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Properties of UV-cured pigment prints on textile fabric

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Abstract

This paper studies and evaluates the UV-curing of pigment prints on textile fabric using a prototype UV scanner. A printing paste comprising synthetic thickener, emulgator, binder, pigment dispersion and photoinitiator was applied using a flat screen printing technique onto the cotton fabric, then dried and exposed to heat or UV-radiation under a mercury vapour lamp (200 W cm^{-2}). The characteristics of cured prints such as paste add-on, colour properties, colour fastness to washing and dry/wet rubbing were evaluated, together with fabric stiffness. The effects of UV-curing were evaluated by Attenuated Total Reflectance Fourier transform infrared spectroscopy (ATR-FTIR). The properties of the UV-cured pigment prints were compared with those of the thermal cured prints. Analyses of the obtained results helped to define the optimum composition of the photo reactive pigment paste, and the UV-curing conditions under which satisfactory results were obtained, comparable with those from the thermal curing method.

Keywords: Textile printing; Pigment dispersion; Binder; Photoinitiator; UV-curing; Printing quality

1. Introduction

Pigment printing is the predominant printing application method for around 65% of prints [1,2]. The main factors why pigment printing has become so widely used are the quality of the prints, applicable to almost every kind of fibre or mixture, and the ability to avoid any washing processes after fixation [3].

The pigment printing of fabrics is an important field in industrial applications where UV-curing is rarely used or investigated. In textile finishing processes such as pigment dyeing, coating and pigment printing, the conventional thermal curing technique is still used irrespective of energy consumption and the cost. UV-curing technology is used in many other industrial applications for curing because of low energy consumption, short start-up period, fast and reliable curing, low environmental pollution, curing at room temperature, space saving, etc. [3,4].

In the past only a few articles, research reports or some patents have been published referring to UV-curing technology in textile printing application. In research work carried out by Fouassier et al. [5] a UV-curable formulation was developed for printing on textile fabrics. As reported, it was important to select a photoinitiating system that can be used under identical conditions, whatever pigment is used. In another research work, which was done as a cooperation between Lamberti Spa., Italy and Fouassier [6], the

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authors concluded that, due to the outstanding results obtained, the technique appears to be very promising and should open up new opportunities for UV-curing technologies in the textile finishing area.

Recently, in an EU project [7] dealing with the possibility of UV-curing for ink-jet printed pigment prints on textile fabric, an investigation was performed using a prototype UV scanner. One of the research phases of the project, presented in this article, was dedicated to investigating the suitability of curing screen printed pigment prints with a UV scanner, a newly developed multifunctional photoinitiator, and the evaluation of UV-cured prints on cotton fabric. Analyses of the obtained results helped us to specify the optimum photo reactive pigment paste recipe and UV-curing conditions, under which satisfactory results were obtained, comparable with those from the thermal curing method.

2. Experimental

2.1. Materials

The experiments were performed on 100% cotton fabric, supplied by Tekstilna tovarna Prebold d.d., Slovenia, which was already desized, scoured, bleached and mercerised. Fabric specifications were: weight 120 g m^{-2}, warp 28 threads cm^{-1}, 20 tex, weft 26 threads cm^{-1}, 20 tex and with fabric stiffness of 4.46 cN cm^2. No further special pretreatment was required as the cotton fabric had been prepared for dyeing and printing.

Two printing pastes were prepared, one type for thermal and another type for UV-curing. The conventional pigment paste for thermal-curing was composed of Tubivis DL-650 synthetic thickener (polyacrylate dispersion fully neutralized), non-ionic Tubigat AFR-20 emulsifier (combination of emulsifier, additives and melamine resin fixing agent), and a Tubifast BN-35 binder (thermally self-crosslinking butadiene copolymer dispersion binding agent). All paste ingredients were supplied by Bezema AG., Switzerland. The composition of the pigment paste for UV-curing was the same, the only exception was the addition of multi component photoinitiator Esacure DP250 from Lamberti Spa., Italy.

