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Colorimetric changes of thermochromic ink printed on smart textile materials exposed to different heat transfer methods

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ABSTRACT

This research aims to determine the influence of heat and printing substrate type on the colorimetric properties of the thermochromic ink printed on various textile materials while subjected to heating simulating realistic conditions of usage. The results of the research can be used as a recommendations for the development of a smart temperature indicators for textile packaging. Four specific groups of textile materials were used as printing substrates and magenta leuco thermochromic water-based screen printing ink (activation temperature 31°C) in order to analize resulting colorimetric properties. Experiment based on analysis of resulting color differences confirmed that the screen thread count influences the rate of the material color change. Namely, the higher the thread count the faster the color change i.e. the sample returns faster from the discolored to the colored state. It was also confirmed that the lower the fabric weight of the material is, the sample returns faster from discolored to the colored state. In addition, this article presents a comparison of the contact and contactless method of sample heating at the same temperature. It was shown that the samples were cooled slower and consequently changed the colorimetric values after the contact method.

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printing, thermochromic, ink, smart textiles, materials properties

1. INTRODUCTION

The most commonly accepted definition of smart materials is that such materials can be activated by physical, chemical or mechanical environmental influences and react to their surroundings predictably and beneficially. Some of the most important smart materials are piezoelectric, shape memory, chromatic and magnetic-rheological [1]. Chromatic materials are materials that change color under the influence of external factors (temperature, light, the presence of solvents, pressure, electricity, friction, etc.) [2]. Material that changes color due to the temperature change is called thermochromic material. Thermochromism founds its application in the textile industry, but textiles produced from thermochromic fibers have not appeared on the market yet. So far the thermochromic effect on textiles is mainly achieved by screen printing with thermochromic inks [3].

In this article, the term activation temperature will be used to define the temperature at which the coloration/discoloration process occurs. Leuco dye-based thermochromic ink is a thermochromic composite that consists a color-former and a color-developer dissolved in a solvent. The composite is microencapsulated in a protective coating to protect the content from the environment [4]. The color-former has two forms in dependence of the temperature – colored and colorless form. In a non-heated state, the composite remains in solid form, and the color-former adopts its colored form. In heated state, the solvent melts and the interaction between the solvent and the color-former destroy the composite, thus causing the color-former to adopt its colorless form. The activation temperature is defined by the temperature at which the solvent changes from solid to liquid state [5].

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Thermochromic leuco dyes are available with different activation temperatures, from -15 to 65 °C. However, the majority of applications are limited to the three standard temperature ranges, cold (~ 10 °C), human body temperature (~ 31 °C) and warm (~ 43 °C) [6]. Usually, the leuco dyes are colored beneath activation temperatures and become discolored or transparent above the activation temperature. All the main types of inks, both water-based and UV-based, are available for paper, plastic and textile applications [7].

Although the colorimetric characteristics of thermochromic inks on textiles are significant from the perspective of the application, the influence of temperature on colorimetric values of thermochromic inks has not been studied to a great extent. Friškovec et al. [8] confirmed that thermochromic inks have poor stability against light and high temperatures. They used the white inked aluminum plate as a substrate for heating at the high temperatures and paper substrate for exposure to light. The stability tests showed that the thermochromic prints were affected by UV light and the temperatures well above 150 °C. Kulčar et al. [9] after tests on paper samples, noted that the discoloration process was not completelly finished at the highest temperatures, even far beyond the activation temperature. The samples retained a bright pastel color from the ink on the sample, and this effect increased with the increase of the ink layer thickness. Kulčar et al. [10] also shown that the color values of thermochromic samples are the function of the temperature. They concluded that the thermochromic samples does not depend only on temperature, but also on its thermal history, so the thermochromic materials belong to several physical systems with a hysteresis. In all this research samples were printed with screen printing technique. Chowdhury et al. [11] applied thermochromic inks to a conductive cotton fabric made of nichrome/cotton core-spun yarn and show the temperature dependence of colorimetric properties on the thermochromic inks. The heat generation and the temperature rise were controlled by the voltage applied. The research related to colorimetric characteristics of thermochromic inks were normally carried out in a controlled manner by precisely varying temperature through specially designed heating and cooling systems. Generally, the samples were heated and cooled on plate materials [6,8,9,10].

