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THE ROLE OF ADHESION ON THERMOCHROMIC PRINTED CARDBOARD BIODEGRADATION

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Abstract: As the packaging industry is growing lately and the amount of packaging waste increases, it becomes one of the major environmental problems. Packaging waste comprising about one-third of all municipal solid waste, mainly plastic and paper based. Anaerobic degradation is one of the most environmentally friendly methods for solid organic waste treatment and widely applied for bio-energy production. In this study, the influence of screen print thermochromic ink on biodegradability aspect of three different cardboard materials (UmcaColor - UC, Propack - PP, Lux Pack - LP) were studied using the soil burial test under anaerobic conditions. Neat and printed cardboard samples were evaluated for changes over 4 months by visual examination, weight loss measurements, volatile solids reduction and surface properties. The surface free energy (SFE) and adhesion parameters were determined for all samples, and showed a very high binding of ink to all cardboard samples. Thermochromic ink reduces the biodegradation of cardboard due to strong ink adhesion on cardboard. If a large SFE interphase is, separation of ink from the substrate occurs. The highest reduction of biodegradation for printed samples was noticed for UC sample. After 120 days the total weight loss for all three neat cardboard samples was similar (about UC 38.4%, PP 34.8%, LP 31.5%). Printed samples showed lower values of biodegradation, up to 36 % reduction compared to neat samples after 120 days.

Keywords: surface free energy, adhesion, biodegradation, cardboard, thermochromic ink

INTRODUCTION

Municipal solid waste continues to be a major environmental problem. Biodegradable municipal waste consists mostly of organic fraction, paper, wood and textiles. Existing methods of waste management include processing and recycling, incineration, composting or aerobic digestion, anaerobic digestion and landfilling. Anaerobic digestion occurs in landfilling sites as natural process but produced methane can be collected or flamed. Paper is mostly recycled by conventional methods like deinking. 40% of paper is wet and is not suitable for recycling but is suitable for composting or digestion (Murphy and Power 2006). Even though paper based materials are often recycled by means of deinking flotation, not all products are suitable for it. Paper based packaging contaminated with food is not desirable in paper recycling facilities due to cleaning difficulties, which leads to contamination issues (Twede, Diana; Selke, Susan; Kamdem and Shires, David; Pira 2015). Besides food contaminants, in the classic flotation deinking process a certain types of prints can cause problems as well. It is known that classic offset prints are easy deinkable by conventional process, but flexographic and UV curing inks can hardly be removed from recycled pulp (Faul 2010). The quality of the waste paper may ultimately decrease as more and more "marginal" paper fractions are collected for recycling and the contents of harmful substances in paper thereby increase (Pivnenko et al. 2015). Study conducted on deinkability aspect of thermochromic inks showed they are very difficult to deink by conventional deinking process (Vukoje et al. 2016). Thus it is crucial to find an alternative method to classic recycling process, such as composting or anaerobic digestion.

Anaerobic digestion (AD) is a promising technology which is widely used in the treatment of various organic wastes, mostly for the treatment of the rapidly biodegradable municipal solid waste, agro-wastes, sewage sludge, etc. It is a natural process where anaerobic bacteria existing in an oxygenfree environment degrade organic matter releasing biogas that primarily contains methane (CH₄) and





carbon dioxide (CO₂). Anaerobic process, if properly applied, can have more advantages than other processes like high degree of waste stabilization, low production of waste biological sludge, low nutrient requirements, no oxygen requirements and useful energy end product – methane (Ismail and Abderrezaq 2007).

Even there are numbers of different methods for biodegradation examination; the soil burial test is one of the simplest methods. The soil beds containing the samples are incubated at a constant temperature for between 28 days and 12 months. The moisture content is normally set at 20–30%, although it is better calculated as a percentage (40–50%) of the soil’s maximum water holding capacity (Chandra 1998).

