INVESTIGATING THE APPLICATION OF TERRA DYE ON COTTON KNITTED FABRICS

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ABSTRACT

Today, sustainable textile dyeing technologies are being researched with a purpose of developing ecofriendly dyes that are cost effective and resource efficient. Natural Earth Pigments also known as native earth pigments, earth colours, earth ochres, iron oxide pigments etc. come from naturally occurring minerals, typically iron oxide or manganese oxide. Terra dye is a sustainable and environment friendly dye which has been derived from pigmented earth and without the use of harsh toxic chemicals. It is 100% natural, obtained from the extraction of minerals. The study investigates the application of 'Terra dye' on cotton knitted fabrics. 100% Cotton Jersey and 100% Cotton Fleece fabrics were used. The terra dyed fabrics were tested for their properties of colour uptake, bleeding, rubbing fastness, resistance to light and washing fastness. The effect of different fixing agents was investigated. The results of the lab trials and testing, conclude that Terra dye has good prospects of being used in dyeing.

KEYWORDS

Application; Natural Earth Pigments; Terra Dye; 100% Cotton fabrics.

INTRODUCTION

New sustainable textile dyeing technologies are being researched and developed with the aim of developing sustainable dyes that are more cost effective, resource efficient causing no harm to the planet. (TS Jeans Care, 2022). Terra is the Latin name for the earth. It is the name of the Goddess that protects the planet. The name has a direct connection to the innovative eco-friendly earth dyes. Terra dye is a sustainable and environment friendly dye which has been derived from pigmented earths and with no use of harsh toxic chemicals. It is 100% natural as it is obtained from the extraction of minerals. Through the process of grinding, geological material can become a pigment powder. Mineral deposits present give colouration to the earth pigments. Earth pigments are insoluble in water and are physically and chemically unaffected by the mediums they mix with. Furthermore, it caters for 50% water savings and 50% less use of energy as compared to a conventional pigment dye. Terra dye has some inherent disadvantages. This pigment dye may not be harmful to the environment but processes such as mining and quarrying required during extraction of earth pigments may be highly polluting. Dyes collected from natural earth pigments may lead to a variance in the colour of the dye due

to factors contributing to the source itself such as climate, location of earth pigment and volcanic eruptions.

Mauritius, is a volcanic island. It has at Chamarel a rare and impressive geological phenomenon of the seven coloured. As seen in figure 1 the colours of the earth are blend together, like ochre and mauve, brown and pink, and shade in-between. The colours are seen to move from brown to ochre, from mauve to pink and into dozens of variations. The island with such diversified earth colour palatte provide scope for exploring terra dyes. The study investigates the application of 'Terra dye' on 100% Cotton Jersey and 100% Cotton Fleece knitted fabrics.

EXPERIMENTAL METHOD

Materials

100% Greige cotton jersey and cotton fleece knitted fabrics were used (Figure 2.1 (a) (b)). Five Terra dyes as seen n figure 2.2 (a) (b) (c) (d) (e), trade names "Giallo Artiglieria" (Yellow), "Verde Similcromo" (Green), "Rosso Laccato Scuro" (Pink), "Rosso Ercolano" (Red Earth) and "Mineral Black" were utilised. The Terra dyes and fabrics were provided by Consolidated Fabrics Ltd.

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Figure 2.1 (a) (b). Greige Cotton Jersey and Fleece knitted fabric







b) Verde Similcromo (Green)



(d) Rosso Ercolano (Red Earth) Figure 2.2 a) b) c) d) e). Terra Dyes

Pre-treatment of Fabric

Semi Bleach: The fabrics were semi bleached in a Fong's industrial dyeing machine as shown in Figure 2.3. Machine was preheated at 50°C for 10 minutes and 158 Litres of water was filled. 160g of Ronwet, a wetting agent, was added. 25 kgs of the fabric was loaded. 160 g of Imerol NFL, a detergent, 64 g of Prestogen FCB, a stabiliser and 32 g of Ronlube, an anti-crease, was added into the machine. After 5 mins, 633g of liquid Caustic Soda was poured into the bath. 640g of Hydrogen Peroxide was added and the temperature was increased at 3°C per minute until it reached 110°C. for 20 minutes. Nevocid, an acid, was injected into machine trough pipe system. The pH was maintained between 6-7. 64g of Bactosol, a peroxide added to neutralize the liquor. The fabric was rinsed with water and dried

