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Forensic Significance of Teal Colored Cashmere and Black Acrylic Fibers

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An Abstract of a Thesis
in Forensic Science

Submitted in Partial Fulfillment
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Abstract

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Textile fibers are a valuable type of trace evidence within forensic cases. They have the ability to connect a perpetrator to a victim and or a crime scene. Some types of fibers are more prevalent than others. The purpose of this research was to conduct a target fiber study in order to determine the significance of the selected fibers in a forensic case. Two fibers were selected from two different garments, black acrylic fiber and a teal colored cashmere fiber. Unknown fibers were collected from three local clothing stores and counted. Using light microscopy, all 20,164 fibers were eliminated as a potential match to the black acrylic target fiber. Microscopic comparison of the unknown fibers to the teal colored cashmere target fiber, produced two potential matches, which were further eliminated by microspectrophotometric analysis. Therefore, of the 20,164 unknown fibers, comparison to both target fibers resulted in no potential matches. It can be concluded that these target fibers may have potential forensic evidential value within a criminal case in the Erie County area.

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Table of Contents

List of Figures

List of Tables

I. Introduction

1.1 Forensic Disciplines in Question

The validity of all types of forensic evidence is currently being questioned. Analysis of fingerprints, firearms, bite marks and alike, are subjected to skepticism among the legal and forensic communities. This may be due to the lack of statistics, lack of reproducibility or the inability to confirm uniqueness of the evidence (Page et al, 2011). Unlike other types of evidence, DNA has a strong influence in the courtroom because analysts can put some weight behind their results with statistics that demonstrate how common or how rare a DNA profile may be. This in turn, helps the jury recognize the significance of the DNA evidence in trial. This is partly due to the empirical values that can be calculated with DNA profile results. These values provide a more effective meaning in interpretation, such as "one in one million people have this DNA profile" or "one in two billion have this DNA profile".

There are other types of evidence that cannot provide such statistical power or meaning like fingerprints or bite marks. Thus DNA evidence is deemed as the 'gold standard' and is used as a baseline for how discriminatory other forensic evidence should be (Lynch, 2013). This often results in the opinion that the other types of forensic evidence should have little weight on the outcome of a trial.

Analysis of fibers is one such case where the significance of particular fibers must be determined. The investigator or analyst must not only be able to determine if fibers found at a crime scene will tell the tale of the committed crime, but also how common or rare these fibers are within a population, much like a DNA profile. It is equally important that

the forensic fiber examiner knows the relevance of the fiber evidence while testifying in court.

1.2 Locard's Exchange Principle

In 1928, Edmond Locard, a French scientist, developed an idea that would revolutionize forensic science. This is known as Locard's *Exchange Principle* which states that when two objects come into contact with one another, they each leave their remnants on the other object by way of transfer (Gaudette, 1988). The results of the exchange of material between objects can leave either macro or microscopic substances left on both substrates from the other (Woods, 2006).

This *Exchange Principle* is the fundamental idea behind trace evidence. Such evidence has the potential, if analyzed correctly, to establish a link between a suspect and a crime (Woods, 2006). This type of evidence can also give facts of the victims and or suspect's whereabouts, collaborate or disprove eyewitness testimony, tell the story of what happened at a crime scene in addition to other unknown information concerning the case. Locard showed that every perpetrator has the potential to leave behind a piece of himself or herself and therefore, evidence.

1.3 Importance of Trace Evidence

All forensic evidence tells a story. Trace evidence is no exception. It is defined as "evidence that is transferred in small amounts when there is contact between individuals and/ or objects" (Deadman, 2004, 127). This type of evidence usually requires microscopic examination as a means of analysis because it is found in small amounts. Types of trace evidence may include fragments of glass, soil, paint chips and fibers.

These minute pieces of the puzzle can often yield important details and give insight to what had occurred during a crime or who had been at the scene of the crime. Linking a perpetrator to a crime is one of the most valuable pieces of intelligence that trace evidence can provide during a criminal investigation.

There are multiple scenarios one can determine in regards to a crime with the use of trace evidence. It can provide one or several links between the victim and the perpetrator, the victim and the crime scene, or the perpetrator and the crime scene, person to vehicle (intermediate point) or person to weapon. Just as trace evidence can provide these links, it can also indicate an absence of these links allowing for elimination of suspects or theories about the circumstances behind the crime.

As previously mentioned, there is a bias within the legal community that causes DNA to be put on a pedestal among other pieces of evidence (Lynch, 2013). Trace evidence is valuable whether or not biological evidence such as; blood and DNA are present at the crime scene (Grieve and Wiggins, 2001). Trace evidence can be especially useful to detectives who may not have the opportunity to collect a suspect's biological samples for comparison to the samples collected at the crime scene.

Trace evidence may also solidify or discredit the biological evidence, or any other type of evidence such as an eye witness testimony (Deadman, 2004). Forensic methods are prone to false positives or negatives to include DNA testing (Woods, 2006). Therefore, all evidence must be used as a type of internal control with respect to other evidence. For example, location of a bullet may not tell the full story of what occurred during a crime. If there is presence of fracture patterns in the glass of a nearby window, the fractures may help determine the path of trajectory of the bullet. The bullet found on

the outside of a building near a window with a bullet hole fracture pattern, may indicate that the gun was fired from inside the building. This example shows how the fractures in the glass work with the bullet evidence, providing a greater picture for investigators.

1.4 Importance of Fibers in Forensic Science

Fibers are of the trace evidence variety. Everyone wears clothing. Therefore, fibers are a common type of trace evidence and can be as significant in a case as DNA evidence (Grieve and Wiggins, 2001). Fibers, like all physical evidence, can serve as a link between the victim and the perpetrator, the victim and a potential crime scene, or the perpetrator to the crime scene (Gaudette, 1988, Houck, 2004, Woods, 2006). Types of cases where fibers are relevant include, but are not limited to: murder, sexual assault, breaking and entering, hit and run, and armed robbery (Gaudette, 1988). One of the key elements to linking victims, perpetrators and crime scenes together is the determination of the significance of these fibers.

The term "significance" with respect to fibers more or less describes how important a type of fiber is to an investigation and whether the fiber holds any evidential value. Evidential value tells the legal entities (jury, judge, attorney) if the fiber in question is common, meaning if it could have derived from thousands of sources or fewer. For example, it has been assessed that black cotton fibers and denim blue cotton fibers are shown to have little evidentiary value because these fiber types are common (Grieve et al, 1988). If it is found that the same fibers are recovered from a suspect and victim, it is crucial to know how common these fibers are within a geographic location. If the fiber type is found to be uncommon within the area, the suspect cannot be excluded from the investigation. However, if the fiber type is found to be exceptionally common, such as

blue cotton fibers from blue jeans, then the value of the fibers is very little from an evidence standpoint. Knowing the value of a type of fiber may save police and forensic examiners precious time and effort when investigating a case.

If the questioned fiber could have derived from few sources, the fiber evidence would be more significant, or pertinent to the case. A different consideration may be that fibers are important in forensic investigations because they can showcase a person's individuality based on wardrobe choices (Grieve and Wiggins, 2001). Every person has a certain style and their fashion choices reflect that style and their personality. The combination of fiber types within a person's wardrobe may provide some evidential value if the same combination of fibers is found to tie persons involved together or to the crime scene.

It is also possible that fibers from an individual's environment may transfer as well (Grieve and Wiggins, 2001). Carpet fibers may transfer to a crime scene from the perpetrator's shoe or clothing. This carpet fiber could tell investigators about where the person has been or possibly what type of carpet the person has in their residence. Finding a white carpet fiber, could be an indication that the perpetrator was in an environment that has white carpet.

Significance of fibers can also be looked at from the quantitative perspective (Gaudette, 1988). Target fiber studies and population fiber studies are a means of trying to quantify fiber evidence (Houck, 2003). It is difficult to generate statistical data from fiber evidence due to the lack of frequency data, or how often a certain fiber type is used within a fiber population. The lack of frequency data derives from the absence of record

keeping by the manufacturers on details such as how often a type of fiber is used for textiles or other products (Houck and Siegel, 2006).

Statistical analysis demonstrates the significance of the evidence, by putting weight behind it (Aitken & Taroni, 2005). As the frequency of certain evidence increases, the relevance of the evidence decreases. For example, as the frequency of a fiber type within a population increases, it's relevance to the case decreases. Blue cotton fibers are a prime example of this in the United States due to the popularity of blue jeans.

There are two schools of thought regarding statistical interpretation of fiber evidence; the classic approach or the Bayesian approach (Grieve, 1999). The classical approach involves calculating the statistical probability, which is based on fiber frequency. The results are presented with statistical odds. Issues with this approach involve the lack of useful data that should be included such as: figures on fiber production, sales/ distribution figures for textiles produced. In order to ensure that the statistical results are accurate, this information should be obtained. Inaccurate results would cause incorrect conclusions about the fiber evidence. For example, stating that one fiber is not as common as others, placing the evidence at higher value than necessary. This inaccurate information could lead investigators and legal professionals down the wrong path of theories regarding the crime in question.

The Bayesian approach expresses a ratio of the likelihood of two opposing probabilities: the suspect committed the crime or the suspect did not commit the crime (Grieve, 1999). Issues with this approach include the use of various estimations and subjective applications of data that is objective. Scientists should include all possibilities that could represent what occurred based on evidence, then from there the scientist may

determine if the hypothesis of the defense or of the prosecution is correct or incorrect (Aitken & Taroni, 2005).

Association errors can be made if Q, the questioned fiber, is similar to K, the known fiber (Gaudette, 1988). However, the error that may stem from this observation is that Q and K are a match, but the fact that materials are mass produced; it may be inaccurate to make such a conclusion. Type I errors may occur when the analysis differs from the actual state, or is the incorrect conclusion of making an exclusion of evidence. Type II errors are more severe and can have a negative impact on the investigation. For example, saying that Q originated from K, when in fact it did not, would be a Type II error. These are of more consequence than type I errors because it is worse to say that the questioned sample is a match when it is not than to say the questioned sample is not a match to the known sample when it is. Therefore, it is better to incorrectly exclude than to incorrectly include a questioned sample and better yet still to make no errors at all.

1.5 Types of Fiber Studies

1.5.1 Target Fiber Studies

The purpose of a target fiber study is to "determine the chance of encountering a particular chosen fibre type in a random population of fibres" (Grieve, 1999, 347). In this way target fiber studies relate to fiber frequency and significance. There are multiple types of requirements that should be met before selecting a target fiber (Grieve and Wiggins, 2001). The requirements are as follows:

- Man-made fibers- have higher evidential value than natural
- Those which have the strongest colors, making them easily visible and thus saving searching time. Stronger colors also lend themselves better to color analysis using MSP and offer possibility of using TLC

- Those which shed best and which are likely to pervade a particular environment, thus offering the possibility of establishing not only a primary, but also secondary transfer
- Those which may be found in the most significant locations (e.g., under clothes in rape case)
- Those displaying a color, polymer type, or a morphological feature that indicates reduced frequency in the general fiber population (Grieve and Wiggins, 2001. p. 837).

1.5.2 Persistence Studies

Persistence of fibers impacts the accuracy of the interpretation of the fiber evidence. The longer the investigators take in locating and identifying the fiber evidence, the more the potential of loss of fiber evidence increases (Gaudette, 1988). Fibers are a type of evidence that are easily lost or transferred (Gaudette, 1988 & Palmer, 2000). Persistence of fibers involves many factors that may influence the fiber persistence, however, this is also an area of fiber evidence that the forensic community researches as a separate topic from significance of fibers. This does play a role in the statistical analysis of target fiber studies as the loss of unknown fibers could include the loss of potential target fibers and therefore, affecting the accuracy of conclusions drawn during a target fiber study. In a controlled target fiber study, it may be still be possible for fibers on a tape lift to fall off of the tape if it did not adhere very well to the adhesive in the first place.

1.5.3 Transfer Studies

Following the Exchange Principle, fibers from one garment or fiber source transfer to the object it comes into contact with. Gaudette states there are multiple factors that affect transferability of fibers; 1) amount of contact pressure between the two garments, objects or people 2) texture of the garments, 3) fiber length, 4) fiber type of the garment, 5) areas of contact and 6) number of times of contact (1988). Gaudette summarizes sources of

how fibers may be transferred between objects and people in addition to what type of case this information may be relevant (1988). These sources of fiber transfer may include from clothing to a vehicle, clothing to a crime scene, clothing to hair and/or body and also carpet to footwear or carpeting to hair and body. There are multiple possibilities, demonstrating the ways a fiber type may be transferred.

There are two types of fiber transfer; primary and secondary transfer. Primary transfer is the first transfer between two sources of fibers or hair, or the source of fibers or hair and an object. Secondary transfer of fibers occurs after the primary transfer has taken place and the fibers that were transferred initially are then transferred again to another object or person. Palmer warns against secondary transfers of fiber evidence as a source of contamination (2000). An example of secondary transfer of fibers could be a police officer arresting a suspect, then that officer also collects an article of clothing from the crime scene. It is possible that fibers from the suspect were transferred to the police officer's uniform (primary transfer) and then were shed when the officer collected the garment from the crime scene (secondary transfer). If fibers from the suspect are found on the garment, it cannot be concluded that these fibers were result of a primary transfer, i.e. when the crime was committed. Therefore, the fibers found to match the fibers collected from the suspect will be of no evidential value.

1.6 Limitations of Fibers as Evidence

The means in which fiber evidence is collected is adjusted to the needs of the particular circumstances surrounding the crime (Palmer, 2000). Collection of fibers may include tape lifts, alternate light sources, vacuuming, scraping, combing, or collection of a single fiber by hand (Gaudette, 1988, Palmer, 2000). The need for variability of

recovering/collecting fibers may cause this type of evidence to be perceived as a subjective field (Grieve and Wiggins, 2001). An example of this perception may be the inconsistent collection or recovery of fibers. This demonstrates a lack of uniformity within fiber evidence collection, even though it is a necessity for collection methods to adapt to the needs of the case.

II. Background

2.1. Polymers

Polymers are the chemical building blocks for all synthetic fibers (Saferstein, 2007). Polymers also serve as the fundamental chemical substance for paints, plastics, synthetic rubber and adhesives. Polymers themselves are made by linking a significant number of molecules and typically resemble a long, repetitive chain of either the same or different monomers. The variation in monomers within a polymer can account for the multiple forms and differences in physical and chemical properties among polymers. More variation of polymer types may allow for the analyst to depict a more specific classification of the fiber. These variations of fibers are also based on how the monomers are weaved together and the chemical structure of the monomers within the fiber (Saferstein, 2007). Important requirements for fiber-forming chemical characteristics are as follows:

- Linear molecular chains which possess some degree of extension or orientation to the fiber axis, thereby giving a structure which is stronger longitudinally than transversely (p.2)
- A high molecular weight imparting both a high melting point and low solubility in most solvents (p.3)
- Streamlined molecular chains allowing for close packing of the polymer chains (p. 3)
- The molecular chains should be flexible and hence impart extensibility to the fibres (David and Pailthorpe, 1999, 3).

2.2 Fiber Dyes

The color of a fiber could be multiple dyes mixed together to produce a specific color, or it may be only one type of dye (Wiggins, 1999). Just like fibers, dyes also may be classified. The manner in which the dye is utilized, its chemical properties, or the specific

fiber type it is used on, are means of classifying dyes (Wiggins, 1999). Dyes are typically organic, and normally have an auxochromic group in their chemical composition. These auxochromic groups include $–N(CH_3)$, $-NH_2$, - COOH and SO_3H, which affect the color intensity of the dye. There are at least eleven classifications of dyes, some of which include acid dyes, basic dyes, azoic dyes, metallized dyes and reactive dyes. ***Table 1*** shows some different classifications of dyes and the fiber type likely to be associated with those dye classifications.

Table 1. Textile fiber type and corresponding class of dye (Wiggins, 1999).

Dye class	Fibre type
Acid	Wool, silk, polyamide, protein, polyacrylonitrile, polypropylene
Basic	Polyacrylonitrile, modified acrylic, polyester, polyamide
Direct	Cotton, viscose
Disperse	Polyester, polyacrylonitrile, polyamide, polypropylene, acetate/triacetate
Reactive	Cotton, wool, polyamide
Sulphur	Cotton
Vat	Cotton
Metallized	Wool, polypropylene
Azoic	Cotton, viscose
Ingrain	Cotton

2.3 Fiber Classifications

Fibers are typically considered class type evidence (Houck and Siegel, 2006). Class evidence is evidence that can only be identified as belonging to a specific class of fibers, such as blue cotton or white cotton fiber. Fibers may be classified as individual evidence if they bear a specific physical characteristic such as a tear or stain that can help differentiate between that fiber and others that are alike. A tear or stain on a fiber may also indicate the source of the fiber, such as a sweater that has the same colored stain as

the individual fiber. Closer analysis with a microscope may determine if that garment was the source of the fiber in question.

