

## **Abstract**

Fibers found at crime scenes can provide useful information to an investigation. Positive associations between fibers from different sources can establish a link in the crime and help direct the case. The key to connecting two fibers is the inability to make distinctions in physical, chemical, and optical characteristics. To further associate two fibers, to the exclusion of other fibers, the rarity of the fibers must be considered. This rarity results from the frequency of the particular fiber type in combination with the color of the fiber. In the past, blue denim has not exhibited characteristics required to distinguish one blue denim product from another. Thus, blue denim fibers were classified as ubiquitous and typically are not used for comparisons. Traditional blue denim is composed of cotton fibers dyed with indigo (Vat Blue 1). According to a study done in Germany in 2006 (Grieve, Beirmen, and Schaub), “approximately 95% of all the denim material examined was made of 100% cotton and displayed the dye spectrum typical of Vat Blue 1”. The purpose of this research is to determine if the manufacturing of blue denim material has changed significantly within the past ten years. If a significant change has been seen, blue denim could be reestablished as a target fiber. Samples of 108 blue jeans material were randomly collected by donation. Each of the samples were analyzed by polarized light microscopy (PLM) to ensure the composition as stated by the manufacture. According to the manufacturer’s labels, there has been a significant change from the traditional 100% cotton composition that was seen 10 years ago. Around 30% of this study’s samples were manufactured using 100% cotton. The samples showed the addition of fibers such as spandex, polyester, and ramie. UV-Vis microspectrophotometry was employed in order to establish if any dyes utilized differed from the standard Vat Blue 1 (indigo). This study has determined that more than Vat Blue 1 is being used to dye blue jean material. It was found that 8% of the samples used solely Vat Blue 1 dye and

85% used Vat Blue 1 in addition to other dyes. The remaining samples made up the 7% attributed to other dyes not including Vat Blue 1. The compositions of blue denim and the variations in dye establish a significant change in the manufacturing of blue denim in the past ten years.

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# Analysis of Dyes in Blue Denim by Microspectrophotometry

## **Introduction**

“Every contact leaves a trace.” The famous Locard’s Exchange Principle is the guiding standard for many fields of forensic science, including trace evidence, such as, fibers, paint, and glass. In general, this principle indicates that people or objects will always leave or take away some form of evidence wherever they go. This concept is exploited by a variety of types of evidence analyzed by forensic scientists. Fiber analysis in Trace Evidence depends on Locard’s Exchange Principle. (Robertson & Roux, 2010)

Fibers are the units which make-up textiles that are woven or knitted into fabrics. These fibers can be natural (plant, animal, or mineral) or manufactured (synthetic or regenerated from cellulose). Fibers can be transferred from person to person, person to object, object to person, or object to object. Objects such as carpet and blankets transfer and accept fibers rather readily. However, a fabric’s ability to shed and the force of the contact will ultimately affect whether a fiber transfer takes place. For example, a tightly woven nylon windbreaker would require much more force to transfer fibers as opposed to a pair of blue jeans. In this example, the blue jeans

may leave fibers behind from the wearer simply by sitting in a car seat, but it may require the force of a moving vehicle in a hit-and-run to allow fibers to be transferred from the nylon windbreaker. (Borland, 2013) Additionally, the analysis of fibers depends on finding the trace evidence. When available and found, fibers can then be collected and compared to determine if they originated from the same source. In most cases the trace analyst will examine debris from both the victim's and the possible suspect's articles of clothing. Articles of clothing will be picked, scraped down, or tape-lifted in order to potentially find any foreign fibers. The evidence will then undergo comparison analysis and a determination will be made as to the association or discrimination between fibers. Positive associations have the potential to establish a link in the crime and help direct the case. This connection, through associations of fibers, can imply contact between people, places, and objects.