The Tubifast BN-35 binder is a butadiene–acrylonitrile copolymer with 38% of active substance. It is known that butadiene itself produces a very soft homopolymer, but because of too high stickiness it must be copolymerised with a stiffer acrylonitrile monomer. Butadiene improves the dry fastness characteristics and, at the same time, softens the print. Acrylonitrile is used for increasing the toughness of polymer and it does not increase the stiffness. Butadiene–acrylonitrile copolymers are known for their strength as well as their solvent and abrasion resistance [8–10].

The Esacure DP250 photoinitiator is a stable water emulsion which contains 32% of active photoinitiator and is composed of a blend of α-hydroxyketone (AHK), monoacylphosphine oxide (MAPO), benzophenone derivatives (BP) and acrylic esters. As reported [11,12] a combination of AHK, MAPO and BP is most suitable for the curing of pigmented systems. AHK gives the coating a good surface cure because it is relatively insensitive to oxygen inhibition, while MAPO was found to give a good through cure [6]. Acylphosphine oxides have the advantage of absorbing in the near UV-range, where the mercury lamp has its strongest emission, generating very reactive benzoyl and phosphinoyl free radicals which are capable of initiating the polymerization of acrylates [13].

The pigment dispersion used was the Bezaprint Red KF based C.I. Pigment Red 146 from Bezema AG., Switzerland (Fig. 1).

2.2. Preparation of the printing pastes, printing and fixation process

The pigment pastes for thermal-curing produced from stock paste, were prepared from demineralised water, 15 g kg^{-1} of Tubivis DL-650 synthetic thickener, 10 g kg^{-1} of Tubigat AFR-20 emulgator and 50–120 g kg^{-1} of Tubifast BN-35 binder. Printing pastes were prepared by mixing the stock paste and 7.5 g kg^{-1}, 15 g kg^{-1} or 30 g kg^{-1} of Bezaprint Red KF pigment dispersion (Table 1).

The pigment pastes for UV-curing were produced from stock paste in which demineralised water, Tubivis DL-650 synthetic thickener and Tubigat AFR-20 emulgator were stirred. UV printing pastes were prepared by the addition of Tubifast BN-35 binder, Bezaprint Red KF pigment dispersion and 40–80 g kg^{-1} of Esacure DP250 photoinitiator to the stock paste (Table 2).

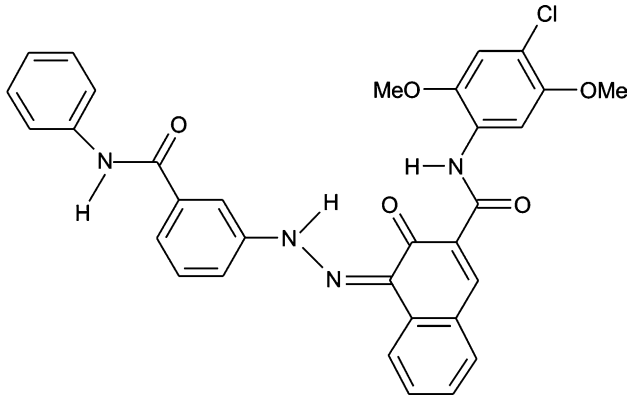

Fig. 1. Structure of C.I. Pigment Red 146.

Table 1
Printing paste composition for thermal curing

Code	Binder conc. ($g\,kg^{-1}$)	Pigment conc. ($g\,kg^{-1}$)
T1	50	7.5
T2	75	15
T3	120	30

The stock pastes viscosity was set at 7 Pa s (measured with HAAKE VT02).

These prepared, and well homogenised, printing pastes were applied to the cotton fabric using a Zimmer laboratory flat screen printing machine. The processing conditions of both thermal and UV-curing are presented in Table 3. UV-curing was performed in the presence of oxygen with a prototype UV Scanner from Ist Metz GmbH., Germany.

