

# The effect of carbonyl components of printing substrates on the durability of UV thermochromic prints

## ABSTRACT

*Special effects on a printed product can increase consumer's interest in a product and therefore lead to higher revenues in consumer industries. For that purpose, graphic industry introduced various technologies and materials that would have such an effect and enhance consumer-goods interaction. One of those solutions is thermochromic printing. Although bringing added value, thermochromic inks have some challenges in application due to their higher sensitivity, especially when exposed to UV light. Therefore, the goal of this work is to determine whether amount of the UV radiation during curing of UV thermochromic prints, as well as exposure after printing would degrade the thermochromic print on various substrates. UV thermochromic prints were made on three different paper printing substrates using the screen printing method and dried in a laboratory controlled unit. Synthetic paper, recycled paper containing 100 % recycled cellulose fibers and bulky voluminous paper were used as printing substrates. During the UV curing of the ink, no photooxidation of the prints occurred. Additional exposure of the prints to UV radiation (after the ink has hardened) leads to their photooxidative degradation, i.e., a change in the initial color. The obtained colour difference ( $\Delta E$ ) is increasing with the increase of the irradiation amount. The highest colour difference is on the synthetic paper while prints on two other substrates are more resistant to UV light. A print on synthetic paper photooxidizes the fastest due to the presence of most carbonyl groups in it. Generated free radicals promote the instability of prints on synthetic paper. Research has proven that when using synthetic paper or substrates with similar characteristics, care should be taken to accurately determine the amount of UV energy required for curing in order to prevent photodegradation of the ink. The results also show that to explain any degradation of prints, the chemical components of the paper needs to be taken into account, which is rarely done.*

## KEY WORDS

Photooxidation, screen printing, thermooxidation, thermochromic ink

Mirela Rožić<sup>1</sup>   
Tomislav Cigula<sup>1</sup>   
Snežana Miljanić<sup>2</sup> 

<sup>1</sup> University of Zagreb, Faculty of Graphic Arts, Zagreb, Croatia

<sup>2</sup> University of Zagreb, Faculty of Science, Department of Chemistry, Zagreb, Croatia

Corresponding author:

Mirela Rožić

e-mail: [mirela.rozic@grf.unizg.hr](mailto:mirela.rozic@grf.unizg.hr)

First received: 5.3.2023.

Revised: 29.4.2023.

Accepted: 5.5.2023.

## Introduction

Printing inks are colored complex mixtures, liquids or pastes consisting mainly of colorants (pigments or dyes), binders (resins), solvents (organic or aqueous-based) and additives (chelates, antioxidants, surfactants, biocides, etc.). Their composition and physical properties

differ mainly with respect to the printing process for which they are intended. When applied to a substrate, the printing inks must change to a solid state. The inks must dry as quickly as possible and can be dried physically (evaporation) and chemically (oxidation, radiation-induced curing) or by a combination of both (White & LeBlanc 1999; Leach & Pierce, 2007).

Radiation-induced curing includes ultraviolet radiation, infrared radiation, electron beams, microwaves, and radio frequency. Ultraviolet (UV) inks are hardened by UV radiation. UV curing (ultraviolet curing) is a process that uses ultraviolet light to initiate a photochemical reaction that creates a cross-linked polymer structure. It can be used in flexographic, offset, screen printing and pad printing processes. UV curing is used in the finishing of instruments (guitars, violins, etc.), the production of billiard cues and other wooden objects. UV printing is possible on different substrates such as plastic, paper, canvas, glass, metal, films and many other materials. The main advantage of curing with UV light is the speed of production, i.e., there is no need for drying time between printing and finishing. Speeding up drying can reduce defects and errors by reducing the amount of time the ink spends wet. This can increase the quality of the finished product (Leach & Pierce, 2007; Robert, 2015; MacLaren & White, 2003).

Thermochromic printing inks change color under the influence of heat (Civan et al., 2020). Instead of classic pigments, they contain microencapsulated leuco dye-developer-solvent systems. They can also contain a mixture of classic pigment and microcapsules. The thermochromic effect is caused by the formation of a leuco-dye developer complex inside the microcapsules. The microcapsules typically have an average particle size of a few micrometers (Phillips, 2000; Ma et al., 2020; Panák et al., 2017).