Scouring: A batch 4 kg fabric was scoured in Rotary machine as shown in Figure 2.4 (a) & (b) with 0.50g/L Asutol and 60g Ronwet, wetting agent to remove impurities present before proceeding with dyeing. The Cotton Jersey and Cotton Fleece



c) Rosso Laccato Scuro (Pink)



(e) Mineral Black

fabrics scoured as per the scouring profile seen in Figure 2.5.



Figure 2.3. Pretreatment



Figure 2.4 (a) & (b). Scouring process in Rotary





Figure 2.5. Scouring Profile (Adapted - Tropic Knits Washplant Dyehouse Recipe Card, 2022)

Dyeing of Cotton Fabrics with Terra Dyes

120 L of cold water was filled into the Rotary machine. 120 grams of Terra dye pigment was mixed with 1 litre of cold water until the pigment was completely diffused into the water. The dye was then poured slowly into the machine through a strainer to filter remaining undissolved dye pigments. The machine was run for 15 minutes and temperature raised to 60°C. The steam valve was opened to allow steam to be gradually released at 1°C per minute from the steam pipe into the machine to control even dyeing. After 15 minutes, hydro extraction was carried out in which the dyed water was drained out of the machine.

The fabrics were rinsed twice with cold water for 5 minutes. 120g of Acasoft, a softener was then added at 40°C and machine was run for further10 minutes. The fabrics were dried. Figure 2.6. shows the dyeing profile used for Terra dyeing.



Figure 2.6. Dyeing Profile Terra dye (Adapted -Tropic Knits Washplant-Dyehouse Recipe card, 2022).

Dyeing of Cotton Fabrics with Terra Dyes using Cationisation method

1 kg of Cotton Jersey and Cotton Fleece were dyed with Terra dye trade name "Sand" which is of a yellow Ochre colour. Cationisation method involves the use of binder, to fix the dye pigment onto the fabric and achieve good colourfastness.

Scouring: The fabrics were scoured with 0.50 g/L Asutol prior to dyeing using the scouring profile as seen in Figure 2.7. After hydro extraction, the machine was filled with 30 litres of water to proceed with cationisation.



Figure 2.7. Scouring Profile (Adapted -Tropic Knits Washplant - Dyehouse Recipe Card, 2022)

Cationisation: 30 grams of Dye prep (Binder) was well diluted, filtered and poured into the Rotary machine and the machine was run for 3 minutes. The machine was heated to 50°C at 2°C per minute. Machine was run for 15 minutes at 50°C. After 15 minutes, 30 grams of Soda Ash was added and machine was run for another 15 minutes. The pH was maintained in the range of 8-10. Hydro extraction was carried out before cold rinsing for 5 minutes twice. Catonisation profile is seen in Figure 2.8.



Figure 2.8. Cationisation Profile (Adapted- Tropic Knits Washplant - Dyehouse Recipe Card, 2022)

Pigmentation: Machine was filled with cold water. 6 grams of Acetic acid added and run for 5 minutes. The pH was maintained at 4.5 before dosing 100 grams of dye pigment into the machine. Machine was heated 1°C per minute to a temperature of 50°C for 20 minutes. 60 grams of Fixacryl CFD, a dye fixer, was added and machine was run for 10 minutes. Water was drained and fabric was cold rinsed for 5 minutes. Figure 2.9 shows the pigmentation profile.



Figure 2.9. Pigmentation profile (Adapted -Tropic Knits Washplant - Dyehouse Recipe Card, 2022).

Softening: Fresh cold water was filled in machine. pH of water was adjusted to 5.5-6 by adding acid and machine was run for 3 mins (Figure 2.10). 40 grams of Acaflakes RT New, softener, was added to the bath. The machine was heated at 2°C per minute to a temperature of 40°C and run for 10 minutes. Hydro extraction was carried out and the fabrics were tumble dried at 105°C for 60 minutes.



