Australian Government

Cotton Research and Development Corporation

HONOURS SCHOLARSHIP REPORT: 2017-18 SEASON

1.	**Project Title** (Maximum 15 words)	:	Design of versatile protective cotton fabrics with colour and patterns
2.	**Proposed Start Date**	:	1st February 2018
	Proposed Cease Date	:	30th November 2018
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SUMMER SCHOLARSHIP REPORT

1. Executive Summary

The project focuses on a coloured coating of waterborne polyurethane (WPU), polydimethylsiloxane (PDMS) and trimethylated silica (TMS) on cotton fabrics by pad-knife-pad method. Different dyes/pigments were applied with TPU paste to bring colour to the coating.

The funded honour student, Ms Olivia Williamson, was enrolled as a Research Assistant in the research group of Dr Xin Wang at RMIT University. She has conducted the experiments of coating and performed related characterisations on the surface properties and protections of both the colour coated fabrics and TPU dyed fabrics. The data colour testing was employed to compare the shade of dyes with the pigments on the coloured fabrics. It has been found that the colour coated fabrics of WPU-PDMS-TMS has brought hydrophobicity and repellency against water, oil, chemicals and aqueous liquids. Comfort properties have also been assessed using thermal resistance and moisture management property tests. The coating technology has the potential to apply coloured cotton in protective clothing and performance textiles in different areas including military, mining and outdoor sportswear.

2. Background

Differential cotton products are highly demanded by customers to provide them with protection, while maintaining comfort during the service of the products. Protection against hazards and threats from environment, such as water, oil, chemicals, biological substances, is necessary for daily routine and for people with special missions. For this reason, protective clothing has been widely used for firefighters, mining workers, military forces and athletes. Coating is a very effective technology to bring extra functions to textiles, especially for performance textiles and coloured protective clothing the coating of extra layer is the core mechanism. Fluoro-based polymers or compounds were applied to fulfil the protection requirements of performance textiles, however, the toxic by-products from synthesis of those fluorobased chemicals have created threats to health and environment. Eco-friendly polymers are being sought for replacing the fluoro-based chemicals in the development of coating. As an example, polyurethane has been widely applied to coat textiles for different purposes. Even though the water proof membrane (WPU) and polymeric coating (TPU) can provide protection to cotton fabrics, it is important to develop versatile protection mechanism to coloured cotton fabrics. A combination of WPU using different pigments and TPU with different dyes have been used to provide a versatile protection and resolve this challenge.

The project focuses on realisation of colour and pattern to the already developed pad-knife-pad coating of TPU-PDMS-TMS on cotton fabrics. The methodology of colouring will be explored by either adding dyes into the coating process or by employing pre-dyeing process. Colour patterns will also be investigated by using multi-step coating or patterned pastes, and protective cotton fabrics with different colour and patterns will be developed in

the end to benefit further commercialisation of the coating technology.

The work aligns with the Strategic Plan 3.2 Differentiated Products and 3.3 Competitive Futures as set by CRDC. It developed high value and novel coloured cotton products to fulfil the increased consumer needs, which will greatly differentiate Australian cotton. It will contribute to value-adding technology to current cotton fabric finishing process with the purpose of widen the application potential of cotton textiles and fulfil performance requirements from a broader range of consumers. The project also aligns with the Strategic Plan 4.1 Workforce Capacity as it has provided student with opportunity to do research in the final year of her study.

3. Aims and Objectives

Design is a very important part to further transfer the already developed versatile coating technology for adoption in products development, and the implementation of colour with patterns is a challenge for the already developed coating technology. This project aims to realise colour and design for the coating of cotton fabrics. It will make the functional protective coating more attractive and favourable to end users, so that the application potential of cotton fabrics will be enhanced to fulfil performance requirements from a broader range of consumers.

The project also aims to provide honours student with the chance to work on cotton and textile related research environment, which will encourage student to pursue a cotton related career, either in research and development or in industry. The objectives of the project are thus as follows: enrolling the funded student as a Research Assistant to work with a PhD student to gain skills related to cotton research; training of the funded student in terms of coating technology and characterisation techniques; performing the pad-knife-pad-cure coating of WPU-PDMS-TMS on coloured cotton fabrics followed related testing; reporting and presenting research outcomes in different ways.