This UV Scanner consists of an CK ultraviolet lamp in which a mercury arc discharge is fired, a BLK reflector system, a cooling system and an electrical supply (Fig. 2) [14,15]. Equally important as the lamp itself is the reflector that reflects the light radiated sideways or backwards onto the object. It plays a vital role that cannot be underestimated. The proportion of light emission reflected onto the object is as high as 55%. The complete spectrum of short wavelengths, visible and IR light is directed onto the object using Al mirrors. The object temperature can be reduced by up to 30% by using so-called cold reflectors. Such reflectors consist of temperature-resistant glass that has been vapour-deposited with so-called dielectric layers on the reflector surface. These reflect UV light but allow visible light and IR light to be directed backwards onto a cooled surface.

2.3. Methods

The paste add-on of dried prints was measured in accordance with SIST ISO 3801:1996. The reflectance measurements of the cured pigment prints were determined using a Datacolor Spectra Flash 500 spectrophotometer, under the following conditions: d/8 measurement geometry, measurement wavelength range from 400 nm to 700 nm, measurement area of 12 mm in diameter, and SIN-specular included measurement mode. XYZ, CIE $L^*a^*b^*$, C^*, h CIELAB 1976 and colour strength values (K/S) were calculated using Datacolor Datamaster software. The conditioned thermal and UV-cured samples were subjected to stiffness measurements which were evaluated according to the DIN 53362 method. The colour fastness to washing, dry and wet rubbing of the thermal and UV-cured samples were determined by the conventional methods SIST EN 20105-C03:1996 and SIST EN ISO 105-X12:2002, respectively. The absorption characteristics of the dissolved photoinitiator, as well as pigment dispersion, were measured using a Varian Cary 50 UV/VIS spectrophotometer in the wavelength range 200 nm–700 nm.

ATR-FTIR spectra of prints were obtained using the Perkin–Elmer Spectrum One FT-IR spectrometer with Golden Gate ATR attachment with diamond crystal. The absorbance measurements were carried out in the range 600 cm^{-1}–4000 cm^{-1}, with 10 scans and resolution of 4 cm^{-1}. The decrease in butadiene copolymer binders' double bonds at a wavelength at 967 cm^{-1} were measured [19–22]. For semi-quantitative measurements, the infrared spectra of the printed cotton fabrics were normalised against the 1314 cm^{-1} band, which can be associated with the bending vibration mode of hydrocarbon structures in cellulose molecules [17,18]. In all measurements the bands limits were adjusted manually. The baseline limits were 980 cm^{-1}–960 cm^{-1}. Perkin–Elmer Spectrum V3.02 software was used in the calculation procedures.

Table 2
UV printing paste composition

Code	Photoinitiator conc. ($g\,kg^{-1}$)	Binder conc. ($g\,kg^{-1}$)	Pigment conc. ($g\,kg^{-1}$)		
A1	40	100	7.5	15	30
A2	40	150	7.5	15	30
A3	40	200	7.5	15	30
B1	60	100	7.5	15	30
B2	60	150	7.5	15	30
B3	60	200	7.5	15	30
C1	80	100	7.5	15	30
C2	80	150	7.5	15	30
C3	80	200	7.5	15	30

Table 3
Printing and curing conditions

Stage of Work	Conditions of work	
	Thermal	UV
Printing	Laboratory printing machine J. Zimmer Type VP-RSF Flat printing screen: 70 mesh Squeegee diameter: 10 mm Printing speed: 4 m min^{-1} Pressure grade: 1 Number of squeegee wipes: one	
Drying	Laboratory dryer Mathis DHE 43687 Hot air 100 °C for 2 min	Laboratory dryer Mathis DHE 43687 Hot air 60 °C for 2 min
Curing	Laboratory dryer Mathis DHE 43687 Hot air 150 °C for 5 min	UV Scanner IST Metz Mercury lamp 200 W cm^{-2} 300 mm s^{-1} Curing in presence of oxygen

