Extraction Efficiency, Quality and Characterization of *Typha latifolia* L. Fibres for Textile Applications

by

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Abstract

The textile uses of the aquatic plant '*Typha latifolia* L.' (genus *Typha*) have not been previously explored. The current research is the first of its kind to examine the extraction, quality and properties of this waste biomass fibre and compare them with the two most widely used fibres: cotton and polyester, wool. It was found that *Typha* leaves and the core spongy tissue could be transformed into fibres under controlled experimental conditions in aqueous alkaline solution giving a yield range of 15% to 60%. The diameter of the *Typha* fibre is much higher than the Cotton and wool while the moisture regain (%) and thermal resistance are comparable to these two fibres.

SEM revealed a unique submicroscopic 'crenelated' structure and FTIR spectrum showed the cellulose rich content in the Typha fibre. The cellulose content helped *Typha* fibre absorb the reactive dyes and the dye exhaustion is similar or better than the cotton. However, the stiffness of the *Typha* fibre is higher than the cotton and polyester, which would make *Typha* fibre difficult to process in the Cotton spinning systems.

Acknowledgements

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Abbribation

American Association of Textile Chemists and Colorists (AATCC)

American Society for Testing and Materials (ASTM)

Analysis of Variance (ANOVA)

Ethylenediaminetetraacetic acid (EDTA)

Food and Agricultural Organization (FAO)

Food and Agricultural Organization Statistical Databases (FAOSTAT)

Fourier-transform infrared spectroscopy (FTIR)

Mixed Stem (MS)

Least Significant Difference (LSD)

Hard Stem (HS)

Scanning Electron Microscopy (SEM)

Soft Stem (SS)

No Stem (NS)

World Wide Fund (WWF)

Advanced Fibre Information System (AFIS)

Image analysis microscopy (IAM)

Polyethylene terephthalate (PET)

Polycyclic aromatic hydrocarbons (PAH)

Acrylic polyacrylontrile (PAN)

Polypropylene (PP)

Green House Gas (GHG)

High Volume Instrument (HVI)

International Organization for Standardization (ISO)

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CHAPTER 1: INTRODUCTION

Over the past few decades, the global production and consumption of textiles has increased due to population growth and improvements in living standards (Wang, 2006). The growth rate of textile industries has helped to develop strong economies; however, these industries face major challenges, especially regarding environmental pollution. At the present time, high demand and low cost, coupled with higher dimensional stability of fibres and the wide range of applications, have created a great expansion of synthetic fibres (Collier et al., 2009; Khan, 2016).

Most synthetic fibres are costly, not easily biodegradable and responsible for environmental pollution (Salit, 2014). The primary resource material for polyester (polyethylene terephthalate or PET) is petroleum, which is acknowledged to be hazardous to the environment (Zupin & Dimitrovski, as cited in Dubrovski, 2010). Polyester and other synthetic polymer fibres (acrylic, polyamide, and polypropylene) are non-renewable and require large quantities of petrochemicals and energy that contribute to environmental pollution (Zupin & Dimitrovski, as cited in Dubrovski, 2010). During polyester (PET) production, polycyclic aromatic hydrocarbons (PAH) are emitted into the air, producing high amounts of toxicity: a concern for human safety (Shen et al., 2010).

More sustainability in textile industries and more sustainable products to meet basic human needs require the development of both ecological integrity and social equity (Sun, as cited by Wool & Sun, 2005). Research is focusing on natural fibres as they have dynamic properties that could be replaceable instead of synthetic fibres. Natural fibre has been used since 7000 BC for different purposes such as paper, rope, and clothing (Kozlowski, 2012a). Presently, many potential plant fibres such as coir (Cocos nucifera L. adapted from Hoffmann, 1884), sisal (*Agave sisalana* P. adapted from Mussig, 2001), jute (*Corchorus olitorius* L., adapted from Curtis, 1828), flax (*Linus*

usatissimum L., adapted from Meyers, 1906), banana (Musa textile adapted from Meyers, 1906), pineapple, hemp (*Cannabis sativa* L. adapted from Pabst, 1887), canola (*Brassica napus* L., adapted from Khan, 2016), ramie (*Boehmeria nivea* H.; adapted from Mussig, 2001), and Kenaf (*Hibiscus cannabinus* L. adapted from www.wikipedia.org/wiki/Kenaf), are used as a resource for industrial materials in composites and reinforced polymer applications (Salit, 2014).

Natural fibre properties depend mainly on the nature of the plant, locality in which it is grown, age of the plant, and the extraction method used. Natural fibres are a widely abundant renewable resource. The traditional natural fibres collected from various sources and their classification are given in Figure 1.1. Nature offers a large number of fibrous cellulosic materials from which fibre may be extracted. Various parts of the plant such as the woody core, bast, leaf, cane, straw, grass, and seed are valuable not only for textiles but also for building materials, human and animal food, agro-fine chemicals, biomass energy and environmentally friendly cosmetics (Kozlowski, 2012a). The part of the plant being used determines the fibre length: stems and leaves yield longer fibres than fruits or seeds (Blackburn, 2005).

Fibres have been defined by the Textile Institute as units of matter having a length at least 100 times their diameter or width (Fedorak P. M., as cited in Blackburn, 2005). The characteristic dimensions of fibres are the basis of their use: individual fibres (or elements of a continuous filament) weigh only a few micrograms, and their length/width ratio is at least 1000:1 (Moncrieff, 1963). The traditional natural fibre sources include cotton, wool, and silk. Bast or leaf fibres – Flax, Kenaf, Hemp and, Raime have been developed to transform a variety of textile materials of all natural fibres. Cotton has botanical name known as *Gossypium* herbaceum, adapted from Meyers, 1906.; it is the most commonly used fibre and has the most globally significant demand, accounting for 35.7 % of the textile market worldwide (Kozlowski, et al., 2012a).

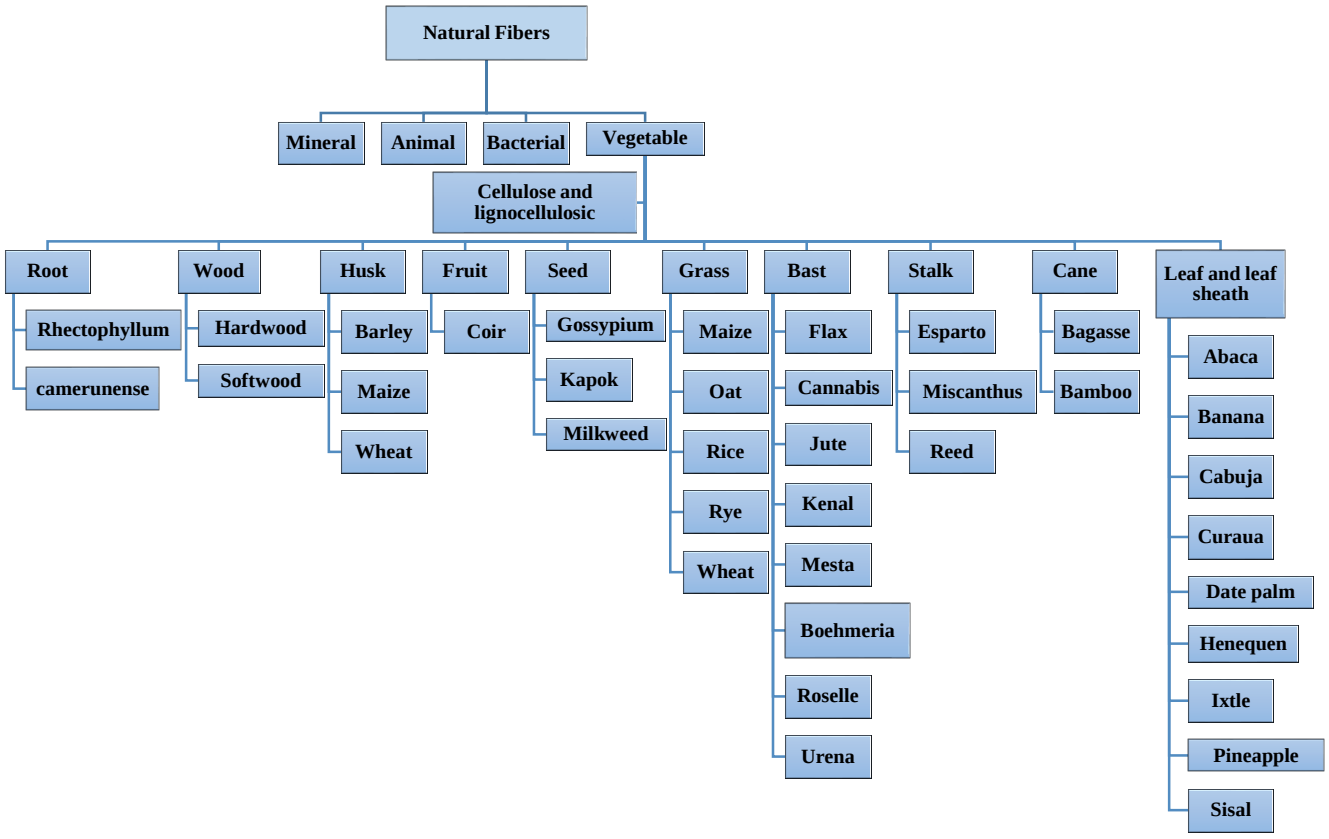


Figure 1.1: Sources and classification of natural fibre (Kozlowski, 2012a).

More than 80 countries produce Cotton, and it represents 2.5% of the entire cultivated crop land of the world (Rashid et al., 2016). Production of Cotton fibre has a significant impact on the environment, as Cotton is considered the most pesticide intensive crop in the world (Rashid et al., 2016). To produce one kilogram of Cotton requires enormous amounts of pesticide and 10,000 litres of water. In other words, it takes 2700 litres of water to make one Cotton t-shirt (WWF, n.d.,). The production of Cotton is responsible for the rapid consumption of natural resources and damage to the fertility of the soil (Judkins, 2008; Khan, 2016). Additionally, agricultural land is often used for Cotton production, thereby having a major impact on food supplies (Judl et al., 2016).

Due to the problems of Cotton and Cotton production, researchers have focused on other natural fibres, called bast fibres, such as linum, corchorus, cannabis, and boehmeria. These are environmentally friendly, and their usability is increasing day by day. Bast fibres have more cost-efficient production and broad product possibilities. All bast fibres have renewable properties with high biomass production per unit land area (Kozlowski, 2012a). Moreover, leaf and crop waste left in the field transforms into organic materials, thereby reducing demand for supplementary chemical fertilizers for subsequent crops (Kozlowski, 2012a). Bast fibre, being of agro-origin, has some unique characteristics such as high strength, good frictional property, tenacity, very high modulus, low breaking elongation, high moisture and good dyeability, using different dyes (reactive direct, vat), good heat and sound insulation properties and low production cost (Kozlowski, 2012a).

Despite their advantages, the share of bast fibres in global clothing industries is very low because of some major limitations such as: insufficient spinning properties (breaking twist angle, single fibre entity, poor bending properties for preparation of yarns), lack of suitable large-scale fibre extraction equipment, and costly methods of degumming (Mather & Wardman, 2015; Kozlowski, 2012a). The fibre yield (%) from bast fibre is also very low (10 to 15%). For example, brassica fibre yield is only 13.82 % and virgin brassica fibre is very stiff, and like all other bast fibres, its usage is very limited in apparel applications (Khan, 2016).

The global market share of bast fibre extracted from the stems of the cannabis plant is only 0.09 %, which is negligible (Figure 1.2). The stiff surface of the cannabis fibre makes it difficult to process on a Cotton spinning system (Ali, 2013). Sometimes the fibre has an irregular surface due to the incomplete removal of the non-cellulosic materials (lignin, pectin). Cannabis production requires higher investments for specialized machinery in order to produce quality spun yarns.

Linum, also a bast fibre, is rigid and difficult to process in Cotton spinning systems and it is costlier on specialized machinery (Minotte & Franck, 2005). According to Kozlowski et al. (2012a), the higher cost of spinning fine yarn comes from the low speed of spinning and the low automation of the process. Recent studies found that brassica fibres solve the environmental problem as fibres are obtained from waste biomass; however, the yield of brassica fibres is low, as mentioned earlier, which is similar to other bast fibres. Further, virgin brassica fibres are stiff and cannot be processed in Cotton spinning machinery without further machine and fibre modifications. For these reasons, bast fibre is not being widely used for fabric.

Considering the above factors, an effort has been made to develop a new textile material with new characteristics, one that is environmentally sustainable, comfortable and industrially suitable. Therefore, researching and developing the extraction and establishment of new cellulose-based textile material is vital.

Table 1.1: Country-based natural fibres production (Source: FAO, 2014).

Fibre production from natural sources												
Countries	**Banana**	**Coconut**	**Pineapple**	**Sugar cane**	**Rice**	**Oil palm**	***Corchorus***	**Kenaf**	***Linum***	***Agave***	**Abaca**	**Kapok**
Brazil	6.90	2.82	2.48	0.74	11.76	1.34	26.71	14.20	0.71	0.25	1.20	na
China	10.55	0.25	1.00	125.54	203.29	0.70	0.17	0.08	0.47	0.15	0.65	0.06
India	24.87	11.93	1.46	341.20	159.20	na	1.98	0.12	0.22	0.21	na	na
Indonesia	6.19	18.30	1.78	33.70	71.28	120.00	0.007	4.35	na	0.03	0.05	0.03
Malaysia	0.34	0.61	0.33	0.83	2.63	100.00	0.002	0.01	na	na	na	0.008
Philippine	9.23	15.35	2.40	31.87	18.44	0.48	0.002	na	0.002	na	0.08	na
Thailand	1.65	1.01	2.65	100.10	38.79	12.81	0.06	1.30	0.01	0.003	na	0.07
USA	0.01	na	0.20	27.91	8.63	na	na	na	0.004	na	na	na
Vietnam	1.56	1.31	0.540	20.08	44.04	na	0.02	8.20	na	0.01	0.01	0.003

*na: Not applicable.

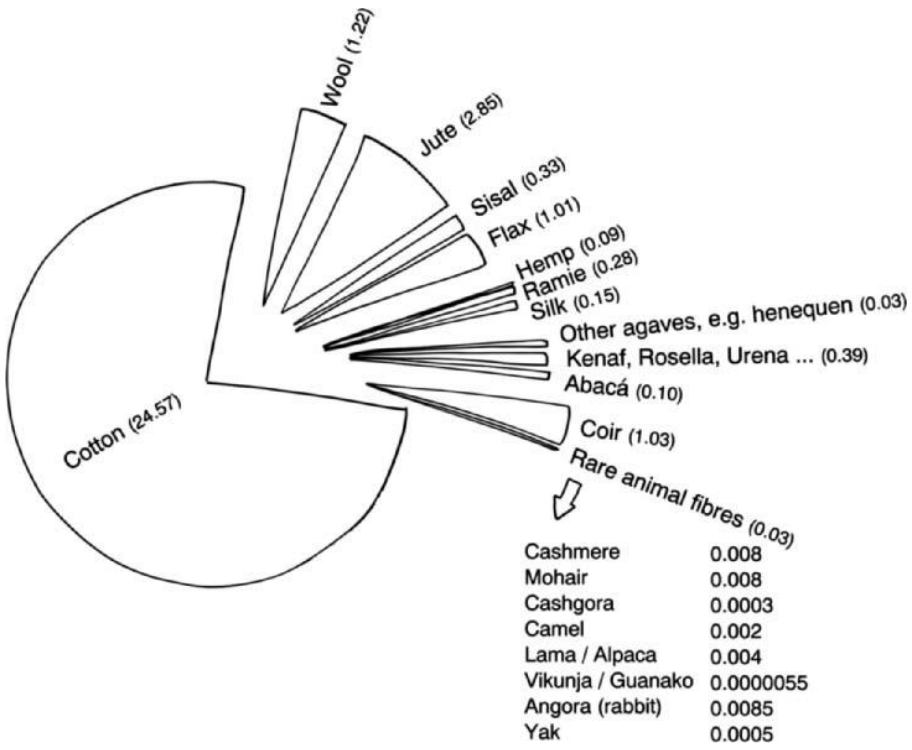


Figure 1.2: World production of natural fibres (million tonnes, average) (Sources: FAOSTAT, 2009 and FAO, 2009).

There are hundreds of thousands of neglected wetland plants all over the world. For many years some natural wetland plants, such as (*Typha latifolia* L., Salix petiolaris, *Typha angustifolia* L., *Phragmites australis* (Cav.), *Carex atherodes Spreng, scirpus fluviatilis (*Torr.*), adapted from* Dubbe et al., 1988) and other species, have been considered potential crops because of their high productivity, interesting chemical composition, abundance, feed value, and medicinal properties (Sana et al., 2014). They also play an important role for wildlife habitat, water filtration and flood protection (Houlahan et al., 2006). *Typha* is a plentiful wetland plant that is being considered as a new natural fibre source (Sana et al., 2014). The native perennial herb *Typha,* known as cattail, originated in Europe, North America and Africa, and has spread over most of the world, including many subtropical areas (Pandey & Verma, 2018). It does not depend upon a harvesting climate or

perfect growing conditions, as do other bast fibres. *Typha* can have significant environmental benefits. The plants are ecofriendly and used to clean soil polluted by heavy metals [cadmium (Cd), lead (Pb), copper (Cu), and zinc (Zn)] (Kozlowski, 2012b). *Typha* grows rapidly, is widely distributed and available locally, is renewable, mouldable, hydroscopic, recyclable, versatile, non-abrasive, porous, viscoelastic, easily available in many forms, biodegradable, and reactive (Maizatul et al., 2012). These features could make the *Typha* fibre appropriate as a raw material for industrial use. For apparel and industrial applications, it is important to understand the physical and chemical properties of *Typha* plants and fibre.

Typha fibre has some unique characteristics; it is environmentally friendly and has multifunctional properties such as thermal insulation (Luamkanchanaphan et al., 2012), reinforced composite, (Wuzellaa et al., 2011), nutrient seizing and watershed management (Grosshans, 2014), and handmade papermaking (Bidin et al., 2015). For these reasons, it is worth exploring if *Typha* can be used in the apparel and textile industry. To start, an effective way to extract the fibre from the plant is needed. A specific method for extracting fibre from *Typha* does not exist yet; traditional methods need to be explored and analyzed for feasibility in textile applications.

It is hypothesized that the *Typha* fibre can be useful for textile uses. Therefore, to determine if this is possible, the following criteria need to be met:

1. Identify a standard extraction method of *Typha* fibre from Typha plants;
2. Optimize the extraction parameters for maximum yield and fibre quality (time, temperature and concentration);
3. Determine major textile properties of *Typha* fibre such as: physical properties, chemical properties, and thermal properties.

CHAPTER 2: LITERATURE REVIEW

2.1 History of textile fibres

Any product made from fibrous materials is considered a textile. Cellulose fibres found in nature have been used for cloth for 4000 to 5000 years (Kozlowski, 2012a; Kozlowski & Mackiewicz -Talarczyk, 2012). Weaving and knitting are the two most common methods used for manufacturing textiles.

Cotton was produced in Egypt around 12000 BC, and in India approximately 1500 BC; Linum fibre use goes back to 6500 BC (Kozlowski & Mackiewicz-Talarczyk, 2012). Silk, a protein filament fibre, is produced from silkworms that Chinese people have bred as early as 3000 BC. The history of Cotton documents its use for mankind since 5000 BC in India and the Middle East (Kozlowski, 2012a).

2.1.1 Natural cellulosic fibres

Vegetable or cellulosic fibres can be obtained from different parts of the plant such as seed (Cotton, kapok, coir), bast (*linum*, *corchorus*, *cannabis*, *boehmeria*, kenaf), and leaf (*agave*, *abaca*), and they are classified according to their source in the plant (Nayak et al., as cited by Kozlowski, 2012b, p. 314 –315). Marques et al. (2010) state that cell characteristics of size, thickness, and the shape and thickness of the lumen are used to identify a vegetable fibre. Natural fibres found in the stem and leaf parts of the plant consist of a complex composite of cellulose, hemicelluloses, lignins and aromatics, waxes and other lipids, ash and water-soluble compounds that are glued together in different layers of the cell walls (Akin, 2010). Cellulose itself is a linear polymer composed of β -d -glucose units, linked together by β -1,4-glycosidic bonds to expand a linear polymer chain (Kozlowski, 2012a). Hemicellulose exists in the natural fibres which are more coherent between cellulose and lignin (Thomas et al., as cited by Kalia et al., 2011). The degree of

polymerization of cellulose varies according to the plant species (7000 dp–15000 dp) (Akin, 2010). Usually the sclerenchyma elongated cells give supporting tissue in plants, providing much strength, and rigidity (Kozlowski, 2012a). Two important properties, xylem and phloem tissue of monocotyledonous and dicotyledonous, are involved fibres of plant stems and leaves (Smole et al., 2013).

Plant cells have distinctive cell walls: the primary wall cover with a loose irregular linkage of closely packed cellulose microfibrils, and the secondary cell wall that is divided into three sub-layers (Kozlowski, 2012a). The primary and secondary cell wall together can be considered a composite matrix of lignin and hemicellulosic polysaccharides (Krässig, 1993). The middle layer has helically arranged microfibrils that give the fibre its mechanical properties. Specific fibres have different microfibrillar orientation in three sub-layers (Krässig, 1993; Smole et al., 2013). When the secondary cell wall is thicker, the lumen becomes smaller. Cellulose forms a crystalline structure with regions of high order orientation amorphous regions of low order orientation.

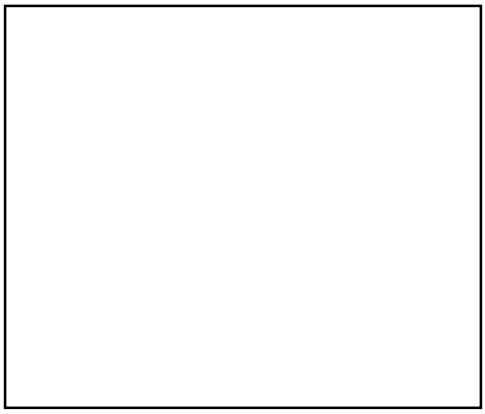


Figure 2.1: Schematic diagram of basic structure of natural fibre cell structure of the plant (Dungani et al, 2016). (Image removed due to copy right restrictions.)

The chemical composition as well as the morphological microstructure of vegetable fibres is extremely complex due to the hierarchical organization of the different compounds

present at various compositions. Depending on the type of fibre, the chemical composition of natural fibres varies. Primarily, fibres contain cellulose, hemicellulose and lignin. The property of each constituent contributes to the overall properties of the fibre. Table 2.1 compares the chemical composition among seed fibre (Cotton), bast fibres (*corchorus, linum, boehmeria, cannabis*), and leaf fibres (*agave, abaca*). The core component of all the fibres is cellulose (Table 2.1); Cotton has the highest cellulosic component.

Table 2.1: Chemical composition (%) of different plant fibres.

Fibre Name	**Cellulose %**	**Hemicellulose%**	**Pectin %**	**Lignin %**	**Wax %**	**Ash %**	**Others**	**Reference**
Cotton	92-95.00	5.7	<1.00	---	0.60	---	---	Dochia and Sirghie, as cited in Kozlowski, 2012.
Corchorus	61-75.50	13.6-20.4	0.20	12-13.00	0.50	---	---	Roy and Lutfar, 2012(b).
Linum	71.75.20	8.6-20.6	2.30	2.2-4.80	1.70	1.10	---	Kozlowski &Mackiewicz-Talarczyk, 2012.
Boehmeria	68-76.20	13-16.7	1.90	<0.70	1.70	1.10	---	Roy and Lutfar, 2012(a).
Cannabis	57-77.00	14-22.4	0.90	3.7-13.00	---	0.80	---	Li et at., 2007.
Agave	47-78.00	10-24.0	10.00	7-11.00	---	0.6-1.00	---	Li et at., 2007.
Abaca	56-63.70	17.5	1.00	15.1.00	---	1.10	---	Baltazar-YJimenez & Sain, 2012.
Kenaf	45-57.00	21.5	3.0-5.00	8.0-13.00	---	---	---	Nayak et al., 2012.
Cattail [a]	42.61	---	23.90	5.75	---	12.56	---	Sridach and Paladsongkhram, 2013.
Cattail [b]	51.30	---	----	22.05	4.25	3.20	3.6	Sana et al., 2016.
Cattail [c]	63.00	8.7	----	9.60	1.4	2.0	---	Sopit, 2007

[a]Narrow leaves; [b] Tunisian *Typha* leaf; [c]*Typha latifolia*

Introduction of bast fibres

Bast fibres have been used for over 8000 years (Smole et al., 2013). They are collected from stems or stalks of dicotolydenous plants. Their woody core structure is surrounded by a stem that consists of a number of fibre bundles or aggregates (Hearle, 1963) having 10 to 25 elementary fibres 2 to 5 mm long and 10 - 50 μm in diameter (Thomas et al., as cited by Kalia et al., 2011). Elementary fibrils and bundles are cemented by lignin and pectin intercellular substances, which must be extracted to obtain the fibres (Mohanty, 2005).

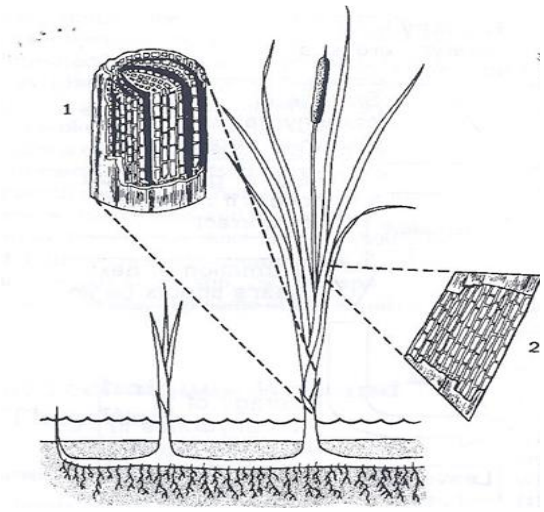


Figure 2.2: Cross-section of *Typha* stem from *Typha* plants (Sojda and Solberg, 1993).

Bast fibres usually contain higher amounts of cellulose (57%–77%), while hemicellulose (9%–14%) and lignin (5%–9%) content is lower compared to woody core fibres (Stevulova, 2014). Currently, bast fibres are raw materials not only for the textile industry, but also for modern environmentally-friendly composites in different applications such as building materials, particle boards, insulation boards, food, cosmetics, and medicine, and are a source for other biopolymers. Several species of Typha exist in North America and other parts of the world. The differences between them must be considered in the selection of a species for a particular site (Dubbe et al., 1988). *Typha latifolia* L. (broadleaf cattail), *Typha angustifolia* L. (narrow leaf cattail), and *Typha x glauca* (a hybrid of the other two species), known as wetland plants and charecteristics are aboveground leaves, below ground rhizome system, and recognizable inflorescence (Dubbe et al., 1988).

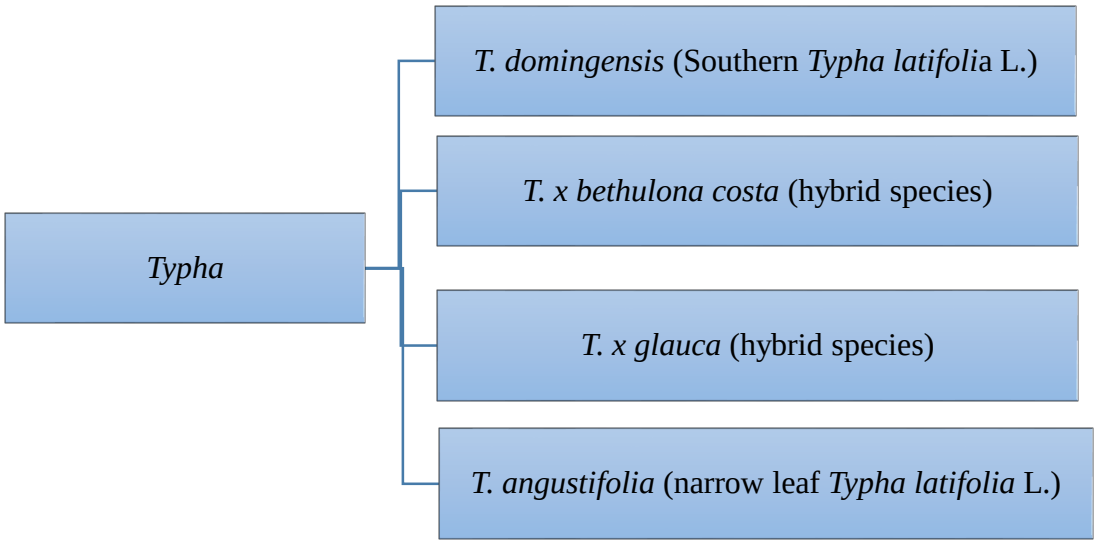
Species selection may affect yield potential, tolerance to various site conditions, nutrient uptake patterns, evapotranspiration rates, characteristics as fibre, and energy (Dubbe et al., 1988). The stem height of *Typha* ranges from 1 to 3 m with collective 12-16 linear and flat leaves that are 15 to 25 mm wide (Grace & Harrison, 1986). The *Typha* stalk is structured in one-piece, consisting of several reciprocally incorporated sheets. Each of these sheets is divided by thin walls into several individual areas. Every area of the sheet is divided into very fine 10 μm to 50 μm open pores. Both individual *Typha* sheets and the stalks consist of enough strong outer walls to fully retain their processing, i.e. cutting, sorting, knotting into bales, and pressing to a given density. The inner layer of *Typha* in the individual areas is filled with very weak and brittle material (Vėjelienė et al., 2011). The special characteristics of *Typha* include good tensile strength of stem fibre and elastic sponge-like tissue, and leaves that are tear and break resistant. These characteristics provide remarkable load-bearing capacity and excellent insulation property (Krus et al., 2014). In the most common form of vegetable fibres, the cellulose structure depends mainly on pectin and hemi-cellulose. In the randomly oriented amorphous regions, the higher the amorphousness, the greater is the vapour water or dye absorption rate of the fibre. In the crystalline regions, where the polymers are oriented or aligned longitudinally or parallel along the fibre axis, hydrogen bonding occurs (Thomas et al., as cited by Kalia et al., 2011).

