



# Surface chemical analysis of the effect of curing conditions on the properties of thermally-cured pigment printed poly (lactic acid) fabrics



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## ABSTRACT

Colouration of poly (lactic acid) textile materials using pigments is particularly interesting because of the commercial importance of pigment prints and the sensitivity of poly (lactic acid) fibres to aqueous treatment; however the processing requires optimisation because of the fibre's heat sensitivity. In this study, poly (lactic acid) fabrics were screen pigment printed and thermally cured at different temperatures, ranging from 120 °C to 150 °C, and curing times, from 3 min to 5 min. Surface sensitive X-ray Photoelectron Spectroscopy (XPS) was used to probe the nature of the outer 1–10 nm of the untreated and heat treated poly (lactic acid) surfaces, in order to characterise the surface functionalities and elemental composition. In addition, the effect of curing temperature and time on the performance characteristics of pigment printed poly (lactic acid) fabrics in terms of colour fastness, burst strength, abrasion resistance, Kawabata Evaluation System for Fabrics (KES-F) parameters and surface morphology were evaluated and optimum curing conditions for the pigment printing of poly (lactic acid) fabrics were established.

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## 1. Introduction

Poly (lactic acid) (PLA) is a versatile, biodegradable, aliphatic thermoplastic polyester which can be derived from 100% renewable sources, such as corn and sugar beets [1]. Polyesters, mainly polyethylene terephthalate (PET), are used extensively in apparel manufacturing and account for almost 40% of world textile consumption. The uses of polyesters are increasing rapidly; however, fossil fuels consumption in the PET manufacturing process and its relatively non-biodegradable nature have been growing environmental concerns. As the world is becoming increasingly sensitive to the protection of the environment, there has been growing interest in using biodegradable materials such as PLA [2].

More than 50% of the total global printed textiles are processed by pigment printing, and it is the single most important and widely used printing method for textile materials because of its simplicity, relatively “clean” processing, and environmentally friendly aspects

[3]. Moreover, there are some distinctive advantages of this technology, such as the quality of prints, ability to apply on most types of fibres and no washing off requirements after printing, which have made this technology very attractive to textile manufacturers [4]. Unlike dyes that are absorbed into the fibres and fixed through interactions or reactions specific to the dyes, insoluble pigments do not have any affinity for the fibres. They are attached by being incorporated into a film of a suitable binding agent on the surface of the fabrics and being fixed after printing at a suitable curing temperature. The paste for pigment printing typically consists of pigment, binder, synthetic thickener and other printing auxiliaries. Thermal energy initiates cross-linking of the binder at a suitable curing temperature, which binds the pigment particles onto the fibre surface [4,5]. Although pigment printing has been used widely, some limitations still exist with this method such as high temperature curing, relatively stiff handle and poor rubbing fastness. Accordingly there has been growing interest to reduce the curing temperature of pigment printing not only to save on energy costs but also to lessen the damage of fabrics [6].

PLA fabrics can be processed in a similar way to PET fibres using conventional dyeing and printing methods; however, the processing temperatures need to be reduced as PLA has a lower melting

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point (130–175 °C) than that of PET (254–260 °C) [7,8]. Moreover, a modified dyeing method is required to dye PLA fabrics as it has low affinity for conventional water soluble dyes [9]. In this study, the effect of thermal curing conditions on the properties of pigment printed PLA and their chemical and physical effect on the outer surface of the printed PLA fabrics was investigated. The surface sensitive X-ray Photoelectron Spectroscopy (XPS) technique was used to probe the nature of the outer 1–10 nm of the untreated and heat treated PLA surface, in order to characterise the surface functionalities and elemental composition and identify optimum curing conditions for pigment printing of PLA fabrics. The prints produced using different curing parameters, such as temperature and time, were assessed in terms of colour fastness, burst strength, abrasion resistance and the KES-F mechanical properties of the fabrics.

## 2. Experimental

### 2.1. Materials

In this investigation, 100% PLA single jersey knit pique fabric, 268 g/m<sup>2</sup>, purchased from Valuable Enterprise, Taiwan, was scoured at 60 °C for 20 min in a solution containing 2 g/L soda ash and 0.5 g/L Kieralon Jet B Conc [7] and air dried. Lyoprint PBA, a self-cross-linking acrylic binder, Lyoprint PTF, an acrylic polymer thickener and Lyoprint PFL, a methylol-melamine fixing agent were supplied by Huntsman, USA. 38% ammonia was supplied by Fisher Scientific and Minerprint Red BTL was supplied by Quality Colours, London, UK.