There are two main classes of fibers that are shown in ***Figure 1***: natural and synthetic. Fibers that are used in a natural state, original chemical and physical characteristics remain with exception of the color of the fiber via dyeing, are known as natural fibers (James and Nordby, 2009). Natural fibers include sources from animals, vegetables and minerals (Gaudette, 1988). Examples of animal or protein fibers include wool, mink, cashmere (goats), hair and silk. Examples of vegetable (cellulose) fibers include: cotton, hemp and sisal. Cotton can be identified by its ribbon-like shape with twists at different locations and may have significance if dyed or has a specific combination of dyes (Saferstein, 2007). Sub-classifications of vegetable fibers are shown in ***Figure 1*** and include fibers that originate from different parts of a plant such as a seed fiber or leaf fiber. Asbestos is an example of a fiber from mineral origin (James and Nordby, 2009 and David and Pailthrope, 1999).

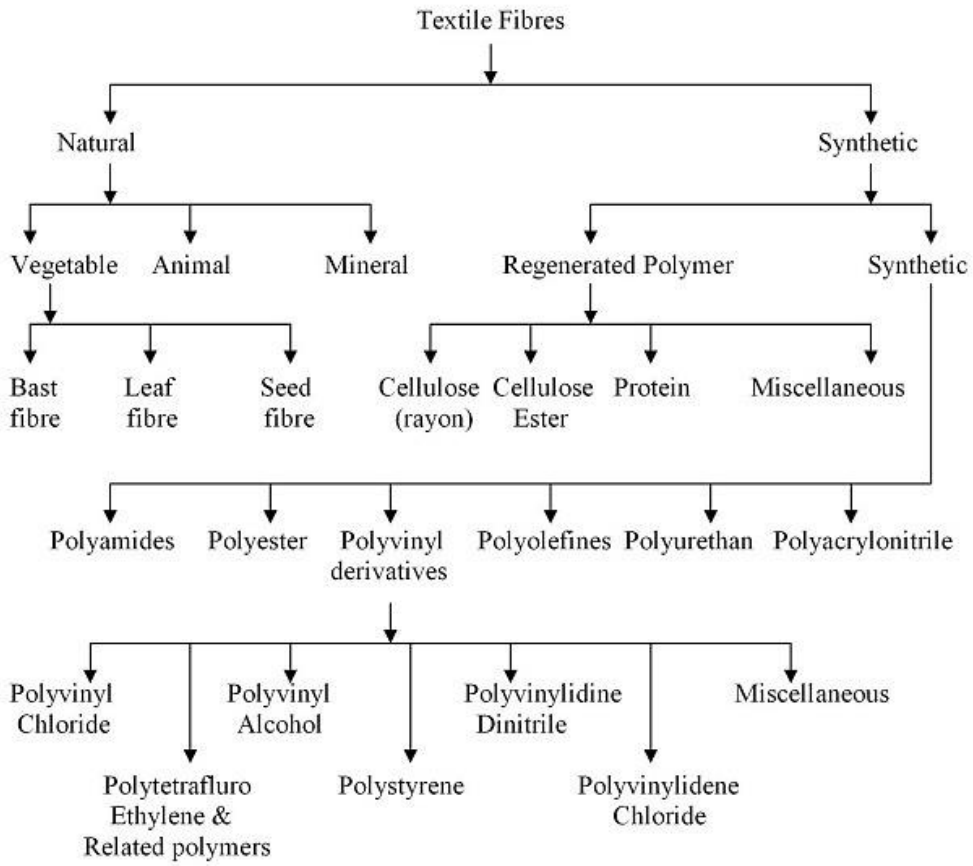

Figure 1. Fiber classification (Roux and Robertson, 2000).

Synthetic fibers are a second main classification of fibers. This classification includes acrylic, nylon, spandex, fluorocarbon (Teflon), and rayon to name a few (Gaudette, 1988). These types of fibers may be made from man-made chemicals alone or in combination with polymers natural, as well as glass (James and Nordby, 2009). Synthetic fibers can be further divided by if the fiber is a regenerated polymer or synthetic. Regenerated polymers are then broken down into even further sub-classifications such as a protein fiber (fiber derived from a protein source), or cellulose rayon fiber (Lyocell). Synthetic fibers may be further classified into several categories separate from

regenerated polymers. Polyamindes, Polyester, Polyacrylonitrile (acrylics) and Polyvinyl derivatives are a few examples of synthetic fiber classifications.

2.4 Characteristics of Selected Target Fibers

2.4.1 Black Acrylic Fiber

Acrylics, or Polyacrylonitriles, are classified as synthetic fibers (***Figure 1***) which are composed of at least 85% by weight of acrylonitrile units (Gaudette, 1988, Bide, 2005 and Causin et al., 2005). The other 15% of the fiber is composed of monomers that aide in dye absorption. The molecular formula of acrylonitrile is C_3H_3N. Its units repeat multiple of times as seen in ***Figure 2*** to produce polyacrylonitrile. ***Figure 3*** shows the multiple types of acrylic fibers that may be produced from acrylonitrile. These include: 1) acrylonitrile (PAN), 2) acrylonitrile/ methyl acetate (PAN/MA), 3) acrylonitrile/ methyl acrylate/ methyl vinyl (PAN/MA/MV), 4) Acrylonitrile/ vinyl acetate (PAN/VA), 5) acrylonitrile/ vinyl acetate/ methyl vinyl \ pyridine (PAN/VA/MVP), 6) acrylonitrile/ methyl methacrylate (PAN/ MMA), 7) acrylonitrile / any other monomer(s). These variations of chemical composition exhibit the variety that exists within the acrylic fiber class and therefore sub-classifications of the fiber are present (Grieve, 1995 and Bide, 2005).

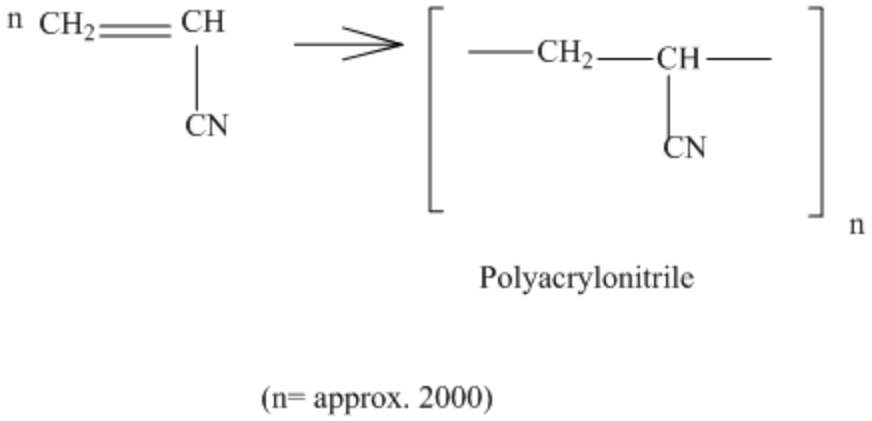

Figure 2. Acrylic fiber structure (Roux and Robertson, 2000).

The sub-classifications of acrylic fibers are based on the type of co-monomers and solvents used during the manufacturing process (Grieve, 1995). As explained by Grieve (1995), co-polymers are used to allow for the acrylic fiber to accept ionic dyes because of the difficulty of dyeing the polymer. Examples of co-monomers include vinyl acetate (VA), methyl methacrylate (MMA) and methyl acetate. The solvent used to reabsorb the polymer in powder form is another chemical signature that may be used in chemical analysis of the polymer (Grieve, 1995).

There are two types of processes to manufacture an acrylic fiber: suspension polymerization and solution polymerization (Grieve, 1995).

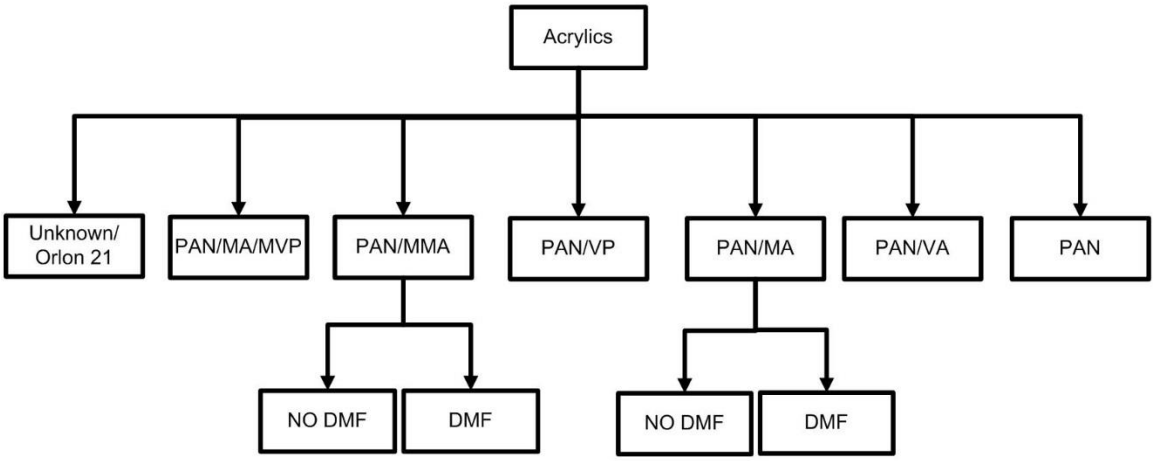

Figure 3. Acrylic sub-classifications by co-monomers and solvent used in manufacturing process (Grieve, 1995).

Suspension polymerization can be described in a few steps. First, the polymer is dissolved in water via a redox initiator (sodium bisulphite or potassium persulphate). Then polymer is dried into a powder and is redissolved into a solvent. The type of solvent used is dependent upon if the fiber will be wet spun or dry spun. Dry spun polymers will be redissolved in dimethylformamide (DMF) if the co-polymer used is MA. If the polymer is to be wet spun and vinyl acetate is used as the co-monomer, then dimethylacetamide (DMA) solvent is used. A summary of these processes can be seen in ***Figure 4***. The type of chemical composition used to manufacture the acrylic fiber also influences the type of dye used to color the fiber.

Solution polymerization can be described as the use of multiple types of solvents (Grieve, 1995). These types of solvents are listed in ***Figure 4***, which may include; DMF, Dimethyl sulfoxide (DMSO) and zinc chloride. The solvent/ polymer mix is then filtered and ready to use in a wet spinning process.

The cross-sectional shape of acrylic fibers may vary (Bide, 2005). Shapes include a dog- bone, peanut, bean or even round. It can also be noted that acrylic fibers are resistant to UV light and environmental factors as well as some inorganic acids (Bide, 2005).

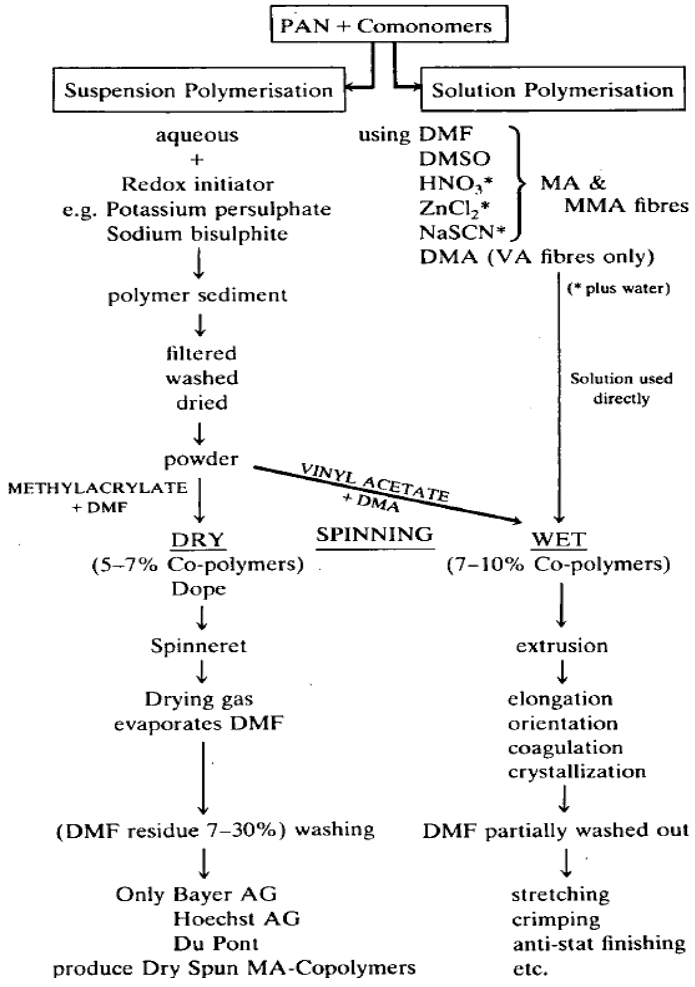

Figure 4. Summary of the manufacturing process of acrylic fibers (Grieve, 1995).

2.4.2 Teal Colored Cashmere Fiber

Cashmere is classified as a natural fiber and may be further classified as a specialty hair fiber, as it derives from the goat family (David and Palenik, 1999). Specifically, cashmere is from the Asiatic goat *Capra hircus laniger.* This species is typically a feral

goat that originates in New Zealand, Scotland and Australia (David and Palenik, 1999). As with all hair fibers its structure is composed of the protein keratin. Keratin is a linear polymer that is high in sulfur and contains many disulfide bonds (Roux and Robertson, 2000).

Besides color, when identifying an animal hair fiber, the scales of the hair are the most important morphological feature of a hair fiber (Palenik, 1999). In ***Figure 5*** a diagram of hair structure displays one type of cell within each of the three layers. The three cell types are the cuticle cells, the cortical cells and the medulla cells (Roux and Robertson, 2000). The cuticle cells are most outer layer of the hair that comprises of overlapping scales. The scale patterns vary depending on the species that the hair originates from. The cortical cells that make up the middle layer of the hair contain pigment granules, which give the hair its overall pigment. The center layer of cells is known as medulla cells. These cells also display a specific pattern pertaining to the origin of species.

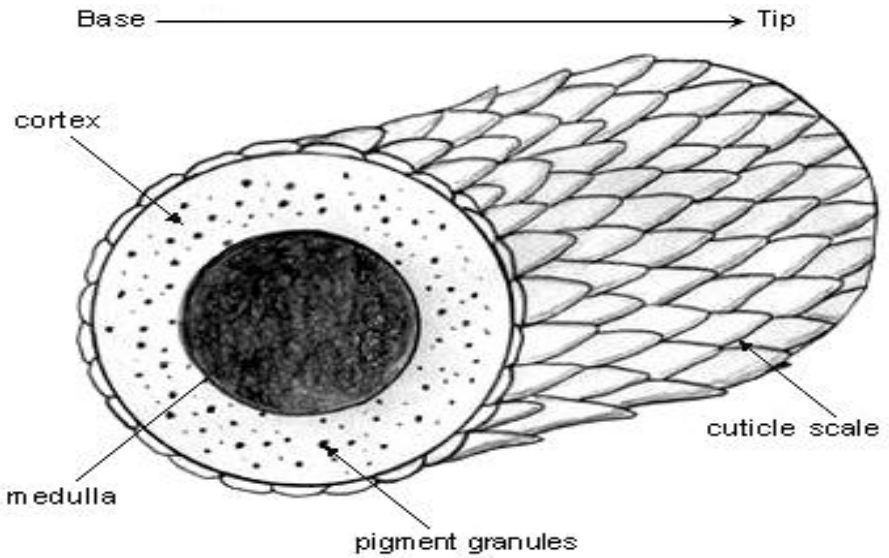

Figure 5. Diagram of hair structure (http://www.ecobyte.com.au/using_.html).

Typically, cashmere fibers are 14-16 µm in diameter and 5-10 cm in length. (David and Palenik, 1999) The scales on a cashmere hair differ from the scale pattern of fine wool as shown in ***Figure 6***. In the picture it can be observed that the fine wool hair has similar size in diameter as cashmere, but has a higher scale count. This is an instance where close examination of the hair scales may help differentiate between the two hair types.

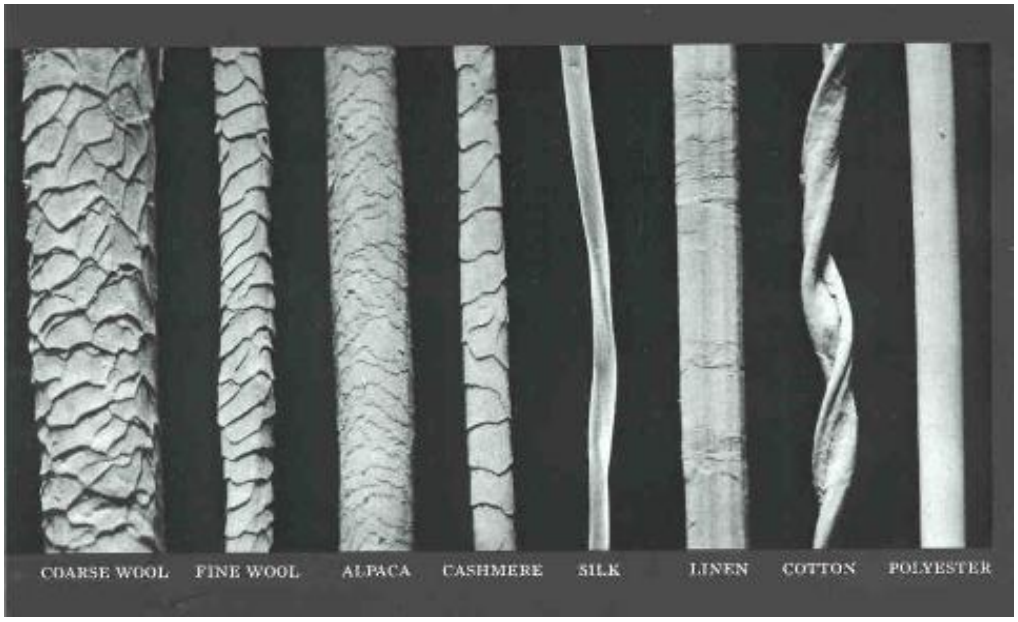

Figure 6. Picture of various fibers under scanning electron microscope. (http://www.rpd611.com/HSL/HSL.html)

Cashmere fibers have the same chemical composition to wool. With the exception of finer wools, cashmere tends to be more vulnerable to chemical damage due to its wetting properties and finer texture (David and Palenik, 1999).

