QUANTIFICATION, IDENTIFICATION AND SOURCE DISCRIMINATION OF ANTHROPOGENIC MICROFIBRES IN MARINE AND FRESHWATER BODIES: A FORENSIC APPROACH

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QUANTIFICATION, IDENTIFICATION AND SOURCE DISCRIMINATION OF ANTHROPOGENIC MICROFIBRES IN MARINE AND FRESHWATER BODIES: A FORENSIC APPROACH

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Declaration

I declare that the work contained in this thesis has not been submitted for any other award and that it is all my own work. I also confirm that this work fully acknowledges opinions, ideas, and contributions from the work of others.

Any ethical clearance for the research presented in this commentary has been approved. Approval has been sought and granted through the Researcher's submission to Northumbria University's Ethics Online System on 31st of July 2019.

I declare that the Word Count of this Thesis is 35,115 words.

Name: Chimdia Nwanneka Adaeze KeChi-Okafor

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Abstract

Microfibres (MFs) – fibres with length <5 mm - are pervasive and pose a danger to aquatic environments. Typically, studies on MF pollution have focused on identifying synthetic fibres; however, forensic science studies have consistently demonstrated that anthropogenic natural MFs are more abundant than their synthetic counterparts. This discrepancy could be due to the different methodological approaches to MF characterisation. Accurate characterisation is critical, as natural MFs may be equal in their environmental threat. Therefore, it is essential to adopt appropriate methods that distinguish between all fibre types that result in accurate quantification. This study aims to address these gaps by using forensic methods to investigate the occurrence of MFs in marine and freshwater environments and a lesser-known pathway to them, namely in the form of tumble drying.

Conventional forensic methodological approaches, including the use of polarising light microscopy (PLM), an instrument capable of distinguishing between all fibre types, were used to identify and characterise MFs collected from marine and freshwater environments along the Kenyan-Tanzanian coast and Lake Victoria respectively. Additionally, the study evaluated lint filters and dryer sheets designed to capture MFs during tumble drying while drying cotton and polyester T-shirts together and quantified the amount of MFs released.

Conventional forensic methodological approaches revealed that natural MFs are dominant, constituting 55% and 78% of marine and freshwater samples, respectively. Among the natural fibres, cotton MFs were the most abundant in all sampled locations. Fine filters captured more MFs than coarse filters during sampling.

Over 90% of the MFs released from vented tumble dryers were cotton, demonstrating the important role of the garment itself to shed fibres that influences their prevalence in the environment rather than the quantities of fibre production.

Natural fibres cannot be excluded from MF pollution discourse as they are as pervasive as their synthetic counterparts. Central to the discussion of MF pollution is the shedding capacity of textile materials i.e. what is shed by a material influences its prevalence in the environment, and quantifying this, is primarily reliant on the pore size of capturing devices used.

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List of Abbreviations

MF-Microfibre

FMP- Fibre Microparticle

FF- Fragmented Fibre

LPM – Low Power Microscope

HPM – High Power Microscope

CM – Comparison Microscope

PLM- Polarising Light Microscope

FLM- Fluorescence Microscope

SEM- Scanning Electron Microscope

FTIR- Fourier Transform Infrared Spectroscopy

UV/Vis MSP- Ultraviolet/Visible Microspectrophotometer

LC-MS- Liquid Chromatography-Mass Spectrometry

CE-MS Capillary Electrophoresis-Mass Spectrometry

TLC- Thin Layer Chromatography

HPLC- High-Performance Liquid Chromatography

CE- Capillary Electrophoresis

PY-GC-MS- Pyrolysis–Gas Chromatography–Mass Spectrometry

FPA-FTIR Focal Plan Array-FTIR

SRS - Stimulated Raman Scattering Microscopy

GN & GS- Global North & Global South

Acknowledgements

As far as I can remember, I've always loved teaching people. Even as a little girl in nursery school, my parents would recall how I used to 'correct' my private tutor. When I eventually decided I wanted to lecture as an adult, I knew I had to undertake a PhD degree. Life happened; I put the idea of doing a PhD on hold to start a family only to eventually begin it at the 'unlikeliest' of times. The past four years have been the busiest and fulfilling time of my life so far having to juggle being a wife, mom of 3 under the age 6, coping with the responsibilities of family and doing a full time PhD! I could only have managed these by the grace of God and the help He provided through the support of my family and academic supervisory team. To Him be all the glory now and forever, amen!

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Publications

Two peer-reviewed journals were published during the PhD program. The titles of the publications and the corresponding result chapters are outlined below.

1. Lant, N. J., Defaye, M. M., Smith, A. J**., KeChi-Okafor, C**., Dean, J. R., and Sheridan, K. J. (2022). The impact of fabric conditioning products and lint filter pore size on airborne microfiber pollution arising from tumble drying. *Plos one*, *17*(4), e0265912.

   This publication is based on the study findings in Chapter 5

   "Exploration of Factors Contributing to the Release of Microfibres by Tumble Dryers"

2. **KeChi-Okafor, C.,** Khan, F.R., Al-Naimi, U., Béguerie, V., Bowen, L., Gallidabino, M.D., Scott-Harden, S. and Sheridan, K.J., 2023. Prevalence and characterisation of microfibres along the Kenyan and Tanzanian coast. *Frontiers in Ecology and Evolution*, *11*, p.67.

   This publication is based on the study findings in Chapter 3

   "Prevalence and Characterisation of Microfibres in Marine Environment Using the Kenyan-Tanzanian Coast as Case Study"

Chapter one

General Introduction: Aquatic Anthropogenic Microfibres and Research Aim

1.1 The Problem: Microfibre Pollution in Aquatic Environments

Marine environments include open and deep-sea oceans (1) and have been the major focus of aquatic anthropogenic microfibre (MF) pollution (2, 3, 4, 5, 6, 7). This is unsurprising as the seas and oceans make up the earth's largest ecosystem and are essential to human existence and life (8). Therefore, efforts to monitor and ensure that it is conserved and used sustainably are paramount (8, 9). The United Nations for example has included the conservation and sustainability of the marine environment in its sustainable development goals (8). However, before the avalanche of studies in aquatic MF pollution, some earlier studies in the 20th century had indicated that MFs were present in the aquatic environment. These 'pioneering' published studies (10, 11) indicated the presence of both synthetic and natural anthropogenic fibres in the aquatic environment. Buchanan's observation was noted in a 1971 publication (11) that during routine examination of suspended matter in the sea, large amounts of synthetic fibres were increasingly found following membrane filtration of water samples collected from the sea areas adjacent to the south of the coast of Northumberland. Nonetheless, Atkins, Jenkins, and Warren (10) had already identified natural fibres in their investigation of suspended materials in sea water also almost two decades before Buchanan's observation. Given their diminutive nature, which render them largely imperceptible to the unaided human eye, they were likely not perceived as posing any risk to the environment. Consequently, there was a lack of concerted efforts towards identifying, measuring, and evaluating the environmental consequences of these anthropogenic fibres at the time.

Paradigm Shift: Macro- to Microparticles

During the period between these 'pioneer studies' (10, 11), plastic manufacture underwent several advancements until its use began to rise, resulting in aquatic plastic pollution (12, 13). This was seen as a major issue because the negative impact was visible unlike the fibres previously observed. Following research into plastic materials polluting the environment, these studies led to the discovery of micro- sized plastics which Thompson et al. later termed "microplastics" (2, 14, 15). Microplastics could be primary or secondary in nature i.e., intentionally produced to be micro sized for specific purposes such as beads found in cosmetics or formed by the breakdown or degradation of larger plastic materials, respectively (16). Furthermore, they may be found in the form of fragments, fibres, pellets/beads, or films (17). Thompson et al. (2) reported in their investigation that fibres i.e. MFs made up a major portion of the microplastics identified which have subsequently led to the avalanche of studies investigating MF pollution either as a domain of its own or in tandem with broader microplastic research (6, 17, 18, 19, 20).

Microfibre Definition in the Context of Pollution Studies

There has been an apparent lack of agreement on the definition of MFs, with some authors restricting the term to synthetic or plastic fibres (21). This is not surprising given the origins of MF study in plastic pollution studies. Furthermore, differences in size specifications have contributed to these disagreements (22). In response to the situation, Liu et al. presents a relatively simple and acceptable definition of microfibre as

> "any natural or artificial fibrous materials of threadlike structure with a diameter less than 50 μm, length ranging from 1 μm to 5 mm, and length to diameter ratio greater than 100" (23)

It is important to state that some authors have argued for a different terminology altogether in order to disassociate the idea of MFs referring only to microplastic (synthetic) fibres. For

instance, some authors have suggested the use of the term fibre microparticle (FMP) (7) whilst others have suggested fragmented fibre (FF) (24). However, the term microfibre (MF) has been used throughout this thesis and follows the definition offered by Lui et al. (23). Furthermore, it is important to differentiate it from MF in the context of textile production. MF in textile production refers specifically to synthetic fibres designed to have a diameter less than 5 µm and denier less than one. Denier is defined as the mass in grams per 9,000 meters of fibre (25, 26).

Adverse Effects of Anthropogenic Microfibres

Anthropogenic MFs have been found on a global scale in water and sediment samples from beaches (27), lakes (28, 29), rivers (30, 31), oceans (4, 5, 32) and even in aquatic biota (33, 34). They are easily transported from one location to another by water or air due to their density (35, 36, 37, 38). It is therefore unsurprising that they have been found in remote areas with scarce human presence such as the Antarctic (37). Because of their ubiquitous nature, MFs pose a significant threat to aquatic biota. Pathogens such as *E. coli, E. faecalis, and P. aeruginosa* can thrive on MFs and persist over time (39). MFs also transport and release chemicals (40, 41). Browne et al. in their 2011 paper (3) addressed the possible impact of MFs as a result of the usage of additives such as dyes and mordants in their manufacture. Other harmful chemicals used during their production include flame retardants like bisphenol A and anti-degradants such as benzotriazole (42, 43). For example, a study conducted on twenty-six clothing samples including baby clothes, found concentrations up to 8.3 mg of benzothiazole in one of the samples (42). Although these were garment pieces (1.5 -2 g cut cloth) rather than single fibres, the inference is that the accumulation of single MFs over time is capable of producing such amounts of chemical substance in the aquatic environment (42). Despite the fact that there is a paucity of studies investigating toxicity at environmental level concentration of this particular chemical, high levels have been linked to a carcinogenic effect (44).

Furthermore, humans may become unwell if they consume contaminated sea food. Pathogens and toxic components present in these MFs may cause illness impacting on human health adversely. It has been suggested that ingestion of these MFs may trigger similar symptoms as in breast and lung cancers, skin lesions, cardiovascular diseases and tumours (45). However, there is a lack of robust studies that accurately assess the risk to human health as a result of ingesting contaminated foods (46) and drinking water (47, 48) including beverages (49, 50). In addition to ingesting MFs, research has shown that inhaling them has a harmful effect on the respiratory system (41, 51, 52). Although synthetic MFs have been found in lungs of humans postmortem (53), similar to studies in mechanism and impact of ingestion of MFs in humans, there is also paucity of studies on impacts of inhalation of environmental level concentration of MFs. Nonetheless, available studies such as one conducted by Van et al. (54) indicated that 16-39 μg/ml and 122 μg/ml of nylon and polyester MFs respectively exposed to human lung organoid had negative impact on the growth and repair of airway organoids. However, there is apparent evidence of the adverse effects of long-term exposure to high level concentrations such as dyspnea, sinusitis, nasal catarrh and decrease in lung functions as observed in individuals with occupation in textile factories (51, 55, 56).

When MFs are ingested by aquatic fauna, laboratory studies have shown that they can cause problems ranging from cellular level to organism level i.e from harmful effect to cells only, within the organism which may not impact significantly on the organism's overall wellbeing to more severe impacts which may lead to death. Examples of these impacts as evidenced in laboratory studies are outlined in Table 1.1.

There is evidence that MFs accumulate more than micro- fragments and beads (57). In a Zebrafish laboratory study (57), MFs accumulated four times the number of fragments and sixteen times more than beads after seven days. The implication being that the sample organism easily egested fragments and beads compared to the MFs. The longer time MFs

were retained in the fish meant that there was greater opportunity of chemical leaching from the MFs and physical damage to internal organs. This could have a negative impact on growth and reproduction, cause inflammation, and disrupt metabolism (6, 57). It is vital to note, however, that fauna's feeding style may influence preference in the intake of MFs. A recent study found that filter feeders preferred MFs while deposit feeders seemed to actively select micro- fragments (58). The reason appeared to be that the feeders preferred what was easily accessible. Another factor that appears to contribute to MF intake by aquatic fauna includes physicochemical properties. In an experiment where Asian clams (*Corbicula fluminea*) were fed polyester, polyester-amide, acrylic, polyamide, rayon and polyvinyl alcohol, it was discovered that polyester MFs had the highest uptake followed by polyester-amide (59). These two MF types had the lowest elastic modulus i.e., they were the softest of all MF types in the experiment. Therefore (60), it appears that ease of ingestion also plays a significant role in MF ingestion by aquatic fauna. Numerous laboratory based studies on MF egestion have been carried out but there appears to be a paucity of information on what influences MF egestion as evidenced in this review (61). One example of how egestion is handled is through regurgitation as evidenced by shrimps fed by different concentrations of polyacrylic MFs (62). Given that regurgitation is a common physiological process among certain aquatic fauna, it follows that the egestion of MFs during digestion may constitute a routine mechanism in such organisms. Accordingly, this phenomenon may enable predictability in the fate of MF egestion, as it appears to follow the natural aspect of their normal digestive process. However, studies need to be carried out to determine if this holds true for different fauna species. MFs can also cause actual skin damage or strangulation in aquatic fauna (63).

Most of the studies carried out to investigate the sources of MF have focused on synthetic fibres (3, 6, 16, 23, 64, 65, 66, 67). Almost a decade ago, Ludwig et al.(68) noted the paucity of data regarding natural fibres in the aquatic MF pollution discourse. Recent studies (30,

69, 70) have begun to opine the same views as Ludwig et. al (68). Nonetheless, it seems that the trend is not changing significantly, or at least not in the anticipated manner, given that both natural and man-made fibres are subject to chemical treatment during the final stages of textile production. Despite the arguments regarding the biodegradability of "natural" MFs, this does not invalidate the fact that they can discharge toxic chemicals like their synthetic counterparts. Nevertheless, it is probable that the likelihood of releasing these chemicals into the aquatic environment increases due to the biodegradability of these MFs after their breakdown (68). A recent study by Detree et al. (71) showed that the natural MFs (cotton and wool) used in their study of impact of MFs on Oysters had more digestive and inflammatory impact than the man-made MFs. The need to therefore investigate natural MFs in aquatic environments cannot be overstated.

Table 1.1: Toxicological Impacts of Microfibres on Aquatic Fauna

Microfibre Type	Concentration	Duration of Exposure	Organism	Toxicological Impact	Authors
Acrylic	10MF/L and 10000MF/L	96 hours	Oysters	Inflammation	Detree et al. (71)
Cotton	3-30 MFs/mL	96 hours	Silverside fish	Behavioural changes	Siddiqui et al. (72)
	3-30 MFs/mL	96 hours	Mysid shrimp	Reduced growth, behavioural changes	Siddiqui et al. (72)
	8 MFs/L and 80 MFs/L	94 days	Mussels	Reduced growth	Walkinshaw et al. (73)
	10MF/L and 10000MF/L	96 hours	Oyster	Impacts enzymatic activities	Detree et al. (71)
Ethylene vinyl acetate	0.03g/pellet	6 weeks	Goldfish	Inflammation of intestine and liver	Jabeen et al. (74)
Lyocell	500 mg/L	48 hours	Brine shrimp	Gut damage	Kim et al. (75)
	1000mg/L and 2000mg/L	48 hours	Waterflea	Mortality, growth inhibition and gut damage	Kim et al. (76)
Nylon	100MFs/mL	24 hours	Copepod	Decrease in feeding activity. Impact on prey selection	Coppock et al. (77)
	50 MFs/mL	6 days	Copepod	Inflammation	Cole et al. (78)
	10MF/L and 10000MF/L	96 hours	Oyster		Detree et al. (71)

Table 1.1: Toxicological Impacts of Microfibres on Aquatic Fauna (continued)

Microfibre Type	Concentration	Duration of Exposure	Organism	Toxicological Impact	Authors
Polyester	3-30 MFs/mL	96 hours	Silverside fish	Reduced growth	Siddiqui et al. (72)
	3-30 MFs/mL	96 hours	Mysid shrimp	Reduced growth	Siddiqui et al. (72)
	500 mg/L	48 hours	Brine shrimp	Gut damage, mortality	Kim et al. (75)
	12.5-100mg/L	48 hours	Waterflea	Mortality	Jemec et al. (79)
	1000mg/L and 2000mg/L	48 hours	Waterflea	Mortality, growth inhibition and gut damage	Kim et al. (76)
	25 MFs/L	5-10 days	American lobster larvae	Mortality	Woods et al. (80)
	31.25-1000µg/L	8 days	Waterflea	Reduced reproduction	Ziajahromi et al.(81)
	25 and 40 MFs/mL	72 hours	Sea cucumber	Lysozymes toxicity	Mohsen et al. (82)
	10MF/L and 10000MF/L	96 hours	Oysters	Inflammation	Detree et al. (71)
	Up to 30 MFs/mL	30 minutes – 9 hours	Blue Mussel	Reduced feeding	Woods et al. (83)
	10-30MFs/mL	48 hours	Waterflea	Increased the toxicity of glyphosate	Zocchi & Sommaruga (84)

Table 1.1: Toxicological Impacts of Microfibres on Aquatic Fauna (continued)

Microfibre Type	Concentration	Duration of Exposure	Organism	Toxicological Impact	Authors
Polypropylene	3-30 MFs/mL	96 hours	Silverside fish	Behavioural changes, reduced growth	Siddiqui et al. (72)
	3-30 MFs/mL	96 hours	Mysid shrimp	Behavioural changes, reduced growth	Siddiqui et al. (72)
	500 mg/L	48 hours	Brine shrimp	Gut damage	Kim et al. (75)
	8 MFs/L and 80 MFs/L	94 days	Mussels	Reduced growth	Walkinshaw et al. (73)
	1000mg/L and 2000mg/L	48 hours	Waterflea	Mortality, growth inhibition and gut damage.	Kim et al. (76)
	> 3 MFs/100L	71 days	Pacific mole crab	Increased mortality and reduced reproduction	Horn et al. (85)
	5 MFs/feeding	8 months	Male Northern lobster	Reduced growth, blood protein and stored lipid	Weldon & Cowie (86)
	50000MFs/mL	3 hours	Grass shrimp	Increased mortality	Gray & Weinstein (87)
	0-1mg/g	4 weeks	Crab	Reduction of food consumption and energy needed for growth.	Watts et al. (88)
	20mg/L	24 hours	Zebrafish	Damage to intestine	Qiao et al. (57)
	0-90 MFs/mL	10 days	Amphipod	Reduced growth	Au et al. (89)

Microfibre Type	Concentration	Duration of Exposure	Organism	Toxicological Impact	Authors
Polyethylene	100mg/L	48 hours	Brine shrimp	Reduced growth	Kokalj et al. (90)
	0-20.000MFs/mL	42 days	Amphipod	Reduced growth	Au et al. (89)
Wool	10MF/L and 10000MF/L	96 hours	Oysters	Decrease in enzymatic activity	Detree et al. (71)

1.2 Types of Microfibres

MFs may be classified broadly into natural, synthetic, or regenerated fibres depending on their origin (Figure 1.1). Natural MFs are fibres that come from either animal, mineral, or a plant source. Animal fibres can be found as hair, fur, or secretions from invertebrates such as the silkworm (91). They are mostly made up of protein, which is a polymer of amino acids (26). Keratin is the most common kind. Minerals, unlike the other two types of natural fibres, are inorganic. Natural fibres of plant origin are usually cellulosic that is, polysaccharide polymers. These fibres occur naturally in plant seeds such as cotton, leaves such as sisal, and stems/bast such as jute (26). Animal fibres, particularly wool, are the second most extensively used natural fibres after cotton. Wool fibres are derived from sheep, goats, and oxen, among other animals (92).

Regenerated fibres are made by chemically processing naturally occurring polymers such as cellulose or protein with the former being more common. Common types include viscose and modal whereas synthetic fibres are made from raw materials such as petroleum and coal (26). The raw materials needed to make regenerated and synthetic fibres are processed into fibre-forming chemicals known as 'spinning dope' (25). The spinning dope is made by converting a solid monomeric substance to a liquid or semiliquid state using a solvent or heat. Synthetic fibres can be produced using a wet, dry, or melt spinning method. Melt spinning is the process whereby a viscous melt of polymer is being extruded through a spinneret containing a number of holes into a chamber, where a blast of cold air or gas is directed on the surface of fibres emanating from the spinneret (93). Following this, the molecules are aligned in a parallel arrangement which enables them to crystallize and orient (94, 95).

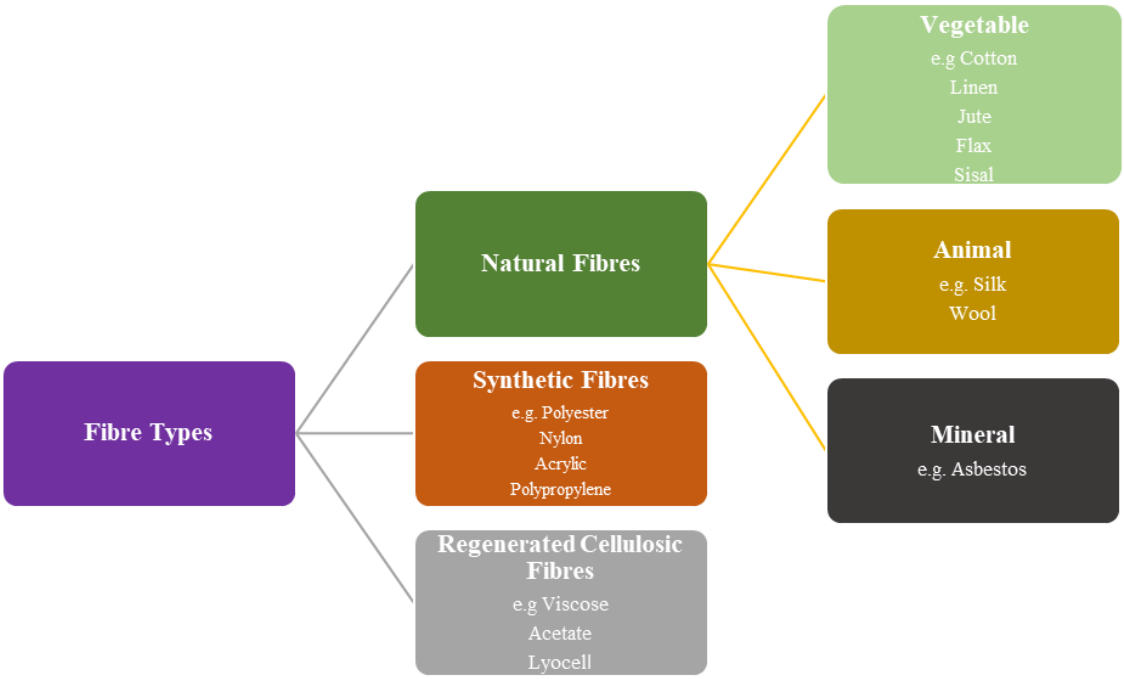


Figure 1.1: Classification of Fibres

The orientation of the polymer chains along the fibre axis, increases its mechanical strength. Examples of fibres formed through melt spinning include polyester, nylon, olefin etc. Wet spinning is the process used for polymers that need to be dissolved in a solvent before spinning. The fibre is caused to precipitate by submerging the spinneret in a chemical bath and then solidify as it comes out e.g., acrylic, lyocell etc. Dry spinning involves fibre formation through a process that transforms a high vapour pressure polymer solution to a solid fibre by controlled fibre evaporation in the spin line (96). Examples include acetate, triacetate etc. The process of extruding a fibre through a spinneret is known as spinning. Spinnerets come in a variety of shapes, resulting in a variety of fibre shapes (97). This feature contributes to their distinguishing characteristics.

1.3 Sources of Microfibres

Textile materials used for both apparel and non-apparel purposes have been identified as sources of MFs to the aquatic environment (6, 66, 70, 98). In 2021, 113 million tonnes of textile fibres were produced globally, with polyester accounting for 61 million tonnes (>50% of total production) as the most manufactured fibre, followed by cotton (99). If current trends

continue, fibre production is estimated to reach 149 million tonnes by 2030 (99). MFs are shed from materials composed of fibres. Shedding occurs from the point of textile production (70) to normal use (100, 101, 102) and laundering (103, 104, 105, 106), including during drying (107, 108).

Textile production involves various stages from fibre formation to finished fabric (Figure 1.2) and MFs are shed at each stage (61, 98, 109, 110). During the first stages of production, the process of extruding fibres through the tiny holes of spinneret could cause fibres to break or shed MFs or in the case of carding for natural fibres. Carding is the process of separating and straightening fibres to prepare them for spinning

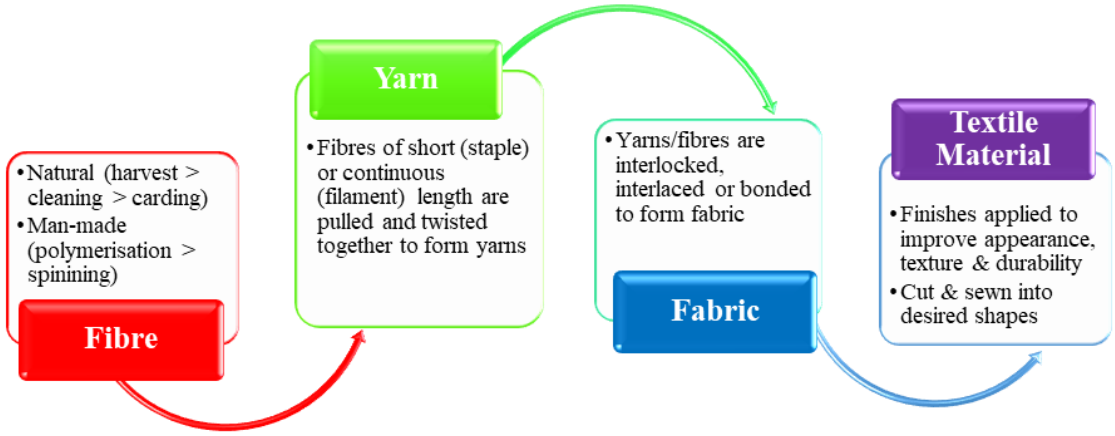
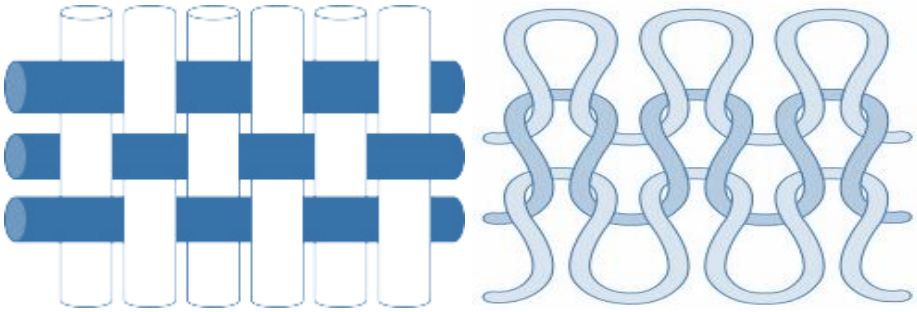


Figure 1.2: Schematic diagram showing the process of fibre production to finished product.

The fibres are passed through a series of carding machines with sharp teeth that comb and align the fibres, removing impurities and creating a consistent web of fibres that may be spun into yarn or other products. The mechanical actions of the combing results also in MF release.

After fibres are formed either naturally or artificially as discussed earlier, they are pulled and twisted through a spinning process into yarns. Fabrics are subsequently produced by

interlacing, interlocking or bonding yarns or fibres through knitted, woven, or non- woven method of assembly. Woven fabrics are made by interlacing two sets of yarn called the weft and warp. They run crosswise and length wise to the fabric respectively (Figure 1.3).



(A) (B)

Figure 1.3: Schematic diagrams of (A) Woven fabric with blue weft and white warp yarns (B) Weft knitted fabric. (Adapted from Houck) (25)

In addition to their use in making garments, woven fabrics are ideal for making curtains and upholstery. Knitted fabrics are made by interlocking a series of yarn loops in a course that run either crosswise (weft knitting) or lengthwise (warp knitting) (25). Since they are generally light weight, warp knitted fabrics are used for making clothing for activity such as in sports. Unlike the first two forms of assembly of yarns, non-woven fabrics are made by bonding fibres together through mechanical, chemical or heat treatment. This type of fabric is mainly used for making hygiene products such as cleaning wipes, medical bandages and car carpets (26). The overall physical, mechanical and chemical actions leading to MF release throughout the production culminate in the disposal of textile products in solid waste and wastewater (98).

Common non-apparel sources of MF include fishing and recreational sailing gears (7, 111, 112). Following a study on the breakdown rate of ropes made from commonly used fibres in fishing gear, it was projected that almost 50,000 tonnes of MFs might be generated each year (113). However, the worrisome number projected in this study is conservative because it does not account for all forms of gear that could potentially release MF into water bodies.

As research into MF pollution expands, more non-apparel sources have been identified, such as facemasks (114, 115) , wet wipes (116, 117, 118) , cigarette butts (65, 119) and fabrics such as polyester and rayon used in reinforcing vehicle tyres (120, 121, 122). Following the Covid-19 outbreak, there was an increase in the use of PPE, particularly facemasks. Although their use helped to reduce transmission, they currently represent a concern due to inadequate disposal practises. A study found 284 abandoned facemasks across a total area of 22,500 m^2 from three beaches within a 15-day period (115). This number was only limited to those that were clearly visible. One can only speculate on how much more is buried in water or soil sediments. In congruence with this study, other authors found that disposable masks were the commonly encountered types in the river investigated especially the KN95 (123). This could be an indicator that consumer's perception of a particular product may influence its availability to environmental pollution i.e., the more consumers use a product, the higher the chances of that product being a pollutant on the long run. In addition to the contribution of facemasks to MF pollution, a recent laboratory study discovered that smoked cigarette filters (SCFs) may release approximately 100 MFs each day (119). The researchers also predicted that this source of MF could contribute up to 0.3 million tonnes of MFs to aquatic habitats each year (119). On the other hand, commercially available wet wipes may be contributing from 11,900 fibres/g to 1,150,000 fibres /g based on a laboratory experiment (118).

1.4 Factors Influencing Microfibre Release

The rate at which fibres are released from a textile material, referred to as its "sheddability", plays a crucial role in determining the presence of MFs in the environment although it is largely unrecognised. Sheddability, which is influenced by several factors such as the fibre type the fabric is comprised of, the type of yarn used (staple or filament), the structure of fabric (knitted, woven, or non-woven), and its texture (smooth or coarse), has a significant impact on the amount of MFs that are released, in addition to the process by which it was

dyed and chemically finished (124). For instance, loosely structured materials such as fleece blankets often shed more fibres than tightly arranged structures like those found in woven fabrics (125). Staple fibres, which refer to fibres of short length, typically exhibit a higher propensity for MF shedding in comparison to filament fibres, which are continuous in length. Nonetheless, an investigation has revealed that a knitted polyester garment composed of filament fibres shed a greater number of MFs than a knit garment comprising staple filament polyester fibres (102). This outcome is surprising, but it may be partly accounted for by the fact that the knit garment made from staple polyester fibres was twisted, while the knit garment made from filament polyester fibres was not. The twisting of staple fibres creates a tensile force that imparts greater durability and reduces the likelihood of fibre breakage, as compared to untwisted fibres. The length of time a garment is in use, or its age, has an impact on the release of MFs. Specifically, newly manufactured garments are more likely to release MFs that have accumulated on their surface or are loosely hanging following the production process (104, 126). However, older, used garments with frayed edges would favour the release of more fibres than new garments with closed edged (127) The frayed edges expose fibres that can be broken through friction and agitation in the washing machine (128). These fibres are also available for fibre-fibre friction leading to breakage and release. Thus, shedding is complex and no one variable is singularly responsible.

Given that textile materials, particularly clothing, are ubiquitous in daily life, they are exposed to various conditions that promote fragmentation and eventual shedding (100, 102). The degree of shedding and subsequent release of MFs from textile materials may be exacerbated by various factors including chemical, physical and mechanical conditions it undergoes (125).

The actions of tumbling, agitation and friction happening within a washing machine's drum result in fragmentation and release of MFs during washing. According to a worldwide survey conducted across more than ten countries, a minimum of 76% of households in each country

reported owning a washing machine (129). Following studies, washing machine has been estimated to release > 1900 fibres/ 4 kg (3) to 6,000,000 per 5kg (130). This goes to show how vital a role the washing machine plays in MF release. A significant amount of research has been carried out to investigative the contribution of washing machine to MF pollution as evidenced in a recent review by Hazlehurst et al. (131). Research on the release of MF by washing machines can be classified into two categories: studies conducted using laboratory simulators and those carried out with actual washing machines. Each of these methods has its advantages. For instance, laboratory simulators allow for easy replication of experiments due to their small size and relatively easy set up. However, their results may not robustly reflect real-life situations, limiting their scope. Conversely, the use of actual washing machines presents an opportunity to replicate real-life situations and generate more realistic data. However, these setups are cost and time-intensive, which may limit the number of experiments that can be conducted.

Various factors exacerbating the shedding of MFs from garments during washing, includes the water volume to load ratio (132), the duration of the wash (133, 134) , the temperature used (131, 135), and the use of fabric care products (126, 136). A study comparing a laboratory wash simulator to real washing machines found that increasing the water volume to load ratio resulted in an increase in the release of MFs during washing (132). The authors proposed that water volume has a greater impact on MF release compared to agitation in washing machines, which may account for the differences in observed levels of MF release between top-loading and front-loading machines. Water volume seems to amplify the effect of agitation. Other studies have indicated that longer wash times are associated with increased MF release. In a recent study by Mahbub and Shams (133), it was found that the amount of MFs released during washing increased from 60.22 ± 13.32 mg/kg to 131.51 ± 21.03 mg/kg when the wash time was increased from 30 to 60 minutes.

There is some conflicting evidence regarding the effect of fabric softeners/conditioners on MF release. Depending on whether the recommended dose by the manufacturer is followed or not, these products may either decrease or increase the amount of MFs released during washing (130). The influence of detergent and fabric conditioner was found to be relatively small in a study (126). Contrary to this observation, De Falco et al. (130) found that detergent increased the amount of fibres released amongst the different fabrics used in their study. Furthermore, they noted that the use of powdered detergent led to the release of more MF than liquid detergent. The increased friction from the granules of powered detergent would favour fibre shedding. For instance, a plain weave polyester released 62 ± 52 MFs per gram of fabric that increased to 1273 ± 177 and 3538 ± 664 when using liquid and powder detergents respectively. Another study found that detergent had no significant influence on MF release (136).

High temperatures have also been observed to increase the number of fibres released when washing (130, 136). For instance Lant et al. found 30% reduction from mean 181.6 ± 87.1 ppm for the 40°C cycle to 129.5 ± 42.9 ppm for the Cold Express cycle (136). Increased temperatures have the potential to induce thermal expansion of the fibres within the fabric, making them expand and loose which is more convenient for the release of MFs during the laundering process. Moreover, elevated temperatures can accelerate the rate of chemical reactions between the detergent and the fabric, which may also promote the shedding of MFs.

In a study of real soiled consumer wash, an inverse relationship was observed between the number of microfibres released and the wash load i.e. the higher the wash load the lower the amounts of microfibres shed (136). Garment made with a blend of polyester/cotton/modal tend to release more fibres (137).The number of MFs may reduce with increasing number of washings depending on the condition of the garment. It was observed that a polyester garment blended with cotton and modal continued shedding and did not appear to reach a

climax rather the cotton fibres continued to shed (137). The hairiness and staple yarns of cotton may explain how the mechanical activities involved in washing would easily break the fibres away from the blend. However Pirc et al. (126) found that the number of fibres released both during washing and drying a polyester fleece blanket decreased with increasing number of washes (126). In contrast, Hernandez et al. (135) did not notice an increase or decrease in MF release with increased number of washing.

Cutting and sewing techniques also lead to release of MFs. A study of two polyester materials comprising of two different sewing techniques indicated that the one with double heated- seal shed less MFs compared to the other sewn with 100% polyester thread (138). Here, the bond holding pieces of fibres together is stronger and therefore not easily loosened.

Regardless of the parameters used in washing, a common theme is the dominance of studies investigating materials composed of synthetic fibres (Figure 1.4). Eight man-made fibre types have been examined including nylon, acrylic, polyester, elastane, viscose, rayon, acetate and polyamide whilst cotton and wool are the natural fibre types studied based on this recent review (131). Blended fabric types include, polyester-cotton, polyester-elastane etc. There is an obvious paucity of data relating to factors influencing the release of natural MFs during garment care using washing machine. In particular, the sheer amount of cotton fibres produced globally (approximately 24.7 million tonnes as at 2021 (99)) and its prevailing presence in aquatic environments (5, 30, 69) necessitates prioritising it in research.

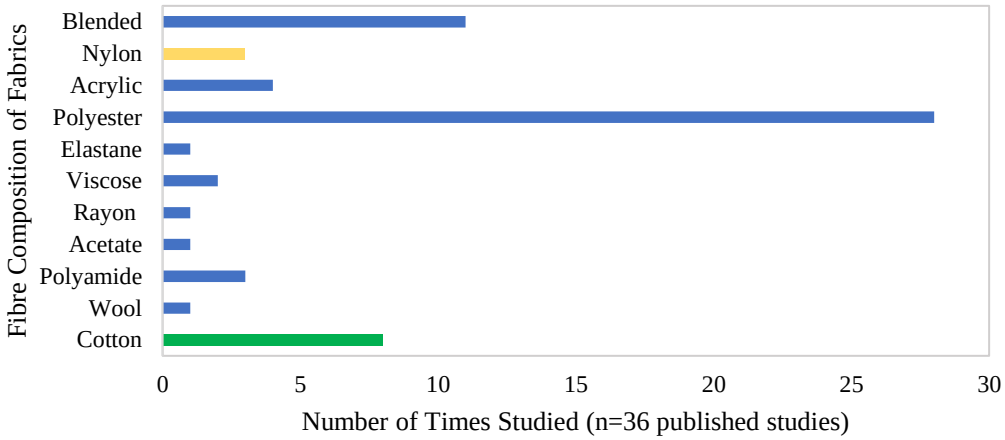


Figure 1.4: Bar graph showing the number of times fabrics composed of different fibre types were studied for MF release during washing (Adapted from review by Hazlehurst et al.(131))

Methodological and reporting inconsistencies make it challenging to compare data from different studies. For example, some estimates report MF release in grams (126, 139), parts per million (108), litres (3) and tonnes (131), making it difficult to make comparisons and truly quantify the scale of MF release.

Inappropriate disposal of clothing items, especially at the end of their useful life, can result in a significant generation of anthropogenic MFs. This often occurs when such items are discarded in landfills, leading to terrestrial pollution that may eventually make its way into aquatic environment through storm or runoff water (61). This point has been exacerbated by the advent of fast fashion. The fast fashion business, defined by its rapid manufacture and marketing of low-cost apparel collections matched with the newest fashion trends, has had a significant impact on this issue (140, 141). This effect is evidenced through several interrelated ways. Notably, fast fashion industry engages in high-volume garment manufacture, the implication being that the availability of more garments leads to more shedding of MFs into the environment. Furthermore, the emphasis on price and convenience leads to the production of lower-quality clothes with increased MF release during use. Consumer culture and a lack of understanding about the environmental repercussions of fast

fashion contribute to the problem's pervasiveness, needing comprehensive mitigation solutions such as sustainable practises and more public education (142, 143).

1.5 Pathways to Microfibre Pollution in the Aquatic Environment

When MFs are released from their sources, they may be transported to the aquatic environment through run offs, precipitation, atmospheric deposition, and effluents from wastewater treatment plants (WWTPs) (6, 16, 144) . However, treated wastewater effluent and air transport have been identified as two major pathways by which they may eventually end up in aquatic environments (20). A study involving 90 samples across different WWTPs in the United States resulted in an estimation of ~50,000 up to nearly 15 million particles with MFs making up almost 60% of the total (145). Effluents generated from laundering garments in washing machine are transported to WWTPs. Due to their large surface area to volume ratio, MFs contained in effluents can escape despite filtration processes in place at WWTPs and eventually accumulate in fresh and marine water bodies (23, 146). The earliest published study showed that effluent from washing machine may contain >1900 fibres per wash when a polyester blanket, fleece or shirt is washed (3). Since this publication, other studies have found varying MF concentrations in washing machine effluent (see review article (147)). For instance, effluent from washing a 6kg load of synthetic fabric may contain between 137,951–728,789 fibres per wash (104) whilst annual effluent from washing machine may contain up to 2.23×10^5 and 9.73×10^5 of polyester and cotton MFs respectively (105). Elsewhere, it is estimated that 17,167 billion to 2,602,080 trillion MFs are released through domestic washing machines in the UK and this evidences an increasing trend of MFs release with more recent studies (131).

