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Non-destructive Blended Fibre Analysis from Forensic Perspective

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Abstract – *The increasing use of fibre blends in clothing requires effective characterisation methods. Fibre analysts have often relied on the methods developed for single fibres to characterise fibre blends. This study was thus carried out using light microscopy and infrared spectroscopy to characterise binary-blended specimens and to evaluate the effectiveness of these tools on blended fibres. The blended samples were successfully characterised with the application of stereomicroscope, polarized light microscope (PLM), fluorescence microscope and attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR). PLM was more effective in discovering the presence of fibre blends compared to other techniques used in this work. Stereomicroscope allowed preliminary screening while fluorescence microscope provided useful information in fibre discrimination. High correlations (>80%) with combined infrared spectra of individual fibre components were reached for most blended samples. Therefore, light microscopy (stereo, polarizing and fluorescence) and infrared spectroscopy (ATR-FTIR) are recommended for the effective characterisation of fibre blends.*

Keywords: Fibre blend, Forensic analysis, Non-destructive

1.0 INTRODUCTION

Fibres are frequently encountered in forensic cases such as hit-and-run, assault and break-in. These fibres can be associated to a suspect by providing a proof of contact and may disprove his or her alibi. Of all the sources of fibres, clothing is most frequently found at crime scenes [1].

Cotton/polyester blend is the most popular choice of clothing material [2]. Such scenario indicates that there are high chances of encountering fibre blends at crime scenes. Fibre blends have different fibre types either in a yarn (blended fibre) or in a fabric (blended fabric) [3]. For instance, warp and weft yarns in a union fabric are made of different fibre types while intimate blends have different fibre types blended in a single yarn [4]. In practice, it is less common to have more than two fibre types blended together [5].

Fibre identification and comparison are two important aspects in forensic fibre examination [6]. For identification of fibres, physical and chemical properties are determined in order to categorise them. On the other hand, the properties of case fibres are compared with known fibres during fibre comparison. Scientific Working Group for Materials Analysis

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(SWGMAT) mentioned that the applications of at least stereomicroscope, comparison microscope and polarized light microscope (PLM) are desirable for such purposes [7]. In addition, stereomicroscope was recommended in ASTM E2228-10 to be the first step in fibre analysis [8]. Infrared analysis was also suggested to be done after examination with PLM, fluorescence microscopy and UV/visible spectroscopy [9]. Blended fibres are usually characterised using the same methods for single fibres that have only one fibre type. Various techniques can be applied on the fibres but non-destructive means are preferred in forensic analysis due to small amount of samples available. Previous studies on fibre mixture had employed analytical instruments including but not limited to optical microscopy [10, 11], scanning electron microscopy (SEM) [12], infrared spectroscopy [3, 13], Raman spectroscopy [14, 15] and microspectrophotometry (MSP) [16, 17]. Microscopy and infrared spectroscopy were considered to be quintessential in fibre identification as stated by Nayak *et al.* [18]. Hence, these two approaches were carried out to characterise binary fibre blends in this work in order to evaluate the effectiveness of the methods in analysis of fibre blends.

2.0 METHODOLOGY

2.1 Materials

Reference samples for 100% cotton and 100% polyester were kindly donated by Maple Tricot Industries (Batu Pahat, Johor). Reference samples for pure rayon, silk and wool as well as 25 types of clothing and fabrics were collected from clothing shops (Skudai, Johor), individuals residing in Selangor and Spark Manshop (Kuala Lumpur). A 5 cm x 5cm sample was prepared for each clothing and fabrics except for fabrics from Spark Manshop, which were analysed as it was received as cloth strips. The samples used in this work were tabulated in Tables A.1 and A.2. These samples contained blends of cotton, polyester, rayon, silk and wool, which are commonly found in Malaysia [19]. Therefore, there are high chances of retrieving the fibres at crime scenes.

2.2 Preparation of reference samples

The fibre content of the reference samples for cotton, polyester, rayon, silk and wool fibres were confirmed by comparing the photomicrographs with a colour atlas [20] and published infrared spectra [21, 22].

