



RESEARCH ARTICLE

PREPARATION OF NOVEL INK-JET INKS WITH UV-ABSORBERS, FOR DIGITAL PRINTING OF SYNTHETIC FIBRES

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ABSTRACT

Two UV-absorbers, Tinuvin P, 2-(2-hydroxy-5-methyl-phenyl)-2H-benzotriazole and Tinuvin 320 (benzotriazole of unknown formula) were used as active agents in this work. These were added in several amounts in water-based disperse ink-jet inks. The surface tension, pH, viscosity and conductivity of the prepared formulations were monitored over a period of 90 days in order to estimate the ink stability and the compatibility of the UV-absorbers with the ink constituents. The inks were used for ink-jet printing of polyester polyamide and polyacrylonitrile samples. Fastness and colour properties of the samples were measured. Quantitative determination of the UV-absorbers extracted of the printed sample was also made.

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INTRODUCTION

Ink-jet printing is one of the fastest growing of imaging technologies; compared to conventional printing methods offers unique advantages over conventional printing such as simplicity, lower production cost, reduced effluent waste, less water and energy consumption together with an unlimited design combinations which gives the possibility of producing innovative personalized finished articles (Fryberg, 2005; Daplyn and Lin L 2003; Holme, 2004; Dawson, 2006). During the last decades has been a growing interest in the preparation of water - based ink-jet inks for digital textile printing (Kang, 1991). Water-based ink-jet inks offer environmental advantages over their non-aqueous based counterparts, through reduced emissions of volatile organic compounds that are otherwise present in solvent-based formulations. With environmental considerations continuing to have ever-greater impact upon industry and legislation, textile producers have to carefully examine the environmental impact of the products used. Water-based inks contain, typically, 30-80% water as a proportion of the total mass of the ink, together with a water miscible organic solvent such as a monohydric alcohol

(methanol, ethanol, isopropanol), the colorant (e.g. disperse dye) and a surface active agent which, in the case of disperse dyes, acts as a dispersant for the dye in the aqueous medium (Kang, 1991; Yamamoto *et al*, 1997). Ink-jet inks must be carefully formulated to yield specific properties that enable optimum droplet formulation within the fine nozzles of a printing head for application at high speed. Such properties are dependent upon the pH, surface tension, viscosity and conductivity of the ink. Furthermore, inks designed for the ink-jet printing on textile materials need to satisfy various fastness criteria namely wash and light (Daplyn and Lin L 2003; Holme, 2004; Kang, 1991). UV-absorbers (UV-abs) have been utilized to protect polymeric materials from photodegradation caused by sunlight or ultra-violet rich artificial light. Recently, interest has developed in the application of UV-abs in the dyeing and printing of textile materials to protect their colour from fading. High light fastness of dyed/printed materials is important especially for automobile coloured upholstery, which can be exposed to direct sunlight and temperatures above 50°C (Kehayoglou, and Tsatsaroni, 1993). Various commercial UV-abs, mainly of the benzophenone (Baumann, 1985; Eichler *et al*, 1985; Ciba-Geigy, A.-G., EP 417040 A1, 1991; Showa Chemical Industries, Ltd, JP 58157881 A2, 1983) and Benzotriazole (Baumann, 1985; Reinert, 1991; Mitsubishi Kasei Corp. JP 02293477 A2. 1990; Rakuto Kasei Industrial Co. Ltd, JP 01124694 A2, 1989; Sandoz GmbH, Ger. Offen. DE 3706915 A1, 1987) and less of other classes

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(benzoic acid esters, hindered amines) (Sandoz GmbH, Ger. Offen. DE 3417782 Al, 1984) used directly for polyester, (Baumann, 1985; Reinert, 1991; Sandoz GmbH, Ger. Offen. DE 3417782 Al, 1984) wool, (Reinert, 1991; Ciba-Geigy, A.-G., EP 417040 Al, 1991) polyamide (Teijin Ltd, JP 49003469, 1974) and polypropylene (San&q GmbH, Ger. Offen DE 2453 146, 1976) fibres caused a light fastness improvement ranging from 0 to 2 units of the blue standard scale (The Society of Dyers and Colourists, 1990) for various dyes. Nevertheless an investigation of light fastness improvement by adding UV-absorber for dyes of lower light fastness is also interesting, especially when these are of low cost, brightness, etc., or applied to fabrics for other uses. An improvement in light fastness has been achieved either by addition of a UV-abs in the ink-jet process. The aim of this work is the preparation of stable disperse ink jet inks for digital printing applications with and without UV-abs as active agents was made. Physicochemical properties of the inks pH, conductivity, surface tension and viscosity were studied. The inks were used for digital ink jet printing of polyester, polyamide and polyacrylonitrile samples. Fastness properties and colorimetric coordinates of the prints are also performed.