In this article, we developed a system for testing the temperature dependence of colorimetric properties based on multipurpose universal devices without specially designed heating and cooling systems. Since textile materials in real use may or may not have be in a direct contact with the heated body, the tests on the samples are carried out after the air heating process under controlled conditions without direct contact between the fabric and the heating surface. In this way, the real conditions in which the product finds itself in practical application are emulated. In this article, the thermochromic sample property will be examined as a function of the temperature in the cooling process, to show the dependence of the material type and the screen mesh selection during printing to achieve a quality print with desired characteristics.

2. MATERIAL AND METHOD

2.1 Materials

Four types of textile materials with dimensions 250 x 350 mm were used in this study. For each of the material samples, the following are determined: fabric weight (ISO 3801:1977) [12], number of threads per unit length (EN 1049-2:1993) [13], linear density of yarn removed from fabric (ISO 7211-5:1984) [14] and material composition (EN ISO 1833-1:2010) [15]. The characteristics of the samples are given in Table 1. In the printing process, commercial SFXC magenta ink (activation temperature 31°C) and acrylic based binder were used [16]. The ratio of the dye and binder was 1:1.

2.2 Color Measurement

Using the diffuse geometry colorimeter HP200 (D65/10°, measurement geometry d/8°), CIELAB values of thermochromic ink were determined over time. Figure 1 shows the experimental setup with the position of devices during the measurements. Color measurement was done in two experiments. In the first experiment samples were heated in laboratory drying oven COLO DRYS53A, while in another experiment the samples were heated using the heating press. After heating, the samples were placed on the Styrofoam. A temperature change of the material was recorded with a thermal imaging camera, and during the cooling process, the color change was measured with the HP200 spectrophotometer.

Table 1. Fabric weight, number of threads per unit length, the linear density of yarn removed from fabric and material composition of the samples

Textile material	Fabric weight [g/m2]	Number of threads per unit length [threads/cm]		The linear density of yarn removed from fabric [tex]		Material composition	
		Warp	Weft	Warp	Weft	Type	[%]
Sample 1	208	44	21	27.00 x 1	26.40 x 1	Cotton	100
Sample 2	123	23	16	30.20 x 1	29.20 x 1	Cotton, Polyester	98.60, 1.40
Sample 3	81	39	25	8.10 x 1	18.20 x 1	Cotton	100
Sample 4	120	52	28	13.60 x 1	12.60 x 1	Cotton, Polyester	14.40, 85.60
Standard	ISO 3801:1977	EN 1049-2:1993		ISO 7211-5:1984		EN ISO 1833-1:2010	

