to characterise with IR spectroscopy because so many different substances are added to alter the properties of the final product or to make the product cheaper. In some cases, in the IR spectrum bands occur in regions where they are overlapped by bands due to other groups making the charactarisation difficult and sometimes even impossible (*Socrates, 2001; Itrić et al., 2011*). ATR (Attennuated Total Reflectance) technique is an excellent, non destructive method but data are collected only from the sample surface thus the absorption can vary depending on topology of the surface (*Proniewicz et al., 2001*). With ATR spectroscopy it's possible to analyse organic binder materials but it find its use in the identification of pigments (*Derrick et al., 1999; Mazzeo et al., 2007*). Although many inorganic pigments have characteristic absorption bands in the mid-IR region, there are many that either do not absorb in that region at all or have absorptions that are at the low wave number end of the region and are not characteristic enough (*Nyquist et al. 1997*). Those inorganic pigments often have no vibrations in the mid-IR region, and their lattice vibrations occur in the far-IR region (*Kendix et al., 2008*).

The depth of penetration (d_p) of the IR radiation into the sample depends on the (Eq 2.) wavelength (λ) of the IR radiation, the angle of incidence of of the radiation (θ), refractive index of the ATR crystal (n_1) and the refractive index of the sample (n_2).

$$d_p = \lambda / 2\pi\, n_1 [\sin^2\theta - (n_2 / n_1)^2]^{1/2} \qquad (2)$$

It follows from Eq 2. that in order to observe any ATR effect, the refractive index of the sample must be lower than that of crystal. (*Willis et al., 1987*)

The FTIR spectra were recorded by FTIR IRAffinity-21 spectrometer in ATR mode for the investigations of paper and inks on paper, before and after ageing procedures. The Specac Silver Gate™ Evolution is single reflection ATR sampling accessory with angle of incidence at 45° and a ZnSe flat crystal plate (index of refraction 2,4). A total of 15 scans were taken, for each sample, with the resolution of 4 cm^{-1}, spectral range 500-4000 cm^{-1}.

3. Results and discussion

Figures 1-5 present experimental results of reflectance spectral measurements before and after natural and accelerated ageing in the visible part of the electromagnetic spectrum, for all examined samples (as well as coated and uncoated papers).

The measured reflectance for coated and uncoated papers (Fig. 1) show the most significant reflectance changes in the blue part of the spectrum at aprox. 430nm, where the unprinted papers exhibit the maximum reflectance values (coated 108%, uncoated 105%). This phenomenon is influenced by the fluorescent whitening agents (organic materials), whose objective is to absorb ultraviolet radiation and transform it to visible light (Pauler, 2001). In this region reflectance values decrease significantly (values for coated paper: $R_{nat.\ ageing}=77\%$, $R_{UV/VIS\ radiation}=78\%$, $R_{130-6}=50\%$, $R_{160-6}=28\%$).

In the red part of spectra wavelength interval ranges from approx. 550 till 700nm and there are no differences in reflectance values after natural and UV/VIS ageing, while papers exposed to thermal ageing show optical degradation (values for coated paper: $R_{130-6}=88\%$, $R_{160-6}=69\%$), but the decrease in reflectance values is not as high as in the blue part of spectrum.

Reflectance spectra for C and U papers show similar behaviour for all treatments (Fig 1), but the effect of optical degradation is higher for coated paper.

After natural and accelerated ageing treatments by UV/VIS lamp for all printed papers, there is no significant reflectance difference in the whole measured wavelength range. On the contrary, thermal ageing treatment cause pigment degradation, especially in the particulate part of the spectrum (Fig. 2-4).

Different printing colours have different intervals of the selective absorption within the measured wavelength range; blue inks absorbs the longest wavelengths of the incident light (575 – 700) nm (Fig. 4), yellow the shortest (400 – 495) nm (Fig. 2) and red absorbs till the middle part of spectrum (400-560) nm (Fig. 3).