Many thermochromic dyes are of the spiro lactone type. A common example is crystal violet lactone (CVL). The thermochromic mixture is about 75–95 mol % solvent, the solvent melting point largely controls that of the mixture. Long-chain alkyl alcohols are most often used as solvents. Microcapsules are dispersed in a vehicle. The most widely used system for microencapsulation of thermochromic inks involves urea, melamine, melamine-formaldehyde, gelatine-gum arabic and epoxy resins (MacLaren & White, 2003; Panák et al., 2017).

The thermochromic inks communicate with the customer by changing appearance as the ambient temperature changes, providing additional information. On the other hand, their composition with microcapsules makes them sensitive in application and use. The microencapsulated thermochromic pigments usually have medium particle size of a few micrometres, which is about ten times larger than the particle size of conventional pigment particles.

In this paper, prints of thermochromic ink that hardens with UV radiation on three different paper substrates were analyzed. The aim of the work was to determine whether paper components affect the UV stability of ink prints.

## Materials and Methods

### Printing substrates, printing ink and printing trials

For this study, three different paper substrates were used for printing with UV curable thermochromic screen printing ink: synthetic paper (Yupo, 73 g/m<sup>2</sup>) extruded from polypropylene pellets, recycled paper (Mondi, 80 g/m<sup>2</sup>) containing 100 % recycled cellulose fibres, bulky voluminous paper (Munken Print White, 80 g/m<sup>2</sup>), which contains woodfree pulp and more than 10 % groundwood pulp.

A leuco dye based, UV curable screen printing ink (Chromatic Technologies, Inc.) with an activation temperature of 31°C (according to the manufacturer) was used to print the paper samples. Below the activation temperature, the thermochromic UV curable screen printing ink was purple in colour; above the activation temperature, the printing ink was pink. The ink was printed using a semi-automated (squeegee motion) screen printing device (Holzschuher K.G., Wuppertal, Germany) with a 60/64 screen printing mesh. The printed samples were dried under UV irradiation (30 W/cm) using the Technigraf Aktiprint L 10-1 device. All samples were printed in solid colour. The samples were denominated as: B – print on bulky paper, R – print on recycled paper, S – print on synthetic paper.

For the determination of UV light degradation, the dried samples were further exposed to UV radiation using the Technigraf Aktiprint L 10-1 device for defined energy in of 1–5 J/cm<sup>2</sup>. The energy was determined using the Technigraf UV-Integrator which measures in the wavelengths between 320–420 nm.

### Kappa number of bulky paper

The lignin content in bulky paper was determined by the method Kappa pulp number, Test method TAPPI/ANSI T 236 om-22 TAPPI/ANSI T 236 om-22. The kappa number is a key test method for determining the lignin content remaining in a sample. The kappa number is based on the reaction of a strong oxidizing chemical, potassium permanganate, with lignin, as well as small amounts of other organic impurities remaining in the pulp. The percentage of lignin in a sample whose kappa number is determined by the procedure in the specified standard test method is approximately determined using the following equation:

$$\text{Lignin level (\%)} = \text{Kappa number} \times 0.13$$

### Infrared (IR) spectra

To analyze the printing substrate composition, infrared (IR) spectra of the papers were recorded using

the attenuated total reflectance (ATR) technique. A Bruker Equinox 55 interferometer, equipped with a Pike Technologies MIRacle ATR holder with a reflection crystal made of diamond and ZnSe, was used to acquire the ATR spectra. The spectra were measured in the 4000-600  $\text{cm}^{-1}$  spectral range at a resolution of 4  $\text{cm}^{-1}$ , averaged over 32 scans. The raw ATR spectra were corrected using the extended ATR correction algorithm.

## Scanning electron microscopy (SEM) of prints

JSM-7000F, thermal field emission scanning electron microscope (FE SEM) manufactured by Jeol Ltd., was used to assess print's surface. FE SEM was coupled with EDS/INCA 350 (energy dispersive X-ray analyzer) manufactured by Oxford Instruments Ltd. The prints inspected by FE SEM were coated with a conductive layer.

## Colour measurement

In order to determine the hysteresis curves, the prints were heated and then cooled on a thermo-regulating metal plate in the temperature interval in which they change color: from 22.5 °C to 30 °C. Measurements of colourimetric CIE Lab values  $L^*$ ,  $a^*$ ,  $b^*$  on printed paper substrates were performed using SpectroDens (manufacturer Techkon).