Experimental

MATERIALS AND METHODS

Double-Jersey 100% polyester fabric white 190g/m² supplied by Greter-Jersey AG (Switzerland) as well as nylon 6.6 (78F46, 1.69 dtexpf) and commercially available polyacrylonitrile fabric supplied by KYKE HELLAS were used. The dye CI Disperse Blue 321 (Yorkshire Farben GmbH) was used as a disperse dye (Figure 1). Commercial grade 2-propanol (Fluka) and ethylene glycol (Riedel-de-Haen, Germany) were used. Alcoospers LFD (KYKE HELLAS SA), a condensation product of formaldehyde-sulphonic acid sodium salt was used as dispersing agent. Tinuvin P, 2-(2-hydroxy-.5-methyl-phenyl)-2H-benzotriazole and Tinuvin 320 (III, benzotriazole of unknown formula) were used as the UV-absorbers.

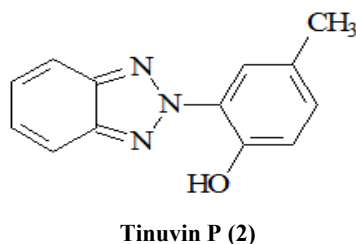
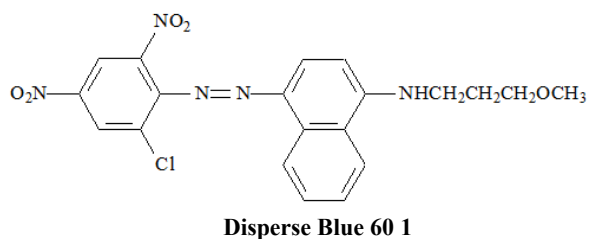


Figure 1. Disperse Blue 60 with Disperse Blue 321

Ink formulation

In Table 1 the water-based ink formulations containing 2% w/v dye, 78% v/v H₂O, a mixture of 2-propanol (IPA)/ethylene glycol (EG) 15/5 v/v as the miscible solvent, 2% w/v

dispersing agent and various % amounts of two uv-absorbers are given. The reference formulation, without active agent, is also included (Table 1). The mixture of ingredients was homogenized for 10 min using a Sonicator UP100H homogenizer at room temperature.

Table 1. Ink formulations prepared with and without active agent

No	Active Agent	
	Tinuvin P % w/v	Tinuvin 320 % w/v
1.1	-	-
1.2	0.1	-
1.3	0.2	-
1.4	-	0.1
1.5	-	0.2