Classification of the *Typha* plant

Typha has mostly colonial, rhizomatous, perennial characteristics in the division of *Magnoliophyta*, class *Liliopsida*, subclass *Commelinidae*, order *Typhales*, family *Typhaceae* (Sana et al., 2014). A monocotyledonous plant, *Typha* are considered important wetland plants of shoreline areas in temperate and boreal North America (Smith, 1986) and are also distributed throughout the tropical and temperate regions of the world in marshes and wetlands (Sana et al.,

2014). The *Typha* family contains two genera and 51 species. The two species having the widest range in the *Typha* family are *T. latifolia* and *T. angustifolia* (Fahlgren, 2017). Differences between these species must be considered in the selection, though species for all plants grow naturally.

Table 2.2 : *Typha* plant species in North America (Dubbe et al., 1988).



Current uses of *Typha latifolia* L. plants

The effects of cultivation of Canadian wetland environments are quite unknown. Challenges include the volume of harvested material, moisture content, general quality of the biomass for energy, calorific value, and the energy conversion technology. Recent studies have found harvesting of *Typha* from wetlands provides both economic and environmental benefits. *Typha* cellulose is considered a new vegetable fibre source (Sana et al., 2014). Studies have found that *Typha* can remove human disease-causing microorganisms and pollutants from water (Sharp, 2002). Moreover, *Typha* has significant environmental benefits by helping to reduce nutrient loading (i.e. phosphorus) in aquatic systems through capture and removal. The harvested *Typha* biomass is not only a renewable, sustainable biomass feedstock for energy production, but also

mitigates greenhouse gas (GHG) emissions. The recovery of phosphorus could be a precious resource for global food security (Grosshans, 2014).

In addition, researchers have established that *Typha* can be harvested and used as a source of "biofuel," meaning the plant material can be dried and burned for energy, reducing consumption of fossil fuel such as coal. *Typha* leaves may even be fermented to produce ethanol which has excellent energy properties for displacing gasoline (Lakshman, 1984 as cited by Grosshans, 2014). The phosphorus from *Typha* ash could be used as a crop fertilizer. *Typha* leaves have an interesting soft open-cell spongy tissue that provides an excellent insulating effect, fire protection, noise protection, elasticity or hydrophobicity (Krus et al., 2014). *Typha* stock comprises resilient, natural monocultures with an annual production rate of 15 to 20 tonnes of dry matter per hectare (Krus et al., 2014). This enormous growth rate of *Typha* may be appropriate as a raw material for industrial use. The *Typha* is a natural neglected plant (NNP) whose chemical constitutions are shown in Table 2.1.

2.1.2 Common textile fibres

Cotton is known as a plant seed fibre of the genus Cotton and the purest form of cellulose available in nature (Nevell, as cited by Shore, 1995). Cotton is very prominent in the textile industry because it has many desirable fibre properties making it a major fibre for textile applications. The properties of strength and good absorbency make it a comfortable and durable apparel fabric.

The commercial development of man-made fibres began in the early 20th century. The study of polymer thread-like molecules found great progress in the 1920s and 1930s, and in the 1930s Wallace H. Carothers developed the first known polymer fibre, Nylon (Khan, 2016), which was developed and manufactured by DuPont in the United States starting in March 1953 (Collier

et al., 2009). The commercial development of man-made fibres experienced much growth during the 1940s and expanded rapidly after World War II. Synthetic/man-made fibres have been in major use for general textiles since the 1950s. Today, many other classes of polymer have been developed for different purposes and applications such as polyester, polyamides, olefins, polyethylene terephthalate (PET), acrylic polyacrylontrile (PAN) and polypropylene (PP) (Cook, 2001).

Polyester fibres can be defined as fibre-forming substances of an aliphatic hydro-carbon (—CO·O·CH_2·CH_2·O·CO—) of linear condensation polymers composed of at least 85% by weight of an ester of a substituted aromatic carboxylic acid produced by melt-spinning and drawing (Cook, 2001). In aliphatic sequence, monomers are flexible at room temperature and bonded with weak van der Waals force. Polyester fibre is known for its many uses, and when blended with other fibres, it helps to increase durability of the product. Since Cotton and polyester are similar moduli they are ideal for blending.

Currently, textile fibres are not limited to traditional cloth manufacturing, but have diverse applications in almost all sectors such as agriculture, aquaculture, horticulture and forestry; reinforced polymer composites are used in aircraft, marine craft, automobiles, civil infrastructure, medical prosthesis and all kind of sports and leisure materials (Salit, 2014; Mohammed et al., 2015; Mussig, 2010).

Global fibre consumption increases as new products are developed. The world fibre consumption is 90% for yarn, 7% for nonwoven, and the rest for filling, cigarette filters and so on (Lawrence, 2003). The latest figures, published in 2001, showed 53% of the world production of textiles is synthetic polyester fibre; whereas, Cotton was only 33.3%. The other natural and synthetic fibre production is: wool 2.3%; cellulosics 4%; corchorus 5.8%; linen 0.3%; boehmeria 1.1%; and silk 0.1%, respectively.

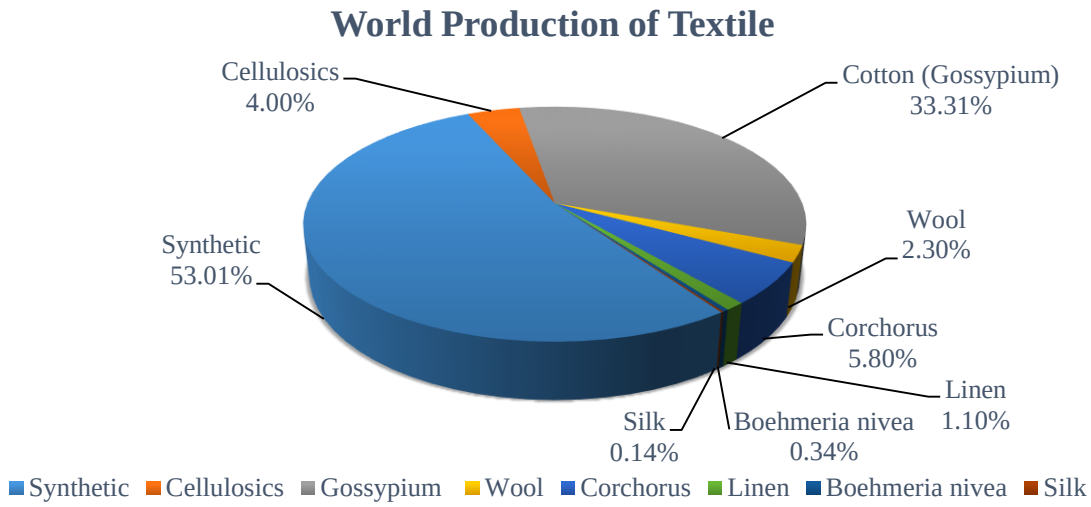


Figure 2.3: World production of Textile (Lawrence, 2003; pp.24).

Table 2.3 Physical properties of polyester and Cotton fibres (Ali, 2013; Kozlowski, 2012a).

Fibre Name	Length (mm) textile fibre from stem	Finished Fibre Length for spinning (mm)	Finished Fibre Diameter (microns, µg/inch)	Density (g/cm³)	Moisture (%) (65% RH, 21°C)	Fibre fineness (µm)	Tenacity (g/den)
Cotton	15-56	15 - 56	2-6.5	1.52 -1.56	8-11.0	16.0-21.0	1.7-6.3
Polyester	Controllable	Controllable	Controllable	1.36 -1.41	0.4	1.3-22.0	3.0-7.0

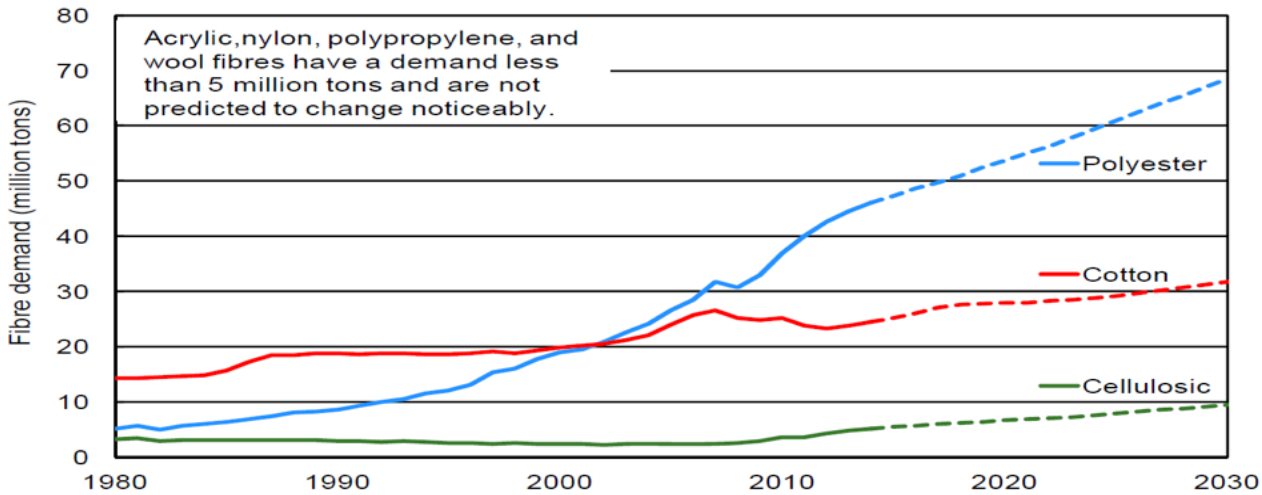


Figure 2.4: Future forecast of global fibre demand out of 2013 as calculated by PCI fibres. (Source: www.textileworld.com).

Figure 2.4 shows the demand for three dominant fibres, Cotton, polyester and cellulose, across history. Throughout the years, the demand for Cotton and polyester fibre has remained fairly constant, with cellulosic fibre experiencing a slight growth of 5 million tons. The consulting firm, England-based PCI Fibres, calculates that the estimated polyester fibre demand will increase until 2030.

2.2 Extraction of bast fibre

Bast fibre is extracted from plant stems through various methods. The most widely used is known as retting--a process of extracting and separating the bast fibre from the woody stems by a controlled degradation (Thomas et al., as cited by Kalia et al., 2011; Tahir et al., 2011) that breaks down or removes the gummy substance which binds the fibres together in the core of the plant, most commonly by dew and water (Kozlowski, 2012a). All retting processes influence the structure, chemical composition and properties of the fibre. Kozlowski (2012a) and Tamburini et al. (2004) found that the degree of retting significantly affects fibre quality and quantity. Different retting methods may be used such as biological, mechanical, chemical or enzymatic (Thomas et al., as cited by Kalia et al., 2011; Tahir et al., 2011).

Table 2.4: Plant fibre degumming processes (Kozlowski & Mackiewicz-Talarczyk, 2012).

Application methods of degumming				
Water		**Dew-retting**	**Chemical**	**Physical**
In river	**In tank**			
Cold water	Cold water	Nitrogen nourishment	Ethylene	Ultrasound oscillation
Warm water	Warm water	Desiccants	Oxalic acid	Electron radiation
Nitrogen nourishment	Nitrogen nourishment	Breaking of straw in root part	Sodium hydroxide	Steaming with application of pressure method
Aeration	Aeration		Potassium hydroxide	Steam explosion
Enzymes	Enzymes		Lithium hydroxide	Osmosis degumming
			Acid and sodium carbonate	High-power electromagnetic pulses
			Oxygenated water Sodium sulphite	

2.2.1 Biological retting

Biological retting is divided into two processes, natural and artificial (Thomas et al., as cited by Kalia et al., 2011). Biological retting involves in-field or dew retting, and cold water retting. Dew or field retting is the most commonly applied process to achieve satisfactory retting as a result of weather conditions, appropriate moisture and temperature ranges (Kozlowski & Mackiewicz-Talarczyk, 2012; Thomas et al., as cited by Kalia et al., 2011; Tahir et al., 2011). Stems from cut crops are spread over the surface of the field for 2-8 weeks depending on the location, and the degree of retting required (Kozlowski, 2012a). The stems are exposed to light, weather and temperature conditions. During the in-field retting process, microorganisms attack the stems and break down the cementations that bind together the fibre bundles (Kozlowski & Mackiewicz-Talarczyk, 2012). When sufficiently retted, the colour of the stems turns dark grey. Under-retting creates difficulty in separating and further processing the fibre; over retting degrades fibre quality; therefore, the process must be stopped at an appropriate time (Thomas et al., as cited by Kalia et al., 2011). Field retting can minimize labour input because it does not require the transport of crops as the retting methods are applied in the field, maximizing agricultural efficiency and cost (Kozlowski & Mackiewicz-Talarczyk, 2012).

The process of submerging plant stems into different water sources, such as a huge water tanks, ponds, rivers or vats, uses anaerobic bacteria to breakdown the pectin of the plant straw bundles (Thomas et al., as cited by Kalia et al., 2011). This retting process depends on water type, the temperature of the retting water and any bacterial inoculum. Water retting is often considered the best separation method compared to other methods as it takes only 7 to 14 days (Kozlowski & Mackiewicz-Talarczyk, 2012, Akin, D.E. as cited as Mussig, 2010). Applying heat between 30°C to 40°C may reduce the process time and provides a high quality fibre (Thomas et al., as cited by

Kalia et al., 2011); however, it leads to environmental pollution due to unacceptable organic fermentation in waste waters (Thomas et al., as cited by Kalia et al., 2011).

Another type of biological retting is artificial retting, which produces homogeneous fibres, works with warm water, and takes 3 to 5 days for high quality, clean fibres (Thomas et al., as cited by Kalia et al., 2011). The plant bundles are soaked in warm water tanks, and the best fibres are separated from the woody parts at completion of the retting process.

2.2.2 Mechanical retting

During mechanical retting, mechanical actions (scotching, hacking, hammer mill, roller mill) separate the fibre from the woody core part of the plant. Mechanical retting is a more economical and easy procedure using either field dried or slightly retting plant straw (Thomas et al., as cited by Kalia et al., 2011); however, it produces poorer fibres which are much coarser compared to dew or water retting fibres (Thomas et al., as cited by Kalia et al., 2011).

2.2.3 Chemical retting

In chemical retting, the plant stems are immersed into heated tanks containing solutions of sulphuric acid, chlorinated lime, sodium or potassium hydroxide and soda ash (Thomas et al., as cited by Kalia et al., 2011). The extraction process of removing non-cellulosic materials uses hot alkaline solution (Williams et al., 2011). This is commonly a dispersion and emulsion-forming process that uses surface active agents to remove unwanted non-cellulosic constituents which bind the fibres. It is the shortest process, taking only few hours, compared to days required for other retting methods. The yield of fibres in the chemical retting process is very good, but it adds an additional cost for the final products (Kozlowski & Mackiewicz-Talarczyk, 2012). From environmental aspect, cotton does not require any retting and hemp and flax needs bio-retting (Thomas et al., as cited by Kalia et al., 2011), whereas, chemical retting process has huge impects

on environmental pollution (Muthu, 2018). Therefore, it is urge to think the chemical retting westage water could be reusable by biological enzymetic treatment (Muthu, 2018), waste water tearment plant (Kumar and Saravanan, as cited by Muthu, 2018) will less impact on environment.

2.2.4 Enzymatic retting

A common process called microbial/enzymatic retting extracts good quality cellulosic fibres from vegetable plants such as cannabis, linum and corchorus (Thomas et al., as cited by Kalia et al., 2011; Tahir et al., 2011). Enzymes produced by fungus or bacteria weaken or remove the pectinic glue that bonds the fibre bundles and release the cellulosic fibres. This process is costly because it needs huge quantities of enzymes and equipment (Kozlowski & Mackiewicz-Talarczyk, 2012).

2.3 Characterization of bast fibre

2.3.1 Physical properties of textile fibres

The properties of natural fibres refer to fibre diameter, structure, degree of polymerization, crystal structure and source. A plant fibre shows various properties due to different growth conditions (Eder & Burgert, as cited by Mussig, 2010). To be suitable for apparel applications, a fibre must demonstrate essential fundamental properties of strength, tenacity and spinning power depending on the end uses. Fibres need strength when twisted together. This property implies a measure of cohesion between the individual fibres which will give strength to the yarn. Spinning of a fibre means there is a certain amount of surface roughness or serration promoted by fineness and uniformity of diameter. An important property of a fibre is flexural rigidity that increases with the decrease in fibre linear density. Fine fibres have lower bending rigidity (Kaushik, Tyagi, & Chatterjee, 1990). Some researchers have studied fibre properties, which are influenced by fibre

type, spinning system and twist on yarn flexural rigidity. Other fundamental properties which are desirable for a fibre are durability, softness, absence of undesirable colour and affinity for dyes.

A potential textile fibre must have some potential textile properties. The ability to withstand tensile force (tenacity) and extension at break are considered important parameters for the manufacturing of yarn in different processing stages such as spinning, winding, warping, sizing and fabric formation (weaving and knitting). In addition, fibre properties and behaviour directly affect fabric performance. The essential parameters for a textile fibre include length, flexibility, cohesiveness, and sufficient strength. Other significant properties include elasticity, fineness, uniformity, durability, and luster. For example of physical and mechanical properties are shown in Table 2.5 and 2.6 which are collected from literature (Franck, 2005).

Table 2.5: Physical properties of major natural bast and leaf fibres (Franck, 2005).

Physical characteristics	Fibre Types						
	Linum	**Cannabis**	**Kenaf**	**Corchorus**	**Boehmeria**	**Nettle**	**Agave**
Length of fibre (mm)	300-600	1000-3000	900-1800	150-360	1500	19-80	600-1000
Ultimate length (mm)	13-60	5-55	1.5-11	0.8-6	40-250	5.5	0.8-8
Diameter (µm)	12-30	16-50	14-33	5-25	16-125	20-80	100-400
Weight per length	1.7-17.8	3.20	50	13.27	4.6-6.4	--	9-400
Density (g/cm^3)	1.4	1.4	--	1.4	1.4	--	1.2-1.45

Table 2.6: Mechanical properties of major natural bast and leaf fibres (Franck, 2005).

Physical characteristics	Fibre Types					
	Linum	**Cannabis**	**Kenaf**	**Corchorus**	**Boehmeria**	**Agave**
Tensile strength (GPa)	0.90	0.31-0.39	0.18	0.22-0.53	0.29	0.08 - 0.839
Specific tensile strength (GPa m^3/kg)	0.60					0.07- 0.42
Flexibility modulus (GPa)	85			2.5-13		3-98
Specific flexibility modulus (GPa m^3 /kg)	71			9.0		3.82
Tensile modulus (GPa)				13		15
Elongation at break (%)	1.8-3.3	1.7-2.7	1.7-2.7	1.0-2.0	2.3-4.6	2.9 - 6.8
Specific tenacity (GPa m^3/kg)				0.37		0.44
Elasticity modulus (GPa)				0.26 - 0.32		0.15 - 0.19

Fibre length

Length is an important attribute of a textile fibre that influences process efficiency, quality of yarn (Kumar, 2015), performance and price (Morton & Hearle, 2008). Short or staple fibres are measured in inches or fraction of an inch: for example, 3/4 inch to 18 inches. All natural fibres except silk are staple fibres. Since staple fibres are a finite length, they are much more uniform in length than filament fibres. Filament fibres are long, often an indefinite length, measured in yards or meters. Silk filament is 360-1200 meters; man-made filament length can be continuous.

Fibre length can be determined from a fibrograph (Kumar, 2015) in which a formation beard of parallel fibres passes through an optical sensing point. This beard is formed when fibres from a sample are automatically grasped by a clamp, then combed and brushed into parallel orientation (Cotton Incorporated, 2016). Fibre diameter, fineness and maturity are three important physical properties that impact the spinning process and end uses of a fibre (Montalvo, 2005).

Fibre maturity in Cotton refers to the degree of cell wall development (Khan, 2016).

Fibre diameter

The fibre diameter, usually expressed in micrometer (μm), defines the distance across its cross section, or the length of a straight line through the center of a circle or sphere. In natural fibres, the diameter varies from one part of the fibre to another because of irregularities in fibre size. In contrast, manufactured fibres usually have a uniform diameter throughout, for example the diameter of polyester fibres used for apparel is presumed to be circular (Stout, 1960). Fibre diameter has significant importance in intermediate (sliver, yarn) and final products (fabric and apparel). Fibre diameter (d) affects numerous physical characteristics of yarn such as: yarn thickness or linear density in Ne (English Cotton count systems) (∝1/d), yarn bending rigidity (∝ d) and extension (∝ d), yarn twist (∝ 1/d) and some important fabric characteristics such as fabric

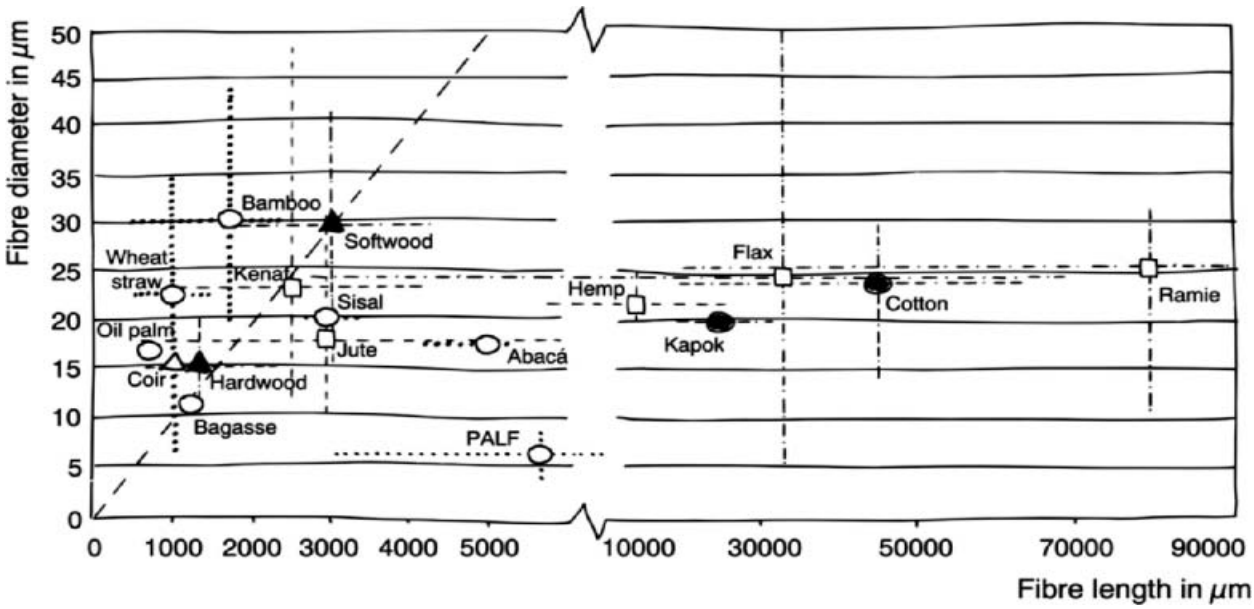


Figure 2.5: The length and diameter of major vegetable fibres. The linear curve represents an aspect ratio of 100 and estimate mean vaule with dotline shows the range of literature data (Ageeva et al. (2005), Aldaba (1927), Angelini et al.(2000), Ashori et al. (2006), Baley (2002), Jarman and laws (1965), Khalil et al.(2008), Kozlowski et al.(2005), Kundu (1956), Leupin (2001), McDougall et al. (1993), Morvan et al.(2003), Mukherjee and Satyanarayana(1986) and Ruys et al.(2002), as cited in Mussig, 2010).

weight, drape, handle, and comfort. Moreover, the price of a textile end product often depends on fibre diameter, as finer materials cost more.

Fibre fineness defines the diameter, cross-sectional area and linear density (weight per unit length) (Kumar, 2015). The mature Cotton fibre cell wall shows 50% to 80% moisture in the cell wall; whereas, immature fibre shows 30% to 45%, and dead fibres less than 25% (Kumar, 2015). A large number of finer fibres results in a stronger yarn. However, an abundance of dead fibre results in difficulties in the spinning process and a low strength yarn. These two attributes of maturity and fineness are measured using several methods including the USTER, Advanced Fibre Information System (AFIS), Shirley Development Fineness Maturity Tester, polarized light microscopy, fibre cross-section image analysis microscopy (IAM), and near infrared spectroscopy (Kim et al., 2014). The micronaire value of Cotton depends on specific surface and is influenced by both fibre fineness and maturity. A low micronaire value indicates finer fibres; whereas, high micronaire values usually indicate coarser fibres, which is problematic in Cotton spinning systems and yarn evenness (Montalvo, 2005).

Textile fibres must demonstrate special characteristics such as minimum length, tensile strength, higher initial modulus and cohesiveness with pliability. Tenacity, as defined by Kumar (2015), is the breaking force of a material divided by the linear density of the unstrained material and can be expressed as tex, lbf / tex, kgf / tex, cN / tex.

$\therefore$ Tenacity = Breaking force/ tex. (tex is a unit of measurement of liner mass density of fibre)

The tensile strength of corchorus and kenaf provides much stronger tenacity than other fibres because of their small elongation at break with higher initial modulus (Krishnan et al., as cited in Franck, 2005). Different plants have different cellulosic morphological alignments producing variations in mechanical properties.

The bonding natures between fibres, the orientation of the fibres within the bundles and micro fibril angle of cell walls (Eder & Burgert, as cited by Mussig, 2010) help to connect neighbouring fibres. Two adjacent fibres have opposite cellulose fibril orientations in the connected cell walls. Because fibres are bonded with tight interlocking, fibre bundles are stiffer than single fibres (Eder & Burgert, as cited by Mussig, 2010). Usually, the Pressley Index (PI) is used to indicate the fibre bundle strength. A fibre bundle with a PI value of 75-80 would be considered medium strength.

2.3.2 Spinning properties of fibres

Conditions of fibre length, fineness, strength, stiffness and single fibre entity determine the spinability of a fibre and the success of the subsequent weaving process. Spinning properties determine the quality of a fibre and increase the values in the textile market. The summary in Table 2.7 provides a ranking of the importance of fibre properties using three Cotton spinning methods: ring, rotor and air jet spinning (Gordon, as cited by Gordon & Hsieh, 2007) which can be used for characterization of the spinning properties of fibres.

Table 2.7: Ranking of fibre properties of Cotton for different short-staple spinning systems and length uniformity index (Gordon, as cited by Gordon & Hsieh, 2007; Kumar, 2015).

Priority ranking of property	**Ring spinning**	**Rotor spinning**	**Air-jet spinning**
1	Flexibility	Flexibility	Flexibility
2	Single fibre entity	Single fibre entity	Single fibre entity
3	Length	Strength	Length
4	Strength	Fineness	Trash
5	Fineness	Length	Fineness
6	N/A	Trash	Strength

HVI length uniformity Index					
Degree of uniformity	Very high	High	Intermediate	Low	Very Low
Range in (%)	>85	83-85	80-82	77-79	<77

Fibre fineness, a factor which limits the spinability of a yarn, is a measure of thickness in the crosswise direction (Kumar, 2015). In a given cross section of yarn, different numbers of fibres can be accommodated depending on fineness of the fibres. When more fibres are in a given cross section they are fine; fewer fibres are coarser. Fineness also controls spinning limit, luster and drape of the textile materials. For Cotton yarn, a micronnaire value of 3 - 4.9 is considered to be medium range fineness (Ali, 2013). Fibre length is another parameter for uniformity and tenacity of fibre spinning. Length uniformity is the ratio of the mean length and upper half mean length of the fibrograph, expressed as a percentage. Kumar (2015) defined fibre length measured by selecting average length of the longer one-half of the fibres, usually from the upper half mean length value obtained from a high-volume instrument (HVI) or 2.5% span length gained from a fibrogram. For example, if the fibres in a bale were all of equal length, then the mean length and upper half mean length would be equal and would give a uniformity index of 100.

Shorter fibres are problematic during the spinning process and produce lower quality yarn. Fibre length impacts spinning limit, yarn strength, yarn evenness, handle and luster of the product, yarn hairiness and productivity.

Fibre cohesive properties contribute to yarn strength. In Cotton, the natural breaking strength of individual Cotton fibres helps in spinning. The yarn strength correlates highly with fibre strength. The minimum strength for a textile fibre is approximately 6 cN/tex (Kumar, 2015). At optimum yarn twist, fibre tenacity has a greater effect on yarn tenacity than any other property. The fibre strength utilization is typically 50% - 60% for rotor yarn and 60%-70% for ring yarns (Kumar, 2015).

Elongation is another important fibre property required to accommodate the stresses applied during the spinning and weaving processes. Through elongation, strength is shared over

the whole yarn. The elongation range for Cotton is 5% to 7.6% (Kumar, 2015). Sometimes increasing fibre elongation may reduce the spinning end breakage and yarn strength. Fibre stiffness is an important factor for twisting during spinning. A more rigid fibre is more likely to break during twist spinning than a softer fibre. The relationship between length and diameter of the fibre affects its stiffness properties. With an increase in diameter, flexural rigidity also increases, so in order to reduce stiffness, the diameter should be a mid-range micronaire value (Ali, 2013). Fibres need to be a single entity to be spinnable and inhibit the thick and thin places in the resulting yarn and fabric. In their natural state, Cotton fibres do not require any physical or chemical processing to make them single entities, unlike bast fibres (Khan, 2016).

2.3.3 Blending properties

The basic spinning properties for any textile fibre can be denoted by fibre length, strength, fineness, elongation, trash, stiffness, individuality and twist ability (Gordon, as cited by Gordon & Hsieh, 2007). Since *Typha* fibre by itself is difficult to spin, it has been blended with Cotton and other natural fibres according to four different ratios before being spun on a ring spinning machine (Liu et al., 2011). The blended Cotton *Typha* fibres gave properties capable for spinning (Khan, 2016). The strength of the blended yarns was higher than that of the pure Cotton yarn (Liu et al., 2011), and the highest strength was found when the blending ratio of *Typha* and Cotton was 20/80. The number of thin places and slubs of blending yarn increase, while the neps and breaking elongation decrease as the content of *Typha* fibre increases (Liu et al., 2011).