2.5 Fiber Comparison Techniques

Before beginning analysis of an unknown fiber, analysis must be first conducted on the known, or target fiber (Gaudette, 1988). A known fiber originates from a known

source. An unknown or questioned fiber originates from a source that is not known. This could include a type of hair found on a victim that does not belong to the victim or other occupants of their residence. The results of the examination of the unknown fiber may be then compared to findings of the known fiber. The purpose of the comparison is to look for differences between the fibers. In general, the order of fiber analysis is as follows: stereomicroscopic examination, polarized light microscopic examination, microspectrophotometric analysis and lastly, Thin- liquid Chromatography analysis if there is enough of the fiber sample to perform the test (James and Nordby, 2009). Infrared analysis may also be included in the fiber analysis process (Grieve, 1995 & Kirkbride and Tungol, 1999).

As differences between the known and questioned fiber are noted, the unknown may be eliminated. If there are no differences noted, that fiber may proceed to the next step of analysis. Since fibers are class type evidence, there can be no definitive "match" without some indication of uniqueness. This may be a blood stain or debris affiliated with the unknown fiber. The blood or debris then may help trace the source of the fiber because the source will also have a blood stain or the same debris as the questioned fiber.

2.5.1 Light Microscopy

Basic characteristics such as color and length may be used. Further examination with the use of a microscope would be required to determine fiber type, cross section, presence of delustrants, optical properties and alike. These are among the first observations made by a fiber examiner. While microscopic methods help identify physical and optical properties, instrumental methods can help identify a more specific fiber type (Woods, 2006) based on dye or polymer analysis. Acrylic fibers, for example, are numerous in

types and may possess the same optical properties under the microscope. Physical properties (color, diameter, length) can be determined as well as the optical properties such as birefringence, refractive index and sign of elongation. These are all useful in fiber comparison, which ultimately leads to the determination of a possible "match" or elimination of the unknown fiber collected at the crime scene to a known fiber collected from a suspect.

Stereomicroscopes operate under low-magnification (Palenik, 1999). Stereoscopic analysis can allow certain information to be ascertained such as the color, luster, cross-section, soil, trace particles on the fiber, and damage to the fiber. This step also is where an initial fiber classification can be made. Polarized Light Microscope (PLM) examination allows for greater magnification and finer details of the fibers to be assessed. Optical properties such as refractive index and birefringence can be determined. Using crossed polars to examine fibers can help determine if a fiber is anisotropic or isotropic (James and Nordby, 2009). Compensators are also a valuable tool when determining the retardation of anisotropic material. The PLM examination is also useful for determining cross-sectional shape of the fiber. The type of cross-sectional shape that a fiber possesses is determined by the functional use of a fiber (carpet or clothing) (James and Nordby, 2009).

Comparison microscopy allows for two samples to be examined side by side simultaneously. A comparison microscope consists of two microscopes connected by an optical bridge so that one or both samples may be viewed (Woods, 2006). When conducting an examination under the comparison microscope, both microscopes should

have the same background color so that the color of both fibers may be assessed equally (Gaudette, 1988).

2.5.2 FT-IR Microspectrometry

Fourier Transform Infrared Microspectroscopy aids in the identification of the polymer or polymers in natural or synthetic fibers (Kirkbride and Tungol, 1999). It can confirm the fiber class and may even lead to subclass information with fibers, such as nylon or acrylic. The sub-classification of a fiber may then in turn identify the manufacturer of that fiber. This method of analysis may also provide information to confirm or discredit observations made under PLM that are questionable. It can also be a faster means of identifying the fiber type instead of determining optical properties that may be difficult to distinguish or classify. However, this technique is typically not useful for natural fibers with similar structures, such as hemp and cotton or wool and cashmere (Kirkbride and Tungol, 1999).

The instrument setup is an FT-IR coupled with a microscope. The infrared source from the instrument passes through the fiber sample causing vibrations between the molecules within the fiber sample. In turn, the type of vibration, caused by the type of chemical bond between molecules, will produce a certain type of peak within an IR spectrum (Bruice, 2007). The focus of the microscope on the sample can influence the quality of spectra produced. The size of area measured on the sample is 100-150 micrometers in diameter (Kirkbride and Tungol, 1999). A spectral match is determined by the absence or presence of peaks displayed in the unknown sample compared to the presence or absence of peaks in the known sample (Woods, 2006).

There are some advantages and disadvantages to employing this technique for fiber analysis (Kirkbride and Tungol, 1999). In addition to the instruments ability to measure fibers that are less than 100 µm, the technique is non-destructive to the fiber. This is important in forensic investigations because this enables further testing to be done on the fiber if needed. FT-IR is also a quick method of analysis and the use of IR databases make polymer identification an easier process. Though sample preparation is minimal, there is some alteration of the fiber during sample preparation. In order to produce quality spectra, it has been determined that the fiber should be rolled flat. This in turn may cause the crystalline/ amorphous structure of the polymer to change (Kirkbride and Tungol, 1999). There is also the potential for the fiber to be rolled with uneven pressure, which may cause different spectral results between different locations on the fiber. This may lower the level of reproducibility of the results. FT-IR Microspectrometry may also be valuable for colorless fiber such as acrylics (Causin et. al., 2005). The co-monomer peak ratios may help differentiate one colorless acrylic fiber from another.

2.6 Fiber Dye Analysis Techniques

There are multiple methods for analyzing the dyes of a fiber. The preferred methods are a non-destructive methods such as Microspectrophotometry and recently Raman Microscopy. Other techniques that may be employed for dye analysis include High Performance Liquid Chromatography (HPLC), Surface Enhanced Resonance Raman Scattering Spectroscopy (SERRS), Capillary Electrophoresis (CE) and Thin layer chromatography (TLC). Thin layer chromatography may be used in conjunction with Microspectrophotometry if further analysis is needed. Microspectrophotometry and/or

TLC are the primary methods for forensic dye analysis and will be the only techniques discussed in more detail.

2.6.1 Microspectrophotometry

Microspectrophotometry (MSP) is used to measure the absorption of radiation from the UV-Vis range and its effects on the excitation of electrons in the chromophores of the sample (Adolf and Dunlop, 1999). It can be considered to have higher discriminating power when compared to FT-IR analysis (Kirkbride and Tungol, 1999). This is due to the lack of variation of polymer types and the thousands of dyes available to manufactures. This analysis is necessary when samples are a metameric pair. A metameric pair can be defined as "two colors that appear visually matched in one illuminate, but which have different spectral curves" (Gaudette, 1988, 245). This means that the two fibers appear to be the same color under a polarized microscope, but produce different UV- Vis absorption spectra. Since it is possible for fibers to be non-uniformly dyed, it is necessary to take multiple spectral measurements at different locations of a fiber sample. This is especially true of a natural fiber since they tend to not absorb dye non-homogeneously (Gaudette, 1988). If the concentration of the dye is too small to be detected, other methods such as micro- Raman spectrometry can be utilized (James and Nordby, 2009).

Once the MSP spectra have been measured on the known fiber and the unknown fiber, a comparison of the spectra may be made. It is the shapes of the spectra that may be used for comparison (Gaudette, 1988). Presence or absence of peaks should be noted on the unknown spectra and compared to the known spectra. It is also possible that only one spectrum from the unknown matches one spectrum from the known and may be

considered a possible color match. MSP cannot identify the type of dye, but can only provide information about the color of the fiber (Adolf and Dunlop, 1999).

2.6.2 Thin- Layer Chromatography

Thin- layer chromatography (TLC) is a separation technique involving analytes (dyes) traveling via capillary action up a coated glass plate, usually silica coated (Bell, 2006). TLC allows for the separation of a mixture of dyes on a thin layer of a stationary medium (Wiggins, 1999). The plate is the stationary phase, while the solvent is the mobile phase of the technique. Solvent is applied to the dyed fibers on the medium and the dye mixture travels with the solvent, until each type of dye is separated. Depending on the polarity of the solvent, one can determine whether the dye is polar or non-polar, based on the distance traveled by each analyte. Polar solvents will attract polar analytes, which would travel a greater distance than non-polar solvents that would not attach itself to the polar solvent. If enough of the questioned fiber sample is available, TLC may be utilized in conjunction with MSP analysis. The amount of fiber sample matters for this type of analysis because of the destructive nature of the technique (Bell, 2006).

2.6.3 Raman Microscopy

Like Infrared spectroscopy, Raman spectroscopy is a technique that measures the vibrations within molecules (Kirkbride and Tungol, 1999). Unlike IR Spectroscopy, Raman measures the scattered energy of polarizable bonds while IR measures the absorbed energy of dipole moments between bonds (Bell, 2006). This technique focuses on the scattering of radiation from a light source, such as a laser. The radiation hits the sample, causing electrons within the molecular structure of the sample to become excited

and give off radiation, which is scattered (Kirkbride and Tungol, 1999). The scattered radiation is then filtered before it is detected. Stokes scattering is what is typically recorded in the Raman spectra.

Raman Microscopy can be utilized if the concentration of a dye within a fiber is too low to detect with MSP analysis (James and Nordby, 2009). Issues that arise from using this technique for dye analysis, is the inability for Raman to detect multiple dyes as well as it may be inhibited by fluorescence properties of the dyes (Massonnet et al, 2012). Though background fluorescence is a current issue for some samples analyzed with Raman microscopy, this problem can be minimized by the use of a laser with longer wave length (Bell, 2006).

Another problem is the low intensity of Raman signals due to the scattering of the radiation. The low intensity of the spectra may also be caused by disintegration of the fiber sample by the power of the laser that is used to cause scattering energy (Bell, 2006). A positive aspect of using Raman microscopy is the sample size may be relatively small. The sample may also be measured while mounted on a glass slide, since this does not interfere with the instruments collection of spectra.

2.7 Research Objective

This research is a target fiber study that aims to determine the significance of two fiber types within a location. The sample fibers selected include a teal- colored cashmere fiber and a black acrylic fiber. Both fibers originated from garments that were labeled 100 percent of a particular fiber material. The location of this study is centered in North Buffalo on Delaware Avenue as three clothing stores within a one mile radius were used as sources of the unknown fibers. The target fibers were searched for among the

unknown fiber samples, while using analytical methods to differentiate the collected unknown fibers from the selected target fibers.

III. Materials and Methods

3.1 Collecting Unknown Fibers

Unknown fibers were collected via tape lift using a 3M Scotch- Brite pet hair roller from the floor of multiple dressing rooms of clothing stores within Erie County on Delaware Avenue. The types of stores were selected based on popularity and price range as to get a wider sample range for the unknown fibers. ***Table 2*** shows the clothing stores and the month and year the samples were collected. Typically the samples were collected later in the day so that more fibers would be present. Two tape lifts were taken from each store: one from a men's dressing room and one from a women's dressing room, with the exception of Kohl's, to provide an equal representation of the fiber population. Some samples were taken in January 2013, while others were taken February 2013.

The floor of each dressing room was rolled under the hanging hooks on the wall. This was determined to be the best area to collect unknowns since clothes hang from the hooks and fibers may be shaken loose from the garments while hanging. Each time fibers were collected with the lint roller a 1 yard ^{2} area was rolled for fibers. The sheet from the lint roller was then placed flat in a plastic bag and sealed with tape. The date, time, location, dressing room type and name initials were placed over the tape where the bags were sealed.

Table 2. Sources of Unknown Fibers

Store	Location	Month &Year Collected
Good Will	Delaware Ave	Feb 2013
Kohl's	Delaware Ave	Jan 2013
Marshalls	Delaware Ave	Feb 2013

3.2 Selecting the Target Fibers

Two types of target fibers were extracted from two different personally owned garments. The teal colored cashmere fiber was extracted from teal sweater that was 100% cashmere shown in ***Figure 7*** at 10x magnification. The black acrylic fiber was extracted from a scarf that was 100% acrylic show in ***Figure 8*** at 10x magnification. All garments were 100% of the fiber type so that no fiber blends were utilized as target fibers. Common fiber types and colors, such as blue and white cotton were avoided when selecting the target fibers, as they were shown to hold no significant evidential value.

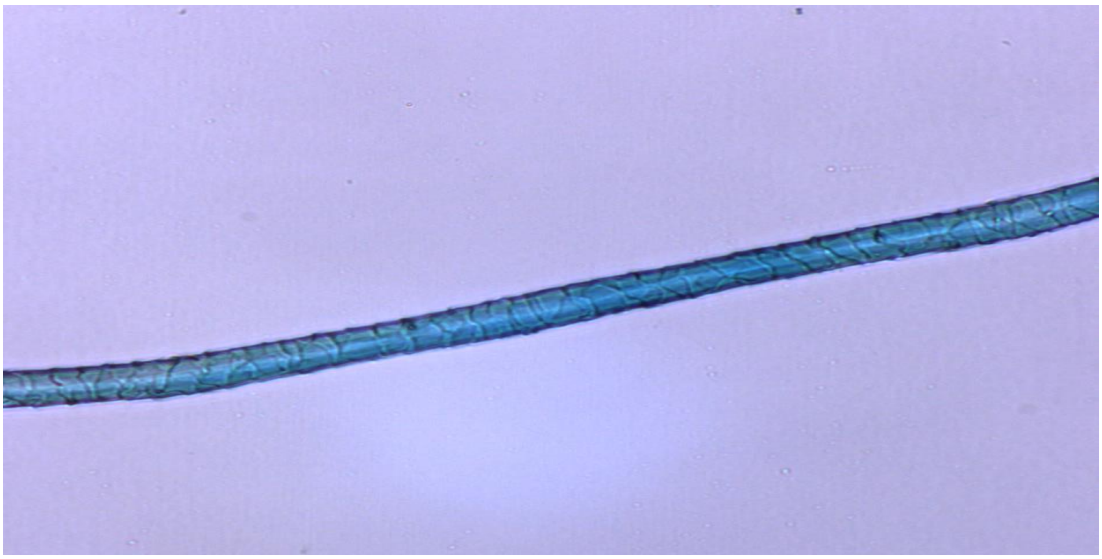

Figure 7. Image of known teal colored cashmere fiber under MSP analysis (10x magnification).

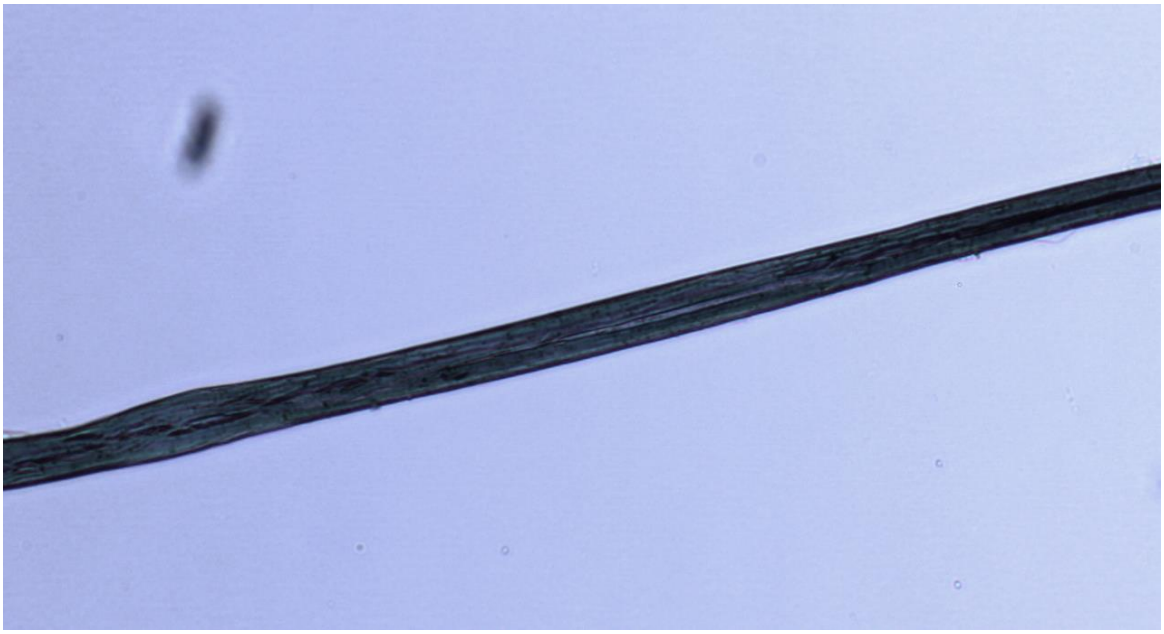

Figure 8. Image of known black acrylic fiber under MSP analysis (10x magnification).