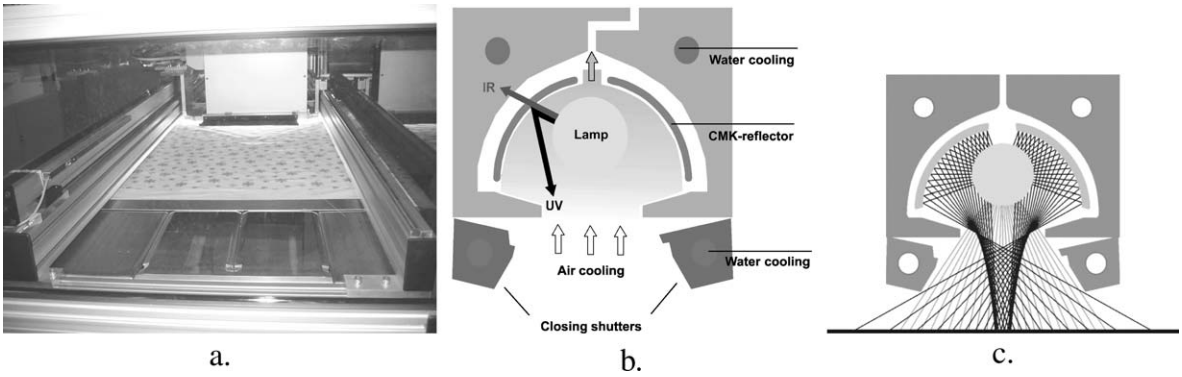

Fig. 2. UV Scanner IST Metz (a), UV unit (b) and BLK-reflector (c) [11].

3. Results and discussion

3.1. Characteristics of thermal cured pigment prints

In the first stage of the investigation, the prepared pigment pastes (Table 1) were printed on cotton fabric using a flat bed Zimmer screen printing machine, dried, and then thermally polymerized with hot air at 150 °C for 5 min. Their properties regarding paste add-on, colour characteristics, and stiffness and fastness levels are shown in Table 4. The paste add-on amount and K/S values, as well as fabric stiffness, increase with the increasing amount of binder and pigment. As expected, the higher amounts of binder caused higher stiffness value, which is reflected in fabric handle. Normally, the stiffness values of thermally cured prints are approximately four times higher (15.60 cN cm^2–18.23 cN cm^2) than the stiffness value of raw cotton fabric (4.46 cN cm^2). Colour fastness to rubbing, regarding washing, decreases with increased pigment concentration. The auxiliary crosslinking agent must be added into the printing paste for improvement of colour fastness characteristics. This could result in rigid fabric handle [10].

3.2. Spectral characteristics of mercury lamp, photoinitiator and pigment

Prior to the pigment prints being UV-cured, we evaluated the absorption properties of the photoinitiator, and the pigment dispersion of C.I. Pigment Red 146. The UV-curing of pigmented prints remains difficult, due to high light absorption by the pigment, which is detrimental to light absorption by the photoinitiator [6]. An efficient cure can only be obtained by achieving the best overlap between the absorption spectrum of the photoinitiator, the transmission spectrum of the pigment, and the emission spectrum of the light [13].

Fig. 3 shows the spectral distribution of the mercury lamp, the absorption characteristics of the photoinitiator, and the dispersion of C.I. Pigment Red 146. The Esacure DP250 photoinitiator in methanol exhibits strong absorption at 249 nm, as well as at 365 nm, with a tail up to 420 nm and, at the same time, coincides with the emitted spectrum peaks of CK Hg lamp. The pigment dispersion of C.I. Pigment Red 146 has absorption maxima at 234 nm, 529 nm and 574 nm.