2.3.4 Thermal properties

The thermal effects of fibres are influenced by factors such as thermal conductivity, specific heat and its variation with temperature, the coefficient of thermal expansion, the melting point, and the latent heat of melting (Morton & Hearle, 2008). It is important to know thermal properties when a

fibre interacts with outside heat sources to see how rapidly the fibre heats up. Specific heat, or the amount of heat required to increase the temperature of a material by 1°C, is a term used to measure this thermal property. The specific heat of several different polymers was measured in a search for transition effects in their structure (Morton & Hearle, 2008). Batra, as cited by Lewin & Pearce, (1998) stated that most bast fibres show a small difference in heat capacity, for example, linum 0.322 (cal /g/°C), Corchorus 0.324 (cal /g/°C) and Cannabis 0.323 (cal /g/°C). Nylon, on the other hand, shows a specific heat of 260°C that is comparatively higher than bast fibres. The thermal conductivity of a textile material depends on the air entrapped within it. Dimensional changes in fabrics due to reversible swelling on moisture absorption are much larger than those due to reversible thermal expansion (Morton & Hearle, 2008). During the dyeing process thermal properties play a vital role; different temperatures are applied by the characteristics of the dyes and fibres (Khan, 2016). When dyeing with reactive dyes, temperature is raised to 90°C for the cellulose fibre dyeing treatment process (King, 2007).

Table 2.8: Specific Heat for Textile fibres (Morton & Hearle, 2008).

Fibre types	**Specific Heat (J/ (g K)**
Cotton	1.22–1.35
Rayon	1.35–1.59
Wool	1.36
Silk	1.38
Nylon 6	1.43
Nylon 6.6	1.46
Polyester (PET)	1.03
Asbestos	1.05
Glass	0.80

2.4 Modification of bast fibre

The bast fibre surface can be improved significantly to improve fineness and single fibre entity and to reduce fibre stiffness using mechanical, chemical or microbial action (Zawani et al., 2015). Treatments using silane, alkali, peroxide, and isocyanate affect the properties of natural fibres (Sgriccia et al., 2008).

2.4.1 Alkalization/Mercerization

Binding materials such as lignin, pectin, waxy substances, and natural oils cover the external cell wall of cellulose fibres and produce a rough surface topography. Through alkalization, these non-cellulosic materials are removed, making a fine structure of the fibre (Mwaikambo & Ansell, 2002; AL-Oqla et al., as cited by Khalid et al., 2014). The most widely used chemical treatments for modification of the surface properties of a bast fibre use sodium hydroxide (NaOH). When treated with different alkalis, bast fibres react differently due to their different chemical compositions. Alkali concentrations penetrate into the inter-crystalline regions swelling and

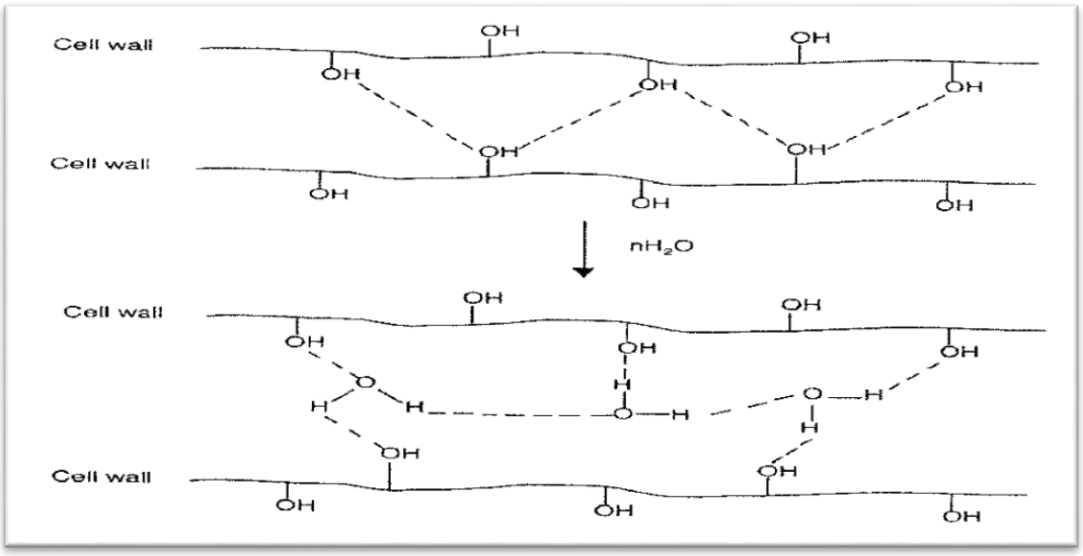


Figure 2.6: Schematic presentation of alkali effect on swelling process in cellulose (Mwaikambo & Ansell, 2002).

modifying these crystalline regions (Ali, 2013) in Figure 2.7.

When alkali is applied to cellulose the following reaction takes place:

$$\text{Cell} - \text{OH} + \text{NaOH} \longrightarrow \text{Cell} - \text{O}^{+}\,\text{NA}^{-} - \text{H}_2\text{O} + [\text{surface impurities}].$$

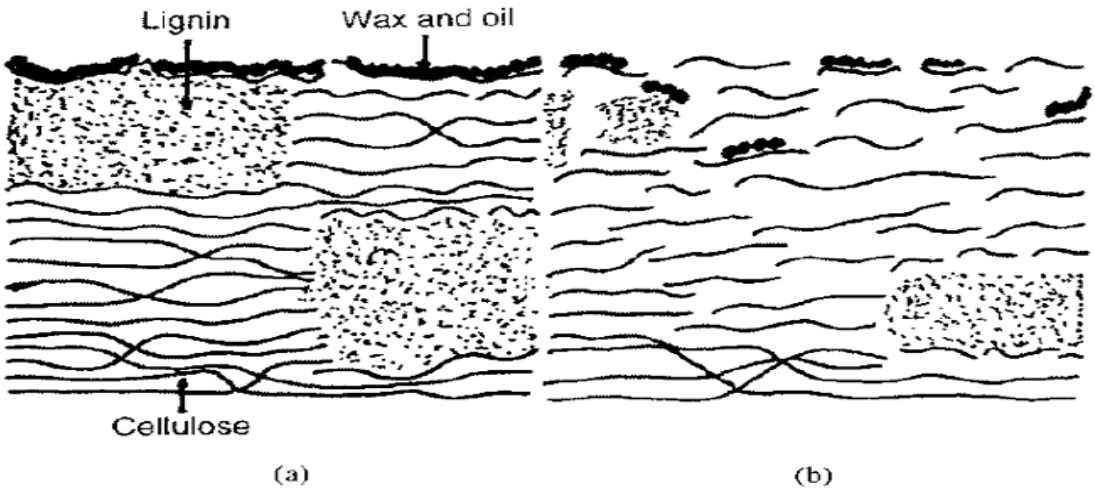


Figure 2.7: (a) untreated molecular structure and (b) alkalization treatment cellulosic fibre (Alkali treatment molecular structure depolymerizes and creates short length crystallites) (Mwaikambo & Ansell, 2002).

Xiao et al., (2001) observed that one of the simplest treatments, using KOH, was the most effective and economical treatment that had the least environmental impact. The effect of varying alkali concentrations, treatment, temperatures and times during mercerization of natural fibres are discussed by Zhang et al. (2005). Alkaline treatment can improve the cellulosic fibril, the degree of polymerization, and the extraction of lignin and hemi-cellulosic compounds (AL-Oqla el at., as cited by Khalid et al., 2014). In addition, NaOH treatment may increase tenacity and improve the surface morphology (AL-Oqla et al., as cited by Khalid et al., 2014). Mercerization used on corchorus under tension results in a fibre with an improved appearance and handling (Kozlowski, 2012b).

2.4.2 Dyeing of textile fibres

Dyeing is a process used to enhance the appearance of fibrous materials. Natural dyes have been obtained from vegetables, fruits, flowers, insects and fish since 3500 BC but due to the dull shade obtained, natural dye use is limited (Patra & Paul, as cited by Fu, 2013). Because of the wider colour range and brighter colours, synthetic dyes such as reactive dyes, direct dyes, vat dyes, and sulphur dyes have been developed for cellulose fibre dyeing (Khan, 2016). The mechanism of direct dyes on cellulose fibre is physical sorption of water-soluble dyestuffs by hydrogen bonds; mechanical retention of water insoluble colours (vat, sulphur and azoic dyes) on cellulosic fibres; and the dyestuffs reacting with fibre molecules to form covalent dye-fibre bonds (reactive dyes). Among these, reactive dyes are most widely used because they are suitable for natural fibres, a wide range of colour is available, fixation is efficient, fastness is good, and the dyes are less costly (Patra & Paul, as cited by Fu, 2013). Vat dyes are most expensive, and disperse dyes have poor colour fastness (Collier et al., 2009). Reactive dyes are classified into three groups: monofunctional, bifunctional, and polyfunctional. Usually the reactive dyeing process is carried out in traditional batch treatments compared to the modern high-speed continuous processes (Patra & Paul, as cited by Fu, 2013).

CHAPTER 3: MATERIALS AND METHODS

This chapter discusses the details of *Typha latifolia* L. material; an extraction method used to obtain *Typha* fibres, the textile properties of the *Typha* fibres, and the improvements to the physical and mechanical properties of the *Typha* fibres to make quality yarns.

For the initial experiments, *Typha* samples were collected from the Assiniboine forest and others were collected from previously harvested and stored samples (Location of Netley-Libau Marsh land, at the south end of Lake Winnipeg). All samples were simply cut using a knife. The plants were collected during the fall season and winter seasons and were identified as *Typha*, a green plant with a cylindrical dark brown flower. The stem is light green in colour, and 6-12 mm in diameter (Figure 3.1). The branches have 4-10 leaves or blades which measure 2.5 m long and 10-27 mm wide.

Figure 3.1: *Typha latifolia* L. plants

The leaf of the *Typha* plant was cut into 5 to 6 cm lengths. Different chemicals were used for the retting of the *Typha* stem to obtain the fibre. Below is the name of chemicals, supplier and

purity of chemicals, which were used for retting purposes.

Table 3.1: Chemicals and other materials used for *Typha* plant treatment.

Chemical	Supplier	Purity
Sodium hydroxide (NaOH)	Sigma Aldrich-USA	≥ 97%
Potassium hydroxide (KOH)	Sigma Aldrich-USA	≥ 97%
Lithium hydroxide	Sigma Aldrich-USA	≥ 97%
Acetic acid	Kimax-35 USA	5g/l
Hydrogen peroxide	Sigma Aldrich-USA	≥ 97%
Softener	R.H. Corporation –Switzerland, EU	0.10 kgs
Ethylene diaminetetraacetic Acid (EDTA)	Sigma Aldrich-USA	99.4 – 100.6%
Pectinase from aspergillus aculeatus	Sigma Aldrich-USA	≥ 3800 units/ml

3.1 Retting and Fibre Extraction

To make fibre bundles appropriate for spinning, the non-cellulose substances must be removed through fibre extraction or degumming processes. Water retting, the most widely practiced method of retting, was used to extract the fibres from epidermis (outer surface) of the plant stem. The *Typha* plants were cut in 4 cm and 5 cm lengths and were placed into separate beakers containing cold water, hot water, acid, alkali, or enzyme treatment, all at room temperature (21°C). To obtain better fibre extraction, a Launder-o-meter (SDL-Atlas) was used with a machine speed of 40±2 rpm and a pre-determined set of times and temperature. Following completion of the treatment, fibres were manually separated from the plants and washed in distilled cold water. The details of all chemical treatments are summarized in Table 3.2.

Table 3.2: *Typha* plant fibre extraction methods.

Sample #	Retting process	Retting temp. (°C)	Time (h)	Concentration of Chemical	Material to liquor ratio
S1-W	Water	20	24	--	1:50
S1-W	Water	20	384	--	1:50
S1-Acid	Water	20	384	--	1:50
S1-Alkali	Water	20	384	--	1:50
S1-Alcohol	Water	20	384	--	1:50
S1-Enzyme	Water	20	384	--	1:50
S1-HW	Hot water	80	2	---	1:50
S2-NaOH	Alkaline solution	80	1	1% NaOH	1:50
S3-NaOH	Alkaline solution	80	2	1% NaOH	1:50
S4-Acid	Acidic solution	80	2	1% Acetic Acid	1:50
S5 -NaOH	Alkaline solution	80	4	3% NaOH	1:50
S6-Acid	Acidic solution	80	4	3% Acetic Acid	1:50
S7-Enzyme	Enzyme solution	40	4	3% Enzyme (Pectinase from Aspergillus Aculeatus)	1:50

*S: sample; *W: water

3.2 Fibre extraction in alkali

The virgin *Typha* plants were scissor cut into 4 cm lengths to fit the Launder-o-meter canister, conditioned at standard atmospheric climate (Temperature 20°C, relative humidity 65%) and weighed using an electronic balance. The extraction chemicals (see Table 3.1 above), were added to canisters in 150 ml aqueous solutions. Then the launder-o-meter was heated to 80°C for water, acid, and alkali treatments, and to 40°C for the enzyme treatment. According to the fibre surface modification recipe, 1% chemical concentration for 1 hour and 2 hours, and 3% for 4 hours

was used as a trial of various times and temperature, to find the best fibre extraction efficiency, absorbency and dye take up of the resultant materials.

After treatment, it was observed that the fibre bundles were clustered, so the individual fibres were separated manually. Five minutes washing with distilled tap water neutralized the treated fibres. Subsequently, the samples were dried and reweighed to calculate the weight loss and the yield percentages. At the end of this process, the fibres were conditioned at standard atmospheric conditions (temperature 20°C and relative humidity 65%).

Based on the first experiment, it was determined the alkali medium treatment worked more efficiently than the others. In the second phase, a different alkali, potassium hydroxide (KOH), was used to compare the fibre extraction efficiency to the sodium hydroxide (NaOH) treatment used in the first experiment. Each experiments was conducted four times for four types of samples (for example S8-K1-MS, S8-K1-HS, S8-K1-SS, and S8-K1-NS) with same concentrations of alkali, and temperatures were replicated, and times were increased to determine the effect on fibre extraction efficiency (see Table 3.3).

Table 3.3: Experimental conditions (3% chemical concentration and 1:50 liquor ratio) for chemical treatment of *Typha* fibres.

Sample#	**Treatment time (h)**	**Retting temp. (°C)**
S8-K1	2	80
S8-K2	4	80
S8-K3	6	80
S8-K4	8	80

*S: sample; *KOH: Potassium hydroxide

To determine the significance of temperature on fibre extraction, samples were treated with KOH in 150 ml aqueous solution and run in the Launder-o-meter at a temperature of 95°C for times of 2, 4, 6, and 8 hours, and the resulting yield percentage was calculated.

Table 3.4: Experimental conditions (3% chemical concentration and 1:50 liquor ratio) for KOH treatment of *Typha* fibres.

Sample#	**Treatment time (h)**	**Retting temp. (°C)**
S9-K5	2	95
S9-K6	4	95
S9-K7	6	95
S9-K8	8	95

*S: sample; *KOH: Potassium hydroxide

Another fibre extraction method using lithium hydroxide (LiOH) in 3% concentration solution was tested to see if it would perform better in the extraction and separation of the fibres. The samples were treated in the solution (Table 3.5) in the launder-o-meter using different time cycles.

Table 3.5: Experimental conditions (3% chemical concentration and 1:50 liquor ratio) for LiOH treatment of *Typha* fibres.

Sample#	**Treatment time (h)**	**Retting temp. (°C)**
S10-L1	2	80
S10-L2	4	80
S10-L3	6	80
S10-L4	8	80

*S: sample; *L: Lithium hydroxide

3.3 Fibre Characterization

In the table below, methods which were used in *Typha* fibre properties characterization are described (Table 3.6).

Table 3.6: Characteristics of extracted *Typha* fibre.

Fibre characteristics	Machine/Method Used	Test Method
Diameter (µm)	Bioquant life science image analyzer	France standard NF G 07-004 (1983)
Length (mm)	Scissor and Ruler	
Moisture regain (%)	Conditioning & Thermo Scientific Oven	ASTM D2564
Fibre softness	Visual and Hand	AATCC, 2010c
Investigate functional groups	FTIR*	
Thermal behaviour	Burner and Tweezers	
Fibre decomposition point	Linkam Scientific Instruments, Model: T95-HS, 2009)	
Microscopic image evaluation	SEM*	
Yield Measurement	Oven	(Gravimetric method) standard: NF G 08- 001
Dyeing	Conventional dyeing method	
Dye absorbency	LabScan XE (Hunter Lab)	
Evaluation of dyeing performance	Colour fastness to hot pressing: dry hot pressing and wet hot pressing	(AATCC) standard: 133-2009 (AATCC, 1985)

*FTIR = Fourier- transform infrared spectroscopy, *SEM = Scanning electron microscopy.

3.3.1 Yield Measurement

The yield of fibres (R %) is measured by the percentage of the ratio between the mass of the fibres after the chemical extraction process (M_f) compared to the mass before the chemical extraction process (M_i). These materials are placed in an oven equipped with a ventilator and a thermostat and dried. The drying temperature depends on the nature of the fibre, and is usually slightly greater than 100 °C (about 105 °C). Dehydration by heating is carried out for 3 to 8 hours

until a dry or almost dry constant weight (M_f) is reached. The gravimetric method was used with the following standard: NF G 08- 001, to calculate fibre yield (%).

$\therefore$ $R\ (\%) = (M_f / M_i) * 100$ (Eq. 1)

3.3.2 Statistical analysis of *Typha* fibre length

The analysis can be considered a completely randomized design in the selection of individual fibres from fibre bundles, and the two treatments selected from the 12 treatments shown in Appendix III.

For Treatment 1: S8-K4-MS and Treatment 2: S10-L4-MS, the model can be expressed as shown in (Eq. 1).

$y_{ij} = \mu + h_i + e_{ij}$ (Eq.2)

In Eq. 1:

y_{ij} = Fibre length of j^{th} observation on i^{th} treatment.

μ = Population mean.

h_i = Effect of i^{th} treatment, i = 1 to 2 and j= 1 to 15;

e_{ij} = error deviation.

From this model, the null hypothesis is, h_O: $h_i = h_i'$ - Effects are same; the alternative hypothesis is h_a: $h_i \neq h_i'$ - Effects are different;

3.3.3 Diameter measurement

The diameter of the fibre was determined using a Bioquant Analyzer, which was connected to a computer, a projection microscope and a camera. The *Typha* fibre was prepared on a glass slide and observed using 40x magnifications. Computer software was used to calculate the fibre

diameter. The same method was used to determine the diameter of *Typha* fibre samples from fibre bundles along the length of each fibre. Statistical analysis was done to compare the fibre diameter of *Typha*, Cotton and wool. To determine diameter, France standard NF G 07- 004 (1983) and unit was considered as micrometer (µm). For this diameter comparison, 10 single fibre samples were chosen and 10 diameter readings from each fibre, which are shown in 5.4 (a), 5.4 (b) and 5.4 (c), were taken and the average diameter of 10 readings (fibre #1 – fibre# 10) with the standard deviation is calculated (Table 5.3(a)). For comparison, two of the most commonly used fibres, Cotton and wool, were also measured. The individual reading for 100 diameter values are given in Appendix III.

3.3.4 Softness Measurement

The softness of *Typha* fibre was evaluated by a survey method, according to AATCC evaluation procedure 5 (AATCC, 2010a) standard, which results in a numeric scale for the purpose of softness grading. Fibres bundles are taped to a piece of cardboard and placed into small polybags (2 x 2 inches), and evaluators graded it for softness in a range of 1- 5. The lowest softness (in terms of easy fibre separation and touch feeling) is given a rating of 1, and 5 indicates the highest softness (in terms of easy fibre separation and touch feeling method). Softness data for textile fibre Cotton, silk, wool, nylon, acrylic, polyester, linum, brassica and *Typha* (virgin) fibre and modified *Typha* fibre were used to develop the test method for this evaluation. The common textile fibres were used to compare the standardization with sample fibres in Table 5.23.

3.3.5 Moisture regain (%) measurements

The moisture regain of *Typha* fibre was measured according to the ASTM D 1776- 4 (2008). For this experiment, the humidity chamber was used at the University Manitoba grain storage building. Before starting the moisture regain test, all samples were oven dried at 105°C for eight

hours. Then the samples were held in a humidity chamber using a range of relative humidity and temperatures of 50%, 59%, 61% and 65% RH and 20°C, 21°C, 24.9°C and 25°C respectively, with various conditioning times. The moisture regain was calculated as the ratio of the amount of water absorbed to the dry weight of the sample.

$$\therefore \text{ Moisture Regain (\%)} = \frac{W}{O} \times 100\% \qquad \text{(Eq. 3)}$$

Where: W= Weight of water in the fibres (g);

O= Oven dry fibre weight (g)

3.3.6 Infrared (IR) spectrum study

Sample preparation

IR spectroscopy helps to determine the functional groups of a fibre through electromagnetic wavelengths (relatively sharp absorption bands and transmission energy source) that are displayed on a monitor. Since *Typha* fibre is considered a lignocellulosic fibre having a heterogeneous structure (Sana et al., 2014), IR spectroscopy was the best application to determine the functional groups and chemical structure for the *Typha* fibres.

IR spectra measurement

Samples were prepared, placed in the sample holder of the instrument, and the spectra were recorded over the range of wavelengths between 500–4000 cm^{-1}.

3.3.7 Thermal Analysis

Burning behaviour

To compare the burning characteristics of *Typha* fibre to Cotton and polyester fibres, the most common test is the burn-test-response method. In this experiment, tweezers hold the *Typha* Cotton and polyester fibre clusters, the fibres are slowly brought toward the flame of a Bunsen

burner at 45° angle, and the reaction on approach to the flame is observed. Further, the burning behaviour (while in the flame) and the reaction following removal from the flame are also observed.

Decomposition temperature

During end use application, fibres go through various processes for dyeing or other high temperature manufacturing and laundering procedures, so it is important to know the thermal decomposition temperature to determine the ability to withstand high temperature. To measure *typha* fibre thermal properties, a decomposing point analyzer was used: Linkam Scientific Instruments, Model: T95-HS, 2009) combined with an imaging station (Linkam Scientific Instrument, UK). The fibre bundle specimens were placed in a glass slide and the temperature was raised at a set rate to a maximum temperature in the analyzer. The decomposition temperature was determined by the visual analysis of the colour change on the sample displayed on the monitor (Brand: Dynax, Model: DX- 22L 150A11, Made: USA). The decomposition points of *Typha* plant, *Typha* fibres and Cotton samples were determined.

3.3.8 Scanning electron microscopy (SEM)

A scanning electron microscope (SEM), located at Manitoba Institute for Material (MIM) at the University of Manitoba, was used to examine the *Typha* fibre surface and cross-section. Samples were prepared from 3% KOH treated fibre (Sample 1), 3% LiOH treated fibre (Sample 2), and untreated *Typha* plant (Sample 3), with and without gold coating, and mounted on the SEM stub for examination. Morphologic details were collected with ETD (Everhart-Thornley detector) in HiVac at low pressure for coated *Typha* samples, and LFD (large Detector Field) LoVac mode at high pressure for uncoated *Typha* samples, Concentric Backscattered Electron (BSE) Detector

(CBS) in Hi and LoVac mode at high pressure for *Typha* samples. The SEM was used for imaging at different magnifications from low to high (100x to 12000x) for context.

3.3.9 *Typha* fibre dyeing with reactive dyes

Natural fibres have the ability to absorb dye materials. In order to find out the dyeability of *Typha* fibre an exhaustion dyeing technique was used. Seven hundred (700ml) liquor (dye solution) of reactive blue 4 in the ratio of 1:700 was prepared in a beaker at room temperature and separated into 7–100ml beakers. To each beaker was added 2.0 gram sequestering agent Ethylenediaminetetraacetic acid (EDTA) to deactivate metal ions, and 0.5 gram non-ionic surfactant was added as a wetting agent. Each of the alkali treated fibres S5-NaOH, S8-K2, S10-L2, S8-K3, S10-L1, S9-K5, and Cotton were immersed into a prepared beaker, and the beakers were placed in a Corning PC-400 hotplate, and heated to 35˚C. After 5 minutes at this temperature, a dosing of 2.5 grams glauber salt ($Na_2SO4{\cdot}10H20$) and 1.5 gram soda ash (Na_2CO_3) was added for dyeing fixation; heating was raised to 60˚C and held for 10 minutes. A further 2.5 grams salt (NaCl) and 1.5 gram soda ash were added to each beaker and heating continued at 60˚C for a further 45 minutes.

During the test, the dyeing process curve was followed. Dye molecules adsorption (%) increases rapidly during the first 5 to 10 minutes, called the primary exhaustion. Alkali in dye bath helps to increase the dye uptake and as the dye particles begin to react with the hydroxyl groups of the *Typha* fibre, more reactive dye is absorbed from the bath. Alkali is added, and the reaction continues slowly until the equilibrium state (secondary exhaustion) is reached at ~ 60 min. For better colourfastness, the post-treatment process is very important. During the post-treatment process, a hot wash confirms that fibre samples are totally free from unfixed dyes. Then the samples are immersed in 100 ml water with 10ml highly concentrated (50% v/v) hydrochloric acid

(HCl) for 15 minutes at 60˚C to neutralize additional salts and alkalis, rinsed in cold water and dried at room temperature for 24 hours.

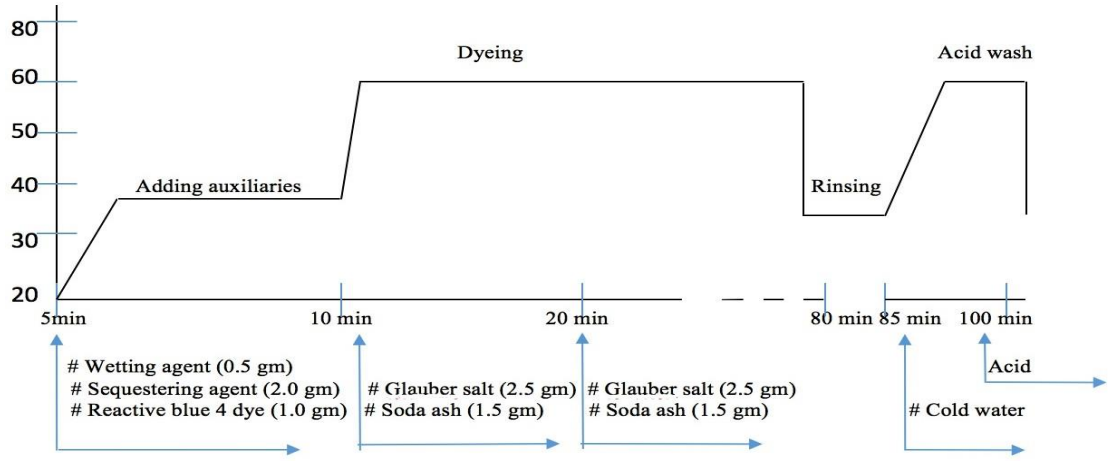


Figure 3.2: Dyeing process curve.

3.3.10 Colour differences of dyed fibres

In order to find the colour differences of dyed material, it is important to know the dyeing absorbency of the dyed fibres. The computer-controlled LabScan XE (Hunter Lab) spectrophotometer was used to indicate the degree of dye take-up by the samples S5-NaOH, S8-K2, S10-L2, S8-K3, S10-L1, S9-K5, and Cotton. Optical geometry was used to measure the colour differences between the after dyeing process liquor colours and the after-treatment dyeing liquor colours. Following this method, the dyeing liquor was measured through the bottom of a clear glass sample cup and the port-up orientation dyeing liquor beaker covered 1.75 inch sample cup viewing port. During the experiment, the recommended colour scale CIE 1976 L*a*b* system and D65 recommended illuminant were used. The Commission International d'Eclairage has established the principle of colour measurement. The colour value is expressed in terms of colour space L*, hue angle, and chroma values (Weatherall and Bernard Coombs, 1992). CIELAB colour

space is shown in the below Figure 3.3. Lightness L* is denoted in vertical axis with white (L* = 100) at the top and black (L* = 0) at the bottom. The value a* and b* axes signifies chromatic colours. The value a* axis – when colour red represents positive values and negative for green. Similarly, positive b* values represent yellow and blue for negative b* values.

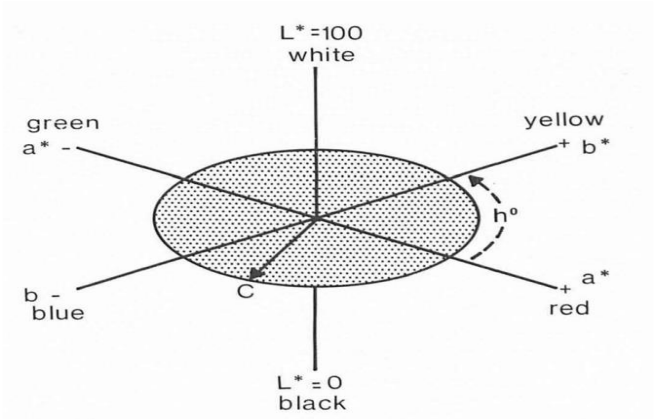


Figure 3.3: CIELAB colour space (Weatherall & Coombs, 1992).

The colour difference (ΔE) is calculated, using coordinate geometry, as the length of the line joining the coordinate positions: The Total colour difference (ΔE), the difference in individual parameters of the standard and a sample are also estimated e.g. ΔL = L (sample) - L (standard).

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad \text{(Eq. 4)}$$

3.3.11 Evaluation of dyeing performance (colourfastness to hot pressing)

End use textile products are always subjected to some major factors including moisture, light, heat, and abrasion during processing (Khan, 2016). Using the American Association of Textile Chemists and Colorists (AATCC) standard: 133-2009 (AATCC, 1985), the subjective dyeing performance (colourfastness to hot pressing) of dyed samples S10-L2, S10-L1, S8-K3, S9-K5, S8-K2, S5-NaOH, and Cotton were evaluated to determine colour resistance of dyed fibres to colour change, and colour transfer when subjected to hot pressing.