1.1-1.5:Dye=disperse blue 321

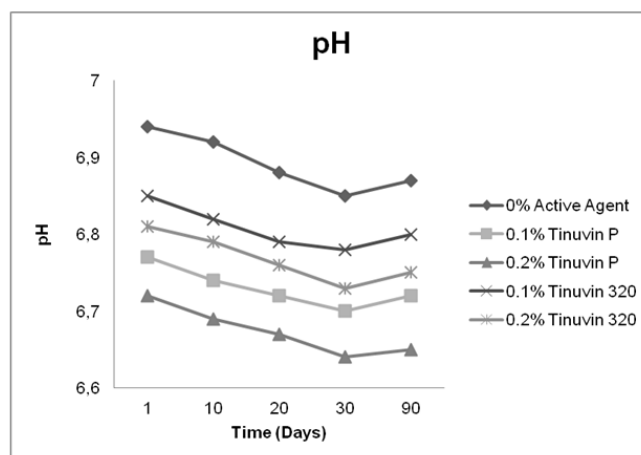


Figure 2. Plots of pH values vs time for the ink formulations

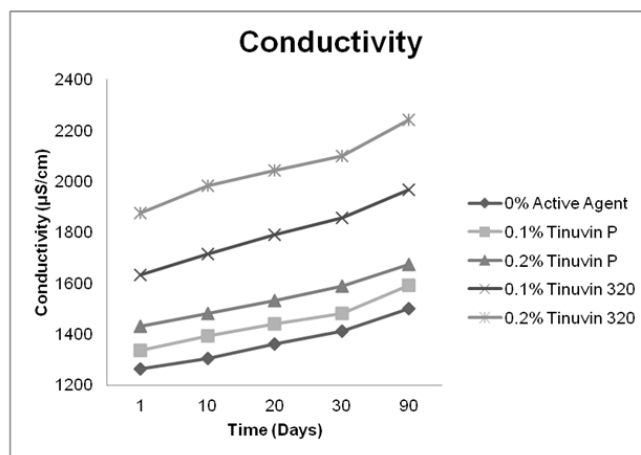


Figure 3. Plots of conductivity values vs time for the ink formulations

Surface tension conductivity, viscosity and pH measurements

Surface tension measurements were made using a KSV Sigma 70 tensiometer fitted with a De Nouy platinum ring. Conductivity measurements were performed using a Crison Conductimeter Basic 30 and pH measurements were made using a WTW Microprocessor 535 pH meter; viscosity measurements were made using a Viscostar plus+ H (Fungilab) viscometer. The above properties were measured at room temperature and atmospheric pressure for 1, 10, 20, 30 and 90 days for the inks containing Tinuvin P and Tinuvin 320 and the reference ink.

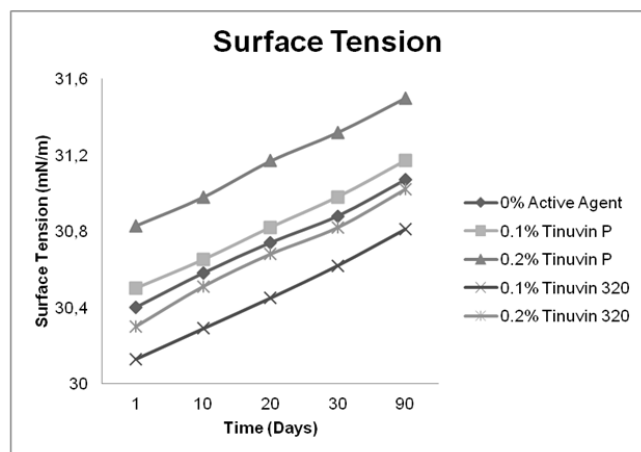


Figure 4. Plots of surface tension values vs time for the ink formulations

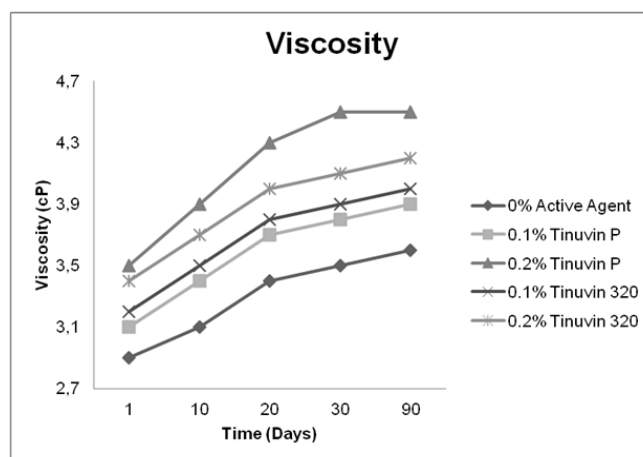


Figure 5. Plots of viscosity values vs time for the ink formulations

Table 2. Wash and light fastness values of the printed samples with/without active agent

