



## Comparative Evaluation of Novel Heterocyclic Azo Disperse Dyes on Poly(lactic Acid) and other Hydrophobic Fibers

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### Abstract

Some novel heterocyclic azo disperse dyes were synthesized from 2-aminothiophenes which were prepared from cyanoacetates and a range of 1,3- dicarbonyl compounds such as, o-acetoacetotoluidide, 4-chloroacetoacetanilide, and o-acetoacetanilidide using the Karl-Gewald one-pot technique. The 2-aminothiophenes were the major precursors for the series of dyes which were used in the work. The dyes were applied to various hydrophobic fibers such as poly(lactic acid), polyester and nylon. The dyes gave results comparable to those of conventional disperse dyes of similar composition. The exhaustion and light fastness and other relevant data were particularly good on the poly(lactic acid) fiber in contrast to some widely reported data. Dyeing behaviour of (PLA) fibre also showed that at slightly higher temperature, the colour strength of PLA was higher as compared to other fibers. The dyes are therefore well recommended especially for application on PLA (poly(lactic acid)) and polyester, where it's cost of production from simple and available starting materials are an obvious and attractive advantage.

**Keywords:** heterocyclic azo dyes; poly(lactic acid); hydrophobic fibers; dyebath exhaustion; fastness properties.

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Received: 26<sup>th</sup> May, 2021

Accepted: 19<sup>th</sup> Aug, 2021

Published Online: 12<sup>th</sup> Nov, 2021

### Introduction

Poly(lactic acid) (PLA) is an aliphatic polyester based on renewable materials. The fermentation of corn, sugar or vegetables provides mostly L-Lactic acid, whose polymerisation produces PLA. Polyester fiber (PET) is a petroleum-based fiber, its life cycle is claimed to cause a global warming (Ajioka *et al.*, 2011). For this reason, a greener polyester fiber has been developed for textile production. Poly (lactic acid), PLA, is considered as the eco-polyester fiber. It is derived from renewable materials e.g.

corn, sugar and starch. Fermentation of the materials yields lactic acid which is then used for PLA polymerization, (Jantip and Potjanart, 2011).

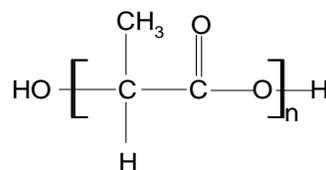
Bio-plastics have gained tremendous attention, due to the increasing environmental pressure on global warming and plastic pollution. Among them, poly (lactic acid) (PLA) is both bio-based and biodegradable, which has been widely used in many disposable packaging applications. The global market for PLA demand doubles every 3–4 years (Jim and Bowen, 2020).

Compared to traditional petroleum-based plastics, PLA is more expensive and usually has less mechanical and physical properties. A recent study by Ozan and Akbar (2010) also established the fact that commercialization of D(-) lactic acid and its polymer PDLA have the potential to improve the mechanical and thermal characteristics of PLA (e.g. by forming stereocomplex PLA) for applications in high-end markets. However, the usage of PLA in some other applications is still limited.

An attempt to use PLA as a textile fiber with the aim of replacing PET, fiber with this green polyester fiber has been a challenge and an interesting prospect for chemists for some time. PLA fibers are more hydrophilic than PET, have a lower density, and have excellent crimp and crimp retention. These PLA fibers tend to be stable to ultra-violet light resulting in fabrics that show little fading. They also offer low flammability and low smoke generation characteristics (Gupta *et al.*, 2007).

PLA fibers have for some time been seen to be a clearly possible replacement for polyester. Since PLA is a hydrophobic fiber, disperse dyes that have a high affinity for other hydrophobic fibers such as PET are expected to have affinity for PLA. PET is commonly dyed at 130°C, which is above the melting point of PLA. PLA is generally dyed at 110°C and for shorter lengths of time. Excessive heat or extending the dyeing time will cause degradation and loss of tensile strength, molecular weight and elongation at break point (Suesat *et al.*, 2003)

Poly(lactic acid) (PLA) is the first melt-processable, renewable, sustainable and biodegradable natural-based synthetic fibre. According to Ozan *et al.* (2020) PLA fibre, as an aliphatic polyester, can be dyed with disperse dyes. Apart from the limited number of commercial disperse dyes, disperse dye exhaustion on PLA is generally lower than that on polyethylene terephthalate (PET).