4. Materials and Methods:

<u>Materials for WPU Coloured Cotton Fabric:</u>

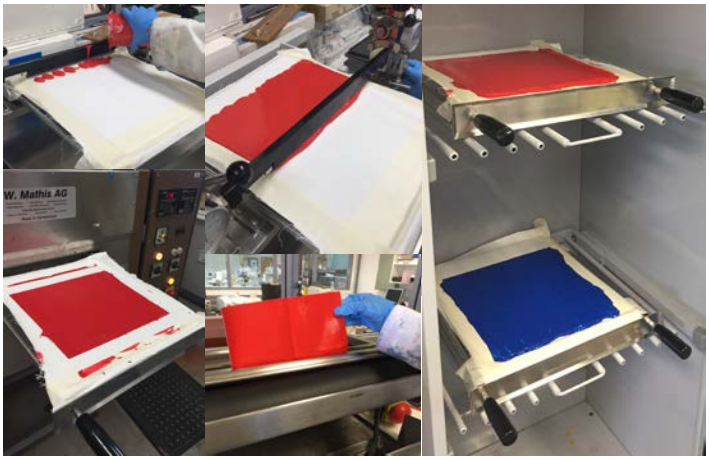
- Cotton (Bruck Textiles, Australia)
- Tubicoat WP 1665 HT (CHT Pty Ltd, Australia)
- Xiameter FBL -0563 (Dow Clothing Pty Ltd, Australia)
- Yorkshire Flexobrite Golden Yellow R #607428 (Australia)
- Yorkshire Flexobrite Brilliant Blue B #511134 (Australia)
- Red FGR (Australia)

Method

Pad- Knife-pad Coating: Preparation of WPU Coloured Cotton Fabric

The cotton fabric was scoured in MCS mini jet dyeing vessel with o.1 w/v Triton X100 and a liquor ratio of 20:1, at 60°C for 20 minutes. After scouring, the fabric was rinsed with warm water and then with cold water, followed by an overnight hanging drying process. The cotton fabrics were then subject to a three-step coating as illustrated in Figure 1. Firstly,

Ernst Benz Pad was used to perform ICB Fix padding for three passages. The solution in the pad contained 30 mL distilled water with 0.15 g of ICB Fix. The sample was then dried at 60° C for 30 minutes. Secondly, the padded fabrics were coated with 30-45 g original paste of WPU (tubicoat 1665) by knife coating. As shown in Figure 1, the edge of knife rolled over fabrics to allow an even coating on the surface of fabrics, and the coated fabrics were then dried at 60° C for 30 minutes. Thirdly, the pad-knife treated fabrics were padded with PDMS-TMS. The PDMS-TMS combined solution was diluted with n-heptane at the concentration of 4%. The sample was dried at 60 °C for 30 minutes followed by a 150 °C curing process for 3 minutes.



Sample	Description
A″	Cotton
B″	4%PT
C′′	WPU
A	WPU+ Red + 4%PDMS-TMS
B	WPU+ Yellow +4%PDMS-TMS
C	WPU-Blue +4%PDMS-TMS
D	WPU+ Red-Yellow-Blue+4%PDMS-TMS

Figure 1 Cotton Coating Process and Sample Coding.

Preparation of TPU Dyed Cotton Fabric

The recipe for the red shade consists of 1.8 g of Cibacron Rot C-2G, 0.5 g Cibacron Gelb C-R, 3 g Sodium Hydroxide, 3 g Sodium Silicate and 100 mL water. The yellow shade consists of 1.2 g Cibacron Gelb C-R, 3 g Sodium Hydroxide, 3g Sodium Silicate and 100 mL water. The blue shade is made up of 3 g Synozol Blue SHF-BRN 150%, 3g Sodium Hydroxide, 3g Sodium Silicate and 100 mL water. After the mixture had been made, each coloured fabric was padded three times before it was rolled up and placed in a sealed bag for 24 hours. The fabric was then washed with cold water and 2 mL acetic acid in 200 mL water boiling at 90 °C for 30 minutes where pH of 3 was maintained. The fabric was again washed with 5 g detergent in 500 mL water at 40 °C for 30 minutes before it was rinsed and dried in the laboratory dryer.

Measurements and characterizations

Data Colour

The HunterLab Color QUEST II Spectrophotometer (Leicestershire, UK) was employed to analyse the colour value. K/S values and the relative strengths between the dyed samples and the pigment coated samples were calculated using Premier Colorscan software (Premier Colorscan Instruments Private Ltd., Mumbai, India). Measurements were made under the following conditions: 10 °C observer, maximum wavelength 600 nm, and D65

light source. Three different angle measurements were conducted to get the averaged surface characteristic of the coloured fabrics.

Water Repellency

The Water repellency test (Toyoseiki, Tokyo, Japan) was performed in accordance with the AATCC 22:2010. A sample (180 mm x 180 mm) was conditioned at 20 ± 2°C and 65 ± 2 % RH for 4 h. During the test, 250 mL of distilled water was poured onto the surface of the sample for 30 seconds in order to obtain the spray rating.

Aqueous Liquids Repellency

Aqueous liquids repellency test was performed in accordance with AATCC 193:2012 standard. Six specimens (2 cm x 2 cm) were placed in petri-dish where three droplets of the chemical (Iso-propyl alcohol, 10 microliter) was applied to the surface of the specimens. The chemical droplets on the fabric were observed after 10 seconds at an angle of 45°. The grade was recorded if the droplet was repelled otherwise a zero was recorded if it penetrated into the fabric.