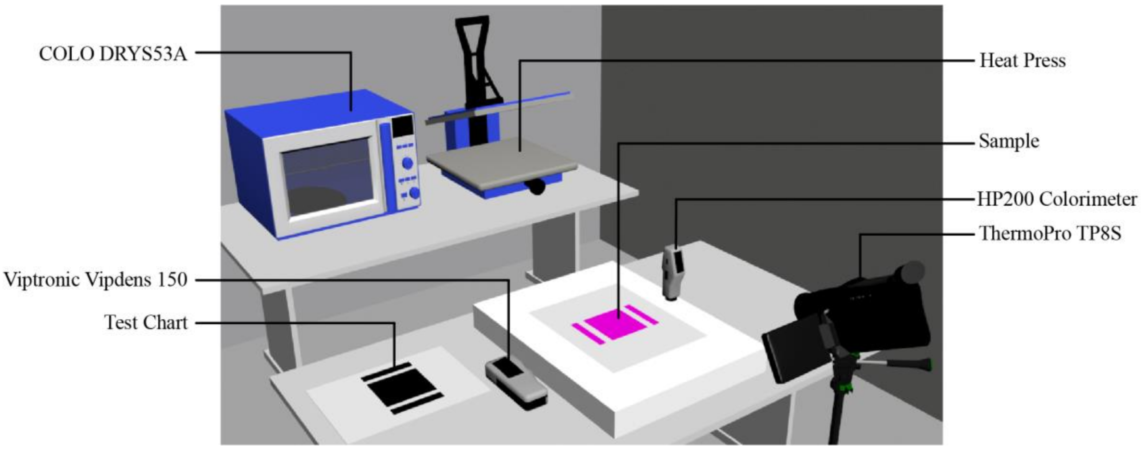

Figure 1. Experimental setup

2.3 Test Chart

The test chart (164 x 100 mm) consists of two 26 x 100 mm surfaces for color measurement, and 100 x 100 mm field for temperature analysis (Figure 1). It was developed on a glossy positive film ColorGate Screenfilm with photosensitive layer Sericol Dirasol 915, 50 lpi, at the angle of 90°. The film was illuminated using the Linotype Hell Linotronic 300 Red laser at its working temperature for 120 s, developed on the Esco Fot Gluns & Jensen machine with a process bath temperature of 32 °C for 30 s and fixed at 25 °C for 30 s. The shape of the raster dot on the film was ellipse. The concentration of the process bath was satisfactory as the regeneration was carried out on time. Minimum and maximum density values were measured on the film using the Viptronic Vipdens 150 densitometer. Obtained values of Dmin = 0.06 and Dmax = 2.70 confirmed that the film was well developed.

2.4 Screen Development

For the fabrication of the screen, silk net finishes (thick fibers), dimensions 500 x 700 mm and two different monofilament weave weights of 54 threads/cm and 120 threads/cm were used. The fabric was attached to the measuring aluminium frame size of 580 x 840 mm. The tensile force of the screen on the frame was 21 N/cm2 during the making of the screen. After screen mesh tightening, the tensile force was 18 N/cm2. The emulsion thickness was 0.30 mm for 54 threads/cm and 0.10 mm for 120 threads/cm, and it was exposed on a screen mesh through an indirect screening process and a drying time of 2 hours at 35 °C.

2.5 Printing Process

The samples were printed on screen printing machine S550. For the printing process, a neoprene-shaped squeegee edged at an angle of 45° was used, hardness 80A. The thickness of the squeegee was 5 mm, and the length 25 mm. The weight of the squeegee was 180 g every 100 mm in length, the total length of the blade was 255 mm. The printing speed was 150 mm/s, the return speed of the squeegee and the ink carrier was 150 mm/s, the distance of the exposed emulsion from the substrate was 4 mm, the distance of the ink application from the substrate 2 mm and the distance of the squeegee from the substrate was 2 mm. The ink (50 ml) and the binder (50 ml) were homogenized using the IKA KS 130 basic orbital shaker at a mixing rate of 400 rpm for 5 min. During the homogenization, printing and measuring process, the following ambient conditions were measured: temperature 22 ± 2 °C, pressure 101 ± 1 kPa and relative humidity 40 ± 2%. For these measurements, we used Extech RH520A.

2.6 Temperature Measurement

In order to measure the temperature values during the cooling of analyzed samples the IR ThermoPro TP8S camera was used. Selected camera has a microbolometric sensor with a resolution of 640 x 840 pixels and generates wavelengths in the range of 8 to 14 µm with a sensitivity of 0.08 at 30°C. The temperature measurement process takes place in such a way that the infrared sensors integrate electromagnetic IR radiation in the wavelength range of operation, and then generate the corresponding electrical signal [17]. The application of IR cameras in this experimental study is particularly useful because the emissivity coefficient of the tested materials, which is determined experimentally, is quite significant and ranges from 0.89 to 0.95. In order to increase the accuracy of the temperature measurement, the values of the relative air humidity, the ambient temperature and the distance between the IR sensor and the object of measurement should be entered as the input parameters in the IR camera. The distance between the IR sensor and the samples was 100 cm (Figure 1).