3.3 Counting the Unknown Fibers

The purpose of counting the questioned or unknown fibers was to be able to generate statistics on the frequency of the known fibers within a location. The known fibers were examined under a stereomicroscope and also a polarized microscope to ascertain physical

properties for the purpose of familiarization with fiber types/ dyes. Next, each tape lift of unknowns was taped to a 10.16 cm by 38 cm glass plate that consisted of 38, 1 cm wide lanes. Using a polarized light microscope, fibers in every 5^{th} lane, starting in lane 2 from the top of the lane were counted. To keep track of the number of fibers counted per lane, the number of fibers counted was periodically recorded and added to the previous count number. If there was a question as to if a specimen was a fiber, it was not counted. Human hair and non- human hair were also not counted unless the hair seemed to be dyed an unusual color such as green or purple, indicating a textile fiber. This precaution ensured that the hair was not from a domestic animal, such as a dog or human hair was not included in the unknowns. There were areas of the tape lift that were heavily concentrated with fibers, which were too difficult to differentiate and count. Fibers that stood out by color were counted since they were easy to separate from the rest of the tangled fibers.

As shown in ***Table 3***, the tape lifts were then labeled with a letter. This allowed the source of the unknowns to be maintained. The unknowns were then given a number after the letter in the order they were collected. For example, sample A1 is an unknown fiber taken from tape lift A and is the first sample located on the tape lift.

Table 3: Sources of tape lifts and assigned tape lift labels

Tape Lift Source	Tape Lift Sample Letter
Kohl's Misses Dressing Room #3	A
Marshall's Female Dressing Room	B
Marshall's Male Dressing Room#2	C
Goodwill Unisex Dressing Room #2	D
Goodwill Unisex Dressing Room #1	E
Kohl's Misses Dressing Room#5	F

3.4 Extracting Possible Matches from Tape Lifts

The location of the unknown fibers that were potential matches to the known fibers was marked during the counting process. First the front of the glass was marked with a marker, and then this reference point was used to circle the back of the tape lift. The circle was made to mark the general area of where the potential match would be within the lane. Then, using a stereomicroscope, the area circled on the tape lift was examined to reestablish the location of the unknown fiber of interest. If the appearance of the fiber looked similar to a target fiber, the fiber was removed with tweezers. A drop of methanol was used to disintegrate the adhesive for easier removal of the fiber. Sometimes the fiber was removed under a PLM to provide confirmation of extracting the correct fiber type as dark or black fibers looked similar under the stereoscope. Once the fiber was removed from the tape lift, it was mounted on a glass slide with glycerol as a medium. Glycerol was the mounting media of choice because of its low fluorescent properties that would otherwise interfere with the microspectrophotometric analysis, as well as its non-hardening properties. The ability to remove the fiber from the slide without requiring a

solvent for its removal was also beneficial because fibers were readily available to be removed for further testing. Each slide was labeled with appropriate letter and number before the fiber was mounted under a stereomicroscope.

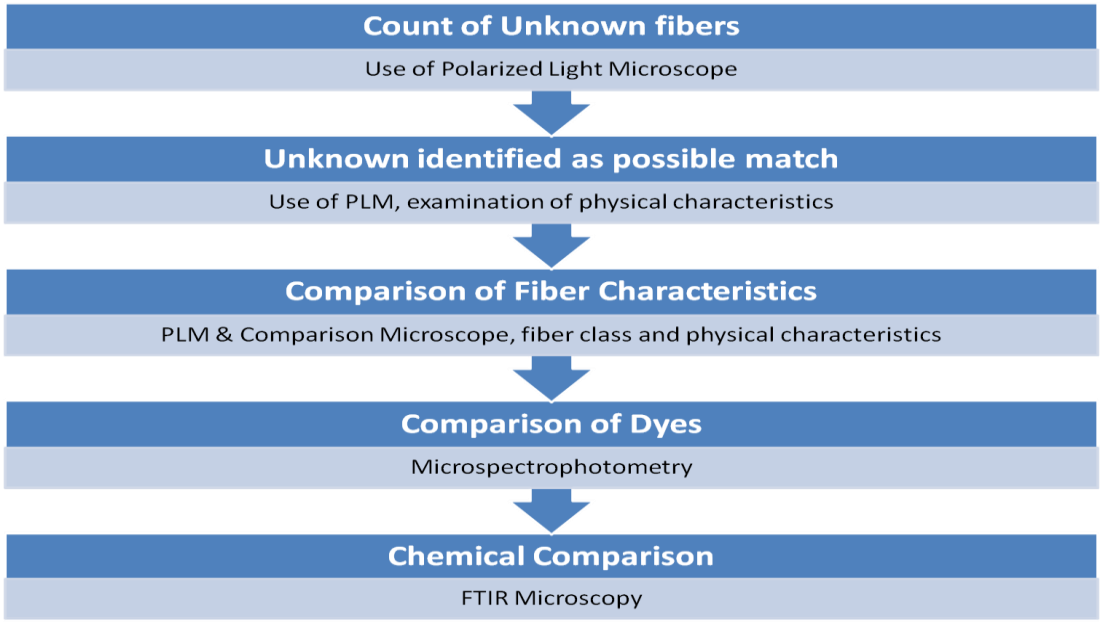

Figure 9. Flow chart showing overall fiber comparison process and techniques used.

3.5 Microscopic Comparison of Fibers

Using the PLM at 10 x magnification and then 40 x magnification, unknown fibers were eliminated based on color, cross-sectional shape and diameter. When looking for potential black acrylic fiber "matches", the presence or absence of delustrants was used to further eliminate or include potential matches. The presence or absence of scales and scale type was used to further determine the possibility of the fibers match to the known cashmere fiber. After the preliminary determination of potential matches, a comparison microscope was used to compare the unknown fibers to the known fibers.

3.6 Measuring Dye Spectra with Microspectrophotometry

Microspectrophotometry (MSP) was used to measure dye spectra of each fiber sample. As previously mentioned, sample preparation for measuring the fiber dye spectra using MSP was mounting the fiber samples on a glass slide in glycerol, placing a glass cover slip over the fiber. Glycerol was used as a mounting media due to its low fluorescence properties, minimizing interference with the microspectrophotometer. A CRAIC 308 PV^{TM} Microscope Spectrophotometer equipped with a Leica DM 2500 Polarizing Light Microscope was used to measure the dye in transmission mode within the visible light spectrum (400 to 800 nm). The optimal integration time was adjusted before each sample to the range of 200-300 milliseconds in accordance with the standard operating procedure. The measurement process was as follows: collection of "Dark Scan", followed by the collection of a "Reference Scan" of the mounting media, then collection of sample spectra. Each sample was measured at a minimum of four different locations along the length of the fiber.

3.7 Measuring FT-IR Spectra with FT-IR Microspectroscopy

Collecting the FT-IR spectra of the known and unknown sample fibers was the last step in the comparison process (***Figure 9***). Sample preparation for the FT-IR analysis required fibers to be removed from the slides and to be washed with water to remove any glycerol from the fiber. Fibers were washed with deionized water, and dried via suction vacuum by use of a glass Gooch crucible with sinter fritted filters attached to a volumetric flask with a side arm. The side arm was connected to a hose that was connected to a built in vacuum line to the laboratory. Fibers were placed back onto

separate, clean, labeled glass slides and held in place with double sided tape. Fibers were then flattened using a metal roller.

A Nexus 470 FT-IR spectrometer (Thermo Nicolet) with a MCT/A detector and equipped with an IR Microscope (Nicolet). The spectral range was from 4000 to 650 cm^{-1}, with a resolution of $4cm^{-1}$ with use of 64 scans viewed in transmission mode. Fiber samples were rolled, size permitting, with a small metal roller and mounted to a KBr salt plate via double sided tape. All of the samples were oriented perpendicular to the microscope. Before samples were measured, a standard polystyrene film was measured twice for quality control of the instrument as shown in ***Figure 10*** and was searched against the IR database to confirm a polystyrene film. Each fiber sample was measured twice on the same spot located on the flattened portion of the fiber to confirm the spectrum. If the two spectrums of the same sample differed, a third spectral measurement was taken to confirm the correct results.

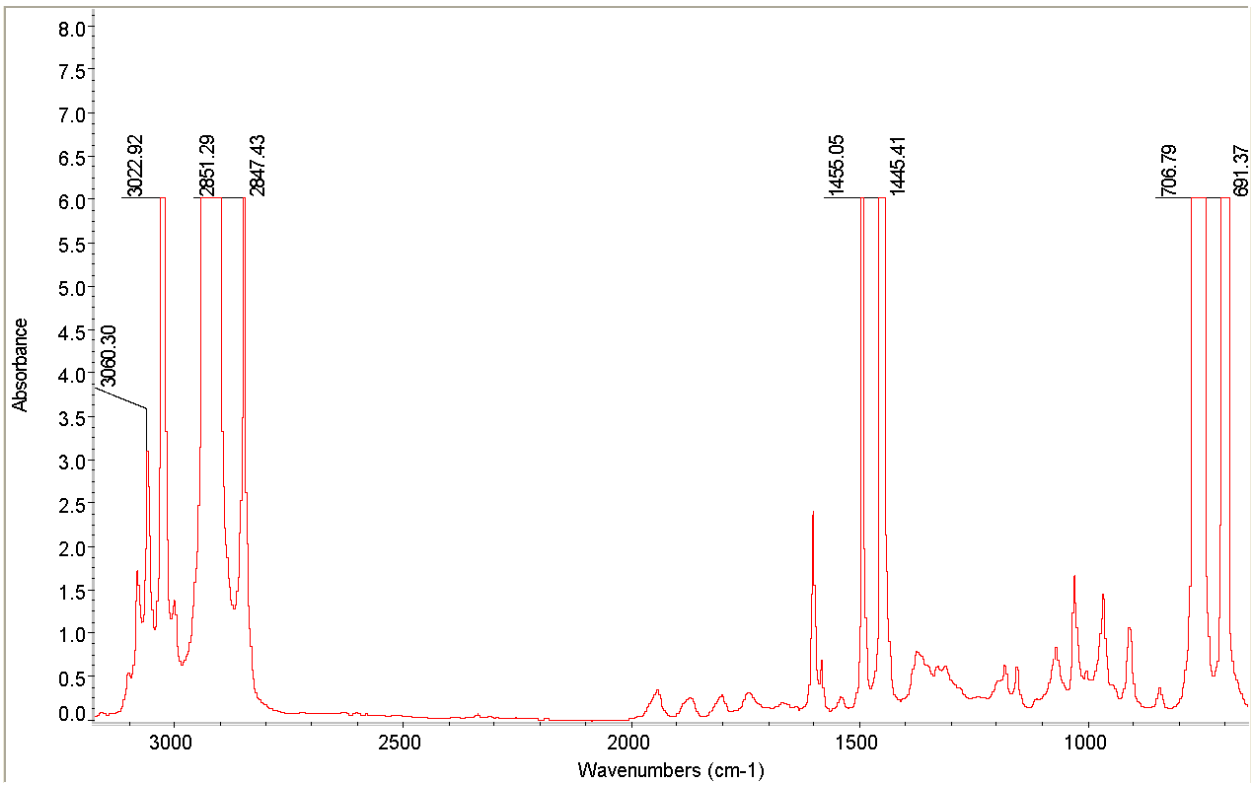

Figure 10. IR spectrum of Polystyrene film standard taken before sample measurement.

IV. Results

4.1 Fiber Counting Data

The total number of unknown fibers counted was 20,164 fibers under PLM at 10 x magnification, shown in ***Table 4*** below. Tape lift A from a female dressing room in Kohl's was the most concentrated with unknown fibers. Tape lift C, from the male dressing room in Marshalls was the least concentrated with unknown fibers.

Table 4: Number of unknown fibers counted on tape lifts

Tape lift Sample	A	B	C	D	E	F
Lane						
2	835	406	26	537	353	183
7	802	361	249	408	389	238
12	763	342	281	504	408	359
17	802	399	348	425	554	287
22	745	356	278	516	535	290
27	684	280	336	322	467	315
32	924	241	239	346	483	326
37	900	199	208	213	375	327
Total Fibers per tape lift	6,455	2,584	1,965	3,271	3,564	2,325

Total Number of Fibers: 20, 164

4.2 Polarized Light and Comparison Microscopic Analysis

Using polarized light microscope (PLM) to observe the physical properties of the unknown fibers, five fibers were designated as possible matches to the known teal colored cashmere fiber and seven fibers were designated possible matches to the known black acrylic fiber. ***Table 5*** is an example of the fiber elimination process using PLM.

Physical characteristics of each fiber such as type of fiber, color, diameter (in ocular scale divisions), cross- sectional shape and presence of delustrants or scales were used for the exclusion of the questioned fibers from the tape lifts. If a fiber was found to be a potential match, its diameter was re-measured with a calibrated ocular stage to provide an accurate measurement of diameter. ***Figure 11*** shows a picture of an unknown hair fiber that was eliminated based on diameter and scale patterns that deviated from the characteristics of the target cashmere fiber.

Table 5: PLM discrimination of samples from tape lift D at 40 x magnification

Sample	Description/Characteristics	Possible Match TC or BA?
D1	Light blue, round, no delustrants, 5 osd, no scales	No
D2	Med blue, round, no delustrants, striations, transparent, like cotton	No
D3	Black cotton	No
D4	Light blue, round, no delustrants, possible acrylic	No
D5	Med grey, round, no delustrants, 3 osd, more transparent than target Black acrylic	No
D6	Black cotton	No
D7	Med blue, round, no delustrants, 2 osd	No

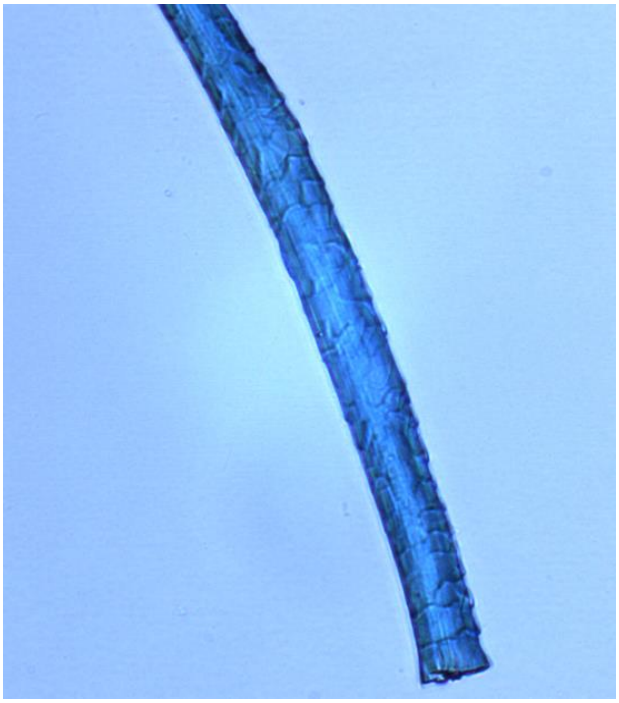

Figure 11. A12 fiber sample with scales that resemble wool under 10 x magnification.

Using the comparison microscope at 10x magnification and 40x magnification, more fibers were eliminated based mostly on color and /or scale patterns. ***Figure 12*** shows an example of sample A6, a questioned fiber, being compared to the acrylic target fiber on the left. Sample A6 appeared to be of the acrylic class, delustrants were present, and it had a round cross-sectional shape. The diameter of the sample appears smaller than the known fiber (left) at the same magnification of 40x. Therefore, sample A6 was eliminated as a potential match to the target fiber.

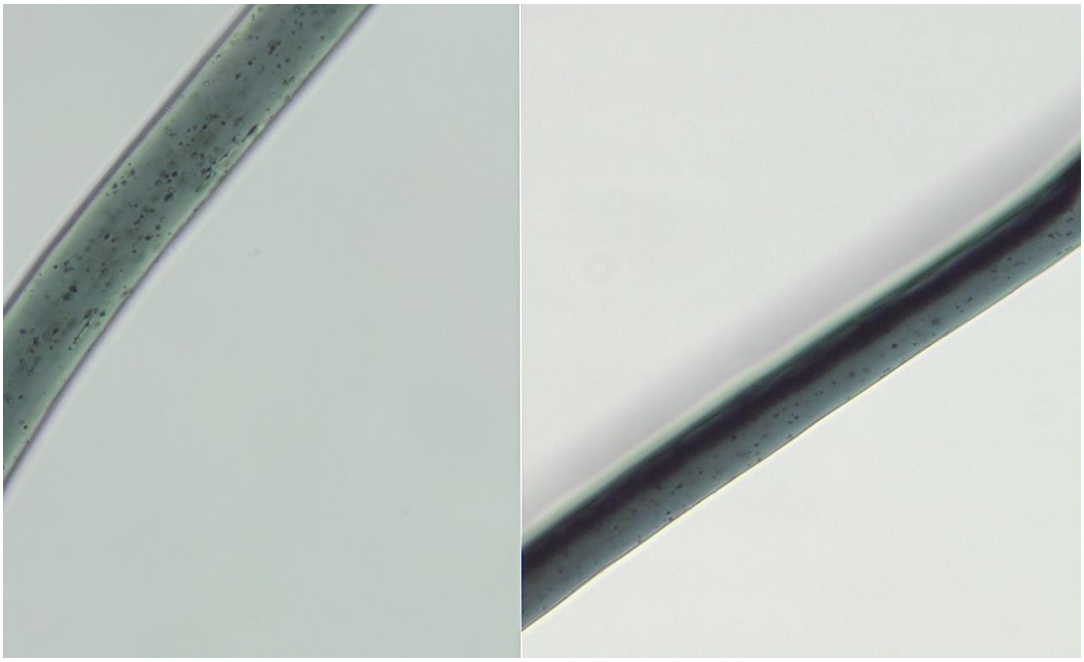

Figure 12. Image of known black acrylic fiber (left) and unknown fiber sample A6 (right) on comparison microscope (40 x magnification).