Figure 2.10. Softening profile (Adapted from Tropic Knits Washplant - Dyehouse Recipe Card, 2022).

Finishing

Dyed fabric samples were padded with different fixatives in separate batches - 200g Sodium Sulphate (Salt), 5 ml Acetic acid, 200g Sodium Carbonate (Soda Ash), 200g Sodium Bicarbonate (Baking Soda) and 40g Hifix, a cationic fixing agent. A rapid pad mangle machine was utilised for padding process.

Padding: 1 litre of water was boiled at a temperature of 100°C. 200 grams of Sodium Sulphate was added, stirred continuously to dissolve and allowed to cool. This solution was used for padding and samples were oven dried. Same process was used for each fixative.

Evaluation

Grams per Square Metre (GSM): The GSM of both Cotton Jersey and Fleece were measured when at "greige" state, after semi-bleaching, dyeing and finishing process. The fabrics were ring cut matched with circular standard template and weighted on an electronic balance.

Pantone CAPSURE: Pantone Capsure apparatus Figure 2.11 was utilised to match dye colour of fabric with pantone colour libraries to obtain colour name and code.

Rubbing Fastness: Dyed Cotton Jersey and Fleece specimens were cut in dimension 25cm x 5 cm for both dry and wet rubbing test. The test was carried out in a crock meter whereby the rubbing fringe was

covered with a dry crocking cloth and held in position with a spring clip. Fabric specimen was placed on rubbing area and was held by the clamping device's pins passing through the fabric and into holes on the base. Along the warp direction, the fringe was moved forth and back 10 times in 10 seconds at the rate of one turn per second. Same procedure is carried out for wet rubbing test. In the latter case, the crocking cloth is wetted, squeezed and dried at room temperature after rubbing which was later matched with greyscale for assessing staining. (Figure 2.12)

Wash Fastness: Microfiber was sewn on top of all dyed samples. Soap solution was prepared with 5g of ECE detergent and 2g of Sodium Carbonate per litre of water. Solution of liquor ratio 50:1 was poured in each container for each sample and was put in machine at 60°C for 30 minutes and was flat dried (Figure 2.13). Samples were assessed with greyscale for staining.

Light Fastness: Pieces of Blue Wool was stick onto a piece of cardboard of dimension 12.8 cm x 4.9 cm and was used to act as control. On 5 other pieces of cardboard, the dyed samples were cut and glued. The machine was set for 103 hours. Samples were cross-checked each 6 hours for any colour change. Changes in colour were matched with greyscale standard in a light cabinet. The blue wool had to fade to a contrast equal to a grade of 2/3 according greyscale. (Figure 2.14(a) (b)).



Figure 2.11. Pantone.



Figure 2.13. Assessing crocking cloth with greyscale for staining.



Figure 2.12. Samples Rub fastness.





Figure 2.14 (a) Greyscale for assessing colour change (b) Samples in light fastness machine.

RESULTS AND DISCUSSION

Pre-treatment of Fabric

Semi-bleaching: With visual inspection, a major change in fabric colour can be observed. The natural yellowish brown colour the fabric was removed to a uniform degree of whiteness. (Figure 3.1 (a) & (b)).



Figure 3.1 (a) (b). Semi-bleached Cotton Jersey and Fleece fabric.

Fabric Mass weight loss: Table 3.1 shows the weight loss occurred due to the amount of fibres being treated with the addition of Hydrogen peroxide(H_2O_2) during semi-bleach process. It can be observed that weight loss for Fleece is less than Jersey fabric.

Table 3.1 Mass of Fabric.

Fabric Type	Before Semi- bleaching	After Semi- bleaching
100% Cotton Jersey	25 kg	21.5 kg
100% Cotton Fleece	25 kg	22 kg

Spectrophotometric Whiteness Test

As seen in figure 3.2 (a) (b), a Standard Whiteness of 72.42 was obtained for semi-bleached Cotton fleece and 70.63 for jersey fabric.