Different proposals have been made for mitigating the problems arising from effluents. For example, packed bed microfiltration (PBMF) was used to filter effluents and drinking water from different locations in one of the most polluted cities in India and was shown to have up to 90% effectiveness in reducing MF pollution (47). PBMF is a process that filters out small

particles and impurities from a liquid using a bed of small particles. The liquid flows through the bed of particles and as it does, the particles trap the impurities and allow the filtered liquid to pass through. So simply put, PBMF is a 'giant' filter. When the height of the sand bed was increased, the ability to filter the MFs greatly improved. The spaces between the fibres became more tangled, which made it harder for the fibres to move through. This increased the time that the fibres stayed within the PBMF system thereby increasing the efficiency of the system. Although the authors argue for its cost effectiveness and simplicity, they did not state how the sand bed would be disposed of as they noted that it had to be changed periodically. This could result in the MF pollution of another environment if not properly disposed. Other current proposals of mitigation processes include bio-based finishes, surface modification (67) and alkali treatment of polyester (148). Previous recommendations include the use of products that capture MFs during washing process such as washing bags (149), Cora balls (150), Lint LUV-R (150) and use of filters with finer mesh (108). It is obvious that no one method would be sufficient at the short term and would require individual to industrial level response in addition to government policies (146). However, the most promising long-term solution is to address the source of the MFs i.e. the material shedding the MFs. Efforts need to be channelled towards manufacturing fabrics that do not readily shed (20, 61, 104, 151).

MFs apart from being transported through effluents when released from their sources may be suspended in and transported by air (20, 23, 102). Studies in this area include investigating MFs suspended in air which is mainly done by vacuum filtration and MF fallout from air (atmospheric deposition) done via bulk deposition samples (69, 152, 153). MFs in the air can travel between 10 to 1000 km (154). Due to MFs being able to suspend in the air and travel long distances, they could eventually deposit in areas far from their source (154). This would in part explain why areas with no tangible anthropogenic activities are still polluted by MFs. At the same time, some researchers have suggested that atmospheric deposition

could be a significant contributing factor to the accumulation of MFs in wastewater effluent (69, 155). The implication being that, if MFs in the air can be controlled, it would invariably lead to reduction in MF concentration in wastewater effluent. Factors influencing air transport of MFs include a combination of fibre morphology and environmental condition such as direction and speed of wind (69, 156).

As this involves direct discharge into the air, it is expected that MF levels in populated regions will be high. Urban areas have been evidenced to have higher atmospheric deposition of MFs compared to sub-urban (155) and rural areas (20). A study of two cities in the United Kingdom showed that MFs were more numerous in atmospheric deposition than in treated wastewater effluent (20). More people wearing garments translates to more fibres releasing MFs. Normal garment wear contributes its quota to atmospheric MF concentrations (101, 102). This abundance of atmospheric MF over MFs in treated wastewater effluent should not be surprising as measures have been put in place to capture MFs in WWTP unlike the absence of mitigation in place for MFs in the air. It also confirms that mitigation actions relating to WWTP are effective.

Unlike the washing machine effluent that contribute to MF concentration in WWTPs, electric clothes dryers have been found to contribute significantly to atmospheric MF concentration. Like studies in MF release through laundering with washing machine, various factors can influence how much MFs is released into the air. For instance, Tao et al. (157) found a positive correlation between the mass of polyester load and number of MFs released into the air compared to the cotton load dried (157). The more the mass of polyester clothing being dried, the higher the number of MFs released. No apparent relation was found for the cotton load. As the fabric structures of the cloth items were not provided, it's not clear the reason for this interaction.

However, the details provided for the cloth items showed that 9/12 of the polyester items were either pants or shorts contrary to the cotton items which had 1/10 pant with the rest being T-shirts. Friction within the items with smaller surface area may have favoured MF release. In all, it appears that the two most important factors influencing MF release from dryers are the garment's sheddability and nature of MF capturing device within the dryer (107, 158).

Unlike research with washing machines, the role of the electric cloth dryer is still in its infancy. Unfortunately, it appears that lessons are not being learnt as the same trend of focus on synthetic fibres is evident here (107, 133). The need to address this knowledge gap is pertinent. Studies have shown the dominance of natural MFs over their synthetic counterparts in air (38, 69). The presence of MFs in indoor and outdoor air (153, 155) should raise health and safety concern and therefore drive research geared towards identifying and quantifying the array of MF in the air (159). There is indication that MFs in indoor are more abundant than outdoor air (153) and also some MFs are more abundant in indoor air as opposed to the outdoors (152). For example, polyester was found to be more abundant indoors whilst acrylic outdoor, reflecting the abundance of materials within these environments that are composed of these fibre types. As MFs have been found to be more abundant indoor, the role cloth dryers play ought to be investigated. A review of published literature on atmospheric deposition also indicates that notable studies from the continents of Africa and South America are lacking (38).

1.6 Prevalence of Microfibre in Marine and Freshwater Environments

Following the release of MFs from their sources, they eventually end up in the aquatic environments through any of pathways described in the previous section. The prevalence of MFs in the marine environment has been extensively studied compared to other aquatic environments (6, 17, 18, 19, 160). In a review published in 2021, the authors found that about 60% of studies reviewed examined marine environment (6). MFs have been found on

marine water surfaces (5, 161), sub-surface, sediments (162, 163) and biota (34, 162). Arguably, more population/human activity appears to correlate with more MFs abundance in marine waters (3, 160). However, a study found that higher concentration of MFs were present in open ocean surface i.e. areas without human population compared to coastal samples (164). This is a result of MFs being transported from coastal areas (area of anthropogenic activities) to open sea through ocean currents (37, 164). It also demonstrates long residency time of MFs and the ease of movement by air.

These authors who investigated five major oceanic basins also found that MFs made up 91% of micro pollutants in samples recovered (164). It further indicated that MFs in marine environments seemed to reflect global production of textile fibres (164) i.e. the relative abundance of the various MFs found in their samples corresponded with the percentage of the various fibre type produced globally in the order of polyester > cotton > others fibre types. However, Suaria et al. (5) found a contrary evidence when they sampled 916 water samples from six oceanic basins and analysed almost 2000 MFs unlike the previous studied that analysed 113 using μFTIR (164). They found 79.5% of the examined MFs to be cellulosic and this was congruent with few other studies which had investigated other aspects of marine environment and found the dominance of natural MFs such as in seafloor sediment (165). It is estimated that the top meter of the world's oceans contains 9×10^4 to 38×10^4 metric tons of fibres.

Freshwater systems are the main transporters of MFs to marine environment (144). Compared to marine studies, less studies have been done reporting the prevalence of MFs in these environments. In a fairly recent review, 23% of reviewed studies were on freshwater (6). For example in the UK, 32%, 52% and 16% of studies have been done on freshwater, marine and estuaries respectively (166). MFs have been found in freshwater fauna (167), freshwater in remote locations (28), surface water (31), and sediment of freshwater (168). Untreated storm water is one of the main pathways of MFs to freshwater environments (29).

Factors impacting MF prevalence include physical conditions such as direction of the wind and proximity of anthropogenic activities (168).

According to the review on MF pollution conducted by Athey and Erdle (6), majority of the studies carried out on both marine and freshwater environments did not account for the presence of natural MFs (Figure 1.5). In cases where these natural MFs were noted, the

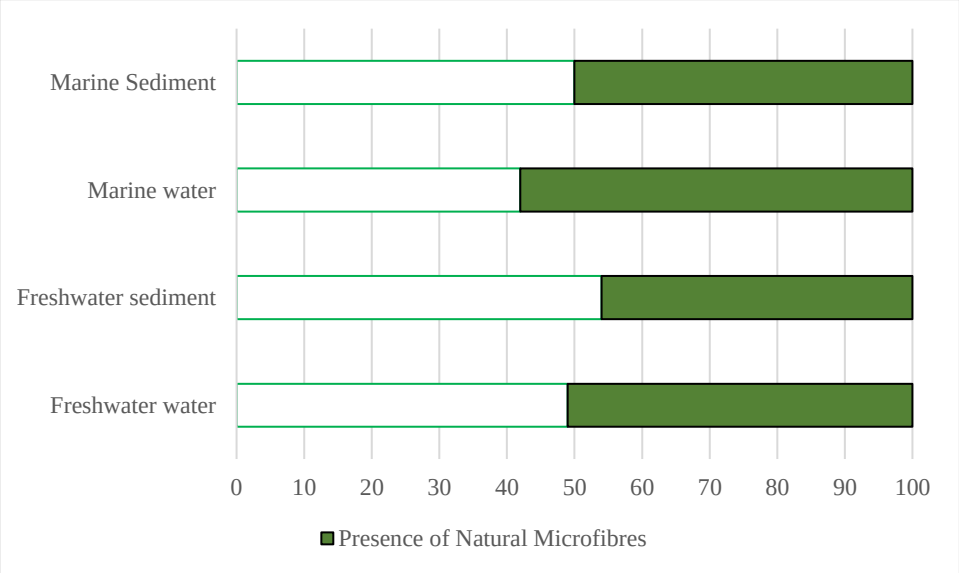


Figure 1.5: Graph showing the proportion of studies that have noted the presence of natural MFs in their findings. (Adapted from Athey and Erdle (6))

authors did not always indicate the relative proportion of these MFs. The studies not accounting for the presence of natural MFs lead to underestimation of the MF pollution

problem. As the ecotoxicity of these chemically treated natural MFs are yet unknown, the extent of harm which they are potentially able to have would not be adequately understood in the absence of their concentration data in the aquatic environment.

1.7 Microfibre Pollution in Africa

Although studies have been carried out globally, the majority of studied environments are in the Asian continent, particularly China (6, 110, 161, 169, 170, 171, 172), whilst the African continent has been understudied in comparison (6, 173).

A factor impacting on the accuracy of global data is the fact that some regions, such as in Africa, are grossly underrepresented due to a paucity of studies. Africa is the second largest continent in the world with an estimated population of over 1.4 billion people (174). The fact that anthropogenic activities directly contribute significantly to MF pollution, in particular the ways in which textile materials are used, laundered, and dried should make the African continent a hotspot for MF pollution study. In particular, the mode of washing clothes by hand which is common practice in Africa and other parts of the global south (175, 176). Hand washing of clothes usually involves discarding wash effluents directly on the ground or into water bodies especially when washing around lakes and rivers. The concentration of MF in these areas is expected to differ from places where washing effluents from washing machines are first treated in WWTP before they reach marine and freshwater bodies. Poor/insufficient wastewater management continues to plague the African continent despite improvements, thereby providing the perfect scenario to study the factors contributing to MF pollution (177). This is a vital aspect as wastewater treatment plants have been shown to be a significant pathway for the transport of MFs to aquatic bodies as earlier discussed. Another point worthy of note is the large influx of second-hand materials especially clothes and shoes from Western countries (178, 179, 180, 181). The poor or lack of sufficient waste management protocols implies that a significant number of these items end up discarded inappropriately and as they breakdown due to the impact of environmental create an avalanche of MFs polluting the continent.

A review published in 2021 found only three studies which had published findings on aquatic anthropogenic MFs on the African continent thereby making it the least studied continent on the planet at the time (6) although this number has now increased. Two of the three studies reviewed were in marine environments. However, none of these characterised natural MFs as the focus of the study was on microplastics. One other study in Guinea-Bissau, West

Africa, omitted from the review, specifically investigated MFs (182) found in coastal sediment and invertebrates. Although they noted the presence of cellulose and protein based MFs, no further investigation was carried out to determine their generic types.

The paucity of studies is unsurprising as MF pollution research mostly require the use of equipment that are sophisticated, and often expensive (6, 183) and require high levels of skill and expertise. Consequently, Alimi, Fadare and Okoffo (2021) (184) recommended that African researchers collaborate with international laboratories as they acknowledge the cost intensive nature of these studies. They observed that more than half of the studies they reviewed depended on identifying MFs visually due to lack or insufficient instrumentation. In addition to collaboration, development of instruments that are simple and easy to use should be encouraged. However, the goal would not be to replace robust traditional methods but to provide alternatives in their absence.

This also provides an avenue to get local communities involved as they would not require expertise but little training. The added advantages of involving locals would mean faster data collection and coverage of a wider geographical area in a relatively short time. This in turn would address the issues of education and awareness which have been identified as major factors that need to be prioritised in Africa to ensure its contribution to the resolution of the global MF pollution problem (185, 186). For example, the Flipflopi project; a circular economy with the world's first recycled plastic sailing dhow which started in 2016 has been exemplary in addressing pollution issues. In 2019, the Flipflopi's sailing expeditions across regions in East Africa began and has been instrumental in raising awareness of aquatic pollution, plastic in particular through the training and education of local citizens (187).

1.8 The Invaluable Role of Citizen scientists

The worldwide scope of contamination caused by MFs has been confirmed and its detrimental impact on the aquatic environment is indisputable. To develop efficient remedies, comprehensive data gathering, and analysis is necessary. Nevertheless, given the amount of work involved, and the limited number of researchers available, the implementation of viable solutions within a reasonable timeframe may not be feasible, necessitating the participation of additional personnel. Encouragingly, citizen scientists have shown significant promise in pollution research thus far, playing an invaluable role. (188, 189).

Citizen scientists have been defined by Cooper as

> "people exercising their rights and responsibilities to participate in collective scientific endeavours through different hobbies or concerns, not necessarily through their professions" (190).

One of the most extensive data collected for the investigation of MF pollution across five major oceanic basins was made possible through the help of citizen scientists(164). In 2019, a group of volunteers undertook the task of collecting sediment samples from 68 locations within the Ottawa river watershed with the aim of assessing the concentration of micro-pollutants. The analysis of the samples revealed that the shorelines might not be an effective sink for microplastics which contrasts with previous beliefs. This illustrates the potential for citizen science in generating a significant amount of data. Nonetheless, certain challenges such as contamination, mislabelling, and inadequate information have been identified, which could impede the accuracy and reliability of the results. These challenges can be mitigated by utilizing field blanks (191), taking note of clothes worn (189) and giving specific instructions through training (191). The concept of citizen science is one of the effective ways of communicating the problem of MF pollution to the general public whilst gathering data that would help in proffering solution to the problem (192).

1.9 Interdisciplinary Approach to Research in Microfibre Pollution

Research in aquatic MF pollution has grown quite rapidly over the years and has been described as a highly complex and multifaceted environmental problem (193). The authors suggest that to address gaps in knowledge related to various aspects of this problem, an interdisciplinary and reflective approach to research is essential. Key gaps in knowledge include how to minimize or control contamination and develop effective methods to collect and identify MFs. Consequently, any discipline that can contribute to solutions in these areas would provide profitable collaboration (194, 195, 196).

Forensic science is a branch of study that utilises scientific processes for the analysis of evidence recovered in relation to an alleged criminal activity for legal purposes. One of such evidence types that is analysed is textile fibres. Textile fibres have been utilized in forensic investigation for several decades to solve criminal cases due to their tendency to shed easily from textile materials, to transfer between surfaces and through the air (101, 197). This enables them to be used to link people, objects, and environments, making them a valuable source of evidence in criminal investigations. The collection, recovery, examination, and analysis of fibre are subject to rigorous scrutiny to guarantee the reliability and robustness of the evidence presented in a court of law (60, 91, 198). The integrity of the evidence is preserved, and contamination is minimized through the adoption of strict examination protocols. Standards such as ISO 9000, ISO 17000, 17020 and 17025 are undertaken in these examinations (199). The examination process involves the use of appropriate PPE and examinations are carried out within minimal contamination.

The differences in fibre type and dye stuff they are made of, are essential components in distinguishing ostensibly similar fibres. Non-destructive microscopic techniques are prioritized over more complex and/or destructive methods. Robust identification procedures primarily using microscopic techniques are used to identify accurately and quickly natural or regenerated cellulosic fibres. Synthetic fibres on the other hand are classified using

polarised light microscopy (PLM) whilst μFTIR is used to determine exact polymer information (200, 201). The main difference between these two techniques is that the former primarily utilises morphological and optical characteristics of the fibres whereas the latter provides information regarding molecular structure of the fibre, including its functional groups and chemical composition. (This is further discussed in Chapter 2). The application of well-established forensic science processes to environmental studies involving MFs will produce rapid, accurate, and reliable results by minimizing contamination and ensuring the accurate identification of all fibre types. Following established processes in forensic fibre examination, MF pollution research would benefit in three key areas namely, fibre recovery fibre processing (sample preparation) and fibre identification (Figure 1.6).

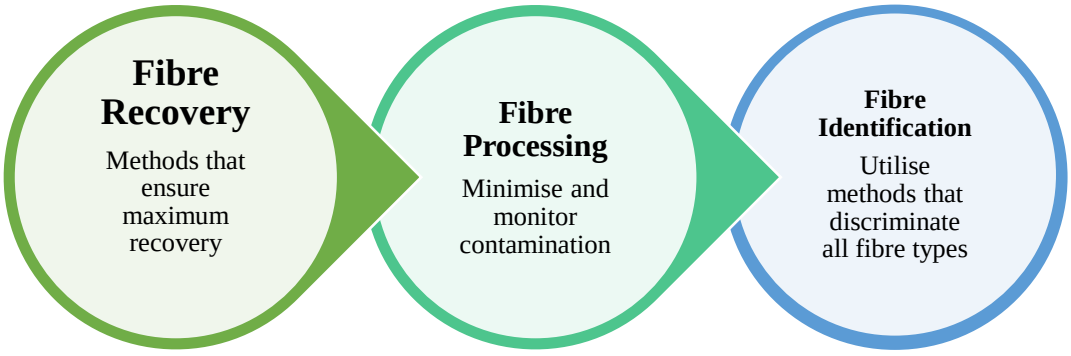


Figure 1.6: Illustration of key points of adaptation from forensic fibre examination

However, it is important to note that choice of sampling method maybe influenced by the nature of the area to be sampled. For example, a pump was chosen in a study due to the presence of narrow and shallow bays (202). On the other hand, cross-sectional river flow affects MF distribution (191) and this may indirectly impact MF quantification. Cross-sectional flow in a river refers to how water moves horizontally across the river channel. The

velocity and direction of the cross-sectional flow can influence the distribution of MFs in the water. For example, if the flow is greater on one side of the river channel as opposed to the other, higher concentration of MFs is expected from the area of weaker flow as MFs are not being transported rapidly increasing their retention time. Therefore, using a method that samples either of the opposites may not give the accurate concentration. It could either be overestimated or underestimated. Alternatively, if the cross-sectional flow across the river channel is somewhat uniform, MFs may be more uniformly distributed within the water. The implication being that method used in sampling may not impact data on MF concentration in the sample area. Sampling method may impact on MF quantification based on the relationship between the sample volume and filter size of sampling device. For example, Forrest et al. recorded low amounts of MFs. The sampling process involved filtering 100 L of river water using a 4 L bottle through 100 µm filter mesh. Water was poured through the filter multiple times, which may have resulted in loss of MFs through the filter pores (191).

It is therefore important to carefully consider and adopt methods that offer the most opportunities for capturing MFs. In forensic fibre examination for instance, recovery of fibres involves methods that ensures all relevant samples are recovered and retained (199). This is primarily done by use of adhesive tape, but other methods include handpicking, brushing, combing etc. depending on the surface where fibres are to be recovered from (91). Tape lifting is the action that involves applying clear adhesive tapes to a surface to gather any fibres that may be present in surface debris. The tape is then secured and kept intact by mounting on a transparent sheet or folding them back on themselves (91, 199). This ensures that fibres are efficiently recovered and retained. The lesson to be adapted at this stage in terms of aquatic MF study is that methods that more efficiently capture MFs are to be prioritised. This translates to using devices with fine pores to prevent or minimise fibre lose.

Following efficient recovery of fibres, forensic process involving the processing of fibre for analysis ensures that fibre is not damaged. This is important as fibre identification and

characterisation is dependent on having the discriminatory features intact. However, current practices in MF pollution research does not always prioritise this. Because organic materials may be present in samples, certain chemicals are introduced to eliminate them. Commonly used ones include NaOH, sodium dodecyl sulfate (202) HCl (203) hydrogen peroxide (204) nitric acid solution (205) or KOH and may be used at different concentrations depending on the sample e.g. 10% KOH (206). However, a study has found that these solutions may cause some level of damage to MFs. It found that Fenton's reagent and HCl caused physical damage in polyamide MFs whilst NaOH and KOH caused both chemical and physical deterioration to almost all polymer used in the study (207). The implication of this being that these MFs may be misidentified resulting in inaccurate data. It is vital to note though, that certain conditions aggravate this damage such as high temperatures or prolong treatment duration. Therefore, it is important to prioritise techniques that preserve the integrity of MFs to be analysed. This would involve research into determining levels of concentrations of these chemicals that are not harmful to samples or completely finding alternatives.

Finally, identification of MFs have routinely involved the use of µFTIR and Raman spectroscopy in aquatic pollution studies as captured in these reviews (6, 208). Both of these techniques are capable of providing information about a fibre's chemical and molecular structure. The major difference being that µFTIR is more useful for the identification of functional groups, such as carbonyls, hydroxyls, and amines, and can provide information about the presence of specific chemical bonds. Raman spectroscopy on the other hand is better suited for providing information about the molecular structure and the vibrational modes of specific chemical bonds. Further information about how these two functions is explained in chapter 2. Beyond the fact that some authors have now referred to the use of these techniques as "laborious" and advocating for more accurate and "high-throughput" techniques (166), is the limitation in discriminating between cellulosic MFs (209). The implication being that when µFTIR is used for identifying cellulosic fibres, the accuracy of

the data may not be reliable. For instance, Frias et al. (210) identified rayon as the most prevalent MF in the samples they analysed. This is contrary to most of the published literature on aquatic MF pollution. The probability that most of these may be cotton cannot be ruled out. Furthermore, identification using µFTIR involves sample preparation that is semi-destructive and therefore not ideal for initial analysis.

1.10 Research Aims and Objectives

Evidently, considerable amount of studies have been carried with the purpose of identifying and quantifying MFs in marine and freshwater environments. However, the emphasis on synthetic fibres is apparently limiting available data as the array of MFs polluting the environments are not adequately captured. It is therefore paramount to formulate research strategies that can account for all MF types. The need to target areas with paucity of research cannot be overemphasized if solution will be proffered on a global scale. To this end, the thesis presented here aims to use a forensic approach to provide data on relative amount of all MF types present in marine and freshwater using the understudied continent of Africa as case study. Furthermore, investigate the sheddability of the fabrics influencing the pollution.

To accomplish these, the following objectives have been set.

1. Critically explore MF pollution studies in aquatic environments and identify limitations to currently employed methods in MF pollution research.
2. Evaluate forensic fibre examination and identify key practices that would be useful in MF pollution research.
3. Employ identification methods used in forensic examination of fibres to examine samples recovered from marine and freshwater samples in Africa.
4. Compare the abundance and distribution of MFs present in African freshwater and marine environments.
5. Determine the efficiency of common methodologies and subsequent accuracy of data.

6. Evaluate parameters that influence the amount MF released through tumble drying.

Chapter Two

Investigating the Case of Microfibre Pollution through Forensic Fibre Examination

2.0 Introduction

Since the mid-twentieth century, forensic examination of textile fibres has formed part of routine analysis carried out in laboratories (198). Textile fibres are ubiquitous, which means they may be found everywhere - in clothing, sofas, carpets, curtains, and chairs - and their diversity discriminated (197, 211). Even though textile materials are usually mass produced, textile fibres, based on these, play an important role in forensic investigations. As a result, forensic examination of fibres employs robust and quality-assured processes to provide the level of transparency and accountability required in evidence presentation at law courts (212, 213). As highlighted in chapter one, the forensic examination process prioritises non-destructive techniques. Furthermore,

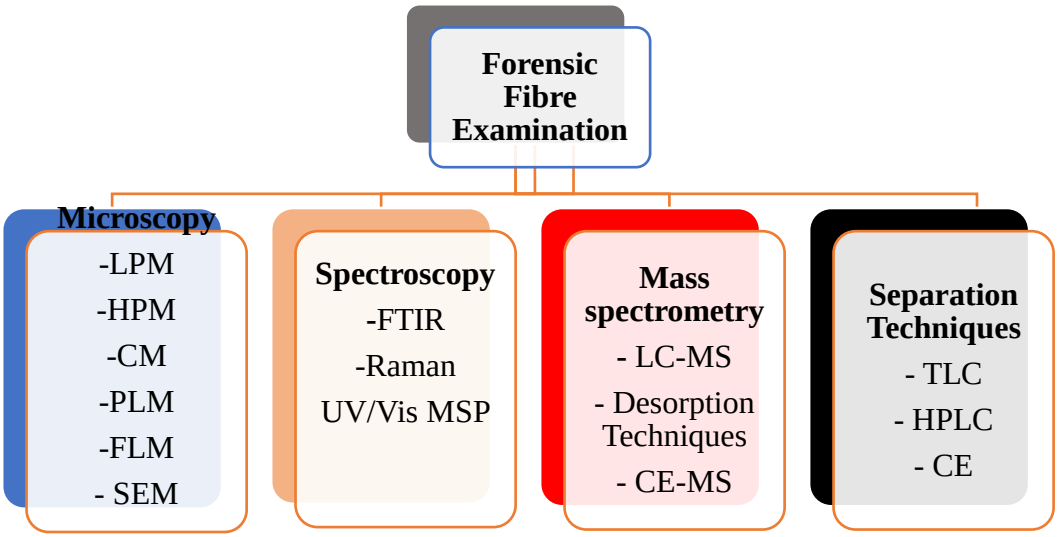


Figure 2.1: Techniques used in forensic fibre examination (L-R, non-destructive to destructive effect on fibre sample)

because its output must be timely, it ensures that procedures that are broad and quick are used before those that are more specific and complex. A generalised sequence for forensic examination of fibres is shown in Figure 2.1. It is important to note that

techniques listed in Figure 2.1 that are not relevant to this research and MF pollution are beyond the scope of this discussion and thus have been omitted.

MF pollution studies seek to identify, characterise, and quantify MFs in a particular environment to determine the probable sources and potential treat, as a result, make informed recommendations for solutions (61, 66, 214). A review of published literature from 2011-2020 showed that FTIR and Raman spectroscopy are the two most commonly used instruments in MF identification (6) with FTIR being used the most (Figure 2.2). Studies published after this review indicate that the trend is likely unchanged (215, 216, 217).

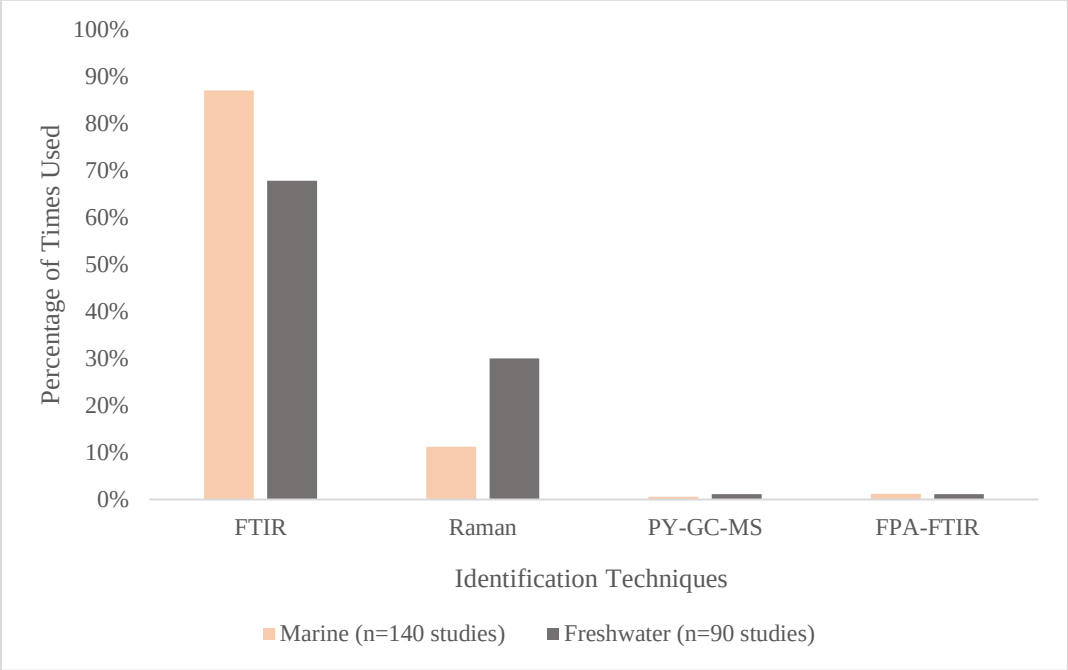


Figure 2.2: Graph showing the frequency of use of FTIR, Raman, Pyrolysis–gas chromatography–mass spectrometry (PY-GC-MS) and Focal plan array-FTIR (FPA-FTIR) in MF identification. (Adapted from Athey and Erdle(6))

Although these instruments have aided in identification of MFs, their use as first point of call in fibre identification is undermined by 1) not being cost effective as they are expensive to purchase (217) 2) time consuming (166), 3) reliance on identification

through spectral libraries (218) and 4) potential misidentification when the polymer is cellulose based (209).

2.1 Forensic Fibre Examination

Following an alleged crime, forensic investigations attempt to recover transferred fibres from surfaces whereby fibres are thought to have been transferred. Typical surfaces include the clothing of the victim and assailant (219, 220), naked bodies (221, 222, 223), the environment of an alleged criminal activity, such as a vehicle (224) or weapons (225). Fibre recovery techniques are adapted depending on the type of surface e.g. for large surface areas, adhesive tape is usually used, where for small, hard surfaces where adhesive tape could potentially destroy other evidence types, individual fibres will be removed by hand, with the aid of a microscope so they can be seen, using a pair of forceps transferred to an adhesive tape or directly to a microscope slide (226).

The examination process begins once the fibres have been retrieved from a surface. The purpose of any inspection is to distinguish one fibre from another. When two fibres cannot be distinguished and all techniques have been exhausted, it must be acknowledged that the two fibres may have come from the same source. The process ends after a fibre has been thoroughly characterised or discriminated. Each stage of investigation is usually more discriminating, time-consuming, and more specific than the one before it. As a result, the analytical procedure frequently begins with microscopic examination before moving on to complex instrumental approaches (201).

2.2 Discrimination of Fibres

Discrimination of fibres is based on differences resulting from their origin and production processes, morphological, optical, and chemical attributes (26, 200, 201). Synthetic fibres are manufactured by different techniques, resulting in varying cross-sectional shapes and diameters, which are employed as discriminatory criteria (200) (Figure 2.3). Morphological features such as thickness, diameter, cross-sectional shape,

and the inclusion of pigments and/or delustrant particles (Figure 2.4) are used to distinguish between natural and synthetic fibres, and discriminate one fibre from another (227, 228). For natural fibres, the maturity of the fibre source can also influence morphological traits. Cotton fibres, for example, have a cross-sectional form that varies from almost round in mature fibres to flattened in immature fibres. They are also characterised by ribbon-like twists (Figure 2.5). The main morphological parameters that may be examined and compared on wool are diameter, scale thickness, prominence, and count (Figure 2.6). These characteristics in turn determine what analytical techniques can be used to examine them (91, 229, 230).

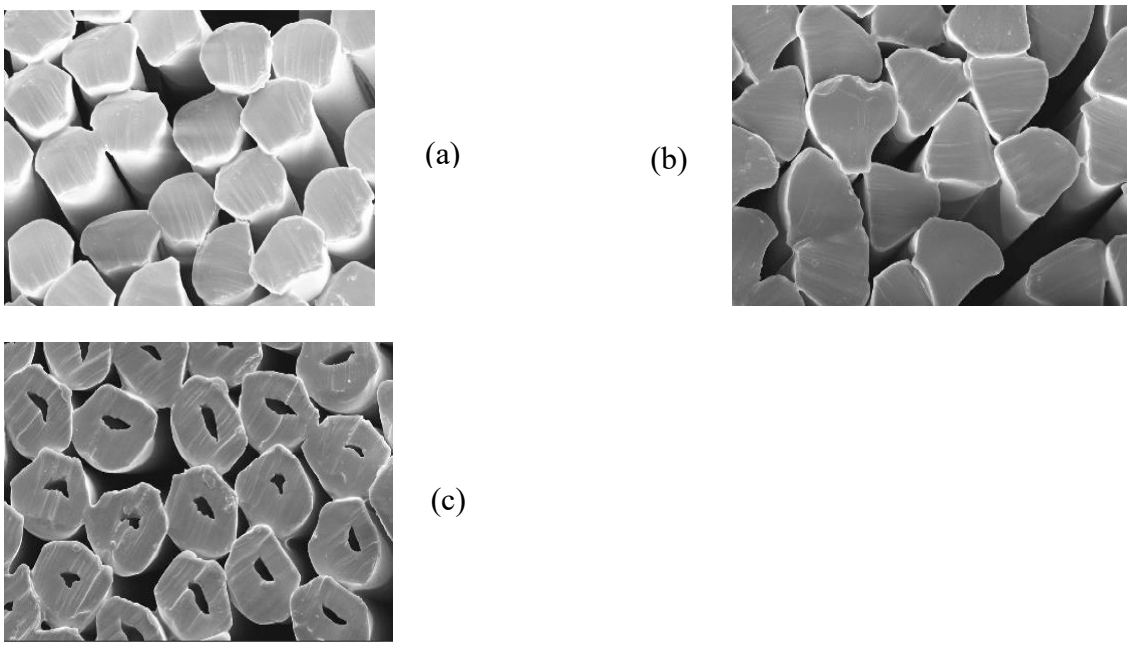
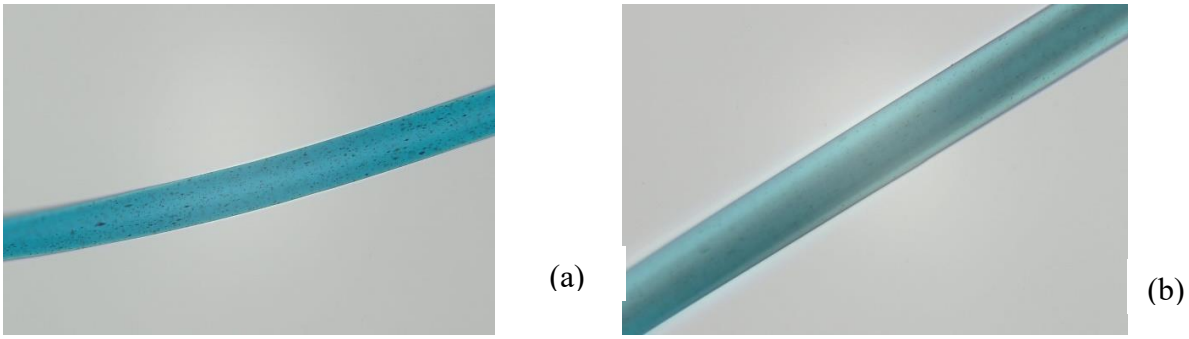
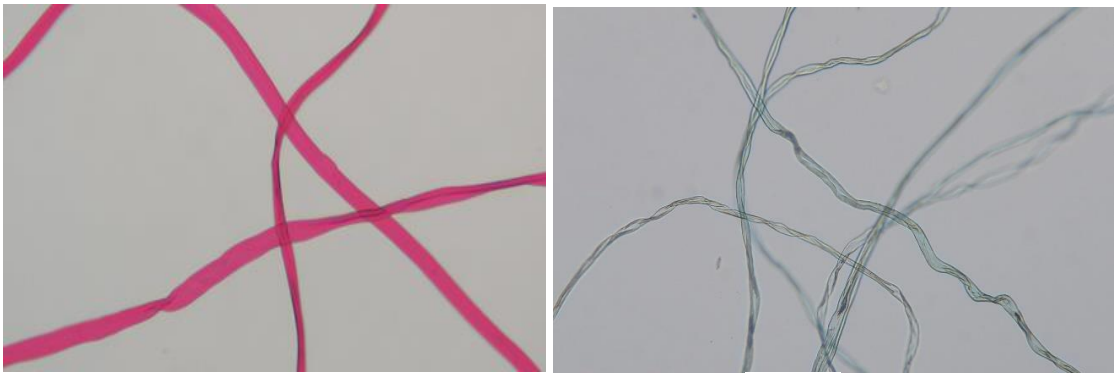


Figure 2.3: Cross sectional images of polyester fibres (a) Round (b)Trilobal (c)Hollow (231)



(a) (b)

Figure 2.4: Images of (a) Delustred acrylic fibre (b) Featureless polyester fibre (x400 magnification).



(a) (b)

Figure 2.5: Ribbon-like features of (a) Red and (b) Blue cotton fibre (x100 magnification).

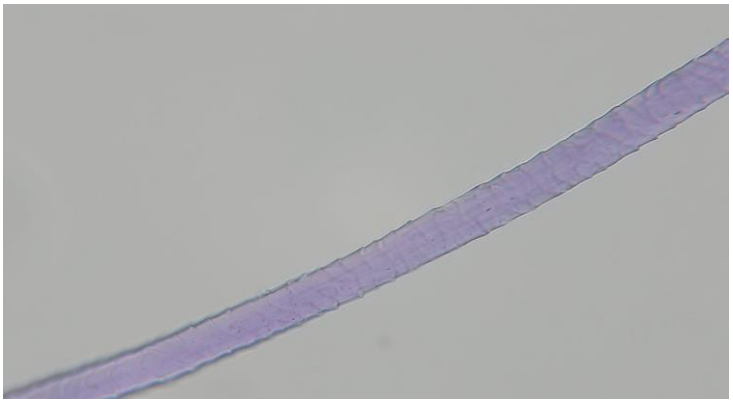


Figure 2.6: Image showing scale-like features on a purple wool fibre.

The techniques utilised in examination of fibres in a forensic science laboratory are presented in the following sections of this chapter. Because they have been proven to be

reliable in forensic research, the purpose is to highlight their advantages in order to inform their best use in MF pollution studies.

2.3 Microscopic Technique

Low Power Microscopy (LPM)

Low power microscopes (LPMs) are usually the first instruments used during fibre examination (Figure 2.7). Nowadays LPMs are stereomicroscopes that consist of two lenses and mainly use light reflected from the surface of the fibre rather than transmitted through it (200, 201, 228). The microscope uses two separate optical paths consisting of two objectives and eyepieces to provide slightly different viewing angles to the left and right eye. The space between the specimen and objective lens and large field of view makes this instrument of choice for performing preliminary examinations of searching and recovery of fibre from adhesive tapings (or tapelifts) at magnifications between x4-x100. Screening for specific target fibres, such as a blue cotton (229) or a blue polyester (232) is relatively quick. Under low magnification, fibres can be broadly discriminated by their colour, shape and size (233). Experienced fibre scientists at this point may be able to generically classify a fibre as being natural or synthetic. Certain fibre types such as cotton can be specifically identified at this stage due to their unique morphology. Whilst fibres may appear to be visually similar to one another and to the target fibre, they could have different morphological characteristics not evident at low magnifications and / or be of a different shade of colour. For this reason the next step in the examination process requires the fibres to be removed from the medium it is on and individually mounted to a microscope slide to allow for more detailed examination (200).

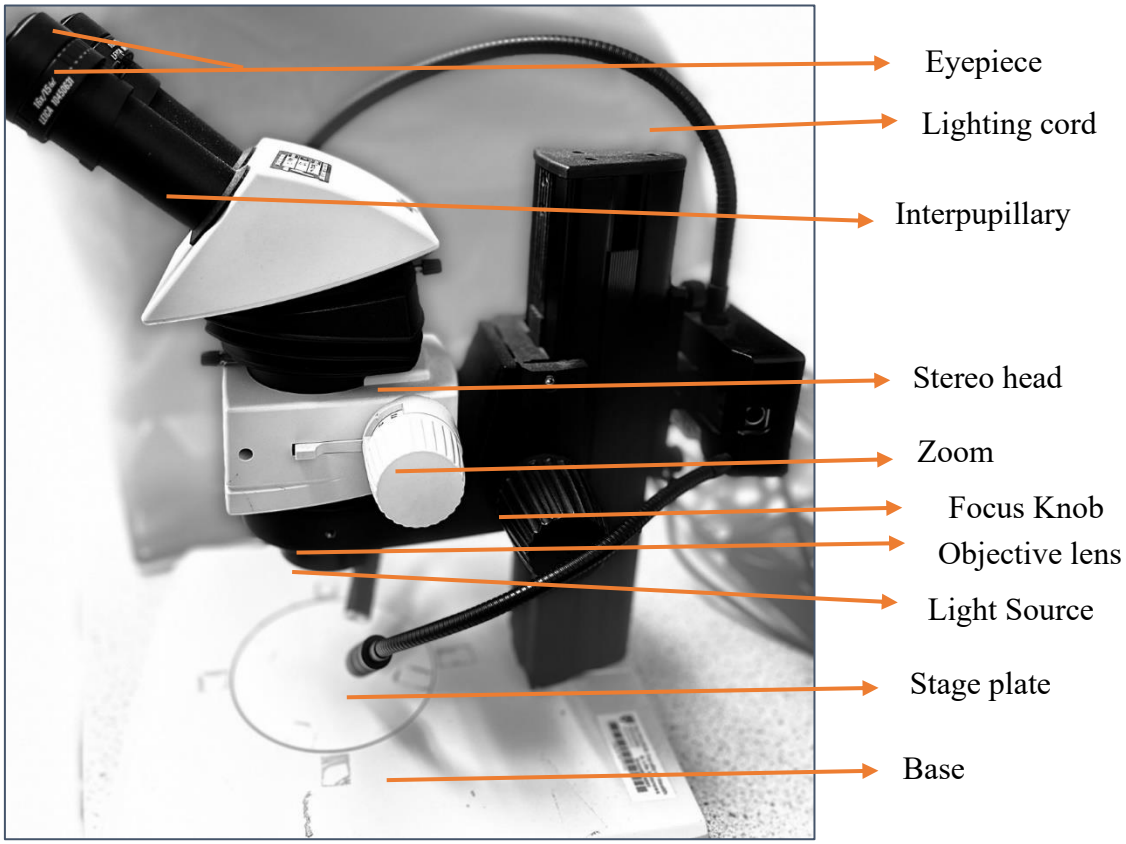


Figure 2.7: A Leica M60 low power microscope.