The inability to make distinctions, positive association, is the key to connecting two fibers from different sources. Distinctions can be made through comparison of two fibers' physical, chemical, and optical characteristics. The rarity of the fibers must also be considered when associating two fibers. The fiber identity (fabric composition), as well as the color of the fiber are considered when determining the rarity of a fiber. The fiber can be examined by Polarized Light Microscopy (PLM) to determine physical characteristics that may help identify it. In addition to microscopic examination, instrumental analysis can provide information regarding the fiber's color. The combination of both fiber identity and color will discriminate only if the combination is not frequently found. Blue denim is an example of a fiber that was previously considered ubiquitous, thus not exhibiting these rare qualities. (Goodpaster & Liszewski, 2009)

Historically, blue denim has been noted to be a weave of cotton fibers dyed using a standard Vat Blue 1 (indigo) dye. Blue denim fibers did not exhibit characteristics required to distinguish one product from another. Due to this, blue denim has had limited use in fiber comparisons and has not held significant evidential value. The denim weave consists of both blue dyed threads and plain white (colorless fibers). The blue dyed threads are known as warps and the plain white cotton threads are known as wefts. Denim is constructed so that the blue threads (warps) are more visible than the white threads (wefts). (Borland, 2013)

According to a study by Grieve *et al.*(2006), “approximately 95% of all the denim material examined was made of 100% cotton and displayed the dye spectrum typical of Vat Blue 1”. This study established that the typical 100% cotton material was still heavily popular for blue denim. The denim/cotton material first came to use for workers because of its strong durability. Since its debut, blue denim has become wildly diverse in the fashion world. In the 2006 study, the 5% that differed from the traditional indigo-dyed cotton denim were found in women’s fashion items. This introduced the possibility of an increasing trend of variation in the manufacturing process of blue denim. As fashion evolves, the manufacturing methods have to change. Manufacturers have begun to incorporate other fiber types in denim including polyester, spandex, and ramie. By incorporating different fiber types, the ability to distinguish between products is established, increasing the evidentiary value of blue denim.

One of the most important aspects in fiber analysis is the color of the fiber. The color is attributed to the dyes and pigments used to color a particular fiber. Originally, an extract from the plant *Indigo tinctoria* was used to dye blue denim clothing. Indigo is now almost exclusively found in a synthetic form. Cotton uptakes the indigo more readily than other fibers, but is still challenging to process. Indigo dye is not soluble in water, which poses a problem in the dyeing

method. The dye must first be reduced to its water soluble form known as indigo white or leuco indigo. The denim can then be submerged several times in order to form layers. These multiple layers and the difficulty of dyeing with indigo explain why denim has a tendency to fade over time. Once pulled from the dye solution, the indigo white will oxidize back to its blue form. When re-oxidized the blue dye will be relatively permanent. However, the indigo will slowly wash out over time. Natural fibers, such as cotton fibers, undergo this tedious process much easier than synthetic fiber types. Referring back to the study by Grieve *et al.* 5% of denim material differed in fiber composition and the dye used. The variation in dyes is likely due to the increase in synthetic fibers used. The indigo dye will not react with synthetics as it does with cotton. The dye will immediately wash out of the fiber; therefore, a cotton/polyester blend will appear to be much paler when using the standard Vat Blue 1 dye. With the increase in blends of denim, hues of the indigo derivatives have been used. There are a number of indigo derivatives available including brominated indigo, chlorinated indigo, etc. At least seven different derivatives have been noted to have been used in fashion dyeing. An increase in the use of these derivatives, as well as any other dyes, would provide distinction due to the rarity of their roles in the market. (Borland, 2013)

Microspectrophotometry is a generally accepted approach that reveals characteristics of fibers imparted by dyes and pigments. Pigments and dyes are designed to absorb in the visible light range (400 nm – 700 nm), which is the range of wavelengths visible to the human eye. Microspectrophotometry uses the visible and UV light ranges (200 nm – 800 nm) in order to differentiate between indigo and its derivatives, although less distinction has been made between the individual derivatives. Using the UV-vis-microspectrophotometer, two important utilities are employed. The instrument contains a microscope and a spectrometer. The microscope provides

enhancement and magnification, while the spectrometer is responsible for the measurement of the absorption of light. The instrument first focuses white light on the sample, as white light is the combination of all colors or wavelengths reflected. This light will be transmitted through the sample, and the absorbed wavelengths will be recorded. Chromophores, molecules responsible for color, in a fiber and dye/pigment will absorb certain wavelengths of light characteristic of the electronic structure of the molecules present in the fiber and dye/pigment. The wavelengths of light transmitted through the fiber will then be recorded. For example, a blue dye or pigment will primarily absorb light in the wavelengths corresponding to blue. Different hues or shades of blue will absorb differently, but will remain in the area of the wavelengths characteristic of each blue color. The absorbance of light at each wavelength is then measured based on the transmission of light. The spectrum, “absorption line,” is displayed as a function of the wavelength vs. the absorbance of the light. Through this approach a spectrum from a fiber can be obtained that is characteristic of the dye or pigment used. Comparisons are made by looking at the similarities and/or differences in the absorption line. The absorption line is characteristic of the chromophore structures present and allows the analyst to make determinations whether the pigments and dyes used are the same between a known and unknown. (Wiggins, Holness, and March, 2005)