Oil repellency Test

The oil repellency test was performed in accordance with AATCC 118:2013 standard. Six specimens (2 cm x 2 cm) were placed in petri-dish and a 10-mL droplet of oil was dropped onto the specimens. Photos of the droplets on the fabric were taken from an angle of 45° after 10 seconds and 300 seconds, respectively. Five types of oils were tested using castor oil, vegetable oil, paraffin oil, n-hexadecane, n-heptane and n-decane.

Chemical Repellency Test

The chemical resistance was measured by the same method as that for the oil and aqueous liquid repellency test. Five fabric specimens with the size of 2 cm × 2 cm were placed in petri-dishes, and three droplets of the chemical (10.0 mL each) were placed on these specimens. Photos of the droplets on the fabric were taken from an angle of 45° after 10 seconds and 300 seconds, respectively. Twelve types of chemicals were tested using Acetic Acid, Sodium Hydroxide, Dimethylformamide, Sulphuric Acid, Acetonitrile, Butadiene, Acetone, Iso-propyl alcohol, n-hexane, Triethylamine, Methanol and Toluene.

Laundering Test

Accelerated laundering test was performed in accordance with the AATCC standard 61: 2013(1A). Fabrics (10 cm x 5 cm) were washed in an SDL Atlas launder-O-meter at 35°C along with 10 steel balls, and the AATCC reference detergent without any optical brightener was used in the laundering. One 45 min washing cycle is approximately equal to five commercial laundering cycles. Each specimen was then rinsed twice in deionized water and then dried in an air circulating oven for 30 min. All samples were conditioned according to ISO 139 at a temperature of 20 ± 2°C and RH 65 ± 5% prior to the testing.

Crocking Test

A crock meter (Toyoseiki, Tokyo, Japan) was used to determinate the durability of the coated fabrics against rubbing, in accordance to the standard AATCC 08:2013. Samples (130 mm × 50 mm) were prepared in both the warp and weft directions for wet and dry testing (10 cycles), respectively. A crock meter test cloth was used to determine the crocking fastness under a downward finger force of 9 N.

Thermal Resistance

Thermal resistance (Rct) was measured on the sweating guarded hotplate (SDL Atlas Pty Ltd., Kendal, UK), according to the standard ISO11092: 1993(E). Three specimens (30 cm × 30 cm) were prepared and conditioned at 20°C and 65% RH for 24 h. The specimen was then placed on the measuring plate for testing. The parameters of the testing were set as follows: measuring unit temperature (Tm) 35°C, air temperature (Ta) 20°C, air circulation speed 1 m/s, and RH 65%.

Moisture Management Property

The moisture management property of fabrics was measured on the SDL Atlas moisture management tester (MMT), in accordance with the standard AATCC-TM-195 (2009). Five specimens (80 mm × 80 mm) were prepared to do the test under the standard condition. A saline solution was penetrating from the top side to the bottom side of the sample in the test, while the wetting time, wetting radius, spreading time, absorption and overall moisture management capacity (OMMC) were reported on the associated computer.

Air permeability

Air permeability was measured on an air permeability tester (SDL Atlas Pty Ltd., Kendal, UK) according to the standard AS-2001.2.34: 1990. During the test, the specimen was clamped over an air inlet of the apparatus while air was sucked through it by a pump. The volume of air passing through the fabric was then measured using a flow meter. Five specimens were tested and the mean air flow (cm3/cm2/s) was calculated. The diameter of the test area was 20.0 cm and the pressure was 100 kPa.

5. Results

Data Colour

As shown in Figure 2, the WPU standard sample (#0) has provided excellent shading in comparison to the dyed TPU fabric (#3). The WPU coloured fabric has provided a strong shade with 80%, whereas the TPU dyed fabric is 17% weaker with a rating of 63%. The TPU coating has altered the colouration of the fabric by making it a pinker shade.

As shown in figure 3, the WPU and TPU fabric are fairly similar, however the TPU sample (#3) is 9.67% weaker in comparison to WPU. The standard colour of the WPU fabric (#0) does not match the TPU shade (#3), as the TPU colour is 2.76% weaker (see Figure 4).

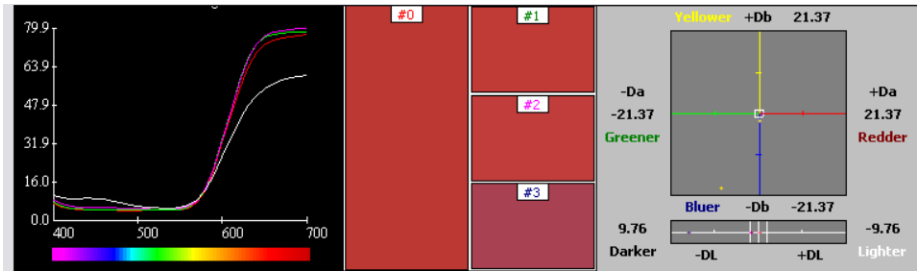


Figure 2 Data Colour Results Sample A.