High Power Microscopy

High Power Microscopes (HPMs) (Figure 2.8) are built to give a higher magnification than stereo microscopes. Their magnification typically ranges from x40-x400. With the aid of HPMs, morphological features that differentiate one fibre from another can be clearly observed. More specialist types of microscopes that have distinct purposes in the forensic examination of fibres can also double up as HPMs due to their magnification capability. Due to a higher resolution compared to LPMs, fibres can be discriminated by their colour. As more details are obtainable with the level of resolution HPM offers, fibres can be compared side to side like in a specialist HPM known as comparison microscope (200, 201).

Figure 2.8: A Leica FS 4000 Comparison Microscope

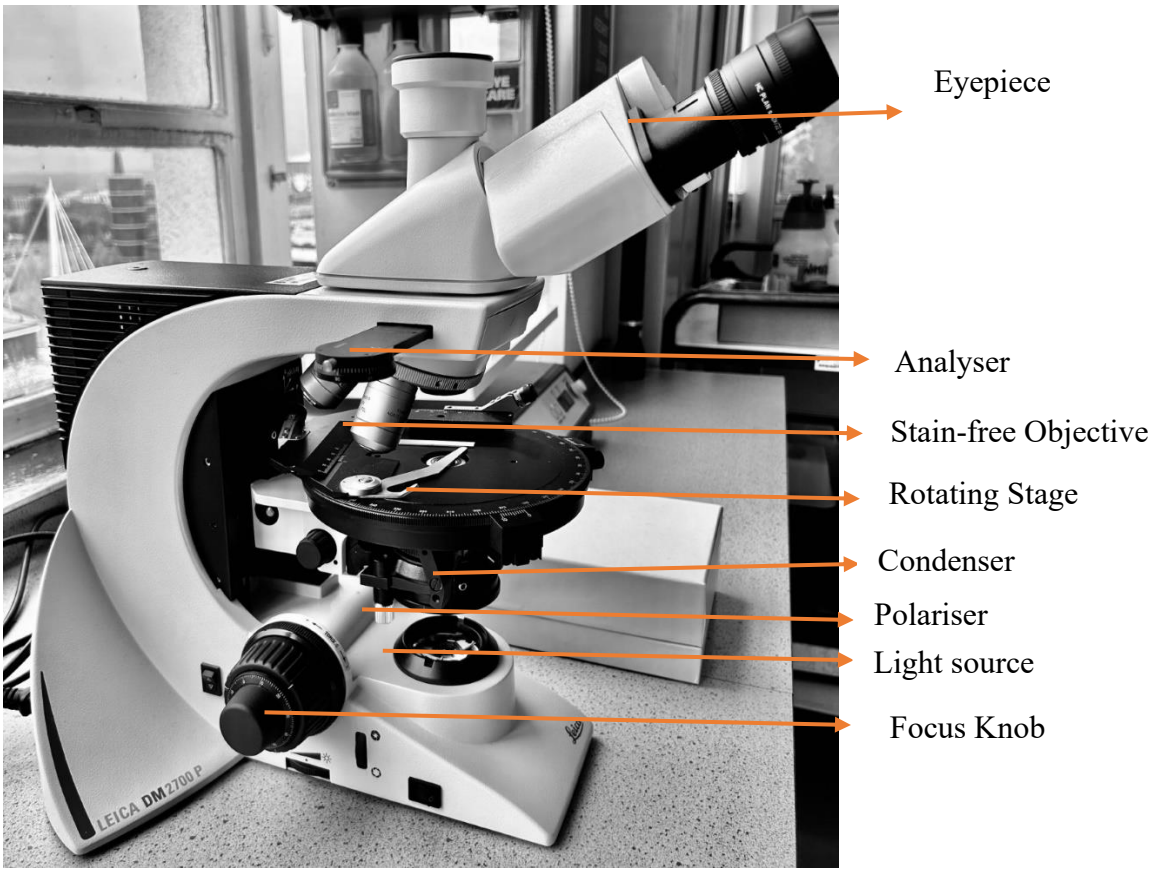


Figure 2.9: A Leica DM 2700 P Polarising Light Microscope

Polarising Light Microscopy

In addition to having a high magnification that aids in making fibres' morphological features to be clearly seen, a polarising light microscope (PLM) allows for the

determination of the generic class of synthetic fibres through its optical characteristics (200, 201). This is particularly useful for identifying synthetic fibres as they are mostly without distinguishing morphological features because of their production process. A PLM is distinguished from other HPMs by possessing two polarisers (one located beneath the specimen and another above the objectives called analyser) (Figure 2.9). When light is emitted from the light source, it passes through the polariser and become plane polarised i.e. instead of travelling in multiple directions it is restricted to one direction as it passes the polariser. The plane polarised light travels through the specimen in this case, a fibre and two separate wave components are formed possessing different refractive indices (201). This happens because fibres are anisotropic i.e., they possess different physical properties when measured at different directions. In the case of fibres, the different directions are along its length ($n_{||}$) and across its path ($n_{\perp}$). As they reach the analyser, they are planed polarised once again. When the polariser and analyser are perpendicular to each other, they are said to be crossed and when the fibre is viewed under crossed polars interference colours are produced. The interference colours produced are dependent on the refractive indices of the wave components produced when the polarised light passed through the fibre. Synthetic fibres can be identified by the interference colours produced or by calculating its birefringence (Δn) (Equation 2.1).

$(\Delta n) = (n_{||} - n_{\perp})$ ---------- **Equation 2.1**

Since not all synthetic fibres have a circular cross-section, an approximate thickness measurement can be achieved by determining the shape of the cross-section by looking at the fibre longitudinally and measuring between the locations. In place of measuring $n_{||}$ and $n_{\perp}$, a tilting compensator or a quartz wedge is used to determine the path difference. These components placed in the path of polarised light between the sample and the observer provide a graded effect that is the opposite of the fibre's path difference. The interference colours are extinguished once the effect is opposite and equal to the fibres path difference; this is known as the "extinction point". The tilting compensator

may be used to read the exact location where this happens, and a calibration table can be used to estimate the path difference of the fibre (in nanometres). The calibrated graticule aids in determining the fibres thickness (201). It is important to note that it may be difficult to pinpoint the extinction point when the fibre is heavily dyed or mostly damaged.

$$(\Delta n) = \frac{\Gamma}{1000 \text{ x } t} \text{ ---------- } \textbf{Equation 2.2}$$

where Γ is the path difference (units) and t is the fibre thickness (units - micrometres)

Fluorescence Microscopy (FM)

This microscopic technique works by exploiting the fluorescence characteristics of dyes, optical brighteners (often found in detergents) and contaminants present in fibres (200). Fluorescence occurs when these materials absorb high intensity and emit light of a lower intensity after being excited by light of a higher energy. The colour of the microscopic image produced is then based on the emission wavelength of the fluorescing material in the fibre. This occurs because fluorescence microscopes are equipped with filters which separates the fluorescent from other radiations. Various excitation filters are used such as UV, blue and green lights at 330-380nm, 450-490nm and 520-560nm respectively.

Fibres of ostensibly similar colour can be discriminated by this technique because of the array of dye stuffs used in dyeing fibres. For example, all blue colours of dye are not the same because they are made using a combination of dyes and different manufacturers will differ in how they produce a colour. In a study involving the examination of 293 blue cotton and 287 red cotton fibres from different sources (234) found that for each group about 90% of the fibres exhibited fluorescence characteristics in one or more of the three types of filters employed. Five groups of blue cotton and three groups of red were formed using FM. When anthropogenic MFs are recovered from aquatic environments, this discriminatory technique would be relevant in speculating how

diverse the source of pollution is. However, caution needs to be applied as false discrimination can occur between fibres from same source as a result of effect of optical brighteners used in some washing detergents (212).

2.4 Spectroscopic Techniques

Fourier Transform Infrared Spectroscopy

Fibres are made up of repeating chains of monomers held by chemical bonds. For example, cotton is made up of a repeating unit of glucose molecules held together by β-1,4-glycosidic bonds (Figure 2.10).

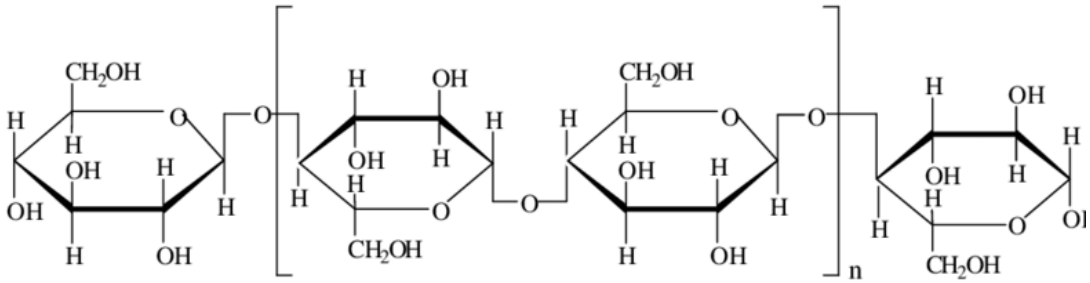


Figure 2.10: Repeating units of glucose held by β-1,4-glycosidic bonds (235)

This technique works by discriminating fibres based on the wavelengths of absorbed light within the infrared region of the electromagnetic spectrum (EMS) following its irradiation with infrared light. The range of wavelength within the EMS is shown Figure 2.11 below. When these bonds are irradiated by infrared light, some of the light is absorbed. The energy of the absorbed light causes the bonds to vibrate. An interferometer then produces an optical signal with all of the Infrared frequencies recorded. The signal is measured then deciphered using a mathematical process called Fourier transformation.

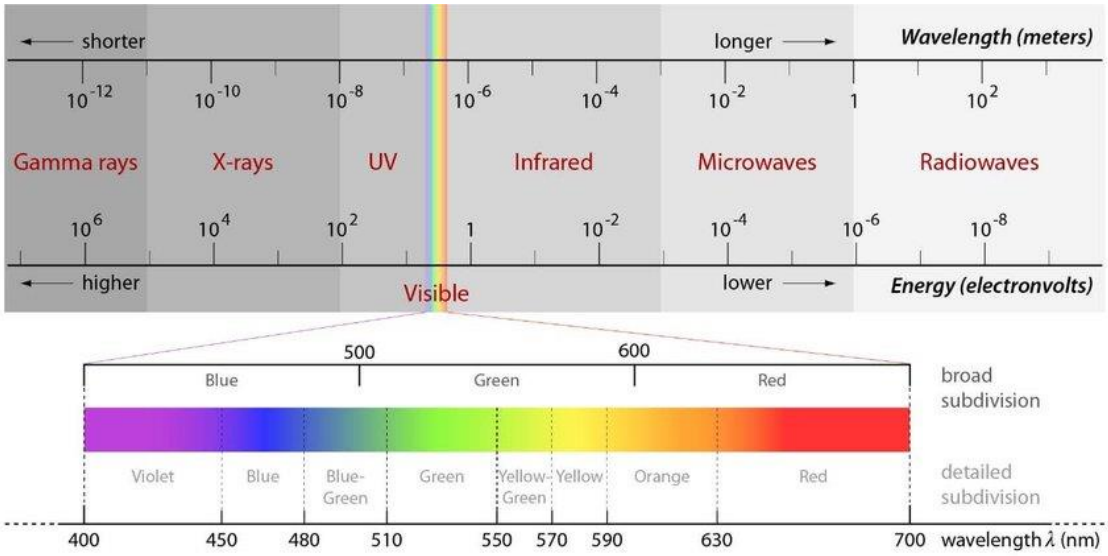


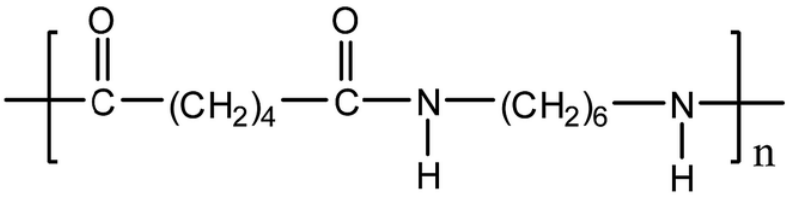
Figure 2.11: The Electromagnetic spectrum showing the range of wavelengths (236).

The spectral information is then generated and displayed on a computer. The resulting graph is the FTIR spectrum.

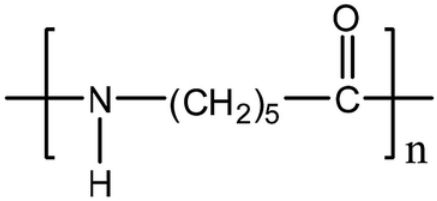
The absorption peaks of an infrared spectrum correspond to the frequencies of vibrations between the bonds of monomers making up the fibre. In order to more accurately ascertain fibre polymers from samples an FTIR is used. Fibre identification is achieved by comparing spectra produced by the fibre being examined to spectral information contained in a lab's database or by following guidelines provided for the interpretation of spectra which include checking for presence or absence of peaks rising from carbonyl and nitrile groups which are found in the range of 1500-1800 $cm^{-1}$and 2245 cm^{-1} respectively.

However, for an FTIR instrument to be able to analyse a fibre sample it would need to be augmented with a microscope increasing the cost of instrumentation. The microscope is needed to position the fibre in such a way that maximum radiation from the IR source is directed to the fibre. The sample processing often damages the surface of the fibre as it involves flattening the fibre. The implication being that when it is the first examination done on a fibre sample, the chances of getting any reliable information from its morphological attributes is slim. Nonetheless, in a scenario where discrimination needs

to go beyond generic level to sub class such as in Nylon 6 and Nylon 66 (Figure 2.12). FTIR would be vital as the main difference between these two are their chemical structures (237, 238, 239). Nylon 66 is composed of hexamethylenediamine and adipic acid monomers, leading to a repeating unit of twelve carbon atoms whereas nylon 6 is composed of caprolactam monomers, resulting in a repeating unit of six carbon atoms.



(a) Nylon 66



(b) Nylon 6

Figure 2.12: Structural differences between Nylon 66 and Nylon 6 (240).

Raman Spectrophotometer (Raman)

Raman is another type of vibrational spectroscopic instrument. This instrument is mostly non-destructive and minimal sample preparation required (241). It uses a powerful laser source like UV to irradiate a sample. Unlike FTIR which irradiates a sample with a wide range of frequency, Raman uses a monochromatic light to strike on the sample resulting in either elastic or inelastic light scattering. A spectrometer then measures the scattered light. Like FTIR, it can be used to identify a fibres molecular composition. However, its utility in dye analysis is prioritised as the identification of fibres' molecular make up usually yield weak signals (230, 239, 241). Due to the detailed spectrum it yields, Le pot et al. argued for its use in the discrimination of natural fibres since colour is their main discriminatory characteristics (242). Da Wael et al. on the other hand, commented on the

shorter processing time of using Raman compared to FTIR for identifying polymer type (polyethylene terephthalate) of the fibre that appeared like the target fibre in their study and confirming the spectra groups produced by microspectrophotometry (232). The technique is limited by its inability to differentiate between the energy loss because of fluorescence from energy change as a result of Raman effect (241). The possibility of thermal decomposition of fibre cannot be ruled out.

2.5 Discrimination of fibres based on their colour.

The colour of a fibre is determined by the colourants (colour imparting substances) used in textile manufacture (243). Colour is a significant factor in distinguishing known and unknown fibres, especially natural fibres like cotton, which have less discriminating morphological features than synthetic fibres. This is generally the initial characteristic that decides whether two samples should be analysed in the first place. For example, if the fibre samples are visibly green and red, comparing them would be pointless.

Dyes sometimes referred to as dyestuffs are substances that give colour to fibres. They are mostly soluble in water. Dyes bind to suitable surfaces by solution, by forming covalent bonds or complexes with salts or metals, physical adsorption, or mechanical retention (244). Their classification is based upon how they are structured chemically and by how they are applied. Figure 2.13 shows chemical structures of some dyes. Pigments on the other hand are colour imparting substances that are insoluble in the solvent or vehicle in which they are suspended (245). Pigments and dyes though colour imparting substances on textile substrates differ significantly (Table 2.1).

The colour of a dye is determined by a group of atoms called chromophores. Chromophores are unsaturated groups in the dye or pigment that absorbs light and reflects it at specific angle to give colour, such as azo-, keto-, nitro-, nitroso-, thio-, ethylene (Figure 2.14) e.tc. while on the other hand, auxochromes are salt forming groups

such as hydroxyl or amino groups containing electrons which either by withdrawing or donating substitutes generate or intensifies the colour of chromophores (246, 247, 248).

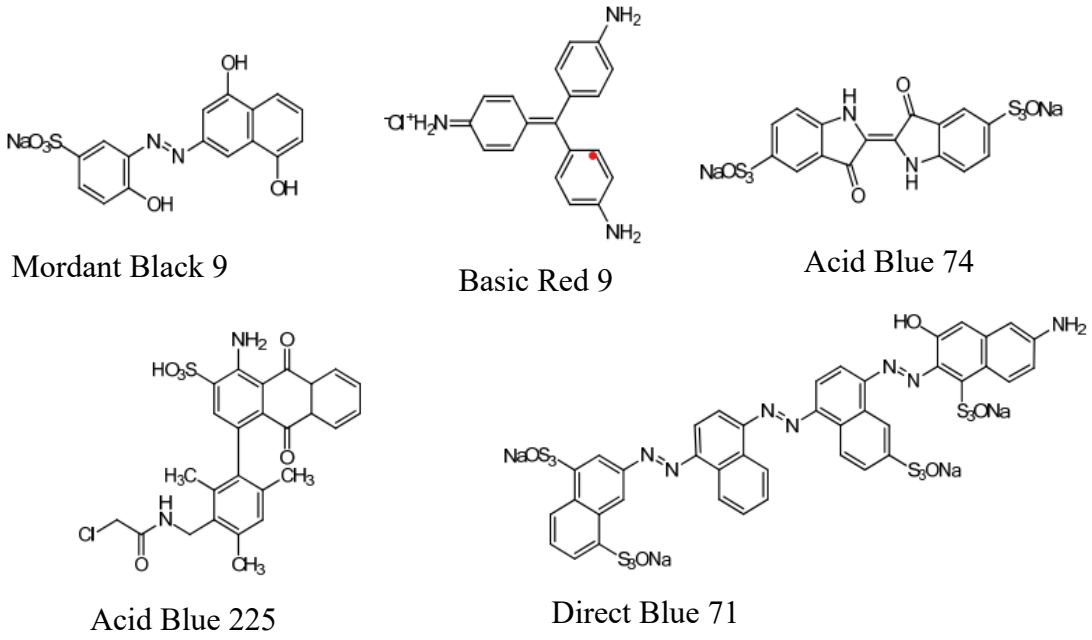


Figure 2.13: Chemical structures of some dyes (249).

Table 2.1: Difference between Dyes and Pigments

Characteristics	**Dyes**	**Pigments**
Durability	Durability depends on chemical bond or linkage	Durability depends on binders used
Molecular size	Small molecular size to aid penetration.	Molecular size varies from small to large
Solubility	Soluble in water during dyeing stage	Insoluble in water and common solvents

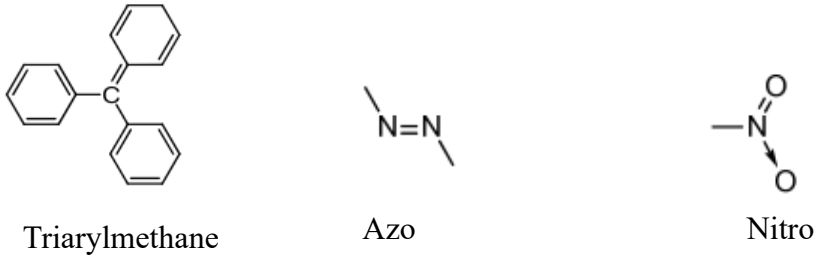


Figure 2.14: Chemical structures of chromophores (249).

Due to the nature of fibres, dye uptake could occur in a manner that results in intra and inter-sample variation. Inter and intra sample variation can occur due to uneven dye uptake caused by fibre structure or dye molecule variation. This is usually observed in natural fibres such as cotton and wool (225, 250, 251). This is because natural fibres are composed of inhomogeneously distributed chemical components throughout the matrix of the fibre (246). Inter-variation occurs also as a result of differences in dye concentration across batches. Other factors include changes in dye bath conditions (temperature, pH, salt added, and so on), the presence of dye precursor residue, functionality of the dye or the presence of "dead" fibres in a bale (252).

When comparing a known sample of fibre to a likely source, heterogeneity increases the potential discrimination power. This happens when individual fibres possess areas having distinctly different colours because each of the areas can be analysed and compared separately (253).The nature of a dyestuff determines the type of fibre it would be used for. For example, the covalent bond between the hydroxyl groups in cellulose and atoms in reactive dyes. Ionic bonds formed between amino groups in wool, silk, and nylon with anionic dyes such as acid dyes(246).

Fibre analysis has advanced in such a way that even single fibres can be analysed to identify the type of dye it is composed of (254). At times, these single fibres are made up of more than one dyestuff. In determining whether a fibre can be linked to a putative

source, the type and number of dyes used in the manufacture of fibres are employed as discriminatory factors. Dye mixtures increase evidential value in situations when they are encountered as it increases the individuality of the fibre (254). Dye mixtures are usually a result of a manufacturer's desired end product. These dye mixtures come as a manufacturer's 'signature' thereby imputing unique characteristics that have the potential to lead back to the source of the fibre. However, determining the composition of the mixtures is accompanied by the limitation of fibre destruction.

2.6 The Way Forward

The uses, advantages, and limitations of commonly used techniques in forensic examination of fibres have been discussed. They are not being presented for the first time in MF pollution studies. As earlier noted, FTIR and Raman are commonly employed in these studies. However there needs to be a paradigm shift in the way fibre examinations are currently carried out. The necessity for standardising methodology in MF pollution research has been noted in published literature as captured by Athey and Erdle (6). Forensic science studies in fibre examination had begun decades before environmental studies of MF pollution (255). As a result, time tested techniques have been developed coupled with the responsibility to present robust and transparent data in court. Adapting a forensic approach to MF pollution studies appears to be a solution to the lack of standardisation of method currently plaguing MF pollution studies. It can achieve this through normalising contamination control and prioritising methods to identify all MF types.

Since Woodall et al. proposed a forensic approach in their 2015 publication (4) some studies have employed PLM though not as a first point of call (256, 257). Although Woodall et al. employed a forensic approach including minimising contamination and identifying MFs using PLM, complete information was not given on fibre types (37). A category was assigned as "cellulosic" which could be any of the natural or regenerated

MFs with a cellulose base. Similar incomplete data was observed in Stanton et al. (69) who employed PLM in categorising MFs into natural and extruded fibres without giving data on the generic types. A more recent study in the Antarctic also employed PLM to only identify synthetic MFs in their study (258). It appears that apart from advocating for the need to prioritise PLM as first point of call, emphasis must be laid on utilising it to identify all categories of fibre.

2.7 Conclusion

The concept of an interdisciplinary approach to MF pollution research is not new, but it is a proposition that should be taken seriously, and deliberate efforts should be made (259). So far, recent investigations have demonstrated the enormous value that forensic fibre examination processes and techniques contribute to the MF pollution discourse. The forensic approach has been proven to be fit for purpose, from assisting in addressing contamination issues during MF pollution studies (4, 183) to quantifying and identifying anthropogenic MFs. The suite of techniques outlined in this chapter presents opportunities to not only discriminate MFs of synthetic origin (37), or just discriminating merely between natural and extruded fibres (69) but to identify all MF individually, present in samples at least to their generic level. This later application has now been implemented in research carried out in both marine (Kenyan-Tanzanian coast) and fresh water (Lake Victoria) environments which are detailed in subsequent chapters of this thesis.

Chapter Three

Prevalence and Characterisation of Microfibres in Marine Environment Using the Kenyan-Tanzanian Coast as Case Study

3.1 Introduction

Anthropogenic Microfibres in Africa's Marine Environment

The occurrence of anthropogenic MFs in marine environments has been extensively researched, and investigations in Asia (64, 161, 163, 172), America (260), Antarctica (258), Europe (210, 261) and Australia (262, 263) have demonstrated that MF pollution is a global issue. However, global estimation of MF pollution appears to be limited by arguably two major factors: first, underestimation of natural MFs because most studies are primarily designed to capture and study microplastics, and secondly, a paucity of data from certain parts of the world, such as Africa (6).

In studies where natural MFs have been quantified, it is mainly as a category without providing numbers or relative proportion of individual fibre types (264). In other instances, the presence of cotton is noted without quantifying them or lumped into the category "cellulosic" (165). Although Suaria et al. (5) provided one of the most comprehensive data of MFs present in oceanic surface water, they were unable to reliably discriminate between natural and regenerated cellulosic MFs because of the limitation associated with the use of FTIR (265). Methods such as Stimulated Raman Scattering (SRS) microscopy, have been noted to be useful in the identification of natural fibres; nonetheless, it destroyed more than 30% of examined fibres (sample size = 71) (266). In Chapter Two, it was demonstrated that relatively simple microscopic techniques, such as optical microscopy and polarising light microscopy (PLM) employed in forensic studies are effective and efficient in identifying all fibre types, while needing minimal sample

preparation and being non-destructive to MFs. Using this forensic approach, this chapter aimed to systematically and objectively identify all MFs present in surface water samples recovered from the Kenyan-Tanzanian coast with the aim of providing robust data describing the types of MF polluting that region of the Indian Ocean.

The Kenyan-Tanzanian Coast and Microfibre Pollution

The Kenyan-Tanzanian coast is in the Western Indian Ocean (WIO), which is a region of the Indian Ocean (IO). In addition to Kenya and Tanzania, the WIO encompasses the shores of South Africa, Mozambique, Madagascar, and Somalia. The Indian Ocean (IO) is a body of seawater that runs over 6,200 miles between the southernmost tip of Africa and Australia, spanning one-seventh of the Earth's surface. It has a depth of 3,741 metres on average. It is the third biggest of the world's five oceans, although its oceanic boundaries are difficult to identify (267). The IO has the inland Red Sea and Persian Gulf to its north, The Arabian Sea to the northwest and the Andaman Sea to the northwest.

Deep seas have been theorised and evidenced to be major sink for microplastics including MFs (210, 268, 269) and therefore investigating the presence of MFs in the Indian Ocean would contribute to the knowledge of the type, mechanism of transport and accumulation of anthropogenic MFs. In the Eastern Indian Ocean for example, there is an indication that atmospheric MFs over the ocean originates from emissions from adjacent continents and inter-oceanic travels and these eventually end up in the ocean’s water column (171, 172). MFs are being ingested by zooplankton in the Arabian Sea, Indian Ocean (270) and therefore emphasises the need to identify all types of anthropogenic MFs polluting the environment.

3.2 Materials and Method

Sites of Study

The Flipflopi sailing dhow embarked on a journey southward, tracing the Kenyan coastline with stops in Watamu, Kilifi, Mombasa, Diani, and Shimoni. It then continued its voyage by crossing into northern Tanzania, reaching Pemba Island, and concluding its expedition in Stone Town, Zanzibar (187). 37 locations along the Kenyan-Tanzanian coast were sampled (Figure 3.1). Locations 1-24 were situated in Kenya whereas locations 25-37 were part of northern Tanzania.

Citizen Scientists

Sampling of the Kenyan-Tanzanian coast were carried out by the Flipflopi crew including citizen scientists. Citizen scientists were locals who joined and departed the expedition at various points along the journey. There were about a total of 12 locals in all who participated. The work that was being done was explained to them and they were taught how to undertake the sampling process and they carried this out following the protocols outlined by the Flipflopi crew.

Sampling Procedure

Surface water samples were collected by the expedition crew including citizen scientists with a specially made simple device from a commercially bought and adapted AeroPress® (Figure 3.2). The AeroPress® sampler device was developed by a Northumbria University member of staff in the Design School. The modification involved replacing the Aeropress®'s original conventional paper filters with nylon filters. These nylon filters were crafted from a 50 μm nylon mesh sheet (Plastok Associates Ltd. in the United Kingdom). They were cut into 50 mm diameter discs using a laser (Epilog Laser Legend 36EXT, United Kingdom). The nylon filter was subsequently positioned atop the AeroPress® metal filter. The AeroPress® was then

affixed to a 1,000 mm flexible High-Density Polyethylene (HDPE) plastic tube with an 80 Ø mm aperture, which remained open through the use of a robust wide plastic tube, facilitating the easy filtration of ocean water (271). Appendix 1 contains details of the sampled locations and can also be accessed here **https://tinyurl.com/flipflopimap**

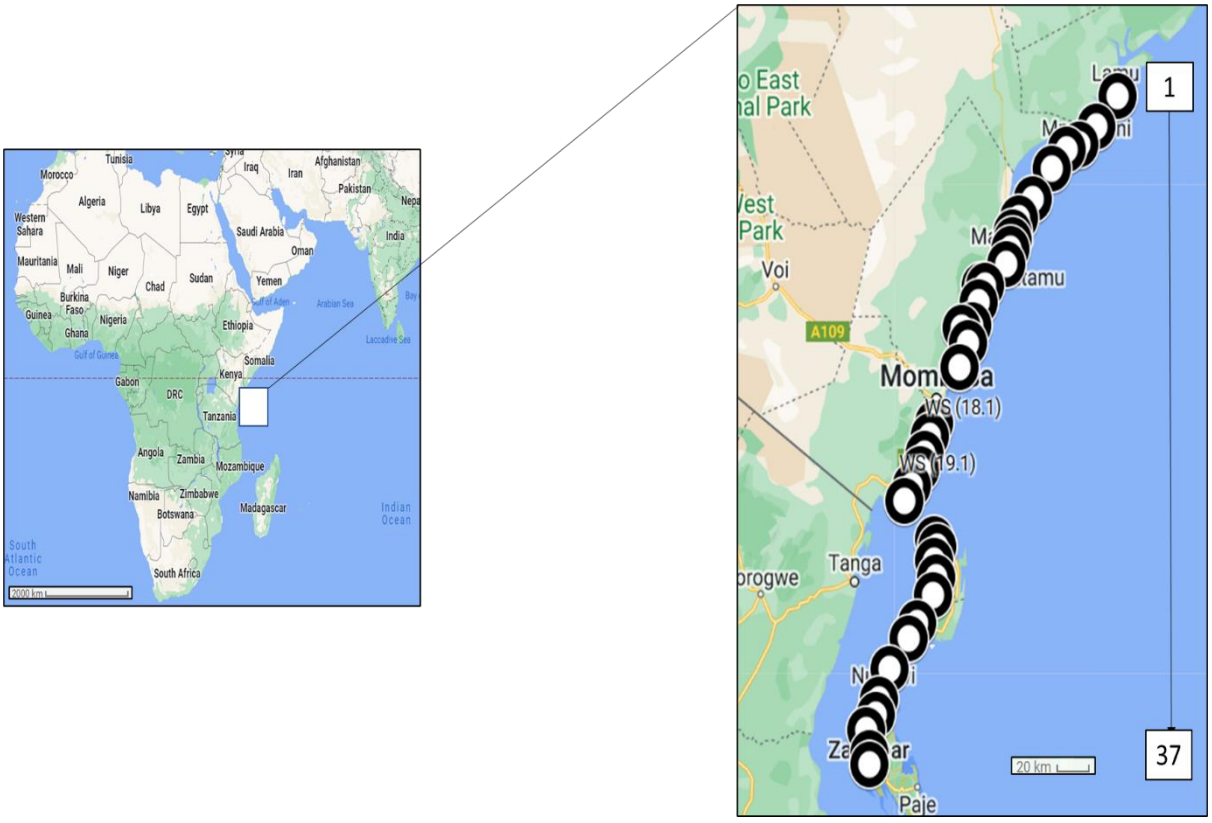


Figure 3.1: Map showing snapshot of 37 sampling locations from Lamu (Kenya) to Zanzibar (Tanzania) with arrow showing direction of expedition.

Figure 3.2: AeroPress® sampling device in use by local citizens

A 2 L plastic jug was used at each sample station to take ocean water from the surface to a maximum depth of 100 mm below the surface. The water was poured down the tube and filtered back to the ocean. This procedure was carried out three times, sampling a total of 6 L of water. Once all the water had been filtered through the sampling device, the nylon mesh filter holding any filtrate was removed from the device using tweezers and placed in a sealed zip lock labelled bag ready to be sent to the United Kingdom for examination.

Quality Assurance and Control

To minimise contamination between sampled locations, the Aeropress® sampling device was rinsed with bottled water stored on board after each sampling process during field sampling. In the laboratory, contamination was controlled by following rigorous laboratory protocols during the MF recovery process. To exclude any coloured fibres potentially contaminating the samples, clean and new laboratory coats made of colourless cotton fibres were worn. The recovered samples were kept in clean and enclosed petri dishes after they were dried. Vikron was used to clean the laboratory

benches and equipment before and after each use. To maintain cleanliness, fresh brown paper was used to line the benches after cleaning. To minimize contamination, all fibre observation and recovery procedures were performed under a stereomicroscope, allowing any extraneous fibres to be promptly identified and discarded (as per standard forensic protocols). Moreover, samples were always covered when not being analysed. Permanent mountant was applied during sample preparation, this ensured that extraneous MFs were not introduced to the mounted slides as the edges were sealed when dried.

Microscopic and Spectroscopic Identification of MFs

Glass slides containing already mounted MFs recovered from surface water filtrates were examined using high-power microscopy with magnification of x100-400 (Comparison Microscope, Leica DMR, Germany) coupled with UV and blue light filters to discriminate fibres based on florescence characteristics. The morphological characteristics of a fibre were used to characterise it as either cotton, wool, viscose or synthetic as per the flow chart depicted in Figure 3.3. As discrimination is based on differentiating physical characteristics/morphology, damages on MFs may lead to inaccurate identification. For instance, if scales on a wool fibre deteriorates, it may prove difficult to classify confidently. However, prior to analysis of samples, the researcher familiarised herself with fibre morphology by examining more than 1000 fibres under the microscope.

Further discrimination was carried out by noting the absence or presence of fluorescence under each of the two filters as well as the colour and intensity. Intensity was noted by indicating ‘bright’ or ‘dull’ to the fluorescent colour.

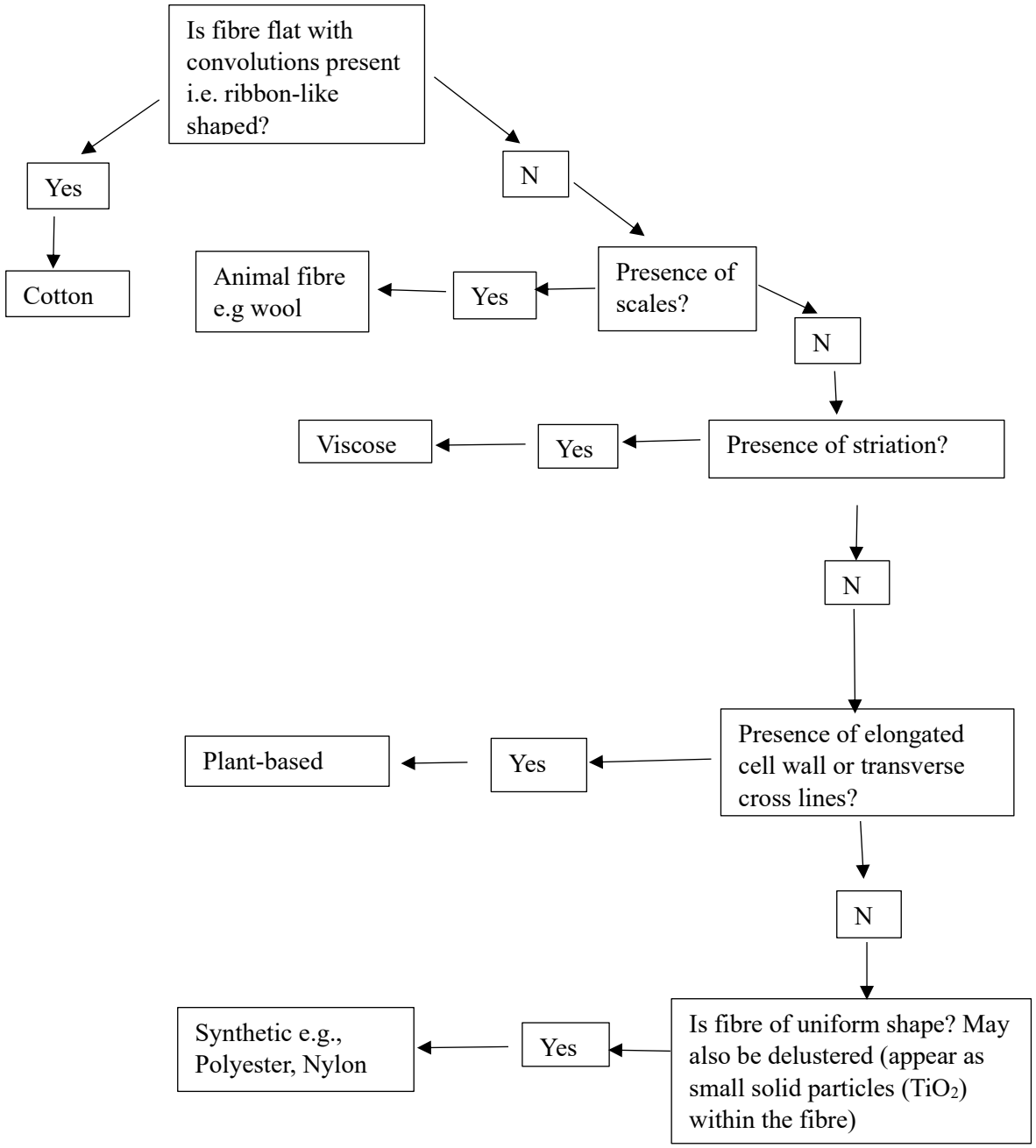


Figure 3.3: Diagram illustrating fibre identification process under High Power Microscope (HPM)(228, 272)

Synthetic fibres were further analysed using Polarizing Light Microscopy (PLM) (Leica DM2700P, Germany) to differentiate between generic types following birefringent measurement (200). When additional confirmation was needed, further examination was performed using a μFTIR (Perkin Elmer Frontier connected to spotlight 150i Microscope), either because the fibre was damaged, extensively coloured, or features were not clear to enable identification. It is important to note however, if the fibre that needed further discrimination due to the afore mentioned reasons was cellulose based for example, using an FTIR would be ineffective to determine its generic class such as discriminating between viscose and cotton. With a wavelength range of 400–500 cm-1, a resolution of 4 cm-1, and an accumulation of 32 scans, a measurement in transmittance mode was made. The internal textile fibre spectral libraries' contents and the spectra of unknown fibres were compared. The spectral library was created using a known, authenticated textile fibre collection donated to Northumbria University from The Forensic Science Service (UK). A correlation value of >0.5 was considered an acceptable match in addition to visual examination of the spectral images.

Colours were identified based on a fibre's appearance under a high-power microscope (HPM). White/colourless/transparent fibres were purposefully not recovered or identified in this study due to the difficulty of being able to locate them. It is important to note that fibres from a pale-coloured fabric, for example, a pale pink T-shirt, would appear colourless under microscopic view. This is because, the background on which an object is viewed may impact on the perception of its colour (273). Black and grey colours were combined because they may appear similar sometimes, making them easily overstated or underestimated as separate groups. The same is sometimes true of red and pink, and in this study, they have been classified as red. Once the identification process was complete all recovered fibres were fully categorised.

Statistical analysis

The data produced were not normally distributed and therefore non-parametric tests were applied. Hence, for comparison between the three fibre categories, the Kruskal-Wallis H test was employed, and for pairwise comparisons, the Mann-Whitney U test was utilised, with a significance level of 0.05 used in all analyses. Microsoft Excel™ of Microsoft 365 Version 2,205 was used for statistical analysis.

3.3 Results

Concentration and Relative Abundance of Natural, Regenerated Cellulosic and Synthetic Anthropogenic MFs in the Kenyan-Tanzanian Coast.

Following microscopic examination, 2,403 MFs were recovered from the nylon filters following filtering of the surface water samples. However, data from 2,382 MFs have been presented here as the rest were lost or damaged during sample preparation. The mean MF concentration of the 37 sampled locations was determined to be 10.73 ± 1.99 fibres L^{-1} with a range of 1.33 fibres L^{-1} to 58.67 fibres L^{-1} per sampled location (Figure 3.4). Nine fibre types (Table 3.1) were identified from the natural, regenerated or synthetic categories. Natural fibres included cotton, wool and other vegetable fibres; viscose was the only type of regenerated cellulosic fibre found and of the synthetic fibre types, acrylic, nylon, polyester, polyethylene and polypropylene were present.