Variety types of paper and board can be used in packaging applications. Cardboard can either be a single or a multi-layer material. It can be made of more than one type of pulp and it often incorporates a recycled fibres. In multi layered cardboards, the inner layers are usually made of from lower quality pulp than the outer layers. In order to improve printability, the cardboard can contain clay or other coating on one or both surfaces. Cardboard is often classified by its thickness, ranging from 410 to 610 µm (Twede, Diana; . Selke, Susan; l Kamdem and Shires, David ; Pira 2015).Cardboards can be classified using a brief description consisting of two letters and a number, for example GC1; where G represents coated, C- chromo and 1- low quality.

Thermochromic inks are temperature sensitive materials which change colour with heat. In their cool state, they exhibit colour, and when heated, they become clear or translucent. The temperature range at which transition of coloured to colourless state occurs is commonly called the activation temperature (TA). The change of colour may be irreversible or reversible (Homola 2008). The reversible inks microcapsule consists of leuco-dye, solvent and colour developer. The main disadvantage of organic microcapsules is their insufficient light stability and high sensitivity to environmental changes (Rožić et al. 2015). Thermochromic inks are available for use on a variety of substrates and they have been developed for many types of printing processes, such as screen printing, gravure and flexography, and more recently for applications in offset lithography (Bamfield, P, Hutchings 2010).

Many studies have reported the degradation of paper based materials in various environments such as aerobic and anaerobic (Yen and Brune 2007; Wang et al. 2015; Fonoll et al. 2016), but there is lack of studies about influence of printing inks on biodegradation, especially for thermochromic inks.

METHODS

Materials

Three different cardboard materials were used in biodegradability test. The used cardboards are representing different classes of cardboards (Table 1) according to their properties and quality.

Table 1 – Cardboard classification

Cardboard samples	Abbreviation	Grammage, g/m ²	Classification
Lux Pack	LP	350	GC1
Propack	PP	350	GT2
UmcaColor	UC	350	GD2

All cardboard samples were analysed in the terms of thickness, moisture, ash and CaCO₃ content. Thickness of all samples was determined according to T411 standard. Moisture content was determined according to T412 om-94, while ash content was determined according to T413 (combustion at 900°C). Alkalinity of paper as calcium carbonate was determined according to T533.

All three cardboard samples were printed with thermochromic ink in order to examine how it affects the process of biodegradation. One leuco dye based, screen-printing UV curable thermochromic ink produced by CTI® was used for printing. The thermochromic ink was coloured in purple below its activation temperature (TA=31 °C) and changed to pink above the activation point. The biodegradation of neat (UC, LP, PP) and printed cardboard (UC-UV, LP-UV, PP-UV) samples was studied using the soil burial test under anaerobic conditions as it will be described in the text.

Printing

The printing trials were carried out using the Siebdruckgeräte von Holzschuher K.G., Wuppertal. The cardboards were printed in full tone, under the same conditions. The printed samples were dried under the UV irradiance (30 W/cm) using TechnigrafAktiprint L 10-1 device.

Determination of surface properties

The evaluation of surface free energy (SFE) of neat and cardboard printed with thermochromic ink was carried out by measuring contact angle of standard liquids. Measurements were conducted on DataPhysics OCA 30 Goniometer, using the Sessile Drop method. Standard test liquids whose surface





tensions are known (Table 2) were demineralized water, diiodomethane and glycerol. Measurements were performed at room temperature of $23.0 \pm 0.2^\circ\text{C}$. The volume of droplet was $1 \mu\text{L}$. Contact angle was captured by CCD camera and measured 1s after the droplet was formed. Average values of ten drops

Table 2 – Surface tensions (γ) of test liquids and their dispersive (γ^d) and polar (γ^p) components