The Atlas Scorch Tester (SDL, Model M247B) containing a heating device was used to conduct colourfastness to hot pressing evaluation using fibres that were wet, dry, and damp at 65 ± 2% relative humidity and temperature 21± 1°C. For dry pressing, the samples were placed on top of the undyed Cotton fabric (130 g/m2) /wool flannel (260 g/m2) pad and subjected to a specific pressing temperature for 15 seconds. The heating device gave an even heat transfer to the sample between the top plate and bottom plates. In damp pressing, a similar process was followed, but one extra piece of undyed cloth (dimension: 12 × 4 cm) was soaked with distilled water, then squeezed until it contained its own weight of water, placed on the top the sample, and held 15 seconds with even hot pressing. For wet pressing, the samples and an extra piece of undyed cloth (dimension: 12 cm × 4 cm) both were wet during the pressing process. Evaluation uses a grey scale for staining (revised 1979, 1981 by AATCC / ISO 105-A03 methods) and a grey scale for colour change (revised 1979 by AATCC / ISO 105-A02). Grey scale is graded in 9 different grades such as 1, 1-2, 2, 2-3, 3, 3-4, 4, 4-5 and 5, where grade 1 denotes the highest staining or colour change, and grade 5 denotes no staining or colour change during washing (Collier & Epps, 1999).

3.3.12 Statistical analysis

Different statistical analytical techniques are employed in this current research in chapters 4.0 and 5.0. A single Factor ANOVA used, two-way ANOVA test, Fisher's LSD and Tukey test were considered to test the hypothesis. The test for the significance of the factor round by defining a linear model and creating an ANOVA-Table and Post Hoc test method used for a multi-comparison. This statistical analyses were performed with Excel-16.0 version and the SPSS version 16.0 statistical program for Windows system (SPSS) and able to test the effect size, main effects and interaction between individual variables, significant difference within the variables. Differences were considered significant at p =0.05.

CHAPTER 4: EXTRACTION OF *TYPHA LATIFOLIA* L. FIBRE

4.1 General overview

The successful extraction of cellulosic fibre from plant biomass depends on various factors, such as, the chemical composition of the plant and methods of extraction (dew retting, mechanical, water, enzyme, acid and alkali) (Dungani et al., 2016). Several methods have been used to separate the fibres from the materials surrounding them, as discussed in Section 3. Fibres that are separated from bast plants are often referred to as 'virgin fibres', are usually coarser and stiffer than Cotton, and are attached to each other (Khan, 2016). Therefore, further processes, for example, enzymatic, mechanical or physio-chemical treatments are needed to make them suitable for apparel and non-apparel applications (Dungani et al., 2016). In this chapter, the extraction methods of fibre from *Typha* plants are presented.

4.1.1 Water retting

In the present study, after five days of water retting, no fibre could be detected, the plant samples were immersed in the same water again, checked daily, and removed after 10 days for further inspection. At the completion of 10 days of water retting, no fibre could be separated, and the plant stems were re-immersed into the same water bath. After 16 days, the samples were finally removed as it is known that bast fibre is usually retted within two weeks in water, i.e. traditional corchorus retting for about 15-18 days (Roy & Lutfar, as cited in Kozlowski, 2012(b), p.24 - 46) and brassica between 10 to 20 days (Khan, 2016). However, for the *Typha* plant, the water retting method (Sample ID: S1-W) did not produce any fibre at 21°C after 16 days. The results are given in Table 4.1. The plant samples before and after 16 days of water retting are shown in Figures 4.1 and 4.2 respectively. The result of water retting was unexpected for 16 days at 21°C; therefore, a

longer retting time of up to 90 days at room temperature (Sample ID: S3-W; Figure 4.4) and higher temperature of 80°C for up to 6 hours (Sample: S2-W; Figure 4.3) were investigated. No *Typha* fibre could be obtained from these two retting processes (Table 4.1).

It can be seen in Table 4.1 that some weight loss occurred during the attempted water retting. To identify the source of the weight loss, fresh plant samples were weighed after drying in the oven for 24 hours at 105°C and found to be 4.8 gram. These plant samples were then washed in water to remove the dirt, mud and other foreign materials, again oven dried for 24 hours at 105°C, and reweighed. The washed samples weighed 4.5 gram. The loss in weight of the washed samples explained some of the weight loss during water retting. Since no *Typha* fibre was obtained after 90 days of water retting at room temperature and 6 hours' treatment at 80°C, it was decided to investigate chemical and enzymatic retting methods in order to extract fibre from the *Typha* plants. The *Typha* demonstrated good resistance to extraction of starch, and it is hard to break the *Typha* cell walls (Fahlgren, 2017). Water retting cannot extract fibre from the *Typha* plant because the *Typha* plants grow in shallow waters, waterlogged ponds, ditches, and wetlands (Lidman, as cited by Fahlgren, 2017, p.7). Further, it is known that 94% of *Typha* can survive in 2.4% saline water in field conditions (Jesus et al., as cited in Fahlgren, 2017). Additionally, previous research has found that *Typha* is considered a highly water tolerant plant (ACT, 2013) compared to other bast fibres such as corchorus that do not tolerate a waterlogged environment (Roy & Lutfar, as cited by Kozlowski, 2012b, p. 27), and have a very high resistance to mold growth (Krus et al., 2014).

Table 4.1: Water retting of *Typha* plant.

Sample ID	Weight of *Typha* plant (g)		Time	Temp. (°C)	Plant specimens after retting	Fibre Yield (%)
	Control	After retting				
Control	4.0	na	na	21	Figure 4.1	na
S1-W	4.0	3.91	16 days	21	Figure 4.2	Negligible
S2-W	4.0	3.63	6 hours	80	Figure 4.3	Negligible
S3-W	4.0	3.50	90 days	21	Figure 4.4	Negligible

*W: retting in water, water to material ratio: 1:50; na: Not applicable

Figure 4.1: Original *Typha* plant

Figure 4.2: *Typha* plant retted in water for 16 days at 21°C

Figure 4.3: *Typha* plant retted in water for 6 hours at 80°C.

Figure 4.4: *Typha* plant retted in water for 90 days at 21°C.

4.1.2 Chemical retting

Since there was no sign of fibre after using different water retting methods, various chemical retting techniques were conducted. For chemical retting, alkali (aqueous), alcohol (pure), enzymes (pectinase) and acid (acetic acid) were used to remove the non-cellulosic materials in the *Typha* plant. In chemical retting, 3% concentration for each chemical was used and retting was carried out at 21°C temperature for up to 16 days. All chemical retting methods produced negligible amounts of *Typha* fibre: Table 4.2 and Figures 4.5 (S1- CH_3COOH), 4.6 (S1-NaOH), 4.7 (S1-CH_3CH_2OH) and 4.8 (S1-Enzyme). The immediate conclusion was that no fibre is produced from the *Typha* plant using different chemicals at 21°C.

Table 4.2: Fibre yield (%) for different chemical retting at room temperature (21°C) for 16 days.

Sample ID	**Weight of *Typha* plant (g)**		**Fibre yield (%)**	**Plant specimens after retting**
	Control	**After retting**		
S1- CH_3COOH	4.0	3.77	Negligible	Figure 4.5
S1-NaOH	4.0	3.64	Negligible	Figure 4.6
S1-Alcohol	4.0	3.68	Negligible	Figure 4.7
S1-Enzyme	4.0	3.71	Negligible	Figure 4.8

* M: L ratio: 1:50

Figure 4.5: Appearance of *Typha* plant treated with acid (Sample: S1-CH3COOH) for 16 days at 21°C.

Figure 4.6: Appearance of *Typha* plant treated with alkali (Sample: S1-NaOH) for 16 days at 21°C.

Figure 4.7: Appearance of *Typha* plant treated with alcohol Sample: S1-CH_3CH_2OH) for 16 days at 21°C

Figure 4.8: Appearance of *Typha* plant treated with enzyme (Sample: S1-Enzyme) for 16 days at 21°C

As room temperature retting using different chemicals did not produce any fibre, retting was conducted at higher temperatures using alkali, enzymes, and acid. For alkali (NaOH) and acid (CH_3COOH), retting was conducted at both 60°C and 80°C, while for enzyme (Pectinase from Aspergillus Aculeatus), the retting was conducted only at 40°C as this temperature is the optimum for enzymatic bast fibre retting and modifications (Khan, 2016). The retting results from enzyme, alkali and acid treatments at higher temperatures are shown in Table 4.3. It can be seen from this table that alkali retting S2-NaOH at 60°C with 1% concentration for 1 hour retting no fibre was observed (Figure not shown here), only at 60°C for 2 hours (Sample: S3-NaOH, Figure 4.9) and at 80°C for 4 hours (Sample: S5-NaOH, Figure 4.11) produced fibre; whereas, none of enzyme treatments at 40°C (Sample: S7-Enzyme, Figure 4.13), and acid treatments at 60°C for 2 hours (Sample: S4-CH_3COOH, Figure 4.10), and at 80°C for 4 hours (Sample: S6-CH_3COOH, Figure 4.12) produced any fibre.

Although the retting at 80°C for 4 hours in acid (Sample: S6-CH_3COOH, Figure 4.12) appeared to break the *Typha* plant into 'fibre-like' pieces, after close examination it was noticed

that most of these pieces were ‘plant flakes’ (flake width ≈ 68 μm – 2 mm) and attached to each other. In order to find out whether a longer treatment time in acid might yield fibre, the *Typha* plant was treated at 80°C for 8 hours; however, no fibre could be seen (Figure 4.14). Further, no fibre was found when the *Typha* plant treated in acetic acid for 20 days either at room temperature or in sulphuric acid for 60 days at room temperature (Figure not shown here).

The fibre was found from NaOH retting depended on the retting temperature. It is worth mentioning here that, unlike other bast fibre, where, fibre is extracted from the outer layer of the plant, for the *Typha* plant, the bulk of the plant could be transformed into fibre, as discussed in Section 4.3.1. A few ‘flakes’ (bark) could be seen in some of the fibre samples, (Figures 4.9, 4.12 and 4.13). The sources of this barks are discussed in Section 4.3.1. Further, the fibre yield (%) for *Typha* plant was much higher for alkaline treated samples (Table 4.3) than for brassica, linum and cannabis where the fibre yield range is between 10 - 15% (Khan, 2016).

It is known that natural fibre can be treated with up to 28% alkali to improve the fibre adhesion for composite applications without a significant reduction in strength (Naveen et al., 2015). Since fibre can be obtained using NaOH, and no damage of the fibre was reported when using up to 28% NaOH (Naveen et al., 2015), *Typha* plants were immersed in 10% NaOH solution at room temperature for 30 days. However, no fibre could be obtained for this experimental condition. Therefore, it is concluded that room temperature is not suitable for fibre extraction from *Typha* using alkali and no further experiments were carried out at room temperature.

Table 4.3: Effect of different chemicals treatment in fibre extraction

Sample ID	Time (hour)	Conc. (%)	Temp. (°C)	Weight of the *Typha* plant (g)		Fibre obtained from chemical retting	Plant specimens after retting (Shown in Figure)
				Before treatment	After treatment		
S2-NaOH	1	1.0	60	4.0	3.73	No	---
S3-NaOH	2	1.0	60	4.0	3.14	Yes	4.9
S4- CH_3COOH	2	1.0	60	4.0	3.62	No	4.10
S5 -NaOH	4	3.0	80	4.0	2.24	Yes	4.11
S6- CH_3COOH	4	3.0	80	4.0	3.47	No	4.12
S7-Enzyme	4	3.0	40	4.0	3.68	No	4.13
S7- CH_3COOH	8	3.0	80	2.8	2.77	No	4.14

* S: sample; Enzyme: Pectinase from Aspergillus Aculeatus; M: L-1:50.

Figure 4.9: Appearance of *Typha* plant treated with alkali (Sample: S3-NaOH) for 2h/1%/60°C

Figure 4.10: Appearance of *Typha* plant treated with alkali (Sample: S4-CH_3COOH) for 2h/1%/60°C

Figure 4.11: Appearance of *Typha* plant treated with alkali (Sample: S5-NaOH) for 4h/3%/80°C

Figure 4.12: Appearance of *Typha* plant treated with acid (Sample: S6- CH_3COOH) for 4h/3%/80°C

Figure 4.13: Appearance of *Typha* plant treated with acid (Sample: S7- CH_3COOH) for 8h/3%/80°C

Figure 4.14: Appearance of *Typha* plant treated with Enzyme (Sample: S7-Enzyme) for 4h/3%/40°C

4.2 Effect of various alkalis on fibre yield

4.2.1 Fibre yield (%) from mixed plants

Since *Typha* fibre was obtained using aqueous NaOH solution, two other alkalis (KOH and LiOH) were used for fibre extraction from mixed or unknown *Typha* plants. The term *'mixed stem'* plants will be discussed later in the section. The fibre yield (%) with experimental conditions for these two alkalis is shown in Table 4.4. As with the NaOH treatment, fibre was obtained under all experimental conditions using both KOH and LiOH [Figures 4.15 (a) - 4.27 (a)].

Table 4.4 shows the effect of time on *Typha* fibre yield (%), where it can be seen that the fibre yield (%) decreases with the increase in treatment time. As seen in Table 4.4, at 80°C in KOH and LiOH the fibre yields were 50.0% (Sample: S8-K1-MS, Figure 4.15), 55% (Sample: S8-K2-MS, Figure 4.16), 35% (Sample: S8-K3-MS, Figure 4.17), 15% (Sample: S8-K4-MS, Figure 4.18) and 66% (S10-L1-MS,Figure 4.23 (a)), 54% (S10-L2-MS, Figure 4.24 (a)), 56% (S10-L3-MS, Figure 4.25 (a)) and 53% (S10-L4-MS, Figure 4.26 (a)); after 2, 4, 6 and 8 hours respectively.

However, a similar trend of decreasing yield with treatment time was not obtained for samples treated at 95°C in KOH solution fibre yield was 20% (Sample: S9-K5-MS, Figure 4.19),

25% (Sample: S9-K6-MS, Figure 4.20), 20% (Sample S9-K7-MS, Figure 4.21) and 34% (Sample: S9-K7-MS, Figure 4.22).

It was found using similar treatment conditions in KOH at 95°C, the fibre yield was much lower than KOH at 80°C for all treatment hours, except the 8 hours treated sample (Table 4.4). It is worth noting here that for the equivalent treatment conditions, the yield (%) using LiOH is much higher than the KOH, particularly after 6 and 8-hours' treatment. For example, after 6 hours' treatment at 80°C, KOH treatment produced 35% fibre [S8-K3-MS, Figure 4.17(a)], whereas the LiOH produced 56% fibre [S10-L3-MS, Figure 4.25(a)], and after 8 hours of treatment, KOH treatment produced 15% fibre [S8-K4-MS, Figure 4.18 (a)] compared to 53% fibre [S10-L4-MS, Figure 4.26(a)] produced in LiOH.

Table 4.4: Fibre yield (%) from ***mixed stem*** plants at 3% concentration in KOH (80°C & 95°C) and LiOH (80°C) solution for different temperature.

Alkali	**Sample ID**	**Temperature (°C)**	**Time (hours)**	**Yield (%)**	**Resultant fibre (Shown in Figure)**
KOH	S8-K1-MS	80	2	50.00	4.15 (a)
KOH	S8-K2-MS	80	4	55.00	4.16 (a)
KOH	S8-K3-MS	80	6	35.00	4.17 (a)
KOH	S8-K4-MS	80	8	15.00	4.18 (a)
KOH	S9-K5-MS	95	2	20.00	4.19 (a)
KOH	S9-K6-MS	95	4	25.00	4.20 (a)
KOH	S9-K7-MS	95	6	20.00	4.21 (a)
KOH	S9-K8-MS	95	8	34.00	4.22 (a)
LiOH	S10-L1-MS	80	2	66.00	4.23 (a)
LiOH	S10-L2-MS	80	4	54.00	4.24 (a)
LiOH	S10-L3-MS	80	6	56.00	4.25 (a)
LiOH	S10-L4-MS	80	8	53.00	4.26 (a)

Figure 4.15 (a): Appearance of *Typha* plant treated with alkali (Sample: S8-K1-MS) for 2h at 80°C

Figure 4.16 (a): Appearance of *Typha* plant treated with alkali (Sample: S8-K2-MS) for 4h at 80°C

Figure 4.17 (a): Appearance of *Typha* plant treated with alkali (Sample: S8-K3-MS) for 6h at 80°C

Figure 4.18 (a): Appearance of *Typha* plant treated with alkali (Sample: S8-K4-MS) for 8h at 95°C

Figure 4.19 (a): Appearance of *Typha* plant treated with alkali (Sample: S9-K5-MS) for 2h at 95°C

Figure 4.20 (a): Appearance of *Typha* plant treated with alkali (Sample: S9-K6-MS) for 4h at 95°C

Figure 4.21 (a): Appearance of *Typha* plant treated with alkali (Sample: S9-K7-MS) for 6h at 95°C

Figure 4.22 (a): Appearance of *Typha* plant treated with LiOH (Sample: S9-K8-MS) for 8h at 80°C

Figure 4.23 (a): Appearance of *Typha* plant treated with LiOH (Sample: S10-L1-MS) for 2h at 80°C

Figure 4.24 (a): Appearance of *Typha* plant treated with LiOH (Sample: S10-L2-MS) for 4h at 80°C

Figure 4.25 (a): Appearance of *Typha* plant treated with LiOH (Sample: S10-L3-MS) for 6h at 80°C

Figure 4.26 (a): Appearance of *Typha* plant treated with LiOH (Sample: S10-L4-MS) for 8h at 80°C

It seems that there is no clear trend in fibre yield (%) with treatment time and temperature when treated in KOH and LiOH. The investigation of the yield variations shown in Table 4.4, revealed that there were three types of plant used for the study and these plant samples are termed ‘mixed samples: (a) plants with a hard stem, (b) plants with a soft stem, and (c) plants with no stem (leaves only plant). While the detailed study of the plants is beyond the scope of the current study, a brief description of these three plant components is given in Section 4.2.2 and the fibre yield (%) from these plants is given in Section 4.3.1 Also, it is worth mentioning here that some of the fibre samples contain ‘flake/bark’ like materials as shown in [Figures 4.15 (a), 4.18 (a), 4.19 (a) and 4.24 (a)].

Figure 4.15 (a): Flake/bark material exists in (Sample: S8-K1-MS).

Figure 4.18 (a): Flake/bark material exists in (Sample: S8-K4-MS).

Figure 4.19 (a): Flake/bark material exists in (Sample: S9-K5-MS).

Figure 4.24 (a): Flake/bark material exists in (Sample: S10-L2-MS).

4.2.2 Fibre yield (%) on hard stem (HS), soft stem (SS) and no stem (NS) plants

Tables 4.5, 4.6 and 4.7 exhibited the fibre yield (%) from hard stem (HS), soft stem (SS) and no stem (only leaves plant) (NS) plants in KOH (80°C and 95°C) and LiOH at 80°C for different treatment times. Similar to mixed plants, fibre was obtained from all three plants as shown in Figures 4.15 (b) - 4.26 (b) for hard stem, Figures 4.15 (c) - 4.26 (c) for soft stem and Figures 4.15 (d) - 4.26 (d) for no stem plants which are given in Appendix I. Again, no clear trend can be seen between fibre yield (%), treatment time and treatment temperature, although at 95°C in KOH and at 80 °C in LiOH, there was tendency for decreasing yield (%) with the treatment time for the three plants, while at 80°C in KOH, no such trend was observed. In general, it can be stated that the yield (%) varies between 50-30, 55-30 and 47-25 for the hard, soft and no stem respectively. An important observation was made in the fibre from hard and soft plants: these fibres contain flakes [Figures 4.17 (b), 4.19 (b), 4.20 (b) 4.23 (b) 4.25 (b) and 4.26 (b) are located in Appendix I], no such flakes could be seen in the fibres that were obtained from no stem plants. Therefore, some components in hard and soft plants may have been responsible in contributing flakes in the fibres, as discussed in Section 4.3.

Table 4.5: Effect of time on the ***hard stems*** plant fibres yield (%) at 3% concentration in KOH (80°C & 95°C) and LiOH (80°C) solution for different temperature.

Alkali	Sample ID	Temperature	Time	Yield%	Resultant fibre (Shown in Figure)
KOH	S8-K1-HS	80	2	50.90	4.15 (b)
KOH	S8-K2-HS	80	4	38.52	4.16 (b)
KOH	S8-K3-HS	80	6	36.24	4.17 (b)
KOH	S8-K4-HS	80	8	41.27	4.18 (b)
KOH	S9-K5-HS	95	2	44.00	4.19 (b)
KOH	S9-K6-HS	95	4	41.00	4.20 (b)
KOH	S9-K7-HS	95	6	35.00	4.21 (b)
KOH	S9-K8-HS	95	8	32.00	4.22 (b)
LiOH	S10-L1-HS	80	2	35.00	4.23 (b)
LiOH	S10-L2-HS	80	4	28.00	4.24 (b)
LiOH	S10-L3-HS	80	6	29.00	4.25 (b)
LiOH	S10-L4-HS	80	8	30.00	4.26 (b)

Figures 4.15 (b) – 4.26 (b) located in Appendix I

Table 4.6: Effect of time on the ***soft stems*** plants fibres yield (%) at 3% concentration in KOH (80°C & 95°C) and LiOH (80°C) solution for different temperature.

Alkali	Sample ID	Temperature (°C)	Time (hours)	Yield%	Resultant fibre (Shown in Figure)
KOH	S8-K1-SS	80	2	31.50	4.15 (c)
KOH	S8-K2-SS	80	4	33.60	4.16 (c)
KOH	S8-K3-SS	80	6	42.00	4.17 (c)
KOH	S8-K4-SS	80	8	32.14	4.18 (c)
KOH	S9-K5-SS	95	2	55.49	4.19 (c)
KOH	S9-K6-SS	95	4	42.37	4.20 (c)
KOH	S9-K7-SS	95	6	33.76	4.21 (c)
KOH	S9-K8-SS	95	8	39.57	4.22 (c)
LiOH	S10-L1-SS	80	2	39.53	4.23 (c)
LiOH	S10-L2-SS	80	4	36.63	4.24 (c)
LiOH	S10-L3-SS	80	6	37.10	4.25 (c)
LiOH	S10-L4-SS	80	8	39.94	4.26 (c)

Figures 4.15 (c) – 4.26 (c) located in Appendix I

Table 4.7: Effect of time on the ***no stems*** (only leaves plant) plants fibres yield (%) at 3% concentration in KOH (80°C & 95°C) and LiOH (80°C) solution for different temperature.

Alkali	Sample ID	Temperature (°C)	Time (hours)	Yield%	Resultant fibre (Shown in Figure)
KOH	S8-K1-NS	80	2	45.43	4.15 (d)
KOH	S8-K2-NS	80	4	35.13	4.16 (d)
KOH	S8-K3-NS	80	6	41.34	4.17 (d)
KOH	S8-K4-NS	80	8	32.04	4.18 (d)
KOH	S9-K5-NS	95	2	35.03	4.19 (d)
KOH	S9-K6-NS	95	4	35.27	4.20 (d)
KOH	S9-K7-NS	95	6	32.23	4.21 (d)
KOH	S9-K8-NS	95	8	31.31	4.22 (d)
LiOH	S10-L1-NS	80	2	46.01	4.23 (d)
LiOH	S10-L2-NS	80	4	33.24	4.24 (d)
LiOH	S10-L3-NS	80	6	31.52	4.25 (d)
LiOH	S10-L4-NS	80	8	27.69	4.26 (d)

Figures 4.15 (d) – 4.26 (d) located in Appendix I.

4.2.3 Data analysis

The statistical analysis was conducted to determine the variation in yield (%) among the treatment hours (2, 4, 6 and 8), treatment temperature (80°C in KOH, 80°C in LiOH and 95°C in

KOH) and two alkalis are significantly different or not. Two way ANOVA was conducted using the treatment conditions, which are time, temperature and alkali as shown in Table 4.8.

Table 4.8: Between-Subject Factors

Time (hour)	N	Temperature (°C)	N	Alkali	N
2	12	80	32	KOH	32
4	12	95	16	LiOH	16
6	12				
8	12				

The output variable was found to be normally distributed and equal variances are assumed based upon results of Levene's test ($F(11, 36) = .848$, $p = .596$). The ANOVA results are given in Table 4.9. The interaction effect between (time and temperature) and (time and alkali) were not statistically significant as ($F(3, 36) = 0.564$, $p = 0.642$) and ($F(3, 36) = 0.361$, $p = 0.782$) respectively. Further, no statistically significant effect was found for treatment time [($F(3, 36) = 1.5$, $p = .226$)], treatment temperature [($F(3, 36) = 1.04$, $p = .314$)] and alkali [($F(3, 36) = .227$, $p = .637$)]. It was found that the effect size was the highest for treatment time ($\eta^2 = 0.11$) followed by temperature ($\eta^2 = 0.028$) and alkali ($\eta^2 = 0.006$).

Table 4.9: Two-way ANOVA Results tests on the effect of temperature, time and alkali type on fibre yield (%).

Treatment factor	df	F	Sig.	Partial Eta Squared ($\eta^{2)}$
Time	3	1.52	0.23	0.110
Temperature	1	1.04	0.31	0.028
Alkali	1	0.23	0.64	0.006
Time * Temperature	3	0.56	0.64	0.045
Time * Alkali	3	0.36	0.78	0.029
Temperature * Alkali	0	0.00	0.00	0.000

Table 4.10: Results of Post Hoc Tests for the effect of time on yield (%)

Time (I)	Time (J)	LSD
2	4	0.230> *p*
	6	0.082> *p*
	8	[S]0.033< *p*
4	2	0.230> *p*
	6	0.572> *p*
	8	0.327> *p*
6	2	0.082> *p*
	4	0.572> *p*
	8	0.674> *p*
8	2	[S]0.033> *p*
	4	0.327> *p*
	6	0.674> *p*

*s=significant, p =0.05.

The LSD post hoc test was used for multiple comparison (Table 4.12) which showed that the yield (%) is significantly different (p=0.033<0.05) between two hours (M =43.24, SD= 4.17) and eight hours ((M=35.78, SD= 4.17).

4.3 Sources of flakes and barks in the fibre

As mentioned earlier, fibre-like pieces (plants flakes and bark) broke off the *Typha* stems during the retting process. The flakes are fibre like materials found in fibre bundles from which fibre can be separated by hand after immersing the stems in water; whereas, bark is a part of the hard/soft woody stems. Fibre cannot be separated by hand from these stems after immersing in water. Since the fibre yield (%) varied widely, within a fibre bundle, all *Typha* plants used in the current study were analyzed for their plant morphology. It was found that three different types of *Typha* plants were used:

(1) Plant with a hard wood stem – Figure 4.27(a) & 4.27(b): Cross-section with different components.

(2) Plant with a soft stem - Figure 4.28(a) & 4.28(b): Cross-section with different

components.

(3) Plant with no stem (only leaves) - Figure 4.29 and Figure 4.30: Cross-section with different components.

There are three major sections that were separated from the plants that have stems (both soft and hard stems): the outer layer comprised of several sheets of leaves [Figures 4.27 (a) & (b)] cross section of whole hard stem plant], hard wood bark, and soft spongy tissue inside the hard wood. Similar components were separated for plants with a soft stem [Figures 4.28 (a) & (b) – whole soft stem plant], leaves, soft wood with soft spongy core and woody bark. Only two components were separated from the plant with no stem: leaves and spongy tissue (Figure 4.29). The spongy tissue in this leaf was found in the core of the leaf. No spongy tissue could be seen at the two edges of the leaf and the thickness of the spongy tissue core, in a quadratic curve shape (Figure 4.30), increased in successive layers of leaf towards the center of the plant.

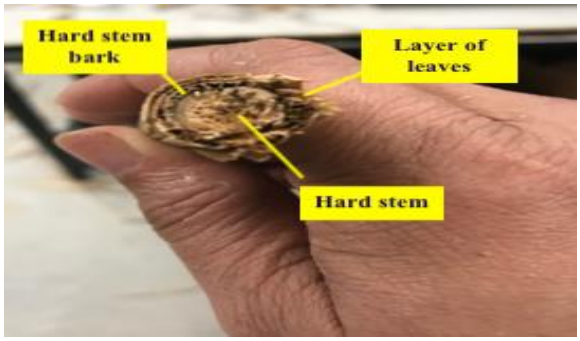


Figure 4.27 (a): Bottom sponge diameter of *Typha* Soft stems (HS) plant

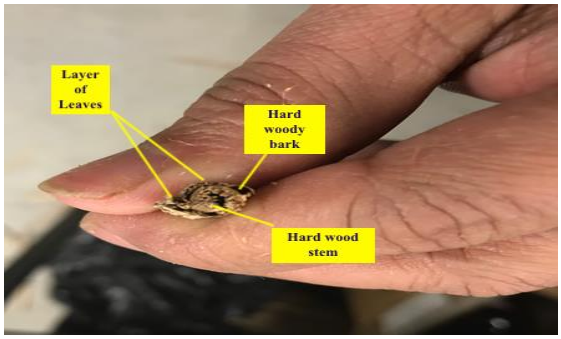


Figure 4.27 (b): Top sponge diameter of *Typha* soft stems (HS) plant

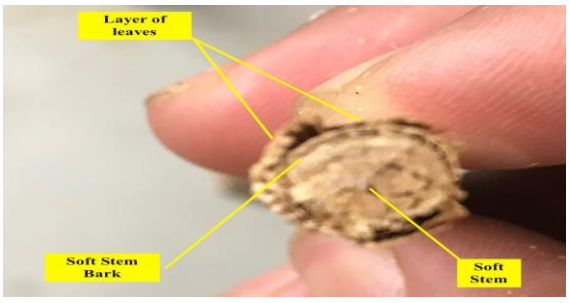


Figure 4.28 (b): Top sponge diameter of *Typha* hard stems (SS) plant

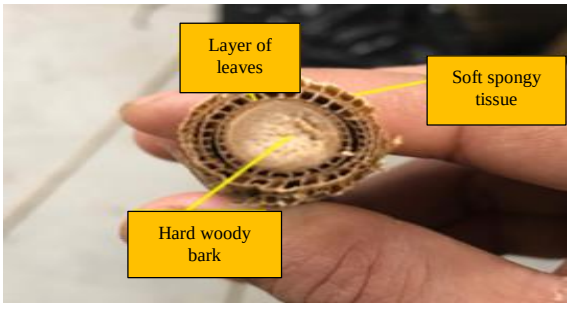


Figure 4.28 (a): Bottom sponge diameter of *Typha* Soft stems (SS) plant

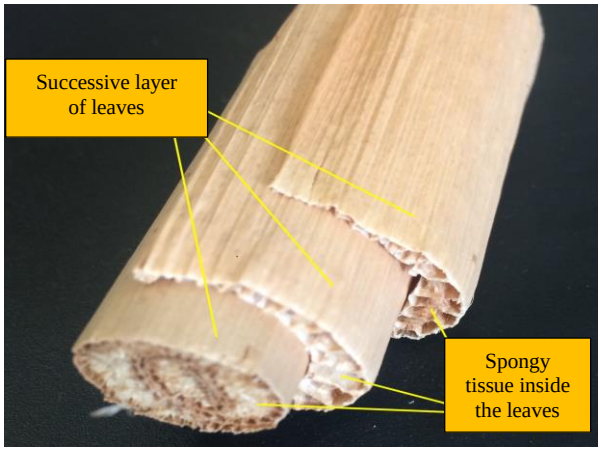


Figure 4.29: Different components of *Typha* plant leaves.