As we can see from Fig. 2-4, the most significant changes in reflectance values during thermal ageing ($\Delta R=R_{before\_ageing} - R_{after\_ageing}$) are in the part of the spectrum in which a particular ink interacts dominantly reflective with incoming electromagnetic radiation, while there are no changes in the region of selective absorption whatsoever, $\Delta R\approx 0$ (yellow, blue) or these changes are negative, $\Delta R<0$ (red).

Among all examined inks, the majority of optical instabilities appear in the process blue ink (Fig. 4) and the most stable pigment (Fig. 3) is red. Every particular ink has specific wavelength in dominant

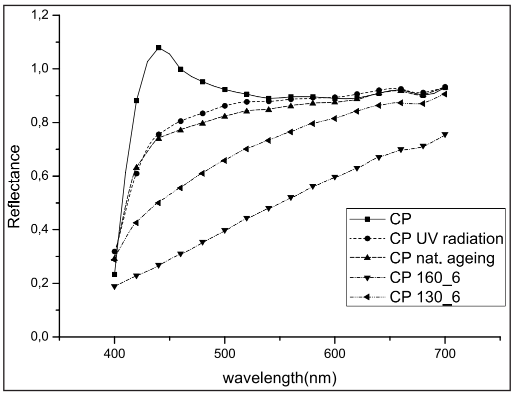


Figure 1. a) Reflectance measurement for all C papers

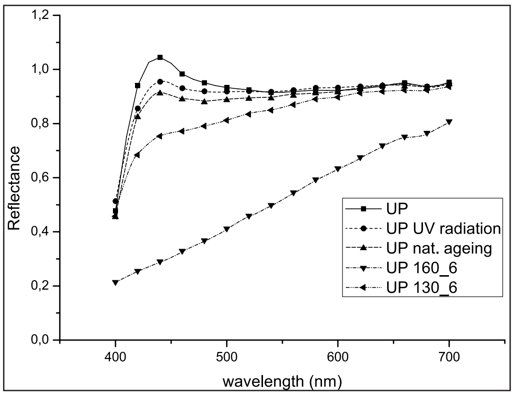


Figure 1. b) Reflectance measurement for all U papers

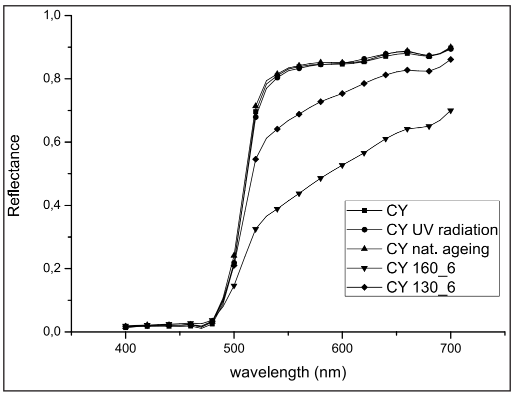


Figure 2. a) Reflectance measurement for all CY prints

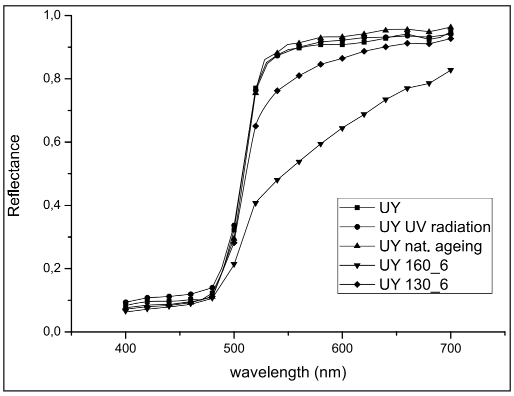


Figure 2. b) Reflectance measurement for all UY prints

reflective interval, which changes in reflectance values achieve their maximum at.