**Fig. 1.** Chemical structure of PLA

The former reaction route needs high vacuum, high temperature and a long reaction time. The latter route involves milder conditions and is consequently preferred industrially. Dimerisation of lactic acid can be controlled to produce three different stereochemical forms of lactide, L-, D-, and meso-lactides. Subsequent ring opening polymerisation of the three lactides produces PLA with different properties. Its use as a fiber in the textiles area has also been extensively studied (Gupta *et al.*, 2007). However, in order to introduce PLA successfully into the textile market it needs to be readily dyeable, but to date only a few dyes are available and even fewer articles have been published on the subject (Sriumaoum *et al.*, 2012).

Recently, substrates based on Polylactide (PLA) fibers have received attention worldwide because of their attractive advantages (Vorabodee *et al.*, 2012), including their availability from a naturally renewable carbohydrate resource. PLA is also a polyester that is subject to high ultraviolet (UV) penetration into fabric composed of its fibers. Also, dye performance is normally different on PLA vs on polyethylene terephthalate PET. For instance, many commercial disperse dyes have exhaustion levels <50% on PLA but have exhaustion levels over 90% on PET (Yang & Huda, 2003). Light fastness levels are also quite different on PLA vs PET. Therefore, in addition to developing alternative dyeing methods for PLA (Lunt & Bone, 2001) (Jamshidi *et al.* 1988), the synthesis of dyes that take the structural nature of PLA into consideration is essential to improving dye exhaustion and light fastness.

An example of the alkaline preparation process is peroxide bleaching used to whiten

the cotton component in the PLA/cotton blend, resulting in a substantial reduction of the fiber strength. Therefore, the preparation, dyeing and finishing processes for PLA should be milder than those used for PET. It is suggested to process PLA fiber at lower alkalinity, processing temperature and time (Phillips, 2004).

Although both polymers are dispersed dyeable, the high dyeing temperature employed for P.E.T must not be used for PLA to avoid degradation of the polymer, manifested as substantial losses in molecular weight, tensile strength, and elongation at break point (Phillips, 2004). In the relatively few studies that have been published on the colouration of PLA fibers, a common theme has been the application of extant commercial dyes with very mixed results obtained.

PLA fibres can be dyed with disperse dyes, just like PET fibres, although modified wet processing processes are employed. Finding the best conditions and methods for wet processing and after-care applications for PLA based textile products will increase the performance of this natural-based melt-spinnable PLA fibre (Ozan Avinc and Akbar Khoddami 2010)

#### **Applications and uses of polylactides**

Several factors have led to the development of polylactic acid (PLA) fibers for textiles application. The fiber potential of PLA was established in the 50s when PLA biodegradable and bioabsorbable surgical sutures were produced (Lipinsky and Sinclair 1986; Shon, 1991). Since then, lower cost processes that eliminate gypsum by-products have catalyzed the use of significant amounts of PLA in an array of new applications that include biodegradable forks, spoons, and knives; biodegradable and environmentally safe plastic packaging with good mechanical and thermoplastic properties; formulations for paper coatings; adhesives for high quality recycled paper; mold-resistant prosthetic devices; and slow release carriers and capsules for herbicides and pesticides (Adedokun, 2011; Ajioka, 1995). Lactic acid is produced naturally during metabolism in

most living organisms including humans and is used in the manufacture of cheese, pickles, yoghurt, and food preservation and acidulation (Louis and Annacleta, 2001). The primary raw materials for commercial production include corn, whey, sweet sorghum, wheat, barley, sugar beets, and sugar cane. Other raw materials for lactic acid include petroleum, coal, and natural gas liquids (Adedokun, 2011; Giita Silverajah, 2012). An understanding of the dyeability of PLA is essential to production of aesthetically pleasing textiles. In an earlier paper by Kameoka *et al.* (1997), they published an article that established the optimum conditions for producing the vibrant colours needed for commercial applications were 60-minute dye applications at 90 – 100°C at pH 5. A dye exhaustion of 88% was achieved under these conditions.