2.7 Ambient Conditions Measurement

In order to reduce the radiation from the additional sources in the surroundings, the temperature measurement should be performed in the dark environment [18]. Using the Eye-One Pro device (D65/10°, measuring geometry 45°/0°), the spectral curves and ambient lighting characteristics were measured (Figure 2). The intensity of light was 5.4 lux while the color temperature was 4594.50 K (corresponds to standard illuminant D50). In the visible spectrum, the light is almost uniform, which confirms that the conditions in which the measurement was performed were favourable and controlled.

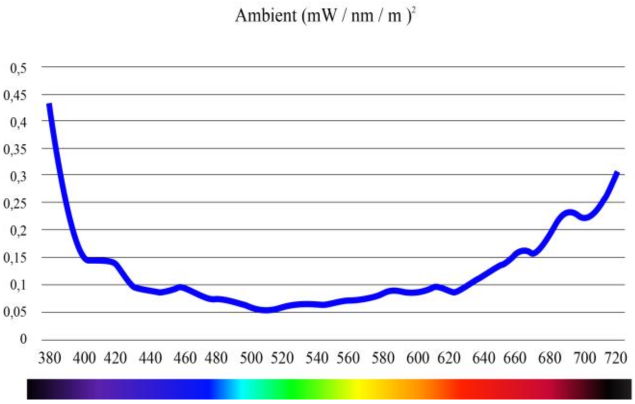

Figure 2. The spectral curve and ambient characteristics

3. RESULTS AND DISCUSSION

3.1 Thermovision Measurements

After heating the samples inside the COLO DRYS53A for 3 minutes at the temperature of 50 °C, the samples were pulled out and left to cool. It was noticed that after one minute of the cooling process, all the samples reached the ambient temperature. The surface temperature of the analyzed material was measured every 10 seconds, and the temperature was measured at the same time as color characteristics. Figure 3 gives an example of the thermal image.

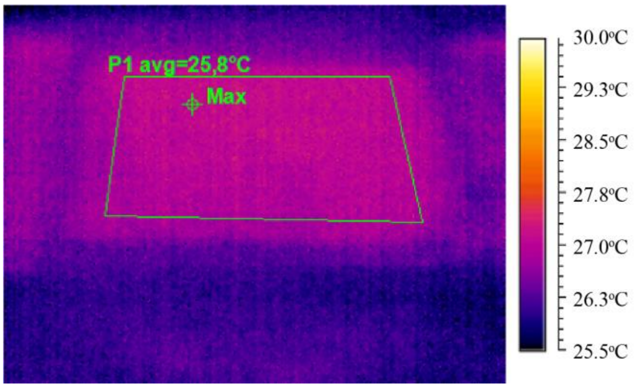

Figure 3. Thermal image of sample 3 printed using 54 threads/cm screen, second measurement, after 40s cooling

Figure 4 shows the dependence of time and temperature for the samples used in the study printed with different screens (differed by screen thread counts). The obtained results show that the cooling process of the printed samples is influenced by choice of a screen thread count, in such a way that the smaller screen thread count leads to the greater samples temperature retention time and, consequently, slower cooling. The cooling rate was slowing down when the screen mesh value decreases because of the larger openings that allow more ink to flow through. [19]. The selection of the screen thread count had the smallest effect on sample 3, where the temperature did not change significantly over time (Figure 4).

3.2 Spectrophotometric Measurements During Cooling of Heated Samples

After heating the samples for 3 minutes in a multi-purpose device, COLO DRYS53A at a temperature of 50 °C, the samples were cooled to ambient temperature (24 °C). During the cooling process, the samples were measured every 10 seconds. In order to minimize the effect of heat transfer from the sample to the measuring surface, the samples were placed on Styrofoam (10 cm thick). During the cooling, CIELAB values for all samples were measured. The results are shown in Figure 5. The same figure also showss color differences. The color difference was calculated using the CIEDE2000 formula [20]. In addition to the color coordinate values and color differences, the relationship between colorimetric values (lightness and chroma) and the cooling time was investigated using Pearson correlation (correlation coefficients (r) and the corresponding degrees of determination (r2) are presented in Table 2).