In some cases the fiber color appeared to be lighter or darker than the color of the target fiber. The side by side comparison was also useful when trying to differentiate between the scale pattern of the unknown hair fiber and that of the cashmere fiber. Of the seven possible matches to the known black acrylic fiber, seven samples were eliminated based on observation under the comparison microscope. Of the five unknown fiber samples that were thought to be a possible match to the target cashmere fiber, two required further discrimination via MSP analysis (**Figure 13**) and three were eliminated.

Figure 13 shows pictures of sample A10 on the left and sample A15 on the right under MSP analysis at 10x magnification. Both samples have the same diameter as the known cashmere fiber. However, the scales of the A10 fiber are difficult to distinguish and therefore difficult to differentiate from the target cashmere fiber. Sample A15

possesses a scale pattern similar to the target cashmere fiber, which in addition to the other similar physical characteristics, was cause for the sample to require further analysis.

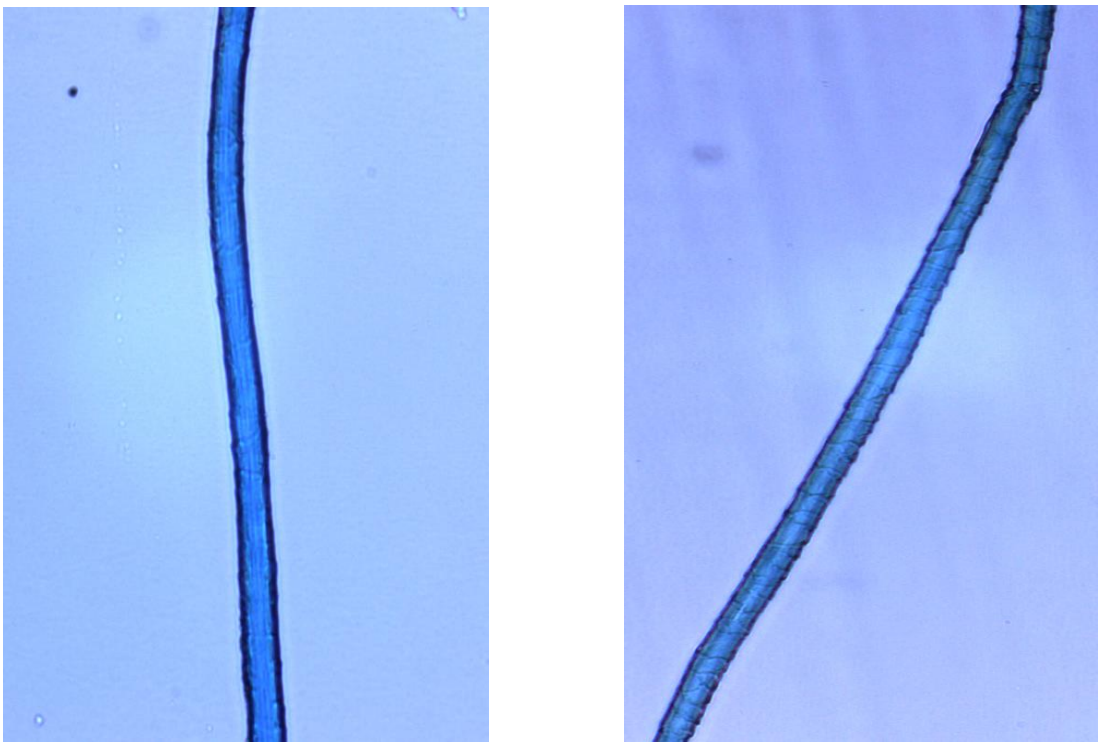

Figure 13. Image of fiber samples A10 (left) and A15 (right) under MSP analysis (10 x magnification).

4.3 Microspectrophotometric Analysis

To determine if a spectral match was present, the spectrum of each unknown (***Figure 15 and Figure 16***) were laid on top of the known cashmere spectrum (***Figure 14***), with the x and y values matching up, and then held up to the light to see how the spectral patterns varied.

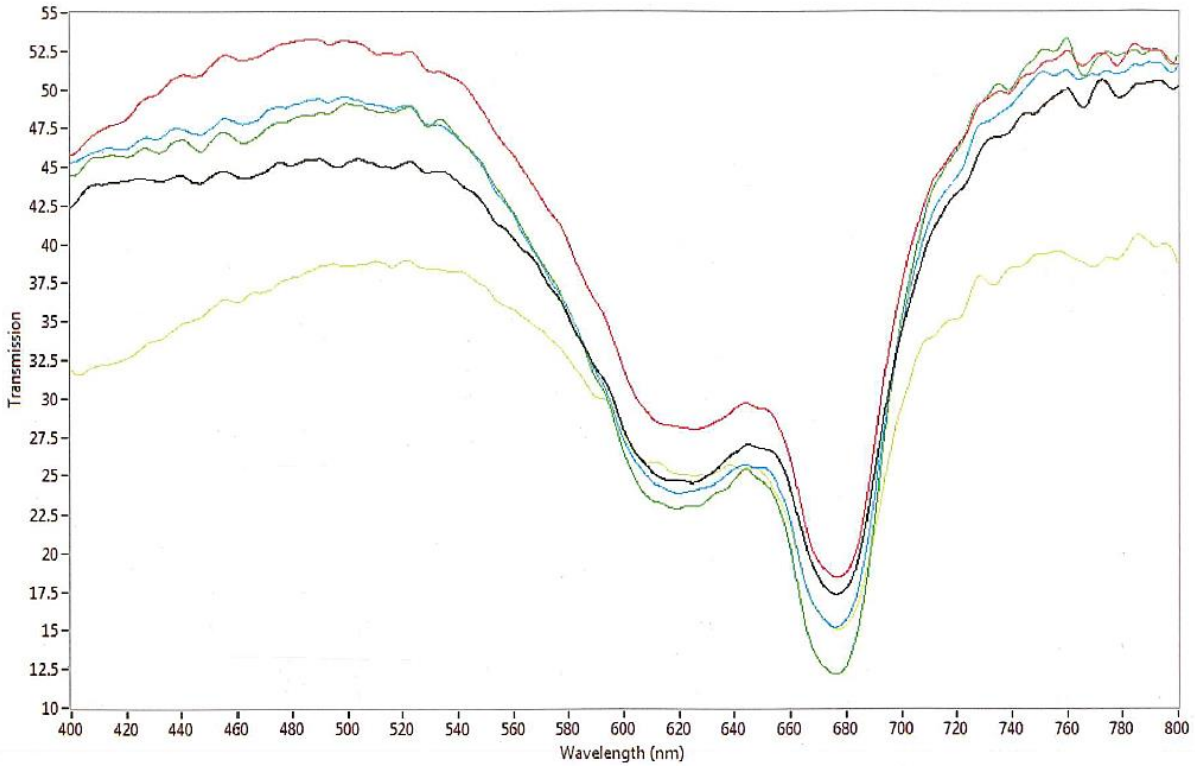

Figure 14. MSP spectra collected at five different locations on known teal colored cashmere fiber.

The transmission scale in ***Figure 14*** ranges from ten to fifty-five. Each spectrum of the known teal colored cashmere fiber, varied in intensity and therefore transmission value. The main characteristics of these spectra are peaks and valleys located between 600 nm to 800 nm. All five spectra show a downward slope beginning at 400 nm before a slight rise at about 610 nm followed by a steep downward slope, which is followed by a significant rise at 670 nm to 800 nm.

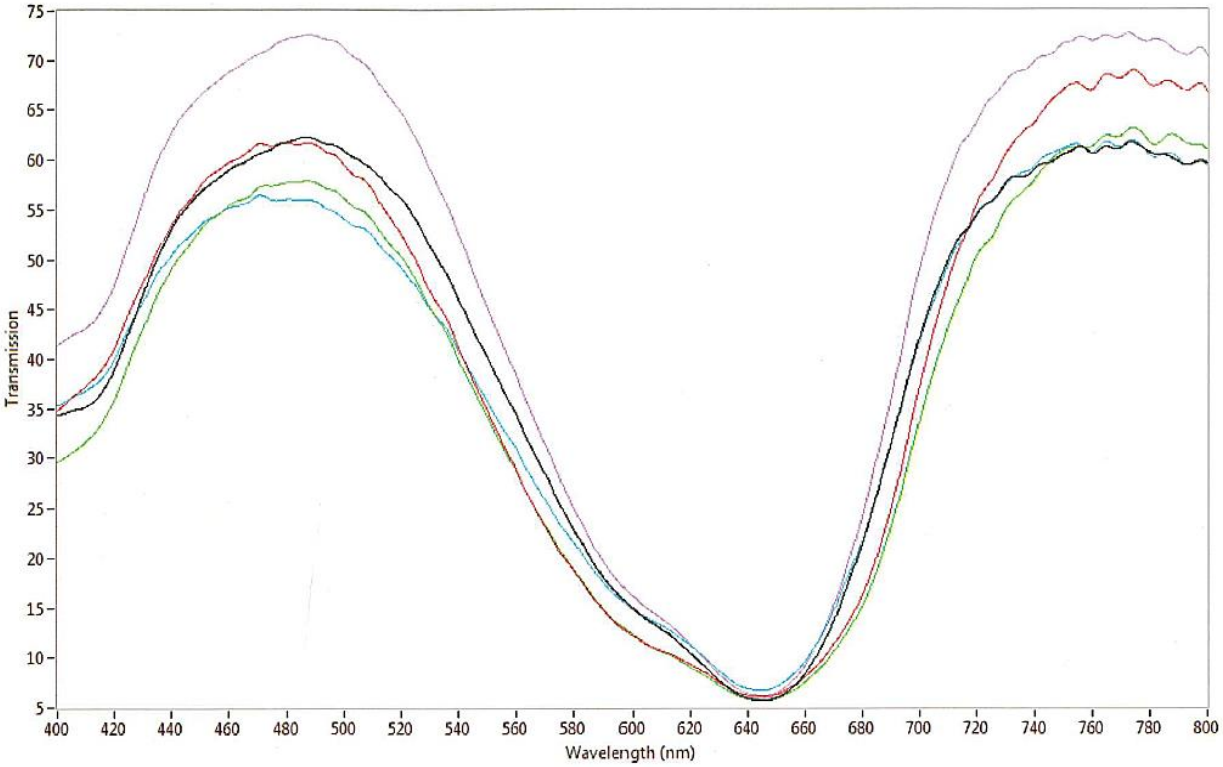

Figure 15. MSP spectra measured at multiple locations on sample A10.

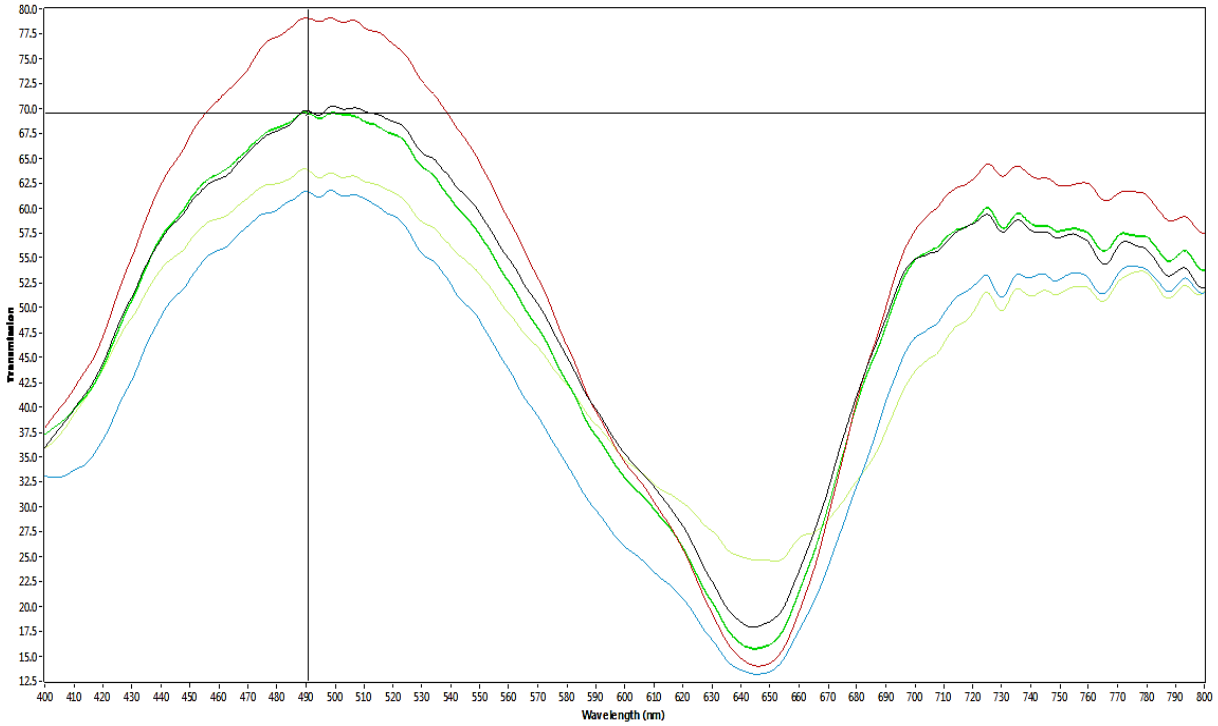

Figure 16. MSP Spectra of sample A15 fiber measured at multiple locations.

In ***Figure 15***, the transmission scale ranges from five to seventy-five. All five spectra of sample A10 show a similar pattern with varying intensities of its transmission value. The main characteristics of these spectra are a broad peak at 480 nm and a broad valley located around 645 nm.

The transmission scale in ***Figure 16*** ranges from 125 to 800. Each spectrum of the spectra of sample A15 vary in intensity, but display the same overall spectral pattern. The main characteristics of these spectra are a broad peak around 490 nm, a narrower valley around 645 nm, followed by an incline in the spectra.

4.4 FT-IR Microscopic analysis

The reproducibility of the IR spectrums varied within each sample. Absorbance was the most varying characteristic among the spectra. The presence of what appears to be a double peak between 2400cm^{-1} to 2300cm^{-1} also varied in same samples. It is possible that two peaks are indicative of carbon dioxide as a result of breathing. Variations in absorbance as well as the presence of these two peaks can be seen in all of the IR spectra (***Figures 17, 18, 19,*** and ***20***). However, the IR spectra of the known cashmere fiber that was not previously mounted, shown in ***Figure 18***, have the best reproducibility of spectral results.

In ***Figure 17,*** the three IR spectra taken from the previously mounted target cashmere fiber are shown. As mentioned, the intensities vary as well as the presence of a minor double peak between 2400cm^{-1} to 2300cm^{-1}. Spectrums 1 and 2 display these peaks, but in reverse of each other, while spectrum 3 does not show the presence of these peaks. In addition to possible CO_2 , there is a presence of a broad band on the known cashmere

spectrum (***Figure 18***) as well as A10 (***Figure 19***) and A15 (***Figure 20***) appearing between 3700 cm^{-1} to 3150 cm^{-1} could be the presence of water.

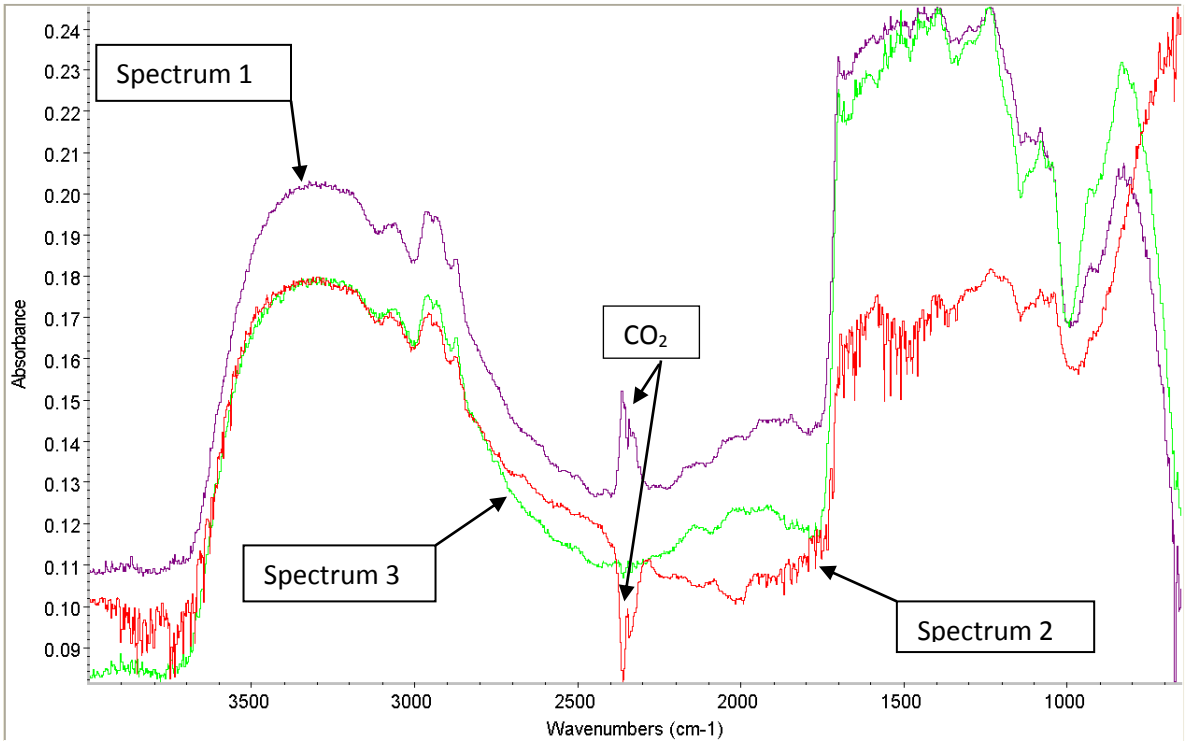

Figure 17. Measurements taken on same spot on previously mounted teal colored cashmere fiber.