The measured reflection spectra of printed samples show the lowest reflectance values for blue offset ink exposed to the process of thermal ageing at 160 °C.

Black ink isn't an exception to the rule, because it absorbs electromagnetic radiation in the whole measurement spectrum (Fig. 5), so we expect $\Delta R \approx 0$ or even $\Delta R < 0$ for whole measurement interval (400-700) nm.

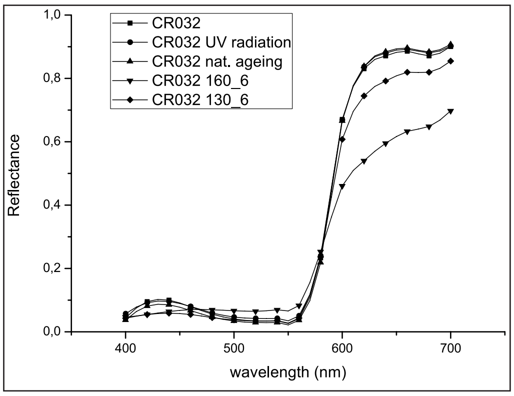


Figure 3.a) Reflectance measurement for all CR032 prints

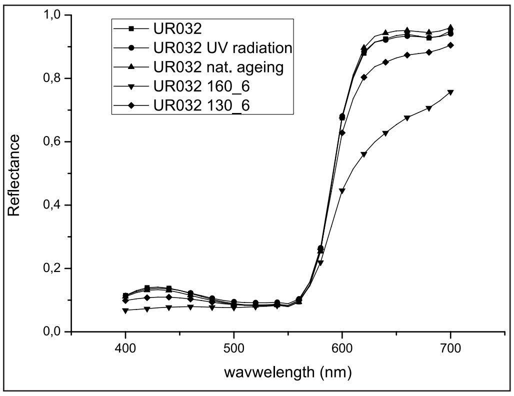


Figure 3.b) Reflectance measurement for all UR032 prints

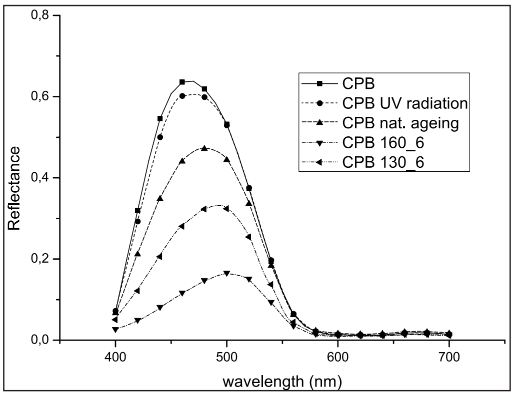
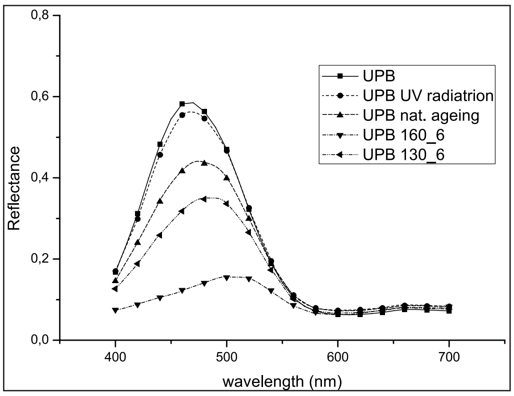