2.3 Stereomicroscope

The samples were observed with a Motic SMZ-168 stereomicroscope (Causeway Bay, Hong Kong) under magnification of 50x. Weave or knit patterns, twist direction and colours of the sample under transmitted and reflected light were noted.

2.4 Polarized light microscope

A Motic BA310 Polarizing Microscope (Causeway Bay, Hong Kong) was applied to examine warp and weft fibres mounted on glass slides with immersion oil (benzyl benzoate, $C_{14}H_{12}O_2$) (refractive index = 1.515-1.517) from Merck (Darmstadt, Germany). The fibres were viewed under a magnification of 400x to note the fibre morphologies and optical properties.

2.5 Fluorescence microscope

A Motic BA410 Fluorescence Microscope (Causeway Bay, Hong Kong) was used to examine the fibres mounted with the immersion oil. Fluorescence activities of the fibres were observed under 4',6'-diamidino-2-phenylindole (DAPI), tetramethylrhodamine-5 (and 6)-isothiocyanate (TRITC) and fluorescein isothiocyanate (FITC) filters (Table 1).

Table 1: Excitation and emission wavelengths of DAPI, TRITC and FITC filters.

Types of filters	Excitation wavelengths (nm)	Emission wavelengths (nm)
DAPI	350 ± 25	460 ± 25
TRITC	540 ± 12.5	605 ± 27.5
FITC	480 ± 15	535 ± 20

2.6 ATR-FTIR

Infrared spectra of the samples were generated using a Perkin-Elmer FTIR spectrometer Frontier (Waltham, U.S.A.) with Universal Attenuated Total Reflectance (ATR) of diamond and zinc selenide (ZnSe) crystals together with deuterated triglycine sulphate (DTGS) detector. Three different areas on each sample (5 cm x 5 cm) were measured. Each area was scanned from 4000-650 cm^{-1} for 32 times with resolution of 4 cm^{-1}. Average spectra from the three areas were obtained with Spectrum 10^{TM} spectra software. The software was also used to generate combined spectra by adding both average spectra of individual fibre components.

3.0 RESULTS AND DISCUSSION

3.1 Stereomicroscopy

Stereomicroscopic examination revealed that 14 out of 30 samples were woven and the remainders were knitted. In the knitted specimens, 12 samples that were mostly T-shirt had plain knit. Weave patterns of the 14 samples could be categorized into plain, twill and satin weaves. Due to its smooth surface, the manufacturers used satin weave to produce the purported silk fabrics, which were found to contain non-silk fibres under PLM and ATR-FTIR.

Under stereomicroscope, fibres with visibly difference in colour and morphology could be noticed (Table 2). Some of these fibres were found to be of dissimilar generic classes under PLM, as in a grey sample coded as 100CG that was labelled as 100% cotton (Figure 1a). Similar to 100CG, most yarns in this study had Z-twist (Figure 1b), which was also reported by Collier *et al.* [23]. In addition, several samples had different twist directions for warp and weft yarns. Thus, it was necessary to investigate the twist direction of yarns from both directions.

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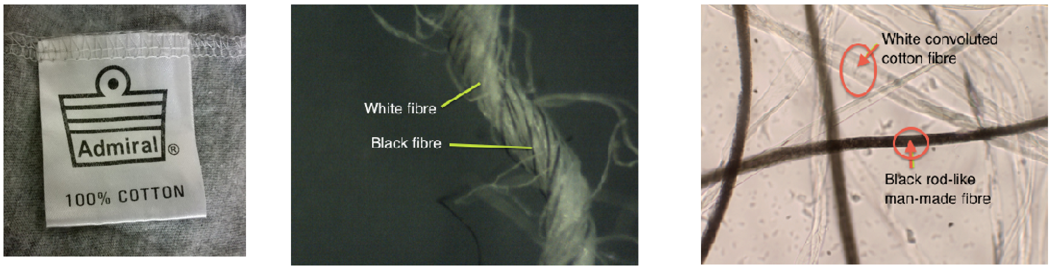

(a) (b) (c)

Figure 1: (a) Label of a sample coded as 100CG showing 100% cotton. (b) Presence of black and white fibres in 100CG yarns with Z-twist under stereomicroscope (50x). (c) Under PLM (400x), the black fibres were found to be man-made fibre while the white fibres were cotton fibres.