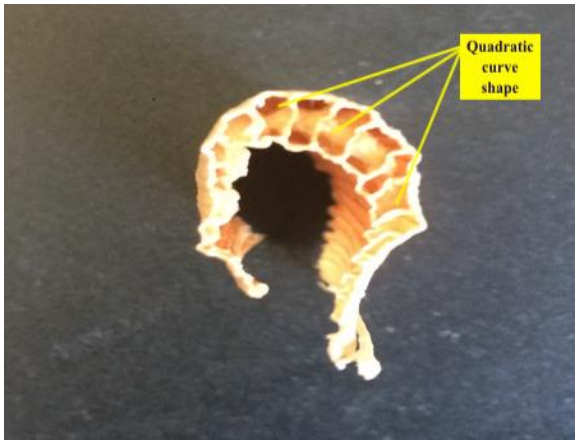


Figure 4.30: Quadratic curve shape of *Typha* plant

Table 4.8 shows *Typha* plant morphology, including components and dimensions – whole plant diameter, leaf, stem, and spongy tissue. The diameter of the no stem plant (30.4 mm) is much higher than the hard (10.59 mm) and soft stem (16.76 mm) plants. This diameter was measured 25 mm above the bottom of the plant. Similarly, larger leaf width, which was measured 2.5 cm from bottom, was obtained for no stem plant (leaf width: 5.1 cm) than the hard (leaf width: 3.0 cm) and soft stem (leaf width: 4.6 cm). After removing the leaves, they could be measured for the no stem plant; the diameter of the hard stem (bark + sponge) and soft stem (bark + sponge) plants was found to be 5.42 ± 0.36 mm and 9.5 ± 0.97 mm respectively. As mentioned earlier, both hard and soft stem plants as well as no stem plant leaf contained spongy tissue and diameter of the spongy tissue was 4.41 ± 0.51 mm for hard stem and 7.69 ± 1.20 mm for the soft stem plant. The diameter of the spongy core of the no stem leaf varies widely due to the quadratic curve shape, however, the diameter of the widest section of the spongy core was found to be 2.2 mm.

Table 4.11: Plant morphology for hard, soft and no-stem of *Typha* plants with dimensions

Plant type	Whole plant diameter (mm)	Leaf	Stem diameter (Stem + bark)	Spongy
Hard stem	10.59 (25 mm above the root)	Yes (width: 3.0 cm, 2.54 cm from bottom)	Yes (average diameter: 5.42 ± 0.36 mm) [5.54,5.43,5.87,4.88,5.31]	Yes (inside stem) Inner core diameter: 4.41± 0.51 mm [4.90,4.29,3.58,4,70,4.58]
Soft stem	16.76 (25 mm above the root)	Yes (width: 4.6 cm, 2.54 cm from bottom)	Yes (average diameter: 9.5 ± 0.97 mm) [9.03,11.23,9.26,9.06,8.95]	Yes (inside stem) Average inner core diameter: 7.69 mm ± 1.20mm [8.44,9.14,7.81,7.03,6.06]
No stem	30.40 (25 mm above the root)	Yes (width: 5.1 cm, 2.54 cm from bottom)	No	Yes, (inside leaf) Inner core diameter (variable): 2.2 mm

4.2.1 Fibre extraction from various plant components of *Typha* plant

In order to investigate whether or not fibre can be obtained from various components of the hard stem (HS), soft stem (SS) and no stem (NS) *Typha* plants, and track the sources of the bark/flakes in the fibre bundle, alkali treatment was carried out using 3.0% KOH at 80°C. The treatment was conducted for 8 hours, except for one component (bark – hard and soft stems) of the plant, where treatment was conducted for up to 12 hours.

The fibre, fibre yield (%), flakes and barks in the fibre, and visual fibre characteristics from the different component of the plants are given in Table 4.9. It can be seen from this table (also shown in figures) that fibre can be obtained from all of the three whole plants: hard stems, soft stems and no stems: [Figure 4.36 (a) – whole plant (SS), Figure 4.36 (b) – fibre (left) and flakes (right)], [Figure 4.31 (a) – whole plant (HS), Figure 4.31 (b) – fibre (left) and flakes (right)], and [Figure 4.41 (a) – whole plant (NS), Figure 4.41 (b) – fibre, no flakes]. Further, it can be seen that only fibres from the NS whole plant are free from flakes [Figure 4.41 (b)], while flakes/barks have

been separated from the fibres obtained from whole soft stem (SS) plant [Figure 4.36 (b), right] and whole hard stem (HS) plant [Figure 4.31 (b), right].

Fibres also can be obtained from the leaves of all three plants as in Figure 4.41 (b) [NS], 4.37 (b) [SS] and 4.32 (b) [HS]. The leaves for these plants are given in Figures 4.41 (a), 4.37 (a), and 4.31 (a), respectively for NS, SS and HS plants. No flakes could be found in the fibres obtained from the leaves. These flakes/barks might have come from bark or/and sponge in Figures 4.36 (b) and 4.31(b).

Fibres from the hard wood + spongy tissue [HS, Figure 4.33 (a)] and soft wood + spongy tissue [SS, Figure 4.38(a)] are given in Figures 4.33 (b) and 4.38 (b) respectively. It can be seen from these two figures [Figures 4.38 (b) and 4.33 (b)] that fibres are mixed with flakes and barks. The flakes/barks have been separated from the fibres and shown in Figures 4.38 (c) fibres (right) and flakes (left) and 4.33 (c) fibres (left) and flakes (right) respectively for SS and HS plants. Woody bark [HS, Figure 4.34 (a)], [SS, Figure 4.39 (a)] and spongy tissue [HS, Figure 4.35 (a)], [SS, Figure 4.40 (a)] were separately treated with alkali. No fibres were extracted from the woody bark as shown in [Figures 4.34 (b) for 6 hours, 4.34 (c) for 8 hours, 4.34 (d) for 12 hours] and [Figures 4.39 (b) for 8 hours, 4.39 (c) for 12 hours]. When spongy tissue separated from hard [4.35 (a)] and soft stem plants [4.40 (a)], and was treated in alkali, fibres could be obtained. These fibres are shown in Figure 4.35(b) and 4.40 (b).

The immediate conclusion is that fibres could be obtained from leaves and spongy tissue and no fibres could be obtained from bark. The sources of the flakes/bark in the fibres have now been established.

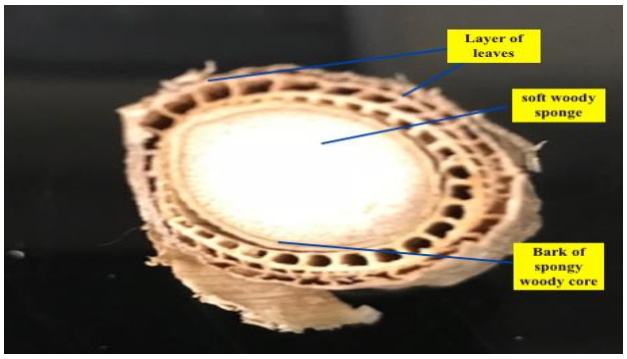


Figure 4.36 (a): Cross-section whole (soft stem plant)

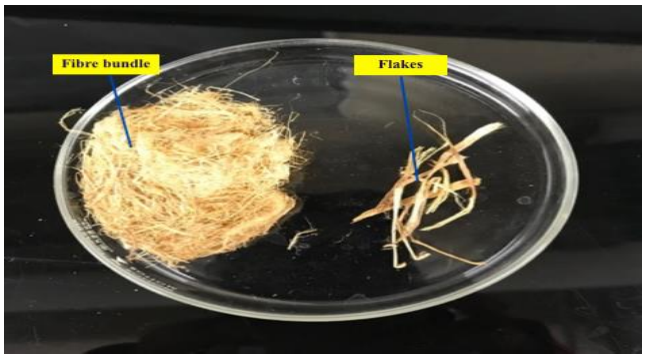


Figure 4.36 (b): Fibre (left) and bark/Flakes, flakes (right) from *Typha* whole (soft stem plant)

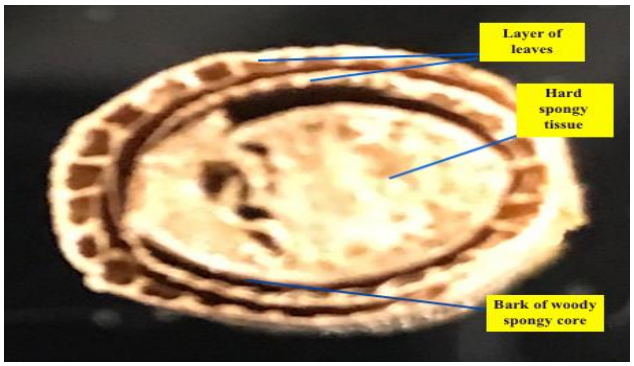


Figure 4.31 (a): Cross-section of whole (hard stem plant).

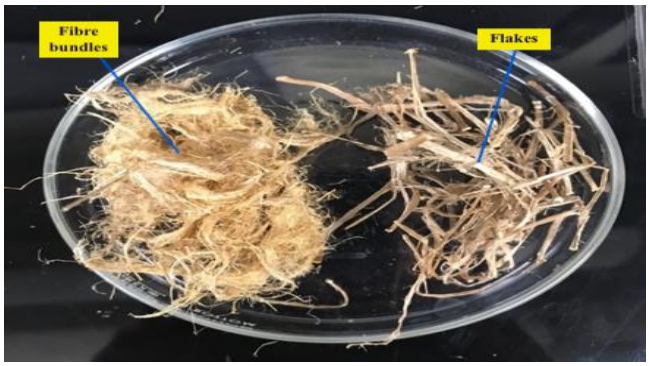


Figure 4.31 (b): Fibre (left) and flakes/ bark (right) from *Typha* whole (hard stem plant)

Figure 4.33 (a): *Typha* plant - hard wood bark with spongy tissue (hard stem plant).

Figure 4.33 (b): Fibre from *Typha* hard wood and with spongy tissue (hard stem plant).

Figure 4.37 (a): *Typha* leaves (soft stem plant)

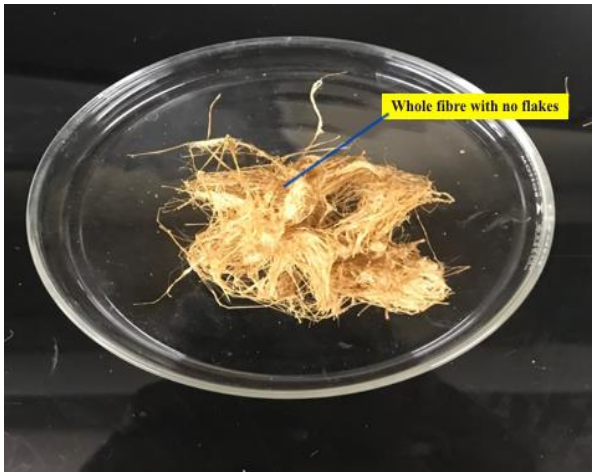


Figure 4.37 (b): Fibre from *Typha* leaves (soft stem plant)

Figure 4.32 (a): *Typha* leaves (hard stem plant)

Figure 4.32 (b): Fibre from *Typha* leaves (hard stem plant)

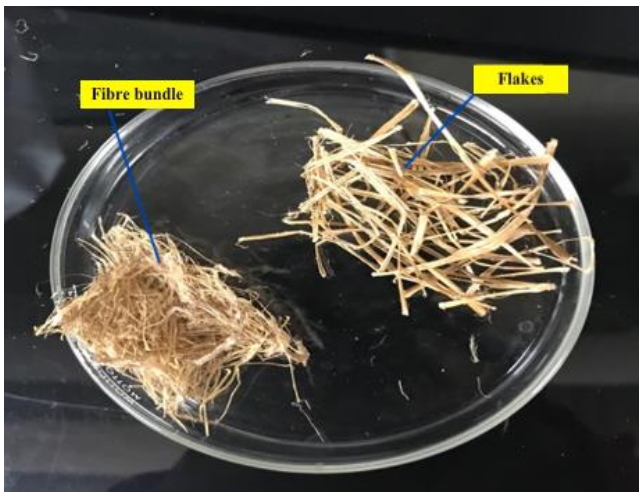


Figure 4.33 (c): Hard stem + sponge [separated from sample 4.33 (b)]

Figure 4.38 (a): *Typha* plant - soft wood with spongy tissue (soft stem plant).

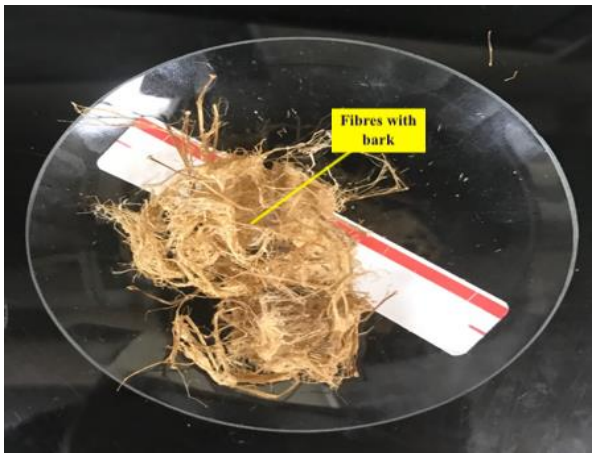


Figure 4.38 (b): Fibre from *Typha* soft wood and with spongy tissue (soft stem plant).

Figure 4.38 (c): Soft stem + sponge
[separated from sample 4.38 (b)]

Figure 4.34 (a): *Typha* plant hard wood bark (Hard stem)

Figure 4.34 (b): *Typha* fibre from hard woody bark treated with 3% KOH for 6h.

Figure 4.34 (c): *Typha* fibre from hard woody bark treated with 3% KOH for 8h

Figure 4.34 (d): *Typha* fibre from hard woody bark treated with 3% KOH for 12h.

Figure 4.35 (a): *Typha* plant soft spongy tissue (hard stem plant).

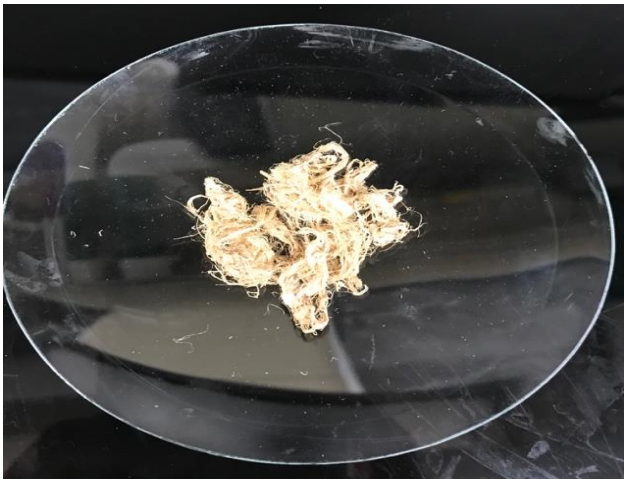


Figure 4.35 (b): Fibre from *Typha* soft spongy tissue (hard stem plant).

Figure 4.39 (a): *Typha* plant soft wood bark

Figure 4.39 (b): *Typha* fibre from soft wood bark, 80°C/3% KOH/8 h

Figure 4.39 (c): *Typha* fibre from soft wood bark, 80°C/3% KOH/12 h

Figure 4.40 (a): *Typha* plant soft spongy tissue (soft stem plant).

Figure 4.40 (b): Fibre from *Typha* soft spongy tissue (soft stem plant), 80°C/3% KOH/8 h

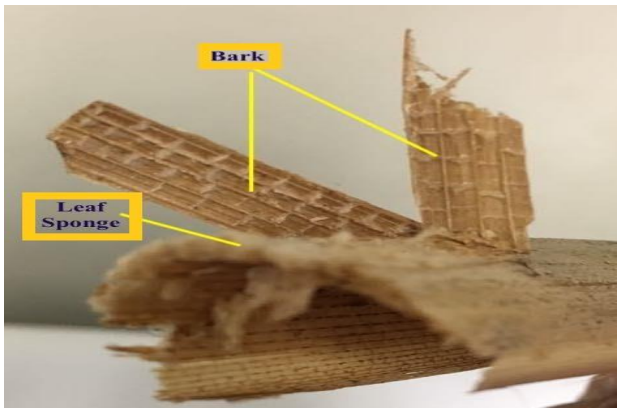


Figure 4.41(a): *Typha* plant leaves (no stem plant).

Figure 4.41 (b): Fibre from *Typha* leaves with soft spongy tissue (No stem plant), 80°C/3% KOH/8 h

The characteristics (fibre quality) of these fibres and flakes are summarized in Table 4.9.

For this experiment all treatments no replica was used. It can be seen from this table that fibre is

obtained from the whole plant, leaves, and spongy tissue, while no fibre can be obtained from the hard-woody part. The materials that are obtained from this woody segment of the *Typha* plants are 100% flake (width: 2mm) for soft stem *Typha* plant (SS) and hard stem *Typha* plant (HS) as shown in [Figure 4.38 (b), (c) and 4.33(b), (c) and (d)], while spongy tissues for (SS) in [Figure 4.40 (b)] and (HS) [Figure 4.34 (b)] and leaves for both (SS) and (HS) provide 100% fibres as seen in Figure 4.36 (b) and Figure 4.31 (b), however, a few flakes could be found in the fibres obtained from the whole *Typha* plant for (SS) [Figure 4.36 (b)] and (HS) [Figure 4.31 (b)].

Further, it appears that the quality of fibres obtained from the spongy tissues is inferior to those obtained from the *Typha* leaves, as the leaf fibres are softer and more easily separable than the fibres from the spongy segment. Fibres from the leaves of the *Typha* plants are similar to those from the whole plant as seen in Figures (SS) 4.36 (b) and (HS) 4.31 (b). The woody bark with spongy tissue is soft but difficult to separate into individual fibres for both (SS) and (HS) plants [Figures 4.38 (b) and 4.33 (b)]. The same characteristics are shown in the spongy part of the *Typha* plant as seen in Figures (SS) 4.40 (b) and (HS) 4.35(b). Fibre yield (%) for the no stem plant was the highest – 41.33%. The yield (%) of fibre and flake in the soft stem plant was found to be 31.90 (%) and 4.76 (%) respectively, while in the hard stem plant this amount was 26.57% and 11.59%, respectively. Fibre from leaves gave a comparatively higher yield (%) in (SS), 38.00; whereas, the (HS) was 29.80; there was no flake (%) for leaves. In (hard wood + spongy tissue), mixed with fibres and flakes fibre yield (%) was 33.33 and flake (%) was 12.57 for (SS); and for (HS) fibre yield (%) was 30.66 and flake (%) 21.33. Fibre extracted from spongy tissue segment was fibre 32.66% for (SS) and 21.33 for (HS). There were only flakes (%), but no fibre in the woody bark section, [1.18 (for 8 hours, SS), 1.64 (for 12 hours, SS); 0.74 (for 6 hours, HS), 1.24 (for 8 hours, HS), and 1.42 (for 12 hours)].

Table 4.12: Fibre yield (%) and visual characteristics of fibres from different plant segment, 3.0% KOH, 80°C after 8 hours.

***Typha* plant segment**	**Plant type**	**Flake obtained**	**Fibre obtained**	**Yield (%)**			**Figure #**		**[d]Fibre quality (visual)**
				Plant weight (g)	**Yield (%) of fibre**	**Yield (%) of flakes**	**Original component**	**After treatment**	
Whole plant	SS	Yes	Yes	2.10	31.90	4.76	4.36(a)	4.36(b)	S, SF
	HS	Yes	Yes	2.07	26.57	11.59	4.31(a)	4.31(b)	S, SF
	NS	No	Yes	1.89	41.33	0.00	4.41(a)	4.41(b)	S, SF
	SS	No	Yes	1.87	38.00	0.00	4.37(a)	4.37(b)	S, SF
Leaves	HS	No	Yes	1.51	29.80	0.00	4.32(a)	4.32(b)	S, SF
Woody bark + spongy tissue	SS	Yes Figure 3.34 (c)	Yes	1.10	33.33	12.57	4.38(a)	4.38(b)	S, difficult to separate
	HS	Yes, 3.29 (c)	Yes	1.59	30.66	21.33	4.33(a)	4.33(b)	S, difficult to separate
Woody bark	SS	Yes	No	1.82	0.00	57.56	4.39(a)	4.39(b)	NA
	[a]SS	Yes	No	2.08	0.00	78.46	4.39(a)	4.39(c)	NA
	[b]HS	Yes	No	1.57	NA	36.63	4.34(a)	4.34(b)	NA
	HS	Yes	No	2.01	NA	60.78	4.34(a)	4.34(c)	NA
	[c]HS	Yes	No	2.03	NA	71.00	4.34(a)	4.34 (d)	NA
Spongy tissue	SS	No	Yes	1.59	32.66	0.00	4.40(a)	4.40(b)	S, difficult to separate
	HS	No	Yes	1.57	21.33	0.00	4.35(a)	4.35(b)	S, difficult to separate

NA: Not applicable; SS: Soft stem; HS: hard stem; NS: no stem; [a]: 12 hours; [b]: 6 hours; [c]: 12 hours; [d]: based on softness (S) and single (SF) fibre.

4.2.1.1 Summary

The most widely used water retting was investigated for fibre extraction from the *Typha* plant. Unsuccessful results from water retting showed that this retting method is unsuitable for the *Typha* plant, so other extraction methods were employed. Similarly, acid and enzyme retting did not work for fibre extraction of *Typha* fibre. Alkali (KOH, LiOH) treatment of less than 2 hours and <3.0% concentration was not suitable for proper fibre extraction; however, treatment between 2 hours and 8 hours with 3% alkali could produce fibres. The plant physiological investigation found that fibre yield (%) was influenced by different factors: composition of plants such as flake (%); presence of an inner woody core section; and plant type: No stems plants provided a higher yield 41.33 % without 0.0% flakes than hard 26.57 % with flake 11.59% and soft stem 31.90% with 4.76 % plants. The optimum parameter for obtaining fibres from all three *Typha* plants was found to be 2 hours / KOH or LiOH at 80°C.

An ideal textile fibre obtain a series of important properties that influence product performance and helps to choose several fibres for any sort of end uses in textile and apperal industry. Once fibres are found from chemical retting, it is essential to investigate major characteristics the textile properties including morphological structure, physical, mechanical, and thermal aspects. The next chapter will discuss these important textile properties in order to find out the suitability of *Typha* fibre for apparel application.

CHAPTER 5: TEXTILE CHARACTERISTICS OF *TYPHA LATIFOLIA* L. FIBRE

For apparel and non-textile applications (medical, geotextiles and bio-composites), fibres must have both textile properties such as length, diameter, moisture regain, chemical properties and absorbency and spinning properties such as softness and single fibre entity. In the current study, selected apparel and spinning properties are measured and discussed. It is worth mentioning that for this part of the research, the whole *Typha* plant was used for producing fibres.

5.1 Fibre morphology

Morphological analysis examines the size, shape, and structure of a material and show relationships between structural properties such as macrostructure, microstructure, submicroscopic structures and fine structure (Hatch, 1993, p.90).

5.1.1 Macrostructure of *Typha* fibre

Macrostructure analysis is one of the powerful techniques for studying visible structure information that helps to characterize the material structure of a fibre. It explores the general nature of the external surface by the naked eye or by using visible properties and components such as fibre length and diameter (Agrawal, 1988).

5.1.1.1 Fibre length

An important characteristic of any textile fibre is length, as machine settings, for example, distance between the drafting rollers during the process of spinning, and depend on fibre length (Figure 5.1). When the fibre length is longer than the distance between the drafting rollers then fibre breakage occurs due to the fibre being gripped by both sets of rollers; also, when the fibre length is shorter than the distance between the drafting rollers then fibre droppings occur (Rohlena, & Plisek, 1975). Furthermore, fibre length is an important consideration for yarn evenness and spinning performance in rotor spinning (Kumar, 2015). Fibres are classified into two categories

according to length: staple fibres that range in length from ¾ to 18 inches such as Cotton, linum, cannabis and brassica; and filament (infinite) length fibres such as polyester, nylon and acrylic. Staple fibres such as Cotton vary in length: Upland Cotton is reported to be between 0.8 to 1.36 inches (Cotton Incorporation, n.d.).

Fibre length influences yarn strength, as longer fibres provide better strength, elongation, smoothness, and evenness in a spun yarn (Kumar, 2015). A lower amount of twist can be used when spinning longer fibres, which translates into better efficiency of the spinning frame, as the speed of the machine is inversely proportional to the amount of twist inserted in a yarn (Rohlena, & Plisek, 1975). The importance of fibre length and uniformity in fibre length were also discussed in Section 2.3.1 and 2.3.2 (Chapter 2).

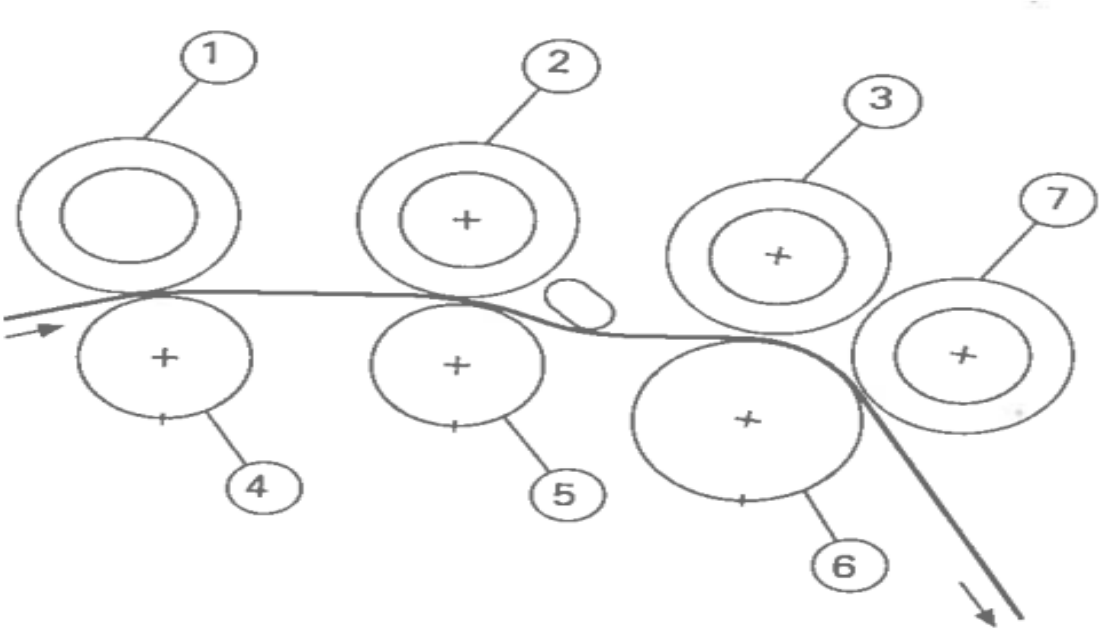


Figure 5.1: A performance of fibre length in spinning processing (A 4 over 3 roller arrangement drafting systems in a draw frame machine, where 1, 4: back drafting roller, 2, 5: middle drafting roller, 3, 6: front drafting roller and 7: guide roller) (Hunter, as cited by Khan, 2016).

5.1.1.1.1 Relationship between *Typha* plant length (cut length) and fibre length

Spin limits is the permissible linear density of yarn. The short staple (Cotton) spinning length varies between 9.5 to 37.5 mm and long staple (wool) spinning length varies usually between 25.4 to 63.5 mm (Stout, 1960). Man-made fibres are cut 1¼ inches for coarse and medium yarn count and 1½ inches for fine count, and for blending with Cotton, man-made fibre must cut to match the length of Cotton. For *Typha* fibre, length should not be a problem as fibre length could be controlled up to a certain length by selecting the cut length of the plant. However, two important characteristics of the fibre should be measured: the relationship between fibre length and cut length; and length uniformity in a fibre bundle.

Table 5.1 contains the sample ID with treatment time (h) and temperature (°C), original cut length of *Typha* plant, average (of 10 fibres) fibre length (mm) with standard deviation. It can be observed from this table that average fibre length is shorter than the cut length of the *Typha* plant. This might be due to the crimp in the fibre. Krowicki et al. (1997) reported that crimp in the fibre was the major source of error when determining the fibre length.

5.1.1.2 Statistical results

To check whether the plant cut length is same as the fibre length, a statistical analysis was used that had been suggested by Booth (1968) for smaller fibre sample size ($n \leq 10$). The t-statistic was conducted by comparing the means and standard deviations of the plant cut length and fibre length. The procedure is given below in which the total mean of seven plant samples and their standard deviations are used. Using pooled estimate of the sample standard deviation S= 4.5, *t*-test was calculated $t_{cal} = 2.40 > t_{12} = 2.179$ (T-table chart), formula given in Appendix XI.

Table 5.1: Statistical analysis on the relationship between plants cut length, and fibre length for different treatments.