Liquid	Surface tension (mJm^{-2})		
	γ	γ^d	γ^p
Water	72.80	21.80	51.00
Diiodomethane	50.80	50.80	0.00
Glycerol	63.40	37.00	26.40

obtained surface free energy and its components are presented in the results section (Table 5). From the obtained SFE, adhesion parameters were calculated. Thermodynamic work of adhesion W_a between two phases was calculated according to (Eq. 1) (Żenkienwicz 2007).

$$W_{12} = \gamma_1 + \gamma_2 - \gamma_{12} \quad [1]$$

where the subscript refers to surface free energy of the each solid, in our case cardboard and ink print, and the γ_{12} denotes their surface free energy of the interphase. Using the Owens-Wendt model the surface free energy of the interphase was determined according to Eq.2 (Żenkienwicz 2007).

$$\Gamma_{12} = \gamma_1 + \gamma_2 - 2\sqrt{\gamma_1^d \gamma_2^d} - 2\sqrt{\gamma_1^p \gamma_2^p} \quad [2]$$

Soil burial experiments

Soil was sieved to less than 2 mm particle size. Large plant materials, stones, and other inert materials were removed. Laboratory soil burial experiments were conducted at room temperature $25 \pm 3^\circ\text{C}$ by placing the neat and printed cardboard samples horizontally in laboratory glass containers filled with soil. Samples were cut in $4 \times 5 \text{ cm}$. All the samples were buried for 14, 32, 50, 80 and 120 days in glass containers filled with the soil. The water content of the soil was adjusted to 60% of its maximum water retention capacity. The commercial available reagent was used in order to allow the development of anaerobic conditions. The existence of anaerobic conditions was proved with Anaerotest (Merck) strips.

Visual observation of surface appearance change

Changes in the appearance of sample surfaces were observed before and after biodegradation. Photos of all samples were taken in order to visually evaluate the substrate degradation over time. A method of visual evaluation can be used in order to describe biodegradation as a first indication of any microbial activity in the terms of visible surface changes (formation of holes or cracks, de-fragmentation, changes in colour)(Shah et al. 2008).

Weight loss measurement and Volatile solids (VS) determination

After the soil burial test, biodegradability was measured through weight loss and volatile solids (VS) reduction. After the incubation in the soil containers, the samples were taken out and rinsed with distilled water to remove soil particles from the surface. Then were dried to constant weight and weighed. The weight loss percentage was calculated according to Eq.3.

$$\% W = \frac{m_0 - m_1}{m_0} \cdot 100 \quad [3]$$

where, m_0 is the initial weight of the sample, m_1 is the final weight of the sample after degradation.

The volatile solids (VS) were determined by ignition of material in muffle furnace at 550°C during 2 h. All the samples were tested for VS.

Colour measurement

The colour measurement was carried out using X-Rite i1Pro spectrophotometer at temperature of $23 \pm 2^\circ\text{C}$. The colour of original printed cardboards and printed samples after biodegradation was determined according to the CIE $L^*a^*b^*$ system. The average of ten values of those measurements was presented in Result section (Table 8). The total colour difference ΔE^*Lab was determined according to the Equation 4 and presented in Figure 11.

$$\Delta E = \sqrt{(L_2 - L_1)^2 + (a_2 - a_1)^2 + (b_2 - b_1)^2} \quad [4]$$

RESULTS AND DISCUSSION

Due to the different constitution materials and quality properties the cardboard samples were examined for some basic properties such as thickness, moisture, ash and CaCO_3 content and results are presented in Table 3. All the samples have similar moisture content. The lowest amount of ash content was found in LP sample, followed by UC and PP. The amount of CaCO_3 increases in a series of: $\text{PP} < \text{LP} < \text{UC}$. This





value can originate from fillers as well as from coatings. All samples differ in thickness, which grows in a row: UC < PP < LP. Due to different degrees of wetting, the thickness of TC UV print is different for all samples, and increases in a row: PP < UC < LP.

In order to obtain surface free energy (SFE) of all samples and adhesion parameters, the samples were examined for contact angle measurements with different liquids. Results of contact angle determination are presented in Table 4, while results of SFE determination and adhesion parameters are presented in Tables 5 and 6.