	Illum/Obs	D65 10 Deg		Illum/Obs	D65 10 Deg
	STD. J858A			STD. J517	
	BAT. 222289			BAT. 222290	
	STD WI.	72.42		STD WI.	70.63
	BAT WI.			BAT WI.	
a)	DELTA.WI		(h)	DELTA.WI	

Figure 3.2 (a) (b) Whiteness test Datacolour Spectrophotometer.

- Illum/Obs D65 10 Deg refers to a light source name which is a simulation of natural day light.
- STD refers to standard of fabric.
- BAT refers to the batch number of fabric lot.
- STD WI refers to the Standard Whiteness

Dyeing with Terra dyes

Table 3.2 shows the colour codes and names obtained for the jersey and fleece cotton fabrics dyed with terra dyes. Pantone CAPSURE apparatus was used to obtain the colour codes and names.

Padding and Finishing

Padding process with Sodium Sulphate: Rosso Ercolano and Mineral black was seen to bleed colour in the bath. For lighter dye shades, no major change was observed in the solution during padding process. White patches of salt were observed on fabric surface after being oven dried and fabric feel became rough.

Padding process with Sodium Carbonate: Slight bleeding was observed for light coloured samples whereas dark shade bleed much more. Fabric were smooth after drying with showed no tendency of curling.

Padding process with Sodium Bicarbonate, Acetic acid & Cationic Fixing agent: Darker dye shades continued to bleed. Fabric remained soft with good drape with even dyeing. Similar results were achieved for acetic acid and cationic fixing agent.

Grams per Square Metre (GSM)

It can be observed that both fabrics gained weight after semi-bleaching process. This may be due to the fact that curling tendency of knitted fabrics and shrinkage occurred increased the weight. Furthermore, an additional increase in GSM was observed after dyeing due to the reason that the dye pigment weight has add up to the fabric weight. Fleece fabric is gaining more mass due to its compact and thick structure. GSM has increased much more after the addition of fixatives, and fabrics have become much heavier.

 Table 3.2: Pantone Color and Code.

Terra Dye	Jersey Cotton	Fleece Cotton
Giallo Artiglieria (Yellow)	PANTONE 12-0817 TCX Apricot Gelato	PANTONE 12-0822 TCX Golden Fleece
Verde Similcromo (Green)	PANTONE 13-6208 TCX Bok Choy	PANTONE 15-6315 TCX Smoke Green
Rosso Laccato Scuro (Pink)	PANTONE 13-1904 TCX Chalk Pink	PANTONE 14-1905 TCX Lotus
Rosso Ercolano (Red Earth)	PANTONE 16-1522 TCX Rose Dawn	PANTONE 16-1516 TCX Cameo Brown
Mineral Black	PANTONE 18-4005 TCX Steel Gray	PANTONE 18-5203 TCX Pewter
Sand (With Binder)	PANTONE 13-1025 TCX Impala	PANTONE 14-0936 TCX Sahara Sun

Terra dye colour	100% Cotton Jersey	100% Cotton Fleece
Giallo Artiglieria (Yellow)	4	4
Verde Similcromo (Green)	4	4
Rosso Laccato Scuro (Pink)	4.5	4.5
Rosso Ercolano (Red Earth)	4	4
Mineral Black	3.5	3.5
Sand (Binder)	4.5	4.5



Figure 3.3 (a) (b). Dry and Wet Rub fastness for treated fabrics.

Dry and Wet Rubbing Fastness of Terra dyes

In the greyscale, the fastness ratings range from 1 (Poor) to 5 (Excellent). Very good results (3.5-4.5) were obtained for all dry rub tests indicating that less staining occurred with amount of unfixed dyes present in fabric. However, for wet rubbing, an average wet rubbing fastness (3) was obtained for non-treated fabrics as unfixed dyes dissolved in water and stained the crocking cloth. Small fuzzy balls(pills) of fibres can be seen on crocking cloth. On the other hand, lighter shade samples treated with Sodium Sulphate, Sodium Carbonate, Sodium Bicarbonate, Acetic acid and Cationic fixing agent obtained excellent results (4-4.5). Only the darker shades wet rubbing fastness could not have been improved with addition of fixatives. (Figure 3.3 (a) (b)).