Sample	Fibre	Light Fastness	Wash Fastness						
			Colour Change (CC)	Colour Staining (CS)					Wool
				Acetate	Cotton	PA	PES	PAC	
1.1	PES	5	5	5	5	5	5	5	5
1.2	PES	6	5	5	5	5	5	5	5
1.3	PES	7	5	5	5	5	5	5	5
1.4	PES	5-6	5	5	5	5	5	5	5
1.5	PES	6	5	5	5	5	5	5	5
1.1	PA	2	5	5	5	5	5	5	5
1.2	PA	2-3	5	5	5	5	5	5	5
1.3	PA	3	5	5	5	5	5	5	5
1.4	PA	2-3	5	5	5	5	5	5	5
1.5	PA	2-3	5	5	5	5	5	5	5
1.1	PAC	4	5	5	5	5	5	5	5
1.2	PAC	4-5	5	5	5	5	5	5	5
1.3	PAC	5-6	5	5	5	5	5	5	5
1.4	PAC	4-5	5	5	5	5	5	5	5
1.5	PAC	5	5	5	5	5	5	5	5

Ink-jet printing

Printing was performed on paper, with an Epson Stylus SX218 printer, followed by transfer printing on paper and then thermostabilization to polyester, polyamide and polyacrylonitrile fabric for 30 sec. The printed samples were then rinsed in cold water and reduction cleared using a solution comprising 2g/l sodium dithionite and 1.5g/l sodium carbonate at 60°C using a liquor ratio of 20:1 for 20 min. The reduction cleared samples were rinsed in cold water and allowed to dry in the open air.

Fastness tests

Wash fastness of the prints and dyeings was carried out according to BS1006:1990CO2 test, whereas light fastness was carried out according to BS1006:1990BO2 test (The Society of Dyers and Colourists, 1990).

Colour measurements

The reflectance values of the prints were measured using a Macbeth CE 3000 spectrometer under D65 illumination, 10 standard observer with UV and specular component included. The CIE1976 L*a*b*C* H* coordinates and the K/S values were calculated from the reflectance values at the appropriate λ_{max} (λ_{max} 620nm for Disperse Blue 321).

Identification and determination of the UV-absorbers adsorbed on the printed samples

Quantitative determination of the UV-absorber of the printed sample was made after extraction of the two active agents with polyethylene glycol 200 and measuring spectrophotometrically the extract at 341nm (λ_{max} of Tinuvin P) and 343nm (λ_{max} of Tinuvin 320). Four extraction cycles (1g sample in a liquor ratio 1:10, 30min, 130°C for polyester and 98°C for polyamide and polyacrylonitrile) were carried out for the quantitative recovery of Tinuvin P and Tinuvin 320 active agents from the fabric.

RESULTS AND DISCUSSION

Physical Properties of the Inks: Monitoring a formulation over a period of time is a widely accepted method for evaluating dispersion stability (Kang,1991; Yamamoto *et al*, 1997). In this study the surface tension, pH, viscosity and conductivity of the formulations prepared were monitored over a period of 90 days for the digital printing inks containing the uv-absorbers Tinuvin P and Tinuvin 320. Figure 2 show the pH values of the inks prepared with Disperse Blue 321 against time of storage. From the above figures can be concluded that the blue formulation without active agent 1.1 (Table 1) have generally neutral pH. This is due to the fact that the blue dye has been milled and processed in neutral pH. Addition of the UV-abs Tinuvin P and Tinuvin 320 in the formulations 1.2 -

1.5 shows lower values of pH compared to the reference formulation 1.1 (without active agent) due to the acid character of the benzotriazole followed by small decrease for the rest of the time. Addition of both UV-abs in the dye formulations results in a slight decrease of pH values compared to the reference formulation 1.1 (Figure 3) followed by small decrease over the period of storage time. Given that acceptable pH values for ink-jet inks are near the neutral region the formulations 1.1-1.5, with pH values 6.9-6.6 (Figure 2) are in this area. Figure 3 show the effect of storage time on the conductivity of the inks. The presence of Tinuvin P and Tinuvin 320 results in a increase in conductivity values of both two inks compared to the reference ink which already present high conductivity values (Figure 3).