This study sought to determine if manufacturers of denim products are differing the composition of their fabrics; thus, differing the dyes used as well. Using a variety of fiber types and dyes in the manufacturing process would allow blue denim fibers to be of more significance in trace evidence, allowing for denim to be a target fiber for comparisons. Polarized light microscopy and microspectrophotometry were employed in this study to confirm the type of fibers in the denim and determine if different dyes were being used.

## **Materials and Methods**

Fibers were taken from 108 samples of blue denim material that were donated. The samples consisted of jeans, shorts, skirts, a jacket, and a hat. Distinguishing information such as brand, country of origin, and identifying numbers were obtained for each of the products and compiled in Microsoft Excel®. Along with this information, a unique identifier was assigned to each of the products. Samples from each of the products were collected by pulling or cutting fibers and placing them in a labeled bag. It was found that the pockets were the easiest source of attached fibers that could easily be removed without damaging the jeans. The samples are listed in Table 1.

Polarized light microscopy (PLM) was used to confirm the fiber composition for each sample. A fiber from each sample was mounted onto a slide using Meltmount® (refractive index 1.539). The Zeiss SteREO Discovery V.8 stereoscope was used in the mounting process. An Olympus BX51 polarized light microscope was used to observe each slide in order to determine if the composition was consistent with the listed composition on the label. This was determined by the physical, chemical, and optical properties of each fiber. The PLM was also used to identify how many different color fibers were found in each sample. This ensured that all dyes used in the process are later identified.

All 108 samples were analyzed using microspectrophotometry to compare the dye spectra. Three fibers from each sample were selected and mounted on a quartz-slide. Glycerin was used to secure the fibers to the slide before covering with a quartz cover slip. In the case of more than one color found in the sample, three fibers of each color were mounted. Nine spectra were obtained from different areas along the length of the fiber. These were recorded in the ultraviolet and visible with a range from 250 nm to 820 nm provided by the CRAIC Technologies microspectrophotometer. Due to the small size of fibers a 40 x objective was used

with an aperture of four. Resolution was achieved by averaging five and ten scans. The instrument was subjected to a wavelength check using holmium oxide and didymium filters, as well as a photometric check using neutral density filters of 0.1, 0.5, and 1.0 g/cm<sup>3</sup>. The “Autoset Optimize” function was used to determine an appropriate integration time.

After obtaining the spectra for the samples, an indigo powder standard was analyzed. The powder was varied between solid, moistened, or crushed. The indigo spectrum was analyzed three different times with the same number of replicates as the fibers. To achieve a similar standard sample to the blue jean fibers, a sample of cotton was dyed. To achieve this, 600 mL of water was heated to between 50 - 60 °C. Two grams of synthetic indigo was added to the heated water and made alkaline by adding 10 mL of 6 M sodium hydroxide (NaOH) with constant stirring. The indigo solution was reduced by mixing 6 g of sodium hydrosulfite (Na<sub>2</sub>S<sub>2</sub>O<sub>4</sub>) into the solution. This gave the leuco indigo the capability of penetrating the fibers in order to complete the dyeing process. Fifteen minutes was allotted to ensure the mixture achieved a yellowish-greenish color. Ten pieces of cotton were submerged in the solution from 5 to 15 minutes. The submersion time for each standard prepared is listed in Table 2.

## Results and Discussion

The blue denim samples tested exemplified changes in both fiber composition and color. Figure 1 demonstrates that only 30% of samples were composed of 100% cotton. This is a large difference to the 95% of samples containing 100% cotton as demonstrated by Grieves *et al.*. Most of the samples in this study proved to be blends of cotton with one or more different fiber types. The fiber types that have been introduced into the blue denim industry are spandex, polyester, and ramie. The most common composition found was a blend of cotton and spandex comprising 36% of the samples. The amount of samples seen to use solely vat blue 1 indigo



dye changed from 100% to 8%. Figure 2 displays the results of the dye composition of the samples separated by whether the standard vat blue 1 indigo dye was used, a different dye, or both. A combination of both the Vat Blue 1 and other dyes was most observed in 85% of the samples. Only 7% of the samples used strictly other dyes. The results were seen to be much different than the study presented 10 years ago.