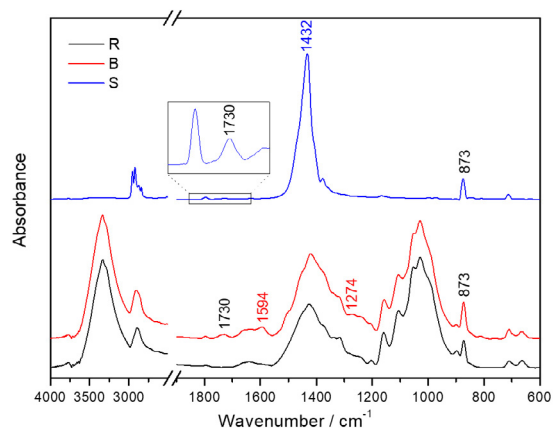
Colorimetric values are the result of averaging 20 measurements for each parameter. Color differences between non-irradiated and UV-irradiated samples and samples at different temperatures were calculated using the  $\Delta E_{\text{Lab}}$  equation (Mokrzycki & Tatol, 2011).

## Results and discussion

### The IR analysis of the printing substrate composition

The qualitative composition of the papers was analyzed by means of the IR spectroscopy (Figure 1). The ATR spectra of recycled and bulky papers corresponded mostly to cellulose, indicated by the strong broad bands of the O-H stretching (3330  $\text{cm}^{-1}$ ), COH and HCC deformation (1500–1300  $\text{cm}^{-1}$ ), C-C and C-O stretching (1160–950  $\text{cm}^{-1}$ ) as well as COC and CCO deformation (899  $\text{cm}^{-1}$ ) of the glucopyranose ring (Proniewicz et al., 2001). Beside the cellulose bands, the phenyl ring vibrational bands (1594 and 1274  $\text{cm}^{-1}$ ) were observed in the ATR spectrum of bulky paper, pointing to presence of lignin in its structure (Derkacheva & Sukhov, 2008). Unlike the cellulose-based papers, the intense band (1432  $\text{cm}^{-1}$ ) observed in the ATR spectrum of synthetic paper, was assigned to the methyl and methylene deformations, confirming that the synthetic printing substrate was mainly

composed of polypropylene. In addition, in the spectra of all the studied papers, a moderate band at 873  $\text{cm}^{-1}$  was observed and associated with calcium carbonate. Interestingly, a weak band (1730  $\text{cm}^{-1}$ ) attributed to the carbonyl stretching was observed in the ATR spectra of bulky and synthetic paper, but not assigned to any of the main constituents of the printing substrates.



» **Figure 1:** ATR spectra of the paper substrates; S-synthetic, B-bulky, R-recycled paper

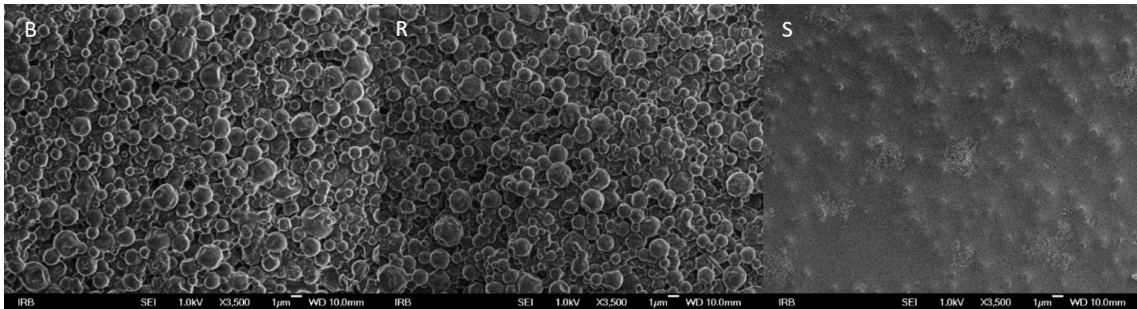
### Scanning electron microscopy (SEM) of prints

The binder in the tested ink is polyurethane acrylate containing chromophoric C=O and N-H groups (Vukoje et al., 2018). It is evident from the SEM images (Figure 2) that the binder was almost completely absorbed into the structure of recycled and bulky paper, and almost not at all into the structure of synthetic paper. SEM analysis shows that the microcapsules and the binder did not degrade (photooxidized) under the influence of UV light used to polymerize the prints after printing.

After coating the ink on the paper, the air inside the system should leave the ink film while the viscosity is still low enough. The atomized air in the system that remains in the system is called microfoam. Such microfoams are visible in the SEM image of the print on the synthetic paper.