There was a strong, negative correlation between lightness and cooling time and a strong positive correlation between chroma and cooling time in cases of all four materials and both screen thread counts. This means that the color is gradually getting darker and more saturated with enhancing time interval after exposing samples to the heat. The high coefficients indicate that colorimetric values can be used with a high level of confidence to determine the time elapsed from the beginning of the cooling process. The higher correlation coefficients in the case of lightness indicate that the lightness value is a more precise parameter for the cooling time prediction than chroma. Furthermore, the extremely high correlation coefficient values in the case of sample 1 indicate that the confidence degree of prediction depends on the choice of material. The influence of material and screen thread count on lightness value was investigated with ANOVA analysis with repeated measures for intervals 10, 20, 30, 40, 50, 60 and 70 seconds after exposure to the heat source. Besides determined significant effect of time interval on lightness, $F= 319.34$, $p<0.0005$, multivariate partial eta squared = 0.99, it was confirmed that there a significant influence of the material type on behavior of lightness ($F= 319.34$, $p<0.0005$, multivariate partial eta squared = 0.99), and also of screen thread count ($F= 42.17$, $p<0.05$, multivariate partial eta squared = 0.79). The influence of the textile material type on the color difference change in relation to the first measured value during the cooling process for the tested screen thread count of 54 and 120 threads/cm are shown in Figure 6.

Table 2. Pearson's correlation coefficient (r) and the corresponding degree of determination (r2) between the lightness and time, as well as the chroma and time for all samples

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 1	Sample 2	Sample 3	Sample 4
Screen thread count (threads/cm)	r(r2) correlation L i T				r(r2) correlation C i T			
54	-0.95 (0.90)	-0.87 (0.75)	-0.93 (0.86)	-0.89 (0.79)	0.92 (0.85)	0.84 (0.70)	0.82 (0.68)	0.77 (0.60)
120	-0.94 (0.88)	-0.91 (0.83)	-0.86 (0.73)	-0.83 (0.69)	0.92 (0.84)	0.88 (0.77)	0.74 (0.54)	0.75 (0.57)

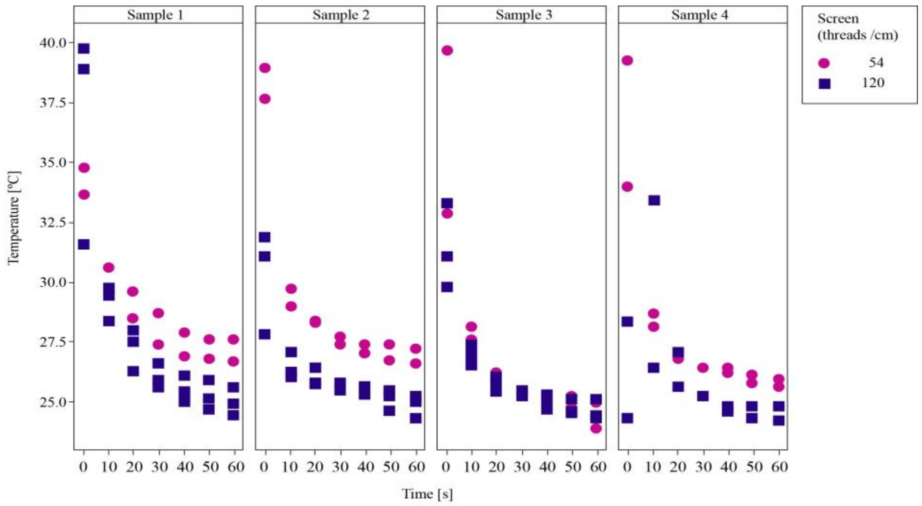

Figure 4. Time and temperature comparison of the samples printed with two different screens