3.2 PLM

PLM was found capable of detecting the presence of fibre blends effectively since different fibre types had dissimilar morphologies (Table 2). This was helpful especially when other fibres were present for clothing with single fibres listed on its label, as evident in 100CG (Figure 1c). It could also confirm the presence of valuable fibres in clothing as labelled. Interference colours observed between crossed-polars aided in identification of silk fibres. Absence of silk fibres was noted for 3 specimens that were claimed to contain silk fibres. Silk fibres in the reference samples had irregular first order interference colours along the shaft in addition of presence of fibrils whereas the interference colours of the fibres in the purported silk samples were more consistent along the length. With the application of PLM, 6 samples were found to have disparities between fibre listing on labels and true fibre contents.

Dichroism of fibres could also be studied using PLM by observing whether there were differences in colour or intensity for parallel and perpendicular positions. Synthetic fibres in 5 samples displayed positive dichroic effect, which had greater intensity in parallel orientation compared to perpendicular orientation. Cellulosic fibres, which include cotton and rayon, were also dichroic for 5 samples. The wool fibres did not exhibit dichroism, which is in agreement with the results reported by De Wael and Driessche [24]. Since not all fibres are dichroic, it can be an additional point in fibre discrimination, as suggested by the authors [24].

Delustrant concentration of the fibres could be grouped into none, very low (0.03-0.05%) and semi-dull (>0.05%) [25]. Most of the samples in this work had semi-dull delustrant concentration. Two samples had different delustrant levels for both warp and weft fibres. These two specimens also had noticeable differences in diameter under PLM.

Perceived colour of the clothing may not be the same with the colour of the fibres present. In some of the samples, the colours of different fibre types differed because of different dye batches or poor dye uptake [16]. Therefore, it is recommended to make colour comparison under PLM instead of stereomicroscope during selection of clothing to be compared with questioned or case fibres.

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3.3 Fluorescence Microscope

When fibres of the same colour were subjected to examination under fluorescence light, the fibres displayed varied fluorescence activities (Table 2). White fibres in 4 samples containing cotton and polyester fibres had different fluorescence intensities even between different fibre types in the same sample (Figure 2). Contrasting fluorescence colour of several samples under different filters also occurred for the dissimilar fibre types. However, Grieve *et al.* cautioned against making inference based on disparities in intensity only [25].