Figure 4. a) Reflectance measurement for all CPB prints



igure 4. b) Reflectance measurement for all UPB prints

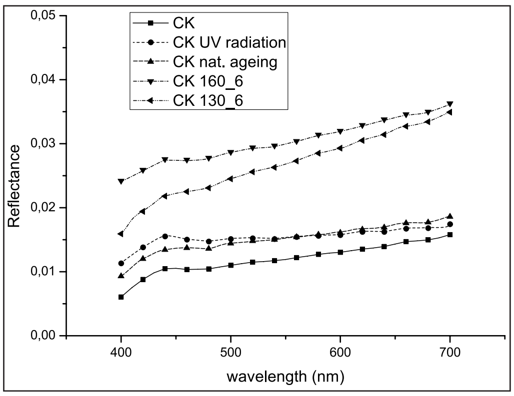


Figure 5. a) Reflectance measurement for all CK prints

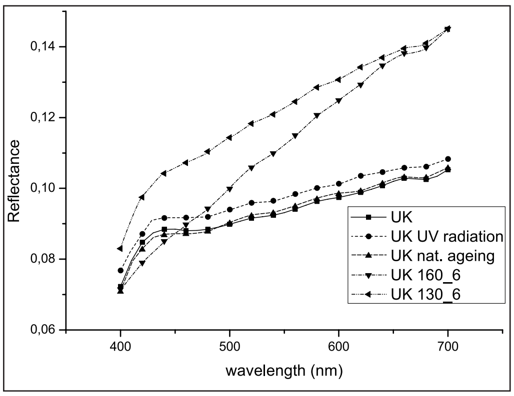


Figure 5. b) Reflectance measurement for all UK prints

So as to be able to monitor optical degradation through reflectance changes better, maximum relative reflectance change has been introduced:

$$\left(\frac{dR}{R}\right)_{\max} = \frac{R_{before\_ageing} - R_{after\_ageing}}{R_{before\_ageing}} \quad (3)$$

The highest grade of optical deterioration is observed in all examined printed samples under elevated temperature, while the influence of natural (3 years) and UV/VIS lamp radiation (12 hours) is negligible. Because of that, maximum relative reflectance changes are compared only for thermal ageing during 6 days, under 130˚C and 160˚C (Fig. 6-7).

Specific properties of black pigment are evident from the maximum relative reflectance differences presented in Fig. 6 and Fig. 7. Only for black ink printed on coated paper reflectance values after thermal ageing become higher in comparison with reflectance values before thermal ageing, especially after exposed in the oven at 160˚C (fading). Chemical stability of black pigment printed on uncoated paper is evident from the insignificant changes in reflectance values (≤20% at 160˚C and ≤10% at 130˚C, Figure 6 and 7).

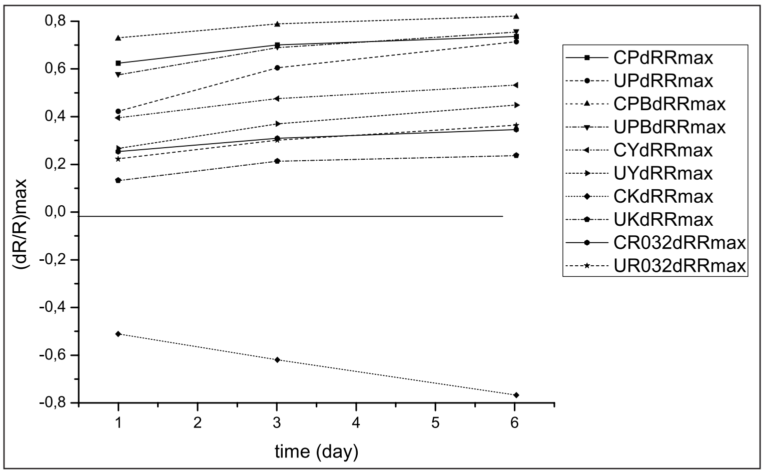


Figure 6. Maximum relative reflectance differences at specific wavelength, $(dR/R)_{max}$, for all measured samples after they had been exposed to heat in the oven at 160˚C for one, three and six days.