Obtained contact angles presented in Table 4 show mostly hydrophilic surface properties of cardboard except for PP which shows hydrophobic surface. The highest hydrophilic surface is noticed for UC sample. All samples of prints show hydrophobic surface (UC-UV > LP-UV > PP-UV).

In the Table 5 the obtained results for surface free energy of neat and printed cardboard samples are given. Neat cardboard samples have a large proportion of polar component in its SFE, as indicated by the γ^p and the dispersion index x^d . The highest polar character of surface is observed for UC sample (13.56 mJ m⁻²), followed by LP (10.01 mJ m⁻²), which is characteristic for the hydrophilic surface. Compared to UC and LP sample, PP sample has hydrophobic surface with the smallest polar component (1.44 mJ m⁻²). SFE of neat cardboard samples increases in a row: PP < LP < UC. By observing the SFE of prints, they are similar in all samples. SFE of prints on all samples mostly originate from its dispersive component, which is characteristic for hydrophobic surface. It is evident that LP-UV sample does not have hydrophilic molecules on the surface ($\gamma^p = 0.00$ mJ m⁻²), and its total SFE is equal to its dispersive component. The other two printed cardboards (PP-UV and UC-UV) show the existence of polar components on their surface according to obtained SFE. From this it can be concluded that an adhesive bond between cardboard and ink is mostly provided by nonpolar interactions.

Table 6 shows the adhesion parameters for cardboards and prints. Optimum adhesion can be achieved when the following conditions are fulfilled: $W_a = \text{maximum}$, $\gamma_{12} = \text{minimal}$, and $\gamma_1 = \gamma_2$. As these conditions are satisfied, adhesion will be optimal. Considering these conditions, it can be concluded that the greatest adhesion is achieved on a UC-UV sample ($W_a = \text{largest}$, SFE of both phases are almost equal) regardless to larger SFE of interphase (γ_{12}). In other two samples (PP-UV and LP-UV) the work of adhesion is similar but noticeably lower than in the UC-UV sample. This can be attributed to the large SFE of interphase (in the case of the LP-UV) and with different SFE of individual phases (in the case of PP-UV). The obtained results show that the interaction between all tested cardboards and TC ink are strong. The work of adhesion is the highest for UC-UV sample (62.77 mJ m⁻²) (Table 6). The smallest work of adhesion is observed for the PP-UV sample (41.30 mJ m⁻²) due to largest differences in SFE of individual phases.

The adhesion of ink onto the cardboard substrate depends on the intermolecular interactions between them. Cardboards with hydrophilic (polar) surface (LP and UC) show higher values of SFE of interphase. The highest SFE of interphase is observed for the LP-UV sample (11.88 mJ m⁻²). In this case the adhesion is provided only by the dispersive bond due to absence of polar components in the print. The high SFE of interphase can be explained by the existence of polar molecules on the surface of LP cardboard which creates resistance to print and thus preventing the binding and absorption of ink on the surface of cardboard. This resistance will result as a higher γ_{12} value. Cardboard with hydrophobic surface (PP) shows the minimum value of SFE of interphase (3.41 mJ m⁻²). Furthermore, SFE of interphase also affects the thickness of print on the cardboard surface. The greater the SFE of interphase, the thickness of the print on the cardboard higher is (Table 3 and 6). Thus, LP-UV results in the thickest print, followed by UC-UV, and PP-UV with a minimum thickness of the print. Due to maximum absorbency of ink on PP

Table 3: Properties of used cardboards

	moisture, %	ash, %	CaCO ₃ , %	Thickness, mm	Thickness of TC UV print, mm
LP	4.63	9.67	13.09	0.563	0.010
PP	4.28	18.51	12.56	0.494	0.003
UC	4.51	16.25	15.28	0.453	0.005