The present study has been carried out to evaluate the performance of some newly synthesized heterocyclic azo dyes on PLA. The study was also extended to examine the application of the dyes on other hydrophobic fibers as well such as PET, Nylon 6,6. Nine disperse dyes were used in the study. These heterocyclic disperse dyes were synthesized in our laboratories (Adedokun, 2011) and in addition, the study sets out to evaluate their dyeing properties and other relevant parameters.

#### **Materials and Methods**

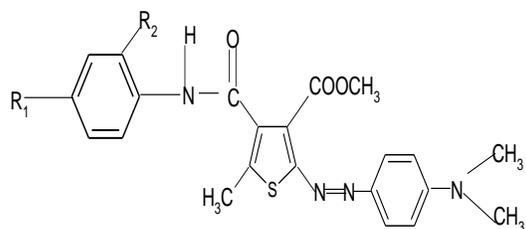
##### **Materials**

100% PET (75 denier/36 filament) fabric and Polylactic acid non-woven material and Nylon 6,6 were used. The PLA material was used as is.

##### **Structures of Dyes used in the Study**

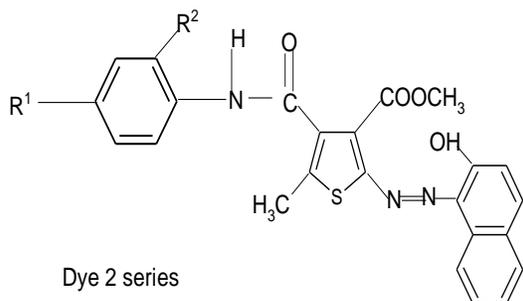
Three series of dyes were used. They were earlier synthesized in our laboratory and labelled as Series 1 to 3. While the three amine (diazo) components were essentially the same, three different coupling components were applied producing a total of nine (9) test dyes. The couplers were N,N-dimethylaniline, 2-naphthol, bis-hydroxyethyl aniline.

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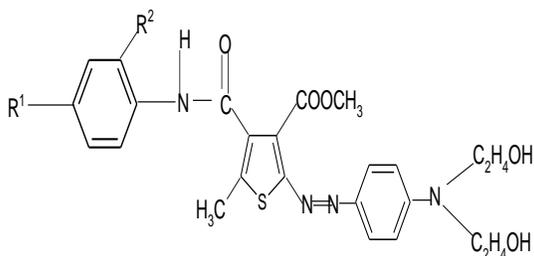
Dye 1 series

- Dye (1a)  $R_1=H$ ,  $R_2=CH_3$   
 (1b)  $R_1=H$ ,  $R_2=OCH_3$   
 (1c)  $R_1=Cl$ ,  $R_2=H$



Dye 2 series

- Dye (2a)  $R_1=H$ ,  $R_2=CH_3$   
 (2b)  $R_1=H$ ,  $R_2=OCH_3$   
 (2c)  $R_1=Cl$ ,  $R_2=H$



Dye 3 series

- Dye (3a)  $R_1=H$ ,  $R_2=CH_3$   
 (3b)  $R_1=H$ ,  $R_2=OCH_3$   
 (3c)  $R_1=Cl$ ,  $R_2=H$

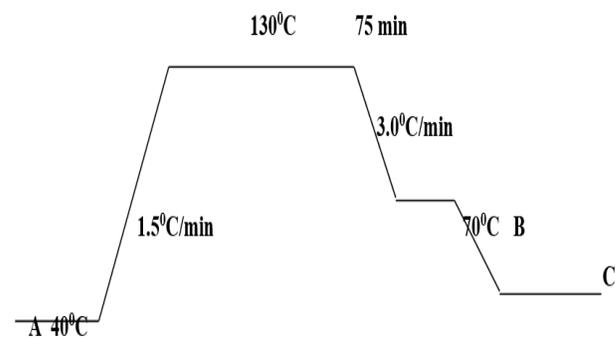
### Preparation of stock and dyebath solution

The stock solutions were prepared by weighing out 0.5g dye powder, and adding it to a beaker containing 100ml water and the dispersing agent 0.75g/L. The volume was brought to 250ml and thoroughly shaken. The dispersion was then further diluted to the final volume of 500ml. This gave a solution

strength of 1g/l (0.1 %) which was found to be a suitable concentration and was used throughout the experiments.