prepared with and without the additives are in this range making them suitable for ink jet applications. Figure 5 show the effect of storage time on viscosity. It is generally accepted that ink jet inks should have a viscosity lower than 2cP to be suitable for digital printing application although it is not unknown for such inks to have a viscosity up to 3cP (Schulz *et al.*, 2003; Siemensmeyer *et al.*, 1993). All inks with and without the active agents showed an initial sharp increase in viscosity followed by a stable increase in viscosity after 20 days of storage. This temporary sharp increase can be attributed to the presence of the dispersing agent present in all inks, Alcosperse LFD, which is a sodium salt of a polysulphonated derivative full dissociation of which produce more anionic repulsions and thus causing an increase in viscosity as a function of storage

Table 3. Color coordinates L*, a*, b*, C*, H* and colour strength K/S values of the printings with/without active agent

Formulation	Fibre	K/S	L*	a*	b*	C*	H*
1.1	PES	0.445	73.77	-3.57	-15.65	16.05	257.14
1.2	PES	0.528	71.37	-6.22	-19.03	20.02	251.90
1.3	PES	0.722	68.14	-3.33	-20.15	20.43	260.61
1.4	PES	0.637	69.37	-6.73	-21.53	22.56	252.63
1.5	PES	1.396	59.49	-0.813	-29.58	30.67	254.63
1.1	PA	0.541	74.09	-6.98	-13.79	15.46	243.15
1.2	PA	0.547	73.43	-11.47	-15.53	19.30	233.55
1.3	PA	0.627	71.55	-12.61	-17.43	21.51	234.12
1.4	PA	0.581	72.53	-11.37	-15.73	19.41	234.13
1.5	PA	0.693	71.94	-7.80	-16.50	18.25	244.69
1.1	PAC	0.171	82.47	-2.41	-10.48	10.75	257.04
1.2	PAC	0.196	81.34	-2.48	-10.84	11.12	257.11
1.3	PAC	0.357	74.37	-3.68	-9.84	10.51	249.51
1.4	PAC	0.292	76.35	-3.34	-8.29	8.94	248.08
1.5	PAC	0.466	71.75	-4.52	-12.75	13.53	250.47

Table 4. Amount (mg/L) of Tinuvin P adsorbed on polyester (PES) polyamide (PA) and polyacrylonitril (PAC) printed samples

Tinuvin P % w/v	POLYESTER	POLYAMIDE	POLYACRILONITRIL
	AMOUNT mg/L	AMOUNT mg/L	AMOUNT mg/L
0.1	133	108	111
0.2	271	201	211

Table 5. Amount (mg/L) of Tinuvin 320 adsorbed on polyester (PES) polyamide (PA) and polyacrylonitril (PAC) printed samples

Tinuvin 320 % w/v	POLYESTER	POLYAMIDE	POLYACRILONITRIL
	AMOUNT mg/L	AMOUNT mg/L	AMOUNT mg/L
0.1	126	103	107
0.2	235	192	204

This can possible be attributed to the presence of ionized phenolic hydroxyl groups of benzotriazole which may give rise to higher conductivity values compared to the reference ink 1.1. Figure 4 show the surface tension values of the inks prepared against storage time. Surface tension values are increasing with time until day 7 remaining then relatively stable for the rest of the time for all formulations 1.1-1.5 (Fig. 4). This slight increase in surface tension can be attributed to the higher surface activity conferred by the two hydrophobic additives of Tinuvin P and Tinuvin 320. Surface tension increase is at the benefit of the adhesional wetting of the ink. Additionally this surface tension stability is a very good indication for the stability of the digital printing inks (Holme, 2009). Typical surface tension values of commercial ink-jet inks for textile printing are in range 21-48 or 30-60 mNm⁻¹ (Holme, 2009; Schulz *et al.*, 2003; Siemensmeyer *et al.*, 1993). As figure 4 show the surface tension values of all inks

time. The initial and final viscosity values of the inks prepared lie between the generally accepted viscosity limits for the digital printing inks making all formulations suitable for ink jet applications. All ink formulations show stable viscosity increase values after 20 days of storage. This stabilization is again a good indication for the stability of the ink formulations.

Determination of the UV-abs from the digitally printed samples

Tables 4, 5 show the amounts of active UV-abs extracted from the printed polyester polyamide and polyacrylonitrile samples after four extraction cycles with polyethylene glycol 200 as a solvent. A number of solvents was tested for the extractions but polyethylene glycol was finally chosen because of its maximum effectiveness in solubilising both active ingredients.