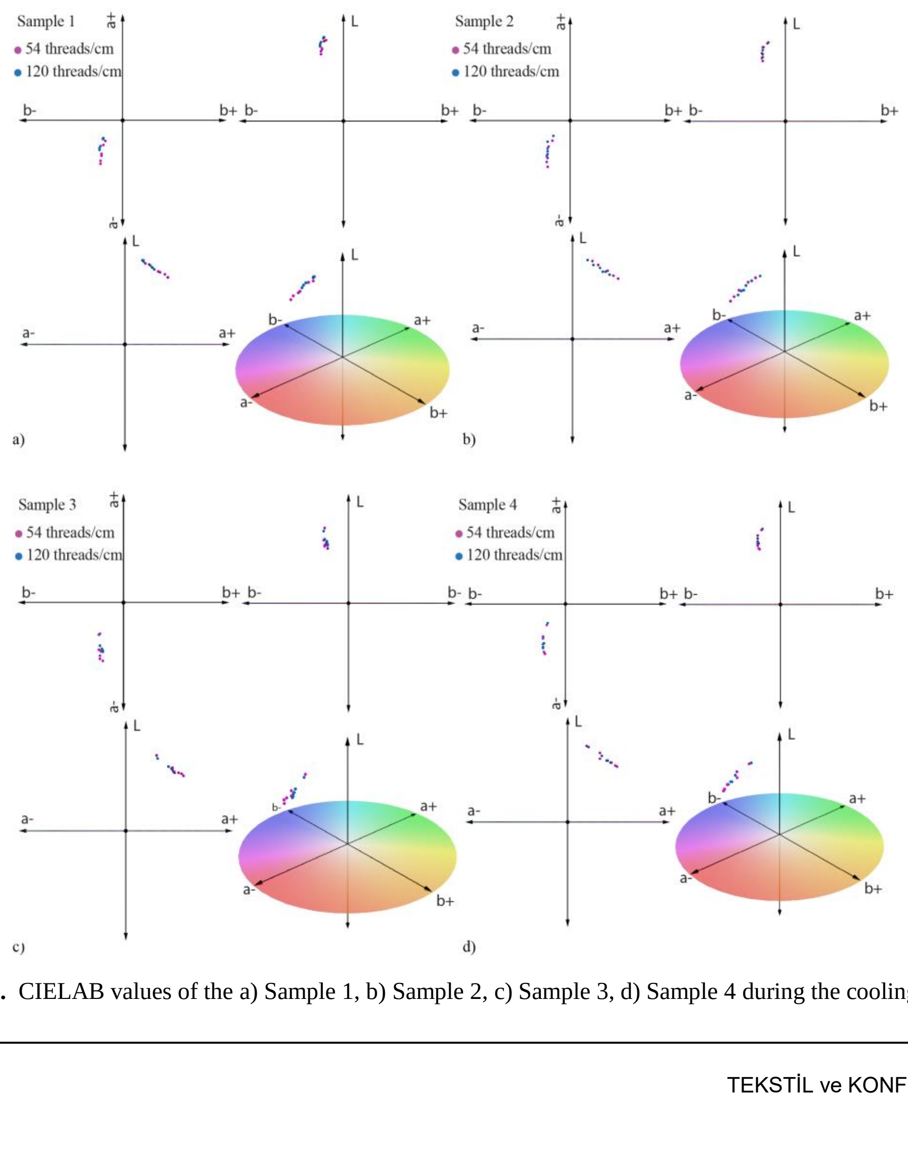

Figure 5. CIELAB values of the a) Sample 1, b) Sample 2, c) Sample 3, d) Sample 4 during the cooling process

After color differences analyzing, it was observed that sample 4 (54 threads/cm) and sample 2 (120 threads/cm) had the most significant color difference change compared to the first measured value. These fabrics are distinguished by the non-linearity of the color difference value between each subsequent measurement. In terms of the linearity of the changes over time, the lowest color difference between each subsequent measurement is characterized by sample 1 (54 threads/cm and 120 threads/cm), and at the same time these samples are characterized by the slightest change in the color difference and the smallest effect of the screen thread count on the color difference. With reducing the screen thread count, the color difference between each subsequent measurement is lower, but the color difference in relation to the first measured value is higher. Thus, for each fabric type, the color difference change in relation to the first measured value is higher for the screen thread count of 54 threads/cm.