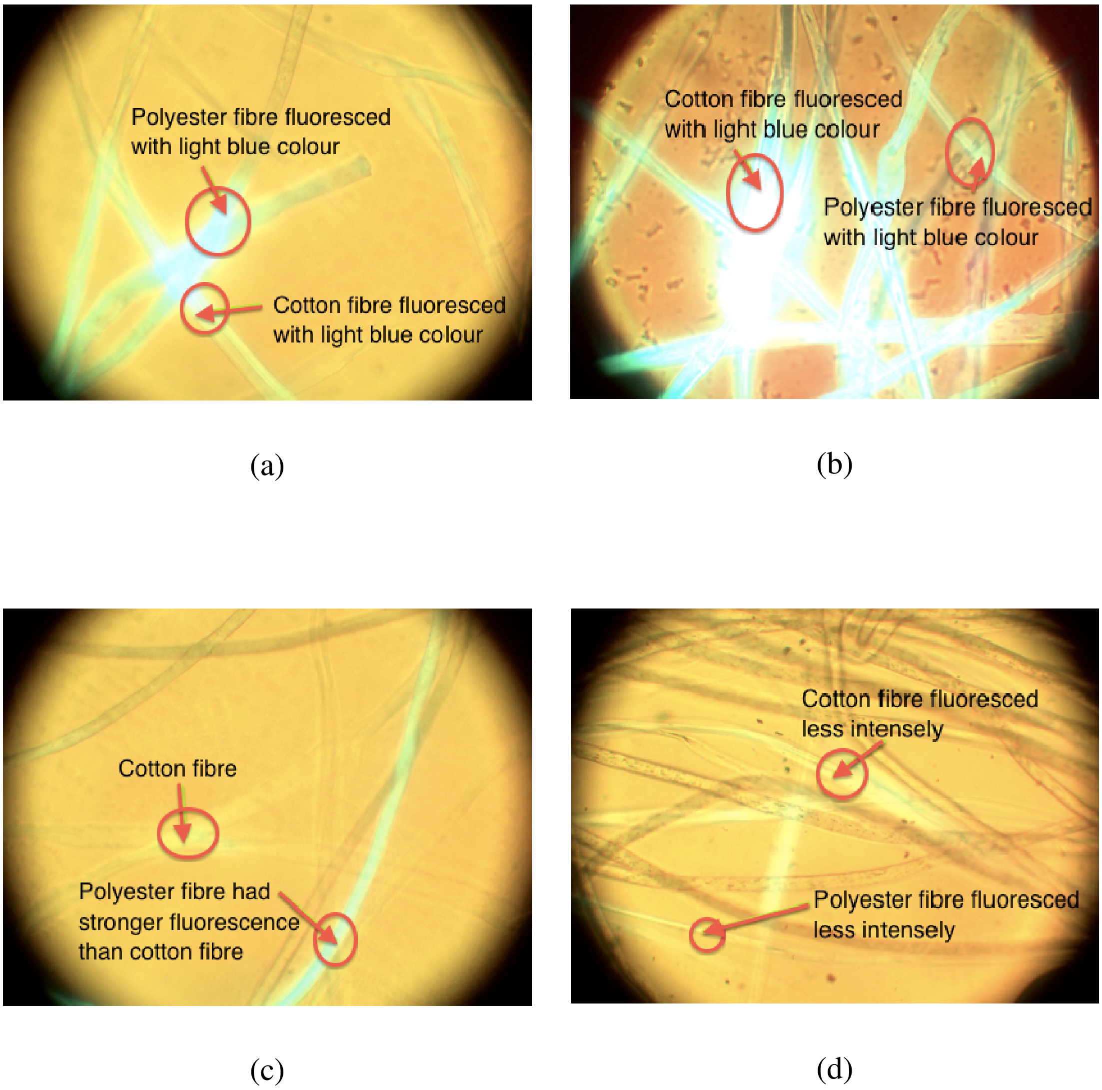

Figure 2: Fluorescence activities displayed by samples coded as (a) 50C50P, (b) 100CW, (c) 55C45P and (d) 100PW.

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Table 2: Characteristics observed using stereomicroscope, polarized light microscope and fluorescence microscope.

Types of microscopes	Characteristics	Examples of samples
Stereomicroscope	Weave or knit pattern could be noticed.	
	Difference in colours and morphologies of different fibre types were visible for some samples.	100CG, 80P20R, W-G
	Twist direction for warp and weft yarns were different for some samples.	WS-P, W-G
Polarized light microscope	Fibre morphologies could be used to identify fibre types.	
	Interference colours helped in identification of silk fibres and thus were useful in detecting fraud.	100SO, S-B, 45S55P
	Dichroism could be discriminating.	
	Delustrant concentrations among the samples did not appear to be the same.	
	Perceived colour may differ from the colour of fibres present (including intra sample variation).	65C35P, 60C40P-Br
	Diameters of warp and weft fibres in a few samples were visibly different.	100SO, 20C80P
Fluorescence microscope	Fibres of the same colour exhibited disparities in fluorescence activities.	100CW, 50C50P, 55C45P, 100PW
	Different fibre types had varied fluorescence colours under different filters.	65C35P

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3.4 ATR-FTIR

Concordant to previous reported results [1, 14], dye was found to have non-detectable effect on the spectral profiles in this work, which involved three pieces of fabrics with the same composition but different colours (Figure 3). The bands in the spectra corresponded to individual fibre types (Figure 4). There were 3 samples that had noticeable differences in spectral profiles, leading to lower correlations with the combined spectra of individual fibre constituents. Other than these samples, high correlations (>80%) with the combined spectra were achieved.

For blended samples, several areas had to be scanned since these areas could produce spectra of one particular fibre type, which may result in misinterpretation as single fibres. Some of the samples had different peak profiles for face and back sides of the fabrics. The same observation was also noted for samples with different fibre types for warp and weft yarns. Table 3 summarises the infrared analysis results.