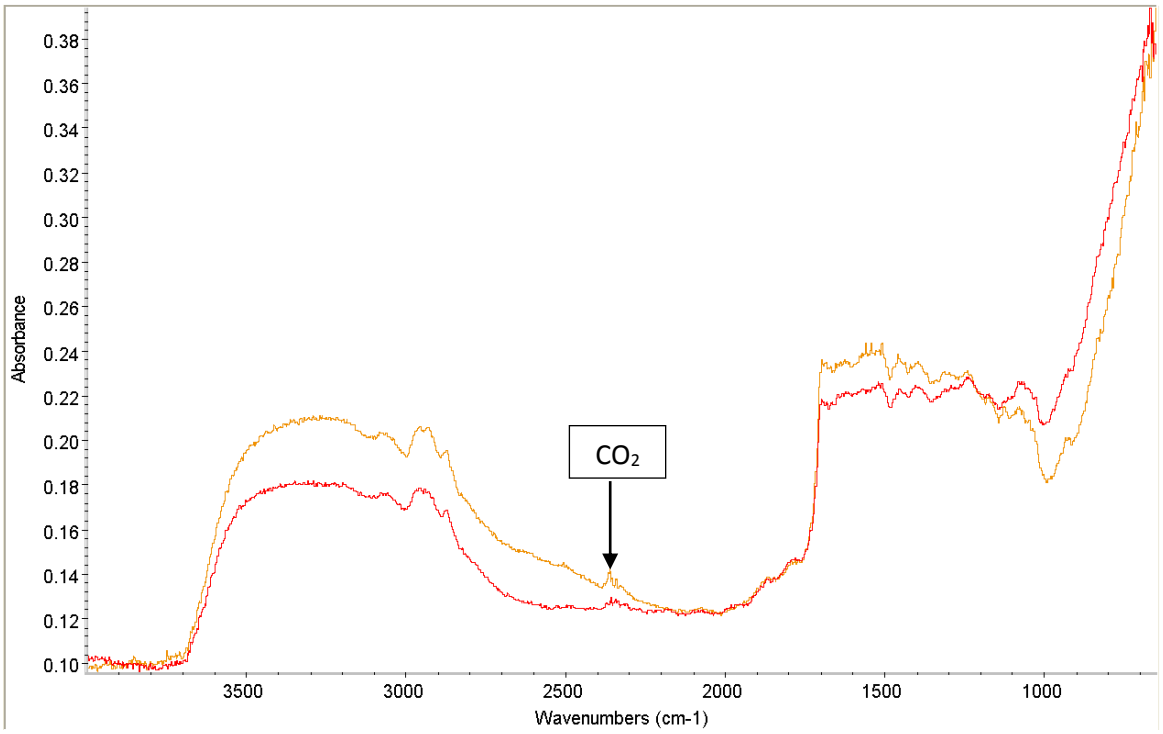

Figure 18. Measurements taken from same spot on teal colored cashmere fiber.

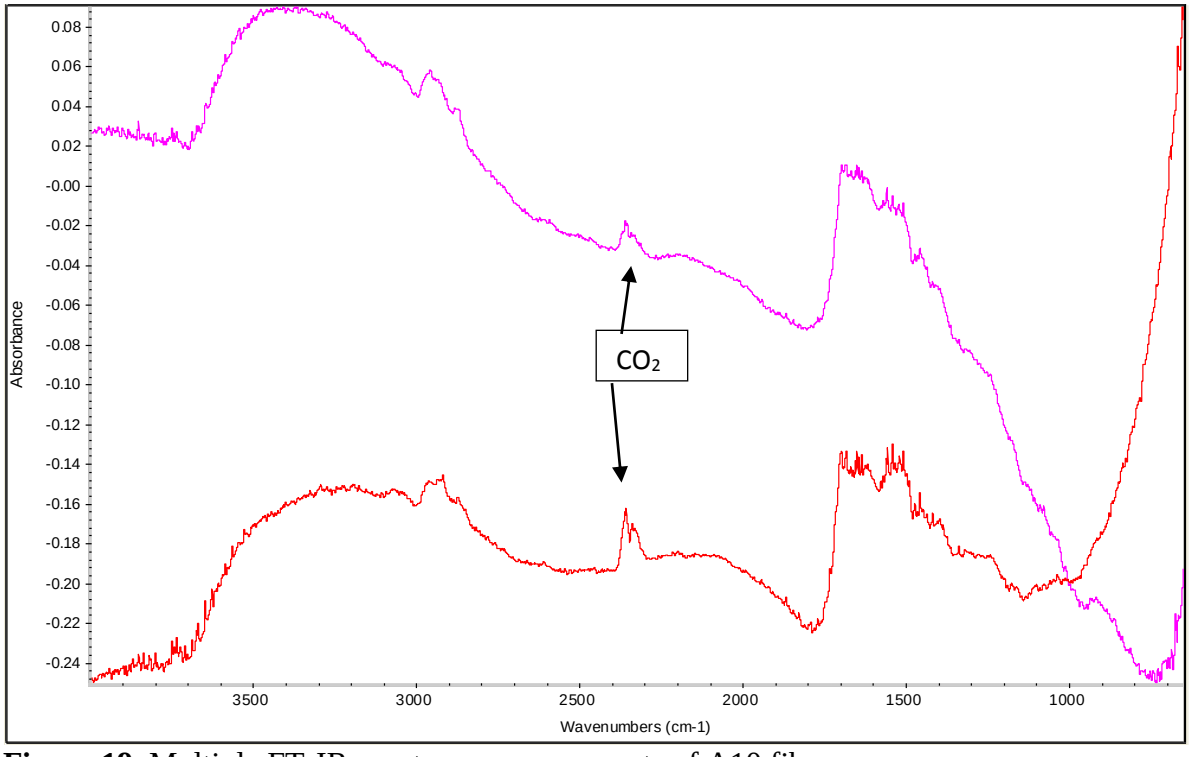

Figure 19. Multiple FT-IR spectrum measurements of A10 fiber.

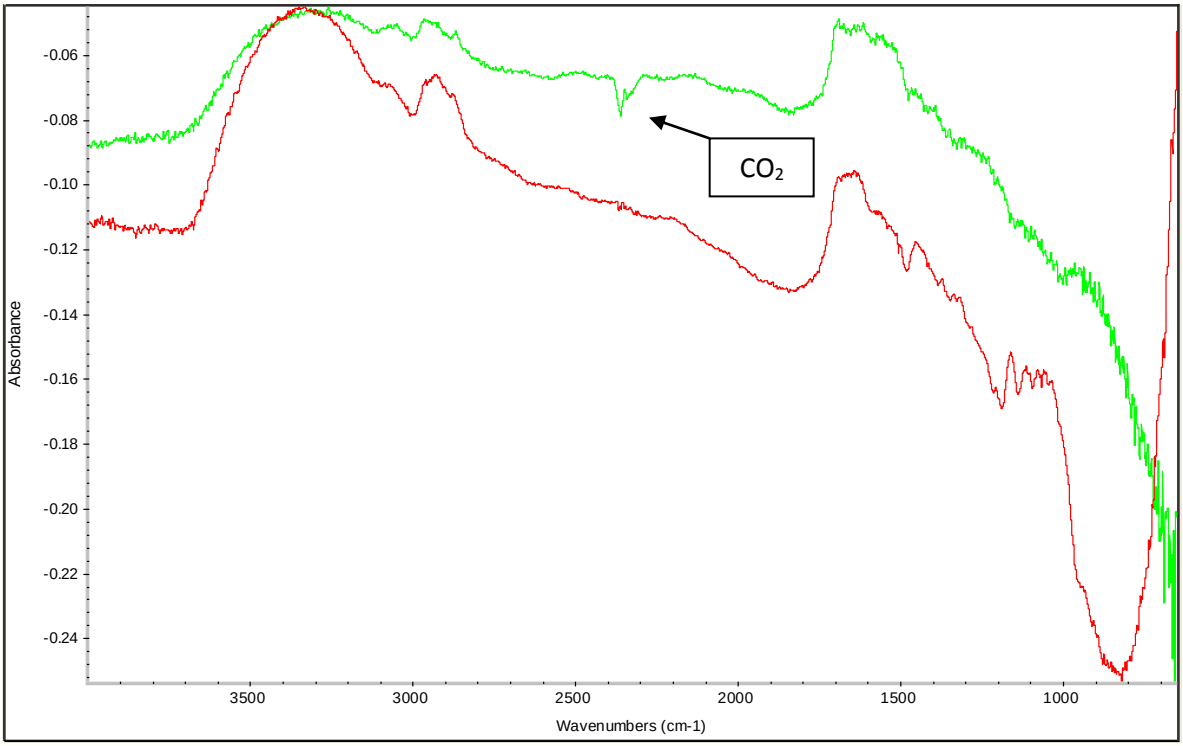

Figure 20. FT-IR measurements of A15 fiber.

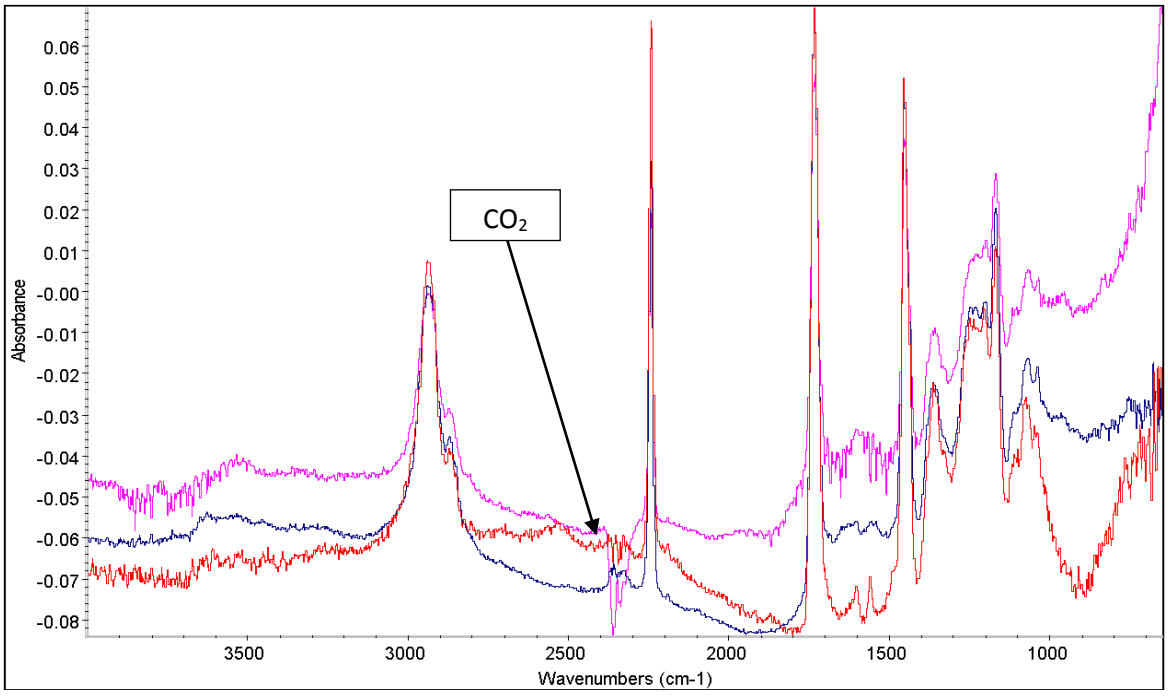

Figure 21. Multiple FT-IR spectra of the washed known black acrylic fiber.

4.4.1 FT-IR Spectra of Questioned Fibers Compared to Known Teal Colored Cashmere Fiber

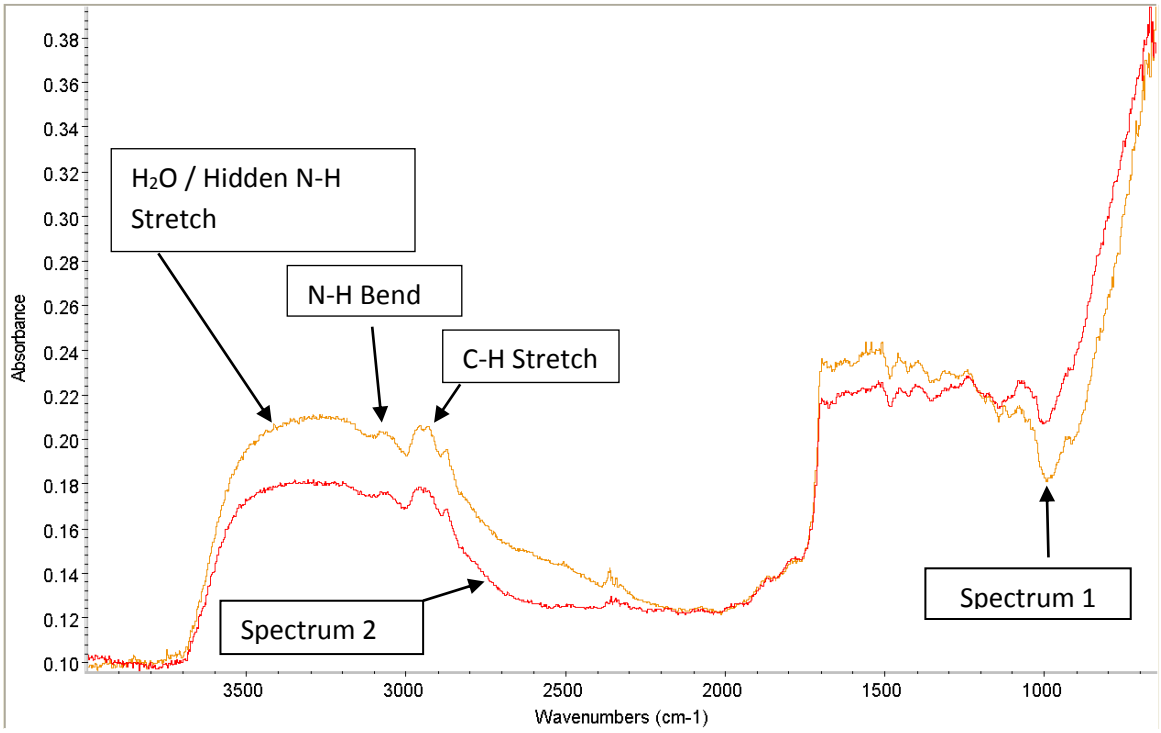

Figure 22. General features of cashmere spectrum, shown on known cashmere spectra.

Looking at ***Figure 22*** of the previously mounted known teal colored cashmere fiber, the minor peak around 3500 cm^{-1} could indicate an overtone of an N-H bend. Minor and barely visible peaks in the 1699 cm^{-1} to 1350 cm^{-1}.