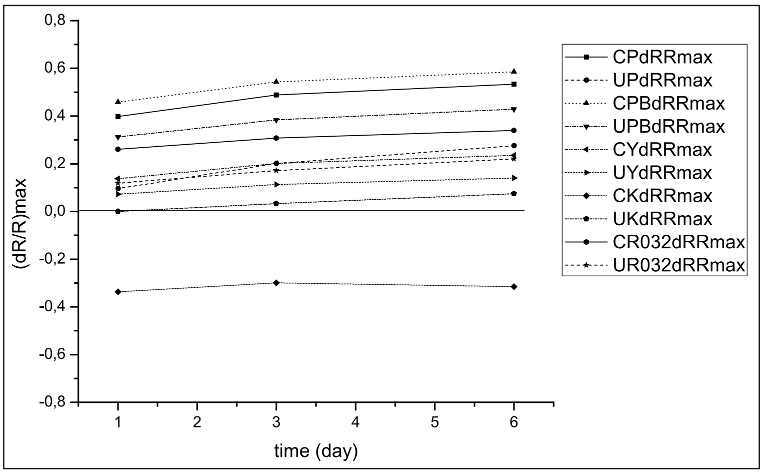


Figure 7. Maximum relative reflectance differences at specific wavelength, $(dR/R)_{max}$, for all measured samples after they had been exposed to heat in the oven at 130˚C for one, three and six days.

Figure 8 illustrates the decrease of brightness (R_{460}) for coated and uncoated papers during the exposure to elevated temperatures. The biggest change is visible in all prints after the first day of thermal ageing (for uncoated paper $\Delta R_{460} \approx 70\%$). Further brightness changes during 3 and 6 days are small (for example coated paper $\Delta R_{460} \approx 10\%$) except for uncoated paper aged at 160°C. When undergoing ageing at a temperature of 130°C, uncoated paper is proven more stable than coated paper at all stages of ageing, while in cases of high temperature (160°C), uncoated paper gradually deteriorates and assumes the same value as the coated paper after it had been aging for 6 days.

From Table 1 it is evident that after all procedures of accelerated ageing, colour differences (ΔE^*) on coated papers and prints on coated papers are higher than on uncoated papers and prints on uncoated papers.

According to tolerance definition $\Delta E^* \leq 2$ is classified as very small noticeable difference for standard observer while $\Delta E^*=5$ is defined like big noticeable difference in the colour whose standard observer can recognized.

Colour difference values caused by natural ageing are minor, with the highest differences values of black prints on uncoated and coated

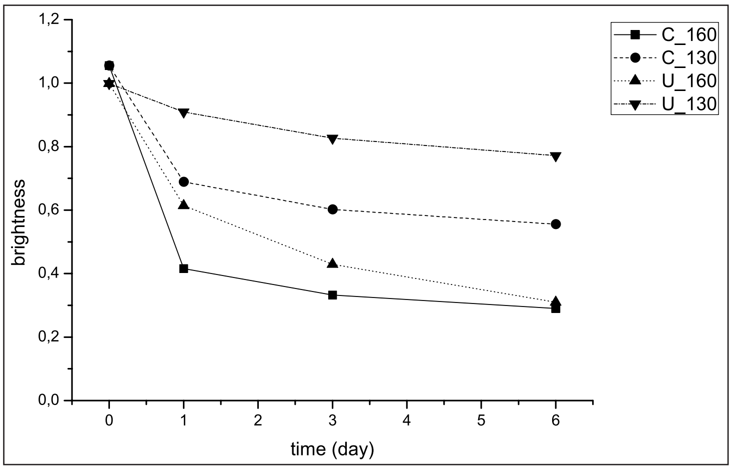


Figure 8. Dependance of paper brightness on the duration of accelerated ageing

Table 1. Colour differences (ΔE^*, ΔL^*, Δa^*, Δb^*) between a non-treated sample and a treated sample