These results support that the manufacturing process for blue denim has changed. Both the change in the fiber type and color could be attributed to the emersion of blue jeans into fashion, which is the main industry for blue denim. This was the materials first usage and continues to be its primary use. However, historically blue jeans were not as prevalent as they are today. Originally they were made for American gold miners in the 1800's because the miners needed material with high durability. Somewhere in the 1930's the popularity of blue jeans expanded to more than just the average worker. They became frequently seen in Hollywood with the production of old western films. Soon after World War 2, young Americans began wearing them as a mean of rebellion. During the 1960's and 1970's, the style of blue jeans began to evolve. The historical use of denim switched from the work wear to a fashion statement so the composition of the jeans has gone from durability to fit and fashion. Blue jean manufacturers have begun to incorporate elastic material into their products in the form of spandex. Blue jeans not only started to incorporate spandex, but also other non-stretchy materials, such as polyester. The manufacturing process of using different blends of fiber types for blue denim can be attributed to the evolution of blue denim clothing made to have a 'fashionable' appearance. (Borland, 2013)

The change in fiber composition of blue denim is of importance to trace evidence. Ten years ago when cotton was the basis of blue denim, it was difficult to distinguish between blue

denim products. Trace evidence relies on distinctions. If all blue denim products were still composed of the same materials, there would be no way to definitively say whether a fiber came from one distinct item. With the composition of blue denim deviating from solely cotton, more distinction can be seen between blue denim products. Therefore, a blue denim fiber found in casework may offer more evidentiary value.

In addition to the fiber type, the fiber color allows for even more differentiation. Previously, it was seen that most blue denim products used the same standard Vat Blue 1 dye. Now different dyes are being used. This could be a result of the addition of different fiber types in the composition. With the addition of different synthetic fibers, variation is to be seen in the dyes being used to dye the blue denim. This is due to the nature of the indigo dye. Cotton fibers undergo the rigorous process of dyeing with indigo better than synthetic fibers do. With synthetic fibers, the indigo dye will not adhere and easily wash out. To compensate for this, blue jean manufacturers have begun to use different derivatives of indigo. These derivatives also provide different hues. In previous literature, it has been stated that “at least seven different derivatives have been noted to have been used in fashion dyeing.” (Grieve) An increase in the use of these derivatives allows products to become more distinguishable from others in the market.

## **Conclusions**

Blue denim has evolved from ten years ago. The changes seen in the fiber type and the color allow for more distinction between different blue denim products. Being able to make more distinctions between blue denim products increases the rarity of blue denim fibers. With the increase in rarity, a positive association or discrimination in a fiber comparison is more likely to occur. Blue denim products today, have the possibility to provide probative information in an

investigation. The composition and dye differentiation in newer denim products prove to have enough discriminating factors to allow them to become target fibers in forensic cases.

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### Tables and Figures

Table 1: Sample List

Unique ID	Brand	Origin	Identifying numbers
Jean001	Route 66	Mexico	WPL6428, RTH15BI, TZS-A936-030, 084084448330
Jean002	Wrangler Jeans Co.	Egypt	RN# 130273, 4CDWHNT, YZS-W862-051, 083625585138
Jean003	Arizona	Mexico	RN# 61252, 430-1117AN, 001, 00012-5, Y7169
Jean004	Levi Strauss & Co.	Lesotho	WPL-423, 409500214
Jean005	Levi Strauss & Co.	Mexico	WPL-423, 429240031
Jean006	Levi Strauss & Co.	Mexico	052178067737, WPL-423, RT3095A, 730S 409500214
Jean007	Anvil	Cambodia	
Jean008	Levi Strauss & Co.	Mexico	WPL-423, CA00342, 429240031
Jean009	Arizona	Mexico	RN #61252, 430-1117AN, 003, 0012-5, X6335