Table 4: Contact angle measurements

Sample	Contact angle θ		
	Water	Diiodomethane	Glycerol
LP	68.4±3.48	56.9±3.59	105.9±2.90
PP	98.5±7.39	73.0±2.98	58.2±5.17
UC	58.2±4.40	48.1±3.01	97.8±2.29
LP-UV	103.0±4.48	46.8±1.70	99.1±1.95
PP-UV	96.6±4.51	44.7±1.73	101.1±1.93
UC-UV	114.8±4.48	32.7±1.67	119.3±2.23

Table 6: Adhesion parameters

Sample	Adhesion parameters (mJ m ⁻²)	
	γ_{12}	W_a
LP - UV	11.88	45.78
PP - UV	3.41	41.30
UC - UV	6.40	62.77





cardboard, the smallest is the difference in the contact angles of neat and printed PP cardboard (Table 4). In order to make a conclusion about biodegradability, changes in the appearance of sample surfaces were observed before and after biodegradation during the whole sampling time. Samples were taken out from the soil containers in different sampling periods (14, 32, 50, 80 and 120 days). Formation of holes, cracks and changes in colour were noticed.

From Figure 1, 2 and 3 it can be noticed that the biggest changes on the surface of neat cardboard samples occur after 80 days. During the 14, 32 and 50 days, the biggest changes are reflected in the change of color, i.e. they tend to yellow. The biggest changes occur after 120 days of biodegradation for all samples. Also, changes in the coating can be noticed, due to formation of wrinkles and cracks.



Figure 1: Biodegradation over 120 days for neat LP cardboard



Figure 2: Biodegradation over 120 days for PP neat cardboard



Figure 3: Biodegradation over 120 days for neat UC cardboard



Figure 4: Biodegradation over 120 days for printed LP - UV cardboard

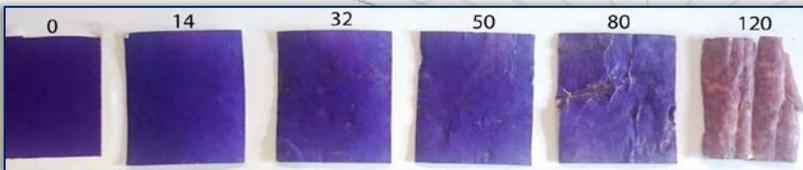


Figure 5: Biodegradation over 120 days for printed PP - UV cardboard

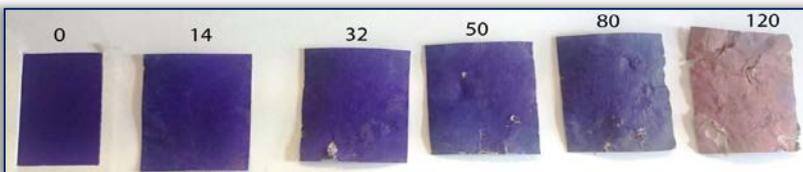


Figure 6: Biodegradation over 120 days for printed UC - UV cardboard

Observing the printed cardboard samples (Figures 4, 5 and 6), the most significant changes are in the colour of the print after 120 days. The print is much lighter on all printed samples. During the first 14 days of biodegradation test, no significant change in colour or on the surface of samples occurred. After 32 days in the LP-UV and UC - UV samples can be noticed the formation of cracks in the print, while in PP-UV sample it is visible after 80 days. In the LP-UV sample cracks in the print are the most significant but also detachment of ink layer from substrate can be observed. This can be attributed to the maximum value of SFE of interphase γ_{12} between the cardboard and ink (Table 6). As SFE of interphase increases in a row: PP-UV < UC-UV < LP-UV, the cracking and lamination of ink layer from the cardboard surface can be noticed.