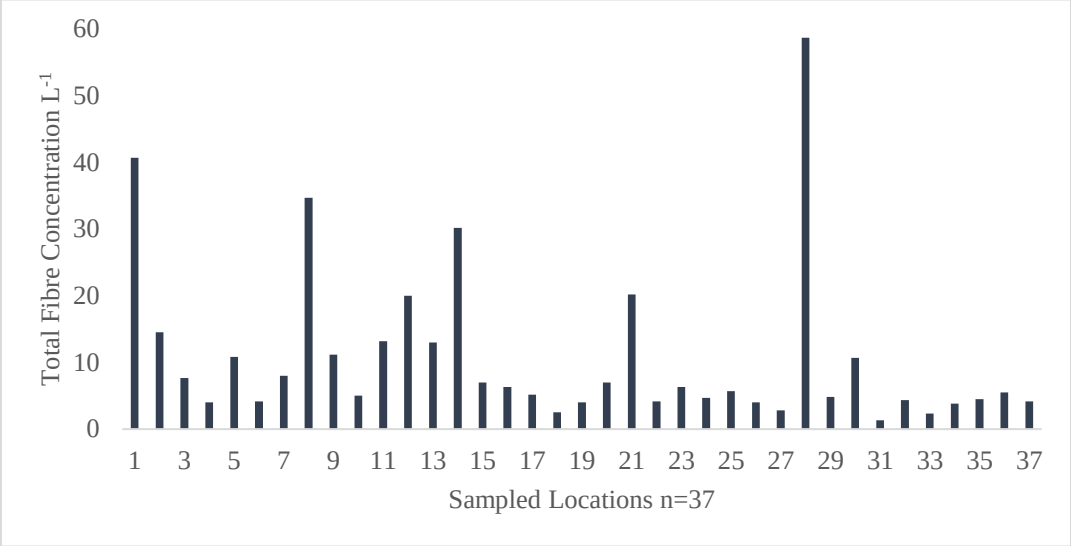


Figure 3.4: Total concentration of MFs per sampled location

Natural MFs had the highest concentration, followed by synthetic then regenerated cellulosic (Figure 3.5). Statistically significant differences were found between the distribution of the three fibre categories (H=52.82, p-value. < 00001).

Table 3.1: Classification of Microfibre Types Found in Present Study

Natural Microfibres	Regenerated Cellulosic Microfibres	Synthetic Microfibres
Cotton	Viscose (Rayon)	Acrylic
Other Vegetable		Nylon
Wool		Polypropylene
		Polyester
		Polyethylene

Further analysis indicated that natural MFs were statistically significantly more abundant than both synthetic and regenerated fibres (Z-Score = 3.35 and $p = 0.001$) and (Z-Score = 6.57 and $p < 0.00001$), respectively. Synthetic MFs were found to be significantly more abundant in distribution compared to regenerated MFs (Z-Score = −5.06 and $p < 0.00001$).

Using the three-group categorisation (natural, regenerated cellulosic and synthetic fibres), natural MFs were most abundant in 33 of the 37 studied locations (Figure 3.6a). However, when regenerated cellulosic and synthetic MFs are combined into one category (man-made) which is commonly done in microfibre pollution studies, natural MFs still had the greater concentration but were found to be more in abundance in 29 instead of 33 of the 37 sampled locations (Figure 3.6b). The difference between the distribution of natural MFs and man-made fibres were also found to be statistically significant (Z-score =1.98, p-value = .047). All results are significant at $p < .05$.

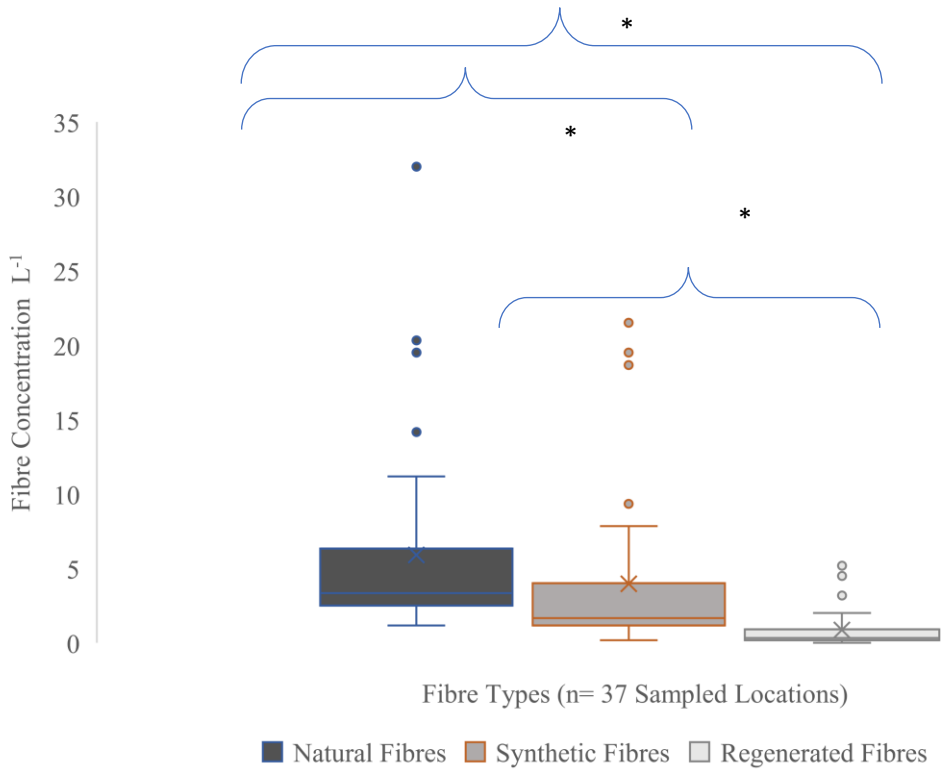
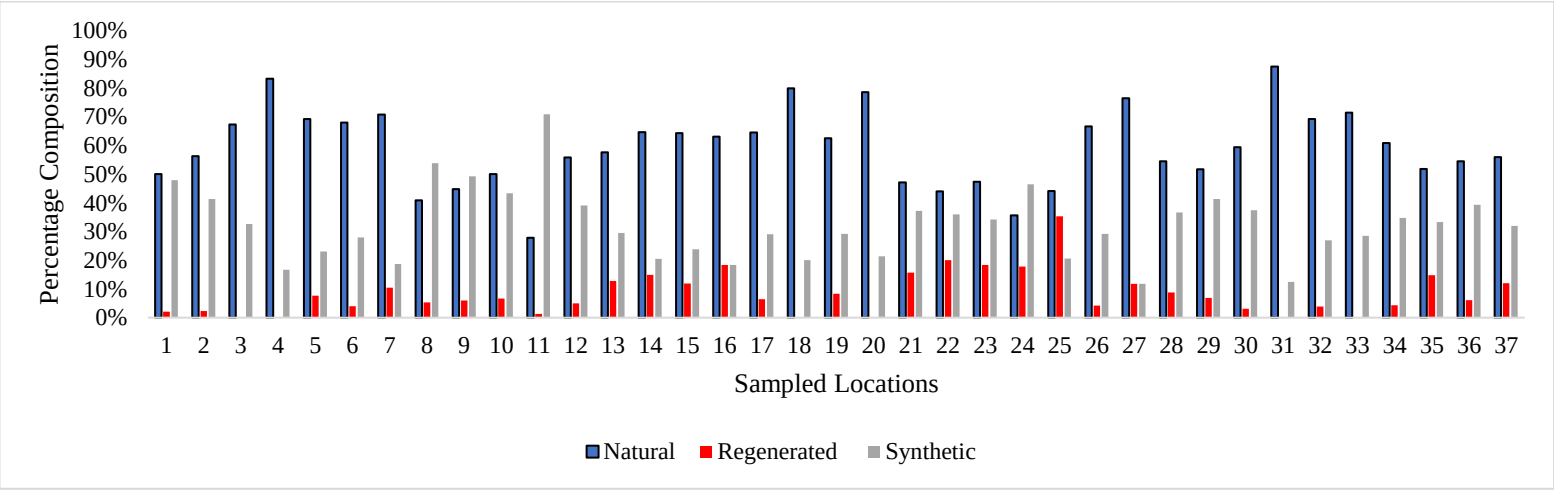


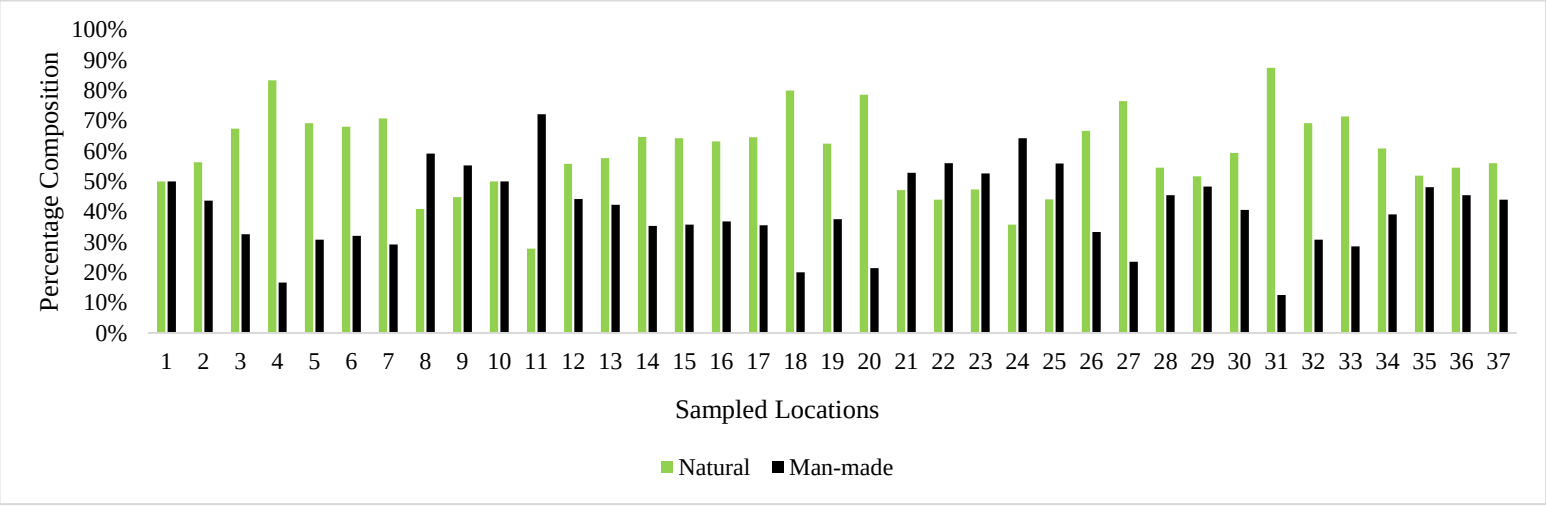
Figure 3.5: Concentration of natural, synthetic and regenerated cellulosic fibres distributed across the 37 sampled locations. * Statistically significant difference at α=0.05

In order of decreasing abundance were cotton, polyester, viscose, acrylic, wool, nylon, other vegetable, polypropylene and polyethylene i.e., cotton had a mean concentration

of 5.24 ± 0.95 fibres L^{-1} (49%), polyester had 2.93 ± 0.63 fibres L^{-1} (27%) and polyethylene was 0.01 ± 0.01 fibres L^{-1} (0.1%) (Table 3.2). Cotton and polyester MFs were the only fibre type found in all of the 37 locations sampled. However, no MF type was found in less than 2 of the sampled locations. Although the individual numbers of viscose and acrylic MFs were greater than wool, the latter was found in more locations than viscose and acrylic. The least occurring MF was equally found in the least number of sampled locations.



(a)



(b)

Figure 3.6 a-b: Percentage distribution of natural, regenerated cellulosic and synthetic MFs across sampled locations.

Table 3.2: Percentage proportion and relative abundance of various MF types

Fibre Type	Category	Mean Concentration (Fibres L^{-1})	No. of sites found	Percentage of total fibres
Cotton	Natural	5.24 ± 0.95	37	48.8
Polyester	Synthetic	2.93 ± 0.63	37	27.3
Viscose	Regenerated Cellulose	0.86 ± 0.19	31	8.1
Acrylic	Synthetic	0.64 ± 0.23	26	6.0
Wool	Natural	0.55 ± 0.11	32	5.1
Nylon	Synthetic	0.23 ± 0.07	16	2.1
Polypropylene	Synthetic	0.15 ± 0.04	16	1.4
Other Vegetable	Natural	0.12 ± 0.03	16	1.1
Polyethylene	Synthetic	0.01 ± 0.01	2	0.1

Prevailing MF Colours

Nine colour groups in total were represented in all recovered samples including blue, black/grey, red, green, black/green, brown, yellow, orange and purple in decreasing order (Figure 3.7) . All major colours were represented. In all the studied locations, blue, black/grey, and red were the most prevalent colours (Figure 3.8). Red fibres were found in more than 80% of examined places, whereas blue and black/grey were present in all the locations. It was discovered that some colours were more frequently associated with a specific MF type than others. For instance, a greater proportion of yellow, black/green and

red were natural MFs whereas blue, orange and purple were mostly synthetic MFs (Figure 3.9).

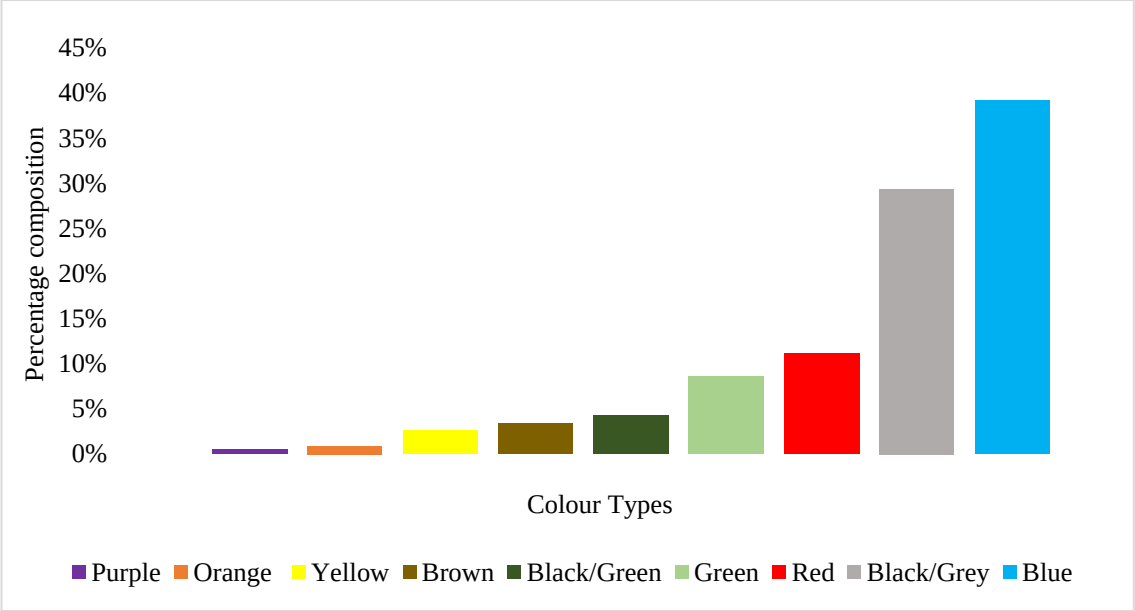


Figure 3.7: Overall percentage distribution of all colour types found in sampled sites.

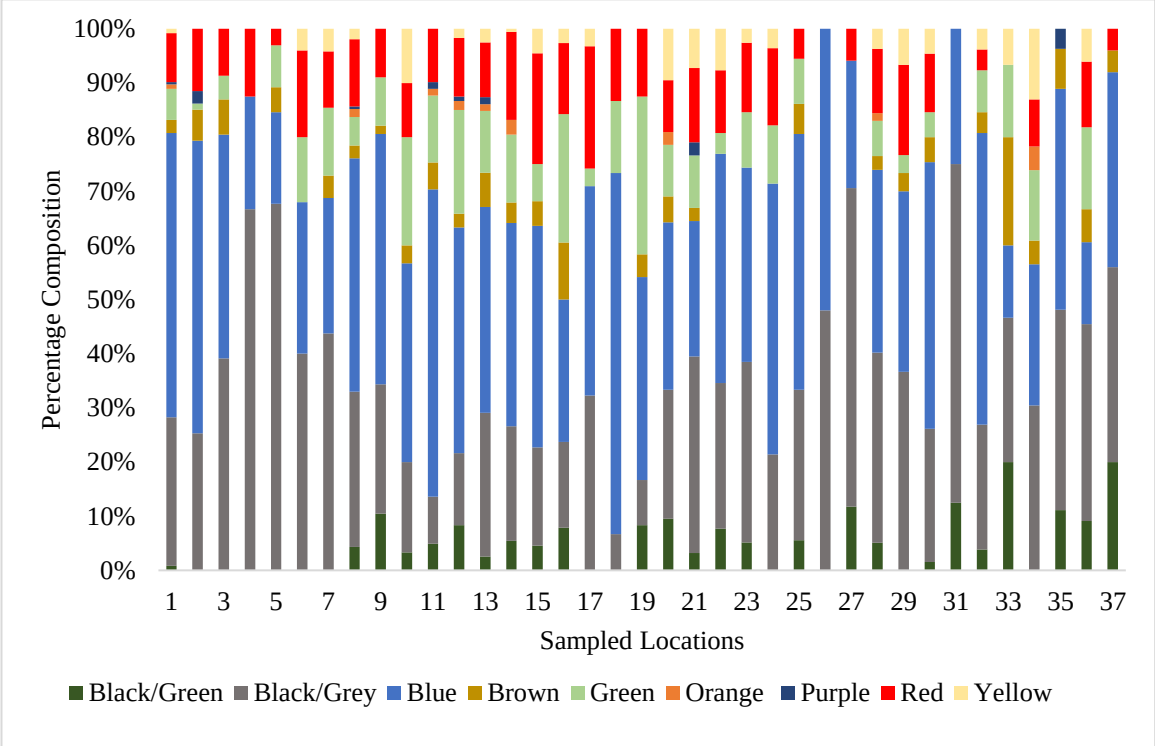


Figure 3.8: Percentage colour distribution across 37 sampled locations

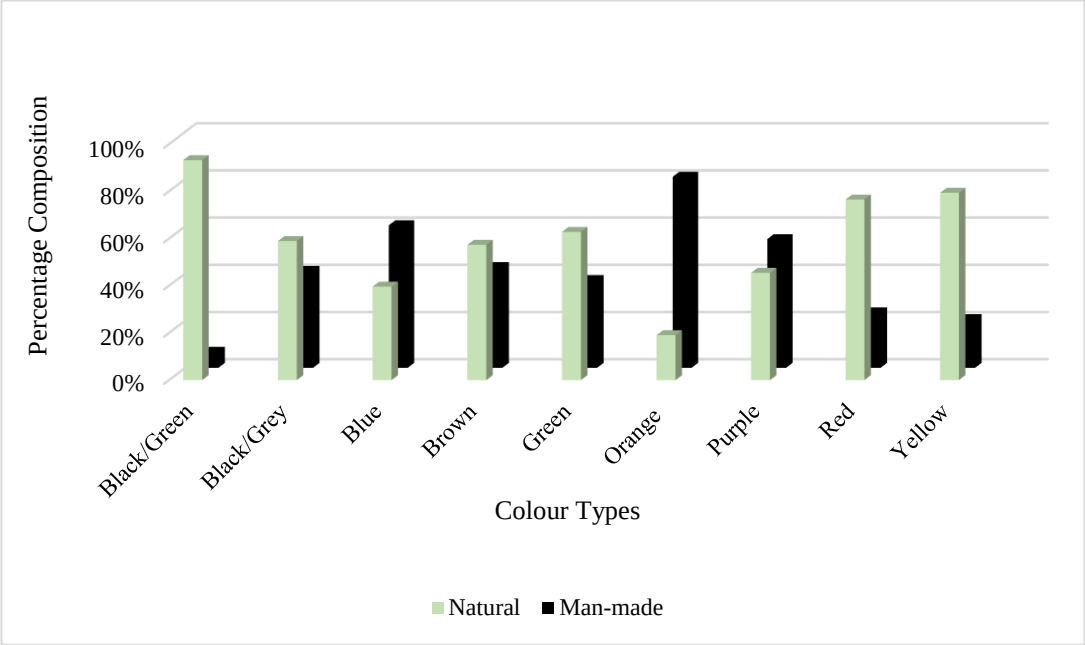


Figure 3.9: Colour distribution between natural and man-made MFs.

Investigating Potential Contamination through Fluorescence Discrimination

Although measures were put in place during laboratory stage of the examination of samples, this was not entirely the case during sampling in the field. Ideally, field blanks should have been taken in addition to control fibre samples from the clothing of crew members in order to eliminate them from the anthropogenic fibres in the water samples (183). However, using discrimination methods applicable in forensic examination, possible contaminants may be hypothesised using available information from fibres identified in the samples. The crew members were provided with Flipflopi branded uniforms made from 100% cotton including white face caps, white or blue T-shirts and beige cotton shorts. The beige fibres would appear pale/colourless. Since colourless/white/transparent fibres were not included in the study these can be eliminated as sources of overestimation of data.

Table 3.3: Discrimination of blue cotton fibres based on fluorescence.

Groups			Fluorescence characteristics			
n=13 groups		n=28 groups			Total number of MFs	No. of sites found
Group	Total MFs		UV Filter	Blue Filter		
		1	Blue	Dull Green	44	19
Group A	93	2	Blue	Green	46	25
		3	Dull Blue	Dull green	3	3
		4	Dull blue	Dull orange	4	3
Group B	21	5	Blue	Dull orange	7	6
		6	Blue	Orange	10	6
		7	Blue	Dull yellow	12	7
Group C	21	8	Blue	Yellow	6	6
		9	Dull blue	Dull yellow	3	3
		10	Dull blue	Dull red	7	5
Group D	36	11	Blue	Dull red	26	11
		12	Blue	Red	3	3
Group E	1	13	Blue	Red-green	1	1
	1	14	Dull Blue	Orange-green	1	1
		15	Blue	None	94	25
Group F	111	16	Bright blue	None	3	1
		17	Dull blue	None	14	6
Group G	2	18	Dull purple	Dull yellow	2	2
Group H		19	None	None	37	13

Table 3.3: Discrimination of blue cotton fibres based on fluorescence (contd)

		20	Dull purple	Orange	1	1
Group I	8	21	Dull purple	Dull orange	1	1
		22	Purple	Orange	6	6
Group J	1	23	Purple	Green	1	1
Group K	1	24	None	Dull green	1	1
Group L	1	25	None	Dull red	1	1
		26	Dull purple	Dull red	3	3
Group M	7	27	Purple	Red	3	2
		28	Purple	Dull Red	1	1

The blue cotton fibres on the other hand are potential sources of contamination. The possible overestimation of cotton MFs could not be ruled out and therefore all 343 blue cotton MFs recovered from the samples were discriminated based on their fluorescence characteristics.

Discrimination was based on the presence or absence of fluorescence when under viewed under UV and blue filters. When categorised based on fluorescence colour without consideration of the intensity, 14 groups were formed (Groups A-M). However, when intensity of the colour was considered 28 groups were formed (Groups 1 – 28) (Table 3.3). To determine whether there was a potential contaminant group, the fibre group would likely have the highest number of MFs and frequency of occurrence in all the sampled locations. This discriminatory process helps to determine if fibres originated from multiple sources and therefore source(s) not limited to the blue T-shirts.

Based on first discrimination without considering of intensity, two groups were of interest. First group comprised of 93 MFs and were present in 31 of the 37 sampled locations. They fluoresced blue under UV and green under the blue light. The second group was made up of

111 MFs and fluoresced blue under UV and had no fluorescence in blue light. They were present in 26 out of the 37 sampled locations. However, further discrimination resulted in 3 sub-groups each based on intensity of fluorescence. 3 out these 6 groups were possible contaminant. One group comprised of 94 MFs and were present in 25 out of the 37 locations sampled. This group fluoresced normal blue (was not perceived as bright or dull) under UV and nothing under blue light. The other two potential groups had different intensities of green fluorescence and comprised of 46 and 44 MFs found in 25 and 19 locations respectively.

Influence of Human Population and Sampling Location on MF Concentration

Not all communities near the sampling locations had data on their population density. Utilizing the population data that was available, sampling locations were divided into five geographic regions (Table 3.4). No discernible pattern was found when MF concentration and population size were analysed (Figure 3.10) and the interaction was not found to be statistically significant (p=0.067). However, a negative correlation was found between the amount of MFs present and the sampling site's proximity to land, with the amount of MF present being higher close to land (Figure 3.11a-i).

Table 3.4: Population of Geographic Regions of Sampled Locations. Population data source: (274, 275)

Sites	**Geographic Regions**	**Population**	**Average Microfibre Concentration (Fibre L^{-1})**
1- 2	Lamu County	143,920	27.59
3- 5	Tana River County	315,943	7.50
6-18	Kilifi County	1,453,787	12.33
19-24	Kwale County	866,820	7.72
25-37	Zanzibar Archipelago	1,300,000	8.67

Nevertheless, when the data was untreated or log-transformed, this relationship was not statistically significant (p= 0.32) and (p=0.18) respectively. The correlation of the

concentration of various MF types to the distance of the sampled location followed the same trend except for the category of 'other vegetable'. Polyester and wool appeared to have a similar relationship between their concentration and distance of sampling location from land. (Figure 3.12 a- i).

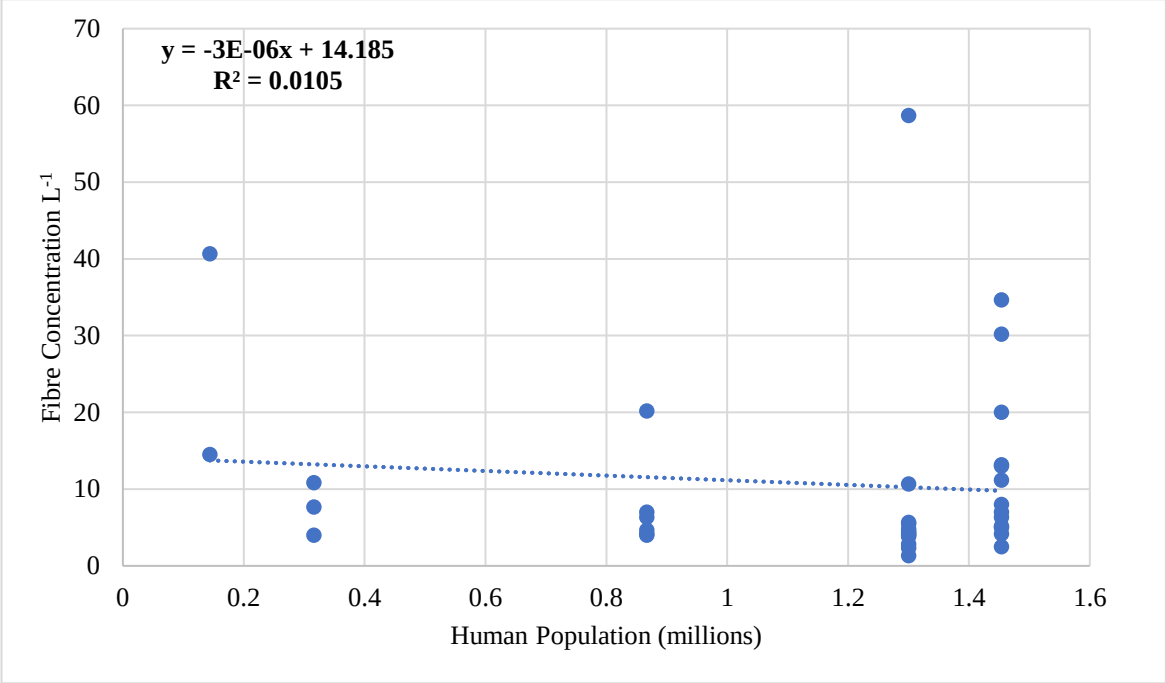


Figure 3.10: Scatter plot showing relationship between MF concentration and human population in sampled locations.

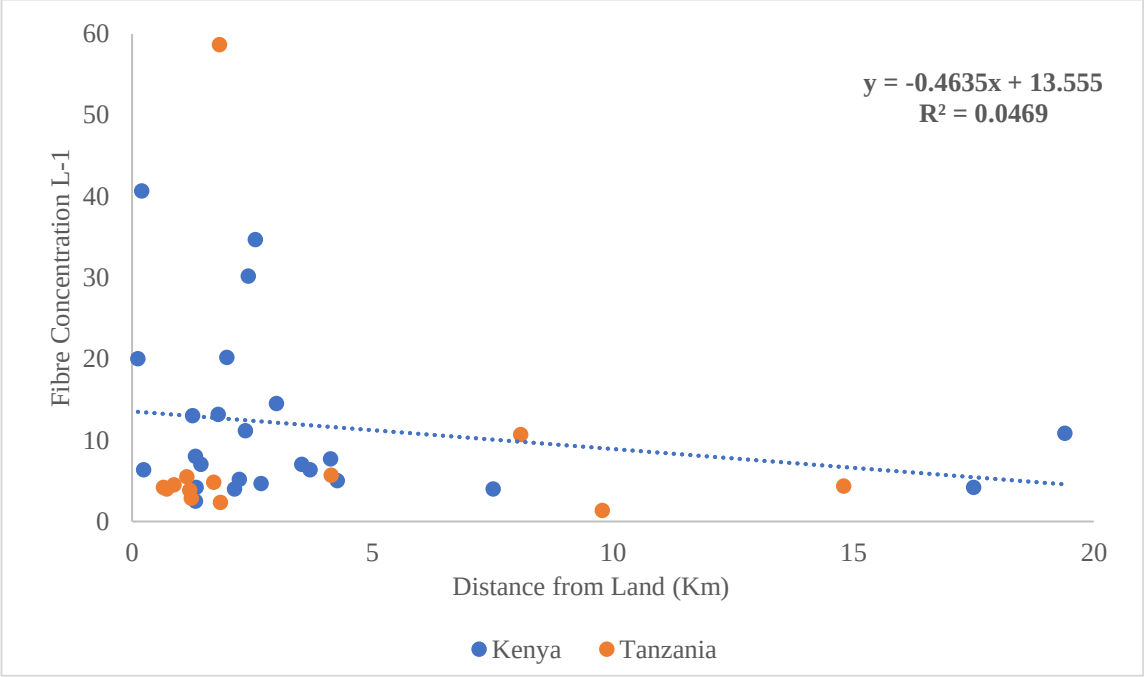
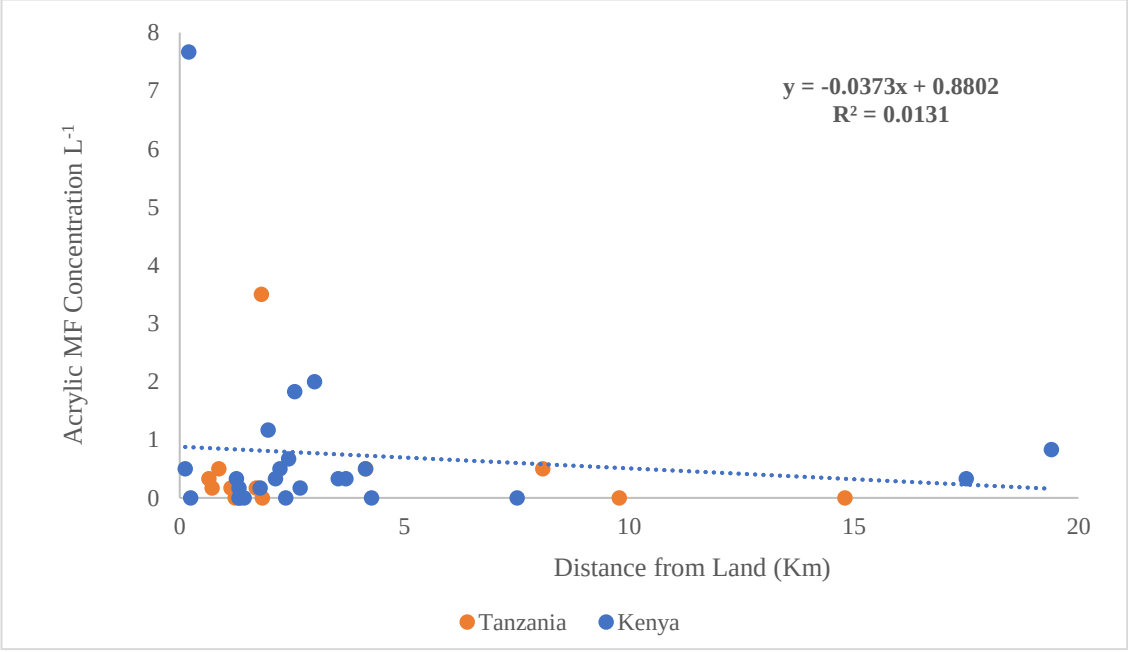
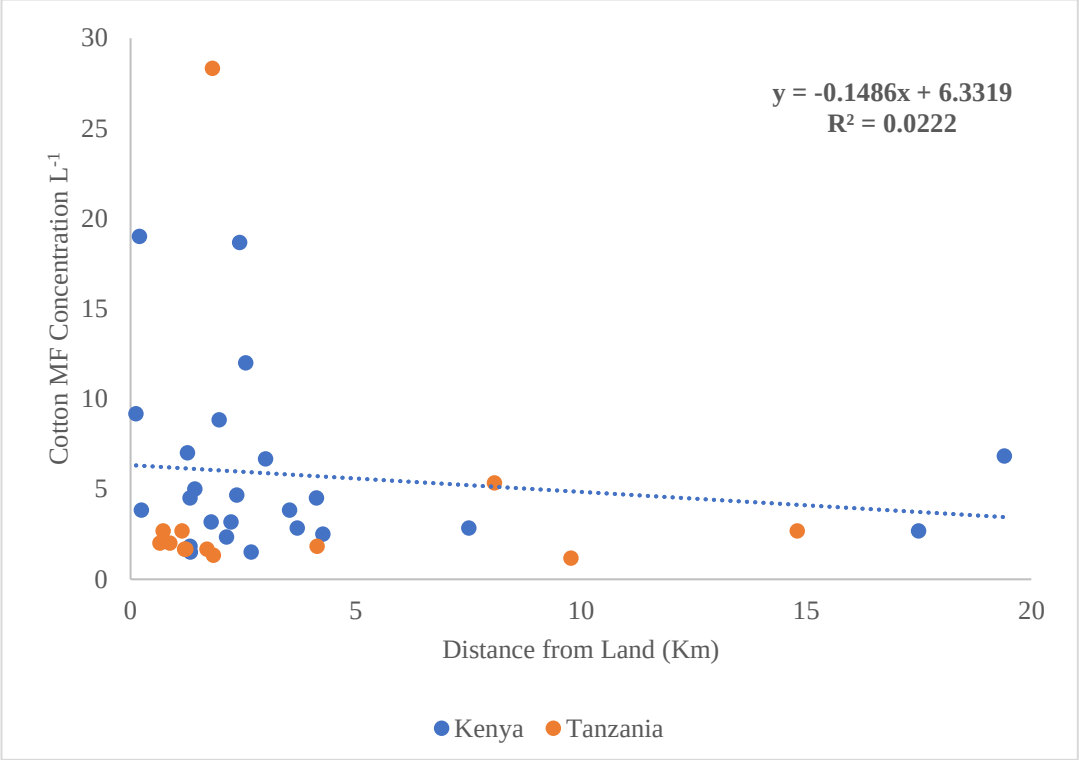


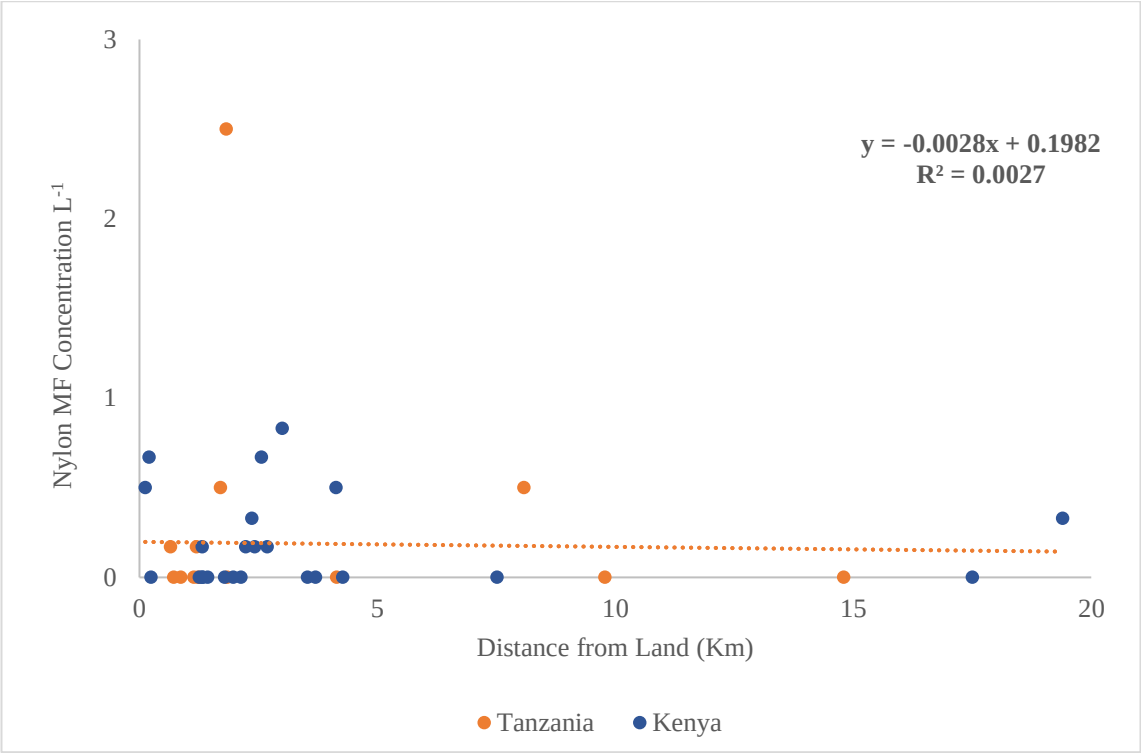
Figure 3.11: Scatter plot showing relationship between MF concentration and distance of sampled locations from land.