Results for Wash Fastness test

Staining on microfiber were assessed with greyscale in order to determine whether the dye used stains other fibres and whether garment with this dye can be washed together in washing machine at home. (Table 3.3). Non-treated terra dye samples slightly stained almost all fibres. Lighter shades did not stain

Terra dye colour	100% Cotton Jersey	100% Cotton Fleece
Giallo Artiglieria (Yellow)	3	3
Verde Similcromo (Green)	3	3
Rosso Laccato Scuro (Pink)	3	3
Rosso Ercolano (Red Earth)	3	3
Mineral Black	3	3
Sand (Binder)	3	3





the multifibre when compared to darker shades. For the Sodium Sulphate samples, the white salt patches present on fabric was washed away. For Sodium Carbonate, Sodium Bicarbonate and Acetic acid, quite good results were obtained as dye shade strength was the same after washing and did not bleed much. Lighter dye shades did not affect the multifibre at all. Excellent results were achieved for cationic fixing agent samples as no major staining was observed

Light Fastness test

After being exposed to UV light for 103 hours, the samples (Figure 3.4.) were assessed with colour change greyscale. The obtained results are seen in Table 3.4. Slight colour changes could be observed in lighter samples whereas a major colour difference was observed in darker samples. The darker the colour, the more the fading away of the shade is noticeable. Sodium Carbonate and Bicarbonate samples fairly resisted to light. Presence of salt in sample has made the colour to fade more rapidly compared to the other samples. Samples with Acetic acid and Cationic fixing agent resulted in good resistance to light.

Table 3.3 Fastness to washing test results.

Sample Type	Dye Colour	_			n Multifibre	-			
Cambio 13be	-	Acetate	Cotton	Nylon	Polyester	Acrylic	Wool		
	Yellow Jersey	5 (No change)							
	Yellow Fleece	4/5	4/5	3(Pink)	4/5	4/5	4/5		
	Green Jersey				change)				
	Green Fleece		-		change)	- 41 - 1			
	Pink Jersey	4/5	5	4/5	4/5	5 (No ch	<u> </u>		
Non-treated	Pink Fleece	4/5	4/5	4	4/5	4/5	4/5		
	Red Jersey	5 (No c	U	4/5	4/5	4/5	4/5		
	Red Fleece	4	4/5	4 4/5	4/5	4/5	4		
	Black Jersey Black Fleece	4/5 4/5	4/5 4/5	4/5	4/5 4/5	5 (No ch 4/5	ange) 5		
	Sand Jersey	4/5	4/5			4/3	5		
	Sand Fleece	5 (No change)							
	Yellow Jersey	5 (No change)							
	Yellow Fleece	5 (No change) 5 (No change)							
	Green Jersey	5 (No change) 5 (No change)							
	Green Fleece	5 (No change) 5 (No change)							
Sodium	Pink Jersey	5 (No c	hange)	4/5	4/5	5 (No ch	ange)		
Sulphate	Pink Fleece	5 (No c		4/5	4/5	5 (No ch			
•	Red Jersey	4/5	5	5	4/5	4/5	5		
	Red Fleece	4/5	4/5	4/5	4/5	4/5	5		
	Black Jersey	5	5	4/5	5	5	5		
	Black Fleece	4/5	4/5	4/5	4/5	4/5	4/5		
	Yellow Jersey			5 (No o	change)				
	Yellow Fleece				change)				
	Green Jersey	5 (No change)							
	Green Fleece			5 (No o	change)				
Sodium	Pink Jersey	4/5	5	4/5	5 (N	lo change)			
Carbonate	Pink Fleece	4/5	5	4/5	5 (N	lo change)			
	Red Jersey	4/5	5	4/5	5 (No change)				
	Red Fleece	4/5	4/5	4/5	4/5	4/5	5		
	Black Jersey			5 (No o	change)				
	Black Fleece	4/5	4/5	4/5	4/5	4/5	5		
	Yellow Jersey			5 (No d	change)				
	Yellow Fleece	5 (No change)							
	Green Jersey				change)				
	Green Fleece		_		change)				
Sodium	Pink Jersey	4/5	5	4/5	5	4/5	5		
Bicarbonate	Pink Fleece			4/5			5		
	Red Jersey					lo change)			
	Red Fleece	4/5							
	Black Jersey				change)				
	Black Fleece			4/5		5 (No ch	nange)		
	Yellow Jersey Yellow Fleece	5 (No change)							
	Green Jersey	5 (No change)							
	Green Fleece	5 (No change) 5 (No change)							
Acetic Acid	Pink Jersey	4/5	5		4/5	5 (No ch	ande)		
	Pink Fleece	4/5		4/5	4/0	5 (No ch			
7,010	Red Jersey		4/5	-10	5 (N	lo change)	ange)		
	Red Fleece	4	-1/0		4/5	lo onungo)	5		
	Black Jersey	4 4/5 5							
	Black Fleece				/5				
	Yellow Jersey								
	Yellow Fleece	5 (No change)							
	Green Jersey	5 (No change)							
		5 (No change)							
	Green Fleece	5 (No change) 5 (No change)							
Cationic	Green Fleece Pink Jersey			5 (No d	5 (No change) 5 (No change)				
Fixing	Pink Jersey				- /				
		4/5			change)	4/5	5		
Fixing	Pink Jersey Pink Fleece	4/5	4/5	5 (No c	change)	4/5			
Fixing	Pink Jersey Pink Fleece Red Jersey		4/5	5 (No c 5 (No chan 4	change) ge)				