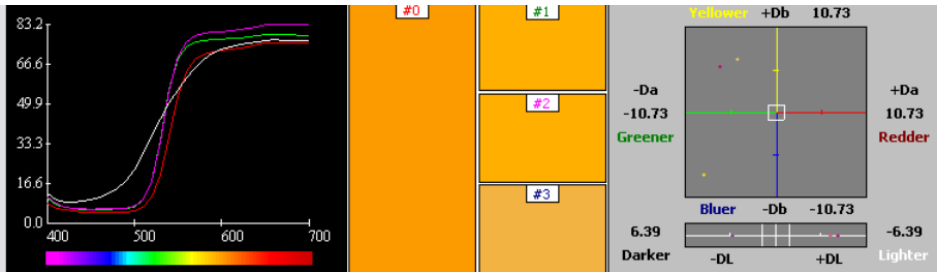


Figure 3 Data Colour Results Sample B

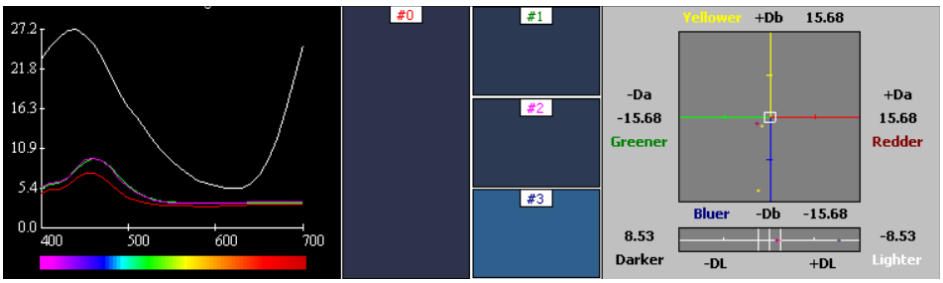


Figure 4 Data Colour Results Sample C

Water Repellency

As shown in Figure 5 and 6, it is evident that the cotton fabric (sample A") has greater wettability and therefore, water is easily absorbed into the fabric. Because of this, the cotton fabric has obtained a rating of 50% (see Table 1). In comparison, the 4% PT fabric (sample B") is highly resistant to water with a rating of 90%. This is due to the fact that the fabric has a low surface tension in comparison to the water surface tension. Furthermore, the WPU membrane and coloured fabrics (sample A-C) are also resistant to water with a rating of 80%, however the rating is 10% less than the 4% PT fabric. This is due to the fact that the WPU membrane has smaller pores.

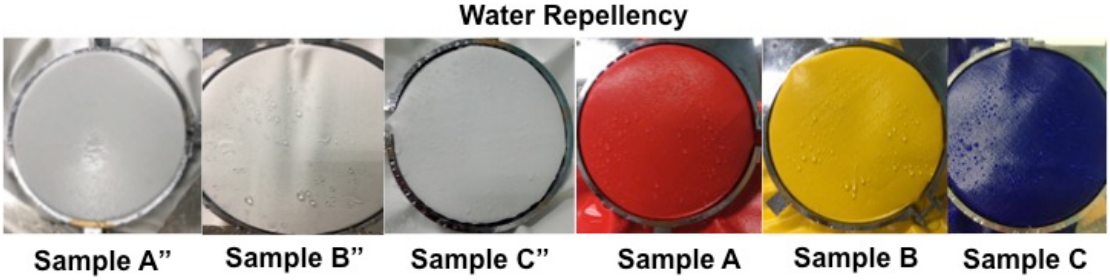


Figure 5 Water Repellency Test

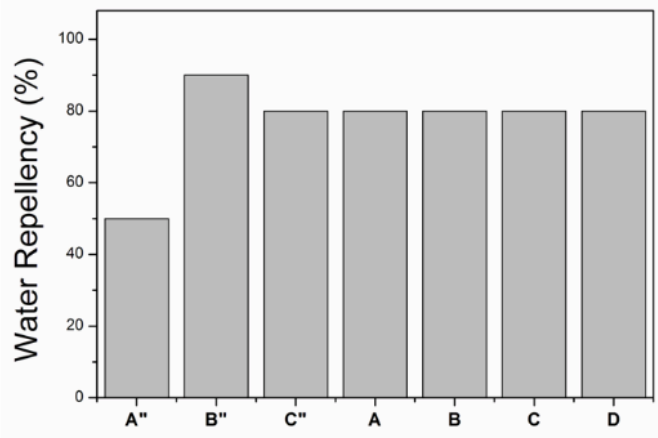


Figure 6 Water Repellency Results

Table 1 AATCC Standard spray test ratings

Rating	Description
100 (ISO-4)	No wetting or non-sticking of the upper surface
90 (ISO-4)	Slightly random sticking or wetting surface at sprayed points
80 (ISO-3)	Wetting of the upper surface at sprayed points
70 (ISO-2)	Partial wetting of upper surface at sprayed points
50 (ISO-1)	Completed wetting of the sprayed surface
0	Wetting of the whole upper and lower surface at sprayed points