### Dye Application [Polyester Materials]

Dispersion of the dyes was produced by grinding in the presence of dispersing agent TRITON-X (1g/l). The wetted-out polyester fabrics was added to the prepared dyebaths (liquor ratio 50:1), which was adjusted to pH 4.75 (with acetic acid) and containing the dispersants and sequestrant (EDTA, 0.5%). Dyeing was performed by raising the dyebath temperature from 70 to 130°C at 1.5degC/min, holding at this temperature for 75 min and rapidly cooling to 70°C. The dyeings were rinsed with cold water and reduction cleared (1g/l sodium hydroxide, 1g/l sodium hydrosulphite, 10 min, 80°C) or alkali cleared (20g/l sodium carbonate, 15 min, 80°C) as required.



- A---Dye bath preparation  
 B---Washing (hot and cold)  
 C---Reduction clearing/Alkali clearing

**Figure 2: HT dyeing profile of polyester used in this study**

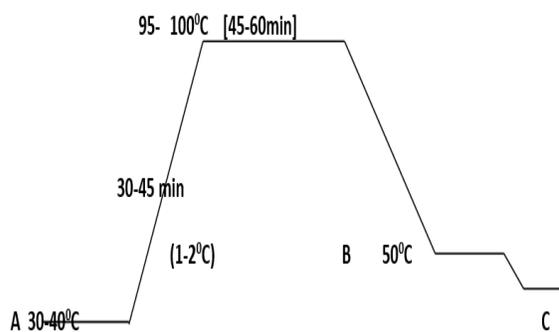
### Dyeing and Reduction Clearing Procedure

Dyeing operations were performed by means of a high temperature dyeing method in the AHIBA NUANCE [DataColor International USA], high temperature dyeing machine. Fabric 5.0g was dyed at a liquor to goods ratio of 50:1 in a dyebath containing disperse dye (2.0% o.w.f) and TRITON-X non-ionic surfactant (1g/L) as dispersing agent. The pH of the dyebath was adjusted to ~5.0 using AcOH/NaOAc and then dyeing commenced at a dyebath temperature of 40°C, which was ramped up to the maximum temperature at a

rate of 2°C/min. These maximum temperatures were 130°C and 100°C for PET and PLA/Nylon 6,6 fibers respectively. After holding at the maximum temperature for 40 mins, the dyebath was cooled back to 70°C at a gradient of 2.5°C/min. Fabric was then removed from the dyebath, followed by a reduction clearing procedure as already described above.

### Dyeing of PLA/Nylon 6,6

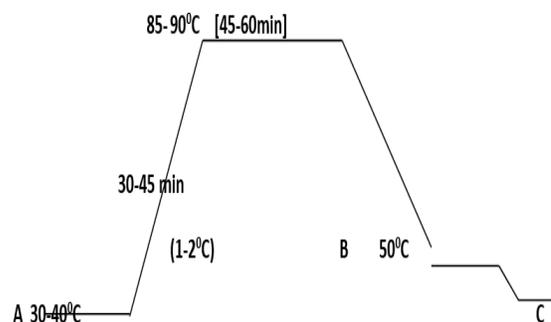
Dye-bath preparation was the same as for the previous fabrics. Nylon may however be dyed up to the boil at 100°C, while PLA is usually dyed at 80 -90°C to avoid fiber degradation. Acetic acid was added to achieve a pH 6-7, and dispersing agent added at 0.75-1.0g/L. The fabric was entered at 30-40°C and raised to the required dyeing temperature within 30-45 minutes at the rate of 1-2°C per minute. The dyeing was continued for 45-60min. The dyed fabrics were rinsed in warm water and then cold water and also allowed to dry.