By observing the neat cardboard samples, the highest level of weight loss shows UC. After 120 days the total weight loss was about 38.4%, followed by the PP with 34.8%. The smallest weight loss has LP, with 31.5% after 120 days of biodegradation. This sequence can be attributed to the thickness of cardboard - the thinner the cardboard, better degradation is. Considering only the printed samples, it is obvious that the largest weight loss shows the sample LP - UV (31.7%), followed by the PP-UV (30.94%). The lowest weight loss was observed at the UC - UV sample (24.6%). The smallest changes in weight loss of all samples are in the first 50 days. After 80 days, there is a significant change in the weight loss of samples, particularly evident for the LP and LP-UV pattern. As with other samples, the biggest changes were observed after 120 days.



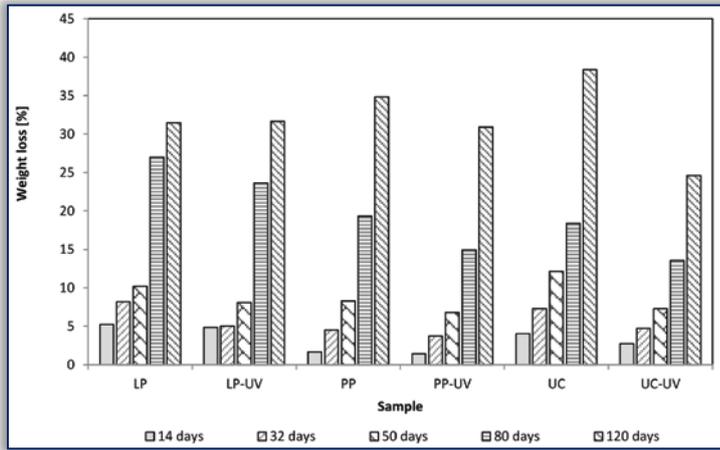


Figure 7: Biodegradation of samples through weight loss over 120 days

Table 7: The influence of thermochromic ink on the reduction of biodegradation over time, compared to neat cardboard

	Time (days)				
	14	32	50	80	120
PP/PP-UV	14.38 %	17.50 %	14.65 %	14.65 %	11.21 %
LP/LP-UV	7.00 %	38.78 %	20.72 %	12.45 %	0.00 %
UC/UC-UV	31.85 %	35.07 %	40.23 %	26.29 %	35.80 %

samples were reduced by about 26 - 40% (Table 7). As in the PP-UV and UV-LP samples the obtained work of adhesion is quite the same (Table 6), reduction of weight loss in the samples was lower than in UC-UV sample. In addition, it can also be attributed to SFE of interphase between the ink and the substrate. LP-UV pattern has the highest SFE of interphase, and during the 120 days the reduction of weight loss differed and cannot be observed by a specific rule. The SFE of interphase in the UC-UV sample was double less than in LP-UV sample and it can be seen that there is less variation in the weight loss reduction during the sampling time, and varies from 26 to 40%. Due to lowest SFE of interphase in PP-UV, the regular reduction of weight loss vs. sampling time ($14 \pm 3\%$), or with very small deviations, can be observed.