Oil Repellency

The WPU membrane and coloured fabrics (samples A-D) are showing excellent resistance to all kinds of oils as compared to the 4% PT (sample B″) which is less resistant to vegetable oil and castor oil and is not resistant to n-Hexadecane and n-Heptane. The cotton fabric (sample A″) is not resistant with oil due to the wettability property of the fabric (Table 2).

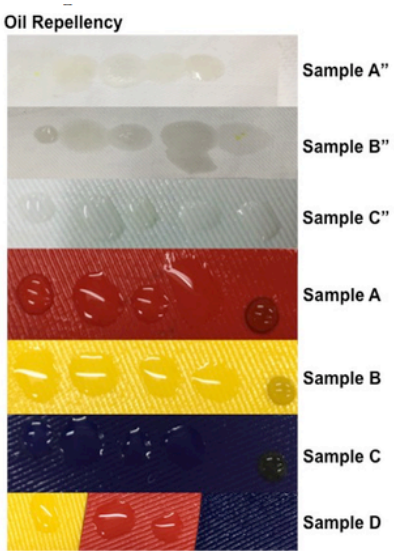


Figure 7 Oil Repellency Results

Oils	Oil resistant time (Seconds)						
	A″	B″	C″	A	B	C	D
Castor Oil	0	240	300	300	300	300	300
Vegetable Oil	0	240	300	300	300	300	300
Paraffin Oil	0	300	300	300	300	300	300
n-Hexadecane	0	0	300	300	300	300	300
n-Heptane	0	0	300	300	300	300	300

Table 2 Oil Repellency Results

Aqueous Liquid Repellency

The WPU membrane and coloured fabrics (samples A-D), show excellent resistance to all types of concentrations of water and alcohol (98-40%). The coated fabrics (samples B-D) all show excellent repellence to aqueous liquids. In comparison, the cotton fabric (sample A") is not resistant with liquid due to the wettability and absorbency property.

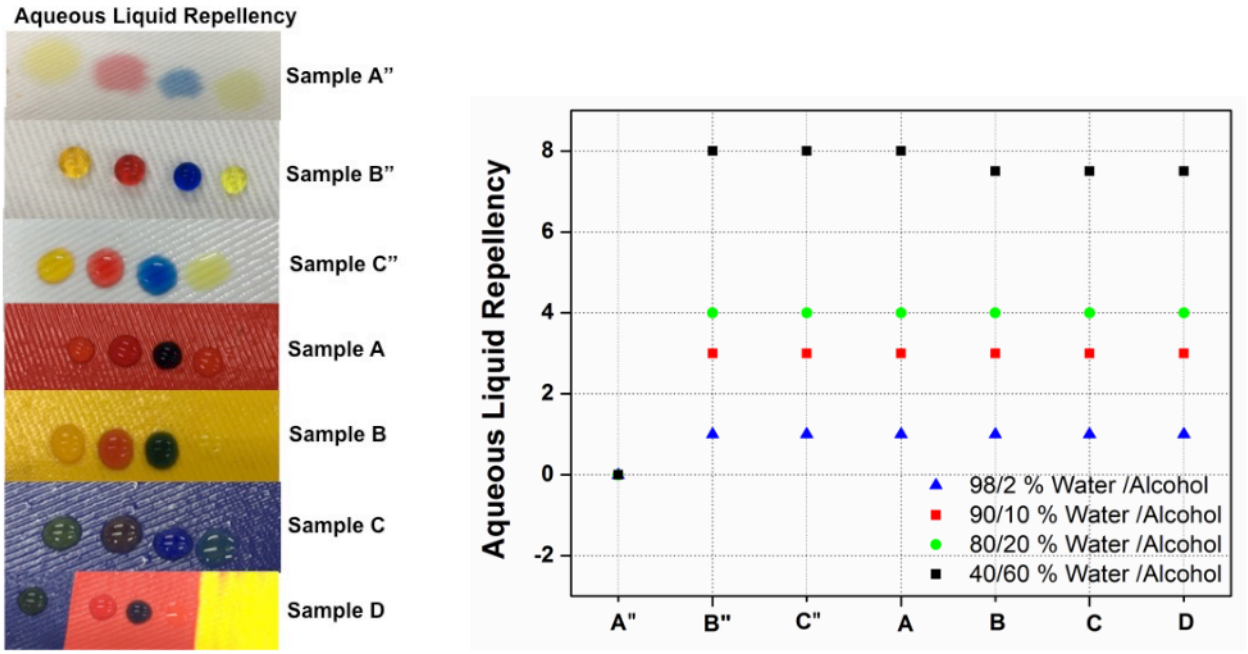


Figure 8 Aqueous Liquid Repellency Test Results.