3.3. Characteristics of UV-cured pigment prints

In the next stage of the investigation the prepared screen prints were UV-cured using a UV Scanner. The aim of these preliminary test was to evaluate the UV-curing efficiency when 15 g kg^{-1} of C.I. Pigment Red 146 prints were UV-cured from only one side, and in another separate experiment from both sides. The prints were cut into two parts after one or double sided UV-curing. The first part was intended for preliminary colour fastness tests to rubbing, while the second part was washed with demineralised water (60 °C, 30 min), dried, and the R% reflections' characteristics was

Table 4
Characteristics of the thermally cured prints of C.I. Pigment Red 146

Code	Pigment conc. (g kg^{-1})	Paste add-on (g m^{-2})	$K/S_{520\ nm}$	Fabric stiffness (cN cm^2)	Colour fastness to washing at 60 °C	Colour fastness to rubbing			
						Warp		Weft	
						Dry	Wet	Dry	Wet
T1	7.5	129.23	6.72	15.60	5	5	4–5	5	4–5
T2	15	132.54	10.33	17.61	5	5	4	5	4
T3	30	134.94	13.67	18.23	4–5	5	3–4	4	3

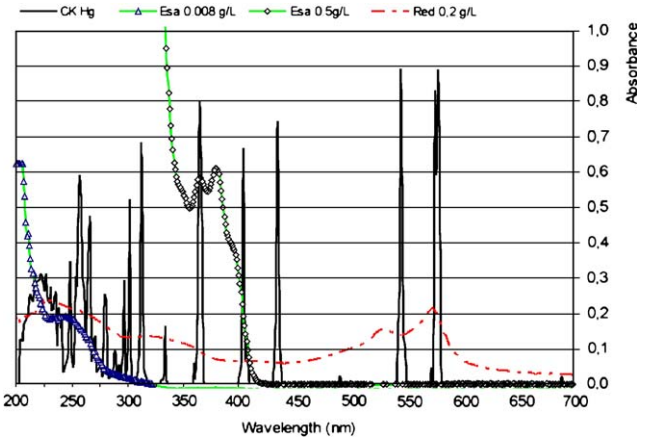

Fig. 3. Spectral distribution of UV Scanner (CK Hg) mercury vapour lamp and absorption curves of photoinitiator Esacure DP250 in methanol (Esa 0.008 g/L, Esa 0.5 g/L) and dispersion of C.I. Pigment Red 146 (Red 0.2 g/L).

measured afterward. The evaluated characteristics of the one and double sided UV-cured prints are shown in Table 5.

The printability of the UV printing pastes was satisfactory with no viscosity problems or screen clogging occurring. No unpleasant or irritating smell was noticed during the phases of paste preparations, printing or UV-curing. Measured add-on amounts of UV-cured prints are comparable to thermal polymerised prints and are in the ranges of 133.28 g m^{-2}–137.61 g m^{-2}.

It is evident from Table 5 that the colour fastness properties of one side UV-cured prints increase with the number of UV light passes and, at the same time, with a higher concentrations of photoinitiator in the printing paste. The very best printing results were obtained in the case of C1 double side (8 passes from the front and 4 passes from the reverse side) UV-cured prints, when 80 g kg^{-1} of photoinitiator and 100 g kg^{-1} of binder were used. Prints have high colour fastness to wet rubbing, and also the highest K/S values. On the basis of preliminary test results, it was decided that prints with higher concentrations of C.I. Pigment Red 146 must be double sided UV-cured.

The properties of double sided UV-cured prints were characterized (8 passes from the front and 4 passes from the reverse side). Table 6 shows the colour strengths of UV-cured, as the fastness properties, meanwhile Fig. 4 shows the stiffness characteristics of raw cotton fabric, thermally and the UV-cured samples.

As shown in Table 6, colour strength increases with an increasing amount of pigment dispersion in the printing paste. The colour strength values of UV-cured prints with 7.5 g kg^{-1} of pigment are similar to the colour strengths of the thermally cured prints, but they are higher in the case of prints with 15 g kg^{-1} and 30 g kg^{-1} of pigments. Colour fastness levels for washing and dry rubbing are surprisingly high (between 4 and 5), the exception being the A1 samples (40 g kg^{-1} of photoinitiator). Meanwhile wet rubbing fastness levels decrease while the pigment dispersion concentrations increase, independent of the photoinitiator concentration in the printing paste. These results can be explained by the fact that higher concentrations of pigment absorb more UV-light, which is reflected in a lower penetration degree of UV-light and hindering the formation of the free radicals [4,7,9,16]. Generally, it is accepted that higher amounts of pigments have an influence on decreasing the fastness levels of thermally cured prints [10].