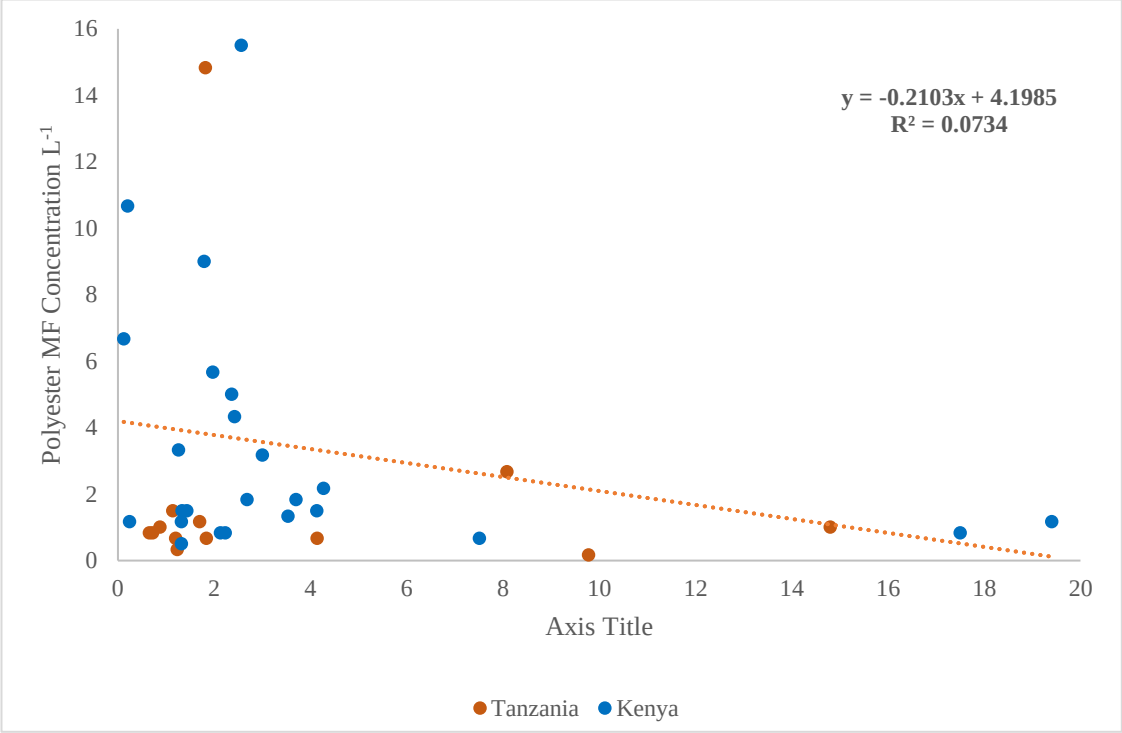
a) Acrylic MFs (p=0.47)



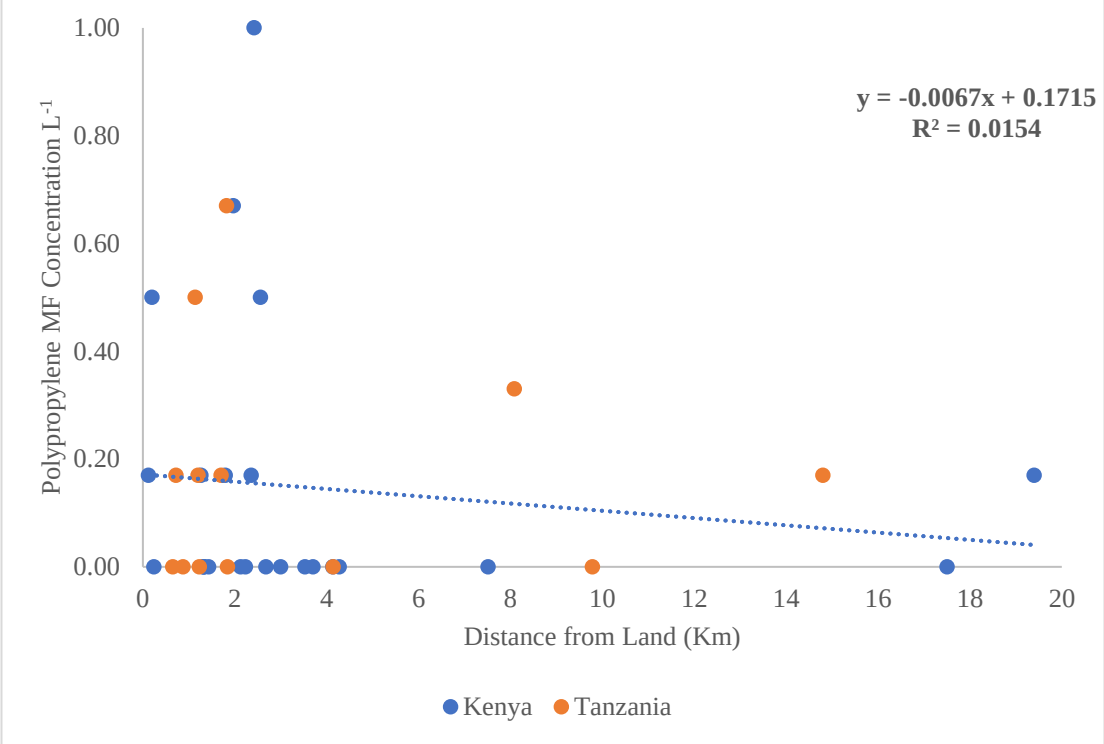
b) Cotton (p=0.49)



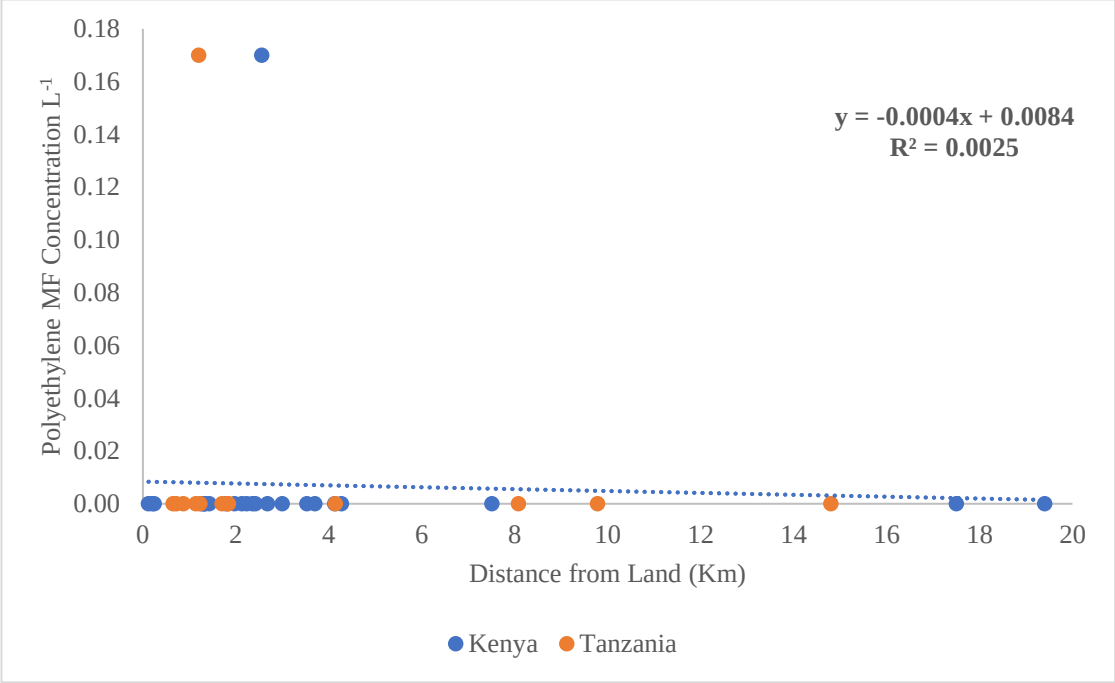
c) Nylon (p=0.64)



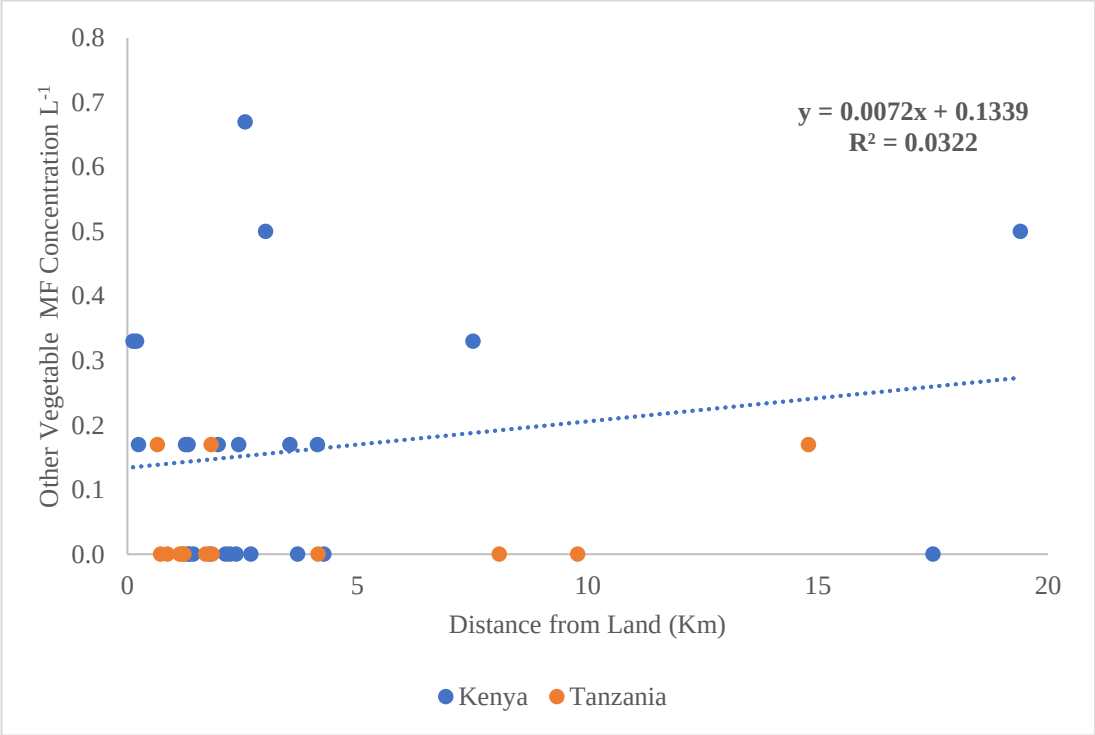
d) Polyester (p=0.21)



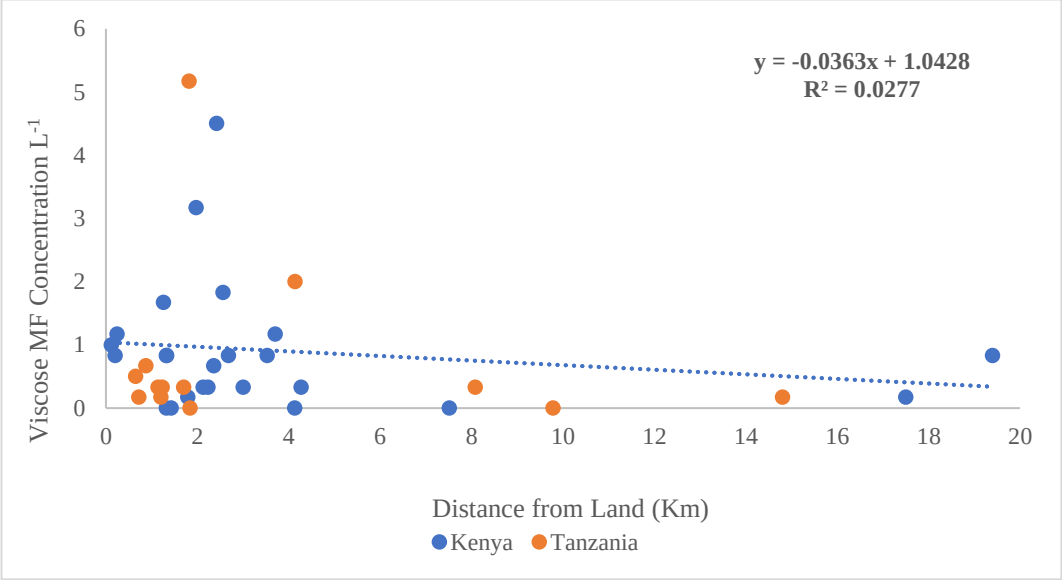
e) Polypropylene (p=0.56)



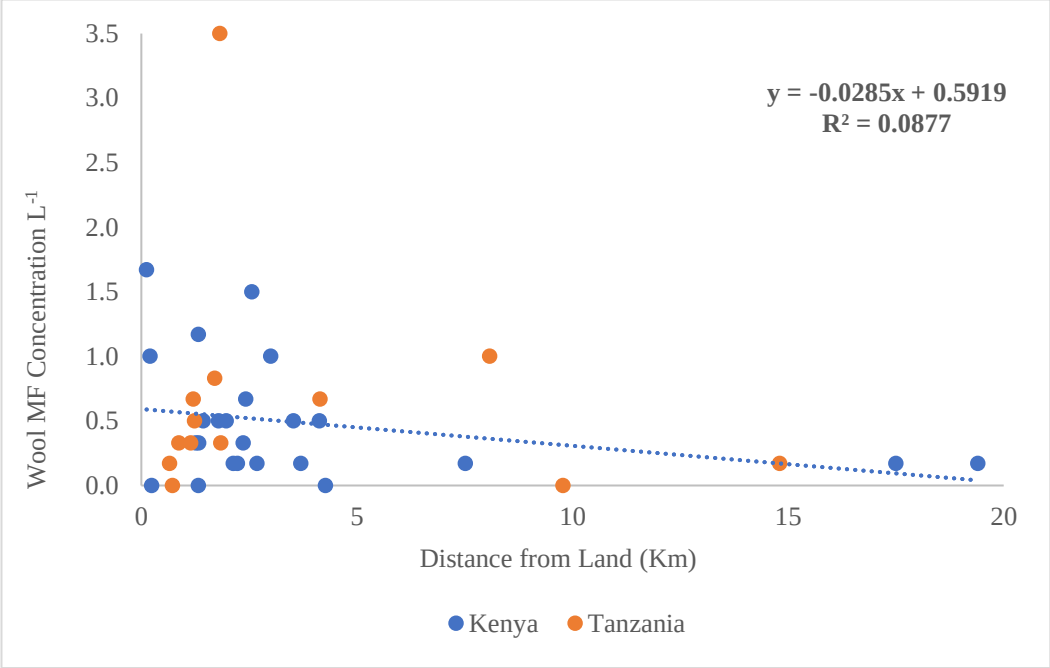
f) Polyethylene (p= 0.57)



g) Other Vegetable (p=0.31)



h) Viscose (p=0.34)



i) Wool (P=0.21)

Figure 3.12: Scatter plots showing relationship between various MF types identified in the 37 sampled locations and distance of sampled location from land.

3.4 Discussion

MF concentrations found in this study ranged from 1.33 fibres L^{-1} to 58.67 fibres L^{-1} per sampled location with a mean concentration of 10.73 ± 1.99 fibres L^{-1} calculated across all locations. Barrows et al. (164) sampled five major ocean basins and found that worldwide marine surface waters contain an average of 11.8 ± 0.6 particles L^{-1} and specifically found an average of 4.2 ± 1.2 particles L^{-1} in their Indian ocean samples. These figures include clear/transparent particles, which made up 47% of the examined samples, and 9% of total particles were non-fibrous, making the findings in this present study double their recorded amounts as this present study did not include clear/transparent MFs. Differences in sampling methods may account for differences in data (6, 276). Differences in sampling volume, where they sampled 1 L versus 6 L in this study, might have an influence on findings. Another study of ocean basins involving 617 locations across 6 oceanic basins also analysed samples from the Indian ocean (IO) (5). Although the average concentration of MF in their IO samples was not given (rather median), the range of concentration per litre across sampled sites are comparable with data found in the present study. However, the data obtained in this present study and from the Southwestern Atlantic Ocean where a mean concentration was calculated to be 10.6 ± 5.3 fibres L^{-1} are one of the highest recorded MF concentrations in published literature (277).

As clothes are hand washed near the coastline, the effluent finds their way into the water body without being filtered through filtration processes applicable in WWTP. It is therefore unsurprising to see high concentration of MFs in locations sampled here. Insufficient management of waste products in the region is also expected to contribute to high levels of concentration (184). However, the absence of a statistically significant relationship in MF concentration and human population and proximity to land is surprising given anthropogenic activities. Nonetheless, a trend shows there is a general increase in microfibre concentration as closer to land. Recovery of replicate samples may have been able to produce more

accurate concentration data. A study in South Africa found a statistically significant relationship between MF abundance and access to piped water infrastructure when they analysed water and sediment samples collected in November of 2014 along south-eastern coastline of South Africa (278). Though no positive correlation was found in that study between population density and MF abundance. Areas without access to pipe water had to do their laundry on the coast which meant that MFs were introduced directly. According to a 2017 survey, more than half of Nairobi residents' hand-wash their clothes and dump the wastewater on the ground. Because of runoff and wind influences, some of these MFs eventually end up in the aquatic environment.

In another study of the South African coastline (279), focused on different locations from a previous study two years earlier, researchers found no significant relationship existed between study sites and MF concentration. However, two harbours were exceptions to their observation as a significant correlation was observed. A combination of the enclosed nature of harbours and the level of anthropogenic activity that goes on means that MFs that are released are retained for longer periods compared to other parts of the coast. Although Suaria et al. found high concentrations of MFs in the Mediterranean Sea correlating to its dense population (5), the Southern Ocean, with little or no anthropogenic activity was found to have high concentration of MFs attesting to the fact that other factors beyond anthropogenic activities influence MF concentration such as temperature, salinity, currents and winds influence MF distribution (36, 37, 280) .

Natural fibres were found to be in greater abundance compared to man-made fibres i.e synthetic and regenerated cellulosic. A study carried out on six oceanic basins also found natural fibres to be more abundant (5). However, the proportion recorded in that study was greater than what was found in this present study. Partly, because all cellulose-based fibres were grouped together as it is not always straight forward discriminating them using μFTIR. The inclusion of optical microscopy to characterise fibres enables the discrimination of

natural fibres based on their morphology. This has enabled discrimination of cellulosic based fibres and provided more accurate data. However, there was a similarity in the proportion of cotton MFs. If viscose (regenerated cellulose) is grouped with natural MFs in this study, the group formed would compose 29 - 88% of recovered MFs across the 37 samples sites. This data is congruent with the result from surface samples recovered from North-West Mediterranean Sea which showed a range of 35-72% when cellulose based MFs were grouped as natural fibres (281).

Cotton was the most abundant and prevalent MF found in this study. This is congruent with studies carried out over the years in forensic science. Using techniques that efficiently discriminates different fibres types, they have found cotton fibres to have the highest abundance in both indoor (282, 283, 284, 285) and outdoor surfaces (286), including human skin (222). As discussed in Chapter One, the sheddability of textile materials largely determine how readily its fibre contents are released to the environment. Cotton fibres are characterised with low abrasion resistance, high hairiness, and low yarn breaking strength as they are staple thereby making them to be readily shed from materials (156). These features make it more readily sheddable compared to other commonly used fibres like polyester (124, 287). The ubiquity of cotton fibres may also be explained by the facts that they are usually blended with other fibre types, mostly consists of 100% material and are most common fibre type weighted by their proportions in blended garments (polyester follows directly after cotton in these statistics) (288). The dominance of cotton as a common base fibre is also reflected in product information found on clothing offline (289) and online (288).

Although cotton fibres are expected to be readily biodegradable in nature (290, 291) and evidenced in laboratory experiments (287), their sheer prevalence in the environment suggests this is not the case. Other factors such as finishes used during textile production may be inhibiting the biodegradation process (292). Furthermore, the effect of processing cotton fibres results in the change of its chemical structure so it is no longer in a 'natural'

state and thus takes longer to degrade (293, 294). Zambrano et al.(292) found that dye finishes did not inhibit degradation of fibres from dyed cotton fabrics in their study, whereas fibres from cotton fabric treated with durable press had the longest degradation time within the period of experimentation. Durable press is a type of finish added to textiles especially cellulose and cellulose blend fabrics during production (295, 296, 297). This finish improves the material by reducing swelling and shrinkage, enhances wet and dry wrinkle recovery, smooths appearance after drying, retains intentional creases and pleats (295, 296).

Nonetheless, one of the issues with biodegradability of processed natural fibres is that as they degrade, the chemicals contained within are released to the environment. For example, Reactive Blue 19 dye was released into the water during the biodegradation experiment by Zambrano et al. (292). As highlighted in chapter two, reactive dyes form covalent bond between the hydroxyl groups in cellulose. During enzymatic hydrolysis, this bond is broken thereby releasing the Reactive Blue 19 dye (292) which contains an anthraquinone nucleus as its chromophore (determines the colour of a dye; see chapter two). Anthraquinone has been found to be toxic and therefore poses a threat to aquatic biota (298). On the other hand, chemicals used in durable press such as formaldehyde are also a toxicological threat to the aquatic environment (296).

Wool fibres were found in 32 out of the 37 sampled location attesting to its prevalence. Wool has been found to be less degradable compared to cellulosic fibres (299). Furthermore, laboratory experiments have shown that wool fibres are more degradable in soil compared to aqueous medium (300). The implication being that wool MFs may be more of a threat to aquatic biota than their terrestrial counterparts.

The fact these natural MFs are intentionally excluded from majority of the MF pollution studies as they are not easily or not able to be identified by routinely employed techniques such as FTIR, underestimates the toxicological threat facing the marine biota. The eventual

implication being that timely solutions would not be implemented as these natural MFs degrade faster than their synthetic counterparts.

About 80% of all analyzed MFs in this study comprised of black/grey, blue and red. This agrees with many marine pollution studies (5, 280) as well as in forensic studies (285, 301). Yellow colour was one of the least occurring in this study but a study of aquatic MFs in South Africa found a relatively significant amount of yellow fibres in sediment and water samples (278). As noted in a forensic study investigating the background population of fibres on knife blades, yellowing of colourless fibres due to exposure to dirt and rust could increase the proportion of fibres categorized as yellow. The tendency of this happening to fibres in water and sediments is quite high because the environment is surrounded by dirt and conditions that lead to rusting. Black, blue and yellow fibres were also found in atmospheric deposition over EIO (171). The study at Burdwood bank (277) also found blue and grey fibres. However, they categorised grey and black separately rather than grouping them which is a more common approach. As a result, the percentage of black fibres in their study was low. This is contrary to prevalent data. Black/grey are usually grouped together as it is not easy to decipher if the greyness is as a result of dye loss in a black fibre.

Studies have shown that aquatic organisms may mistake their diet for fibres possessing the same colour as their food. For example, Amberstripe scad (*Decapterus muroadsi*) (302) and (*Girella laevifrons*) (303), tend to ingest blue and red fibres respectively as these have the same colour as their natural diets. The Kenyan coast is home to aquatic flora that come in the same colour as the prevalent colours found in anthropogenic fibres such as the red *Halymenia spp* (Figure 3.13a) and *Gracilaria spp* (Figure 3.13b), the green *Caulerpa spp* (Figure 3.13c) and the *Sargassum spp* (Figure 3.13d) (304, 305). The implication is that since organisms have a propensity to mistake these fibres for food, the more knowledge that is accessible about the distribution of different MF colours in different environments, the better predictions that can be made about which fauna are at a higher danger.

A

B

C.

D.

Figure 3.13: Images of seaweeds resembling coloured MFs A) *Halymenia spp* (306) B) *Gracilaria spp* (307)C) *Caulerpa spp* (308) and D) *Sargassum spp* (309)

Control samples from clothing worn by the sampling crew were not taken and so could not be used to eliminate potential contamination. A forensic approach through discrimination by fluorescence microscopy was utilised to investigate potential contamination of blue cotton MFs from their blue T-shirts. Different groups of blue coloured fibres were formed attesting to variety of sources. The hypothesis is that one of the large groups may have originated from the blue T-shirts. For each instance where these groups were deducted from the total recovered fibres, natural MFs were overall still higher in abundance than man-made MFs.

The efficiency of discriminating blue cotton fibres using fluorescence microscopy has been shown in forensic studies (229, 234, 310). The use of an added light filter such as green used by Biermann (234) could discriminate these larger groups further. Nonetheless, studies have shown that a combination of fluorescence microscopy and UV-Vis MSP leads to greater discrimination (225, 229). This is an indication that any contamination which may have occurred is negligible. Furthermore, additional discrimination with UV-Vis MSP and comparison microscopy would further reduce the group size. A Microspectrophotometer is an instrument with a microscope and spectrophotometer. The latter measures the intensity of light absorbed or reflected by dyes contained in fibres. Different dyes will absorb or reflect different colours of light, resulting in a characteristic spectral pattern. Grieve, Biermann and Davignon (311) in their study showed that dyes with the same generic name produce similar spectral curves. In 97% of the cases they studied, it was relatively straightforward to determine the dye class from the spectrum.

Tores et al. (194) and, more recently, Gwinnett and Miller (183) have demonstrated that contamination can be reduced by at least 36.9% during both sampling and processing of samples when strict quality assurance and controls are followed. Future study might benefit from highlighting this issue by offering clothing in somewhat uncommon hues like orange and purple, as well as being careful of the garment's rate of shedding for those participating in sample collecting.

When adopting a citizen science approach in sample collection, it is crucial to consider simplicity and repeatability. The straightforward and relatively easy to use sampling device, adapted from a commercially available AeroPress® made sample recovery for the citizen scientists practicable. This device did not require extensive expertise, allowing for focus on the experimental procedure to maintain high-quality standards and reliability of collected data. The effectiveness of this approach is demonstrated by the positive scientific outcomes of the study.

3.5 Conclusion

Research quantifying the amount of MFs in marine environments have greatly increased in the past decade especially following the work of Browne et al. (3) in 2011. However, there has been a focus on synthetic MFs because this research trajectory was founded on microplastic studies. As a result, the methods employed are usually not suitable for the recovery and identification of other MF types namely, natural and regenerated cellulosic fibres, resulting in inaccurate data. Prior to the emergence of the use of fibres in the context of environmental pollution, the discipline of forensic science had already been investigating fibres and has developed effective procedures that prioritise methods capable of identifying all types of fibres. This chapter presents research that utilizes these techniques to quantify and identify all MFs retrieved from surface waters located in the Kenyan-Tanzanian coast, an area that has not been extensively studied before.

Natural MFs were found to be the highest occurring fibre type, followed by synthetic fibres and lastly, regenerated cellulosic fibres. More specifically, cotton was the highest occurring MF followed by polyester. The findings in the research are congruent with studies carried out over the years in forensic science that consistently demonstrate the abundance of natural fibres in the environment. The ease with which cotton fibres are shed from garments implies that they are always available to be transported and deposited in the environment.

The sheer presence of cotton and wool in the sampled locations indicates that factors such as finishes during production are inhibiting biodegradability. Even when they biodegrade, they release harmful chemicals to the environment endangering biota. The studies excluding this category of fibre are underestimating the number of MFs polluting the marine environments and undermining the impact of these MF types thereby not confronting the toxicological threat posed by them.

This study has demonstrated that microscopic techniques used in forensic examination of fibres are fit for purpose. All fibre types were able to be identified by employing high power

microscopic and PLM as it is able to differentiate both natural and man-made fibres. Future research would benefit from prioritising microscopy as it is non-destructive and equally discriminative of all fibre types.

Chapter Four

Assessment of Filter Size and Sampling Depth Effects on Microfibre Quantification and Distribution in Freshwater Environment: The Lake Victoria Example

4.1 Introduction

Identification and Quantification of Freshwater Microfibres

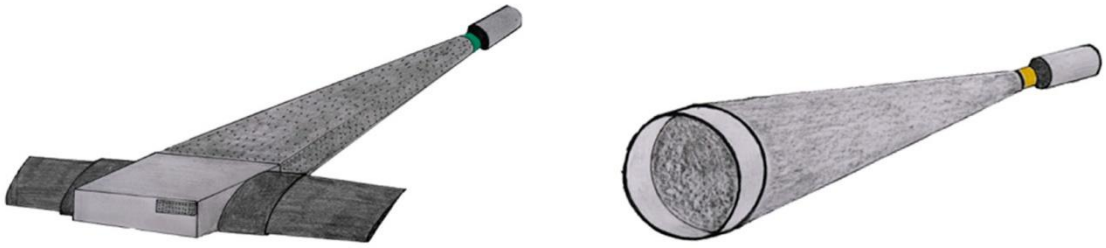
Lakes are one of the pathways that transport MFs into seas and oceans; consequently, studying them is important (169, 312). Determining the anthropogenic MF content of lakes and rivers may thus provide insight into what is to be expected in the marine environment. The growing literature and emerging evidence of MF pollution reiterates the threat they pose to both marine and freshwater biota. Until recent times, studies quantifying anthropogenic MFs had centred on the marine environment (6, 18). However, this has now seen a tremendous increase. As at 2021, there were about 100 published studies on Lakes according to a review (313). Majority of the studied lakes were in Asia (China), Europe (Italy) and South America (Argentina).

Studies have shown the presence of MFs on surface and near-surface lake water (314, 315), in lake's green algae (33) water column (28) and lake sediments (316). It is important to note, however, that these, like marine studies, are primarily concerned with quantifying and identifying synthetic MFs. Yet, a recent freshwater study (69) provides evidence that natural MFs originating from textile fibres dominate freshwater MF population. Therefore, the need to correctly identify and quantify MFs cannot be overemphasised. To ensure accuracy of data from studies in MF pollution, it is paramount that methods used in sampling are effective and efficient (6).

Earlier studies involved the use of manta trawls, neuston and plankton nets (Figure 4.1) with mesh sizes that ranged from 300 μm to 500 μm which were adapted from microplastic

research (6, 313, 317). These work by filtering large volumes of water as they are being towed in a body of water. Their effectiveness has been questioned as a result of the coarse mesh size which means that MFs are flushed out through the pores during towing (317, 318). However, current studies now employ trawls and nets with smaller mesh sizes, such as 20 µm (205) or 50 µm (29) to increase their efficacy. Yet, grab sampling, as opposed to using a tow net, is reported to collect more varied microplastics and minimises the likelihood of contamination (319). Grab sampling involves collecting relatively small volumes of water in non-netted materials such as glass jars (28) bottles (168) ,Van Dorn sampler (320) etc. and filtering them with choice of filter such as Whatman filter of 0.45 µm (164, 321), 1 µm (210) or stainless-steel sieve 20 µm (322). MFs generally have a diameter of around 10 to 20 µm (200, 227), a sampling method ought to be suited to capture and retain them.

A study compared samples collected using a Van Dorn sampler and then filtered using a 0.22 µm filter with samples collected with a 60 µm plankton net and found the former yielded more concentration of MFs (320). Unsurprisingly, more MFs are recorded with finer mesh size.



(A) (B)

Figure 4.1: Types of sampling devices used in MF study (A) Manta net and (B) Plankton net (323)

Combining two or more strategies concurrently to achieve accurate representation of freshwater MF pollution can improve estimation of global data (276). However, the simplest

technique to evaluate the effect of filter size is to run the same sample through successively finer filter (276).

Microfibre Pollution in Lake Victoria

Lake Victoria is the largest freshwater lake in the world after Lake Superior in North America and Africa's largest lake, with a surface area of 68,800 km^2 (324). Lake Victoria is 412 km long from north to south, between latitudes 0°30' N and 3°12' S, and 355 km long from west to east, between longitudes 31°37' and 34° 53' E. It reaches a maximum depth of 80 m and an average depth of 40 m. It is surrounded by Kenya (6%), Uganda (45%), and Tanzania (49%), and has a volume of 2,750 km^3, a coastal length of 3,440 km, and a catchment area of 184,000 km^2 (324) . It supports over 30 million people which invariably means it would be impacted by anthropogenic activities. In addition to eutrophication and excessive fishing, microplastic pollution has been shown as one of the threats facing the Lake (325, 326, 327). Some of the pollution sources in this lake have been identified as goods lost during transit, discarded fishing gears, and water runoff (325). Although clothes composed of only 2% of solid waste recovered in this study (325), these materials disintegrate into MFs and become ubiquitous.

As at time of writing (October 2023), to the best of the author's knowledge, only three peer reviewed studies have been published specifically addressing microplastic pollution in Lake Victoria. In a pioneer study, Nile perch and Nile tilapia were examined in 2015 for the presence of microplastics as a means of monitoring the presence of microplastics in Lake Victoria (326). This study did not report the presence of MFs as it was not the aim of the study. Subsequent studies investigated surface water (327) and lake sediment (328) and employed methods limited to examining synthetic MFs.

The adoption of a forensic approach that prioritises the use of microscopic techniques in the identification of all MF types, as documented in Chapter 3, has been found to be fit for purpose. The study presented in this chapter employs this approach to identify and quantify

MFs present in the water column of Lake Victoria. Additionally, Chapter 3 confirms that global MF estimations is underestimated if studies neglect natural MFs. Consequently, this chapter investigates how sampling method could further underestimate MF data.

4.2 Materials and Method

Sites of Study

Thirteen locations along the lake (Figure 4.2) were sampled and map coordinates can be found in Appendix 2. Description of anthropogenic activities of sampled area are given in Table 4.1 The three border countries were represented during sample collection as follows 2 samples from Kenya, 7 from Uganda and 4 from Tanzania. The sites ranged from areas of obvious anthropogenic activities to areas with little or no apparent anthropogenic activities.

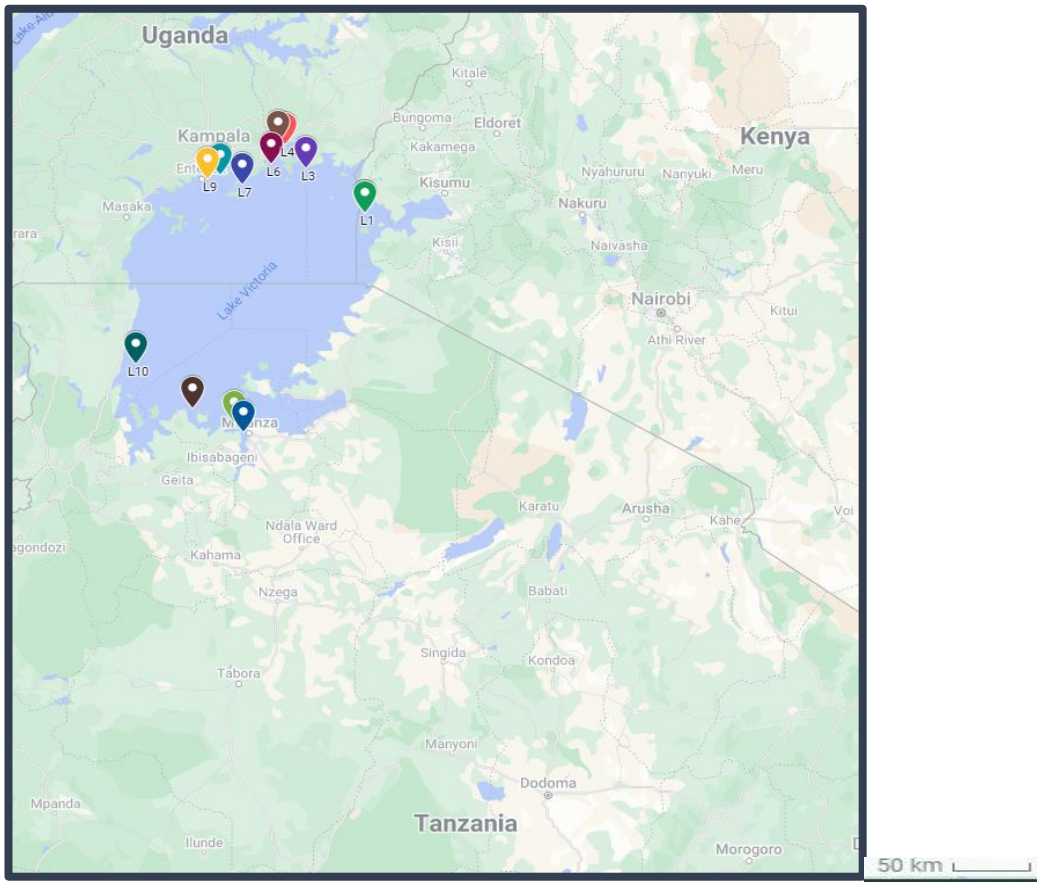


Figure 4.2: Map showing snapshot of sampled locations along Lake Victoria.

Table 4.1: Description of Anthropogenic Activities in Sampled Locations

Site ID	Country Location	Description
L1	Kenya	Area associated with tourist destination.
L2	Kenya	A small population that is dominated by fishing activities.
L3	Uganda	Associated with fishing activities.
L4	Uganda	Large scale fish farming and urbanisation.
L5	Uganda	Fishing, tourism, urbanisation and close to Nile River mouth.
L6	Uganda	Fishing, ecotourism, sparse population and small islands.
L7	Uganda	Near Kampala city, fishing village and surrounded by islands.
L8	Uganda	Close proximity to a city.
L9	Uganda	Close proximity to two cities.
L10	Tanzania	Remote island with sparse population.
L11	Tanzania	Long history of fishing and farming.
L12	Tanzania	Close proximity to a Gulf channel.
L13	Tanzania	Surrounded by rock island and close proximity to a city.

Sampling Device

Van Dorn sampling chambers modified to accommodate a filtration system were designed at Northumbria University Faculty of design by a member of staff (Figure 4.3). The Van Dorn water chamber has an internal diameter of 72 mm, a length of 500 mm and captures approximately 2 L of water when activated within any depth of the lake. Samples were collected at depths of 0, 1, 2, 4, 6, 8, 12, 16, 20, 30, 32, 34, 38, 49 and up to 50 meters depending on the maximum depth of sampled location. It works by capturing water when lowered in a horizontal plane. When water is captured at the required depth, the stoppers at the ends of the chamber shuts it, trapping the water collected at that particular depth in the clear water chamber ready to be brought to the surface by the lowering rope.

The filtration system houses Nylon filters made from 50 µm and 330 µm nylon mesh sheets (Plastok Associates Ltd, UK) that were laser cut into 50 mm diameter discs using a laser (Epilog Laser Legend 36EXT, UK). These filters are used to successively filter the grabbed water.

Figure 4.3: A picture of a Van Dorn sampler with modified filtration system

Anti-contamination Protocol Pre-Sampling

Following lessons learned from the marine water study (Chapter 3), added anti-contamination protocols were followed. Prior to the filters being used at the lake, these were eradicated of extraneous fibres (blanking) in the lab to avoid contamination of water samples during filtration. Nitrile gloves, hair net and white cotton lab coat were worn during this process. The work bench was thoroughly cleaned with wipes containing 70% alcohol. Following cleaning, the bench was lined with fresh brown paper. Using adhesive tape (TapeIt™, 3L Office, Denmark) both surfaces of each filter was blanked by pressing and rubbing the adhesive part of the tape on the filter's surface. This press and rub method were

also used to blank resealable clear Ziplock plastic bags before storing away the blank filters in them. Each blanked filter was examined under low power microscope (10x-40x, Leica S6E, Germany) to ensure no fibres remained. Any remaining extraneous fibres were removed with a pair of stainless-steel tweezers before storing in the blanked Ziplock bag. Once fibre-free, the filter was immediately stored in a clean, blanked Ziplock plastic bag.

Surface and Deep-Water Sampling

Water samples were collected between 3rd and 28th March 2021. During the FlipFlopi expedition of Lake Victoria, samples were collected from 13 sites on the lake using the specially designed Van Dorn sampler. Samples were collected from the lake's surface waters until the lake bottom was reached. The number of samples collected differed by location due to the depth of the lake. For example, the maximum depth collected at location 4 was 4 m whilst locations 7 and 10 had maximum depths of 50 m. Each water sample's depth was determined by utilising a pre-marked rope. Sampling time ranged from 30-45 minutes per station, depending on the weather and depth profile. During sampling, coordinates, temperature, wind speed, and direction were all recorded.

Water Filtration

Following water collection, the sampling device was brought on board and immediately connected to the filtration system. Water was filtered via two filter sizes, 50 µm and 330 µm, in this order: 330 µm first, then 50 µm. Filtration times ranged from 30 minutes to 1 hour each location, depending on the number of plankton and turbidity, which occasionally clogged the filters. Due to the enclosed nature of the Van Dorn sampler and filtration system, contamination by atmospheric deposition was controlled. Following filtration, each filter was immediately transferred to a pre-labelled clean petri dish and tightly wrapped in aluminium foil before being shipped to the United Kingdom for further analysis.

Fibre Recovery and Examination

Using a pair of stainless-steel tweezers (EM-Tec, 5 AM, Switzerland) coloured MFs were removed and individually mounted on glass slides (CIMED®, 1-1.2 mm thick, 25 x 75 mm) using Glycerol (VWR® CAS number: 56-81-5) as a mountant and covered with round cover slips (9 mm, Thermo Scientific®, Germany). Recovered MFs were identified using brightfield microscopy (x100-400) (Comparison Microscope, Leica DMR, Germany), followed by Polarizing Light Microscopy (PLM) where necessary (Leica DM2700P, Germany), as described in Chapter 3. Using an Olympus CX22 microscope coupled with Euromex camera with Image Focus 4.0 software, measurements of MF length and width were taken following calibration of system either in mm or µm.

Atmospheric Deposition Monitoring in the Laboratory

To prevent contamination of the samples from microfibres in the environment, strict laboratory protocols were followed during the microfibre recovery process. Since colourless cotton fibres were not examined, clean, white cotton laboratory suits made of colourless cotton fibres were worn. As the need to monitor possible contaminates was noted from study in Chapter 3, atmospheric MF deposition was monitored in the laboratory. After cleaning the work bench, upturned adhesive tape (TapeIt™, 3L Office, Denmark) was placed alongside the workstation. After a whole session of MF recovery was completed, the adhesive tape was fastened to a clear acetate sheet and examined for the presence of fibres. Atmospheric deposition was monitored in 58 of 61 laboratory sessions. On average, atmospheric MF deposition was 0.1 fibres per minute. Given the time it was taken to recover MFs from the filter and strict laboratory processes followed, the impact of atmospheric deposition is negligible and therefore MF count was not adjusted. Further proof of the negligible impact of atmospheric deposition was evidenced by the fact that no MFs were observed in 6 samples examined at different sessions. Recovery of MFs was carried out throughout under a microscope and filters were only exposed when actively examined. MFs

recovered from lake samples were significantly higher than atmospheric depositions (Figure 4.4) during MF recovery at the laboratory (U=174, Z=-3.56, P=.0004).

Statistical Analysis

Kruskal-Wallis Test and Mann Whitney U tests were used to determine statistically significant differences between variables. Wilcoxon signed ranked test was used for testing significant difference between number of MF recovered on the two filters as the same sample was evaluated under two different conditions. These non-parametric statistics were chosen as data were not normally distributed. All statistical calculations and graphs were conducted using either Microsoft Excel™ for Microsoft 365 Version 2205 or an online statistical calculator found at https://www.socscistatistics.com/.

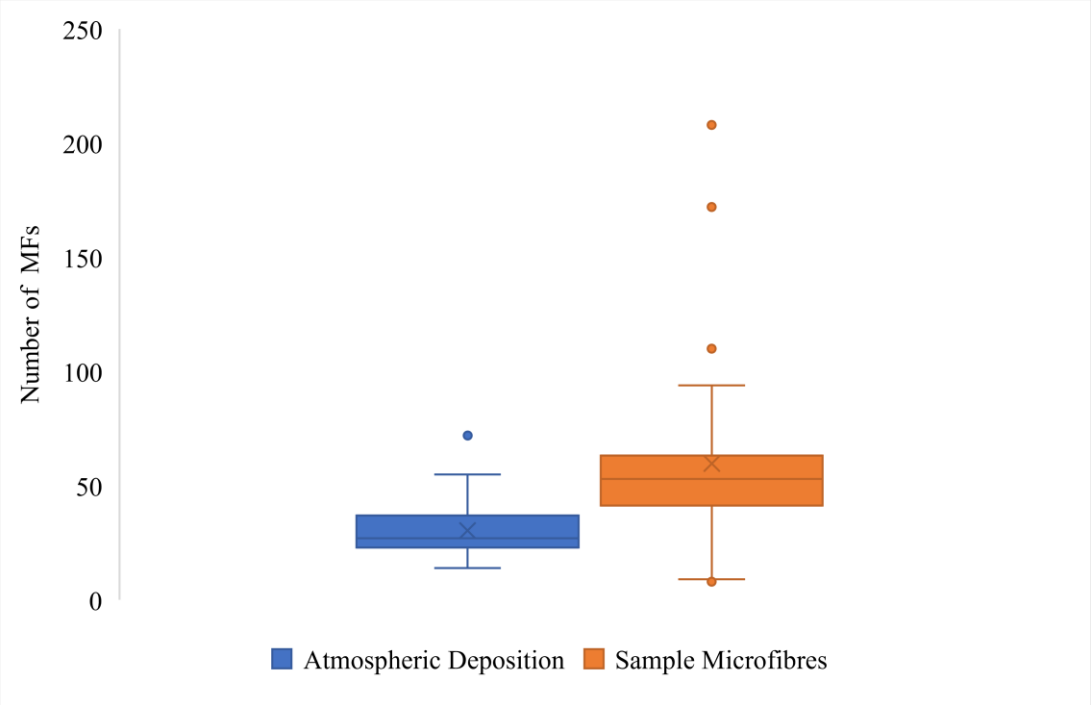


Figure 4.4: Box plot showing number of MFs recovered from lake samples vs. atmospheric deposition in the laboratory (n=58 laboratory sessions)

4.3 Results

Overall MF Distribution across Sampled Locations

MFs were present in all 13 locations sampled. A total of 1,888 MFs were recovered and discriminated based on generic type and colour except 30 MFs whose generic type could not be clearly determined using microscopic methods alone as they were either heavily dyed or with unclear features. Further analysis using µFT-IR would help determine the generic classification of these MFs but could not be carried out due to time constraint. Therefore, all statistical analysis carried out are based on the 1,858 fully classified MFs.