Table 3 AATC Standard Aqueous Liquids

Aqueous repellency grade number	Composition	Surface tension (dynes/cm)
0	None (fail 98% Water)	-
1	98:2 Water: Isopropyl alcohol	49.0
3	90:10 Water: Isopropyl alcohol	32.0
4	80:20 Water: Isopropyl alcohol	33.0
8	40:60 Water: Isopropyl alcohol	23.0

Chemical Repellency

As shown in Table 4, the WPU and coloured fabrics (samples C"- C) are all resistant to Acetic Acid, Sodium Hydroxide, Dimethylformamide, Sulphuric Acid, *Iso*-propyl alcohol, Toluene, Acetonitrile and Butadiene, however the fabrics are less resistant to Acetone and Triethylamine due to the similar surface tension of the coated fabrics with the chemicals. The high surface tension chemicals show excellent resistance with less surface tension coated fabrics. In comparison, the cotton fabric (sample A") is not resistance to any chemicals due to the wettability and absorbency property. When comparing the WPU coloured fabric with the 4% PT, it is evident that the 4% PT fabric is resistant to some chemicals but not others (see Table 4).

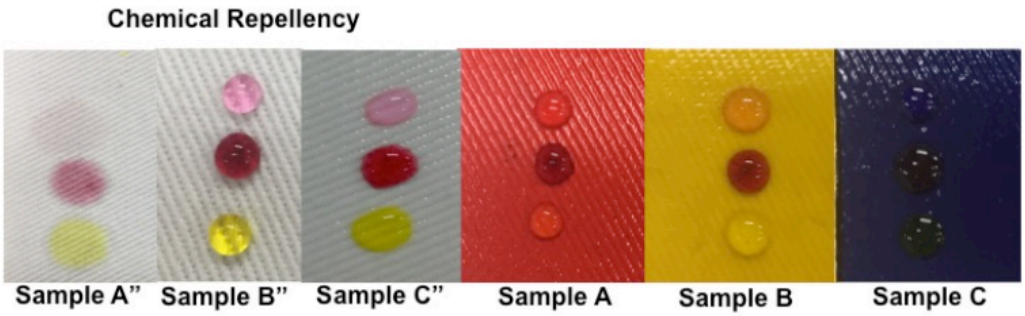


Figure 10 Chemical Repellency Test.

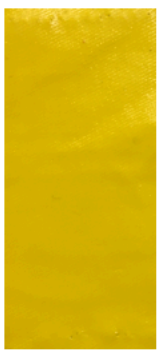
Table 4 Chemical Repellency Results

Chemicals	A"	B"	C"	A	B	C
Acetic Acid	0	300	300	300	300	300
Sodium Hydroxide	0	300	300	300	300	300
Dimethylformamide	0	300	300	300	300	300
Sulphuric Acid	0	300	300	300	300	300
Acetonitrile	10	57	223	300	300	300
Butadiene	0	12	300	300	300	300
Acetone	0	0	49	167	300	132
Iso-propyl alcohol	0	0	300	300	300	300
n-hexane	0	0	10	187	204	300
Triethylamine	0	0	10	102	102	30
Methanol	0	0	257	300	300	300
Toluene	0	0	300	300	300	300

Laundering Test

As shown in the images, sample A" and sample B" show no staining on the multi fibre fabric. After the laundering test, sample A and B show moderate staining onto the multi-fibre fabric (Nylon) with a rating of 4. Sample C also shows a fair staining on the multi-fibre (Nylon) with a rating of 3. Sample D shows no staining on multi-fibre fabric after laundering test which represents a good result. Sample A", B" and D all show no staining on multi-fibre fabric after the laundering test, in comparison to sample A, B and C which show some staining onto the multi fibre (nylon) fabric.