A---Dyebath preparation, B---Washing [hot and cold]

C---Reduction clearing/Alkali clearing

**Figure 3. Dyeing profile of nylon 6, 6 used in this study.**



A---Dyebath preparation, B---Washing [hot and cold]C---Reduction clearing/Alkali clearing

**Figure 4: Dyeing profile of PLA used in this study.**

### Measurement of Dye Exhaustion

Dye uptake was determined by measuring the absorbance of diluted dyebath samples at the wavelength of the maximum absorption. The bath was sampled before and after dyeing. A 1ml aliquot was taken from the bath and diluted in 20ml acetone. This is to ensure that the absorbance falls within the readable range of 1.5. The percentage of dyebath exhaustion (%E) was calculated using the equation below as suggested by Jae-Hong Choi *et. al* (2008); and Ozan Avinc *et. al.* (2006)

$$\%E = [(A_0 - A_1) / A_0] \times 100$$

Where  $A_0$  and  $A_1$  are the absorbances at  $\lambda_{max}$  of the dyebath prior to dyeing and after dyeing, respectively.

### Application Properties of the Synthesized Dyes.

The fastness to light, was assessed in accordance with AATCC Test Method No 16-1998, sublimation according to AATCC Test No 117-1999 and perspiration was assessed in accordance with AATCC Test No 15-1997. The rub fastness test was carried out using a crockmeter (Atlas) in accordance with AATCC-Test Method 8-1996 and the wash fastness test in accordance with AATCC 61-1996 TEST Method NO 2A. The details of the various fastness tests are highlighted below.

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### Light Fastness Test

The light fastness study was carried out using an Atlas Ci-3000 Xenon Fade-O-meter. Procedure E of AATCC Test Method No. 16-1998 was used for the experiment. The light source was a water-cooled xenon-arc lamp. The machine parameters were set according to the standard method. Lamp wattage was set at 6.5 kilowatts, the wet bulb depression was 15°C, and the radiant energy source was set at 1.10 watts/m<sup>2</sup>. Exposure time of 20 hours was adopted.

### Wash Fastness Test

Fastness to washing was assessed using Test No 2A in AATCC Test Method 61-1996 (Colourfastness to Laundering, Home and Commercial: Accelerated)

Fastness to washing was assessed by an ATLAS automatic Launder-O-meter [Textile Test Equipment] under the following conditions; the washing solution contained neutral soap 0.15% detergent of total volume, a washing temperature of 49°C, the duration of washing was 45 minutes; and the material-to-liquor ratio was 1:50. After washing, the fabric samples were removed and rinsed with hot water (35°C). They were then squeezed and air dried. A staining test was also evaluated with the stitching together of the AATCC Multifiber strips No. 10.

The effect on the colour was expressed and defined by the use of AATCC grey scale for change in shade and also that of staining, (Maradiya, 2001). An AATCC Grey scale for colour change and Grey scale for assessing staining was used for the evaluation.

### Perspiration Fastness Test

Specimens of the textile in contact with specified adjacent fibres were treated in histidine solution, drained, and placed between two plates of 11.5 cm x 6 cm size

and of 0.15 cm thickness under the pressure of a weight-piece of mass 5kg in a testing device. It was kept in an oven for 6 hours ±5 minutes at 38± 1°C. The specimen and the adjacent fibres were dried separately overnight for proper conditioning. The change in colour of each specimen and the staining of the adjacent fibres were assessed with the grey scales, (Maradiya, 2001).

### Acid Perspiration Test

An acid solution containing 5g/litre sodium chloride (NaCl), 2.2g/litre sodium dihydrogen orthophosphate (NaH<sub>2</sub>PO<sub>4</sub>.2H<sub>2</sub>O) and 0.5g/litre 1-histidine mono-hydrochloride mono-hydrate (C<sub>6</sub>H<sub>9</sub>O<sub>2</sub>N<sub>3</sub>HCl.H<sub>2</sub>O) was freshly prepared. The solution was brought to 5.5 with a 0.1N acetic acid solution.

### Alkaline Perspiration Test

An alkaline solution containing 5g/litre sodium chloride (NaCl), 0.5g/litre 1-histidine mono- hydrochloride mono-hydrate (C<sub>6</sub>H<sub>9</sub>O<sub>2</sub>N<sub>3</sub>HCl.H<sub>2</sub>O) and 2.5g/litre disodium hydrogen orthophosphate (Na<sub>2</sub>HPO<sub>4</sub>.2H<sub>2</sub>O) was freshly prepared. The solution was brought to pH 8 with a 0.1N sodium hydroxide solution.