	160°C 6 days				160°C 3 days				UV radiation				nat. ageing			
	ΔE*	ΔL*	Δa*	Δb*	ΔE*	ΔL*	Δa*	Δb*	ΔE*	ΔL*	Δa*	Δb*	ΔE*	ΔL*	Δa*	Δb*
CP	37.50	-19.20	1.57	32.17	26.31	-5.51	2.90	25.56	15.37	-0.81	-4.72	14.62	1.18	-0.07	-0.38	1.11
UP	35.27	-18.61	3.35	29.77	12.38	-2.00	0.51	12.21	4.43	0.04	-1.43	4.19	1.59	0.48	-0.06	1.51
CY	38.58	-18.22	14.29	-30.86	24.82	-2.31	19.27	-15.47	9.14	-2.29	8.80	0.89	2.86	1.00	-0.38	-2.65
UY	28.68	-15.57	14.91	-18.92	17.68	-0.82	17.43	-2.82	8.83	-2.29	8.39	1.54	4.37	-0.31	-1.45	-4.11
CR032	21.58	-3.14	-21.35	0.40	25.51	6.88	-5.57	23.93	2.00	0.59	-1.34	1.36	4.13	-067	1.50	3.79
UR032	16.77	-6.67	-15.21	2.32	18.53	6.46	-3.26	17.06	1.77	0.62	-1.51	0.69	1.61	0.06	0.59	1.50
CPB	46.25	-17.16	-15.49	40.06	35.62	-15.26	-30.32	10.80	5.04	-0.14	-3.93	3.16	2.31	-2.24	0.20	0.54
UPB	42.58	-11.66	-3.84	40.77	20.55	-8.20	-14.68	11.81	3.29	0.46	-0.78	3.16	2.56	0.47	1.21	-2.34
CK	9.58	9.57	0.06	-0.45	8.82	8.56	1.30	1.70	3.04	2.21	-0.32	-2.06	5.22	5.10	0.21	1.10
UK	7.04	3.83	0.68	5.87	6.79	5.76	1.47	3.29	1.09	1.06	-0.17	0.19	5.22	-5.10	-0.21	-1.10

papers. After the exposure to UV/VIS radiation, with *ΔE**=15.37 coated papers have the highest colour difference value. Unprinted and printed samples on coated papers have a very high colour difference values upon the procedure of both thermal treatments, with the exception of black prints.

The highest colour differences values appeared in blue prints on coated paper during thermal accelerated ageing (*ΔE**= 46.25 at 160, 6 days).

The most significant changes on unprinted papers after ageing (natural or accelerated) can be observed in hue changes (+b), which makes papers yellowing. After thermal treatment at 130°C and the exposure to UV/VIS radiation yellow prints (C and U) show major changes toward to +*a**, red hue, while the exposure to thermal treatment at 160°C has a stronger impact toward to -*b**, blue hue. Red prints (C and U) exposed to UV/VIS radiation exhibit insignificant alterations in hue. The procedures of natural ageing, UV/VIS radiation and thermal ageing at 130°C result in the appearance of yellowness on the prints, whereas thermal ageing at 160°C causes significant changes in -*a**, green hue. Upon thermal ageing at 160°C, samples become greenish, while other conditions of the ageing process result in yellowish samples. Moreover, there is a distinction among two types of thermal ageing on blue prints (thermal ageing at 130°C induces significant changes in -*a**, green hue, whereas thermal ageing at 160°C causes alterations in +*b**, yellow hue). Changes in colour due to the process of natural ageing are negligible, whereas after UV/VIS radiation considerable changes in greenness occur on blue prints on coated paper. Black prints (C and U), regardless of the method of accelerated ageing, show the most considerable changes in lightness, with the exception of black print on uncoated paper after heating at 160°C, where the prints have changes as well in the hue (+b). These colorimetric results are in accordance with spectrophotometric measurements.

Changes in the visible reflectance spectra and in the total colour difference ΔE^* (CIE $L^*a^*b^*$) of all examined proved the most significant damage of blue prints on coated paper, especially upon heat treatment. After six days, the maximum relative reflectance difference for blue prints is approximately 80% at wavelength of 430nm for coated paper, and ΔE^* is 46,25 (160°C) and 35,62 (130°C).