Sample ID (hour / temp °C)	[a]Length(mm)	[b]Length(mm)	[c]Significant difference	[d]Significant difference
S8-K1-MS (2/80)	60.2± 0.00	54.00± 5.30	$t_{cal}\,(3.71)^{S} > t_{.05}\,(2.101)^{S}$	$t_{cal}\,(2.8)^{S} > t_{.05}\,(2.101)^{S}$
S8-K2-MS (4/80)	60.3± 0.00	54.00± 3.36	$t_{cal}\,(5.9)^{S} > t_{.05}\,(2.101)^{S}$	$t_{cal}\,(4.44)^{S} > t_{.05}\,(2.101)^{S}$
S8-K3-MS (6/80)	50.0± 0.00	47.00± 6.90	$t_{cal}\,(1.37)^{n} < t_{.05}\,(2.101)^{n}$	$t_{cal}\,(3.03)^{S} > t_{.05}\,(2.101)^{S}$
S10-L1-MS (2/80)	50.0± 0.00	42.00± 2.83	$t_{cal}\,(8.9)^{S} > t_{.05}\,(2.101)^{S}$	$t_{cal}\,(0.45)^{n} < t_{.05}\,(2.101)^{n}$
S10-L2-MS (4/80)	50.3± 0.00	41.00± 5.43	$t_{cal}\,(5.26)^{S} > t_{.05}\,(2.101)^{S}$	$t_{cal}\,(0.65)^{n} < t_{.05}\,(2.101)^{n}$
S10-L3-MS (6/80)	50.0± 0.00	42.00± 7.84	$t_{cal}\,(3.2)^{S} > t_{.05}\,(2.101)^{S}$	$t_{cal}\,(0.16)^{n} < t_{.05}\,(2.101)^{n}$
S10-L4-MS (8/80)	50.0± 0.00	39.00±10.10	$t_{cal}\,(3.95^{S} > t_{.05}\,(2.101)^{S}$	$t_{cal}\,(1.02)^{n} < t_{.05}\,(2.101)^{n}$
Mean	52.97 ± 4.97^{S}	45.57 ± 6.24^{S}		
t-Value, $_{(0.05)}$		2.45 >2.179		

[a]Original plant cut length; [b]Average fibre length; [n]No significant variation; [S]Significant variation; [c]Original cut length vs. Average cut length; [d]Original cut length vs. crimp adjusted length; S: Sample; K: KOH, L: LiOH; MS: Mixed Stem.

The *t*-test results are show in Table 5.1, which exhibited that the difference between the sample means of plant cut length and fibre length is statically significant as t_{calc} (2.45)> $t_{.05}$(2.179). This result rejects the null hypothesis and accept the alternate hypothesis (HA: $\mu > \mu 0$), the significant difference between the plants cut length and average fibre length as the original cut length of *Typha* plant (M=52.97, SD=4.97) had higher than average length of *Typha* fibre (M=45.57, SD =6.24). Moreover, the pair of each fibre sample means to the corresponding mean of the plant cut length demonstrated a statistically significant difference, as shown in Table 5.1, except the sample S8-K3-MS, which is not significant.

In Table 5.1, LiOH treated samples (S10-L1-MS, S10-L2-MS, S10-L3-MS and S10-L4-MS) presented shorter average length 42, 41, 42, and 39 compared to KOH treated average fibre length S8-K1-MS: 54, S8-K2-MS: 54 and S8-K3-MS: 47, respectively. Further, the visual observation of the fibre samples revealed that the LiOH treated samples [Figure 5.1 (a)] contain more short fibres as well as variation in fibre length than the KOH treated fibre [Figure 5.1 (b)].

The researchers found the ratio of 1:1.2 between the measured fibre length and crimp

adjusted length for Cotton fibre (Krowicki et al., 1997). The crimp adjusted length of the plants cut length and average fibre length in Table 5.1 showed that also all KOH treated fibre, S8-K1-MS (2/80), S8-K2-MS (4/80) and S8-K3-MS (6/80) had a significantly difference in *t*-test, whereas, all LiOH treated samples were not significant (Table 5.1). For the current study, statistical results established that using the ratio of 1:1.2 and considering standard deviation, the fibre length is almost equal for crimp adjusted four LiOH treated samples length to the cut length of the plant.

As mentioned earlier, for spinning machineries the fibre lengths must be uniform within 1/8 inch (reference). To verify the fibre length variation between two different alkali samples, data given in Appendix III and shown in Figures 5.1 (a) and 5.1 (b), 15 fibre samples (n = 15) were randomly chosen from the samples of S8-K4-MS and S10-L4–MS as suggested by ASTM standard D1440 - 07(2012). Fibre length variation for these two samples is shown in Figure 5.2. The average and standard deviations were calculated and found to be 40.0 mm ± 6.0, and 23.0 mm ± 10.0 respectively for S8-K4-MS and S10-L4-MS.

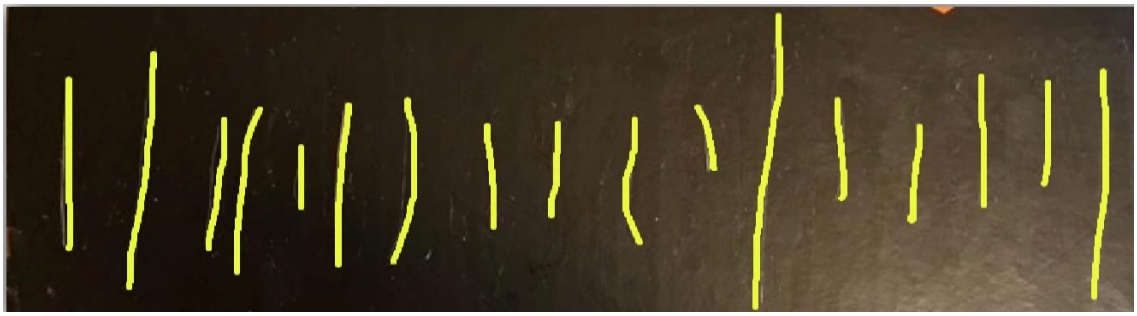


Figure 5.2 (a): *Typha* fibre length uniformity treated with LiOH (Sample: S10-L4-MS) (Painted by paint s).

Figure 5.2 (b): *Typha* fibre length uniformity treated with KOH (Sample: S8-K4-MS) (Painted by paint s).

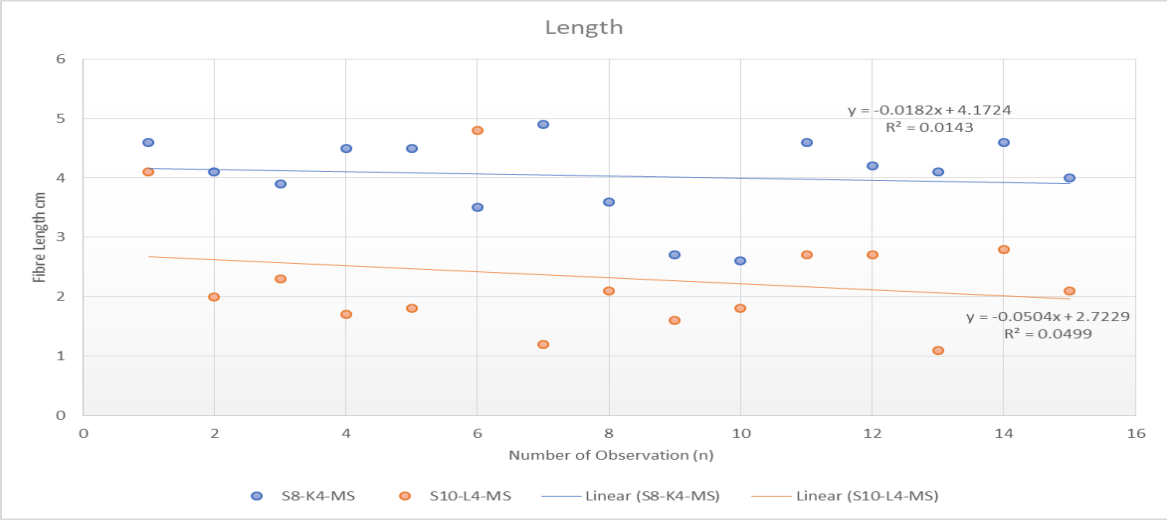


Figure 5.3: Fibre length variation between treated Sample S8-K4-MS and S10-L4-MS.

5.1.1.3 **Statistical analysis of *Typha* fibre length: Effect of treatment on fibre length**

Using this model a statistical analysis ANOVA was tested to find the effect of difference in treatment was examined. From the F-test results ($F_{1, 28}$) = $F_{critical}$ (4.20) < F_{cal} (29.50), it can be stated that the difference in the fibre lengths between the two treatments (T1 = S8-K4-MS; T2 = S10-L4-MS) was significant at p = 0.0001 <0.05. Since the difference is significant, the null hypothesis is rejected. Therefore, the effects of the treatments were shown to be different.

From the F-statistic, it was confirmed that the treatment effects are different. Least squares difference is used to compare the two treatment means. From the table, the estimate of least squares means for treatment S8-K4-MS = 4.0267 and the estimate of least square means for treatment S10-L4-MS = 2.3200. However, to examine whether both treatment effects are necessary for the model, a *t*-test was conducted for each estimated treatment effect, where $t_{critical}$ = 1.701, which is less than $t_{cal.}$ In both cases, $t_{cal} > t_{critical}$ and p < 0.05; therefore, the null hypothesis is rejected. In conclusion, the two alkali-treated samples S8-K4-MS and S10-L4-MS are individually significant and have different length variations.

5.1.1.4 **Implications**

The statistical results demonstrated there is two different alkali (NaOH & LiOH) treated samples which were significantly different in their lengths. During spinning, it must be considered that fibre has to be selected from individual lots, if there are mixed samples taken from different lots then length variation will increase and it will create problems in Cotton blending and roller drafting spinning system.

5.1.1.5 **Fibre Diameter**

Typha fibre diameter was measured using randomly chosen fibre. This was conducted by weighing the fibre of between 0.1 and 0.4 gram from each *Typha* samples. This was necessary in order to obtain an equal number of fibre in all thirteen *Typha* samples. The mean diameter from 15 different readings (the individual diameter readings are given in Appendix II) for each sample with their standard deviation and CV% results are given in Table 5.2 (a). It was found that the diameter varies between 45.27 (Sample ID# S10-L4-MS) µm to 107.47 µm (S5-NaOH-MS). It was also noticed the diameter variation was very high as seen from the standard deviation.

Table 5.2(a): *Typha* fibre diameter from various alkaline treatments.

Fibre diameter (µm) for *Typha*					
Sample Id	**Diameter**	**CV%**	**Sample Id**	**Diameter**	**CV%**
S5-NaOH-MS	107.47 ± 43.12 [a, n]	40.13	S9-K7-MS	65.27 ± 48.61 [a, n]	74.48
S8-K1-MS	93.67 ± 50.65 [a, n]	54.07	S9-K8-MS	77.33 ± 34.46 [a, n]	44.55
S8-K2-MS	37.67 ± 16.28 [a, n]	43.41	S10-L1-MS	93.73 ± 48.72 [a, n]	51.98
S8-K3-MS	58.47 ± 25.97 [a, n]	44.41	S10-L2-MS	82.73 ± 40.29 [a, n]	48.70
S8-K4-MS	76.87 ± 31.13 [a, n]	40.49	S10-L3-MS	78.00 ± 43.67 [a, n]	55.98
S9-K5-MS	82.07 ± 60.41 [a, s]	73.62	S10-L4-MS	45.27 ± 47.71 [a, n]	105.39
S9-K6-MS	46.27 ± 16.83 [a, n]	36.38			
p-value	0.247 > (0.05)				
Fcritical	1.95				
Fstatistical	1.31				
LSD, α=0.05	52.2				

[a]Mean ± Standard Deviation, [s]Significant, [n]Not Significant,

Table 5.2(a) showed that *Typha* fibre samples obtained higher diameter CV% value, when it was taken by random selection method. The alkali differences and due to the presence of group fibre in a single fibre. The resultant CV% was higher which indicates larger the diameter variation. With such larger diameter variation, it would be difficult to make yarn with uniform diameter.

A statistical experiment was performed using a single Factor ANOVA to determine whether there was a difference in diameter between the *Typha* samples. The difference in diameter is not found to be significant as $p = 0.247 > 0.05$ (Table 5.2 (a)). However, the Fisher LSD test exhibits a significant difference between the means of S5-NaOH-MS and S8-K2-MS (69.8>52.2), S5-NaOH-MS and S9-K6-MS (61.2>52.2), S5-NaOH-MS and S10L4-MS, (62.2>52.2), S8-K1-MS and S8-K2-MS, (56>52.2), S9K7-MS and S10-L1-MS, (93.73>52.2), S8-K2-MS and S10-L1-MS, (56.06>52.2), respectively (Table 5.2(b)). No other comparisons were found to be significant, which was due to the larger variation among the diameters.

Table 5.2(b): Fisher's LSD test to compare the difference
Between pairs of means for diameter of the thirteen *Typha* fibre.

Group 1 (Y1)	**Group 2 (Y2)**	**|Y1- Y2|**
S5-NaOH-MS^{S}	S8-K2-MS^{S}	69.8(>52.2)^{S}
S5-NaOH-MS^{S}	S9-K6-MS^{S}	61.2(>52.2)^{S}
S5-NaOH-MS^{S}	S10-L4-MS^{S}	62.2(>52.2) ^{S}
S9-K7-MS^{S}	S10-L1-MS^{S}	93.7(>52.2)^{S}
S8-K1-MS^{S}	S8-K2-MS^{S}	56.0(>52.2)^{S}
S8-K2-MS^{S}	S10-L1-MS^{S}	56.1(>52.2)^{S}

^{s}Significant, $LSD_{(\alpha=0.05)} = 52.2$

The diameter data obtained from random sampling of *Typha* fibre showed the larger diameter than the Cotton and wool fibres as the diameter of these two fibres varies between 10 to 25 µm (Hatch, 2006). Therefore, no comparison was made between *Typha* fibres with Cotton and wool. However, during diameter measurements, it was noticed that the *Typha* fibre was not a single fibre as the fibre comprised of multiple single fibre. To compare the diameter of *Typha* with Cotton and wool, single Typha fibres were collected from biological *Typha* sample S8-K3-MS.

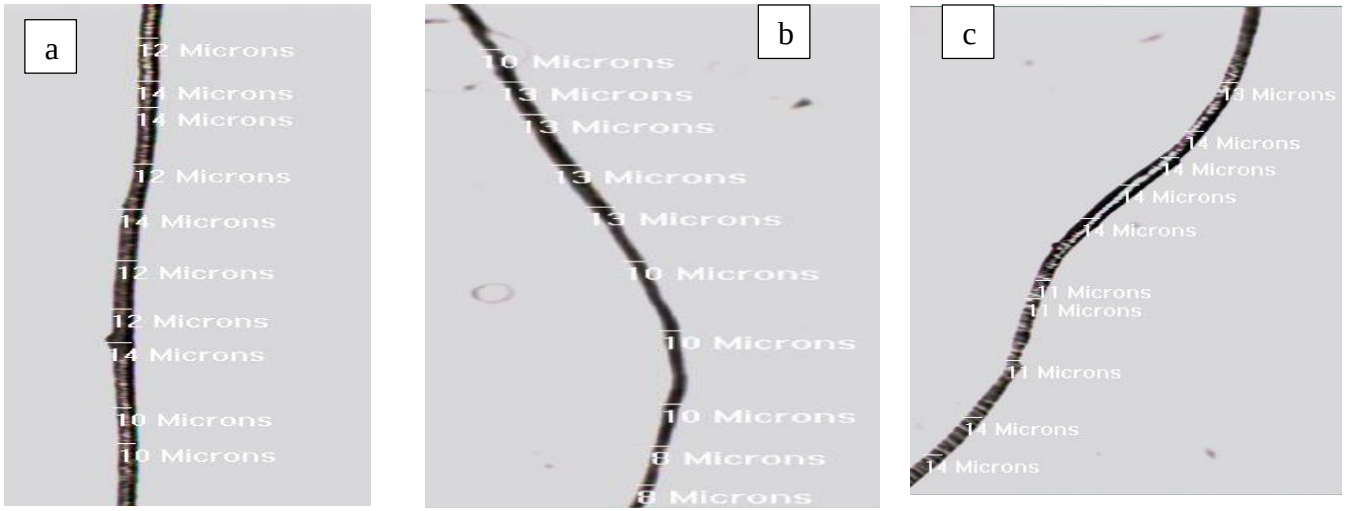


Figure 5.4: Diameter variation in 10 different places of *Typha* (a), Cotton (b), Wool (c).

Table 5.3(a): Diameter (µm) of *Typha*, Cotton, and Wool fibre.

Fibre type	**Fibre #1**	**Fibre #2**	**Fibre#3**	**Fibre#4**	**Fibre#5**	**Fibre #6**	**Fibre#7**	**Fibre#8**	**Fibre #9**	**Fibre #10**	**Grand Fibre#**
Typha (S8-K3-MS)	12.4 (±1.5)[a]	15.4 (±0.69)[a]	14.4 (±0.96)[a]	12.8 (±0.63)[a]	12.2 (±1.03)[a]	12.7 (±3.03)[a]	14.0 (±1.4)[a]	14.6 (±1.64)[a]	8.4 (±1.50)[a]	10.2 (±2.04)[a]	12.7 (± 2.5)[b]
Cotton	11.2 (±2.9)[a]	11.0 (±2.10)[a]	10.8 (±2.04)[a]	12.4 (±1.57)[a]	13.3 (±1.88)[a]	11.6 (±0.84)[a]	14.1 (±0.99)[a]	11.7 (±1.56)[a]	12.5 (±1.58)[a]	13.6 (±0.96)[a]	12.2 (±1.94)[b]
Wool	15.0 (±0.0)[a]	15.5 (± 1.84)[a]	14.2 (± 0.42)[a]	7.9 (±0.56)[a]	12.0 (±0.0)[a]	13.2 (±1.39)[a]	11.8 (±0.42)[a]	12.1 (±0.73)[a]	13.0 (±1.41)[a]	13.9 (±0.87)[a]	12.8 (± 2.25)[b]

[a] Mean± Standard Deviation, [b]Grand Total Average of 100 readings;

5.1.1.6 **Statistical analysis**

A statistical test was carried to measure the significant difference of the fibre diameter between *Typha* and Cotton and *Typha* & Wool fibres using SAS II.

Table 5.4(a): Statistical summary of *Typha* and Cotton fibre diameter.

Fibre	**Mean (µm)**	**Std Dev**	**Std Err**	**Minimum (µm)**	**Maximum (µm)**	**Variance**
Typha **(**S8-K3-MS**)**	12.71	2.54	0.26	7.00	18.00	6.43
Cotton	12.22	1.94	0.19	8.00	15.00	3.75
F_{cal}			1.72			
$F_{critical}$			1.39			
Levene's F test (*p*-value)			0.0078 (< 0.05)			
t-test (*p*-value)			0.1263 (> 0.05)			

N = 100 samples each of *Typha* and Cotton fibres.

Table 5.4(a) summarizes *Typha* and Cotton diameters, where the maximum value for *Typha* is 18 µm, and for Cotton is 15 µm and the minimum value for *Typha* is 7 µm, and for Cotton is 8 µm. The mean value of *Typha* and Cotton is almost similar: 12.7 µm and 12.2 µm.

Equality of variances (Table 5.5) between *Typha* versus Cotton shows that p= 0.0078 < 0.05 and $F_{critical}$ (1.39) < F_{cal} (1.72). So the null hypothesis is rejected. Therefore, the variances for *Typha* and Cotton are not equal.

Since the variance analysis determines that the variances between *Typha* and Cotton were not equal, therefore, the Satterthwaite method was used to conduct the *t*-test for unequal variance. The *t*-test formulas are given in (Appendix VIII). Null hypothesis: $d_1 = d_2$. The diameters of *Typha* and Cotton are equal. Alternative hypothesis: $d_1 \neq d_2$. The diameters of the *Typha* and Cotton are not equal. The *t*-test results revealed that p= 0.1263 > .05, so, the null hypothesis has been shown in Table 5.4(a). Therefore, the diameter of Cotton and *Typha* were deemed to be equal.

Table 5.4(b): Statistical summary of *Typha* and Wool fibre diameter.

Fibre	Mean (µm)	Std Dev	Std Err	Minimum (µm)	Maximum (µm)	Variance
Typha **(**S8-K3-MS**)**	12.71	2.54	0.26	7.00	18.00	6.43
Wool	12.86	2.25	0.22	7.00	18.00	5.05
F_{cal}			1.27			
$F_{critical}$			1.39			
Levene's F test (*p*-value)			0.2314 (> 0.05)			
t-test (*p*-value)			0.6585 (> 0.05)			

N = 100 samples each of *Typha* and Wool fibres.

Table 5.4(b) shows that the maximum and minimum value for *Typha* and Wool are both the same value. The mean value of *Typha* and Wool is almost the same: 12.7 µm and 12.8 µm. First, the homogeneity of the variances was examined in order to perform the *t*-test correctly. From the equality of variances between *Typha* and Wool it was found that $F_{critical}$ (1.39) > F_{cal} (1.27), p = 0.2314 > .05, so we accept the null hypothesis. Therefore, the variances are equal. The *t*-test was performed using the pooled method considering the variances are equal. Null hypothesis: $d_1 = d_3$. The diameters of *Typha* and wool are equal. Alternative hypothesis: $d_1 \neq d_3$. The diameters of the *Typha* and Wool are not equal. The *t*-test revealed that p= 0.6585 > .05. So, we accept the null hypothesis. Therefore, the diameter of *Typha* and wool were deemed to be statistically equal.

It can be concluded that there is no significant difference in the diameters of *Typha,* Cotton and wool fibres. Therefore, *Typha* fibre could be a replacement for wool or Cotton in Cotton spinning system. It should be mentioned that in case of multiple *t*-test comparisons, type I (α) error rate is inflated. However, the comparison error rate (α_c) was .05, and the experimental error rate (α_E) was .00975.

5.1.1.7 **Summary**

It can be concluded that from the macrostructure analysis, *Typha* fibre is suitable for Cotton spinning systems, though fibre length uniformity of *Typha* depends on the type of alkaline chemical treatment. The hypothesis of *t*-test established that, *Typha* fibre is similar to wool fibre

and Cotton fibre.

5.1.2 Microstructure of *Typha* fibre

The microstructure of fibres and plants provides information on surface contour and cross-sectional shape using a light microscope. The microstructures of textile fibres affect the physical properties and behaviours of a material, for example, the scales on the wool surface are responsible for the warmth characteristic of wool [Figure 5.7 (c)].

5.1.2.1 Cross-sectional features

A variety of cross-sectional shapes such as round, triangular, dog-bone, kidney bean, flat, and multil-Oval shapes have been identified for various textile fibres (Hatch, 2006, p.91). As seen in Figure 5.5, wool has irregular cross-section (a) Cotton fibre has a kidney shape (b); man-made fibre cross sections depend on the shape of the extruder, which can be circular (c). Other cross-sectional shapes such as trioval, penta-oval, and so forth can also be manufactured (Hatch, 2006, p.91). Each shape provides distinct features that will make the fibre suitable for specific end products. For example, an irregular cross-section is desirable for a lustrous fabric.

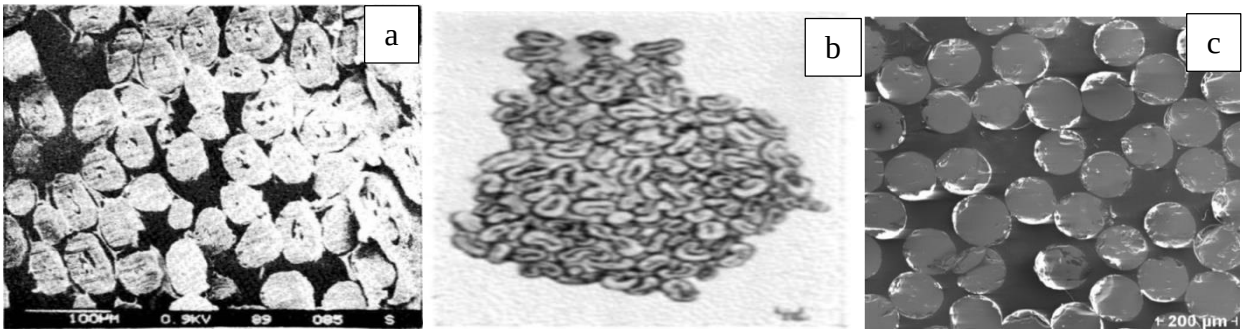


Figure 5.5: Cross-section of wool (Rao & Gupta, 1991), Cross-section of Cotton (Rowland et al., 1976) Cross-section of polyester (Brauer et al., 2008)

A cross-section analysis of *Typha* plant was carried out using an ordinary microscope; however, a Scanning Electron Microscope (SEM) was used to measure both *Typha* plant and fibres

cross-section. The results of SEM are discussed in submicroscopic structure section.

The cross-sections of two *Typha* plants show two distinct features. Figure 5.6 (a) shows the cross-section of a no stem plant (layers of leaf) and Figure 5.6 (b) shows the cross-section of a hard stem plant. It can be seen that the no stem plant does not have any inner hard sponge, however, spongy tissues can be seen inside the leaf; whereas, the hard stem plant has an inner spongy core as shown in Figure 5.6 (b). The brief description of these two different *Typha* plants was provided in Section 4.2 (Chapter 4).

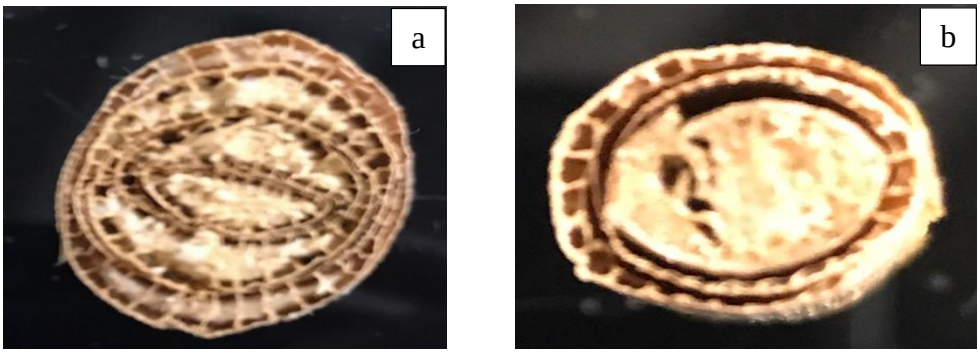


Figure 5.6: Cross-section of no stem plant (a) and hard stem plant (b).

5.1.2.2 **Longitudinal view of fibre**

The longitudinal view of a fibre shows the surface contour which varies depending on the fibre type, and it can be smooth, serrated, lobed, striated, pitted, scaly or convoluted (Hatch, 1993, p.91). The longitudinal view of *Typha* fibre along with Cotton, wool and polyester fibres are shown in Figure 5.7 (a), (b), (c), and (d), respectively. Unlike Cotton, which shows a twisted structure [Figure 5.7 (b)], and wool, which shows a scaly surface [Figure 5.7 (c)], *Typha* fibre [Figure 5.7 (a)] does not have such distinct microscopic features. However, it appears that the surface of the *Typha* fibre is rough, and not as smooth as polyester fibre [Figure 5.7 (b)].

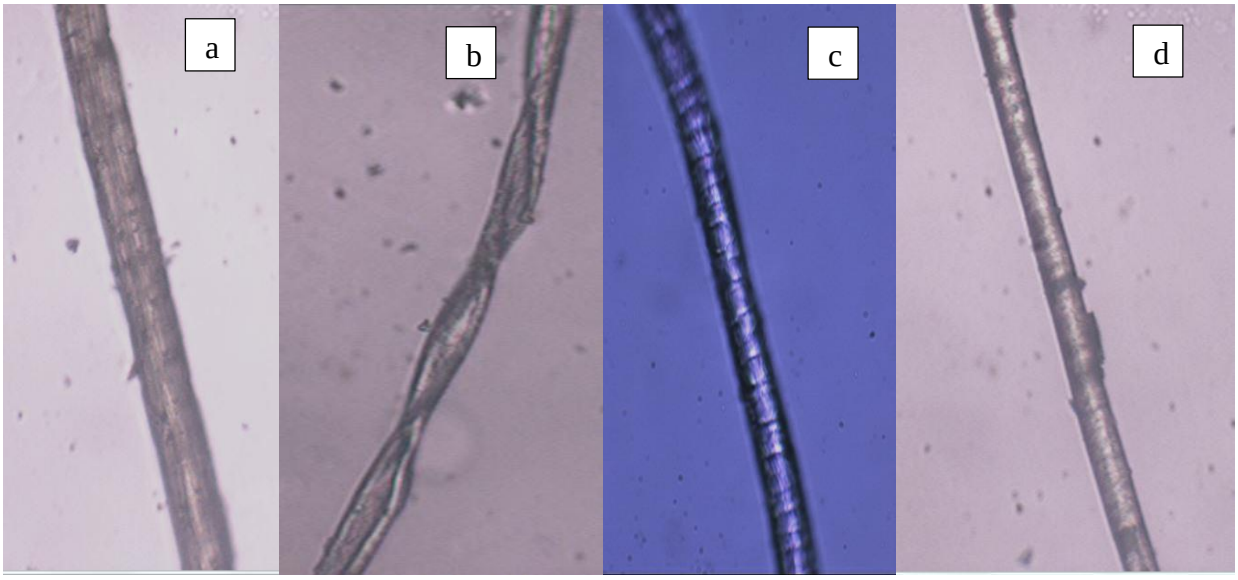


Figure 5.7: Longitudinal view of *Typha* (a); Cotton (b); Wool (c); Polyester (d).

5.1.2.3 **Submicroscopic structure**

The submicroscopic structure refers to a cross-sectional view of a fibre or plant as observed through a scanning electron microscope. Most natural fibres have distinct submicroscopic layers. The microscopic view wool fibre obtains a cuticle, a cortex, and a medulla whereas, man-made fibres and silk tend to be uniform throughout and lack readily visible submicroscopic properties (Hatch, 2006, p.92). The outer layer of material forming the fibre is coarser than the material forming the centre. In general, bast fibres such as linum, cannabis and corchorus are extracted from the outer layer (epidermis) of the plant. As a result, the yield (%) of these fibres ranges between 10 to 20% (Khan, 2016). Current research on the *Typha* plant found that after treatment in alkaline solutions, the whole *Typha* plant except bark could be transformed into fibre, making the yield (%) as high as 40 to 60%, depending on the extraction temperature and time in section (4.1.2). Therefore, the submicroscopic structures of *Typha* fibre might be different than that of other natural cellulosic fibres. Further, a structural analysis using scanning electron microscopy (SEM) offered an explanation of *Typha* fibre behaviour that can affect its textile properties.