### 2.2. Methods

#### 2.2.1. Pigment print paste preparation and printing

To prepare the print paste, 5 g/kg 38% ammonia was added to deionised water and Lyoprint PBA binder was added to that solution as required depending on amount of pigments to be used, as shown Table 1. Lyoprint PFL (10 g/kg) was then mixed into the formulation and the solutions were stirred for 5 min. Finally, 15 g/kg Lyoprint PTF was added to the mixture and the required viscosity of print paste was achieved by stirring for 10–15 min using a high speed automatic stirrer. Minerprint Red BTL pigment was added to print paste to give a pigment content of 7.5 g/kg, 15 g/kg and 30 g/kg as required, and stirred vigorously to ensure homogeneous mixing of the colours with the print paste. The pH of the print paste was maintained in the range of 7–7.5.

Pigment printing onto PLA fabrics was carried out using a hand screen of 62/cm mesh and 10 mm squeegee diameter; all the samples were dried at 90 °C for 5 min using a Werner Mathis laboratory dryer. The pigments were fixed onto the fibres using curing temperatures, ranging from 120 °C to 150 °C, and curing times, from 3 min to 5 min. The fabric samples for XPS analysis were printed as described but using the print paste for 3% shade with the pigment addition omitted.

**Table 1**  
Printing paste composition for pigment printing of PLA.

Pigments and chemicals	Formulation compositions		
	0.75%	1.5%	3%
Minerprint Red BTL (g/kg)	7.5	15	30
Lyoprint PBA (g/kg)	60	100	130
Lyoprint PFL (g/kg)	10	10	10
Lyoprint PTF (g/kg)	15	15	15
Ammonia (g/kg)	5	5	5

### 2.2.2. XPS analysis

A Kratos Axis system spectrophotometer was used to perform the XPS analysis. The fabric samples were cut from the middle of the specimen and attached to the sample holder using a double sided tape. The samples were irradiated using monochromatic Al K $\alpha$  X-rays (1486.6 eV) with a power of 150 W and wide scan spectra were recorded with pass energy of 160 eV from which the surface composition (C, O and N) was determined. High resolution Carbon (1s) and Oxygen (1s) spectra were recorded with pass energy of 40 eV and the binding energy (BE) values were calculated relative to the Carbon (1s) photoelectron peak at 285.0 eV. All samples were analysed in duplicate and data analysed using the CASA XPS software [10].

### 2.2.3. Colour fastness measurement

Colour fastness to washing was carried out according to BS EN ISO 105 C06 C2S, by treating the printed fabrics in a solution containing 4 g/L ECE detergent with 1 g/L sodium perborate and 25 stainless steel balls, adjusted to pH 10.5, at 60 °C for 30 min. The fabrics were rinsed subsequently in running water at ambient temperature and air dried at room temperature prior to colour analysis. SDC multi-fibre fabrics were used as adjacent fabrics in this test [11].

The colour fastness to rubbing of printed fabrics, both dry and wet rubbing, was measured according to the BS EN ISO 105-X12:2002 test method using a crock meter.

The colour fastness to light was measured following the BS EN ISO 105-B01:1999 test method and graded on 1–5 after the exposure of test samples to simulated day-light using Xenon Light fastness tester.

### 2.2.4. Measurement of colour strength (K/S) and colour difference ( $\Delta E$ )

The colour strength (K/S) was calculated from reflectance measurements using the Kubelka–Munk equation (equation (1)):

$$(K/S)_\lambda = (1 - R_\lambda)^2 / 2R_\lambda \quad (1)$$

where  $K$  is the absorption co-efficient,  $S$  is the scatter co-efficient and  $R$  is the reflectance expressed as a fractional value at wavelength  $\lambda_{\max}$ . A Datacolor Spectroflash 600 was used to measure  $K/S$  and CIE  $L^*a^*b^*$  values and the mean value was calculated from the average of four measurements. Colour measurements were taken before and after washing of printed samples and colour difference values were also noted.

### 2.2.5. Measurement of burst strength

The burst strength of the pigment printed samples was measured using a TruBurst Burst Strength Tester Model 500/610 according to BS EN ISO 13938-2:1999. The area of the samples to be tested was clamped over an elastic diaphragm and an increased air pressure was applied to the underside of the diaphragm until the specimen burst. The pressure required to rupture the PLA samples was taken as the burst strength. Six measurements were taken per sample and the mean value was calculated from the average of these six measurements.

### 2.2.6. Measurement of flat abrasion resistance

The Martindale abrasion tester was used to measure the flat abrasion resistance of pigment printed fabrics according to the BS EN ISO 12947-2:1999 procedure. Four circular specimens were cut from each fabric and placed in the specimen holders and were rubbed against an abrasive media under a 12 KPa load. The abrasion resistance of those specimen fabrics were determined from the number of cycles to the breakdown of single yarn in a specimen, and the mean value was calculated from four measurements.

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