Infrared beams can penetrate into almost all coatings, so the reflectance spectrum of coated paper is usually a composite spectrum of the substrate (paper) and the coating. Consequently, if a coating medium or a pigment is to be identified, its spectrum must be different from that of the paper. Figure 9 shows the IR spectra of coated samples printed with blue ink since the most significant changes in visible reflectance spectra and in the total colour difference occurred in those samples.

Kaolin showed up clearly (Fig 9.), with several peaks (1160cm^{-1}, 912cm^{-1},785cm^{-1}, 695cm^{-1}), and the biggest changes after the ageing process occurred in the 1160cm^{-1} peak (significant drop in the absorbance for all samples which underwent thermal ageing and UV/VIS radiation). Except for kaolin a major difference occurred with the peak at 730cm^{-1} which we believe is associated with the blue pigment.

It is evident that IR spectra of coated samples printed with Black ink before and after ageing (Fig. 10) confirm the stability of black pigment. There are no changes in the chemical composition caused by accelerated and natural ageing.

From these results it is obvious that the use of ATR-IR for pigment identification is very limited. This can be explained by the fact that many pigments have high refractive indices (1.9-3.1), so they absorb in the low-wavenumber end of the region (550-230 cm^{-1}). In addition, intense absorptions of other paint components, most importantly organic binder (starch and proteins) in the mid-IR region, complicate the identification of pigments.

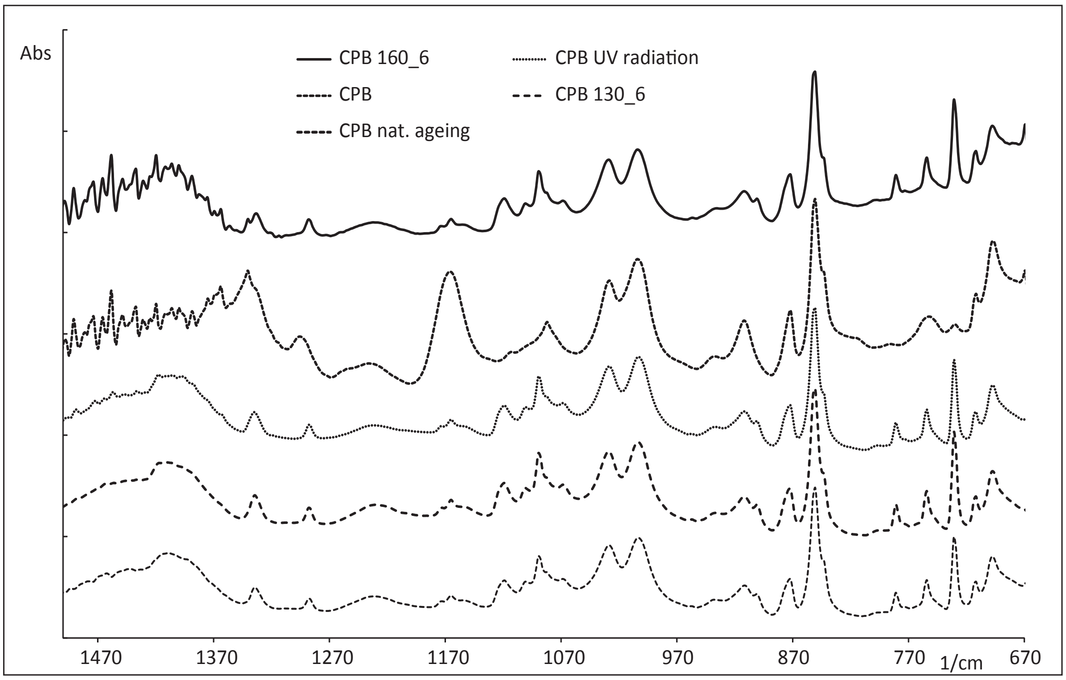


Figure 9. IR spectra of coated samples printed with Process Blue ink before and after ageing (spectra are shifted in absorbance for clarity)