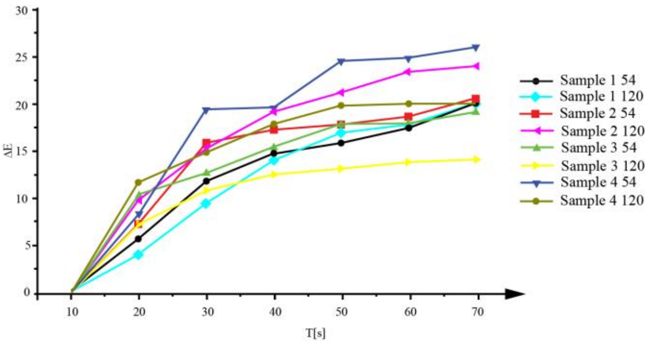

Figure 6. The color difference in relation to the first measured value for screens of 54 threads/cm and 120 threads/cm

In the analysis of color differences between each subsequent measurement, it was determined at which moment the most significant color change occurred, and then this change was slightly reduced until the ambient temperature was reached. For samples printed with a screen of 54 threads/cm, it was noted that the most significant change occurred in the first 30 seconds of measurement, excluding the cotton material with the smallest fabric weight at which the most significant color change occurred in the first 20 seconds of measuring. On samples with a screen 120 threads/cm, this change took place at 20 seconds of measurement, excluding the cotton material with the most significant fabric weight at which this change took place for 30 seconds of measurement. Compared to materials with polyester additives, cotton materials are characterized by higher absorption, resulting in a higher amount of thermochromic ink in the material and longer temperature retention. To determine how the type of material influences the change of thermochromic ink, four specific groups of materials applicable to various textile packaging were used in the experiment. In this way, we created the basis for precise temperature identification via thermochromic ink printed on various textile packaging materials. Based on this analysis, it has been confirmed that the screen thread count influences the change of the thermochromic ink on the material in such a way that the higher the screen thread count is, the faster the color change, meaning that the sample returns faster from the discolored to the colored state. The screen with the higher thread count transfers less ink, and therefore the material, will release the heat faster. With the higher screen thread count, screen the ink will return faster from the colored to the discolored state [21]. It has also been confirmed that the smaller the fabric weight of the material, the faster is the return from the discolored to the colored state. It was shown that depending on the fabric weight of the material, the heat changes was different. The material with a greater fabric weight had the ability to absorb a larger amount of ink, so it kept the heat for a longer period of time [22]. Higher volume of thermochromic ink absorbed in the fabric causes higher color changes in thermochromic effect as a result of higher concetration of thermochromic pigments inside the material. The liquid absorption of fabrics decreases with increasing warp and weft densities as the fabric structure become very compact and dense. The effect of fabric constructions without any hydrophilic finish on liquid or water absorption was studied by researchers [23, 24, 25].

To compare the contact and contactless heating method, the measurements were repeated under the same conditions on samples that were cooled after heating using the heating press. Color differences were estimated between the first measured color value and the consequent measurements. The relation between color difference and temperature for the samples printed with different screens and dried within the drying oven and using heat press are shown in Figure 7a and 7b, respectively.

By analyzing the relation of color difference and temperature, it is noticeable that the samples measured after heating with heat press linearly increased the color difference with decreasing the temperature because they maintain the heat within the material for a longer time. These results show that the heating method influences the samples’ cooling time. Hence, in order to create real conditions in which the smart textile packaging material could be found, it is necessary to use a device for contactless heating.