The cross-section of a *Typha* leaf is interconnected by a number of ribs. Microscopic examination shows a 'crenelated' (rectangular indentation) structure: small ridges corresponding to the fibres can clearly be seen and felt with a thumbnail (Figure 5.12). The ribs and outer surface of the leaf act mechanically and build a protective shield to bending deflections. The (SEM) reveals that the *Typha* fibres lie inside the epidermal layer of the leaf.

The cross-section of the *Typha* plant is divided into two distinct layers: the outer layer (epidermis, cross-sectional area 38.5 mm^2) and the inner layer (probable fibre layer, area 20.1 mm^2) (Figure 5.8). The ratio of fibre to plant area is 52.0%, which is within the range of fibre yield (%). The general structure of the cross section of a *Typha* fibre consists of numerous small elliptical, radially-shaped cells, each about 4.5 – 6.0 µm, (Figure 5.9) and about 0.70 µm apart in a group cell of 740 µm (Figure 5.10). Each cell has a lumen (≈1.03 µm) (as shown in Figure 5.5) in the middle that indicates the maturity of the fibre cell (Hatch, 2006) and defines fibre strength and fineness. The fibre cells are not attached to each other, as there is a 'canal' between the cells. The structure of the cells provides the potential for multiple excellent textile properties (dyeing absorbency, diameter, thermal properties, fibre flexibility and easy fibre separation).

The crenelated structure may positively contribute to good dye absorbency and thermal insulation; the fibres are very easy to separate, making them suitable for spun yarn conversion; the diameter of the *Typha* fibre is comparable to Cotton and wool; the fibre cell has a lumen, and the cells are separated by canal, a feature that might have significant implications in biomedical applications for trapping antibiotics and other chemicals. The yield (%) is about 54%, which is close to the ratio of total plant area to inner (probable fibre) area. From the morphological studies, it is concluded that the *Typha* fibre could be used for both textile and biomedical applications.

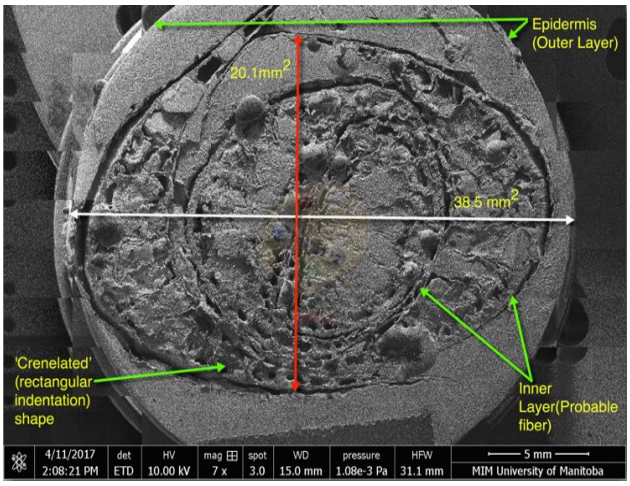


Figure 5.8: Cross-section area of *Typha* plant with two distinct layers

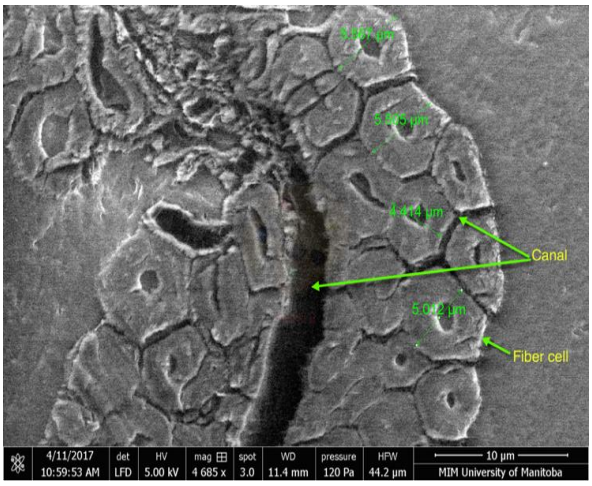


Figure 5.9: Individual cell size and distance of *Typha* Fibre

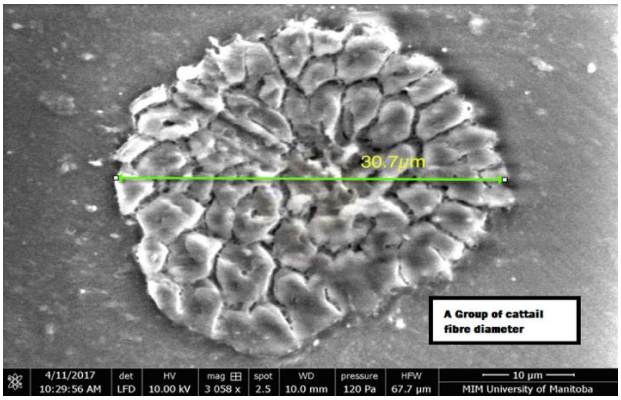


Figure 5.10: A group of *Typha* fibre diameter

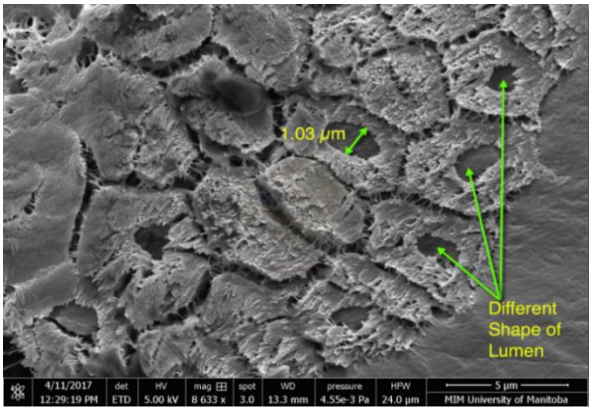


Figure 5.11: Polygonal ultimate cells with lumen size

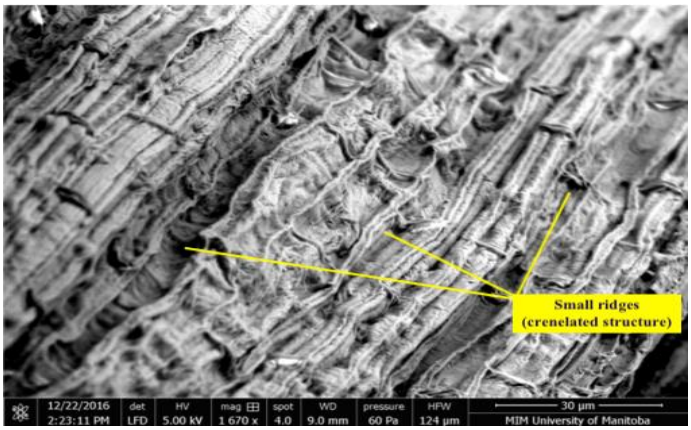


Figure 5.12: Small ridges ‘crenelated ‘structure of *Typha* leaf (rectangular indentation) structure.

Table 5.5: Summary of *Typha* microscopic study.

Microscopic Characteristic	Properties	Figure no.
Two distinct layers	38.5 mm^2 (outer layer area) and 20.1 mm^2 (inner layer area)	5.8
Individual cell size and distance of *Typha* fibre	4.5 – 6.0 µm/cell	5.9
Typha fibre diameter in group	30.0 µm	5.10
Cell lumen size	1.03 µm	5.11
'Crenelated' structure of T*ypha* leaf (rectangular indentation)	Small ridges	5.12

5.2 *Typha* fibre chemical composition analysis by Fourier Transform Infrared Spectroscopy (FTIR)

The primary polymer components of the cell wall, cellulose, hemicellulose, and lignin, are comprised in natural plants (Zheng et al., 2017). Cellulose, the main element of polysaccharides, which are principle components in natural plant fibres, acts as the supporting material in the cell wall (Zheng et al., 2017). The cellulose molecules are bonded in a microfibril structure in which there is extensive hydrogen bonding between cellulose chains, producing a strong crystalline structure (Thomas et al., 2013). FTIR is one of the most important analytical and advanced tools used to obtain information on the super-molecular structure and chemical compositions of *Typha* fibre. FTIR spectra were taken between 500-4000 cm^{-1} wave numbers for *Typha* no stem plant (leaf + sponge), and *Typha* fibres obtained from alkaline treatments. The FTIR spectrum of *Typha* (leaf + sponge), fibre was compared with Cotton, *Typha* leaf and cannabis fibre from the literature.

Table 5.6: Peak Assignments (cm^{-1}) of IR Spectra for *Typha* fibres.

***Typha* fibre and sample ID#**						**FTIR Spectrum value obtained from literature**				
***Typha* (leaf + sponge)**	**S8-K2-MS (55%)**	**S5-NaOH-MS (56%)**	**S8-K3-MS (35%)**	**S10-L1-MS (66%)**	**Cotton***	***Typha* leaf***	**Cannabis ***	**From Literature (cm^{-1})***	**Peak Sources**	**Reference**
3404.75	3433.25	3430.08	3415.95	3452.24	Yes	Yes	Yes	3550 -3100	H-bonded OH stretch with cellulose, hemicellulose content	Dave et al.,2014; Sana et al., 2011;
2918.83	2920.96	2918.82	2917.15	2919.54	Yes	Yes	Yes	2800- 2980	C-H stretching with cellulose content	Cesar et al.,2015
2850.79	2850.79	2850.79	2849.75	2850.79	Yes	Yes	Yes	2849	Symmetric aliphatic CH_2 stretch with wax content	Dave et al.,2014; Cao et al.;2016; Jacek et al., 2011 Sana et al.,2014
1733.66	-	-	1737.58	-	Yes	Yes	Yes	1734- 1750	C=O stretch in -COOH, hemicellulose, lignin, pectin content	Dave et al.,2014; Kaczmar et al., 2011 Cao et al.,2016
1635.49	1635.02	1634.94	-	-	Yes	Yes	No	1631-1644	Adsorbed H_2O, no crystalline region of cellulose	Dave et al.,2014; Sana et al.,2014 Cesar et al.,2015
-	-	-	1651.47	1652.25	Yes	No	No	1648	Absorbed H_2O	Cesar et al.,2015 Pandey, 2007
1516.49	1507.23	-	-	1506.39	Yes	No	Yes	1506, 1550	C=C stretching of the aromatic ring, lignin	Sana et al.,2014; Dave et al.,2014;
1456.66	-	-	1467.55	1456.60	No	Yes	Yes	1429, 1465	C-H asymmetric bending in CH_3 with lignin content	Dave et al.,2014; Kaczmar et al., 2011
1383.63	1384.08	1384.08	1384.11	1384.37	Yes	No	No	1380	CH_2 and C-H bending (deformation stretch) with lignin and alpha cellulose content	Sana et al.,2016 Cesar et al.,2015
1249.39	1265.10	-	-	-	No	No	No	1249.5	C=O stretching vibration of ketones, carboxylic group and esters in lignin and acetyl ester groups in xylan.	Cao et al.,2016; Abdullah et al., 2010
1157.47	1165.15	1162.58	1165.15	-	Yes	Yes	Yes	1155, 1157,1160	Asymmetric bridge stretching with cellulose and hemicellulose	Sana et al.,2014; Dave et al.,2014; Kaczmar et al., 2011
1049.91	-	-	1058.61	1059.54	Yes	Yes	No	1057,1061	C-O Stretching	Cesar et al.,2015; Dave et al.,2014;
893.93	896.85	896.85	896.85	-	Yes	Yes	Yes	894-900	Asymmetric out of phase ring stretch: C1-O-C4, β-glucosidic bond	Sana et al.,2014; Dave et al.,2014; Kaczmar et al., 2011
600.82	591.36	-	-	610.41	Yes	Yes	Yes	658, 675	OH out-of-plane bending and atmospheric CO_2 (deformation vibration) contamination.	Sana et al.,2014; Cesar et al.,2015;

*Collected from literature.

Table 5.6 shows the peak assignments of FTIR spectra using KBr pellet method obtained from *Typha* (leaf + sponge), and fibre (S8- K2-MS, S5-NaOH-MS, S8-K3-MS and S10-L1-MS) are given in Appendix IV (Figures 5.13 and 5.14(a) to 5.14(e)). IR spectra for Cotton, *Typha*, and Cannabis fibres collected from literature are compared with *Typha* (leaf + sponge) and fibres.

The most important component of cellulosic materials is cellulose, a fibre forming group. As with Cotton and other cellulosic materials, this group is dominant in *Typha* fibres. The cellulosic groups in *Typha* (leaf + sponge) and fibres can be found at wavenumber of 3433.25 cm^{-1} (peak range: 3550 -3100) that corresponds to H-bonded OH stretch with cellulose, and hemicellulose content. This band can be found in *Typha* (leaf+sponge), *Typha* fibres (S8-K2-MS, S5-NaOH-MS, S8-K3-MS and S10-L1-MS), Cotton (Dave et al., 2014), *Typha* (Cesar et al. 2015) and Cannabis fibre (Kaczmar et al., 2011). The band at 2918.83 cm^{-1} in the *Typha* (leaf+sponge) and fibres falls within the frequency range of 2800 – 2980 cm^{-1} which is C-H stretching with cellulose content. Such groups can also be found in Cotton (Dave et al. 2014), *Typha* (Cesar et al., 2015) and Cannabis fibres (Kaczmar et al., 2011). The cellulose group (asymmetric bridge stretching) is found in the frequency range of 1155 cm^{-1} to 1165 cm^{-1} for *Typha* (leaf + sponge), *Typha* fibres except S10-L1-MS samples as well as Cotton, *Typha* leaf and Cannabis fibres (Table 5.12). Another component of cellulosic materials is wax which can represent up to 5.0% and is removed before wet processing treatment by scouring (McCall & Jurgens, 1951). The wax group attributed to symmetric aliphatic CH_2 stretch in *Typha* materials [Figures 5.14 (a) – 5.14(f)], Cotton (Dave et al., 2014), *Typha* (Cao et al., 2016) and Cannabis (Kaczmar et al., 2011) can be found at 2850 cm^{-1}. The hemicellulose, lignin, pectin content can be found at the wave number 1733.66 cm^{-1} due to the C=O stretch in –COOH. These peaks can be found only in *Typha* (leaf + sponge) and one *Typha* fibre sample (S8-K3-MS), Cotton, *Typha* and Cannabis fibre. The absence of this group in samples S8-K2-MS, S5-NaOH-MS and S10-L1-MS may be because of the removal of

hemicellulose, lignin, and pectin during alkaline treatment. The peaks due to the absorbed water, in the range of 1631-1644 cm^{-1} and 1648 $cm^{-1,}$ can be seen for both *Typha* plants and Cotton. However, only *Typha* fibre samples of S8-K2-MS and S5-NaOH-MS exhibit a water group at 1635 cm^{-1}; S8-K3-MS and S10-L1-MS samples showed a water group in the range of 1651 cm^{-1} to 1652 cm^{-1}. No such peaks can be found for Cannabis, and only in the range of 1631-1644 cm^{-1} for *Typha*.

The lignin groups are observed at multiple peaks. These peaks, at wave lengths 1516 cm^{-1}, 1456 cm^{-1}, 1383 cm^{-1} and 1249 cm^{-1} for *Typha* plants, belong to an aromatic ring in lignin, C-H asymmetric bending in CH_3 with lignin, CH_2 and C-H bending (deformation stretch) with lignin, and C=O stretching vibration of ketones, carboxylic group and esters in lignin as well as acetyl ester groups in xylan, respectively. For *Typha* fibres, only 1507 cm^{-1}, 1384 cm^{-1} and 1265 cm^{-1} can be found for S8-K2-MS sample; 1384 cm^{-1} for S5-NaOH-MS; 1467 cm^{-1} and 1384 cm^{-1} for S8-K3-MS; and 1506 cm^{-1}, 1456 cm^{-1} and 1384 cm^{-1} for S10-L1-MS samples. Such presence and absence of lignin groups in *Typha* fibres cannot be explained without further research. However, this behaviour is also found for Cotton (Dave et al., 2014), *Typha* leaf (Cesar at al., 2015) and Cannabis fibre (Kaczmar et al., 2011) as recorded in Table 5.12. One of the major bonds is the β-glucosidic bond which connects repeating units of cellubiose in the cellulosic materials. This bond can be found at the frequency range of 894 cm^{-1} to 900 cm^{-1} in all samples except S10-L1-MS. It was noticed that fibres in this sample were broken into smaller lengths during extraction and the absence of β-glucosidic bond may be the reason for the shorter fibre length.

Finally, the IR spectra of untreated *Typha* (leaf+sponge) and *Typha* fibres show similar characteristic features related to cellulose, lignin, hemicellulose, and wax components that also can be found in Cotton, *Typha* leaf and Cannabis fibres. It was noticed that *Typha* leaf does not exist CH_2 and C-H bending (deformation stretch) with lignin and alpha cellulose and C=O

stretching vibration of ketones, carboxylic group and esters in lignin and acetyl ester groups in xylan content whereas *Typha* (leaf+sponge) and fibre sample S8-K2-MS have that contents may be the presents of spongy materials.

5.3 Moisture Regain (%) of *Typha* plants and fibre

Moisture is an important term in textiles that relates to wearer comfort, shrinkage, rate of drying and static electricity (Taylor, 1972). Moisture regain (%) is calculated according to the formula given in Section 3.3.4. The moisture regain (%) of *Typha* plants and fibre obtained from various treatments and conditions (relative humidity and temperature) is given in Tables 5.7(a) For comparison, wool fibre (raw), whose moisture regain has already been established, is also determined. The use of wool fibre helped to determine the accuracy of the moisture regain measurement process as literature values for wool are available.

Table 5.7(a): Moisture regain (%) of *Typha* plants at various humidity and temperatures.

Sample type	Conditioning atmosphere (RH% and temp °C)	Conditioning duration (hour)	Drying time (hour) temperature (°C)	Moisture regain (%)
Virgin *Typha* plant	50%, 20°C[a]	12	4/105	9.4
Virgin *Typha* plant	50%, 20°C [a]	12	8/105	12.7
Virgin *Typha* plant	70%, 16°C [c]	12	8/105	9.8
[1]Virgin *Typha* plant	61%, 24.9°C [b]	72	10/105	10.6
[2]Virgin *Typha* plant	59%, 25°C [b]	72	10/105	9.6
Wool fibre(raw)	70%, 16°C [b]	12	8/105	9.6
[1]Wool fibre (raw)	61%, 24.9°C [b]	72	10/105	11.1
[2]Wool fibre (raw)	59%, 25°C [b]	72	10/105	8.8
Wool (raw)	65%, 21°C [a]	*≥8	Until constant weight/105	11.0

[1] sample was conditioned from atmospheric condition; [2] sample was conditioned from dry state.
[a]conditioning room, [b]humidity chamber, [c]outside weather.*Literature value of Wool (Booth, 1968)

The moisture regain (%) for *Typha* plant ranges from 9.4 to 12.7 (Table 5.7(a)). For the same relative humidity (RH% = 50), the increase in drying time from four hours to eight hours enhances the moisture regain (%) by about 35% (Table 5.7(a), Rows 1 & 2). To determine the

hysteresis of *Typha* plants, samples were conditioned from atmospheric condition and dry state. The moisture regain (%) was 10.6 and 9.6 for *Typha* samples conditioned from atmospheric and dry states respectively (Table 5.7(a), Rows 4 & 5) and the difference in moisture regain was found to be 1.0%.

A similar experiment was conducted for wool fibre and the hysteresis was found to be 2.3%. The hysteresis value for wool fibre varies depending on the wool fibre type (woolen, worsted), yarn type and other factors. However, a more reasonable value is 2.1% as reported by (Cookson and Slota, 1993). The experimental moisture regain (%) obtained from the current study for wool fibre is similar to the published value (Booth, 1968). The measured moisture regain (%) following chemical treatment for *Typha* fibre, (considering relative humidity) is shown in Table 5.7(b).

Table 5.7(b) shows the average moisture regain at 50% relative humidity and 20°C temperature, standard deviations and CV% of various *Typha* samples. The average moisture regain (%) for the *Typha* fibres varies between 3.69 (S8-K3) and 10.9 (S8K2). The moisture regain for most of the *Typha* fibres is lower than the Typha plant (9.57%) except S8-K2 sample (10.90%). A *t*-test experiment was conducted (formula given in Appendix XI) to investigate significant difference between *Typha*, Wool and Cotton shown in Appendix VI. It was found *Typha* and wool fibres has significant difference t_{cal} (2.461) > $t_{statistical}$ (2.179), however, *Typha and* Cotton fibre were not statistically significant, t_{cal} (0.759) < $t_{statistical}$ (2.179) in Table 5.7(b).

The statistical analysis of ANOVA test found no significant difference among these samples (Table 5.7(b)). However, the Fisher's LSD test was conducted and found significant variation between *Typha* plant and S8K3-MS (5.87>4.58), S8K3-MS and S8K4-MS (5.34>4.58),

S5-NaOH-MS and S8K2-MS (4.87>4.58), S8K2-MS and S8K3-MS, (7.21>4.58) in Table 5.7(c).

Another statistical *t*-test analysis was conducted to compre *Typha* fibre with between common fibres Wool and Cotton (data was taken from literature: Cotton (Liggett et al., 1968,) Wool (Kopke & Lindberg, (1966),) and *Typha* (experimental data from Table 5.7(b)). The *t*-test results showed between *Typha* vs. Wool (Appindix VI) were significant and *Typha* vs. Cotton (Appendix VI) were not significant as shown in Table 5.7(b).

Table 5.7(b): Moisture regain (50% relative humidity and 20°C temperature) of *Typha* plant and fibre obtained from various treatments and statistical analysis (ANOVA and Fisher's LSD test).

	Sample type							
Experiment #	Virgin *Typha* plant	S5-NaOH-MS	S8K1-MS	S8K2-MS	S8K3-MS	S10L3-MS	S9K3-MS	S8K4-MS
1	9.4	5.90	10.1	9.40	2.08	7.90	5.50	7.80
2	12.7	8.60	4.50	9.80	5.10	6.40	9.50	13.80
3	6.60	3.60	9.70	13.50	3.90	8.10	7.30	5.50
Mean	9.57	6.03	8.10	10.90	3.69	7.47	7.43	9.03
	$\pm 3.1^{a, n}$	$\pm 2.5^{a, n}$	$\pm 3.12^{a, n}$	$\pm 2.26^{a, n}$	$\pm 1.52^{a, n}$	$\pm 0.93^{a, n}$	$\pm 2.00^{a, n}$	$\pm 4.29^{a, n}$
CV%	31.92	41.48	38.57	20.74	41.17	12.44	26.95	47.44
p-Value				0.10(>0.05)				
$F_{critical}$				2.66				
$F_{statistical}$				2.12				
LSD, α= 0.05				4.58				
Typha (fibres) vs. Wool^{s}				$P_{0.05}=t_{cal}$ (2.461) > $t_{statistical}$ (2.179)^{s}				
Typha (fibres) vs. Cotton^{n}				$P_{0.05}=t_{cal}$ (0.759) < $t_{statistical}$ (2.179)^{n}				

[a]Mean± Standard Deviation, N=3, [n]Not significant, S: Sample; K: KOH, L: LOH,

Table 5.7(c): The comparison of moisture regain (%) between pair of means for *Typha* plant and fibres by Fisher's LSD test.

Group 1 (Y1)	Group 2 (Y2)	|Y1- Y2|	Group 1 (Y1)	Group 1 (Y1)	|Y1- Y2|
Virgin typha plant^{n}	S5-NaOH-MS$^{n\,n}$	3.53(<4.58)^{n}	S5-NaOH-MS ^{n}	S8K1-MS ^{n}	2.07(<4.58)^{n}
Virgin typha plant^{n}	S8K1-MS ^{n}	1.47(<4.58)^{n}	S5-NaOH-MS ^{S}	S8K2-MS ^{S}	4.87(>4.58)^{S}
Virgin typha plant^{n}	S8K2-MS ^{n}	1.33(<4.58)^{n}	S5-NaOH-MS ^{n}	S8K3-MS ^{n}	2.34(<4.58)^{n}
Virgin typha plant^{S}	S8K3-MS^{S}	5.87(>4.58)^{S}	S5-NaOH-MS ^{n}	S10L3-MS ^{n}	1.43(<4.58)^{n}
Virgin typha plant^{n}	S10L3-MS ^{n}	2.10(<4.58)^{n}	S5-NaOH-MS ^{n}	S9K3-MS ^{n}	1.40(<4.58)^{n}
Virgin typha plant^{n}	S9K3-MS ^{n}	2.13(<4.58)^{n}	S5-NaOH-MS ^{n}	S8K4-MS ^{n}	3.00(<4.58)^{n}
Virgin typha plant^{n}	S8K4-MS ^{n}	0.53(<4.58)^{n}	S8K2-MS ^{S}	S8K3-MS ^{S}	7.21(>4.58)^{S}
S8K1-MS^{n}	S8K2-MS ^{n}	2.80(<4.58)^{n}	S8K2-MS ^{n}	S10L3-MS ^{n}	3.43(<4.58)^{n}
S8K1-MS ^{n}	S8K3-MS ^{n}	4.41(<4.58)^{n}	S8K2-MS ^{n}	S9K3-MS ^{n}	3.47(<4.58)^{n}
S8K1-MS ^{n}	S10L3-MS ^{n}	0.63(<4.58)^{n}	S8K2-MS ^{n}	S8K4-MS ^{n}	1.87(<4.58)^{n}
S8K1-MS ^{n}	S9K3-MS ^{n}	0.67(<4.58)^{n}	S8K3-MS ^{n}	S10L3-MS ^{n}	3.77(<4.58)^{n}
S8K1-MS ^{n}	S8K4-MS ^{n}	0.93(<4.58)^{n}	S10L3-MS ^{n}	S9K3-MS ^{n}	0.03(<4.58)^{n}
S8K3-MS ^{n}	S9K3-MS ^{n}	3.74(<4.58^{n})	S10L3-MS ^{n}	S8K4-MS ^{n}	1.57(<4.58)^{n}
S8K3 -MS ^{S}	S8K4-MS ^{S}	5.34(>4.58)^{S}	S9K3-MS ^{n}	S8K4-MS ^{n}	1.60(<4.58)^{n}

^{n}No significant variation, ^{S}Significant variation, S: Sample; K: KOH, L: LOH,

Summary

The properties of textile are strongly affected by moisture regain. In addition, moisture regain will help to determine dimension, mechanical strength and elastic recovery and rigidity. The *Typha* plant and fibre absorbed moisture in different humidity atmosphere and provided various moisture regains, *Typha* plant was found 9.57% (average) and *Typha* fibre was found 10.90% (average). Statistical analysis said that moisture regain of *Typha* is similar with Cotton but different with Wool.

5.4 Thermal Properties

In textiles, when fibres are exposed to a heat source, some ignite and burn while others are non-combustible. Fibres that ignite but do not continue to burn, whether or not the ignition source is removed, are called flame resistant. Fibres that do not shrink or burn readily when heat is applied have a property known as thermal stability. The flammability of a textile may be used as a basis of selection for its use in different products. A thermal-resistant fabric has the ability to act as thermal insulator, (in garments) preventing a dangerous elevation of skin temperature. A fabric

also needs to be heat-resistant and heat durable to maintain properties during and following exposure to high temperature.

Depending on the end-use, a manufacturer can make thermal fabric suitable for firefighters, foundry workers, welders, race-car drivers and astronauts, who are all exposed to intense radiant heat, flame, and/or molten metal splashes in the work environment. Burning of small quantities of textile fibres may be used as a means of distinguishing one fibre group from another. Precise identification of individual fibres by burning is not usually possible; however, burning behaviour can readily establish the general fibre group to which the fibre belongs. Cellulosic fibres exhibit flammability characteristics much like those of paper; protein fibres burn in a manner similar to hair and some man-made fibres melt when they burn. Some fibres produce an odour when they burn, and the remaining ash residue can also assist to identify the fibre.

5.4.1 Burning behaviour of *Typha* fibre

The Burn-test response was carried out to observe how *Typha* fibres respond to flame. Two widely used fibres, Cotton and polyester, were used to compare the burning behaviour of *Typha* fibre. The burning behaviour of these three fibres is shown in Table 5.8.

Three different steps were evaluated during this test: when fibres approach a flame, when they are in the flame and when they are removed from the flame. When Cotton and *Typha* fibres approach flame, they do not shrink or fuse but produce white smoke and have a smell of paper or wood burning; whereas, polyester shrinks, fuses, produces black smoke and has a chemical and sweet smell. In the flame, Cotton and *Typha* rapidly burn, but polyester burns slowly. Polyester melts and does not continue to burn in the flame, while Cotton and *Typha* continues burning with an afterglow, but do not appear to melt in the flame. This result is given Table 5.8.

When polyester is removed from the flame, it extinguishes itself and leaves a hard residue of the fibre [Figure 5.15 (d)]. *Typha* and Cotton do not show self-extinguishing properties, however, but possess grey, feathery ash when removed from the flame [Figure 5.15 (e) and (f)]. From these foregoing tests, *Typha* mirrors Cotton, and could be used in the textile and apparel industry in similar applications to Cotton.

Table 5.8: Burning Behaviour of *Typha,* Polyester and Cotton fibres.