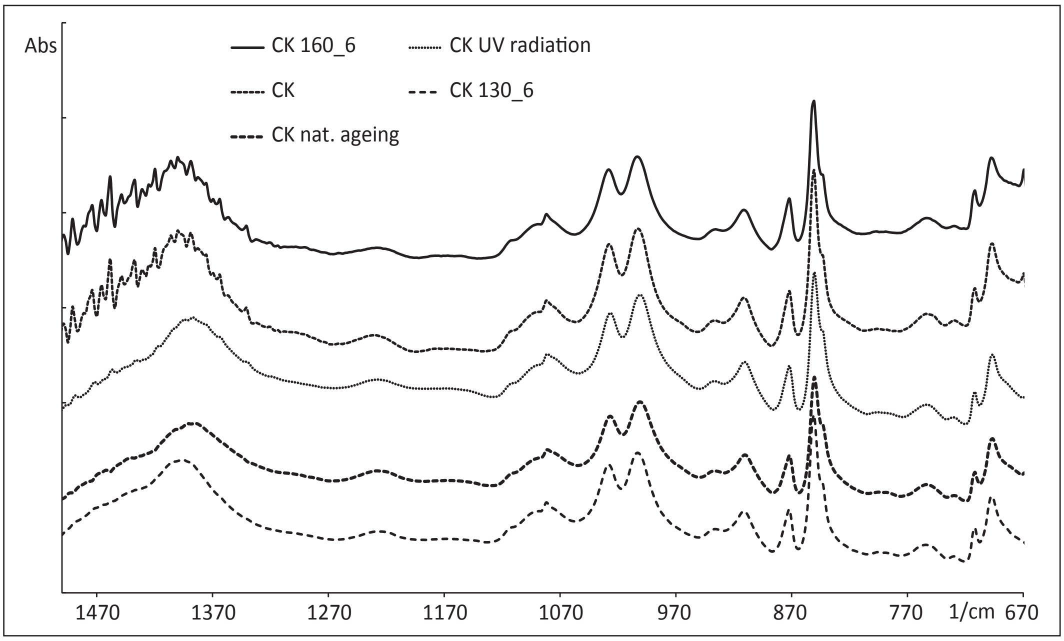


Figure 10. IR spectra of coated samples printed with Black ink before and after ageing (spectra are shifted in absorbance for clarity)

4. Conclusion

This research has brought about several conclusions:

- after natural and accelerated ageing both unprinted papers (C and U) demonstrate significant optical deterioration in the blue part of the electromagnetic spectrum, while after thermal accelerated ageing the optical degradation occurred whole observed in the red part of spectra.
- in respect of brightness, which is the optical parameter for paper quality, one can observe exponential dependence on the time period, the exponent of which differs according to the ageing conditions (temperature and RH) and the substrate used. The study of this dependence has introduced a new field for subsequent research.
- effect of optical deterioration and colour difference values is higher for coated paper and prints on coated papers.
- coated and uncoated papers as well as all chromatic prints, regardless of the method of ageing, show the significant changes in hue, while differences in lightness are minor.
- from reflectance measurements and colour difference values it can be concluded that influences of natural ageing and UV/VIS radiation on prints are very similar and negligible. The highest optical deterioration of all examined sampled were detected after thermal accelerated ageing at 160 °C.
- a major optical change appears in the Process blue pigment, while the most stable pigments are Red 032 and Yellow.
- after natural and artificial ageing, black prints (C and U) demonstrate significant changes in the lightness, while the differences in the hue are minimal.
- changes in the chemical composition of prints caused by accelerated ageing by means of FTIR spectroscopy were not perceived due to the physical limitations of ATR

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