It could be summarised, from results shown in Fig. 4, that when a lower concentration of photoinitiator (40 g kg^{-1}) was used (A group of samples), the

Table 5
Characteristics of one and double sided UV-cured prints with 15 g kg^{-1} of C.I. Pigment Red 146

One side UV-cured					Double sided UV-cured				
Code	No. of passes	Colour fastness to rubbing in warp direction		$K/S_{520\ nm}$	No. of passes		Colour fastness to rubbing in warp direction		$K/S_{520\ nm}$
		Dry	Wet		Top	Back	Dry	Wet	
A1	2	4	3	8.67					
	4	4	3	8.93	4	4	5	3	9.38
	6	4–5	3	9.54					
	8	4–5	3	9.65	8	4	5	3–4	9.70
B1	2	4	3	9.62					
	4	4	4	9.88	4	4	5	4	10.14
	6	5	4	9.94					
	8	5	4	10.12	8	4	5	4–5	10.81
C1	2	4	3	12.19					
	4	5	4	12.16	4	4	5	4	12.77
	6	5	4–5	12.59					
	8	5	4–5	12.83	8	4	5	4–5	12.98

Table 6
Colour fastness properties and colour strength of UV-cured prints (8 passes from the front, 4 passes from the back)

Code	Pigment conc. ($g\,kg^{-1}$)	$K/S_{520\ nm}$	Fabric stiffness ($cN\,cm^2$)	Colour fastness to washing at 60 °C	Colour fastness to rubbing in warp direction	
					Dry	Wet
A1	7.5	5.70	9.13	3–4	5	3–4
	15	9.70	11.74	3–4	5	3–4
	30	15.62	12.78	3	5	3–4
A2	7.5	6.66	11.75	5	5	4–5
	15	11.61	11.14	5	5	4–5
	30	14.54	13.72	4–5	5	4
A3	7.5	5.96	9.63	5	5	5
	15	12.24	11.10	5	5	4–5
	30	15.14	11.18	4–5	5	4–5
B1	7.5	5.65	16.60	5	5	4–5
	15	10.81	17.60	5	5	4–5
	30	13.72	15.48	4–5	5	4
B2	7.5	6.24	13.56	5	5	4–5
	15	9.77	13.95	5	5	4–5
	30	15.51	13.92	5	5	4–5
B3	7.5	6.05	10.44	5	5	5
	15	11.01	11.07	5	5	5
	30	14.99	12.39	4–5	5	4–5
C1	7.5	6.60	27.61	5	5	4–5
	15	12.98	24.16	5	4	4–5
	30	14.64	23.27	5	5	4
C2	7.5	5.88	17.15	5	5	4–5
	15	8.96	22.49	5	5	4–5
	30	15.14	18.79	4	5	4–5
C3	7.5	5.44	14.21	5	5	4–5
	15	10.67	14.07	5	5	4–5
	30	14.54	14.31	4	5	4

UV-cured samples with $150\,g\,kg^{-1}$ of binder in printing paste have the highest fabric stiffness. Expectations that an increase of binder ($200\,g\,kg^{-1}$) would give higher stiffness values did not meet expectations. At higher concentrations of photoinitiator (B and C group of samples with $60\,g\,kg^{-1}$ and $80\,g\,kg^{-1}$) the trend is the opposite. The fabric stiffness values were highest when the lower amount of binder was used ($100\,g\,kg^{-1}$), B1 and C1, respectively. UV-cured samples of the C1 group are hard to the touch, independent of pigment concentration, which could be attributed to higher rates of photopolymerization and through-cure.