TINNIN • ANALYSIS OF DYES IN BLUE DENIM BY MICROSPECTROPHOTOMETRY 12

Jean010	Levi Strauss & Co.	Columbia	WPL-423, 439350002
Jean011	Levi Strauss & Co.	Pakistan	WPL-423, 409500006
Jean012	Roz & Ali	China	RN 120246
Jean013	Lane Bryant	China	RN 116640, AF11071580, S 500314 12
Jean014	Levi Strauss & Co.	Mexico	WPL-423, 005500033
Jean015	Diesel Industry	Italy	RN# 93243, OOE8M200014
Jean016	Levi Strauss & Co.	Mexico	WPL-432, 409500006
Jean017	Jolt	China	RN# 39831, IMP#82411
Jean018	Jolt	China	RN# 39831, IMP# 35025
Jean019	Clarion	Turkey	CLJ0M105-0078
Jean020	Abercrombie & Fitch	Mexico	RN# 75654
Jean021	Mossimo Supply Co.	China	RN# 17730, VN1181300
Jean022	VF Jeanswear LP	Bangladesh	3071748, QVS- LMDH50301, 083625338024
Jean023	Fashion Bug	China	RN# 72882, ZD11070930
Jean024	Fashion Bug (#1)	China	RN# 72882, CS11070022
Jean025	Fashion Bug (#2)	China	RN# 72882, CS11070022
Jean026	American Eagle Outfitters	China	RN# 54485
Jean027	L.A. Blues	China	RN# 72882, CS11070022
Jean028	Venezia	Cambodia	RN# 118641, CS11470225
Jean029	Arizona	China	RN# 93677, 73090-7
Jean030	Fashion Bug (#3)	China	RN# 72882, CS11070022
Jean031	New York & Company	China	RN# 23243
Jean032	Hydraulic	China	RN# 99678, CUT# J122288, STYLE# J6442
Jean033	Jessica Simpson	China	PO# 4500145929, Style# JSB20100-NVW
Jean034	Hollister Co.	Guatemala	RN# 102573
Jean035	Kim Rodgers		RN# 31104
Jean036	Riders		130707L, SPS-V616-019, 08362542297
Jean037	Croft & Barrow		RN# 73277
Jean038	Wrangler	Mexico	RN# 130273, 94LSWDV, 9P4SWDV, TZS-D341- 036, 672787956452
Jean039	Lee	Mexico	RN# 130273, 2055512, TZS-K285-022, 755107829930
Jean040	Lee	Mexico	RN# 130273, 2055516, JIM-Z879-019, 084084327604
Jean041	Levi Strauss & Co.	Columbia	WPL-423, 005592765
Jean042	Lane Bryant	Vietnam	RN# 118641, CS13010262
Jean043	New York & Co.	China	RN# 23243
Jean044	New York & Co.	China	RN# 23243
Jean045	Levi Strauss & Co.	Mexico	WPL-423, 155161101
Jean046	Roz & Ali	China	RN# 111052
Jean047	Zanadi	China	PJA492S4
Jean048	Levi Strauss & Co.	Indonesia	WPL-423, 095203486
Jean049	Banana Republic	Turkey	RN# 54023