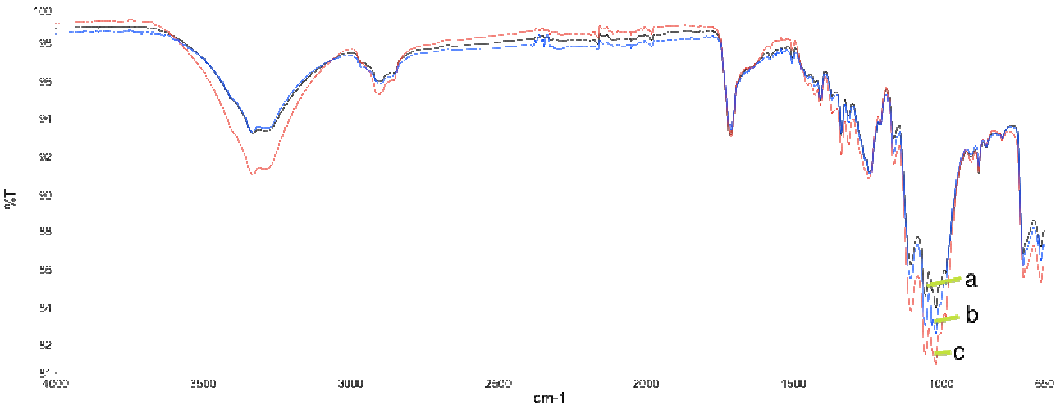

Figure 3: Average infrared spectra of three 60% cotton/40% polyester samples, which are (a) brown (b) red, and (c) white colours.

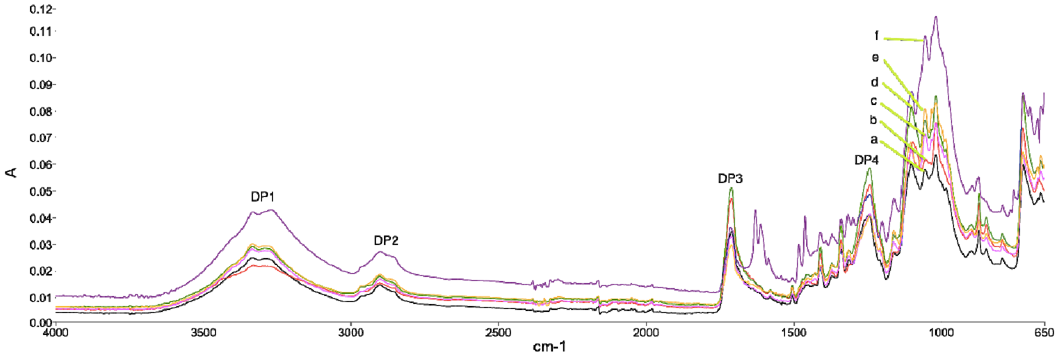

Figure 4: Average infrared spectra of samples with (a) 20% cotton/80% polyester, (b) 35% cotton/65% polyester, (c) 50% cotton/50% polyester, (d) 55% cotton/45% polyester, (e) 60% cotton/40% polyester and (f) 65% cotton/35% polyester. DP denotes diagnostic peaks examined for each spectrum where DP1 and DP2 represented O-H and C-H stretching vibrations respectively from cotton fibres while DP3 and DP4 were assigned for C=O and C-C-O stretching bands respectively from polyester fibres.

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Table 3: Overall results for ATR-FTIR analysis.

Types of blends	Results
Cotton/polyester	- All the polycotton samples except 100CG had characteristic peaks for the fibre components present. - Strong correlations (>80%) with the combined spectra were achieved except for 65C35P and 100CG.
Rayon/polyester	- The samples had similar diagnostic peaks with cotton/polyester blends but the O-H stretch was broader and only had one maximum. - All the samples had strong correlations -with the combined spectra.
Silk blends	- Samples other than WS-P did not have fibres consistent with silk fibres. - Warp and weft yarns for S-B were made of rayon and nylon 6 respectively.
Wool/polyester	- Samples including PW-B, 100WB and W-B150 had different peak profiles for face and back sides. - Strong correlations (>90%) with the combined spectra were obtained for all the samples except W-B150.