### Hysteresis

The color change from purple to pink on all prints (Figure 3) occurs between temperatures of 22.5 °C and 28 °C which is below the temperature specified by the manufacturer (31 °C). By heating the print, the solvent inside the microcapsules dissolves. The blue-colored complexes between the dye and the developer inside the microcapsules are separated under the influence of the solvent, the blue color changes to colorless. Only the pink color of the classic pigment remains. At temperatures below the melting point of the solvent, the classic pink pigment and



» **Figure 2:** SEM micrograph of UV print on bulky (B), recycled (R) and synthetic (S) paper under magnifications of 3500x

blue colored microcapsules give a purple color. Based on the colorimetric values of the prints from Figure 3, it is evident that all the prints are stable to the diffusion of the components from the microcapsules. The heating curves are equal to the cooling curves. The photos show prints taken after a warm-up cycle.

If the initial  $-b^*$  value is taken as a measure of the concentration of blue-colored microcapsules, and the  $+a^*$  value is taken as a measure of the concentration of pink pigment, then based on  $a^*$  and  $b^*$  values it can be concluded from Figure 3 that the highest concentration of microcapsules is on recycled paper (the smallest  $b^*$  value), and the pink pigments together with binder is on synthetic paper (the largest  $a^*$  value). The reason for the highest concentration of pigments and binder on the surface of the synthetic paper is the weak absorption of the binder into the structure of the synthetic paper.

According to the literature, microcapsules are polar. They contain hydroxyl and amino groups. In the tested UV ink, they are contained in a polyurethane acrylate binder that contains C=O and NH groups. Hydroxyl and amino groups of microcapsules enter hydrogen interactions with NH groups of binders and dipole-dipole interactions with carbonyl groups of binders. Therefore, the same concentration of microcapsules in the binder can be assumed before application to the paper. Microcapsules are adsorbed on paper surfaces as part of the binder. If the binder has a higher affinity to the paper substrate, more microcapsules will be adsorbed.

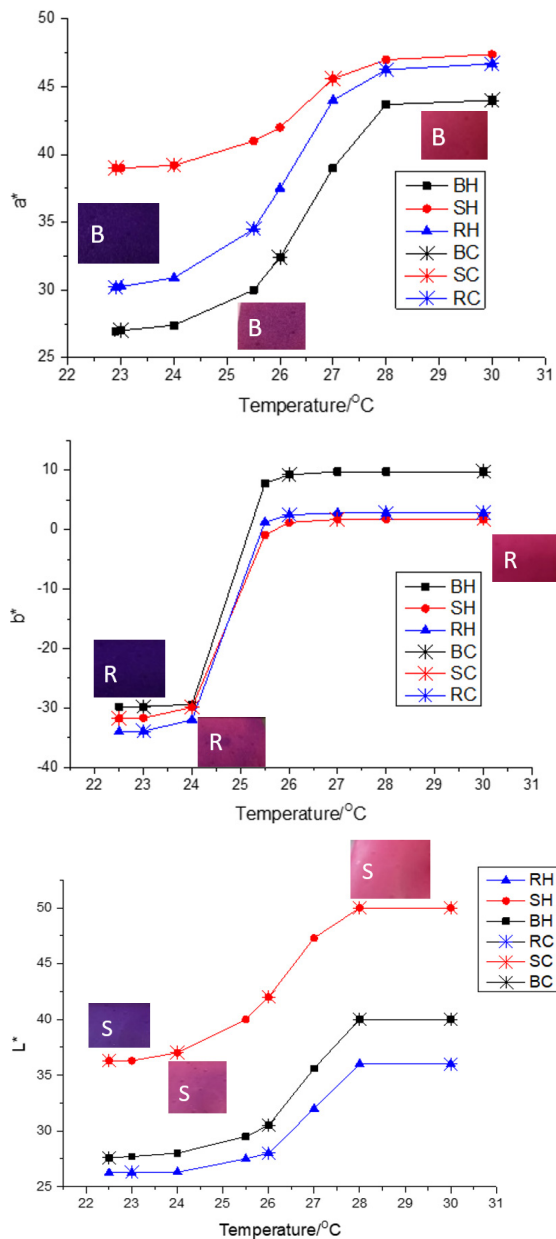
According to the obtained colorimetric values, the most microcapsules were adsorbed on the surface of recycled paper, and somewhat less on the surface of bulky paper. Bulky paper contains 1.17 % lignin, which reduces the hydrophilicity of cellulose and fillers. However, this proportion is quite small, so it causes only a slight decrease in the sorption of binders and microcapsules on its surface. The SEM images show that the entire surface of the recycled and bulky paper has been used as far as microcapsules are concerned, while the synthetic paper has not. Synthetic paper contains 44.5 wt% of calcium carbonate (Rožić et al.,