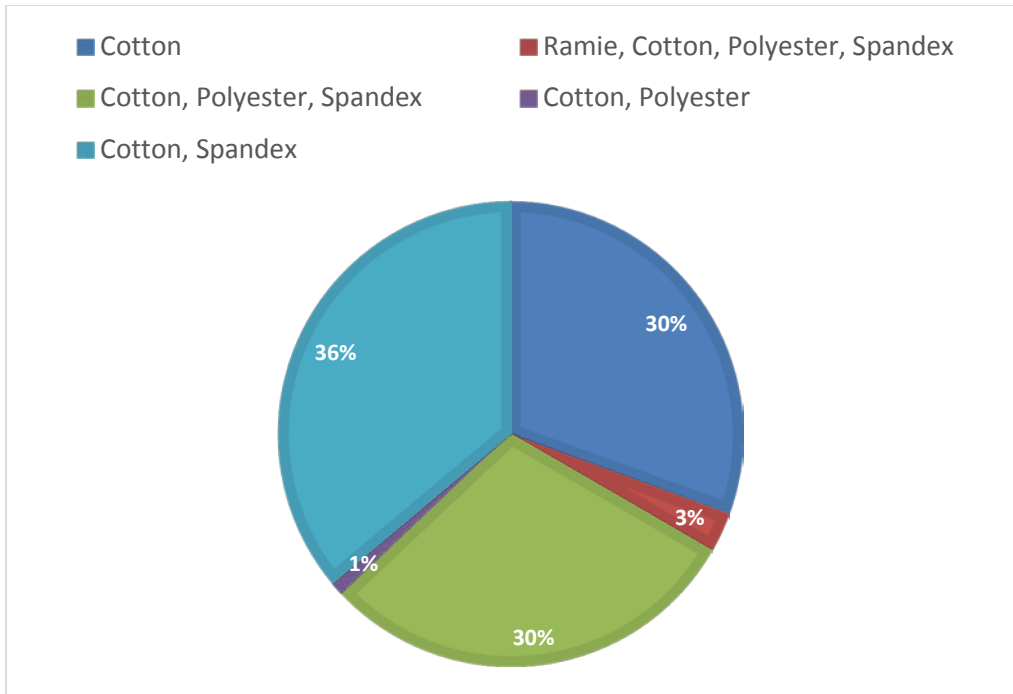
TINNIN • ANALYSIS OF DYES IN BLUE DENIM BY MICROSPECTROPHOTOMETRY 13

Jean050	DKNY Jeans	Philippines	RN# 52002
Jean051	Calvin Klein Jeans	Mexico	RN# 36009, ZD85C5467, 2502834, 000145
Jean052	Old Navy	Cambodia	RN# 54023, S/741258-02, V/1195
Jean053	L.A. Blues	Bangladesh	RN# 72882, CS11160106
Jean054	New Direction	China	RN# 31104
Jean055	Lane Bryant	China	RN# 118641, CS11071947
Jean056	SK <sup>2</sup> Apparel	China	RN# 114936, 692050778062, 1795409858
Jean057	GAP	U.S.A.	RN#54023, 121379BZ22 8002, PO.GQ238-1A 9/00, 121515-00-2 1603
Jean058	Old Navy	Sri Lanka	RN# 54023, S/562760-10, V/700091159
Jean059	GAP	Mexico	VDNR03873 POUJ252, 121846-7L 68609, 121846-00-1 1403
Jean060	J. Crew	Guatemala	P.O.# 3375651, Cor. 110488
Jean061	Wrangler Jeans Co.	Mexico	RN# 130273, 11A00019AP 71896, 9MWZSD, JIM-Q404-008
Jean062	GAP	Mexico	VDNTO 873PO RL655, 121557SZM9L, 12J846- 00-1
Jean063	Dana Buchman	Hong Kong	RN# 52002
Jean064	DKNY Jeans	Turkey	RN# 52002
Jean065	Kut from the Kloth	China	RN# 58539
Jean066	Faded Glory	China	RN# 57116 AB3
Jean067	GAP	U.S.A.	VDN 3190, RN# 54023, 121379BZ22 8002, 121515-00-1 1603
Jean068	DKNY Jeans	U.S.A.	RN# 52002
Jean069	Red Camel	China	RN# 31104
Jean070	Banana Republic	U.S.A.	RN# 54023, VDN 0145
Jean071	Ann Taylor	China	ID#8054C, 127952
Jean072	GAP	Canada	RN# 54023, VDN 300673
Jean073	Wrangler Jeans Co.	China	WPL 6428, WKPH85154, 11MWZTN
Jean074	Banana Republic	India	RN# 54023
Jean075	GAP	U.S.A.	RN 540232, 200055-2 38553, XI410-AA 0145
Jean076	Old Navy	Bangladesh	RN# 54023
Jean077	Lee	Vietnam	RN# 130273, 3789730
Jean078	J. Crew	Guatemala	RN# 77386
Jean079	Style & Co. Jeans	Jordan	RN# 89828
Jean080	Lee	Bangladesh	RN# 130273, 3535735
Jean081	Kut from the Kloth	China	RN# 58539
Jean082	Lee	Egypt	RN# 13073, 3532930
Jean083	L.A. Blues	Mexico	RN# 72882, CS134008
Jean084	L.A. Blues	Hong Kong	RN# 72882, CS 11010014