The mean MF concentration of the 13 sampled locations regardless of depth or filter size was determined to be 71.46 ± 10.44 fibres L^{-1} with a range of 37 fibres L^{-1} to 176.5 fibres L^{-1} per sampled location. Due to the fact that number of samples varied per location as a result of varying depths, total MF concentration per sampled location was normalised and data is shown in Figure 4.5. Sampled locations in Kenya had the highest concentration of fibre L^{-1} followed by Uganda then Tanzania.

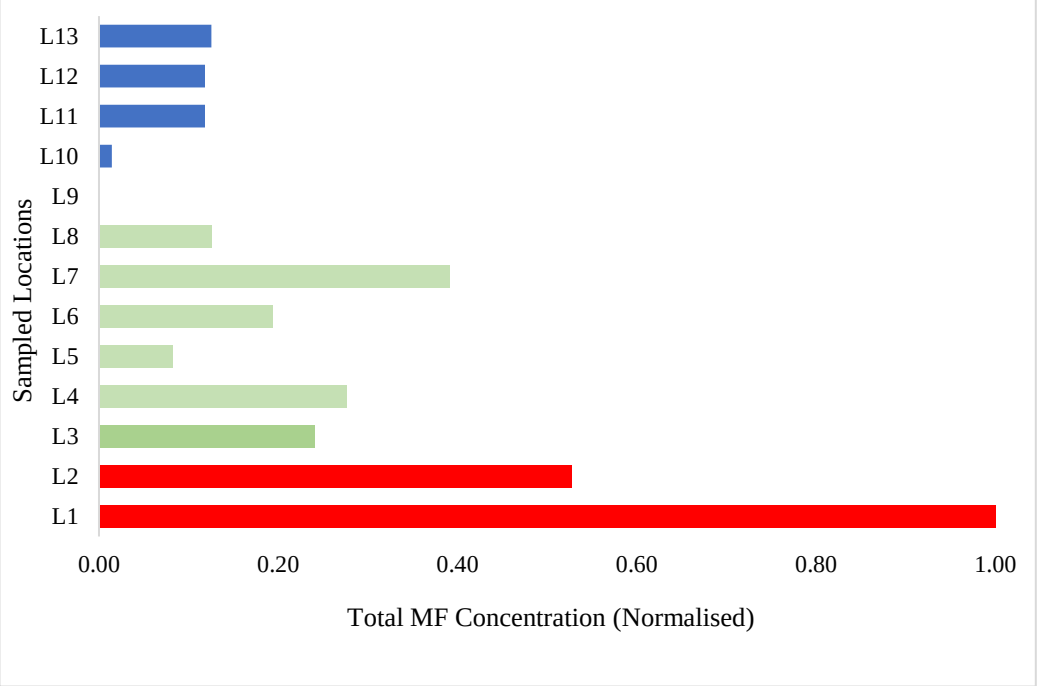


Figure 4.5: Total number of MFs recovered per sampled location with red colour signifying locations in Kenya, green for Uganda and blue for Tanzania.

Natural MFs had the highest concentration, followed by synthetic then regenerated cellulosic (Figure 4.6). Statistically significant differences were found between the distribution of the three fibre categories (H=26.07, p-value. < 00001).

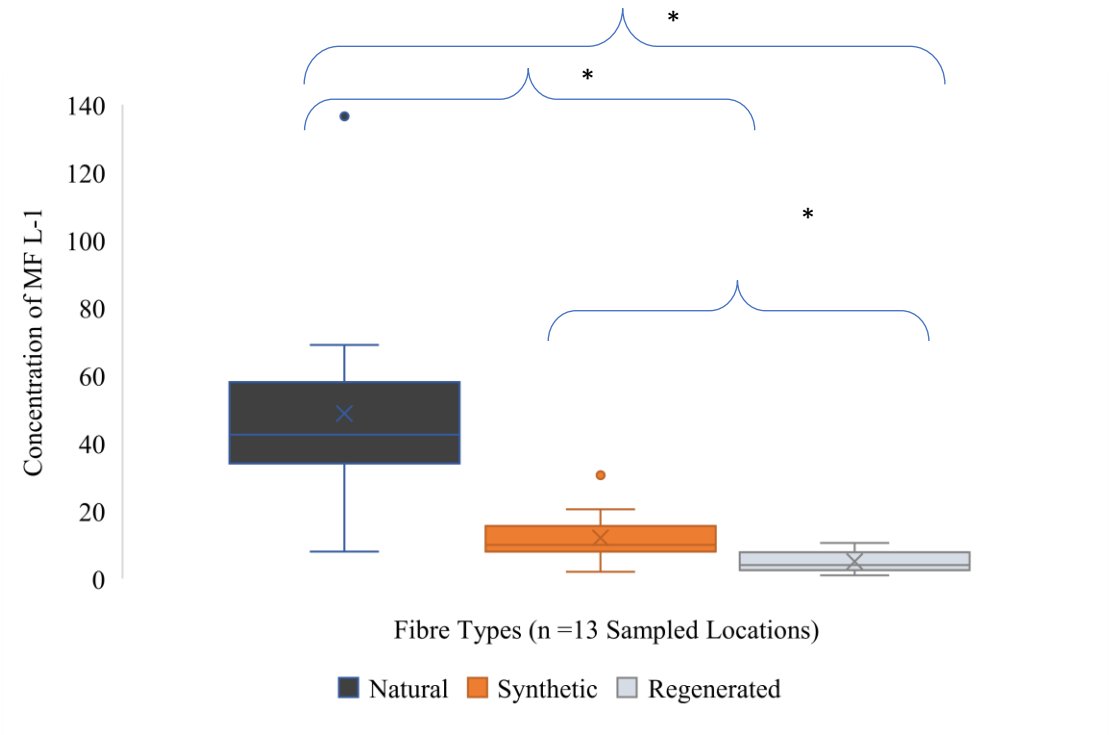


Figure 4.6: Concentration of natural, synthetic and regenerated cellulosic MFs distributed across the 13 sampled locations. * Statistically significant difference at α=0.05

Further analysis showed that natural MFs were statistically significantly more abundant than both synthetic and regenerated MFs (Z-Score = 3.69 and p < 0.001), respectively (Z-Score = 4.18 and p < 0.00001). When compared to regenerated MFs, synthetic MFs were found to be statistically more abundant (Z-Score = 3.03 and p=0.002).

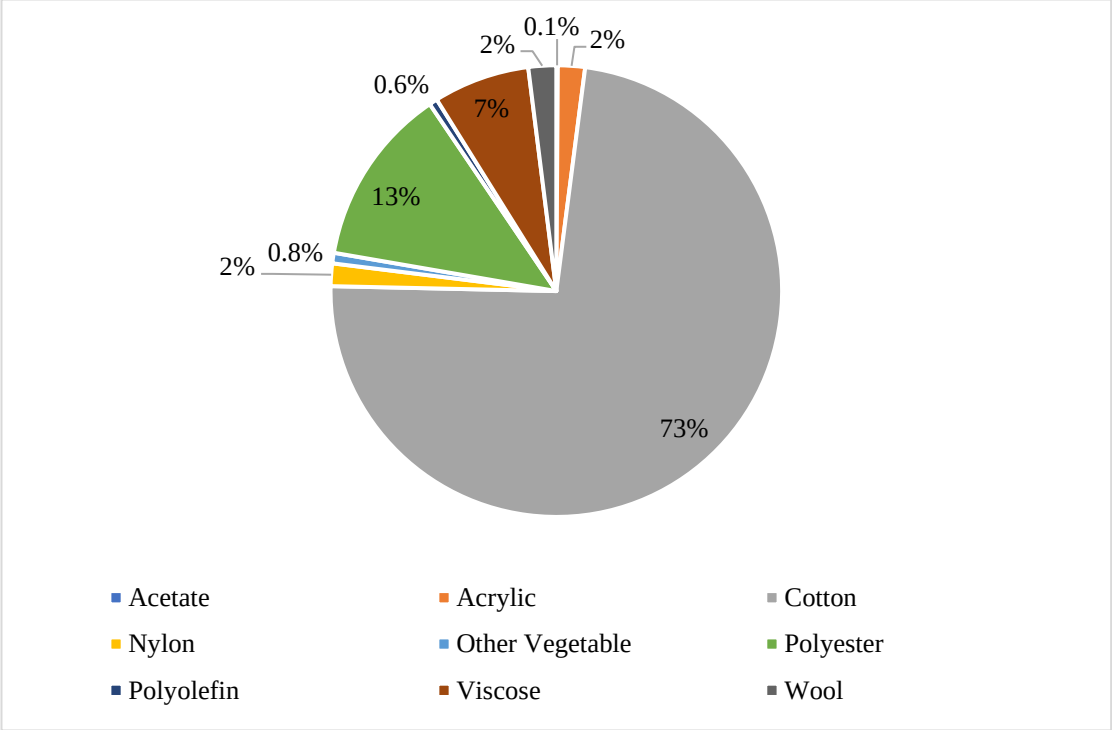


Figure 4.7: Percentage distribution of all recovered MFs across 13 sampled locations.

Across all 13 sampled locations, nine MF types were identified namely acetate, nylon, polyolefin, acrylic, other vegetable, viscose, cotton, polyester and wool. The MF type with the highest proportion regardless of filter type or sampled depth was cotton at 73%, followed by polyester (13%), viscose (7%) with the rest of the MF types comprising 7.5% of the total recovered MFs (Figure 4.7).

Effect of Filter Pore Size on Microfibre Quantification

50 µm filter captured more MFs compared to 330 µm filter across all sampled locations except in locations 11 and 12 where they were relatively comparable (Figure 4.8). There was an average of 34.62 ± 8.50 fibres L^{-1} and up to 76% difference between the number of MFs captured by the filters. The difference between the MF capture between the two filters was found to be significant ($Z = -2.97$, $p = .002$, $\alpha=0.05$).

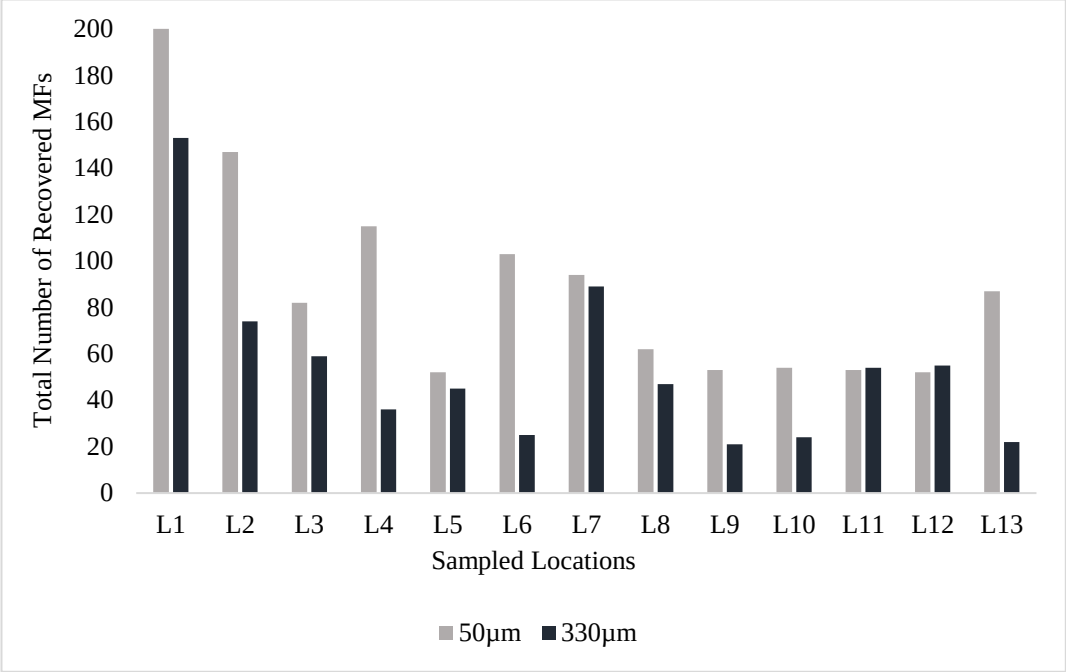


Figure 4.8: Difference in total number of captured MF between the two filters across the sampled locations,

Measurements of width and length of randomly selected MFs captured on 50 µm and 330 µm filters were taken (n= 77 and 21 respectively). The average length of MF captured by 50 µm was 1338.64 ± 138.29 µm and width 21.57 ± 0.93 µm. Whereas the average length of MF captured by 330 µm was 1903.4 ± 447.18 µm and width 17.23 ± 1.36 µm.

Effect of Sampling Depth on MF Distribution

Most studies investigate MF prevalence on surface waters, therefore, for ease of comparison between this study, the marine study in chapter 3 and the published literature, this section describes the abundance and distribution of MF types in terms of surface water (sampled depth of 0m) and subsurface water (1-50 m). The average concentration of surface water MF across all 13 sampled locations was determined to be 11.81 ± 1.47 fibres L^{-1}. The total concentration of MF per sampled location ranged from 4 to 22 fibres L^{-1}. In Table 4.2, the percentage proportion and relative abundances of MF prevalent on the surface water of sampled location are given in decreasing order of abundance.

Table 4 .2: Percentage proportion and relative abundance of MF types on surface water

Fibre Type	Category	Mean Concentration (Fibres L^{-1})	No. of sites found	Percentage of total fibres
Cotton	Natural	8.38 ± 1.16	13	71
Polyester	Synthetic	1.69 ± 0.39	13	14.3
Viscose	Regenerated Cellulose	1.05 ± 0.22	11	7.5
Nylon	Synthetic	1.00 ± 0.28	4	2.6
Wool	Natural	0.58 ± 0.06	6	2.3
Acrylic	Synthetic	0.50 ± 0.00	4	1.3
Other Vegetable	Natural	0.50 ± 0.00	2	0.7
Acetate	Regenerated Cellulose	0.50 ± 0.00	1	0.3

Cotton and polyester MFs were present in all locations sampled followed by viscose and wool. The rest of the MFs were not found on the surface water of up to 50% of the sampled locations. No polyolefin was found on the surface water of the sampled locations.

Table 4.3 shows the percentage proportion and relative abundance of MF types in subsurface water of sampled locations in decreasing order of abundance. The subsurface data captures depths of 1 -50m. The average concentration of MF in the subsurface of the sampled locations regardless of sampled depth is 59.65 ± 9.56 fibres L^{-1}, ranging from 21 to 154.5 fibres L^{-1}. In addition to the MF types found on surface waters, polyolefin was present in the subsurface water samples. The concentration of each MF across the water column is shown in Figures 4.9 -4.16. All MF types were found in higher concentration at the surface water of sampled locations compared to other depths below surface water. This correlation between fibre type and sampled depth was stronger for cotton, polyester and viscose.

Table 4.3: Percentage proportion and relative abundance of MF types in subsurface water

Fibre Type	Category	Mean Concentration (Fibres L^{-1})	No. of sites found	Percentage of total fibres
Cotton	Natural	44 ± 7.42	13	73.8
Polyester	Synthetic	7.69 ± 1.38	13	12.9
Viscose	Regenerated Cellulose	4.04 ± 0.75	13	6.8
Acrylic	Synthetic	1.60 ± 0.29	10	2.0
Wool	Natural	1.5 ± 0.15	10	1.9
Other Vegetable	Natural	1.20 ± 0.21	5	0.8
Nylon	Synthetic	1.14 ± 0.19	7	1.0
Polyolefin	Synthetic	0.92 ± 0.14	6	0.7
Acetate	Regenerated Cellulose	0.50 ± 0.00	1	0.1

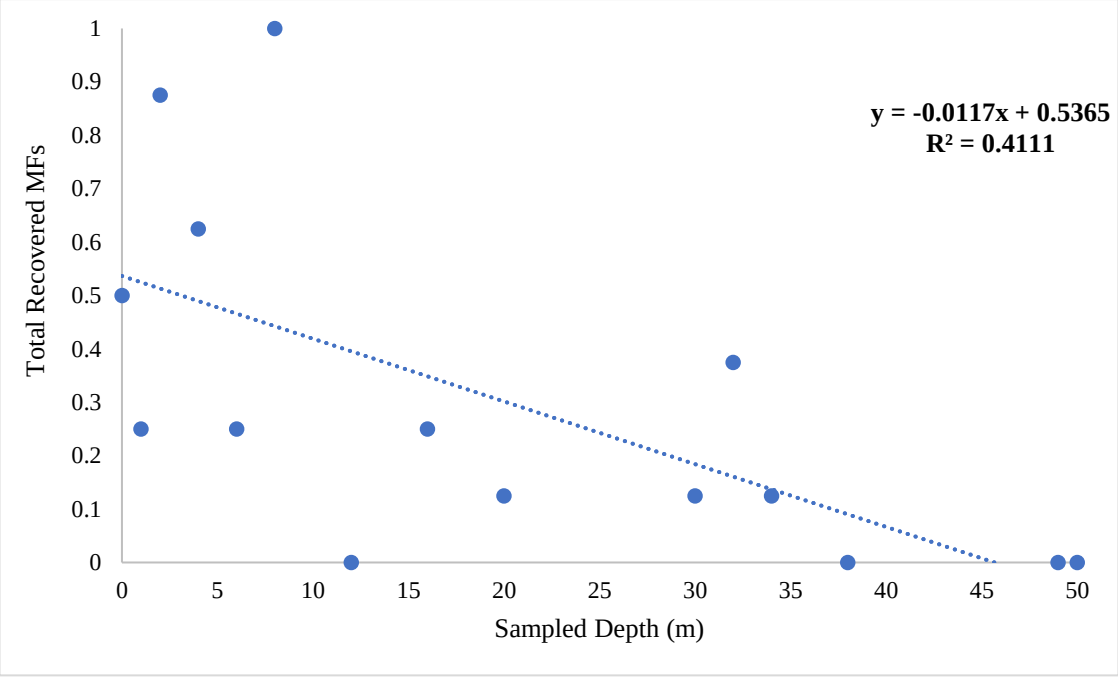


Figure 4.9: Correlation between water depth and Acrylic MF concentration

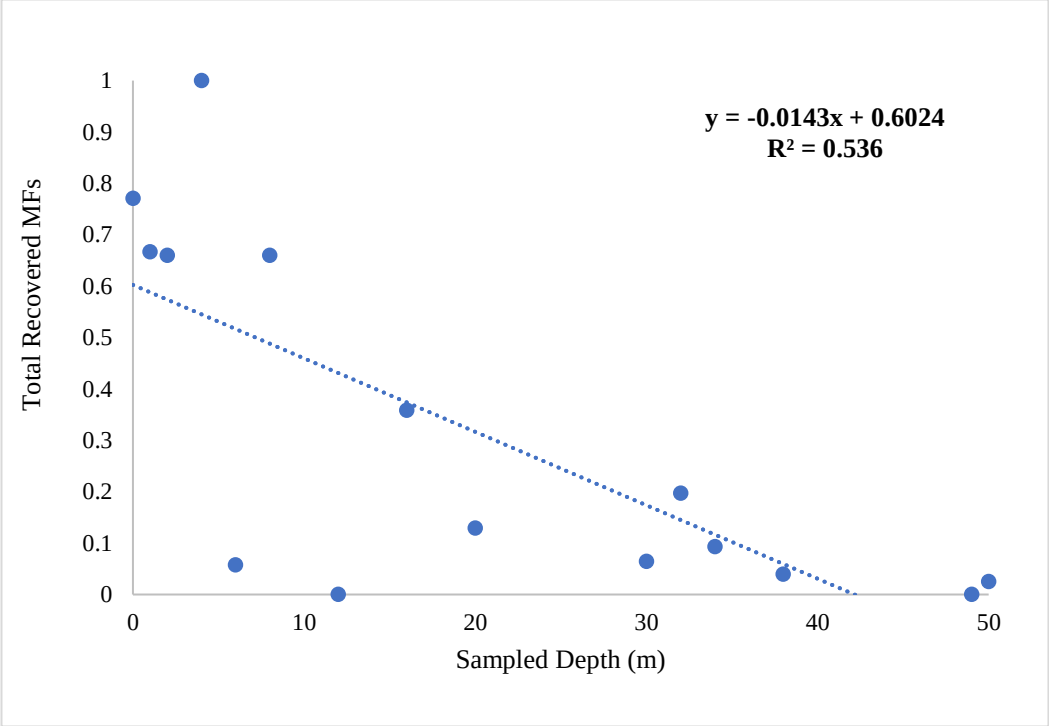


Figure 4.10: Correlation between water depth and Cotton MF concentration

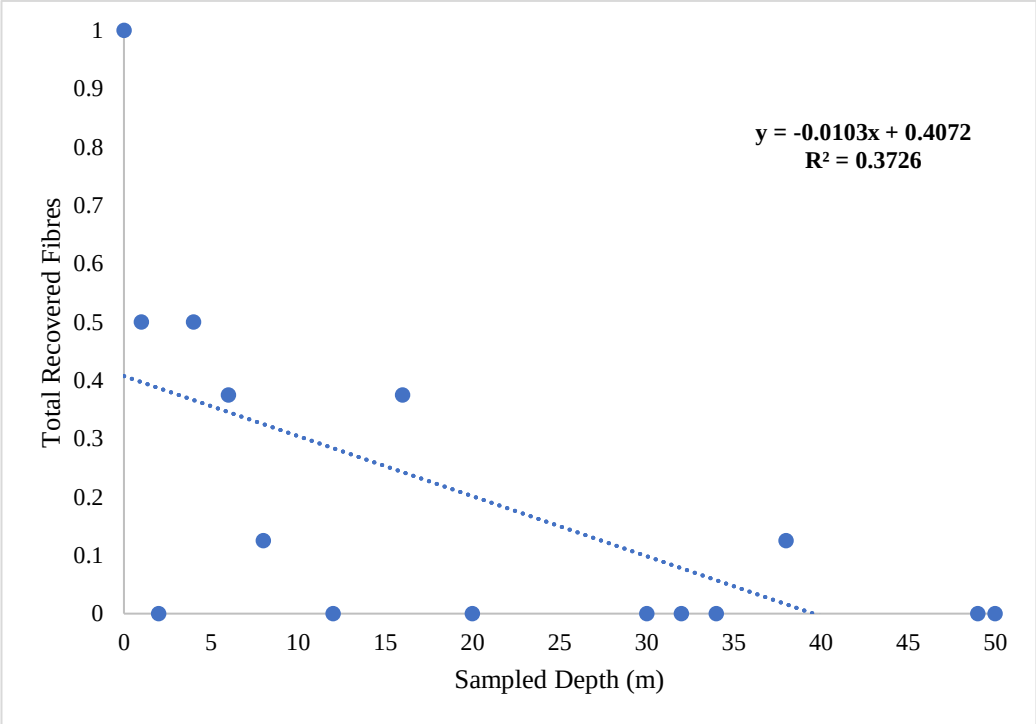


Figure 4.11: Correlation between water depth and Nylon MF concentration

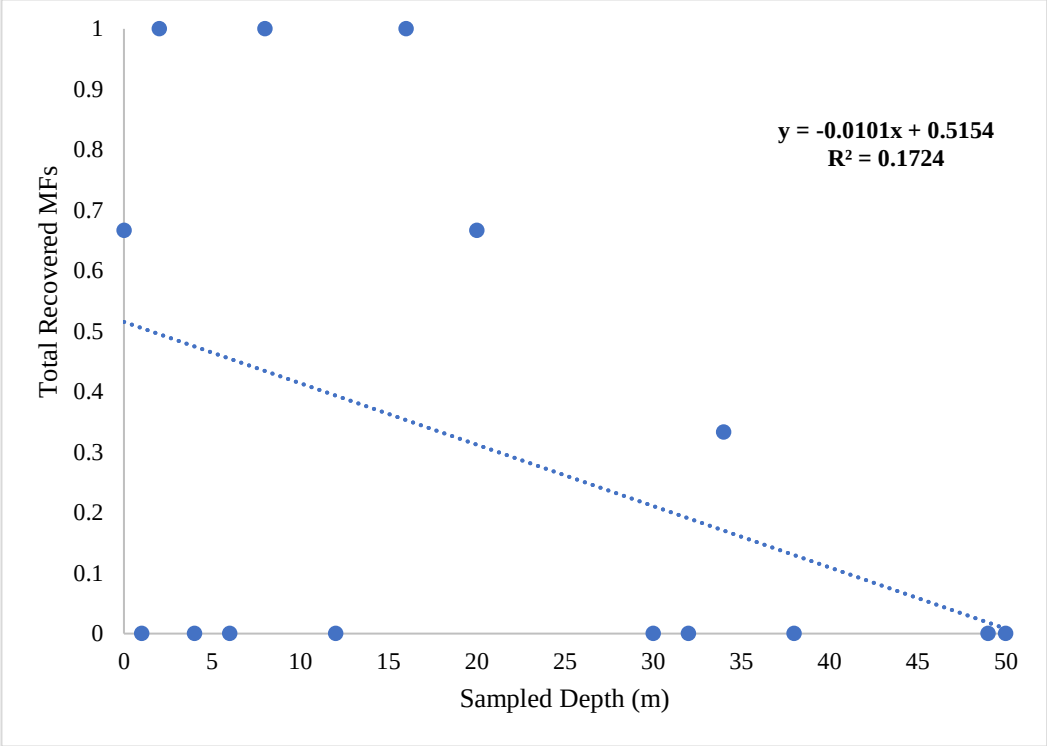


Figure 4.12: Correlation between water depth and 'Other Vegetable' MF concentration

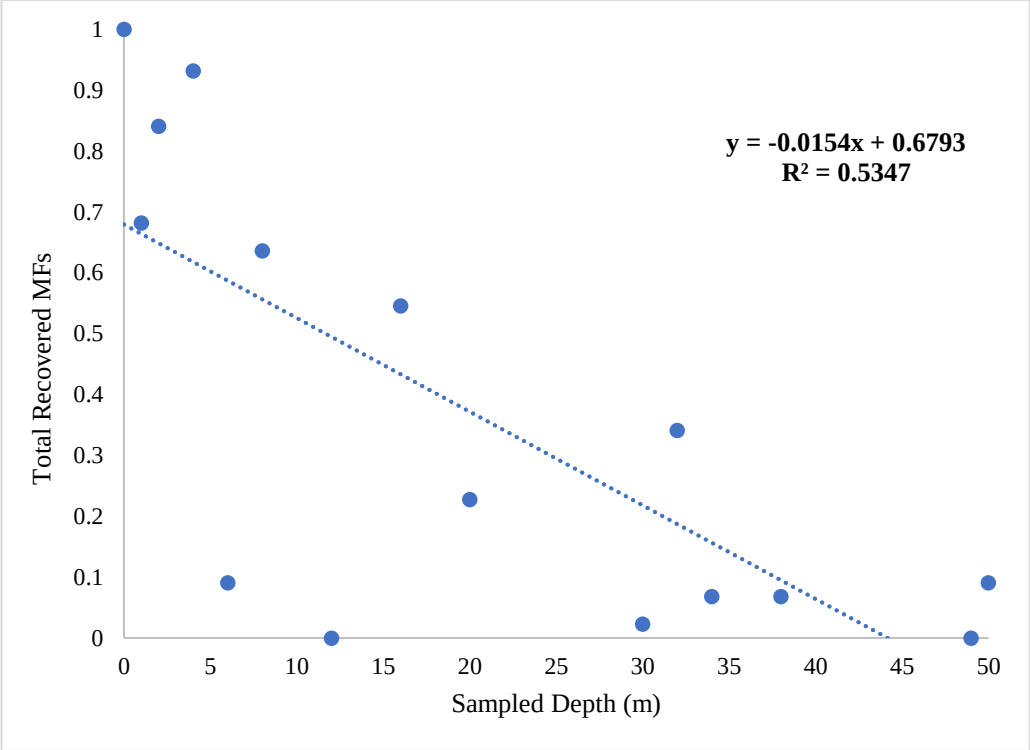


Figure 4.13: Correlation between water depth and Polyester MF concentration

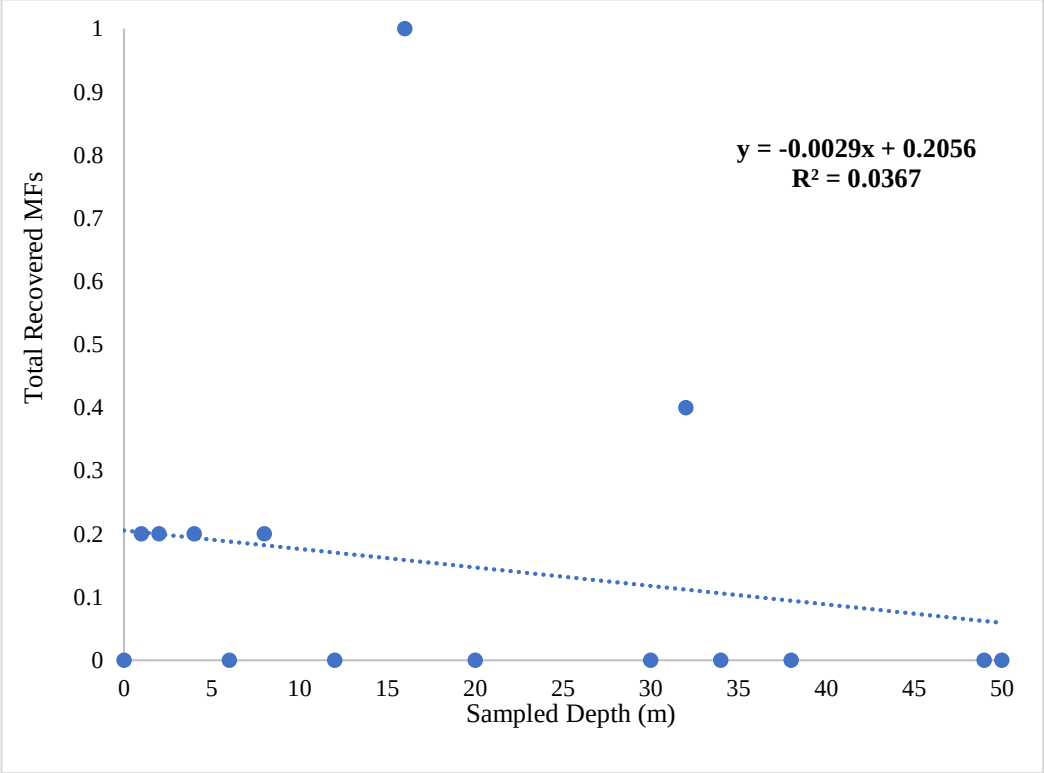


Figure 4.14.: Correlation between water depth and Polyolefin MF concentration

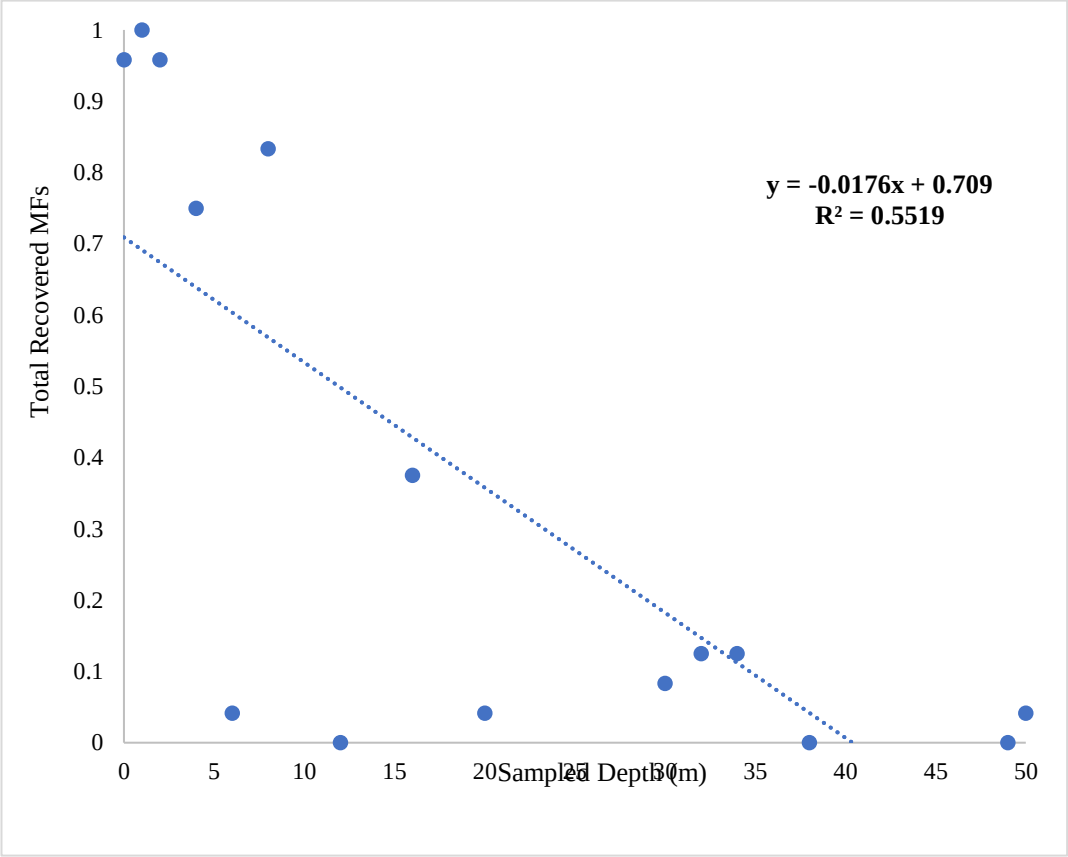


Figure 4.15: Correlation between water depth and Viscose MF concentration

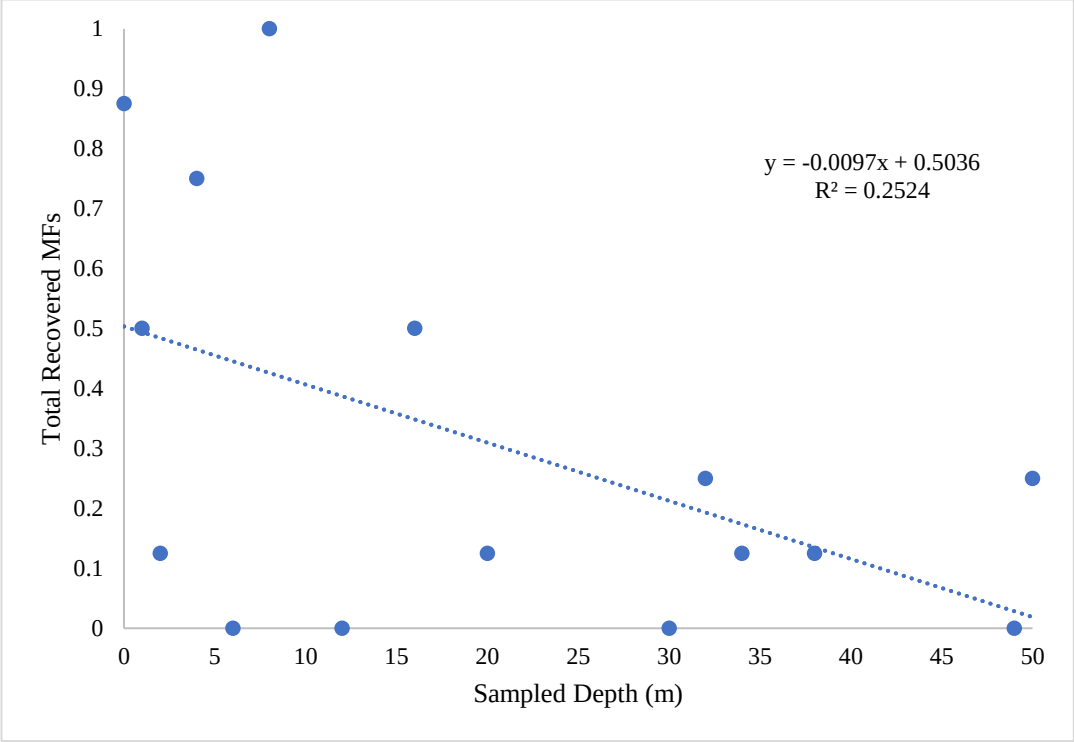


Figure 4.16: Correlation between water depth and wool MF concentration

Although the densities of the various MF types (Table 4.4) apart from polyolefin are higher than that of freshwater (1 gml^{-1}), these MFs are still able to float. As indicated by the low correlation factor R^2 however, other factors are influencing MF distribution along the water column.

Table 4.4: Densities of various fibre types

Microfibre Type	**Fibre Density** g cm^{-3}
Polyolefin	0.90 to 0.96
Viscose	1.52
Polyester	1.39
Cotton	1.55
Acrylic	1.19
Nylon	1.14
Wool	1.30

Colour Distribution based on Filter Size

MF colour was classified into 10 groups in total including blue, black/grey, black/green, red, green, brown, yellow, purple, other and orange in decreasing order (Figure 4.17). All major colours were represented. The group "other" includes MFs with more than one colour. In all the studied locations, blue, black/grey, and black/green were the most prevalent colours. Red fibres were found in more than 80% of examined places, whereas blue and black/grey were present in all the locations.

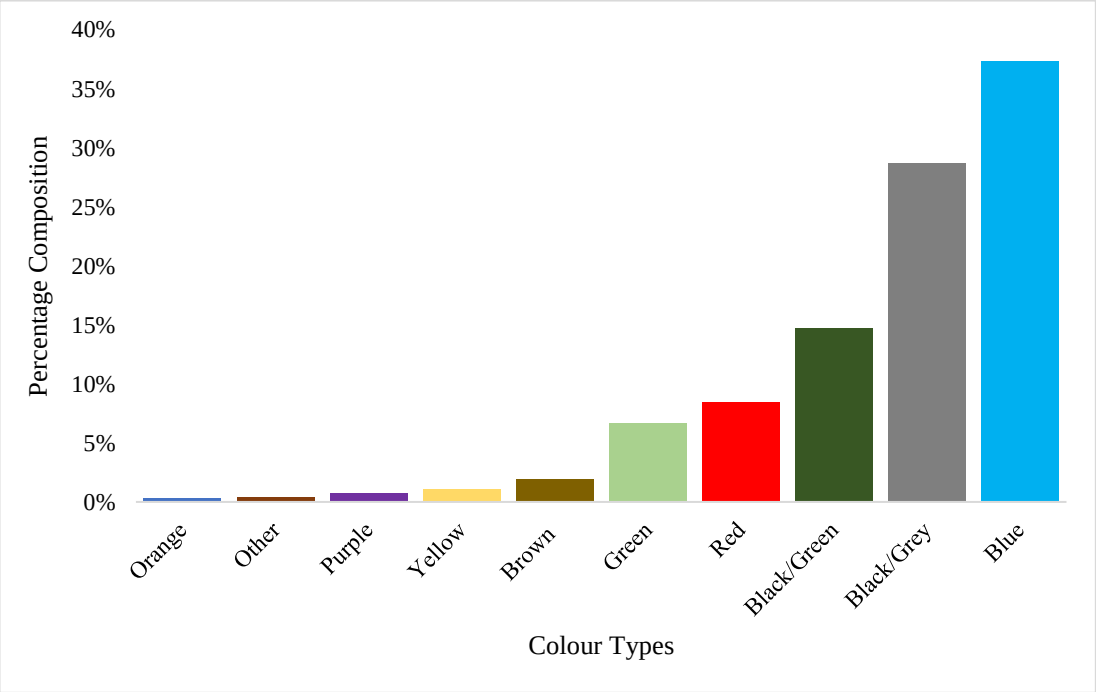


Figure 4.17: Overall percentage distribution of all colour types found in sampled sites.

4.4 Discussion

An average of 11.81 ± 1.47 fibres L^{-1} was found on the samples recovered from the surface water. This is higher than what was found in the Kenyan coast (10.73 ± 1.99 fibres L^{-1}) although more locations were sampled along the Kenyan coast. Overall, MFs were more abundant towards the lake's surface and could in part be due to their morphology (329). Because they are easily damaged by abrasion or friction while being transported, dents may form, trapping air and allowing them to float despite their densities. However, there's evidence from the relationship between MF concentration and sampling depth that

morphology is not the only factor determining their distribution in water. Water movements caused by boats or aquatic animals may also lead to the redistribution of these MFs. A lot of fishing activities are reported on the Lake which means constant movement leading to redistribution of MFs. High concentrations of MFs on the surface of oceans have also been seen to average 2.6 times more than concentrations at 5m (276). Potential sources of surface MFs are atmospheric deposition, run off water (330) and waste water (16). One of the sources of pollutants at Mwanza (sampled location) have been reported to be drainage ditches filled with urban waste (326). Transport of MFs along the lake may also be carried by free-floating vegetative form.