### Crock [Rubbing] Fastness Test.

The principle involves rubbing a piece of standard white cloth against the dyed fabric sample under controlled conditions of pressure and speed. The rubbing finger was covered with white cloth, for both the dry test and wet test. The white test fabric was soaked in deionized water before testing for the wet crock-fastness. The colour transferred to the white cloth was compared with a standard grey scale for colour transfer. The grey scale for the alteration of colour consisted of grades 1-5, (Maradiya, 2001).

## Results

**Table 1: Dyeing properties of the 2-aminothiophene dyes on polyester fabric**  
(2% Depth of shade. 50: 1 Liquor Ratio) TRITON-X as Dispersing agent

Dye no.	Colour hue on polyester	Wash fastness			Exhaus. [%]	Rubbing fastness		Perspiration fastness						Su bl m.
		CC	SP	S C		Dry	Wet	Alk			Acid			
								CC	S P	S C	CC	S P	SC	
1a	Violet	4	4	5	73	4	4	5	5	5	5	5	5	5
1b	Light violet	3-4	4	5	78	5	4	5	5	5	5	5	5	5
1c	Brown	4	4	5	77	4	4	5	5	5	5	4	5	5
2a	Brown	4	4	5	69	5	4	5	5	5	5	5	5	5
2b	Light yellow	5	5	5	72	5	4	5	5	5	5	4	5	4
2c	Yellow brown	5	5	5	73	5	4-5	5	5	5	5	5	5	3
3a	Yellow brown	4	4	5	73	5	4	5	5	5	5	5	5	5
3b	Brown	5	5	5	65	5	4	5	5	4-5	4-5	5	5	5
3c	Orange	5	5	5	78	5	4	5	5	5	5	5	5	5

Alk--- Alkaline

CC-----Change of shade of dyed fabric

SP-----Staining of polyester

SC-----Staining of cotton

Rating scale—1 (lowest) to 5 (highest) for washing, rubbing, and perspiration fastness.

**Table 2: Dyeing properties of the 2-aminothiophene dyes on PLA**  
(2% Depth of shade. 50: 1 Liquor Ratio) TRITON-X as Dispersing agent

Dye no.	Colour hue on PLA	Wash fastness			Exhaust ion [%]	Rubbing fastness		Perspiration fastness						
		CC	SD	S C		Dry	Wet	Alk			Acid			
								CC	S D	S C	CC	S D	SC	
1a	Violet	4	4	5	80	5	4	5	5	5	5	5	5	5
1b	Violet	3-4	4	5	77	5	4	5	5	5	5	5	5	5
1c	Light purple	4	4	5	75	5	4	5	5	5	5	5	5	5
2a	Red	4	4	5	68	5	4	5	5	5	5	5	5	5
2b	Light yellow	5	5	5	70	5	4	5	5	5	5	5	5	5
2c	Red	3	4-5	5	76	5	4-5	5	5	5	5	5	5	5

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2c	Red	2-3	4	5	69	5	4	5	5	5	5	5	5
3a	Red	3	4	5	68	4-5	4	5	5	4-5	4-5	5	5
3b	Orange	3	4	5	84	5	4	5	5	5	5	5	5
3c	Orange	4-5	4	5	75	5	4	5	5	5	5	5	5

Alk--- Alkaline

CC-----Change of shade of dyed fabric

SD-----Staining of diacetate

SC-----Staining of cotton

Rating scale—1 (lowest) to 5 (highest) for washing, rubbing, and perspiration fastness.