The band at $967\,cm^{-1}$ presents a C–H deformation bending mode of the butadiene binders' double bonds, which decrease with UV irradiation of the pigment prints. The band area of uncured print is the highest and

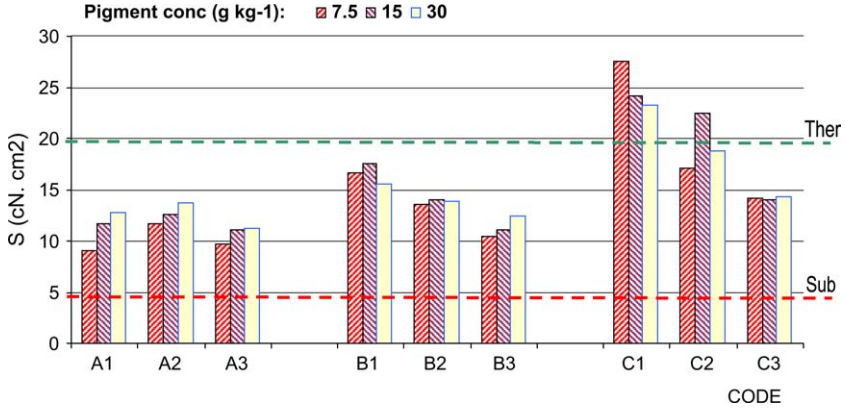

Fig. 4. Fabric stiffness S of double sided UV-cured samples with $7.5\,g\,kg^{-1}$, $15\,g\,kg^{-1}$ and $30\,g\,kg^{-1}$ of pigment, raw cotton fabric (Sub) and thermally cured prints (Ther – represents the averages value of T1, T2, T3 samples).

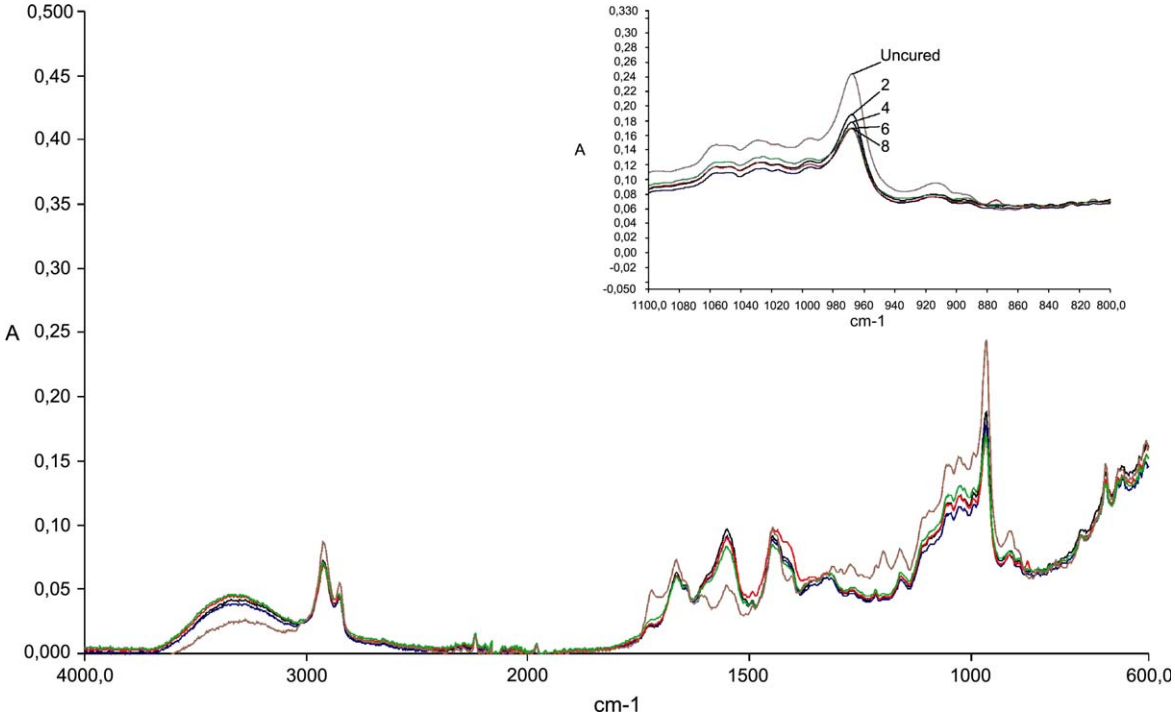

Fig. 5. ATR–FTIR spectra of A3 uncured and UV-cured prints (2, 4, 6 and 8 passes) with 40 g kg^{-1} of photoinitiator, 200 g kg^{-1} of binder and 7.5 g kg^{-1} of Red KF pigment dispersion in printing paste.