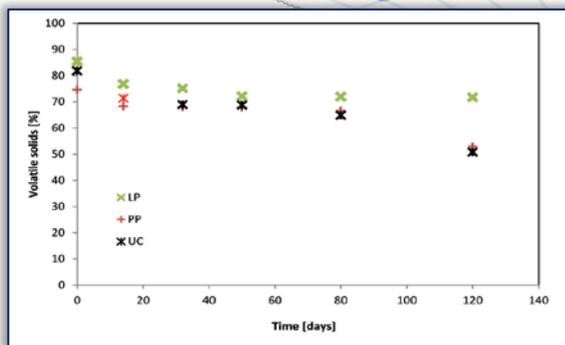


Figure 8: Reduction of volatile solids over time for neat cardboard

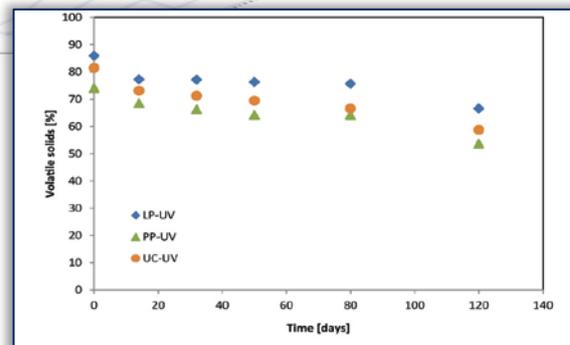


Figure 9: Reduction of volatile solids over time for printed cardboard

Concerning the percentage of ash in neat samples (Table 3), it is expected that the LP sample will have the largest share of VS. Observing neat samples (Figure 8), after 120 days the highest percentage reduction in VS was observed at UC (37.9%), followed by PP (27.6%) and LP (16.1%) compared to the initial share of VS in all samples. These results confirm the results of the weight loss determination. For all printed cardboard samples (Figure 9), percentages of VS decrease are very similar. After 120 days, for UC-UV a reduction of VS was around 27.9%, for PP-UV 27.6% and for LP-UV 22.48%. In the printed samples decrease of VS originate from cardboard and from ink degradation. From this it can be concluded that weight losses during 120 days originates not only from decomposition of organic matter but also from defragmentation and washout of coatings and fillers.

Table 8 shows that all samples over time bright, ie their colour start to fade. The a^* value is reduced to almost half of its value but still remains in the red area of $L^*a^*b^*$ colour space, while the value of b^*

Thermochromic ink significantly influence the degree of cardboard biodegradation. Although sometimes ink can serve as a food source for the microorganisms and thus improve the degree of biodegradation (Lucas et al. 2008), here the different trend was observed. Degree of biodegradation was observed through weight loss, in all printed samples compared to neat cardboard. The total weight loss of PP - UV after 120 days was reduced by about 11% compared to the neat cardboard. The greatest impact of thermochromic ink on biodegradation was observed in UC-UV sample. The total degradation after 120 days compared to neat sample was reduced by 35%. This can be attributed to the high adhesion of ink to the substrate. The sample UC-UV has the greatest adhesion of TC ink to the substrate (62.77 mJ m^{-2}). During all the time interval of biodegradation, printed





changes from negative to positive values. This means that the blue thermochromic microcapsules are biodegradable during conducted degradation process. Blue thermochromic microcapsules in the formulation of the print are responsible for the purple coloration of the print (Figure 10). By mixing blue leuco dye to a conventional pink ink will result in a purple ink formulation.

Table 8: Colorimetric properties of thermochromic ink at 23±2°C

DAYS	LP-UV			PP-UV			UC-UV		
	L*	a*	b*	L*	a*	b*	L*	a*	b*
0	34.70	40.47	-35.34	34.70	37.67	-33.95	35.66	45.12	-25.51
14	37.29	30.65	-31.81	37.14	30.13	-32.35	36.37	31.37	-27.76
32	36.39	26.11	-29.98	38.43	28.59	-28.99	42.15	24.44	-22.26
50	39.74	24.81	-27.15	38.31	23.03	-26.33	42.46	21.57	-20.05
80	41.75	23.19	-19.93	43.92	26.35	-21.15	46.62	20.21	-12.63
120	57.79	33.25	8.50	50.34	24.63	4.43	59.40	25.41	8.16

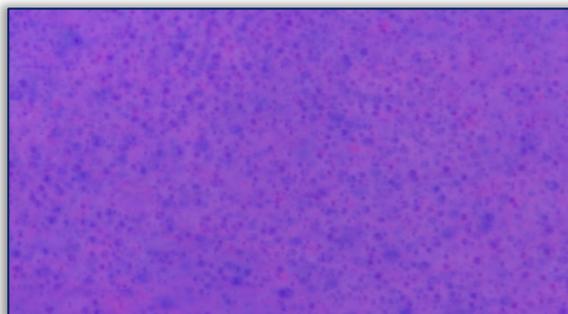


Figure 10: Blue microcapsules in ink formulation (original sample) at magnification of 2000x