Figure 3.4 Samples after light fastness.

Table 3.4 Light fastness test results.

Number of hours	Non- treated	Sodium Sulphate	Sodium Carbonate	Sodium Bicarbonate	Acetic acid	Cationic Fixing agent
		-	Light dye shade	s	<u> </u>	
6 hours	5	4/5	4/5	5	4/5	5
18 hours	5	4	4/5	5	4/5	5
42 hours	4/5	4	4	4/5	4/5	4/5
103 hours	4/5	3/4	4	4	4	4/5
		•	Dark dye shade	s		
6 hours	4/5	4/5	4/5	5	4/5	5
18 hours	4	3/4	4	5	4/5	5
42 hours	3/4	3	3/4	4/5	4/5	4/5
103 hours	3	2/3	3	4	4	4/5

CONCLUSIONS

This study has provided an in-depth understanding of Terra dye. The study investigates the application of 'Terra dye' on 100% Cotton Jersey and 100% Cotton Fleece knitted fabrics. The fabrics were semi bleached in an industrial dyeing machine with Asutol and Ronwet to remove impurities present before dyeing. Data colour spectrophotometer was used to test the degree of whiteness. The scoured fabrics were dyed in a rotary dyeing machine. Five colours of Terra dye namely Giallo Artiglieria (Yellow), Verde Similcromo (Green), Rosso Laccato Scuro (Pink), Rosso Ercolano (Red Earth) and Mineral Black (Black) were used. The fabrics were rinsed thoroughly and treated with Acasoft, softener. The dyed fabrics were treated with fixing agents namely Sodium Sulphate (Salt), Acetic acid, Sodium Carbonate (Soda Ash), Sodium Bicarbonate (Baking Soda) and Hifix, a cationic fixing agent using the rapid padding mangle. Dyeing using the Cationisation method was carried out with 'Sand Terra dye'. Cationisation method involved the use of binder to fix the dye pigment onto fabric surface to achieve good colourfastness. Pantone Capsure apparatus was utilised to match the fabric dye colour with pantone colour libraries and obtain the pantone colour name and code. Very good results were obtained for all dry rub tests and an average wet rubbing fastness was observed that stained the crocking cloth. The dye could not achieve darker colour strength or fix the dye pigments permanently on the fabric surface. It was seen that the bleeding of dye from the fabric could be reduced by use of cationisation method and cationic fixing agent. Samples with Acetic acid and Cationic fixing agent

resulted in good resistance to light. The results of the lab trials and testing, conclude that Terra dye has good prospects of being used in dyeing. These Eco-friendly pigments obtained from natural sources show potential to make it into the mainstream fashion.

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