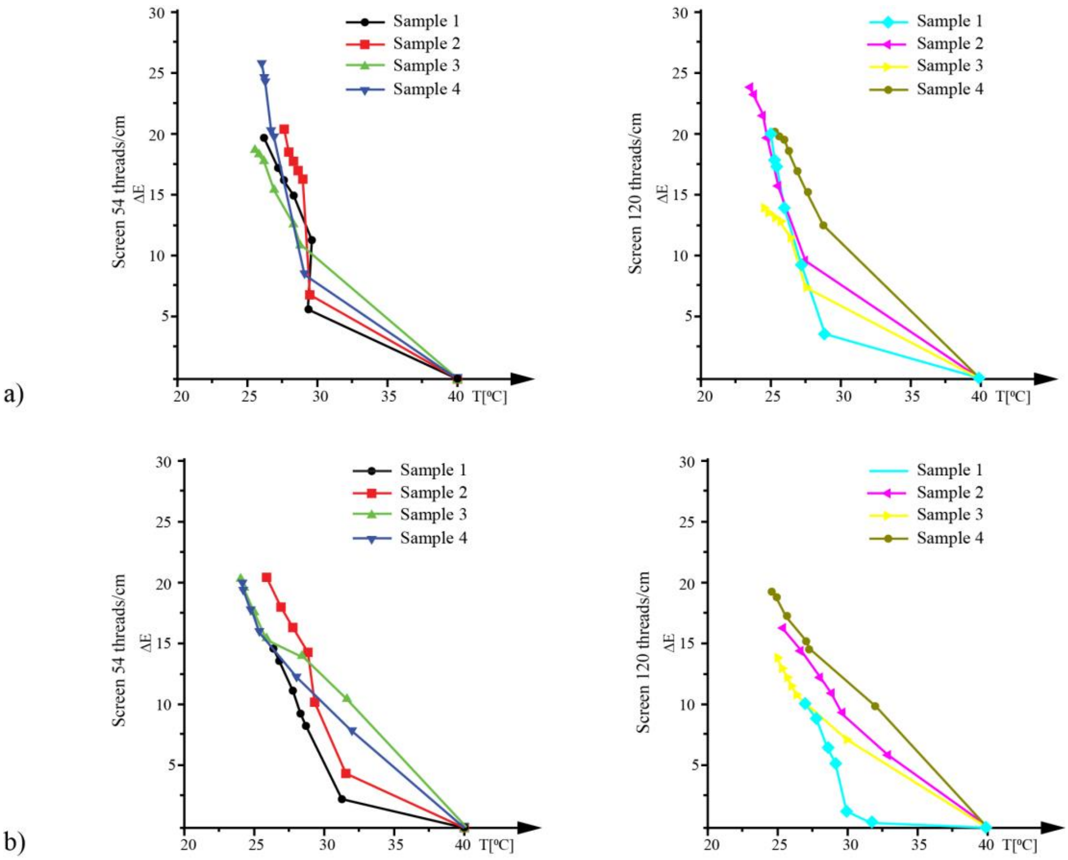

Figure 7. The color difference in relation to the first measured value for screens of 54 threads/cm and 120 threads/cm: a) samples dried within the drying oven, b) samples dried using heat press

4. CONCLUSION

In this article we assessed the change of the colorimetric values of leuco thermochromic ink in screen printing with the change of the temperature during cooling. The printed samples were heated by contact and contactless method. The color measurement was done after the heating, during the cooling of the samples. The samples changed from achromatic to the chromatic after lowering the temperature below the activation temperature. During the cooling, the colors changed significantly in the first 30 s of the measurement. After that time and until the ambient temperature was reached the color difference was very small. It was also noticed that in almost all the samples (excluding cotton fabric with the small fabric weight) the influence of the screen thread count on the material temperature is noticeable. It is observed that using the lower screen counts in screen printing led to higher temperature retention within the printed material, and vice versa. The choice of screen thread counts in printing did not have a significant influence on the temperature change during cooling in case of the cotton sample with small fabric weight.

In addition, it was shown that the heating method influences the cooling time. Although the samples were heated to the same temperature in both methods, the samples were cooled more slowly and therefore slowly changed the colorimetric values after the contact heating.

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