Sample A″	Multifibres	Grading	Sample B″	Multifibres	Grading
		*Wool 4/5* *Acylic 4/5* *Poluyester 4/5* *Nylon 4/5* *Cotton 4/5* *Acetate 4/5*			*Wool 4/5* *Acylic 4/5* *Poluyester 4/5* *Nylon 4/5* *Cotton 4/5* *Acetate 4/5*

Sample A	Multifibres	Grading	Sample B	Multifibres	Grading
		*Wool 4/5* *Acylic 4/5* *Poluyester 4/5* *Nylon 3.0* *Cotton 4/5* *Acetate 4/5*			*Wool 4/5* *Acylic 4/5* *Poluyester 4/5* *Nylon 4/5* *Cotton 4/5* *Acetate 4/5*

Sample C	Multifibres	Grading	Sample D	Multifibres	Grading
		*Wool 4/5* *Acylic 4/5* *Poluyester 4/5* *Nylon 3.0* *Cotton 4/5* *Acetate 4/5*			*Wool 4/5* *Acylic 4/5* *Poluyester 4/5* *Nylon 4/5* *Cotton 4/5* *Acetate 4/5*

Crocking Fastness Test

As shown in figure 11, Sample B″ and B show a similar result, with excellent rubbing property of the fabric for both wet and dry tested in the warp and weft directions (4-4.5). In comparison, Sample A the weft direction (3-4/5) in both dry and wet tests shows a better staining property as compared to the warp direction (2). Furthermore, Sample C presents a good staining property in the weft direction (3) however; colour has transferred onto the fabric in the warp direction for both wet and dry tests (2/3).

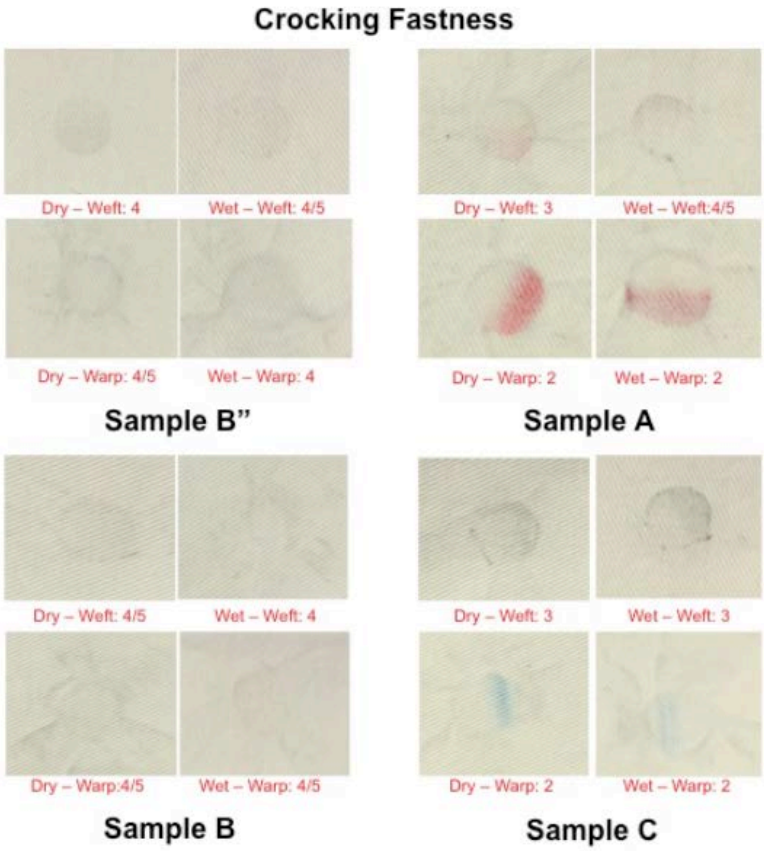


Figure 11 Crocking Fastness Test.

Thermal Resistance

Cotton	WPU	4%PT	RED	YELLOW	BLUE	MULTI
0.0184	0.0274	0.0125	0.0123	0.0117	0.0027	0.0152

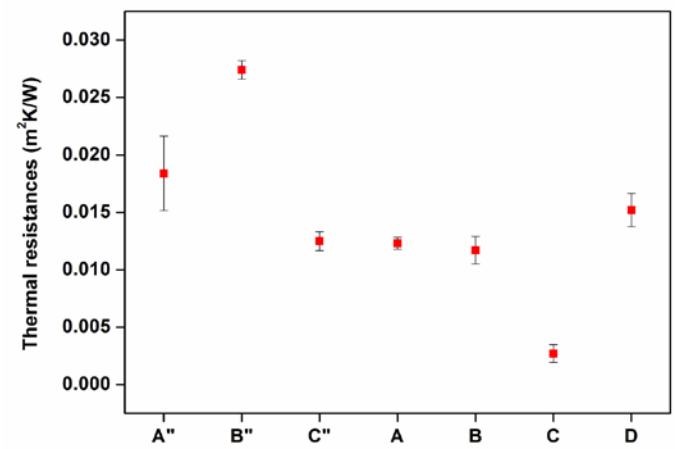


Figure 12: Thermal Resistance Test

Moisture Management

Due to the wettability of the cotton fabric, sample A shows the moisture management property of the fabric, water can easily be absorbed on the top and bottom of the fabric. In

comparison, the 4% PT and WPU coloured fabrics (samples B"- C), have a water proof membrane which allows water to show on only the top surface of the fabric (see Figure 13).