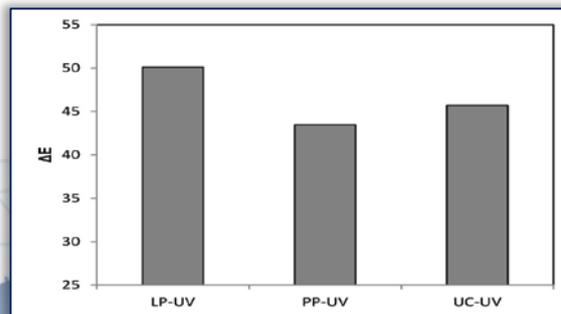


Figure 11: Total colour difference after 120 days of biodegradation

Figures 4, 5, 6, 11 and Table 8 shows that the print was mostly discoloured after 120 days. In the period to 80 days, changes in colour were not so significant. The highest change in colour (ΔE) was observed in the case of LP-UV cardboard while the lowest in the case of PP-UV cardboard (Figure 11). This could be associated with an SFE of interphase. The higher the SFE of interphase is, the greater the change in colour after 120 days can be noticed. As the γ_{12} increases in a row: PP-UV < UC-UV < LP-UV; the difference in colour (ΔE) increases in a series of PP-UV < UC-UV < LP-UV. Stronger bond between the ink and cardboard will result in better colour stability. The higher the value of SFE of the interface is, the forces of binding to the substrate are less.

CONCLUSION

Thermochromic ink reduces the biodegradation of cardboard. The greatest role plays the ink adhesion on cardboard, but also its SFE of interphase. The greater the adhesion of ink on the surface will result in lower weight loss, i.e. lower degree of biodegradation. If a large SFE of interphase is, separation of ink from the substrate occurs. This can improve the biodegradation of the sample.

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Note

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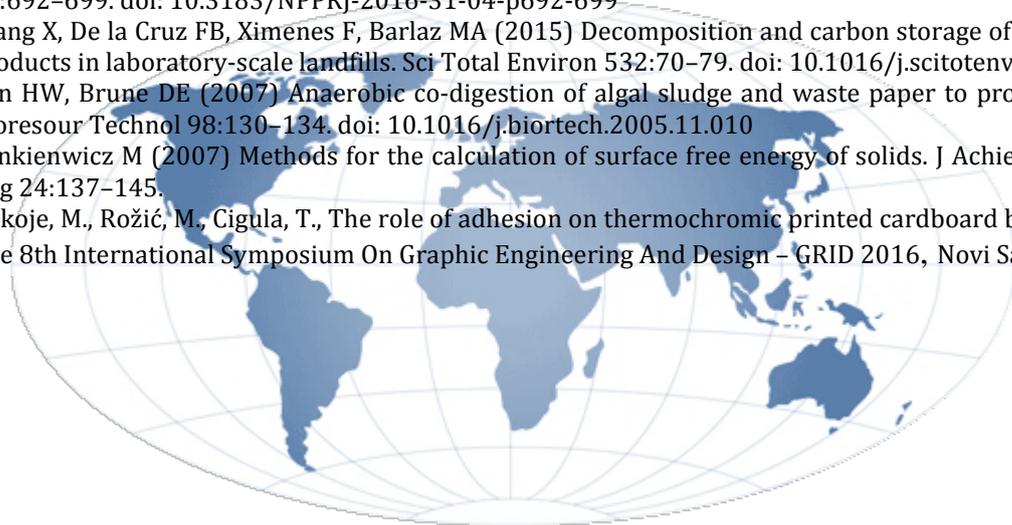
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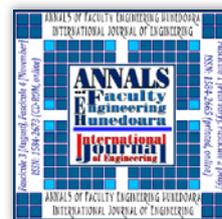




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