4.0 CONCLUSION

The fibre blends were successfully characterised using light microscopy, which involves stereomicroscope, PLM and fluorescence microscope, and infrared spectroscopy (ATR-FTIR). PLM was superior in detecting fibre mixture compared to other techniques employed in this study. Stereomicroscope may indicate possible presence of fibre blends. Fluorescence microscope offered additional comparable attributes in fibre discrimination. Most blended samples had high correlations (>80%) with the combined infrared spectra of individual fibre components. Hence, these approaches are recommended for the characterisation of fibre blends. True fibre contents could also be checked against fibre listing on labels using these characterisation methods. Future researches can include other analytical techniques and fibre types.

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APPENDIX

Table A.1: Description of reference samples for 100% cotton, polyester, silk, rayon and wool.

No.	Sample Code	Fibre content on label	Apparent colour	Type of garment
1	S100C	100% cotton	Brown	Fabric
2	S100P	100% polyester	Blue	Fabric
3	100SBr	100% silk	Brown	Collared shirt
4	100RB	100% rayon	Black	Shirt
5	100WBL	Superfine luxury wool	Blue	Fabric

Table A.2: Description of clothing and fabric samples.

No.	Sample Code	Fibre content on label	Apparent colour	Type of garment	Brand	Country of manufacture
1	100CR	100% cotton	Red	T-shirt	Kiko	NS
2	100CB	100% cotton	Black	T-shirt	Sheriff	Thailand
3	100CW	100% cotton	White	T-shirt	Basic Tees	NS
4	100PG	100% polyester	Grey	Sportswear	Uniqlo	China
5	20C80P	20% cotton/80% polyester	Blue	Collared shirt	SSK	Japan
6	35C65P	35% cotton/65% polyester	Blue	Blouse	Canada	U.K.

NS – not stated on cloth label

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Continuation of Table A.2: Description of clothing and fabric samples.

No.	Sample Code	Fibre content on label	Apparent colour	Type of garment	Brand	Country of manufacture
7	50C50P	50% cotton/50% polyester	White	T-shirt	Fourskin	China
8	55C45P	55% cotton/45% polyester	White	Taekwondo uniform	Taekwondo Koryo	NS
9	60C40P	60% cotton/40% polyester	Red	Shirt	Seed	Malaysia
10	60C40P-Br	60% cotton/40% polyester	Brown	Shirt	Zenana Outfitters	Indonesia
11	60C40P-W	60% cotton/40% polyester	White	Shirt	Aéropostale	Cambodia
12	65C35P	65% cotton/35% polyester	Blue	Denim jeans	Applemints	NS
13	100CG	100% cotton	Grey	Sportswear	Admiral	Bangladesh
14	100PW	100% polyester	White	Fabric	Victory Mycotex	Japan
15	25P75R	25% polyester/75% rayon	Green, brown	Shirt	West Kei	U.S.A.
16	80P20R	80% polyester/20% rayon	Grey	T-shirt	Uniqlo	China
17	53R47C	53% rayon/47% cotton	Yellow	Shirt	Aquagirl	China
18	100SO	100% silk	Orange	Fabric	Moschino	NS
19	45S55P	45% silk/55% polyester	Pink, white	Scarf	NS	NS
20	S-B	(Claimed to be silk)	Blue	Scarf	NS	NS

NS – not stated on cloth label

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Continuation of Table A.2: Description of clothing and fabric samples.

No.	Sample Code	Fibre content on label	Apparent colour	Type of garment	Brand	Country of manufacture
21	WS-P	Warp: 100% silk, Weft: 100% wool	Pink	Scarf	Roberta di Camerino	China
22	W-G	Super 120 wool	Grey	Fabric	Taylor & Littlewood	NS
23	PW-B	Polyester and wool	Black	Fabric	Raymond	NS
24	100WB	All wool	Black	Fabric	Trabaldo Togna	Italy
25	W-B150	Super 150's	Black	Fabric	Vitale Barberis Canonico	Italy

NS – not stated on cloth label