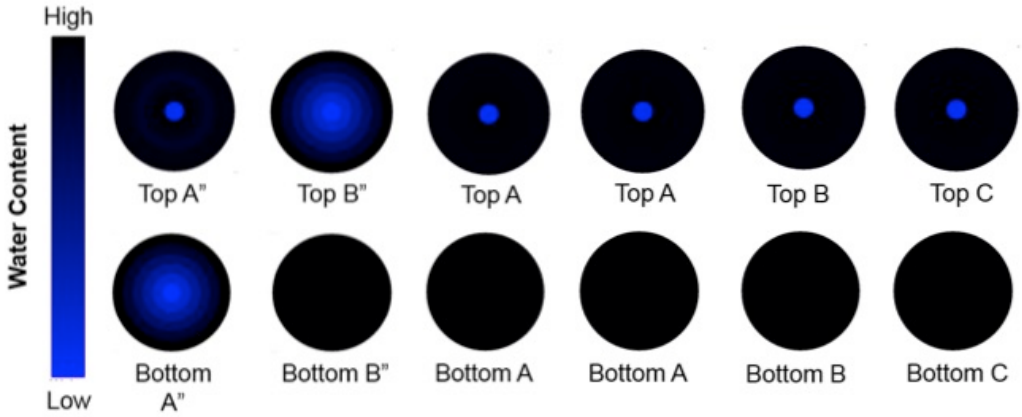


Figure 13 Moisture Management Test.

6. Discussion and Conclusions

In conclusion, the honour student Ms Olivia Williamson was enrolled in the project to learn the knowledge of cotton and coloured cotton. She has conducted a series of experiments to gain the skills of developing cotton-based performance textiles/protective fabrics and performed cotton related research in a research group at RMIT University.

Coloured cotton fabrics coated with waterborne polyurethane combined with polydimethylsiloxane and trimethylated silica were successfully developed by implementing colouring technology into the pad-knife-pad-cure methods. The results showed that the WPU membrane with pigments provided an excellent shading as compared to the pre-dyed fabrics. The repellency of all coloured cotton fabrics is similar with a water repellency rating of 80 as well as excellent resistance to oil, aqueous liquids and chemicals. The coloured fabrics after the laundering and crocking test showed excellent resistance. Furthermore, the cotton fabric exhibited an excellent moisture management property as compared to the coloured fabric because of the waterproof membrane which did not allow the moisture to transfer from the top to the bottom surface of the fabric. The developed coating technology has a great potential in developing performance textiles/protective clothing from cotton fibres.

7. Highlights

Knowledge on cotton fabrics together with technology and characterisation techniques were obtained through the progressing of this project. The cotton fabrics were successfully coated with WPU and PDMS-TMS and the coated cotton fabrics showed excellent protection against water, aqueous liquids and oils.

8. Future Research:

After facing technical issues when developing patterns for the coloured fabrics, it would be beneficial to undertake future research in order to overcome the issues faced and create patterns and designs into the fabric (see image below).

9. Reference

1. Arsheen Moiz, Arun Vijayan, Rajiv Padhye, Xin Wang. Chemical and water protective surface on cotton fabric by pad-knife-pad coating of WPU-PDMS-TMS, Cellulose, 23, 3377-3388 (2016).
2. Arsheen Moiz, Rajiv Padhye, Xin Wang. Coating of TPU-PDMS-TMS on Polycotton fabrics for versatile protection. Polymers, 9, 660 (2017).
3. Xin Wang. Pad-Knife-Pad coating of cotton fabric for versatile protection. Australian Cotton Research Conference, Sep 4-6, 2017.

Please email within 30 days after Summer Scholarship to: research@crdc.com.au

Appendices

Appendix A: Aqueous liquid Repellency Results

Description of solution	**Aqueous liquid grade**						
	A″	B″	C″	A	B	C	D
98/2	0	1	1	1	1	1	1
90/10	0	3	3	3	3	3	3
80/20	0	4	4	4	4	4	4
60/40	0	8	8	8	7.5	7.5	7.5

Appendix B: Air permeability Results

Fabric Samples	**Test**	**Pressure (kpa)**	**Diameter of Test Area (cm^2)**	**Rating**
A″	1	100	20	6 L/min
	2	100	20	7 L/min
	3	100	20	7 L/min
	4	100	20	6.5 L/min
Average Rating				**5.53 mL/sec**
B″	1	100	20	5.5 L/min
	2	100	20	5 L/min
	3	100	20	5 L/min
	4	100	20	5 L/min
Average Rating				**4.27 mL/sec**
A	1	100	20	0
	2	100	20	0
	3	100	20	0
	4	100	20	0
B	1	100	20	0
	2	100	20	0
	3	100	20	0
	4	100	20	0
C	1	100	20	0
	2	100	20	0
	3	100	20	0
	4	100	20	0