The filter pore size made a major difference in the amounts of MFs captured. The 50 µm filter significantly captured more MFs than the 330 µm. This is congruent with other studies which have compared the efficiency of pore sizes used in MF filtration (276, 331). The implication being that studies which have employed finer pore size present a relatively more accurate data than those that used coarse filters to present data on MF concentration (169, 332, 333). Thus, the reliability of the data reported in Chapter 3 and here has been shown not only by measuring all MFs but also by using a sampling approach that recovers more MF data than previous published data in aquatic environments.

The average length of MF captured by 50 µm was 1338.64 ± 138.29 µm and width 21.57 ± 0.93 µm. Whereas the average length of MF captured by 330 µm was 1903.4 ± 447.18 µm and width 17.23 ± 1.36 µm. This agrees with the findings of Ryan et al.*(276)* that mesh size had no effect on the diameter but on the length of MFs. This explains in part why they are still able to be captured by filter sizes which are up to 10x more than their diameter. The longer length means that may be entangled on the filter and therefore not easily flushed out during filtration.

MFs were found at all the depths that were sampled. However, no polypropylene or polyethylene (polyolefin) was found in the samples recovered from the surface water of Lake

Victoria. However, these were found in the surface water of samples from the Kenyan coast. Nonetheless, they were found subsurface in 6 of the sampled locations. Other lake studies have found polypropylene and polyethylene in surface water. However, these were microplastic fragments (204, 327, 334)

Most of the locations with evidence of anthropogenic activities were unsurprisingly high with MF concentrations such as locations 1, 4, 6 and 7. However, location 9 was noted as a possible hot spot due to its proximity to a city. However, it had the least MF concentration. Attesting to the multiple factors impacting on MF pollution.

Blue and black/grey were the two most abundant colours in samples from Lake Victoria and Kenyan-Tanzanian coast. Although, red, black/green and green colours had different proportions between the two studies, they in addition to blue and black/grey make up more than 80% of colour types. These colours have also be found in other lakes such as these (29, 314). The dominance of blue colour in this study agrees with the result found in the microplastic study of Lake Victoria's surface earlier (327). However, they reported very low proportion of red coloured fibres contrary to the findings in this present study. The fact they only quantified synthetic MFs may be one of the reasons for the contradiction. It was demonstrated in the previous chapter that red colour was more associated with natural MFs than man-made.

The laboratory recorded higher levels of atmospheric deposition than in published studies. Differences may be as a result in monitoring techniques. Most published studies use wet filter papers. Adhesive property of the tapes may increase the persistence of MFs unlike the paper filters. It is important to note that the tapelifts were entirely exposed to the environment in each case, while the samples were studied under the microscope throughout, acting as a protective cover. When not being examined, they were safely covered and preserved. While the tape was exposed throughout a session of MF recovery (a session is the time from laboratory set up till the end of examination of a total number of samples), each sample was

only exposed for the duration of the time it was being examined. In addition to the considerations given above, estimating the number of fibres that may be deposited per minute based on the data collected minimises the possibility that the data provided here has been overestimated as a result of contamination rather than true estimation of pollutants from Lake Victoria. It is critical to emphasise that the filters were stored in sealed petri dishes and were only exposed for examination. The absence of coloured MFs on six samples demonstrated the validity of the stringent techniques adopted from forensic fibre examination.

No procedural blanks were taken during field sampling to monitor contaminants from the atmosphere and therefore contamination may not be entirely ruled out including control samples from the clothing of field samplers (38). However, the enclosed nature of the filtration device reduces the likely hood of any significant contamination. Other possible source of contamination which may not be ruled out is the bottled mineral water used in rinsing the Van Dorn sampler before collecting lake water. As replicate samples were not taken, the data provided in this present study may be a rough estimate of MF concentrations (276).

4.5 Conclusion

The presence of MFs on lake's surface water and sediments have been evidenced in published literature. However, data on MF distribution in lake's column is scarce. Using the forensic approach confirmed in Chapter 3, the study laid out here demonstrates that different MF types are distributed along the lake's column although higher concentration was found on the surface. The high concentration of MFs demonstrates how freshwater bodies are the primary recipients of these pollutants which are eventually transported to the marine environment. Similarity of distribution of MF types also confirms that freshwater bodies are one of the sources of MF pollution to marine. The implication being that surface sampling

should be prioritised in the midst of scarce resources to sample water column. Fresh water bodies are good indicators of what to expect in marine bodies.

The finer pore size of filter captured significantly more MFs than the coarse one. This demonstrates that apart from underestimation of MF data as a result of excluding natural MFs from pollution data, MF estimation has also been grossly underestimated through the use of coarse sampling devices. For more accurate data, sampling method employing filtration with fine pore size should be used. Other areas that would benefit from using fine filter sizes to capture anthropogenic MFs should be explored.

The nature of anthropogenic activities recorded for each location was not always directly proportional to MF concentration found, indicating that other factors such as atmospheric transport of MFs influence MF concentration. Being such a vital pathway, sources contributing to MF concentration in air ought to be investigated.

Chapter Five

Exploration of Factors Contributing to the Release of Microfibres by Tumble Dryers

5.1 Introduction

Cloth dryers and their Contribution to Microfibre (MF) Accumulation

In the preceding chapter, evidence was presented, highlighting the critical influence of sampling method choice on the accurate assessment of MF pollution, with a specific focus on the pivotal role played by the pore size of the sampling device. The intrinsic relationship between pore size and the quantity of MFs retrieved from aquatic environments for subsequent analysis was elucidated, revealing that the smaller the pore size, the greater the amount of MFs recovered from the aquatic environment for analysis leading to more accurate quantification and identification. This increased precision bears significant ramifications, as it empowers researchers and environmental advocates with the means to comprehensively address the growing issue of MF pollution in aquatic environments. By facilitating more accurate identification and quantification of MFs within these environments, the utilisation of smaller pore-sized sampling devices presents an invaluable opportunity to devise and implement effective and efficient strategies for mitigating MF pollution.

It is therefore pertinent to explore the efficacy of smaller pore-sized devices, particularly in contexts where the challenge lies in capturing MFs released from textile materials, thus averting their accumulation and consequent contribution to environmental pollution. A prime example of such a challenge resides in the domain of airborne MF pollution, notably stemming from the utilisation of electric cloth dryers.

In contrast to the well-established research on washing machines, the study of the impact of cloth dryers on MF pollution is a relatively new field (126). At the time of writing, there have been only seven published studies on this topic including the published version of this

chapter (Table 5.1). Of these studies, six have utilised vented domestic/household dryers, while one has examined portable washer/dryer machines. Despite these limited studies, inconsistencies in reporting and methodology have already begun to emerge. The implication being that comparison of results would be difficult. Furthermore, with the exception of this present study (108) and Tao et al. (157), the studies have only evaluated one fibre type per drying cycle, which does not accurately reflect real-world scenarios. Despite evidence of the dominance of natural MFs in the aquatic environment as evidenced in Chapters 3, 4 and elsewhere (5, 69), these drying studies have all focused on synthetic fibres except (157). Other crucial factors such as fabric-fabric interactions, which also contribute to MF release, have not been considered. This can only be accounted for when more than one fabric/ garment is put in the dryer.

Vented dryers work by pumping hot air through wet garments, evaporating the moisture, and then venting the damp air outside (335). Within the vented damp air is contained MFs released from the dried garments. As a result, these are a major source of atmospheric MF pollution (107, 336). Atmospheric MFs have been found both indoors and outdoors (153, 330) and the implications of these on humans have been raised (159). When inhaled, they can cause toxicity through particle and chemical effects (159). For instance, it has been demonstrated through laboratory experiments that chemicals from MFs could be leached to cells when they have accumulated through inhalation (337). As MFs are buoyant, they can travel long distances in air. As a result, atmospheric deposition of MFs contribute to MF accumulation in aquatic environments (20, 171). Other sources of atmospheric MFs include normal wear of clothing (101, 102).

Various methods have been utilized to estimate the amount of MFs released through the exhaust vent of vented dryers, but the most effective approach involves capturing the MFs at the vent outlet before they enter the atmosphere (157) or settle on the ground (107). This is to minimise contamination of samples.

Table 5.1: Studies investigating the release of MFs through electric cloth dryers.

Publication	Type of dryer	Fibre Type	Air Sampling Device	Duration of Drying	Reported MF Release Vent	Lint Filter
Pirc et al. (126)	Tumble dryer	Polyester fleece blanket	Fibres collected from lint filter	18mins 40 ºC 1 blank per cycle	NA	32 ± 24 mg
Kapp and Miller (107)	Vented household electrical dryer	Polyester fleece blanket	100 µm white nylon mesh filter bags attached to vent	1-hour low heat 1 blanket per cycle	Site 1 35 ± 16 mg Site 2 70 ± 77 mg	Site 1 68 ± 47 mg Site 2 27 ± 22 mg
O'Brien et al. (338)	Vented domestic dryer	Polyester fleece	Air sampler	20 mins 56-59 ºC 1 blanket	NA	77 ± 22 mg
Kärkkäinen and Sillanpää (134)	Front-load tumble dryer	Polyester Polyamide Polyacrylic clothing	Collected from lint filter (mesh size 60 µm)	50 mins Low heat	NA	10mg - 1700mg/kg
Choi et al. ((158)	Heat pump type drum dryer	Polyester	Lint filter	I hour 40 mins 60 ºC 5 pieces of fabric	NA	20.6 ppm - 59.9 (±20.1) ppm
Tao et al. (157)	Vented household electric tumble dryer	Polyester Cotton Clothing	Air sampler	15 min High heat >1 clothing per cycle	93,635 ± 17, 026 fibres/kg 72,188 ± 11,813 fibres/kg	NA
Mahbub and Shams (133)	Portable washer and dryer	Acrylic	Multistep vacuum filtration	30 mins, 45 mins and 60 mins 1 fabric per cycle	NA	162 ± 44 mg/kg 60 ± 13 mg/kg

Factors such as the structure of the garments (125), the duration of drying cycle (133, 134) and load (157) can influence MFs release during drying. For example, Tao et al. (157) found a positive correlation between the mass of polyester load and number of MFs released into the air.

To mitigate the release of MFs from laundry instruments such as dryers and washing machines, they are built with filtration devices known as lint filters. These filters are designed to capture MFs in order to control environmental pollution. They come in varying pore sizes such as 0.06mm (133), 1mm (107) etc. However, there are other capturing devices that are external to the laundry instruments such as Cora Bell and Lint LUV-R (339) XFiltra filter and Guppyfriend (149). For example, Napper et al. (149) found that XFiltra was 78% efficient at capturing fibres. Conversely, McIlwraith et al. (339) discovered that the number of MFs captured by Lint LUV-R was nearly three times greater than the amount captured by Cora Ball. Although these studies have been done to investigate the efficiency of the capturing devices in washing machine, none has been done for dryers.

The aim of the study laid out in this Chapter is to assess the influence of both lint filter design and dryer sheets on the emission of airborne MFs from tumble dryers. In addition, investigation was made to see if using fabric softener and conditioner when washing made a difference to MF amount released when same wash load was dried. These would help in providing estimations for the air quality following emission of MFs into the atmosphere.

5.2 Materials and Method

Test Garments and MF Capturing Devices

20 T-shirts were used for the drying experiments. Full descriptions of the garments are given in Table 5.2 below including details of MF capturing devices.

Table 5.2: Details of Test Garments and MF Capturing Devices

Fibre Type	100% Cotton	100% Polyester
Brand	Fruit of the Loom® Original T-shirts	Fruit of the Loom® Performance T-shirts
Number of Garments	10	10
Density	145 g/m^2	140 g/m^2
Size	Large	Large
Dryer Load	48%	52%
Dryer Sheet	Bounce® Outdoor Fresh dryer sheets Bounce® WrinkleGuard Mega dryer sheets	

Washing and Drying Procedures

T-shirts were washed using North American and European washing procedures. North American washing procedures involved using 6 grains per U.S gallon hard water and a high efficiency top loading washing machine (Maytag® Bravo, Model MVWX655DW1). The washing machine's 'Medium soil, Fabric Conditioner' knob was set to "ON", Extra rinse knob was set to "OFF". Other parameters such as washing temperature, main wash volume, rinse temperature and rinse volume were 25°C, 38 L, 15°C and 43 L respectively. The total duration of wash was 52 minutes. Apart from when the T-shirts were washed, this procedure was used without garment load (washout cycle) to avoid cross contamination. European washing procedure on the other hand involved the use of 19 grains per U.S. gallon hardness water. A side loading washing machine (Miele® W3622) was used. To wash the T-shirts, the machine was set at 30°C Cotton Short program. This setting had a total of 85 minutes wash time and 1600 rpm spin speed. However, unlike the North American washing procedure, the washout cycle was carried out on a different setting as to when the garments

were washed. This setting had a total of 30 minutes, temperature of 40°C and rpm spin speed of 1600 (108).

Following the washing of the 20 garments together in four successive washing cycles, the load comprising of 20 garments was dried after each cycle in a vented tumble dryer (Indesit®, model IDV75) for one hour on a high heating. Two identical dryers (Indesit®, model IDV75) were used and drying was rotated after each treatment. Mechanical and thermal energy did not vary for the loads. To measure the process of drying, the dryers were kept on a balance in a well-ventilated research facility. At the time of drying, internal temperature and power consumption were noted between drying to ensure consistency. Dryer sheets were added at the start of drying and removed with the garments at the end of drying.

MFs existing through the dryer exhaust were collected using a 20 µm CellMicroSieve® (BioDesign Inc., Carmel, N.Y., U.S.A.), attached to the dryer exhaust (Figure 5.1A) using a 100 mm plastic pipe connector (model 414c, Manrose Manufacturing Ltd., U.K.) (Figure 5.1B). The sieve was connected to one side of the plastic pipe connector using 450 mm long, 10 mm wide cable ties (product 90526, Screwfix Direct Ltd., U.K.) as shown in Figure 5.1C and using an electrical tape, it was connected to the vent pipe (Figure 5.1D). At the end of each drying, the fibres collected on the CellMicroSieve® were re-suspended by washing the CellMicroSieve® properly.

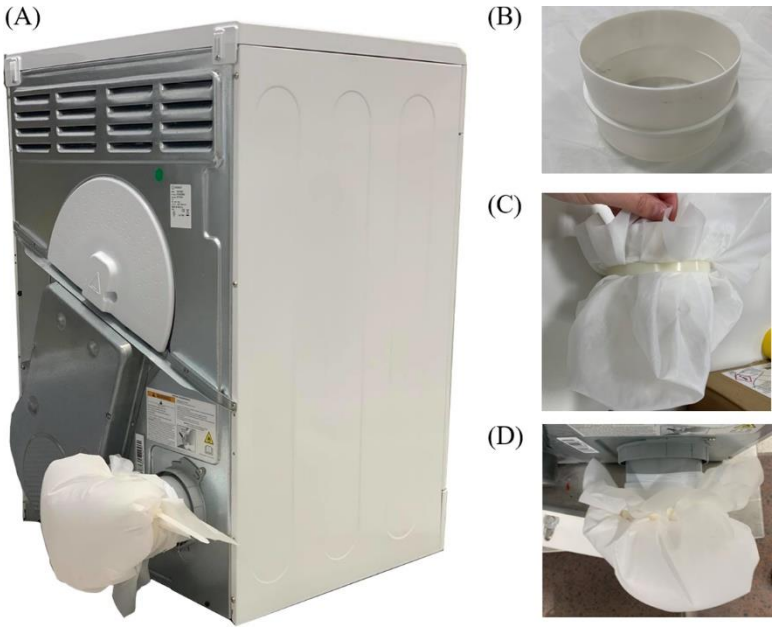


Figure 5.1: Set up of the drying process (108) showing A) the CellMicroSieve® connected to the dryer exhaust B) pipe connecter C-D) coupling process

Throughout the four cycles of drying, the lint filter (Figure 5.2) captured MFs. The tumble dryer lint filter utilized in all experimental trials, with the exception of one trial assessing the influence of lint filter design, was the original filter belonging to the Indesit® IDV75 dryer and had a pore size of 0.2 mm^2. Post-drying cycle, the nylon mesh was thoroughly cleaned with water to ensure proper suspension of the collected MFs. Apart from the impact of capturing devices on MF release during tumble drying, additional parameters were investigated such as the impact of fabric softener and conditioner.

Figure 5.2: Lint filter from Indesit tumble dryer (108)

To investigate the impact of lint filter pore size, two pore sizes were compared. The original lint filter (Figure 5.2) with pore size of 0.2mm^2 and a finer filter of 0.04 mm^2 (Figure 5.3). For a lint filter with the finer pore size, extra Indesit lint filters were obtained, and their mesh was replaced with that taken from Miele® Tumble dryer lint filters (Miele® component number 6244611), which had a pore size of 0.04 mm^2. To bind the Miele® mesh to the Indesit® lint filter frame, Loctite® All Plastic Super Glue (Henkel Ltd., U.K.) was utilised, while preserving the same filtering surface area of 270 cm^2. Before each drying cycle, vented dryers were cleaned to eliminate any leftover fibres by running a 5-minute cycle with no garment or filter.

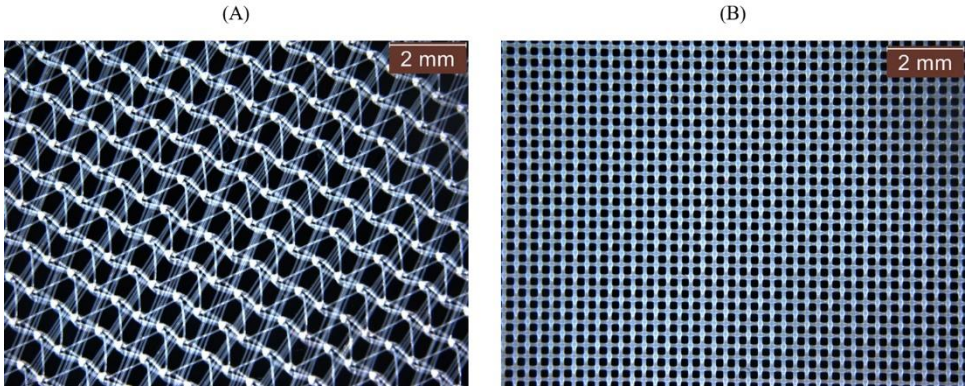


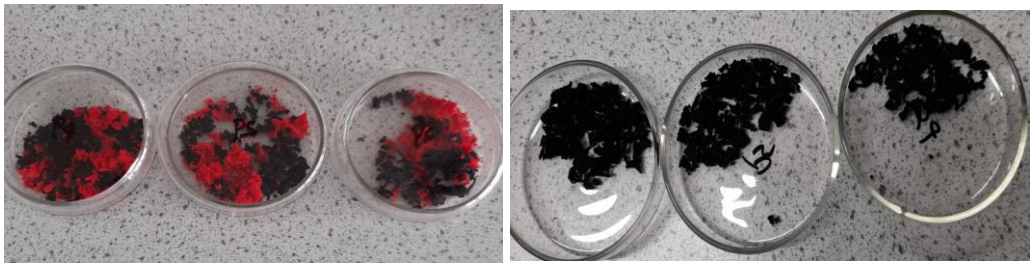
Figure 5.3: Microscopic image comparing the A) coarse and B) fine lint filter meshes.(108)

Validation of Chemical Method and Microfibre Composition

Following the fourth drying cycle, the weight of MFs recovered from the lint filter and the dryer exhaust were calculated and recorded for all treatments. An empty, covered glass Petri dish was weighed and recorded using a balance accurate to 0.0001 g (Sartorius AX124, Germany). Recovered lint was then placed in the Petri dish, covered, weighed, and recorded. The lint's weight was calculated by taking the difference of the two weights. The lint collected from the lint filter and dryer exhaust composed of a mixture of polyester and cotton fibres as the garments were dried together. As it was impracticable to manually count the fibres under the microscope, the relative composition of each fibre was calculated using Chemical Test Method No. 5 of Test Methods D629 (340). Validation of this method (340) was carried out in order to determine (i) if method works, (ii) the amount of reagents that are appropriate for current study samples and (iii) other parameters to adapt to suit samples. Three sets of tests were performed on control samples from the red cotton and black polyester garments used in the drying study. Weights of test run samples ranged from around 0.1g to 0.4g.

Test Run

Pieces of fabrics were cut from the 100% red cotton and 100% black polyester T-shirts. These fabrics were further shredded using a pair of scissors to mimick appearance of lint fibres from tumble dryer (Figure 5.4 A & B). Samples from the shredded fabrics were collected and their weight determined by subtracting the weight of the empty weighing container (watch glass) from the weight of sample + weighing container using a weighing balance (Sartorius AX124, Germany) accurate to 0.001 g. In a fume cupboard, the sample was removed from the watch glass and placed in a 100 ml beaker and covered with 50 ml of 70% H_2SO_4 (Analytical Reagent Grade, Fisher Scientific, UK) using a 50 ml ± 0.05 ml pipette (Volac®, UK) and allowed to stand for 15 minutes at room temperature. The liquid was then decanted through a 560 µm



A B

Figure 5.4: Shredded Samples of (A) mixture of red cotton and black polyester fibres (B) black polyester fibres only

stainless-steel sieve (VWR® Test Sieve BS ISO 3310- 1, Germany) returning the filtrate to the beaker. 50 ml of 70% H_2SO_4 was added again to the filtrate and allowed to stand for 30 mins stirring at 5 minutes interval. At the end of 30 minutes, the liquid was decanted through a 180 µm stainless-steel sieve (VWR® Test Sieve BS ISO 3310- 1, Germany) and filtrate was washed in the sieve under running de-ionized water for about 10 minutes. The fabric pieces were then placed in a 2 % $NaHCO_3$ (Laboratory Reagent Grade, Fisher Scientific, UK) and allowed to stand for about 5 minutes before being washing in running de-ionized water as above. The residue was collected, and blot dried on paper towels. Care was exercised not to lose fibres by using a pair of tweezers. The remaining polyester specimen was then placed uncovered to dry in the oven (UM 200 Memmert, Germany) at 105 to 110^oC for about 1.5 hours.

Following this step, the specimen was then placed in a desiccator containing $CaSO_4$ allowing it to cool for 30 minutes. Another watch glass was placed over the specimen, removed from dessicator, and weighed. The specimen was removed, and both watch glasses weighed.

used to test this. The low amount of loss in S1 may be because of the surface area. This sample was not as shredded as subsequent test runs. 50ml of sulfuric acid proved to be sufficient for samples. Therefore the volume (100 to 150 ml) stated in Chemical Test Method No. 5 of Test Methods D629 was adapted to 50ml. The specimen was then washed in running

de-ionized water between 1 to 5 minutes depending on its quantity in a 180-µm sieve. This was reduced to 1-5 minutes from the 10 minutes recommended in the procedure. This adaptation was made because of the quantity of the sample in order to reduce that amount of fibre that may escape through the pores of the sieve due to pressure from running water. This was evidenced in percentage loss recorded for the polyester samples ranging from 3-14% as shown in Figure 5.5. The validation process demostrated the method was effective as samples from the cotton T-shirt were digested and the polyester left largely unaffected. The remnant maybe additives added during manufacturing process (Figure 5.6).

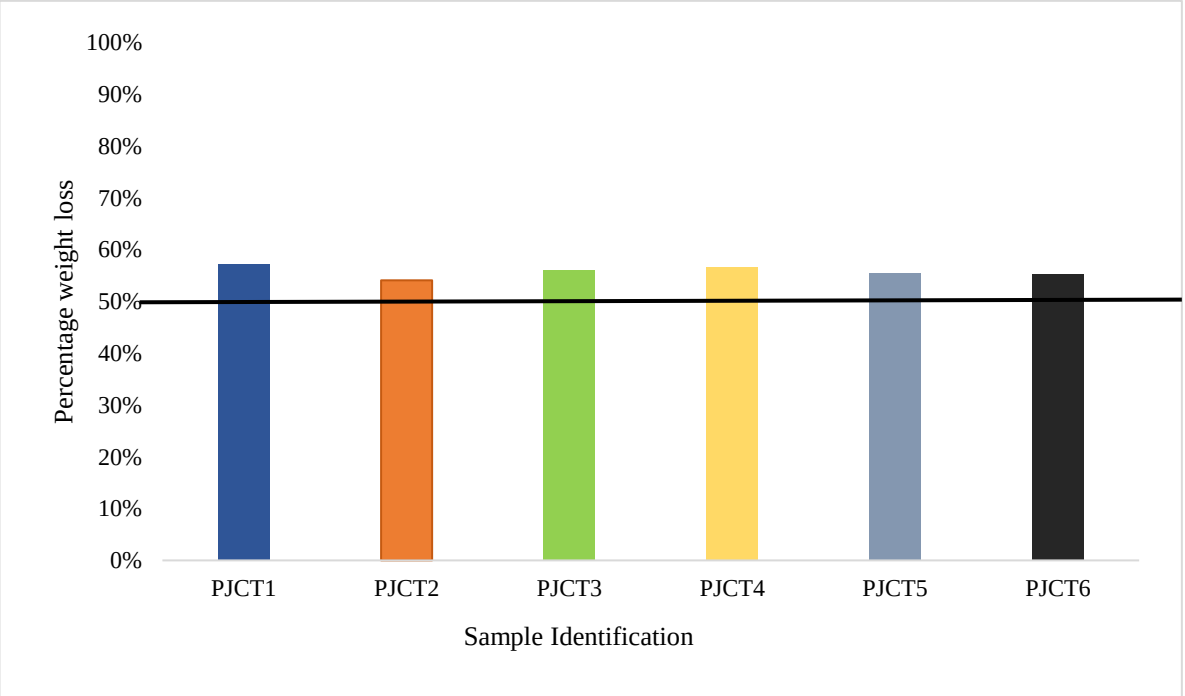


Figure 5.5: Percentage loss of sample weight following the process of acid digestion. The line shows the 50:50 contribution of polyester and cotton fibres in the mixed test samples (Figure 5.5. A). PJCT= Sample mix from Polyester + Cotton T-shirt fibres. The area below the line shows the complete digestion of the cotton fibres. The area above shows percentage loss of polyester fibres likely due to the washing process. This informed the decision to reduce the wash time when analysing the main samples from the drying study.

Figure 5.6: Red residue following acid digestion.

During the test run, it was also observed that polyester samples were lost at an average of 9.5% ± 1.5% when analysed alone or 8.7% ± 1.2% when mixed with sample from red cotton T-shirt (Figure 5.7). The apparent loss of polyester is likely during the washing steps which occurs twice in the procedure and also when solution is decanted using the 560 µm sieve. It is important to note that fewer polyester fibres were lost in the mixed samples compared to when analysed alone. During the digestion process, the cotton sample is sticky at first before it is finally digested. The polyester fibres are stuck to them at this stage.

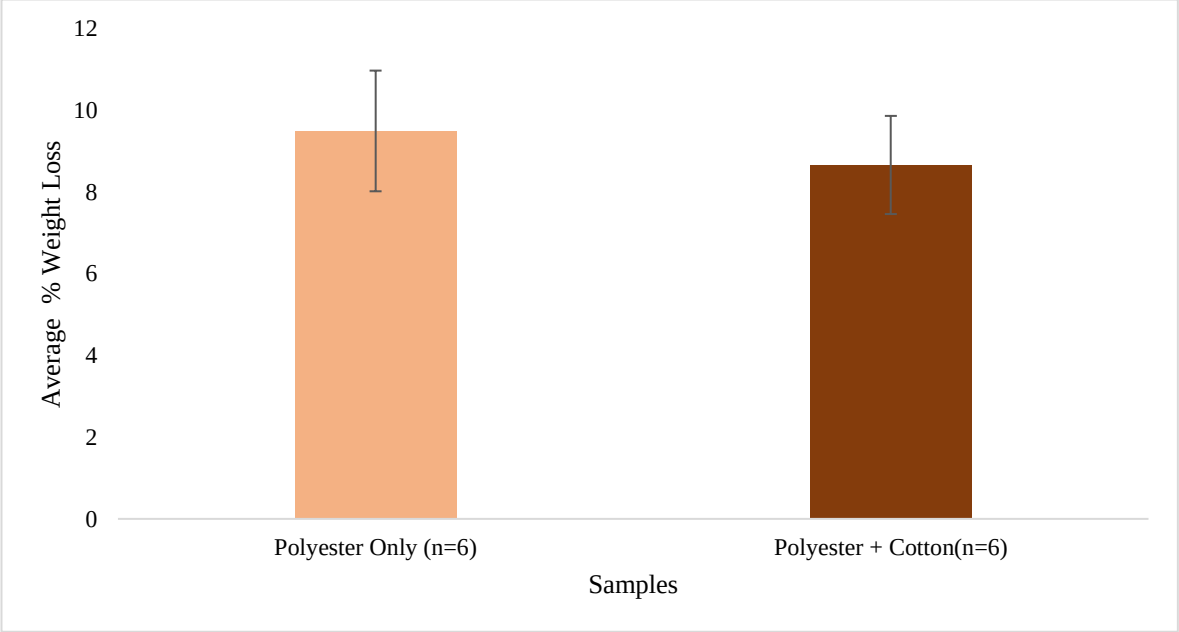


Figure 5.7: Average percentage weight loss in polyester samples

Sheddability Test

A sheddability test was carried out to determine how readily fibres were released from the surface of the garments. To do this, the "press and rub" method was employed to assess the sheddability of the cotton and polyester T-shirts utilised in the drying process. To prevent inaccurate counting, all fibres not part of the garment itself that were on its surface were removed from the testing area. To remove extraneous fibres from the surface of the garment, an adhesive tape (TapeIt™, 3L Office, Denmark) was placed, pressed down, and removed. The now-"blanked" portion of the garment was then covered with a fresh piece of adhesive tape (TapeIt™, 3L Office, Denmark) measuring 17 cm by 5 cm. A forefinger was used to rub along the length of the tape once after pressing the tape's end down into the surface. For further examination, the tape was taken off and fastened to a piece of clear acetate sheet. The tape was partitioned into 1 cm^2 squares, and the number of fibres within one randomly chosen square was counted using microscopy (Leica S6 E Greenough stereomicroscope, Leica Microsystems, Germany). Studies into textile sheddability have employed the use of known weight to standardize the pressure applied during the procedure. For example, Skokan, Tremblay and Muehlethaler (341) in a recent study attempted to standardize

pressure by using ~2 kg weight (417 kg/m^2). Here, to make up for variability, the 'press and rub,' method was repeated six times over the front of the garment and the average number of fibres detected per 1cm^2 sample was computed.

To estimate the number of fibres that are shed per total area of garment, measurements were taken as follows (Figure 5.8).

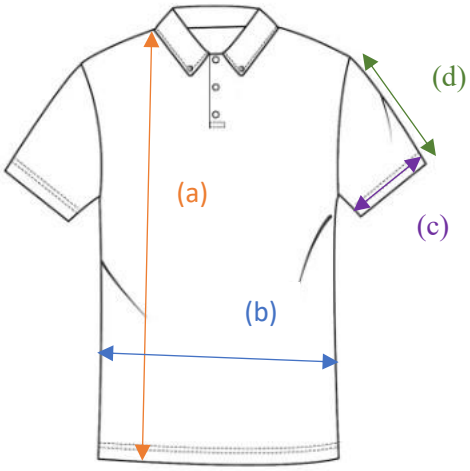


Figure 5.8: Image showing where measurements were taken.

a) Full length
b) Body Width
c) Sleeve Width 1
d) Sleeve Width 2

Total fibre shed from the garment's outer surface is therefore estimated by taking the average of the product of multiplying the fibres counted per cm^2 and the area.

Area of garment (one side) = (a x b) + (c x d) ⟶ Equation 1

Total surface area= (Area of garment (one side)) x 2 cm^2 ⟶ Equation 2

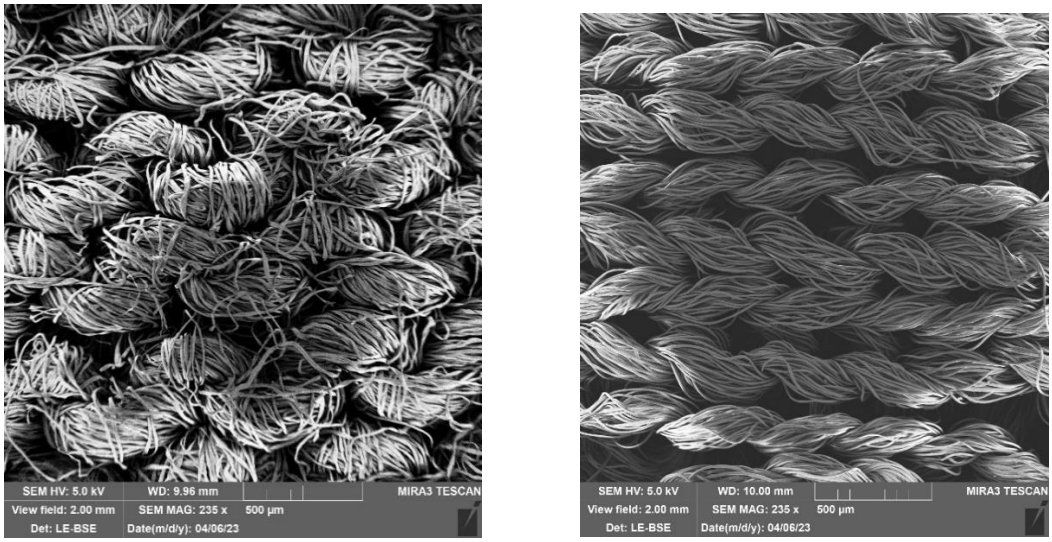
Estimated number of fibres released from the garment surface = Area of garment x Number of fibres counted in 1 cm^2 window. ⟶ Equation 3

Measurement of Fibres

Twenty fibres were measured at random from the sheddability tapelifts taken from each garment. These target fibres were identified and marked using a low power microscope (Leica Microsystems, Germany). Incisions were made on the tape lifts' marked areas, sticky stuff remover (De-Solv-it®, United Kingdom) was applied, and fibres were removed and mounted individually on glass slides (CIMED®, 1-1.2 mm thick, 25 x 75 mm) using glycerol (VWR® CAS number: 56-81-5) and covered with round cover slips (9 mm, Thermo Scientific®, Germany). Following calibration of the system in the relevant units, measurements of fibre length and width were performed using an Olympus CX22 microscope (J.B Microscopes Ltd., U.K.) combined with a Euromex camera with Image Focus 4.0 software.

Scanning Electron Microscopy of Garment Surfaces

Scanning Electron Microscopy (SEM) is a form of microscopy that employs an electron beam to create high-resolution, three-dimensional pictures of a sample's surface. A focussed beam of electrons is scanned across the surface of the sample, and the interaction between the electrons and the atoms in the sample generates signals that may be utilised to create an image. A scanning electron microscope (SEM) was used to observe the fabric structures of the red cotton and black polyester T-shirts. The images (Figures 5.9 A & B) show the loose and compact nature of the cotton and polyester fabrics respectively.



A B

Figure 5.9: SEM images showing A) loose staple fibres on the cotton fabric B) compact filament polyester fabric. Both are knitted fabrics.

Statistical analysis

T-test was used to determine significant difference between treatment effects. Mann–Whitney U test was used to determine statistically significant differences between the length and diameter of polyester and cotton fibres. Statistical calculations and graphs were conducted using Microsoft Excel™ for Microsoft 365 Version 2,205. At 95% confidence level, comparisons with a p-value of < 0.05 were deemed significantly different.

5.3 Results

Fine versus Coarse Lint Filter Pores

Regardless of the lint filter pore size, the lint filter captured more MFs than were emitted from the dryer exhaust. Significantly higher amount of cotton MFs were released from the dryer exhaust when coarse filter was used compared to when fine filter was used (Figure 5.10). This followed logic as fibres would easily escape from the larger pores of the coarse filter. A very small proportion of polyester was released through the dryer exhaust when either of the filters were used. There was a significant difference between amount of cotton

MFs emitted through the exhaust versus the amount captured by the lint filter t(2) = 7.68, p = 0.01. No Significant difference was found between amount of polyester MFs emitted versus amount captured t(2) = 0.48, p = 0.68.

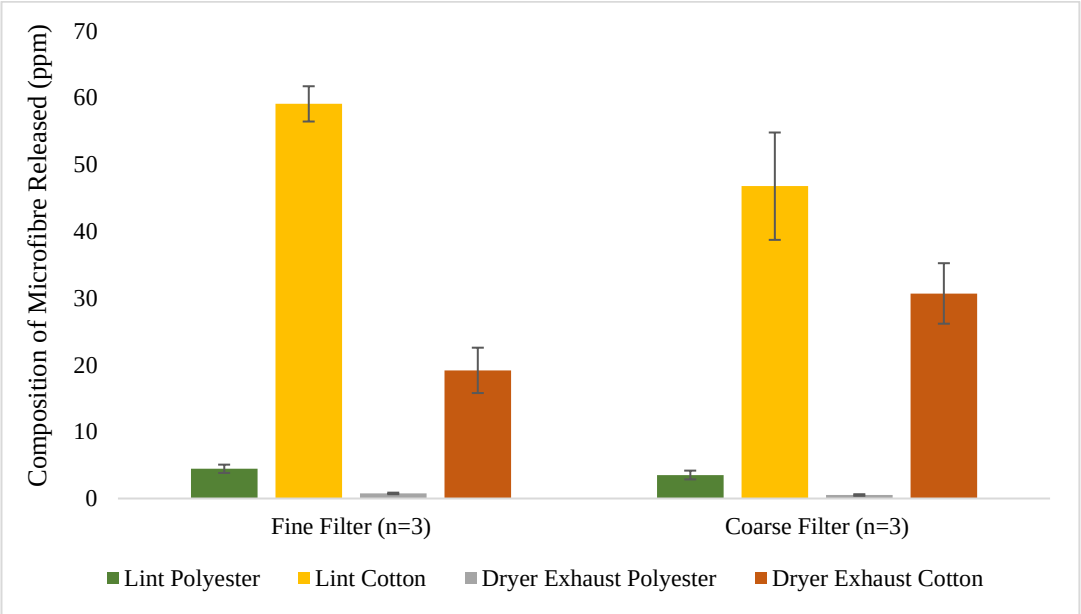


Figure 5.10: Impact of dryer lint filter pore size

Effect of Dryer Sheets

In all the parameters tested, the MFs captured by the lint filter were more than the ones emitted from the dryer exhaust. The amounts of cotton and polyester MFs emitted from the dryer exhaust decreased as the number of dryer sheets increased (Figure 5.11). The amount of cotton MFs emitted from the dryer exhaust when the mega dryer sheet was used, was statistically lower than when no dryer sheet was used t(2) = 30.38, p = 0.001. Conversly, the difference in amounts released from the exhaust dryer and captured by lint filter was not statistically significant for polyester MFs when the mega dryer was used t(2) = 2.64, p = 0.12.

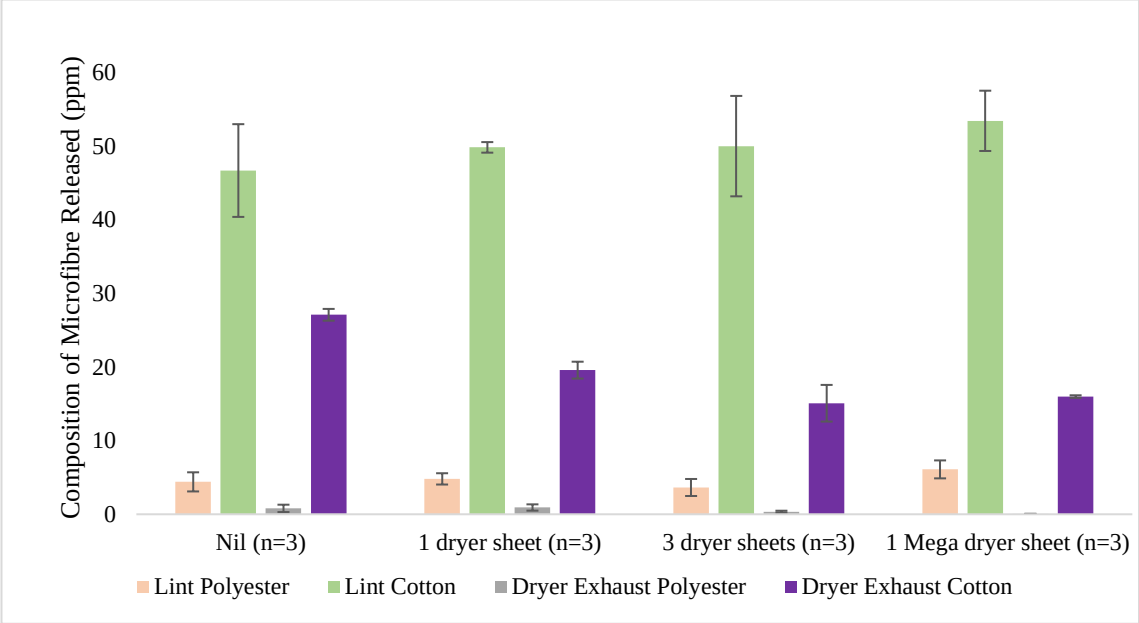


Figure 5.11: Impact of dryer sheets on microfibre release

Combined Effects of Fabric Conditioner and Dryer Sheets

The combination of Downy wrinkle guard and mega sheet dryer resulted in lower amounts of cotton fibres released via the dryer exhaust compared to when no treatment was made (Figure 5.12). This was also the same for the polyester fibres. The amount of cotton fibres emitted via the dryer exhaust when the conditioner was combined with the mega dryer was statistically lower than when there was none $t(2) = 24.36$, $p = 0.002$. There was no statistically significant difference in the case of polyester fibres in this regards $t(2) = 1.84$, $p = 0.21$. In both treatments, the number of fibres captured by the lint filter was more than the amount emitted via the dryer exhaust.