2020). Ion-dipole interactions are stronger compared to hydrogens and dipole-dipole. Despite the stronger attractive forces, the sorption of microcapsules on synthetic paper should be significantly lower considering its composition. Microcapsules are adsorbed in places rich in calcium carbonate, and the binder is adsorbed in these areas by ion-dipole interactions. Microcapsules are densely packed when adsorbed on areas rich in calcium carbonate. So, they are not in one layer but in several layers. Therefore, the  $b^*$  values of prints on synthetic paper are similar to prints on recycled paper containing 100% active polar and ionic components.

At 22.5 °C, the lightest print is on synthetic paper (the highest  $L^*$  value) due to the highest concentration of pink pigment on its surface. The darkest print is on recycled paper due to the highest concentration of blue colored microcapsules.

At a temperature of 25.5 °C,  $b^*$  values take on positive values.  $b^*$  values move into the yellow area. Print on bulky paper is the yellowest ( $b^*=9.78$ ), followed by recycled ( $b^*=2.82$ ) and synthetic paper ( $b^*=1.75$ ). The  $b^*$  value of the bulky paper (Rožić et al., 2020) itself cannot cause such a large  $b^*$  value of the print on the bulky paper at the end of heating. It is possible that some component from the ink was adsorbed on the surface of the bulky paper in a larger amount than on the other substrates, which causes a positive  $b^*$  value. Some possible additives in UV ink include adhesion promoters, pigment dispersing agents, and gloss promoters.

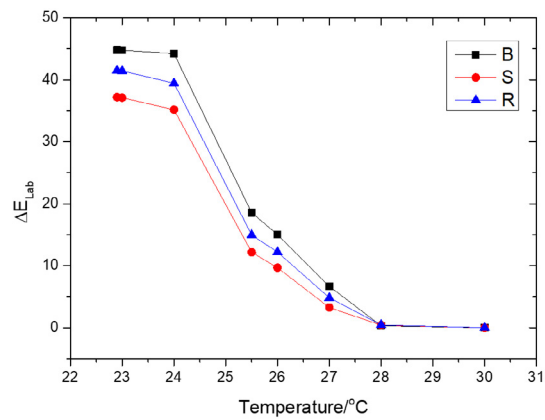
Based on the chemical structure, it can be assumed that the increased  $b^*$  values are caused by gloss enhancers such as hydroxypropyl methylcellulose and lecithin (Wazeska et al., 2002). Hydroxypropyl methylcellulose and lecithin are weakly polar molecules that may have a greater affinity for lignin in bulky paper. The dispersion, polar and dipole components of hydroxypropyl methylcellulose and lecithin are similar to those of lignin because their chemical structures are also similar.



» **Figure 3:**  $L^*$ ,  $a^*$ ,  $b^*$  values of prints depending on heating and cooling temperatures; H=heating, C=cooling- Photographs of prints taken at temperatures of 22 °C, 24.5 °C and 28 °C; S=synthetic, B-bulky, R-recycled paper

Figure 4 shows the color contrasts of the prints at different temperatures with respect to the colorimetric values of the prints at 30 °C as reference values. Color contrasts are greatest up to a temperature of 24 °C, which is to be expected, because prints are the most purple at those temperatures. They are the greatest for print on bulky paper. The smallest color contrast on synthetic paper is a consequence of its insufficient porosity. This is why it already has a high concentration of pink pigment on the surface and approximately the same concentration of blue-colored microcapsules as other papers. As far as savings or green printing is concerned, it is favor-

able. However, because of this, it gives slightly less color contrast compared to other prints, but the color contrast is clearly visible in any case. Color contrasts decrease in the temperature range from 22 °C to 28 °C.



» **Figure 4:** Color contrasts of thermochromic offset prints with respect to the colorimetric values of the prints at 30 °C; S=synthetic, B-bulky, R-recycled paper

## Photooxidation of prints

Although the prints proved to be stable to UV radiation during printing, the research involved additional UV light (after drying) since these inks are sensitive to UV light. The results of the colour measurement of UV exposed prints is presented in Table 1.