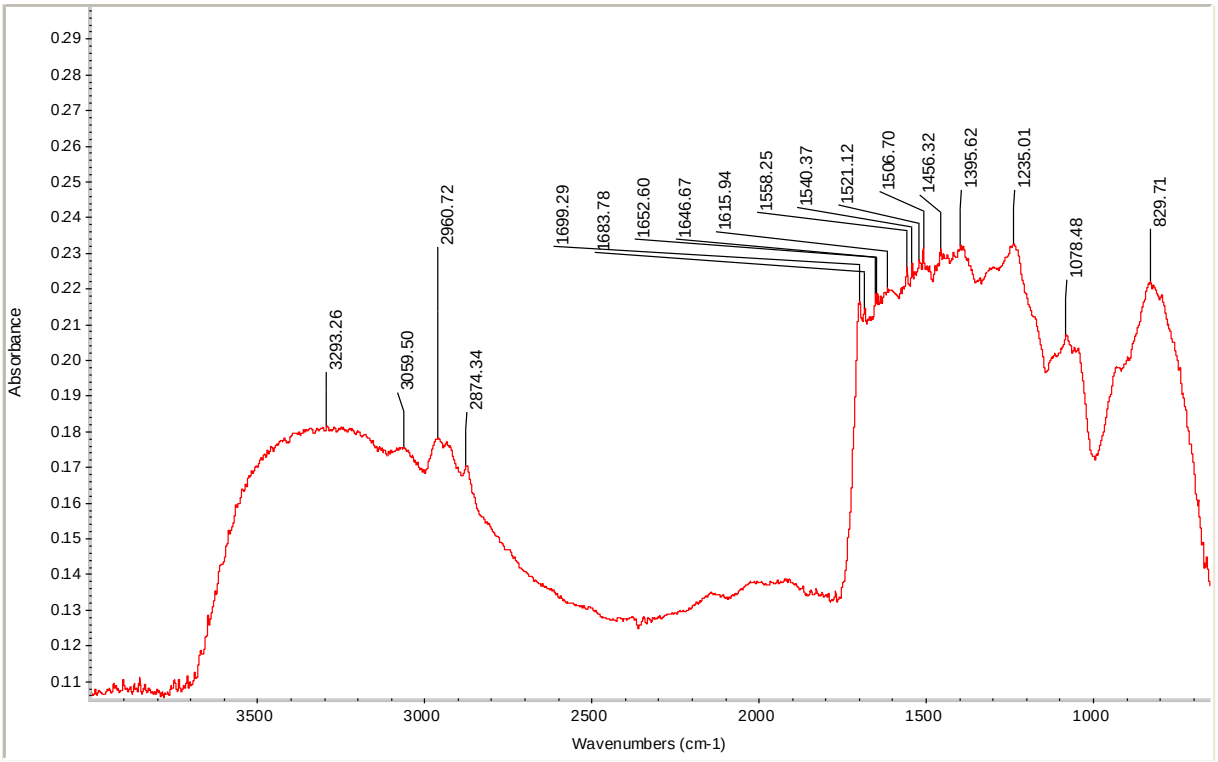

Figure 23. FT-IR spectrum of washed known teal colored cashmere fiber.

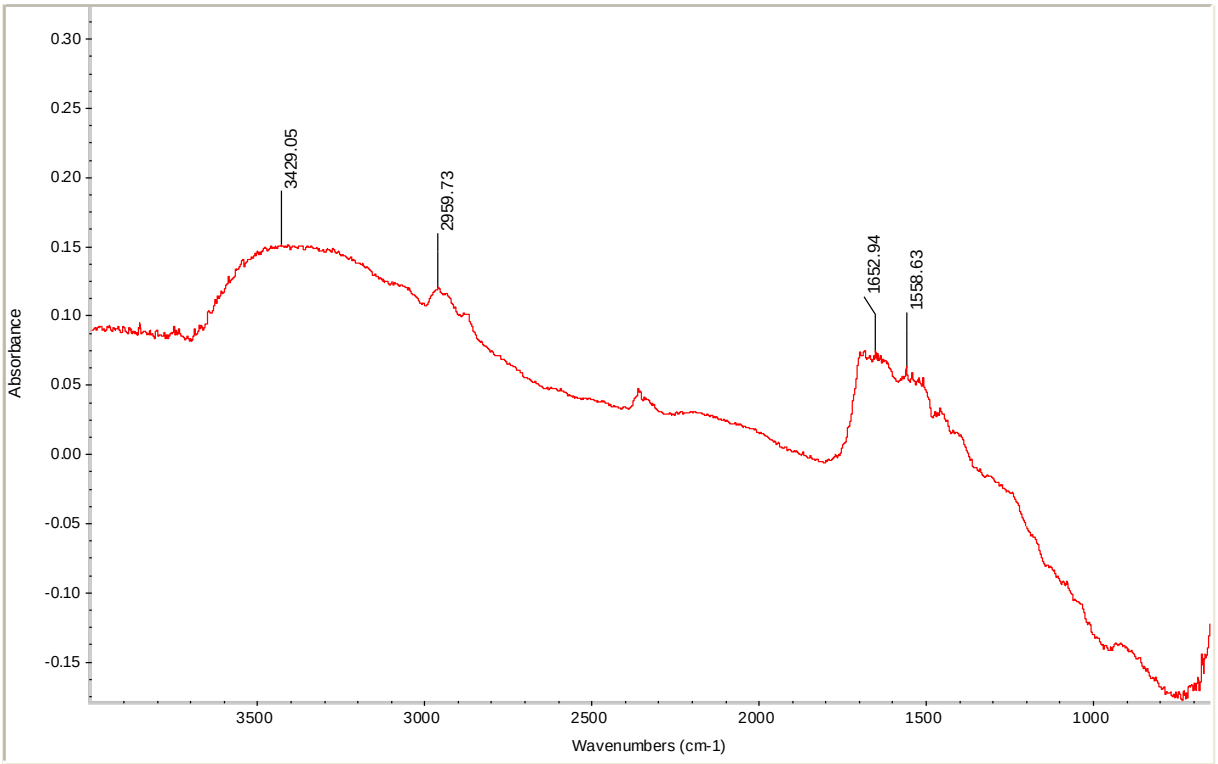

Figure 24. FT-IR spectrum of A10 fiber.

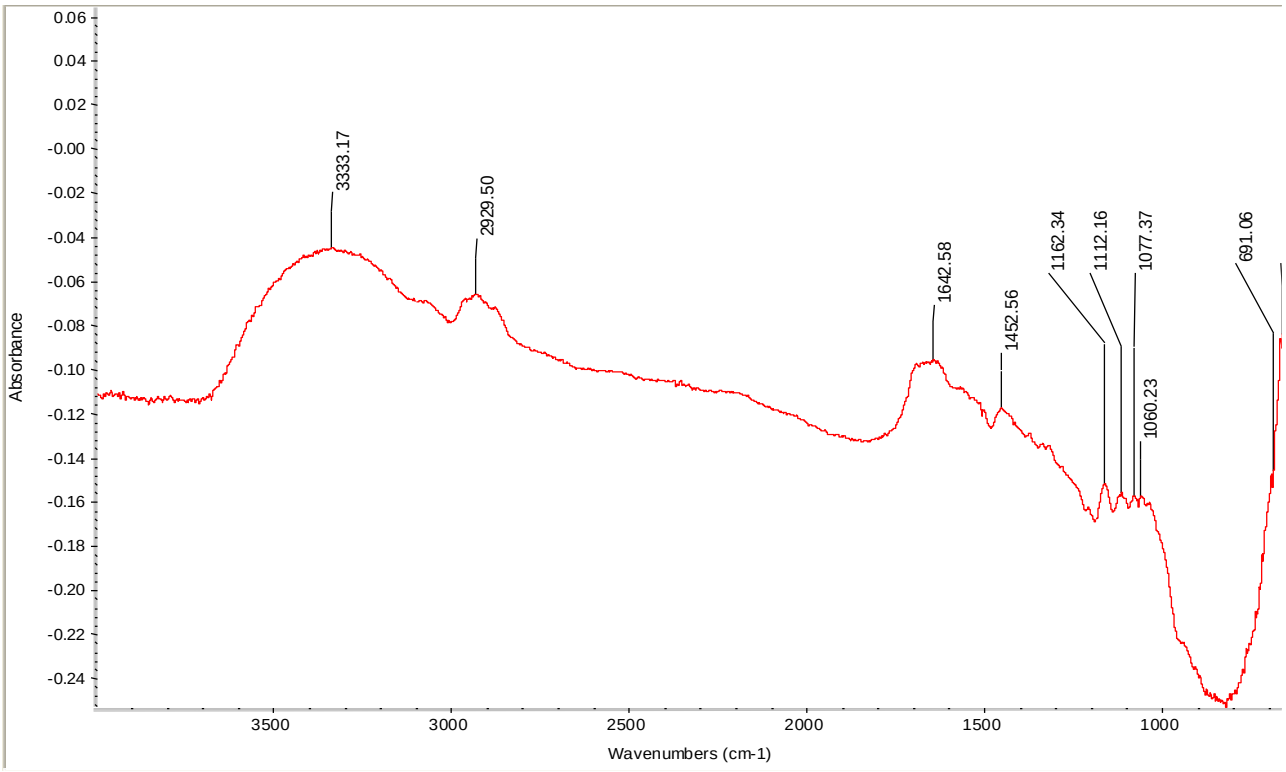

Figure 25. FT-IR spectrum of A15 fiber.

4.5 Integrity of Known Fibers Analysis

Since the unknown fibers were mounted in glycerol, it was necessary to subject the known samples under the same treatment to maintain consistency in the results. In order to be certain that the chemical structures of the fibers were not compromised by the glycerol mounting media, FT-IR measurements of known fibers that were not mounted in glycerol were taken for comparison to spectra of the known fibers mounted in the media. In general, it appears that the mounting media had no significant chemical effects on the known fibers.

4.5.1 FT-IR Spectrum of Known Teal Colored Cashmere Compared to Fiber Mounted in Glycerol

Comparison of the target cashmere fiber that was not mounted in glycerol (Figure 26) to the target cashmere fiber that was mounted in glycerol (Figure 27), some variations can be seen. The intensity of the broad peak at 3291 cm^{-1} in Figure 26, and at 3293 cm^{-1} in Figure 27 vary. This is due to the fiber in Figure 27 being washed with deionized water after being mounted in glycerol. It is possible the fiber did not dry fully before measuring the fiber with FT-IR microscope. However, the overall intensity of the washed cashmere fiber is significantly higher than the unwashed cashmere fiber.

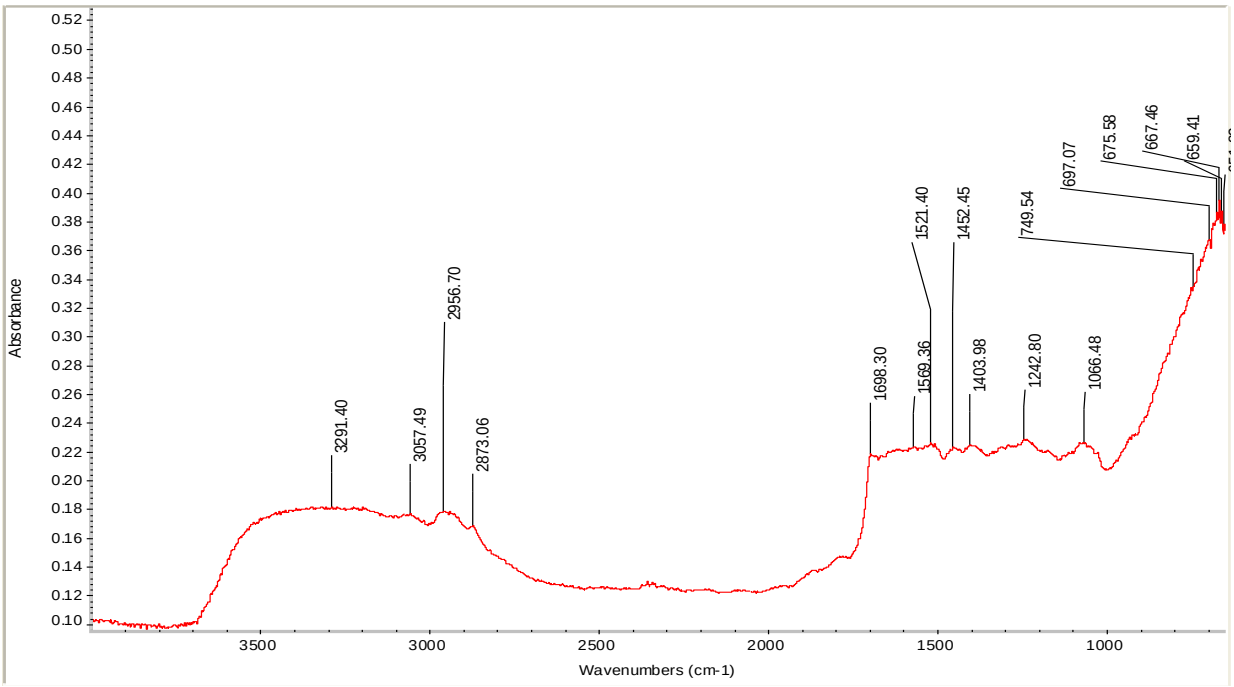

Figure 26. FT-IR spectrum of known teal colored cashmere fiber.

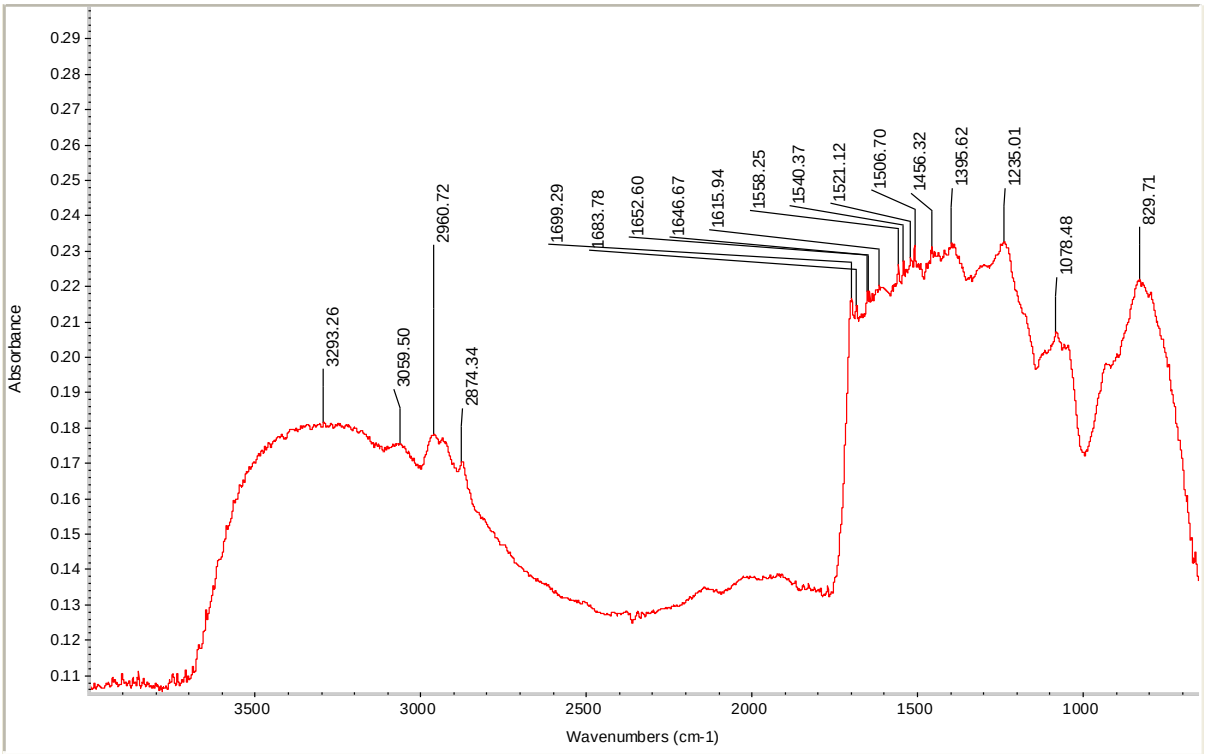

Figure 27. FT-IR spectrum of target cashmere fiber that was washed with H2O, showing variation in artifacts between 2300-2400 cm-1 and broad peak at 3293 cm-1.

4.5.2 FT-IR Spectrum of Known Black Acrylic Fiber Compared to Fiber Mounted in Glycerol

Looking at the spectra of the black acrylic fiber, there are a couple minor differences. First, looking at ***Figure 28*** the spectrum of the acrylic fiber that was not mounted in glycerol, there is a presence of two minor peaks at around 1600cm^{-1} and 1550 cm^{-1}. The baseline in the spectrum of the acrylic fiber that was previously mounted in glycerol is raised from the x-axis, which may have caused the two peaks to be hidden in the baseline.

The intense peak at around 1730 cm^{-1} is indicative of a carbonyl (C=O) stretch, which many acrylics tend to have. The peak at around 2245cm^{-1} is a key indicator that the fiber is from an acrylic or modacrylic classification. Upon further analysis, using information provided by Grieve (1995), it is possible that the IR spectra of the black acrylic fiber

resemble that of PAN/MA/MVP. Orlon 28 is an example of this type of fiber. However the spectrum shown by Grieve (1995) of this fiber, does not match the IR results from the known black acrylic fiber. There were several peaks in the Orlon 28 spectrum that are not present in the known black acrylic spectrum ***Figures 28*** and ***29***. This is also the case for the second possible identification of the known acrylic, PAN/MA/Basic additives+ DMF acid dying type, for which Dralon A is an example. Based on spectral analysis from two sources (Grieve, 1995 & Kirkbride and Tungol, 1999), the sub-classification of this fiber remains inconclusive, but never the less can be identified as an acrylic fiber due the presence of a nitrile peak at 2245 cm^{-1}.