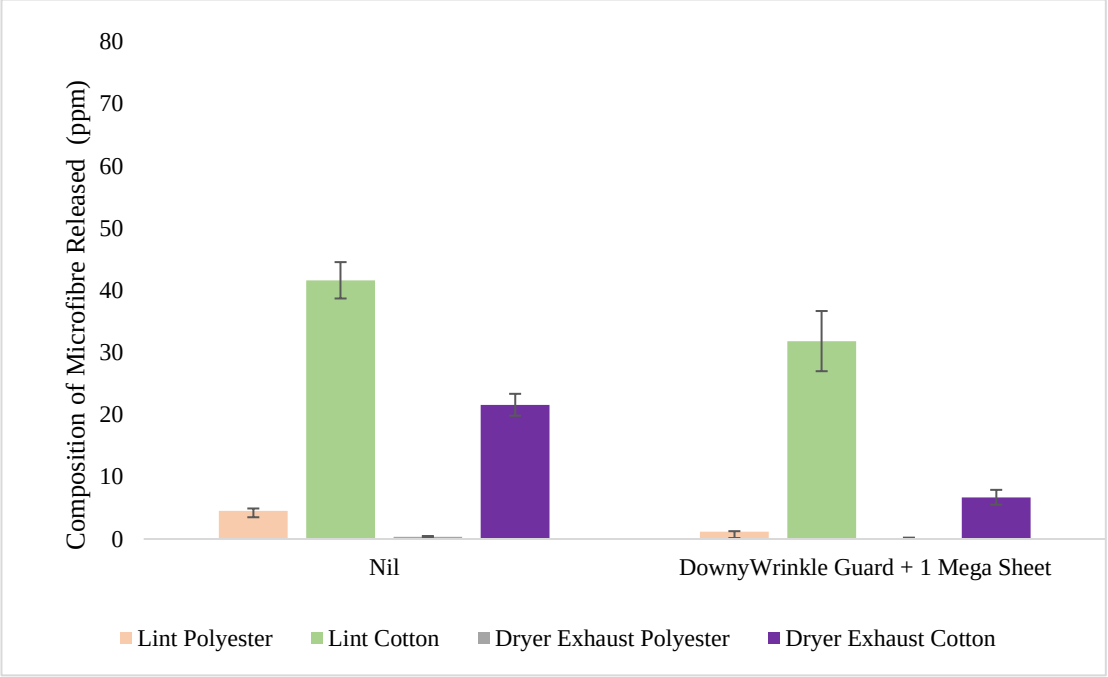


Figure 5.12: Impact of the combination of DownyWrinkle guard and mega sheet

The results presented subsequently do not involve comparisons between fibre capturing devices. These loads were tumble dried following treatment with either fabric softener or anti-wrinkle conditioner during the washing process (108). This involved washing the loads with either recommended, 1.5 x or 2 x the recommended doses.

Impact of liquid fabric softener

Congruent with the results above, regardless of the amount of doses used during US washing procedure, the lint filter captured more fibres than were emitted from the dryer's exhaust (Figure 5.13). Although the amount of fibre emitted through the exhaust reduced above recommended dose, no significant difference was found between using no softener versus recommended, 1.5 and 2 x the recommended doses ($t(2) = 0.42$, $p = 0.71$, $t(2) = 1.21$, $p = 0.35$ and $t(2) = 3.84$, $p = 0.06$)

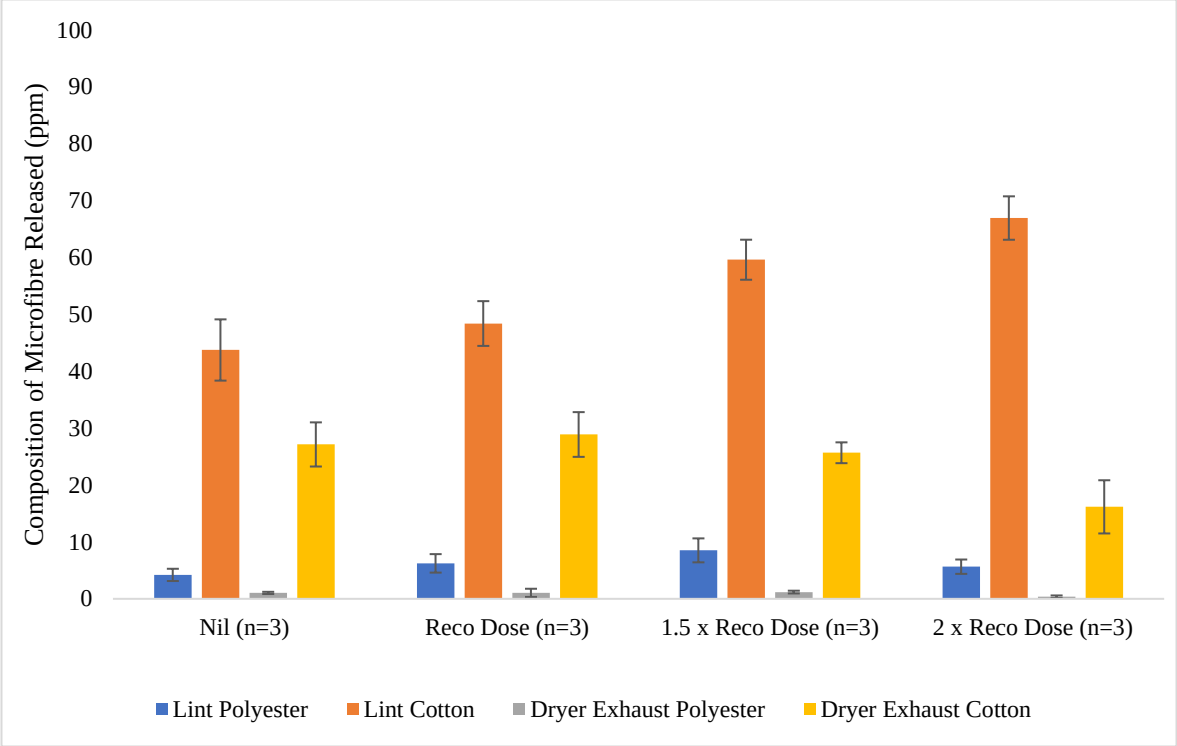


Figure 5.13: Impact of liquid fabric conditioner (North America Conditions)

Similar observations as above were made when the loads washed in EU conditions and treated with different doses of softener were tumble dryed (Figure 5.14). More fibres were

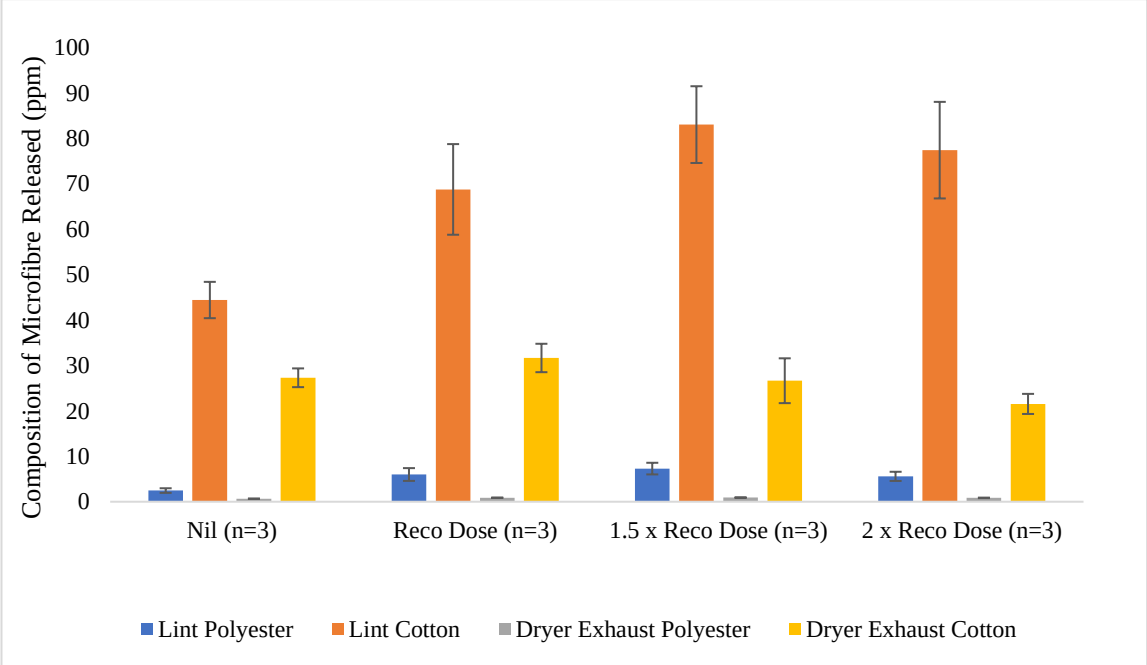


Figure 5.14: Impact of liquid fabric conditioner (EU Condition)

captured by the dryer's lint filter compared to the amount emitted from the dryer exhaust for all fibre types. Drying, following washing treatment with anti-wrinkle fabric conditioner

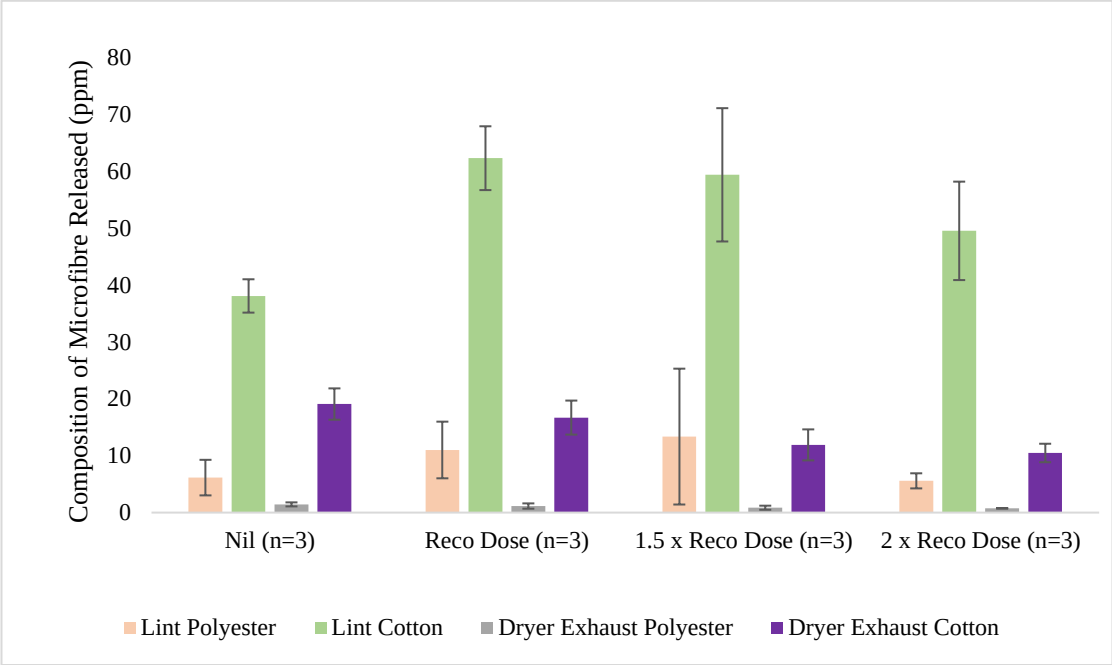


Figure 5.15: Impact of anti-wrinkle fabric conditioner on microfibre release

Showed (Figure 5.15) that significant amounts of cotton MFs were captured within the lint filter similar to the parameter described above. In all cases, the amount of cotton MFs released from the vent decreased as the amount of fabric conditioner increased. Consequently, the highest amount of release was observed in the absence of anti-wrinkle fabric conditioners. This same trend was observed for the amounts of polyester MFs released via the exhaust vent following addition of anti-wrinkle fabric conditioner.

Regardless of treatment, more polyester fibres were captured by the lint filter than were released via exhaust vent, but the reverse was observed to be the case for cotton MFs (Table 5.3).

Table 5.3: Relative composition of cotton and polyester fibres captured by lint filter or emitted via dryer exhaust at different treatments.

		Lint		Dryer Exhaust	
		Cotton	Polyester	Cotton	Polyester
USA Fabric Softener	Nil	91.30	8.70	96.27	3.73
	Recommended dose	88.67	11.33	96.47	3.53
	1.5 x Recommended dose	87.50	12.50	95.52	4.48
	2 x Recommended dose	92.27	7.73	97.92	2.08
EU Fabric Softener	Nil	94.79	5.21	97.61	2.39
	Recommended dose	91.97	8.03	97.31	2.69
	1.5 x Recommended dose	91.96	8.04	96.60	3.40
	2 x Recommended dose	93.28	6.72	96.29	3.71
Lint Pore Size	Fine	93.02	6.98	96.03	3.97
	Coarse	93.01	6.99	98.26	1.74
Dryer Sheets	Nil	91.44	8.56	97.19	2.81
	1 Sheet	91.21	8.79	95.60	4.40
	3 Sheets	93.02	6.98	97.80	2.20
	1 Mega Sheets	89.74	10.26	99.79	0.21
Fabric Conditioner	Nil	86.41	13.59	92.98	7.02
	Recommended dose	85.19	14.81	93.71	6.29
	1.5 x Recommended dose	83.40	16.60	93.46	6.54
	2 x Recommended dose	89.94	10.06	93.25	6.75
Dryer Sheet + Fabric Conditioner	Nil	90.25	9.75	98.39	1.61

Sheddability of Sampled Garments and Length Distribution

The sheddability test showed that the red cotton T-shirt shed more fibres than the black polyester jersey (Figure 5. 16). The estimated average number of fibres released from the surface of each garment is calculated as shown below and presented in Table 5.4. Using Mann Whitney U Test, the difference in estimated number of fibres shed is statistically significant ($z = 2.80$, p-value = .01).

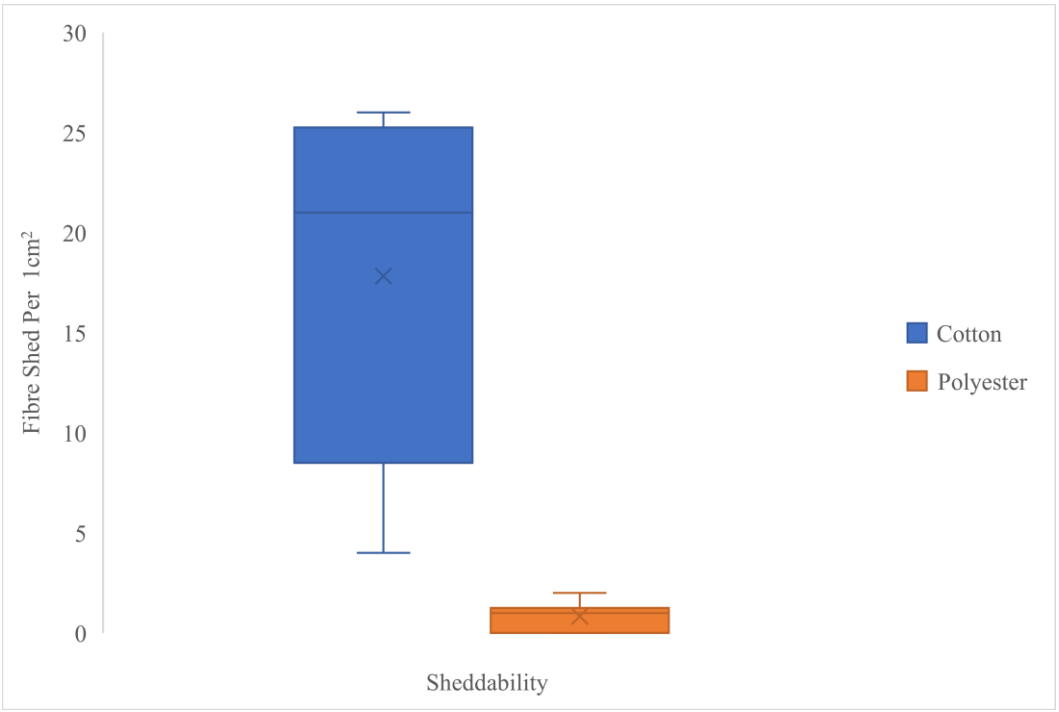


Figure 5.16: Sheddability of cotton versus polyester T-shirts

Table 5.4: Estimated average number of fibres shed from the outer surface of sampled garments.

	Fibres Counted / cm²		**Fibres Shed from Garment's Outer Surface**	
Tapelifts ID	**Red Cotton T-shirt**	**Black Polyester T-shirt**	**Red Cotton T-Shirt**	**Black Polyester T-shirt**
1	4	2	31133	19022
2	10	0	77834	0
3	25	0	194584	0
4	26	1	202367	9511
5	17	1	132317	9511
6	25	1	194584	9511
Total	107	5	**832819**	**47555**
Average	**17.83**	**0.83**	**138803**	**7926**

Regarding fibre length, cotton MFs shed from garments were longer than the polyester MFs sampled in this study (Figure 5.17). Polyester fibres ranged from length of 0.37 mm to 10.78 mm whilst cotton fibres had lengths ranging from 0.99 mm to 8.84 mm. 80% were less than 5 mm in length for cotton whilst 95% of polyester fibres had length less than 5 mm. 50% of

cotton fibres was made up of width measuring 0.03 mm whereas 75% of polyester fibres had width size of 0.01 mm. Fibre length and width difference between the two fibre types were found to be statistically significant at ($z = 4.45$, $p < .00001$) and ($z = 4.92$, p-value $< .00001$) respectively.

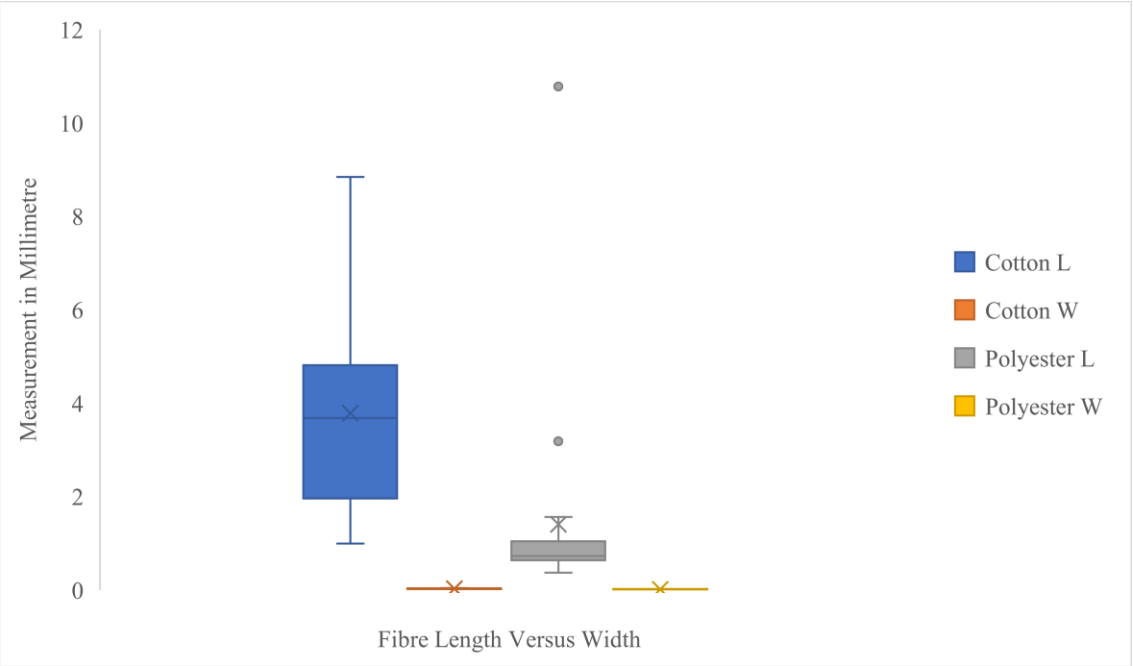


Figure 5.17: Fibre length versus width of cotton and polyester fibres recovered from sheddability test.

5.4 Discussion

Lint filters are fitted to tumble dryers to capture microfibre with the aim of minimising amounts expelled through the dryer exhaust vent. It also prevents accumulation of microfibre within the drum. The finer the lint filter pore size, the greater its ability to capture microfibres. In this present study, replacing the lint filter pore 0.2 mm^2 with 0.04 mm^2 reduced microfibre released through exhaust vent significantly. Kapp and Miller (107), utilised two household dryers comprising of 1mm^2 lint filter. They found that microfibres escaping through the vents travelled up to 30ft from the vent. An average of 404 ± 192 -1169 ± 606 microfibres were released across plots examined in two sites (107). The fact is that the more effectively the lint filter captures fibres the less the amount of fibres that are released

via the vent (107) Dryer type, age, vent installation and lint trap characteristics influences how much microfibres are released. Other studies have utilised lint filters of varying sizes such as 0.06 mm^2 (134) and 0.18 mm^2 (126). The efficacy of these devices is contingent on proper usage, which necessitates the regular cleaning or replacement of collected lint. If the pores become obstructed, MFs within the dryer drum may eventually be discharged through the exhaust vent, resulting in both indoor and outdoor airborne pollution. As demonstrated in this study and elsewhere, augmenting the dryer lint filter with dryer sheets, Cora Ball, Guppy friend, or similar devices can improve the trapping efficiency of microfibers released during the drying process (134). The goal is to minimize the amount of MF that escapes the dryer via its vent.

The tendency of clothes to shed fibres during normal use, as well as during laundering and drying processes, is significantly influenced by their structural design and the type of fibres employed in their composition. Shedding is a complex phenomenon driven mainly by the fundamental qualities of the garment's manufacturing and fibre type. Furthermore, the extent of fibre loss can be significantly affected by a variety of factors such as the garment's manufacturing technique, fibre shape, and fibre-to-fibre and fibre-to-fabric interactions, all of which contribute to the garment's shedding behaviour. As a result, the sheddability of garments is governed by various factors, including their construction and the qualities of the fibres utilised. (100, 125, 341). The sheddability test on the tested cotton T-shirt revealed fibres readily detach from their yarn component. This is evidenced in the SEM images as the yarn were loosely structured as well as the ease with which staple fibre detaches from the garment. The more readily it sheds, the higher the MF loss. As tumbling and spinning motions take place within the tumble dryer, these loose fibres are easily removed from the clothing and become part of the lint that is caught within the instrument or discharged out of the vent. The reverse is true for polyester. Because it does not have easily accessible fibres on its surface, there is less to be influenced by the mechanical operations occurring within

the dryer, resulting in less MF discharge. A study of the shedding capacity of blended polyester and cotton garment by Skokan et al (341) showed a ratio of 93 – 97 % : 3- 7% for cotton and polyester respectively. This agrees with the ratio of cotton to polyester microfibre released in this research when the two fibre types were washed together.

Cotton fibre is hydrophilic whereas polyester is hydrophobic (342, 343). Although polyester's strength is not changed by its wetness, cotton's affinity for water results in a bond which makes cotton fibre stronger when wet (343). It appears that as the drying process occurs within the dryer, the weaker the bond and therefore ease of fragmentation leading to its massive release compared to polyester. However, when it has absorbed excess water, it may lead to swelling and eventual fragmentation (344). Hairiness favours fibres releases as well as staple over filament. (24) The length of polyester fibres from the sheddability test is congruent with microfibres released during washing as evidenced by these authors except for the outlier (345). A garment comprising of a blend of polyester, cotton and modal was found to release more fibres compared to other garments of 100% materials (137). Fibre length of polyester fibres from sheddability test is congruent with 0.3-25 mm fibre length found by (126) and these dryer studies (338). This evidence the suitability of the shedding technique employed here for the identification of shedding potential of garments. The relationship between fibre length/diameter to filter pore size needs to be investigated as it is not clear in this study.

Fabric softener tends to reduce the effect of aging and invariably roughness on knitted polyester (346). Surface roughness or hairiness which are usually associated with garment aging promotes microfibre shedding and loss from the surface of garments. Fabric conditioner acts as lubricant which helps in minimising pilling and friction (347). This then prevents the formation of loose fibres which would easily rub off through the mechanical actions and surface interactions happening during mechanical drying (348). Garment softness would reduce brittleness and invariably fragmentation.

In this study, it appears that the fabric softeners/conditioners had a binding effect that caused fibres to accumulate in the dryer's lint filter, thus decreasing the number of fibres, particularly polyester, that escaped through the exhaust vent. While exceeding the recommended dosage in the study did reduce the release of cotton fibres through the exhaust vent, it is essential to consider the environmental impact of dispersing excessive amounts of chemicals. Further research, similar to Chiweshe and Crews' (347) study conducted more than ten years ago, is necessary to explore this topic.

5.5 Conclusion

The results of the present study provide important insights into the emission of microfibres from domestic tumble dryers. The study found that cotton fibres are significantly more emitted through tumble dryer exhaust than polyester fibres, suggesting that the choice of clothing material can have an impact on the amount of microfibres released into the atmosphere. Furthermore, the use of a lint filter with fine pores was found to be more effective in capturing microfibres than coarse filters, demonstrating that the design of the filter play a role in reducing emissions.

It is worth noting that all tested microfibre capturing devices were effective in capturing microfibres, although some devices were more efficient than others. This finding suggests that there is room for improvement in the design of microfibre capturing devices, and future research should focus on developing more efficient and effective devices.

The emission of microfibres from tumble dryers has important environmental and health implications. Microfibres can accumulate in the environment, where they can have a negative impact on ecosystems and wildlife. Additionally, microfibres can be inhaled by humans, potentially leading to respiratory problems and other health issues. Therefore, the findings of this study have important implications for the development of policies and regulations aimed at reducing microfibre emissions from domestic appliances.

In conclusion, the present study provides important insights into the emission of microfibres from tumble dryers. The study highlights the importance of using appropriate filters in tumble dryers to minimize microfibre emissions and suggests that there is room for improvement in the design of microfibre capturing devices. Future research should focus on developing more efficient and effective devices and investigating the potential environmental and health impacts of microfibre emissions from domestic appliances.

Chapter Six

Conclusions and Recommendations for Further Work

6.1 Current State of Affairs

The pervasiveness of anthropogenic microfibres (MFs) in marine and freshwater environments have been demonstrated. Studies have shown that they are a global threat to the aquatic environment. While much research has been done to identify and quantify MFs in marine and freshwater environments, the focus on synthetic MFs has limited data on other types. The current lack of reliable methods for identifying natural and regenerated cellulosic MFs in pollution studies can be attributed to the historical focus on microplastics. As a result, data on these types of MFs is lacking. However, forensic science, which has been involved in the examination of MFs for many years, has developed processes that can ensure accuracy in fibre examination, including their characterisation. It too, has extensively investigated the root causes of fibre prevalence – namely, how well a garment shed its fibres and the factors that dictate it.

The aim of the research presented in this thesis was to obtain accurate and reliable information regarding the types and quantity of MFs present in both marine and freshwater environments. To achieve this, a forensic approach was employed to enable the identification of all types of MFs accurately. Additionally, the study aimed to investigate the impact of fabric sheddability on the MF pollution problem. The accomplishment of these aims has drawn attention to five main themes, and these are described hereafter.

The Need to Prioritise an Interdisciplinary Approach to MF Pollution Research

Whilst the idea of taking an interdisciplinary approach to MF pollution research is not new, it is important to give it serious consideration and make intentional efforts to implement it. This is because MF pollution is a complex issue that involves various aspects, including

environmental science, materials science, and forensic science. In Chapter 1, this theme was introduced with more emphasis in Chapter 2 where the invaluable contribution of a forensic approach was explored. The studies laid out in Chapters 3 and 4 have demonstrated this point through addressing contamination issues during MF pollution studies and prioritising microscopic techniques capable of identifying all MF types. There is no point 're-inventing the wheel' when methods that have been found fit for purpose can be reliably used in another discipline. Since forensic science has been involved with fibre research for so long, all the environmentalist need do is to glean from the wealth of experience. This approach encourages knowledge sharing, the use of proven methods, and the avoidance of redundancy in research efforts.

Citizen Science as a Useful Tool in MF Pollution Monitoring

In chapter 3, the impact of getting the locals involved in some aspects of the study gave a sense of responsibility, that is something very important. People are then influenced to wash, wear, and dispose textile materials more responsibly. Studies investigating the impact of washing and drying of textile materials have evidenced the significant role these activities play in MF pollution. The need to therefore involve individuals in quantifying the impact of their actions may not be overemphasised if MF pollution would be addressed effectively. In general, citizen scientists allow for a more extensive and diverse data collection process, enabling researchers to collect data from many locations and increase the coverage of the study area. This allows for a better understanding of the extent and distribution of MF pollution. Citizens may become more informed about the topic and comprehend the influence of their activities on the environment by participating in scientific research. Citizen science can give important insights into the behaviour and impacts of MF pollution on a local scale. Citizens may gather information on the types and quantities of MF pollutants present in their neighbourhood, as well as how they affect local ecosystems and animals. It is a cost-effective technique to monitor MF pollution, which is especially important in

resource-constrained environment like the study areas in this current research. Researchers may collect data at a lesser cost while still acquiring high-quality information by incorporating citizens.

Guilty Fibres: Natural Versus Man-made

Natural MFs have been found to be significantly more abundant than synthetic and regenerated cellulosic MFs (man-made) in both marine and freshwater and are distributed along the water column as evidenced in Chapters 3 and 4. The overwhelming presence of natural MFs in both fresh and marine waters demonstrate that studies which have excluded them have grossly underestimated MF pollution. Any prediction models based on those data are far from accurate as the major pollutants have not been accounted for. This also indicates that previous data likely classified natural MFs as synthetic. Their sheer presence points to the fact that they are not biodegrading as readily as they ought to. The implication is that careful considerations ought to be made when advocating them as alternatives to their man-made counterparts. The fact that 'natural' fibres undergo modification during production make them as much of a threat as the man-made ones. This calls for a shift from the debate of natural versus man-made textiles to careful considerations of the production processes both types of fibres undergo.

The Role of Filtration Devices in MF Sampling and Pollution Mitigation

In chapters 4 and 5, the influence of filter size in MF sampling and pollution mitigation were demonstrated respectively. In both cases, finer filters should always be prioritised over coarse filters to ensure effectiveness of process. The use of finer filters was found to be useful for collecting MF since it allowed for the capture of a higher number of MFs from water samples. The fact that finer filters have smaller pores allow them to retain more MFs than coarser filters. As a result, using finer filters for collecting MFs increases the accuracy of the gathered data. Similarly, in the context of pollution mitigation, finer filters were shown to be more successful in decreasing atmospheric MF pollution by increasing the amount of MFs

captured in devices compared to the amount expelled. These findings have significant relevance for researchers as well as practitioners working in the disciplines of air quality monitoring and pollution management and can be used to guide filter selection in their respective applications.

Textile Sheddability as a Core Issue

Textile sheddability is at the centre of the MF pollution discourse. Throughout the life cycle of a textile material i.e. from raw material to manufacturing to consumer use and finally end of life, MFs are shed. Regardless of the type of fibre, MFs are released in each of these phases. As noted in chapter 1, one of the main factors influencing MF release is the way a textile material is constructed. Solving MF pollution ought to therefore begin from here. If textile materials do not shed fibres, they would not be available to pollute the environment. How do you stop them from being released in the first place? That should be the question to drive research. Whilst innovative solutions are being sought, it is important to intentionally make consumers aware of this issue and advocate for sustainable choice. Until such textile materials that are characterised by little, or 'no shedding' become readily available to consumers, manufactures and other stakeholders within the textile and fashion industry ought to find ways to mitigate the impact of shedding in the meantime. MFs released through textile shedding be it into the air, terrestrial or aquatic environments pose a threat to biota including humans and therefore must be taken quite seriously. For example, prolonged inhalation of considerable amounts is detrimental to health as cited in chapter 1.

6.2 Practical Implications of Research Findings in the African Context

Although there is an apparent paucity of research on the implications of laundering and drying of textile materials in Africa compared to more extensively studied scenarios in the global north (GN), insights into the effects of these activities can be extrapolated from studies conducted in that region as well as from the established understanding of textile sheddability, as previously expounded. Less households compared to the GN utilise electric

cloth dryers in Africa, nonetheless, it is important to remember that drying of clothes outside also contributes to the accumulation of MFs in air. Due to climatic conditions i.e. the prevalent hot weather in Africa, cotton materials are favoured. However, the staple and coarse nature of this fibre type implies that it would shed readily during normal wear and when spread outside to dry. Although data is lacking which compares the amount of MFs released during drying outside versus using tumble dryer, logic follows that caution should be taken in both cases knowing that MF is released regardless. However, as there are more mechanical forces acting on clothes in electric dryers, it can be hypothesised that more MFs would be released in that case as opposed to drying clothes outside making the risk level in the African context low. On the other hand, because washing effluents are being directly deposited in water bodies without filtration, MF pollution risk associated with washing is high within the African context.

The African textile and fashion industry is grappling with the influx of second-hand clothing (SHC) from the GN. Although the GN's shipment of these SHC is ostensibly aligned with the promotion of a circular economy, aimed at addressing fibre pollution by prolonging the lifespan of textiles, these exports have inadvertently caused adverse consequences within Africa. Particularly, the proliferation of fast fashion has led to a surge in the export of SHC to Africa. Given that fast fashion typically yields clothing of subpar quality, a considerable portion of these SHC items find themselves underutilized or rapidly discarded. The predicament lies in the inadequacy or absence of efficient waste management practices in Africa, leading to the improper disposal of these textile materials on land and in aquatic environments. As these discarded materials degrade in these settings, they contribute to the generation of secondary MFs alluded to in Chapter 1, which accumulate and yield detrimental consequences.

Moreover, the substantial volume of textile materials in circulation, driven by the upsurge in the SHC trade, implies an increased propensity for these materials to shed MFs as they are

worn. This is further exacerbated by the fact that a significant proportion of these materials are of inferior quality, rendering them more prone to MF shedding. Consequently, the entreaties made by various stakeholders for governmental intervention in the form of policies to regulate these importations ought to be addressed with a sense of urgency. A shift away from a reliance on SHC towards local textile production would ensure the availability of high-quality materials to consumers, extending the useful lifespan of these products. Given the current constraints posed by global economic challenges and limited resources, initiating production on a smaller scale becomes a pragmatic approach. This scaled-down approach offers potential benefits in terms of curbing the proliferation of textile materials available for MF shedding.

6.3 Recommendation for further work

This thesis has contributed to the current understanding of MF pollution in marine and freshwater environments, in addition to the influence of garment shedding in airborne MF. However, the findings have also revealed areas where further research is needed to address knowledge gaps.

Investigation into the environmental impact of natural MFs must be prioritised. While synthetic MFs have received much attention due to their persistence in the environment, this study highlights the need to investigate the potential environmental impact of natural MFs. Future work could focus on the biodegradability of natural MFs and their potential ecotoxicological impact. It is important these studies use concentrations found in the environment.

It is commonly known that washing practises differ greatly across the GS and GN. Hand washing is the major way of washing garments in many underdeveloped nations, whilst washing machines are more widely used in developed ones. This variance in washing practises might have serious consequences for MF accumulation. Previous research has discovered that washing machines considerably contribute to MF pollution, with each wash

releasing thousands of MFs into the environment. Hand washing, on the other hand, is less likely to contribute to MF contamination due to reduced agitation levels and the lack of synthetic fibres in detergents. However, the fact that these hand washing practices are mainly carried out around the water body could make a significant difference. A comparison of MF concentrations in the GN and GS might give useful information on the influence of laundry practises on MF pollution. The collected data might be utilised to discover any variations in MF concentrations between the two locations as well as the role of laundry practises to MF pollution. It would further shape governmental policies and public awareness initiatives focused on lowering MF pollution and enhancing environmental and human health.

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Appendices

Appendix 1

Table 1A: Detailed sampling locations, from Lamu (Kenya) to Zanzibar (Tanzania)

Sample Number	Location	Date	Time	Latitude (DMS)	Longitude (DMS)
WS (1.1)	Lamu County	240119	04:50	2°18'12.0"S	40°54'56.0"E
WS (2.1)	Lamu County	240119	08:30	2°28'36.0"S	40°46'9.0"E
WS (3.1)	Tana River County	240119	10:28	2°34'42.0"S	40°39'3.0"E
WS (4.1)	Tana River County	240119	11:48	2°37'7.0"S	40°33'31.0"E
WS (5.1)	Tana River County	240119	13:35	2°43'31.0"S	40°27'48.0"E
WS (6.1)	Kilifi County	240119	16:01	2°53'58.0"S	40°19'43.0"E
WS (7.1)	Kilifi County	240119	17:19	3°0'29.0"S	40°14'3.0"E
WS (8.1)	Kilifi County	250119	10:07	3°5'13.0"S	40°11'30.0"E
WS (9.1)	Kilifi County	250119	11:04	3°7'56.0"S	40°11'11.0"E
WS (10.1)	Kilifi County	250119	12:09	3°11'3.0"S	40°10'49.0"E
WS (11.1)	Kilifi County	250119	13:48	3°16'12.0"S	40°8'45.0"E
WS (12.1)	Kilifi County	260119	07:17	3°23'20.0"S	39°58'11.0"E
WS (13.1)	Kilifi County	260119	07:51	3°23'21.0"S	39°59'49.0"E
WS (14.1)	Kilifi County	270119	07:40	3°29'27.1"S	39°57'18.7"E
WS (15.1)	Kilifi County	270119	09:21	3°37'16.8"S	39°54'47.7"E
WS (16.1)	Kilifi County	280119	12:50	3°37'58.7"S	39°50'29.5"E
WS (17.1)	Kilifi County	280119	14:10	3°43'34.6"S	39°52'54.4"E

WS (18.1)	Kilifi County	280119	15:56	3°51'43.3"S	39°49'32.6"E

WS (19.1)	Kwale County	300119	10:00	4°10'41"S	39°39'11"E
WS (20.1)	Kwale County	300119	11:00	4°15'33.4"S	39°36'49.3"E
WS (21.1)	Kwale County	010219	11:14	4°21'26.0"S	39°34'49.0"E
WS (22.1)	Kwale County	010219	12:29	4°26'51.0"S	39°32'53.0"E
WS (23.1)	Kwale County	010219	13:55	4°32'50.0"S	39°29'60.0"E
WS (24.1)	Kwale County	010219	14:53	4°37'36.0"S	39°26'10.0"E

WS (25.1)	Zanzibar Archipelago	030219	09:15	4°50'48"S	39°38'56.8"E
WS (26.1)	Zanzibar Archipelago	040219	07:46	4°53'52.0"S	39°40'16.0"E
WS (27.1)	Zanzibar Archipelago	040219	08:50	4°57'55.0"S	39°39'17.0"E
WS (28.1)	Zanzibar Archipelago	040219	10:15	5°4'2.0"S	39°39'50.0"E
WS (29.1)	Zanzibar Archipelago	040219	15:06	5°10'11.0"S	39°38'21.0"E
WS (30.1)	Zanzibar Archipelago	050219	06:21	5°20'3.0"S	39°31'55.0"E
WS (31.1)	Zanzibar Archipelago	050219	08:17	5°24'51.0"S	39°28'27.0"E
WS (32.1)	Zanzibar Archipelago	050219	10:51	5°35'45.0"S	39°20'40.0"E
WS (33.1)	Zanzibar Archipelago	060219	10:32	5°46'1.0"S	39°15'57.0"E
WS (34.1)	Zanzibar Archipelago	060219	11:45	5°51'25.0"S	39°14'53.0"E
WS (35.1)	Zanzibar Archipelago	060219	13:12	5°56'28.0"S	39°10'30.0"E
WS (36.1)	Zanzibar Archipelago	060219	14:50	6°4'24.0"S	39°11'53.0"E

WS (37.1)	Zanzibar Archipelago	060219	15:53	6°8'37.0"S	39°11'50.0"E

Appendix 2

Table 2A: Description of sampled locations along Lake Victoria

Sampled Location	Region	Date	Link to Map Location
Location 1	Kenya	09/03/2021	https://goo.gl/maps/5P6Youc4YKZY788B8
Location 2	Kenya	11/03/2021	https://goo.gl/maps/5P6Youc4YKZY788B8
Location 3	Uganda	12/03/2021	https://goo.gl/maps/jiz2QZAoUJhk3k7C7
Location 4	Uganda	13/3/2021	https://goo.gl/maps/RuMM6w9Gy2oJHUBD8
Location 5	Uganda	15/03/2021	https://goo.gl/maps/RFSCWw1oug9d9Vnw5
Location 6	Uganda	16/03/2021	https://goo.gl/maps/NGwXb1EATgzD4B736
Location 7	Uganda	17/03/2021	https://goo.gl/maps/ndez458sznCYgF5F7
Location 8	Uganda	19/03/2021	https://goo.gl/maps/A6Bti6VneK2YTjDE7
Location 9	Uganda	21/03/2021	https://goo.gl/maps/mB4SExJgebpCJbvP9
Location 10	Tanzania	26/03/2021	https://goo.gl/maps/w4Wj6sAx8p9MAuAeA
Location 11	Tanzania	27/03/2021	https://goo.gl/maps/1xFrnGbUQHFCoauHA
Location 12	Tanzania	28/03/2021	https://goo.gl/maps/YXgMCo1XDwv8fa4g6
Location 13	Tanzania	28/03/2021	https://goo.gl/maps/Vxz6tZdeb5VP1uFC7