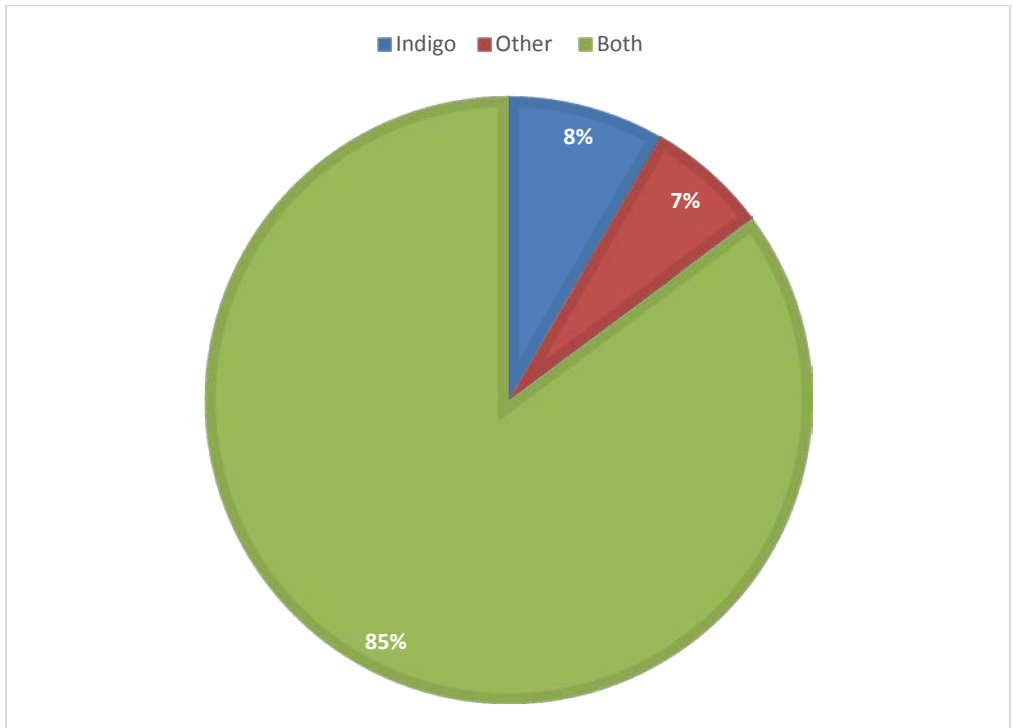
Jean085	Fashion Bug	Vietnam	RN# 72882, CSI3010262
Jean086	Joe's	Mexico	RN# 106214
Jean087	Lucky Brand	Mexico	RN# 80318
Jean088	J.Crew	Indonesia	RN# 77388, CA56445
Jean089	7 For All Mankind	U.S.A.	RN#115561
Jean090	Faded Glory	Mexico	RN# 58287
Jean091	Faded Glory	Bangladesh	RN# 52469
Jean092	Faded Glory	Mexico	RN# 58287
Jean093	Faded Glory	Mexico	RN# 58287
Jean094	Faded Glory	Bangladesh	RN# 52469
Jean095	Lane Bryant	China	RN# 118641, CS11070022
Jean096	Lane Bryant	Vietnam	RN# 118641, CS13011039
Jean097	Lane Bryant	China	RN# 118641, CS11071947
Jean098	Daisy Fuentes	Sri Lanka	RN# 105543
Jean099	New York & Company	Indonesia	RN# 23243
Jean100	Calvin Klein Jeans	Egypt	RN# 36009
Jean101	Apt 9	Cambodia	RN# 73277
Jean102	Old Navy	Bangladesh	RN# 54023
Jean103	Denizen from Levi's	Pakistan	WPL-423
Jean104	Daisy Fuentes	Sri Lanka	RN# 105543
Jean105	Seven 7	China	RN# 109890
Jean106	Old Navy	Cambodia	RN# 54023
Jean107	New York & Company	China	RN# 23243
Jean108	Bandolinoblu	Jordan	RN# 89828

**Table 2: Dyed Cotton Standards**

<b>Total Time submerged</b>	<b>First Submersion</b>	<b>Second Submersion</b>
5 minutes	5 minutes	n/a
10 minutes	10 minutes	n/a
15 minutes	15 minutes	n/a
20 minutes	10 minutes	10 minutes
25 minutes	15 minutes	10 minutes
30 minutes	5 minutes	25 minutes



**Figure 1: Composition of Samples**



**Figure 2: Dyes of Samples**