The amount of extracted Tinuvin P from polyester and polyamide was higher to the extracted amount of Tinuvin 320. The extracted amount of active agents show that about 10% of active agent UV-abs over mass of fabric is deposited by the digital printing head on the textile substrate. This amount is responsible for the UV protection activity of the digitally printed samples.

Colour measurements of digitally printed samples

The ink formulations were applied to polyester polyamide and polyacrylonitrile fibres by transfer printing followed by thermostabilization. Almost 100% transfer of the printed ink from the paper to the textile substrate is taking place under the transfer conditions employed. It is believed that the transfer of the ink from the paper to the textile substrate is being made not through a conventional sublimation transfer mechanism but through a melt or wet transfer mechanism where a thin layer from the digitally printed paper is being deposited on the polyester or polyamide substrate.

Table 3 shows the relevant colorimetric data and colour strength values for the polyester polyamide and polyacrylonitrile printings. L*, a*, b*, C*, H* co-ordinates and the K/S values were calculated from the reflectance values at the appropriate λ_{max} (620nm for C.I Disperse Blue 321).

From table 3 the following are concluded: The presence of two active ingredients in the blue digital ink formulation has resulted in a significant dye increase as this is expressed by the higher K/S values of the blue inks containing Tinuvin P and Tinuvin 320 compared to the blue reference inks. This can be explained in terms that the active ingredients, may be capable to form hydrogen bonds between their hydroxy group and the amine groups or the N-heteroatom of the disperse blue dye respectively and thus increasing the solubility of the sparingly soluble disperse dye in the aqueous face and thus possibly acting as a "carrier", promoting higher dye uptake by the polyester, polyamide and polyacrylonitrile fibre compared to the reference digital printing ink. This higher uptake was especially noticed with the bigger and bulkier blue disperse dye which posses two free amine groups and a long aliphatic chain in the molecule making the dye bulkier and more difficult to penetrate the rigid polyester fibre. It can be claimed that Tinuvin P and Tinuvin 320 with the presence the phenolic ring in their structure and their low water solubility posses characteristics of commonly known phenol based carriers for polyester dyeing (Arcoria *et al*, 1985).

Fastness properties

The wash and light fastness properties of the digitally printed polyester and polyamide samples are given in Table 2. The wash fastness for all printed samples after the reduction clearing treatment performed is excellent with no staining of the adjacent fibres of the multifibre strip (values 5) and no color change of the digitally printed samples (values 5). Light fastness of the printed polyester (values 5-7) were very good to excellent, in polyacrylonitrile samples were medium to very good (values 4-6), while polyamide samples presented a low (value 2-3) light fastness value. Addition of the UV-abs in the ink formulations resulted, in almost all cases, in an improvement in light fastness ranging from 0.5-2 units on the blue standard scale. Comparing the effectiveness on the light fastness of the two UV-abs, it is evident that Tinuvin P showed

the best results, and Tinuvin 320 the worst. Nevertheless, the addition by a higher amount of Tinuvin P and Tinuvin 320 caused an improvement in light fastness. Comparison between the samples, showed that the light fastness of the polyester and polyacrylonitrile were higher (Crews and Clark, 1990).

Conclusions

The UV-abs Tinuvin P and Tinuvin 320 were successfully incorporated in water based ink-jet inks prepared for the ink-jet printing of polyester polyamide and polyacrylonitrile fabrics. The physical properties pH, surface tension, conductivity and viscosity of the inks were monitored over a period of one year and their stability and suitability for ink -jet printing was confirmed. The inks were used for the digital printing of paper, transfer on polyester, polyamide polyacrylonitrile and fabrics and thermostabilization. Colour and wash- light fastness measurements of the prints were made and presented. Quantitative determination of the two agents adsorbed on the fibre was also made and the results regarding the UV protection activity of the textiles were excellent. Addition of the UV-abs in the ink formulations resulted, in almost all cases, in an improvement in light fastness. Comparing the effectiveness on the light fastness of the two UV-abs, it is evident that Tinuvin P showed the best results, and Tinuvin 320 the worst.

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