**Table 1**

Colour difference between initial and samples exposed to UV energy

Sample	Irradiation (J/cm <sup>2</sup> )	ΔE lab
B	0	0
	1	2.24
	3	4.28
	5	5.39
R	0	0
	1	2.03
	3	3.87
	5	4.78
S	0	0
	1	5.89
	3	8.88
	5	9.24

The highest colour difference can be seen on synthetic paper where even lowest additional irradiation energy causes significant colour change. Color changes visible to the eye are greater or equal to 5 (ΔE). On the other substrates it is visible that even the highest additional irradiation did not cause colour change obtained on synthetic paper after 1 J/cm<sup>2</sup>.

The results of the research by Rožić & Vukoje (2018) show the heterogeneous nature of the photooxidative degradation of thermochromic UV prints.

The UV ink microcapsules and the polar polymer binder can form strong hydrogen and dipole-dipole bonds between the NH groups of the binder and the –OH and –NH (–NH<sub>2</sub>) groups of the microcapsules. The number of available chromophoric groups of binders and microcapsules is thus reduced. The areas where the microcapsules and the binder are connected by hydrogen bonds are stable. Only the binder on the surface of the microcapsules and possibly the pigment are photodegraded. However, since polyurethane acrylate contains much more polar groups compared to microcapsule materials, the number of bonds between microcapsules and the binder is small. This means that the binder on the surface of the microcapsules still has a high concentration of chromophore groups. Since the polypropylene areas of synthetic paper is less absorbent than the others used in this study, the number of surface chromophore groups is the highest on the print on its surface but not too much compared to the surfaces of prints on bulky and recycled paper. Prints on bulky and recycled paper have a slightly lower surface number of carbonyl and amino groups. Bulky paper has the lowest chromophore groups concentration. Therefore, a print on bulky paper should degrade the least, and it degrades faster than a print on recycled paper. According to the results of the IR analysis, bulky and synthetic papers contain carbonyl groups that cannot be linked to their other ingredients.

Carbonyl compounds can be added to polymers as additives that accelerate photooxidation degradation. Such an initiator is, for example, benzophenone (Geuskens & Kabamba, 1987). Besides ketones, quinines and peroxides are also initiators of photodegradation reactions of organic compounds. All of them absorb light up to about 380 nm, which causes their excitation or cleavage into radicals (Chew, Gan & Scott, 1977). The influence of low-molecular organic compounds such as benzophenone, anthraquinone and benzoyl peroxide on the photodegradation of polystyrene was investigated in the work of Yousif & Haddad (2013). The results show that these additives accelerate and increase the photooxidation of polystyrene. The presence of carbonyl groups as defects in the structure of poly(p-phenylenevinylene) (PPV) is widely known.

In the paper (Papadimitrakopoulos et al., 1994) it was proved that carbonyl defects can be introduced thermally and photochemically. Although PPV is quite thermally stable, its defects containing hydroxyl groups are very sensitive to reaction with traces of oxygen at elevated temperatures resulting in the formation of ketone-based carbonyl impurities. Photooxidation of PPV involves cleavage of the vinyl double bond and formation of terminal aldehyde groups.

In the work (Kunwong, Sumanochitraporn & Kaewpirom, 2011), 2,2-dimethoxy-2-phenyl acetophenone was used as a photoinitiator in the polymerization reaction of a UV-curing coating based on urethane acrylate oligomer. Accordingly, carbonyl compounds are also used as photoinitiators in ink UV curing.

The reason for the higher rate of photooxidation of prints on synthetic paper should be sought in the paper structure itself. As mentioned, carbonyl compounds are added to polymers to accelerate photooxidation. According to IR analysis, certain carbonyl compounds are also found in bulky paper, so this is the reason for its faster decomposition even though it contains the least binders.

## Conclusions

This research was conducted to determine the influence of the substrate on the UV stability of a thermochromic UV curing ink applied in the screen printing process. In addition, the goal is to determine the effect of higher exposure to UV light than is used to cure the ink film.

The research has proved that used substrate has significant influence on the thermochromic effect, but on the resistance to additional UV light exposure as well. Furthermore, when using synthetic paper, or substrate with similar characteristics one should take proper care to precisely determine UV energy amount needed for curing in order to prevent ink's photodegradation.

## Funding

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

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