Table 7
The band areas (peak at 967 cm^{-1}) of uncured and UV-cured prints (8 passes from the front, 4 passes from the back)

Code	Pigment conc. (g kg^{-1})	UV-curing				
		Uncured	2 passes	4 passes	6 passes	8 passes
A1	7.5	18.63	18.22	17.71	16.32	15.67
	15	19.81	18.17	18.03	15.17	16.17
	30	19.54	17.15	17.59	16.47	16.60
A2	7.5	26.08	26.00	25.57	25.18	25.02
	15	26.49	26.07	25.26	25.07	25.00
	30	26.01	25.79	26.24	24.83	24.94
A3	7.5	25.13	25.18	25.24	25.38	26.02
	15	25.32	25.40	25.86	25.80	25.57
	30	25.50	24.40	24.89	24.64	24.94
B1	7.5	25.87	16.85	14.96	14.69	14.69
	15	26.85	17.19	15.88	14.26	15.35
	30	20.60	12.59	12.82	13.19	13.86
B2	7.5	25.99	20.33	19.31	17.18	16.94
	15	25.49	19.33	19.00	18.32	18.58
	30	25.78	19.03	17.87	17.63	16.66
B3	7.5	25.64	24.20	22.80	22.11	22.186
	15	25.31	23.77	22.50	21.64	20.74
	30	25.20	24.53	22.41	20.00	19.66
C1	7.5	26.66	20.06	19.81	17.77	17.84
	15	21.28	18.32	18.30	17.80	17.76
	30	24.208	20.57	19.54	17.80	18.30
C2	7.5	25.93	24.03	22.51	20.97	21.17
	15	26.03	24.02	22.44	17.45	17.40
	30	26.07	25.12	24.13	21.77	21.83
C3	7.5	25.08	25.50	25.78	25.71	25.86
	15	25.06	24.73	25.50	25.39	24.92
	30	24.90	25.36	25.85	25.99	26.19

decreases due to UV irradiation, which is shown graphically in Fig. 5 and numerically in Table 7. ATR-FTIR measurements show (Table 7) that the number of passes has an influence on the efficiency of UV-curing. The minimum 967 cm^{-1} bands area was achieved, in general, at 4–6 passes of UV lamp, although there were some unexpected exceptions. It is evident that the number of UV-lamp crossings has the greatest influence in the case of B1 samples, where 2 passes of UV lamp cause a dramatic decrease of band area, irrespective of 7.5 g kg^{-1}, 15 g kg^{-1} or 30 g kg^{-1} of C.I. Pigment Red 146 was added to the printing paste. The amounts of photoinitiator (60 g kg^{-1}) as butadiene–acrylonitrile copolymer binder (100 g kg^{-1}) were equal in the printing pastes of B1 samples. The possible explanation could be that the higher amounts of binder (150 and 200 g kg^{-1}) slow down the photopolymerization process.

4. Conclusions

UV-curing appears to be a very effective technique in the case of pigment prints on textile fabric. Our research showed promising results when C.I. Pigment Red 146 prints were cured with a prototype UV-Scanner. The overall characteristics of UV-cured pigment prints are surprisingly good. The results of present investigation shows that it is possible to achieve quality and durable UV-cured pigment prints on cotton fabric using a prototype UV scanner, multifunctional photoinitiator and, butadiene—acrylonitrile copolymer binder. However, the introduction of new pigment dispersions (Yellow, Blue, and Black) for optimizing the printing paste recipes, and curing conditions will be scope for further investigations.

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