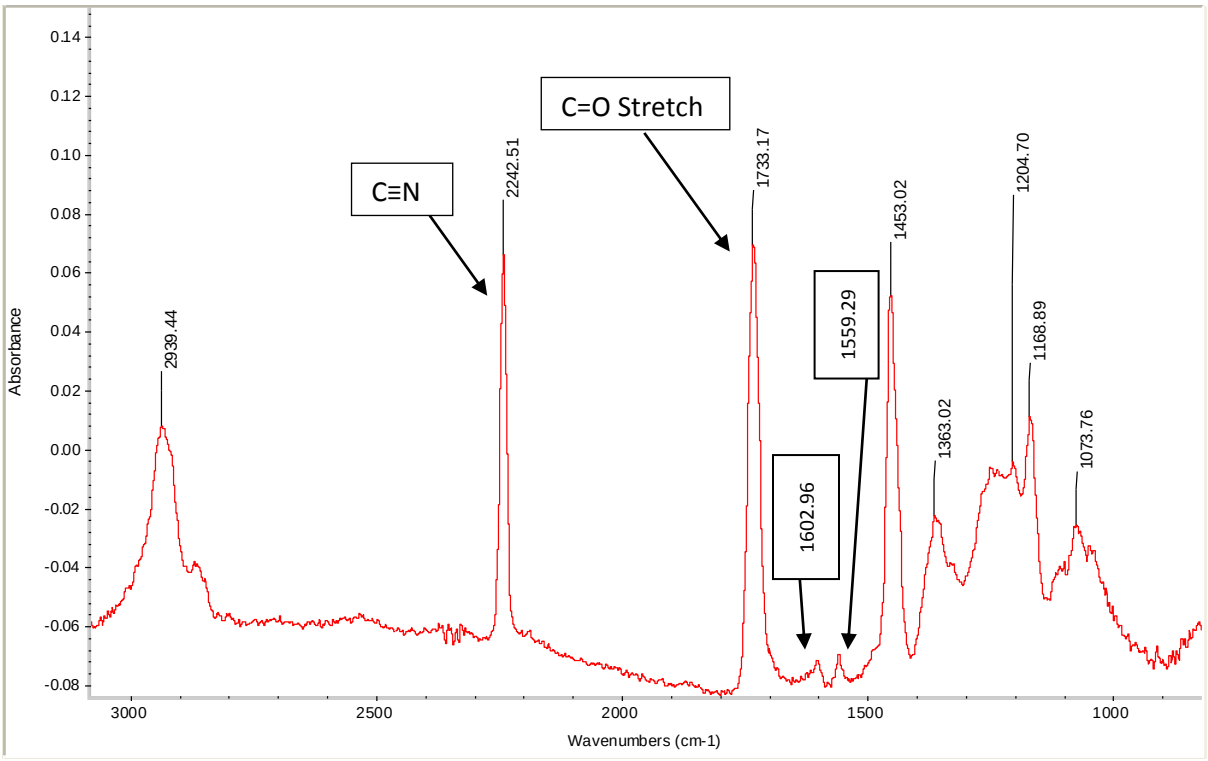

Figure 28. FT-IR spectrum of known black acrylic fiber.

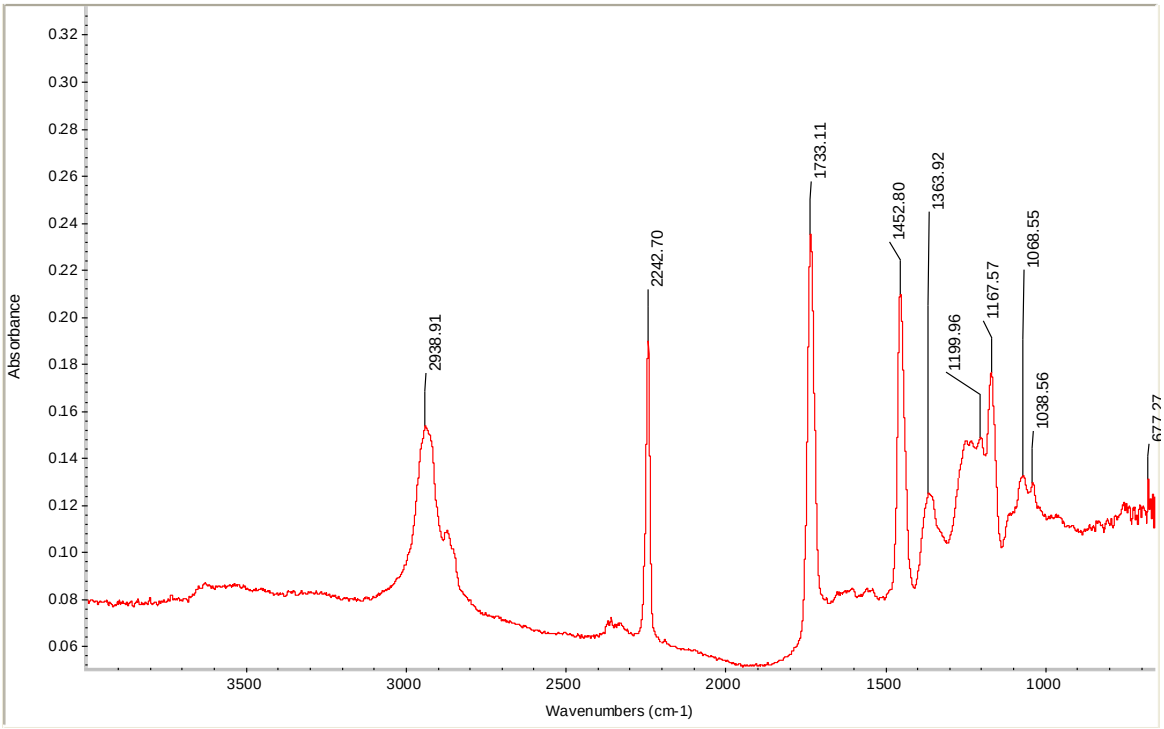

Figure 29. FT-IR spectrum of known black acrylic fiber that was removed from glycerol.

V. Discussion

5.1 Discussion of MSP Results

The spectra of the known cashmere fiber in ***Figure 14,*** with the exception of one measurement, have the same overall pattern. The spectrum with the lower transmission measurement may have been taken at a location on the fiber which may have differed in thickness or color uptake from where the other measurements were taken. Since this sample is a natural fiber, it makes sense that there would be some variation between spectra because the thickness or color would vary in a natural fiber.

Figure 15, shows spectra of sample A10, which also vary in intensity of transmission measurements. As with sample A15 in ***Figure 16***, the spectra of A10, appear to have the same overall pattern. However, variation in transmission measurements may be a result of inconsistent size of thickness throughout both fibers as they are also classified as an animal fiber.

Compared to the known spectra of the teal colored cashmere fiber in ***Figure 14***, the difference of the spectral patterns of sample A10 (***Figure 15***) and sample A15 (***Figure 16***) are quite obvious. This indicates that the dye used for the A10 fiber and the A15 fiber may be different from the known cashmere fiber. Another possibility for the spectral differences between samples may be that the material of the unknown samples did not absorb the dye the same as the known fiber. This provides justification to eliminate samples A10 and A15 as potential matches to the target cashmere fiber.

5.2 Discussion of FT-IR Results

Although there were no potential matches to either target fiber, FT-IR analysis was conducted on the two target fiber and samples A10 and A15. The fiber type should be identified for stronger evidential value in court (Gaudette, 1988). FT-IR results show some similarities of the known cashmere fiber (***Figure 23***), sample A10 (***Figure 24***) and sample A15 (***Figure 25***) in the 3700cm^{-1} to around 2500 cm^{-1}. All show a broad peak with the high of the band between 3343 cm^{-1} and 3293 cm^{-1} , which may be from water. The samples also show same spectral features around 3060 cm^{-1} (N-H bend) and between 2930 cm^{-1} and 2960 cm^{-1} (C-H stretch). The three samples began to differ between 1700 cm^{-1} to 700 cm^{-1}. There is an incline in each spectrum around 1700 cm^{-1} and the pattern begins the same, but then varies. This may be due the thickness of the samples affecting the resolution of peaks and absorbance values. This problem may be rectified by rolling the fibers before IR analysis with even amount and right amount of pressure. However, the unknown fibers too small to roll and increased the risk of losing the samples.

The tightness of the condenser of the microscope was also an issue. Adjustment of the condenser affected the sharpness of the peaks, as seen in the live bench mode of the experiment. The condenser appeared to be broken as it was unable to retain its tightness during scans. This was confirmed before and after checks of the focus of the microscope on the sample. The focus on the sample after the measurements took place was not as good as before the scans took place. Also, the adjustment knob of the condenser was not to the original tightness.

Despite these issues, ***Figure 28*** and ***Figure 29*** clearly show samples of the acrylic class. ***Figure 28*** is the IR spectral measurement of the black acrylic fiber that was not

previously mounted in glycerol. ***Figure 29*** shows the IR spectrum of the black acrylic fiber that was once mounted in glycerol. Both spectra display two peaks that indicate an acrylic fiber as the classification and sub-classification of the samples; a strong Nitrile peak around 2245 cm^{-1} and a strong carbonyl peak around 1730 cm^{-1}. It was inconclusive as to the sub-classification of the fiber via the co-monomer.

Database searches did not yield conclusive results. No samples, not even the known fibers, had above a 75% match. For example, the known cashmere fiber not mounted in glycerol had a 42.92 % match to Isophorone, which is a solvent. Ideally this fiber should have produced results of keratin, which is the protein that is present in hair. Poor database matches could be the result of several factors. The IR instrument used to generate the IR spectrum for the databases could cause the discrepancies when comparing spectra. The resolution between instruments may vary, along with different practices in sample preparation. As seen in section 4.4, it is difficult to produce the same spectrum twice when working with hair fibers.

5.3 Potential Use in Forensic Casework

Based on light microscopic analysis, all potential matches to the target black acrylic fiber have been eliminated based on diameter, lack of delustrants and color comparison. ***Figure 30*** shows a summary of the exclusion of 20,164 unknown fibers as a potential match of the known black acrylic fiber. ***Figure 31*** shows a summary of the elimination of 20,164 unknown fibers as a potential match of a known teal colored cashmere fiber. Since all 20,164 fibers were eliminated as possible matches to either target fiber, the selected fibers are possibly good candidates for linking a perpetrator to a crime or victim around the location of the stores on Delaware Avenue. Each target fiber was shown to

originate from, in this case, a unique fiber source. The microscopic properties of the black acrylic fiber were shown to be distinct due to the combination of color, diameter and delustrants. PLM and comparison microscopic examination were of no use regarding discrimination between the known teal colored cashmere fiber and collected samples A10 and A15.

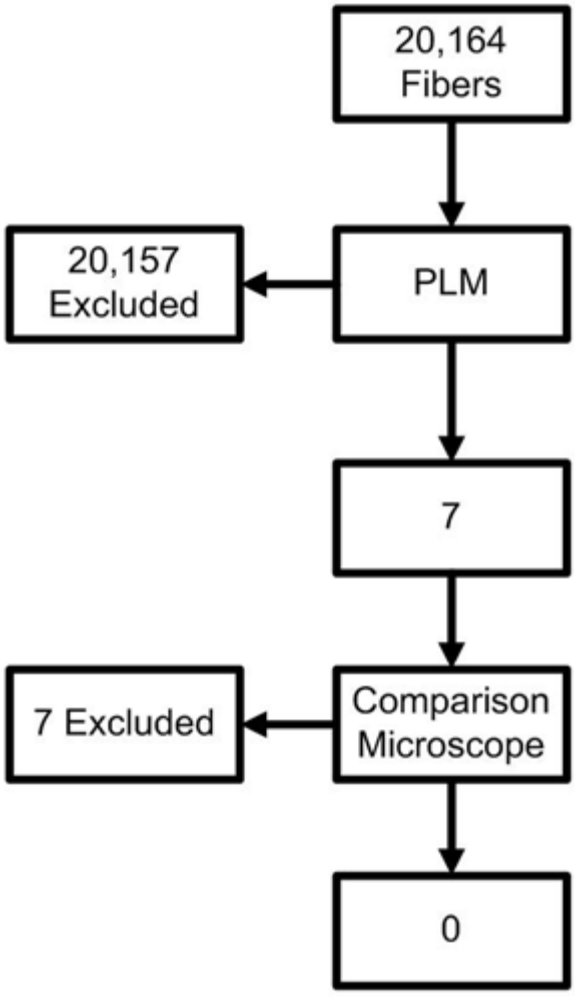

Figure 30. Analytical flow chart showing results of black acrylic fiber comparison.

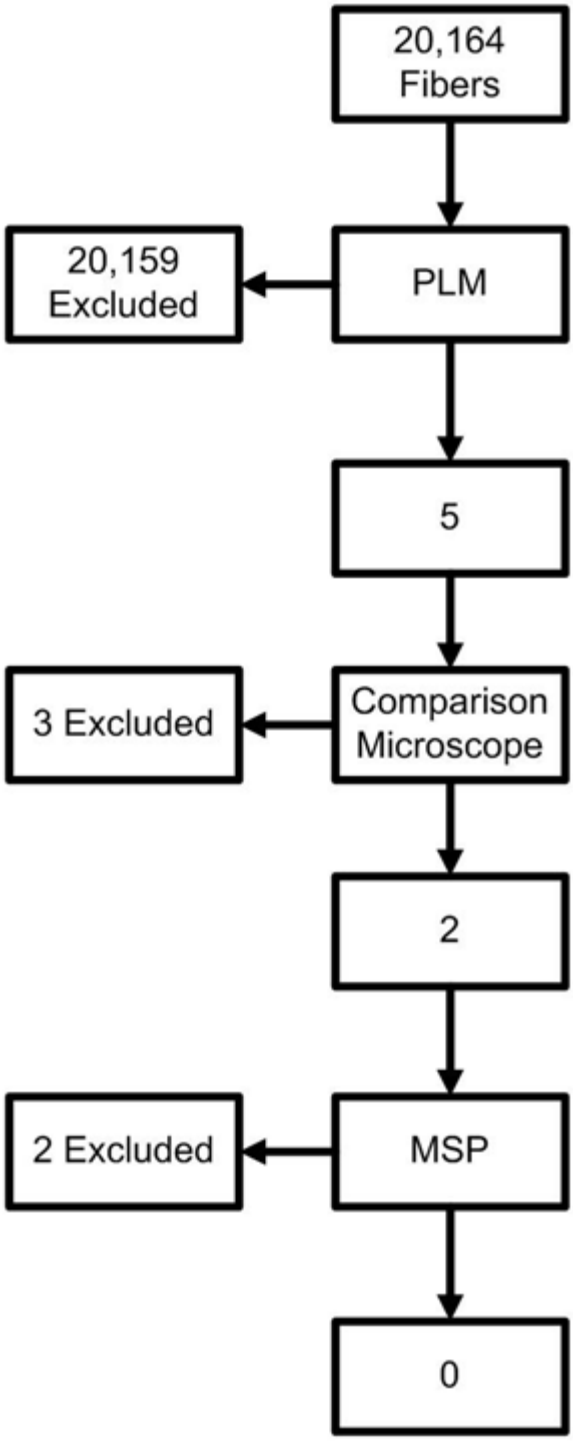

Figure 31. Analytical flow chart showing results of teal colored cashmere fiber comparison.

5.4 Recommendations for Improvements

In order to generate more precise data of fiber frequency/ significance, a broader area of stores should be included in the collection process, especially between neighboring counties, such as Erie and Niagara Counties. This would allow for possibly more fiber variation and give more weight to target fiber significance. This study attempted variance by collecting fibers from both male and female dressing rooms in addition to varying the budget range of selected clothing stores. However, if a wider scope could be added, data would be more useful for generating statistics for several reasons. First, the wider population collection would tell us for certain if neighboring areas have the same common fiber types and similar uncommon fiber types. Second, the higher the number of unknowns, the more weight the statistical data holds. This would make the significance of uncommon target fibers greater as the total number of unknown fibers increases, similar to statistics generated in DNA Profiling.

The total unknowns counted only included 22 percent of each tape lift. Counting fibers is time-consuming. It is also not error proof. Counting only 22 percent of each tape lift relays a general idea of target fiber significance. It is possible that more target fibers are present within the unexamined areas. Counting a greater area of each tape lift will generate greater statistical weight. Ideally, 100 percent of the tape lifts should be counted and examined for more accurate statistical data. Having a few people who receive the same fiber recognition training would be ideal for counting and spotting target fibers. Using an automated fiber finding system may save time locating target fibers. However, counting total number of unknown fibers is still necessary.

It is also recommended that the fibers be analyzed via FT-IR Microscope before analyzing the fibers with MSP. Working the procedure in this order will reduce the risk of losing fiber samples. Removal of the fibers from the mounting media and washing the fibers will not be necessary if MSP is the last step in analysis. Fibers can be mounted after FT-IR analysis is completed. The negative aspect of performing FT-IR analysis first is that because fibers may be short, the entire length of the fiber may be flattened. This change may not yield true MSP results. As with the FT-IR Microspectroscopy, non-uniform pressure while rolling the fiber may affect the spectral pattern. One last minor suggestion would be to have separate hand tools, such as tweezers and roller for both the known fiber and the unknown fibers.

VI. Summary

The manner in which certain types of forensic evidence are analyzed is being questioned. Providing statistical analysis with evidence, such as with DNA evidence, allows for investigators and the judicial system to determine the significance of the evidence. Analysis of fibers is one such case where the significance of particular fibers must be determined in order to give greater meaning to those fiber types as evidence. Target fiber studies must be conducted in an effort to provide forensic fiber examiners with some source of information in order to produce statistical analysis of fiber types. This target fiber study has provided the local forensic community with two types of fibers that have the potential to be of significance in local criminal cases. Through microscopic examination and microspectrophotomic analysis, 20,164 unknown fibers were eliminated. Results of this study concluded that potential matches to neither the known teal colored cashmere fiber nor the black acrylic fiber were not able to be distinguished.

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