**Table 3: Dyeing properties of the 2-aminothiophene dyes on nylon fabric**

(2% Depth of shade. 50: 1 Liquor Ratio) TRITON-X as Dispersing agent

Dye no.	Colour hue on nylon	Wash fastness			Exhau. [%]	Rubbing fastness		Perspiration fastness						
		CC	SN	SC		Dry	Wet	Alk			Acid			
									CC	S	S	CC	S	SC
1a	Violet	3	4	5	79	5	4	5	5	5	5	5	5	5
1b	Violet	3	4	5	80	5	4	5	5	5	5	4-5	5	5
1c	Light violet	3	4	4-5	78	5	4	5	5	5	5	5	5	4
2a	Light violet	3	4	5	69	5	4	5	4-5	5	5	5	5	5
2b	Light yellow	3-4	5	5	79	5	4-5	5	4	5	4-5	5	5	5
2c	Light yellow	5	4	5	80	5	4	5	5	5	5	5	5	5
3a	Red	3	4	5	77	5	4	5	5	5	5	5	5	5
3b	Red	3	4	5	71	5	4	4-5	5	4-5	5	5	5	5
3c	Orange	3	3	5	86	5	4	5	5	5	5	5	5	4-5

Alk--- Alkaline

CC-----Change of shade of dyed fabric

SN-----Staining of nylon

SC-----Staining of cotton

Rating scale—1 (lowest) to 5 (highest) for washing, rubbing, perspiration and fastness.

**Table 4: Light fastness results of tested dyes**

Dye No	Light fastness		
	Polyester	PLA	Nylon
1a	3-4	2	1-2
1b	2-3	2	1-2
1c	2	2	1
2a	1-2	1-2	1
2b	2	2	1-2
2c	2	2	1
3a	2-3	2	1-2
3b	2	2	1
3c	2	2	1-2

P----Polyester

T----PLA

N----Nylon

Rating scale—1 (lowest) to 5 (highest) for the light fastness.

### Discussion

From the data presented, it was observed that the exhaustion of the dyes was slightly higher on PLA than on polyester. This of course was to be expected in view of the high crystallinity of the polyester compared with PLA and Nylon 6,6.

The color fastness of dyed PLA tends to be slightly lower than that of PET at similar depths of shade. Yang and Huda (2003) reported that the disperse dyes studied exhibited lower washing and rub fastness on PLA than on PET. Choi and Seo (2006) reported that wet fastness of each PLA dyeings (at 4.0% o.w.f.) was either similar or up to 1 point lower than its PET counterpart. However, Scheyer and Chiweshe (1999) claimed that most disperse dyes on PLA fibre would have acceptable wash fastness for apparel applications. The wash fastness of high- and low-D PLA fibres exhibit very little difference due to their close glass transition temperatures (Blackburn *et. al* 2006).

However the wash fastness of the dyes on PLA gave results which are comparable with that of polyester and in some cases even better. This is encouraging in view of the fact of the limited availability of suitable dye range for PLA. Similarly data from other application tests are also encouraging. Results from the light fastness test however returned quite poor results which may make

the application of the dyes for outdoor use not suitable.

Overall, extensive work would still need to be done especially in the area of improved exhaustion and better wash fastness. However the encouraging thing is that the application areas of the PLA are so wide and the dyes would no doubt find utility in a number of situations relevant to the specific areas of strength displayed by the dyes.

### Conclusion

The results proved that the tested dyes with further studies and some modifications in the dyeing protocols could give a major boost to the coloration of PLA. Advancement in the chemistry of PLA seems to be moving at a considerable pace and faster than that of suitable colorants for it. Further studies are however needed to encourage production of dyes with substantially higher exhaustion level as well as better light fastness. Achieving both objectives will widen the scope of available colorants for PLA and other hydrophobic fibers.

While the use of PLA fibre is still at its infancy compared with conventional synthetic fibres such as PET, PLA fibre consumption is projected to rise in the near future due to its ecological advantages and other technical usefulness as a textile fibre. Finding the best conditions and methods for wet processing and after-care applications for

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PLA-based textile products will certainly lead to increase in the consumption of this natural renewable resource-based melt processable PLA fibre. New research and improvements in chemistry and wet processing of PLA fibres will obviously further expand the use of this fibre and new applications

### Acknowledgement

Special thanks to Professor H.S Freeman at the Wilson College of Textiles, North Carolina State University (NC State), Raleigh, USA, who provided the use of his laboratory facility as well as supplied most of the consumables used in the research. Also to the Board of the Tertiary Education Trust Fund (TETFUND), Abuja, for sponsoring the trip to NC State University as a Research Scholar.

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