Fibre	**Approaching flame**			**In the flame**			**Removal from flame**	
	Shrink	**Fuses**	**Smoke /Smell**	**Rapidity**	**Melt**	**Continues to burn**	**Self-extinguishes**	**Residue**
Cotton	No	No	White smoke, Paper or Wood Burning smell	Rapidly	No	Yes, with afterglow	No	Grey, feathery ash
Polyester	Yes	Yes	Black smoke, Chemical, sweet smell	Slowly	Yes	No	Yes	Hard, black bead
Typha	No	No	White smoke, Paper or Wood Burning smell	Rapidly	No	Yes, with afterglow	No	Grey, feathery ash

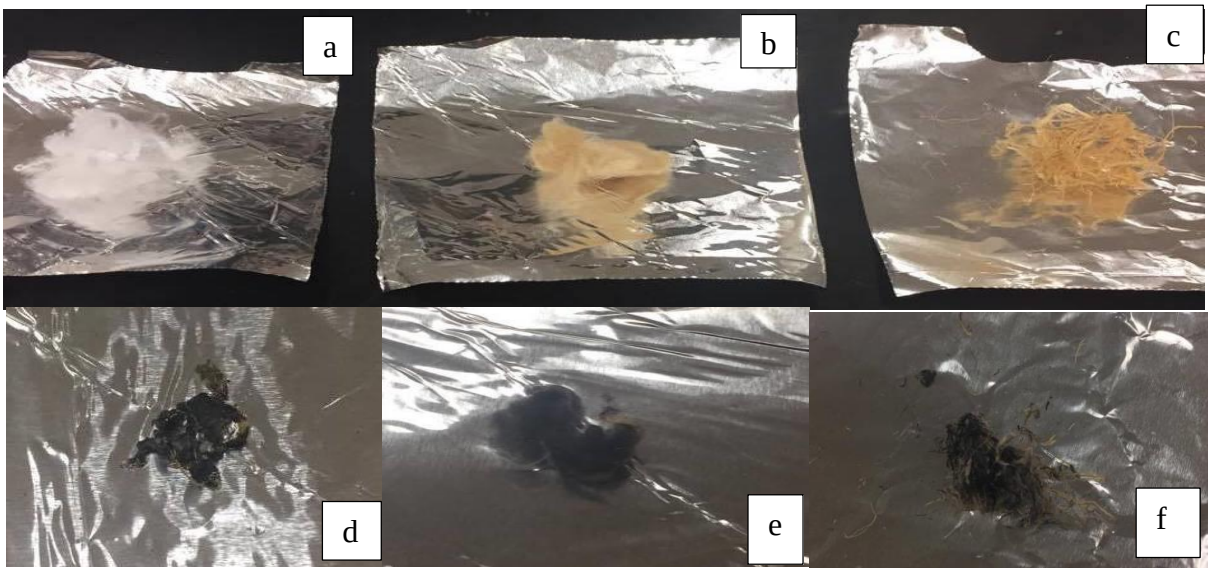


Figure 5.15: Original fibres before burning test: polyester (a), Cotton (b), *Typha latifolia* L. (c) and after burning test of polyester (d), Cotton (e) and Typha latifolia L.(f).

5.4.2 Evaluation of *Typha* fibre thermal properties

Fibre decomposition temperature is measured to determine the ability of a fibre to withstand dyeing or other high temperature manufacturing and laundering procedures. The thermal behaviour of *Typha* plant, *Typha* (S10L3-MS) fibre and Cotton fibre are presented in Table 5.9. In order to obtain the thermal decomposition point, the machine was set to run up to 300°C with no intermediate holding temperature or holding time. The decomposition point was recorded when the fibre turned dark brown or yellow colour as shown in Figures 5.16, 5.17, and 5.18 for *Typha* plant, *Typha* (S10L3-MS) fibre and Cotton fibre, respectively. The virgin *Typha* plant samples [Figures 5.16 (a)] that turn dark brown at 234.9°C [Figure 5.16 (b)] and chemically treated *Typha* (S10L3-MS) fibre [Figure 5.17 (a)] which turn dark brown at 268.7°C in Figure 5.17 (b) and likewise Cotton fibre Figure 5.18 (a) decomposed at 248.3°C shown in Figures 5.18 (b). The result indicates that the *Typha* (S10L3-MS) fibres are thermally stable up to 268.7°C, while the Cotton decomposed at 248.3°C. The lower decomposition temperature for *Typha* plant than the *Typha* (S10L3-MS) fibre may be due to the presence of lower amounts of non-cellulosic materials in the *Typha* fibre.

Table 5.9: Thermal properties of *Typha* plant, *Typha* fibre and Cotton fibre

Material type	**Decomposition temperature (°C)**	**Comments**	**Figure #**	
			Virgin sample	Decomposed sample
Typha latifolia L. Plant	234.9	Fibres turn dark brown	5.16(a)	5.16(b)
Typha fibre (S10L3-MS)	268.7	Fibres turn dark brown	5.17(a)	5.17(b)
Cotton fibre	248.3	Fibres turn dark brown	5.18(a)	5.18(b)

Figure 5.16(a): Virgin *Typha* plant.

Figure 5.16(b): Decomposed *Typha* plant.

Figure 5.17(a): Virgin *Typha* fibre.

Figure 5.17(b): Decomposed *Typha* fibre.

Figure 5.18(a): Virgin Cotton fibre.

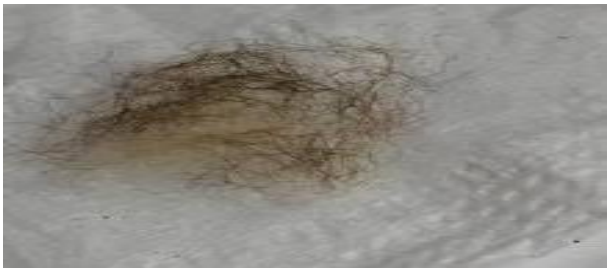


Figure 5.18(b): Decomposed Cotton fibre.

In textile applications, thermal stability is considered as an important parameter. For household textiles and many industrial applications, fibres are exposed to high temperature for long periods of time, such as curtains, rugs, tarpaulins and other products. To determine the thermal behaviour at different temperatures and holding times, seven individual samples of *Typha* (S10L3-MS) and Cotton fibres were prepared from a fibre bundle. For this test, the holding time used to evaluate changes of sample colour performance by colour software (Colour Hexa) at specific temperature and after a specific time. *Typha* (S10L3-MS) and Cotton sample images were imported to graphics software called paint application; using this application all images were

cropped measured by scale (gridline). Each image was cropped 4x4 (gridlines) and created image palette. These images are given in Appendix VI.

Then these image colour palettes exported into image software image colour picker (from https://www.colorcodepicker.com). The software gave HEX color code of the pixel value (for example TTF1 HEX value is #baad90) given in details correspondent pictures and HEX value are given in Appendix VIII. The original pictures Figure 5.19 (a) – 5.32(a) for 10 minutes holding time and Figure 3.19(b) – Figure 5.32(b) for 20 minutes holding time which are taken from Linkam are given in Appendix VIII. To convert HEX pixel value to Hunter-Lab L* value, by software called ColorHexa (from https://www.colorhexa.com). The L* value of colour descriptions was provided in Table 5.10(a). It is worth mentioning here that the lower the L* value, the darker the sample.

It can be seen from this Table 5.10(a) that with the increasing temperature the L value has been decreased for both Cotton and Typha fibres at both 10 and 20 minutes holding times. The changes in colour with the heating temperature and holding time are shown in Figures 5.19 (b) – 5.32 (b). These figures are given in Appendix VIII.

It is seen that for Typha fibre, increase of temperature from 120°C to 300°C with 30°C interval, the L value was decreased to 6.75 (Sample code: TTF7) from 65.07 (Sample code: TTF1) and 7.55 (Sample code: TTF7) from 45.99 (Sample code: TTF1) for 10 and 20 minutes holding time, respectively. Similar trend was observed for Cotton fibre 11.82 (Sample code: TGF7) from 60.70 (Sample code: TGF1) and 8.3 (Sample code: TGF7) from 47.03 (Sample code: TGF1). A graphpical presentation given in Figure 5.33.

5.4.2.1. Summary

Longer stability in heat is a sign of a good quality fibre that is suitable for textile applications. It was observed that *Typha* (S10L3-MS) fibres have heat resistance capacity, exceeding that of Cotton when heated directly from 0°C to 300°C without any holding time (Table 5.9). However, it appears that Cotton is more heat resistant than *Typha* fibre when both fibres are heated using holding times (Table 5.10(a)).

Table 5.10(a): Thermal properties of *Typha* and Cotton fibre with different temperatures range.

Sample type	Temperature Range (°C)	Colour changing effect							
		Holding Time: 10(min)				Holding Time: 20 (min)			
		HEX value	L* Value	Colour description	Figure	HEX value	L* Value	Colour description	Figure
TTF1	120	#baad90	65.07	GO	5.19 (a)	#a2854d	49.99	DMO	5.19 (b)
TTF2	150	#897e61	45.94	DDO	5.20 (a)	#8f703e	42.16	DMO	5.20 (b)
TTF3	180	#795c2c	34.50	DMO	5.21 (a)	#574126	24.39	VDDO	5.21 (b)
TTF4	210	#8c6d3b	41.02	DMO	5.22 (a)	#523c25	22.71	VDDO	5.22 (b)
TTF5	240	#331f12	13.14	VDO	5.23 (a)	#4f3a24	21.95	VDDO	5.23 (b)
TTF6	270	#2e1a0a	11.58	VDBO	5.24 (a)	#2b1b12	11.58	VDBO	5.24 (b)
TTF7	300	#130d0d	6.75	VDBR	5.25 (a)	#151011	7.55	VDBR	5.25 (b)
TGF1	120	#a4a49c	60.70	DGY	5.26 (a)	#81827d	47.03	DGY	5.26 (b)
TGF2	150	#a4a49b	60.67	DGY	5.27 (a)	#82847b	47.62	DGY	5.27 (b)
TGF3	180	#9c9c8e	57.27	DGY	5.28 (a)	#83847e	47.79	DGY	5.28 (b)
TGF4	210	#6c5d47	33.87	VDGO	5.29 (a)	#7c6a4c	38.89	MDDO	5.29 (b)
TGF5	240	#675a44	32.58	VDGO	5.30 (a)	#5e4229	25.37	VDDO	5.30 (b)
TGF6	270	#3c2c1d	16.89	VDDO	5.31 (a)	#36281e	15.48	VDDO	5.31 (b)
TGF7	300	#291d13	11.82	VDBO	5.32 (a)	#1e1310	8.83	VDBR	5.32 (b)

TTF: Temperatue of *Typha* Fibre; TCF: Temperature of Cotton Fibre; Figure 5.18 (a) – 5.31(a) and Figure 3.18(b) – Figure 5.31(b) shown in Appendix V; *GO: Grayish orange; *DDO: Dark desaturated orange; *DMO: Dark moderate orange; *VDO: Very dark orange; *VDBO: Very dark (mostly black) orange; *VDBR: Very dark (mostly black) red; *DGY: Dark grayish yellow; *VDGO: Very dark grayish orange*VDDO: Very dark desaturated orange; MDDO: Mostly desaturated dark orange.

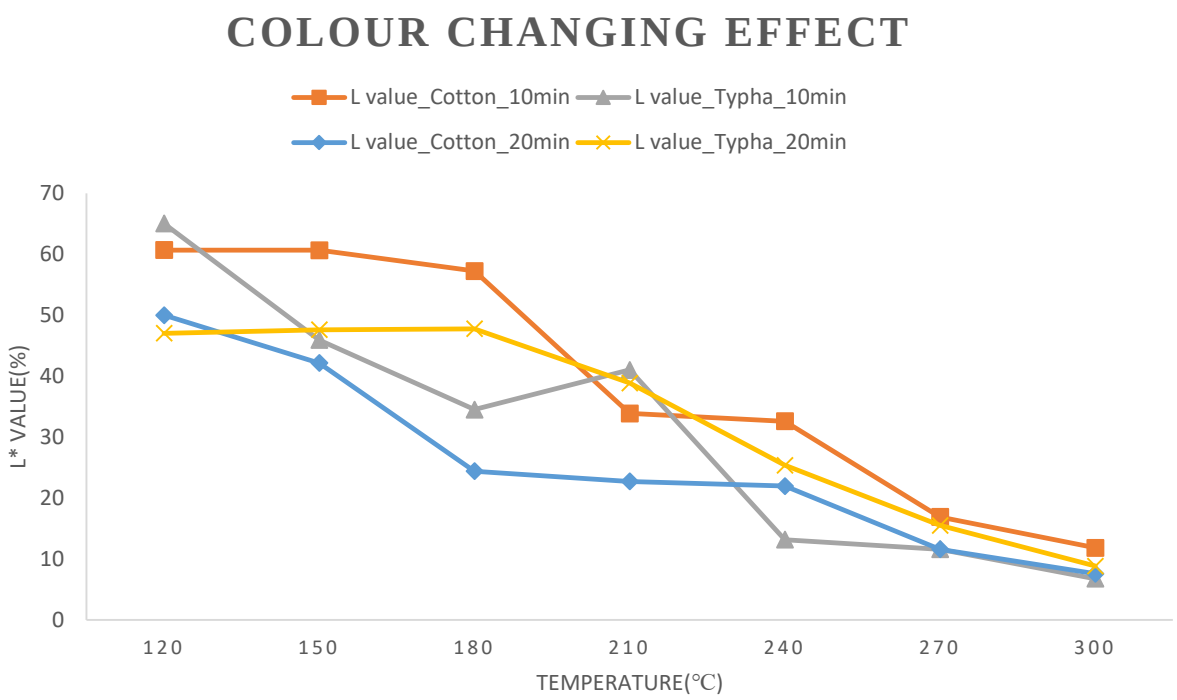


Figure 5.33: Changes in L* Value with temperature for Cotton and *Typha* fibres.

5.5 Dyeing of *Typha* fibres with reactive dye

Cellulosic fibres are dyed predominantly with reactive dyes in the presence of salt and in alkaline conditions. The dye fixation efficiency of reactive dyes on cellulosic fibres varies between 50-90% (Cai et al., 1999). As shown through the IR spectra (Section 5.2), *Typha*. fibres consist mainly of chemical groups related to cellulose.

Typha fibres from different extraction treatments were dyed, and then washed with HCl 50% (v/v) to determine the dye retention by the samples (shown in Figures 5.34 – 5.40). It can be seen that the fibres retain the reactive colour. For comparison, Cotton fibre was also dyed (Figure 5.34) with the same dye and dye bath parameters.

One of the objectives of dyeing is to produce a uniform colouration throughout a material. Visual observation showed no colour variation within the sample from various extracted fibre

samples; however, the colour of the samples was different for different samples as seen in Figures 5.34 to 5.40. The darkest blue colour was observed for sample S8-K2-MS/4h/80°C (Figure 5.36), while the lightest blue colour can be seen for S10-L1-MS/4h/80°C (Figure 5.39); a medium shade was obtained from sample S5-NaOH-MS/4h/80 °C (Figure 5.35). While the shade of the dyed Cotton was blue (Figure 5.34), some samples showed a greener tone (Figures 5.36, 5.37, 5.39 and 5.40). One of the reasons for these variations could be the non-uniform heat and higher temperature of the hot plate that was used for dyeing.

In order to check the effect of dye bath temperature on the resultant shade, dyeing was carried out for *Typha* (S8-K2-MS) fibres in four different temperatures using a launder-o-meter at 50°C, 60°C, 70°C and 80°C (shown in Figures 5.41 to 5.44). The dyeing results showed at 50°C (Figure 5.41) and 80°C (Figure 5.44) fibres turned blue colour tone, whereas, at 60°C (Figure 5.42), 80°C (Figure 5.43) fibres colour changed (faded) from blue colour to green colour tone. However, this result could be affected by dye baths (canisters) that had leaked out and the end of the dyeing the two canisters were almost empty. It was thought that the green tone had been produced because of the dry heat inside the canisters.

Figure 5.34: Reactive blue 4 treated Cotton fibre

Figure 5.35: Reactive blue 4 treated T*ypha* fibres. (Sample: S5-NaOH-MS/4h/80°C)

Figure 5.36: Reactive blue 4 treated Typha sample: S8-K2-MS/4h/80°C).

Figure 5.37: Reactive blue 4 treated T*ypha* fibre (Sample: S10-L2-MS/4h/80°C).

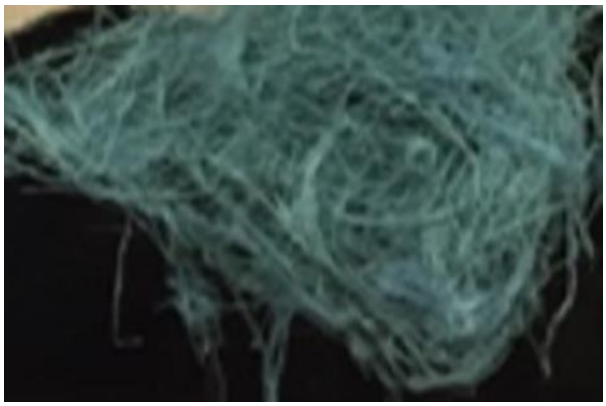


Figure 5.38: Reactive blue 4 treated *Typha* fibre (Sample: S8-K3-MS/ 6h/80°C).

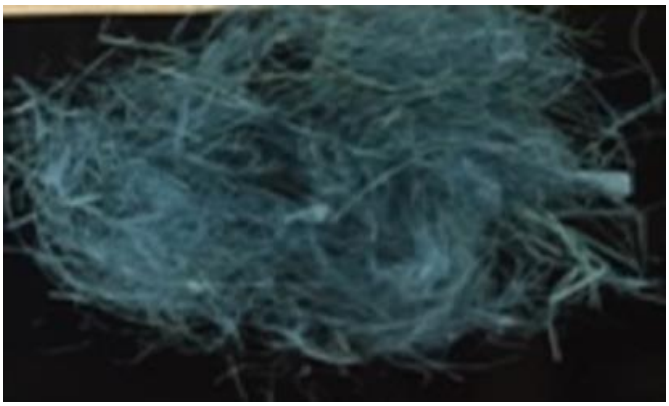


Figure 5.39: Reactive blue 4 treated *Typha* fibre (Sample: S10-L1-MS/2h/80°C).

Figure 5.40: Reactive blue 4 *Typha* fibre (Sample: S9-K5-MS/2h/95°C).

Figure 5.41: Dyed *Typha* (S8-K2-MS) fibres sample at 50°C.

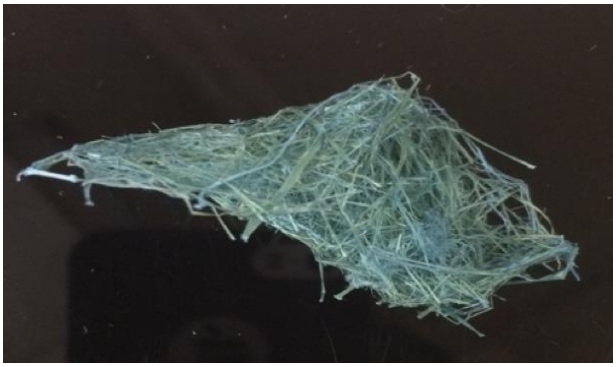


Figure 5.42: Dyed *Typha* (S8-K2-MS) fibres sample at 60°C.

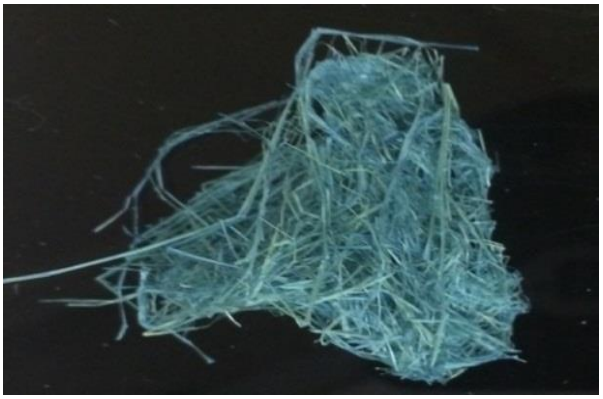


Figure 5.43: Dyed *Typha* (S8-K2-MS) fibres sample at 70°C.

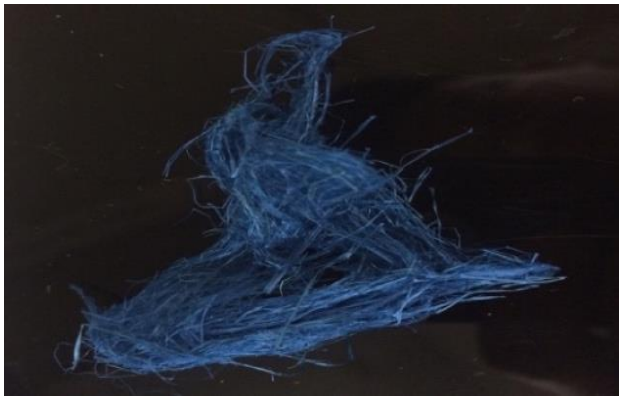


Figure 5.44: Dyed *Typha* (S8-K2-MS) fibres sample at 80°C.

Subsequently, another experiment was carried out using a hot plate to investigate the reason for shade variation during reactive blue dying for three beakers that were placed on a hot plate machine at 60°C. The uneven temperature was noticed between three beaker dye solutions in the hot plate (Figure 5.45). At 50°C and 60°C both temperature, the shade of blue colour was observed (Figures 5.46(a) and 5.46 (c)), however the temperature below 50°C the shade appeared greenish blue (Figure 5.46 (b)). The standard dye liquor value was recoded as -0.57, which indicates (green tone) in the colourimeter; it could be due to the possibility of a greenish tone in the reactive blue 4 dyed sample.

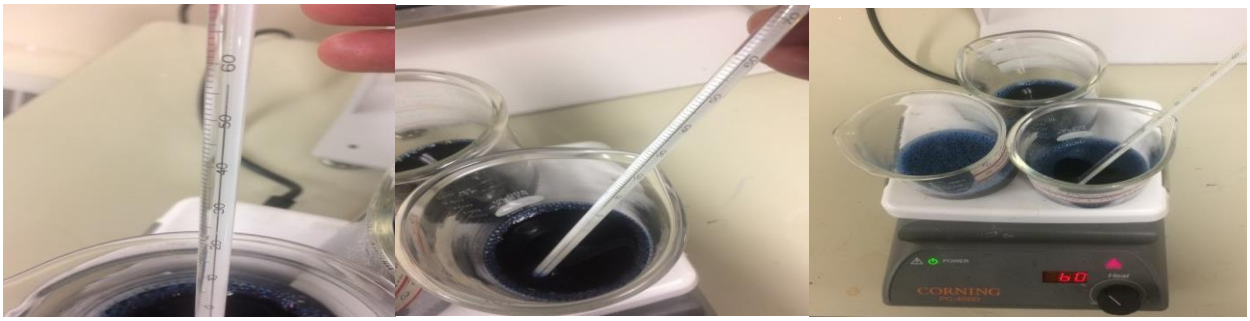


Figure 5.45: Temperatures variation on dye liquor solution in different place of the hot plate.

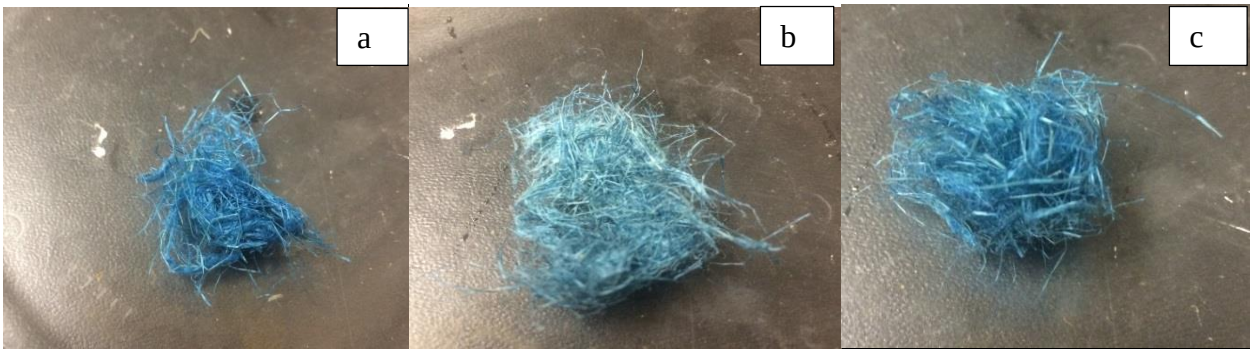


Figure 5.46: Shade variation of dyed *Typha* sample in hot plate test (a: at 60 °C) (b: less than 50 °C) and (c: 50 °C temperature).

5.5.1 Evaluation of colour fastness of dyed *Typha* fibres

The colourfastness of dyed textile materials is required to ensure that there is no colour transfer during washing, colour removal by light/heat/perspiration/ironing/pressing, or colour rubbing off by crocking/abrasion during end uses or on exposure to such conditions. For the current research, only colour fastness to hot pressing was conducted. In the hot-pressing colour fastness method, the dyed samples are placed between two hot plates and pressure is applied as outlined in Section 3.3.10 (Chapter 3). In this colourfastness test, three methods were adopted: dry, damp and wet pressing according to AATCC standard test method: 133-2009 (AATCC, 1985).

Before the colour fastness test, ten (10) evaluators were trained to evaluate the colour difference and staining. No colour changes or staining was obtained for the dry test and therefore, no data is provided. The colour changes and staining data from the ten evaluators for damp and

wet tests are provided in Tables 5.11 (a) and 5.12 (a). The colour change of the dyed *Typha* fibre to the wet undyed Cotton fabric was evaluated because water often is sprayed onto fabric during ironing/pressing. For ease of identification, a two-character sample ID is given with the existing sample ID for each data point which is provided in the parenthesis (Table 5.11(a)). For example, existing sample ID S10-L2-MS is given 1.A as identifier and the wet undyed Cotton fabric at 110 °C/15 second is identified as 1.B [Table 5.11(a)].

In damp pressing, the lowest rating obtained was between 3.18 (Sample: 3.A, data: 3.C) among other samples at 110°C. All dry undyed Cotton samples stained less than the wet undyed Cotton samples. At 110°C, the rating of the difference of staining between dry undyed Cotton fabrics and wet undyed Cotton fabric was between 0.6 (Sample: 3.A) and 0.10 (Sample: 2.A). At 150°C, the largest difference, 0.35, obtained for sample 1.A and the smallest difference, 0.05, found for sample 7.A. The difference was almost similar at 200 °C: 0.3 for sample 6.A and 0.04 for sample 5.A. The rating of colour change for the damp test was lower than the staining rating for all the samples in all three temperatures. For example, for sample S10-L2-MS (1.A), the staining rating is 4.7 in wet undyed Cotton fabric; whereas, the colour change was 4.05 [Table 5.11(a)]. Further, it was noticed that the change in colour for four samples (1.G, 2.G, 6.G and 7.G) was higher (lower rating) when temperature was raised from 110°C to 200°C. For the other three samples (3.G, 4.G and 5.G), the rating was higher. It is worth mentioning here that Cotton fibre and *Typha* fibre behave almost the same: the colour change rating was found 4.4 at 110 °C/15 second (7.C); 4.49 at 150°C/15 second (7.E) and 4.19 at 200°C/15 second (7.G).

For the wet pressing test, the staining rating of wet undyed Cotton fabric was lower than the dry undyed Cotton fabric for all samples and all temperatures as seen Table 5.12 (a). Further,

the wet staining rating is lower at 150 °C and 200 °C for all *Typha* fibres than the wet staining at 110 °C, and the changes in colour are higher at 150 °C and 200 °C than 110 °C.

Two-way ANOVA was conducted for both damp pressing and wet pressing samples in order to identify the difference of colour fastness and staining among the fibre samples. The dependent variables for this analysis were two staining conditions (Dry undyed Cotton fabric for staining and wet undyed Cotton fabric for staining) and one colour change condition (Rating for colour change). The comparison was made for all three treatment temperatures: 110°C/15 sec, 150°C/15 sec and 200°C/15 sec and interactions among treatment conditions (dependent variables) and temperatures.

Table 5.11(a): Colour fastness rating in hot press method (damp pressing) for *Typha* fibres.

Damp pressing temperature (°C) / time (second)									
	110 /15			150/15			200/15		
Sample ID	**Rating for staining**		**Rating for CC**	**Rating for staining**		**Rating for CC**	**Rating for staining**		**Rating for CC**
	Dry undyed Cotton fabric	**Wet undyed Cotton fabric**		**Dry undyed Cotton fabric**	**Wet undyed Cotton fabric**		**Dry undyed Cotton fabric**	**Wet undyed Cotton fabric**	
S10-L2-MS (1.A)	4.95 ±0.15	4.7±0.25 (1.B)	4.05±0.43 (1.C)	4.95 ±0.15	4.6±0.35 (1.D)	3.94±0.60 (1.E)	4.95 ±0.15	4.85±0.33 (1.F)	4.18±0.45 (1.G)
S10-L1-MS (2.A)	5.0 ±0.0	4.9±0.21 (2.B)	4.35±0.41 (2.C)	4.95 ±0.15	4.85±0.33 (2.D)	3.74±1.17 (2.E)	4.9 ±0.21	4.7±0.42 (2.F)	3.75±0.40 (2.G)
S8-K3-MS (3.A)	5.0 ±0.0	4.4±0.31 (3.B)	3.18±0.60 (3.C)	4.95 ±0.15	4.6±0.40 (3.D)	4.08±0.42 (3.E)	5.0 ±0.0	4.95±0.15 (3.F)	3.95±0.68 (3.G)
S9-K5-MS (4.A)	4.85 ±0.24	4.4±0.31 (4.B)	3.54±0.80 (4.C)	4.95 ±0.15	4.7±0.34 (4.D)	4.25±0.26 (4.E)	4.95 ±0.15	4.7± 0.34 (4.F)	3.67±0.95 (4.G)
S8-K2-MS (5.A)	5.0 ±0.0	4.6±0.31 (5.B)	3.44±0.80 (5.C)	4.95 ±0.15	4.8±0.25 (5.D)	4.75±0.26 (5.E)	4.94 ±0.15	4.9±0.21 (5.F)	4.13±0.69 (5.G)
S5-NaOH-MS (6.A)	4.95 ±0.15	4.7±0.34 (6.B)	4.19±0.37 (6.C)	4.95 ±0.15	4.7±0.34 (6.D)	3.89±0.62 (6.E)	5.0 ±0	4.7±0.26 (6.F)	3.75±0.84 (6.G)
Cotton (7.A)	5.0 ±0.0	4.8±0.34 (7.B)	4.4±0.39 (7.C)	4.9 ±0.21	4.85±0.34 (7.D)	4.49±0.60 (7.E)	4.95 ±0.15	4.9±0.21 (7.F)	4.19±0.92 (7.G)

Table 5.12(a): Colour fastness rating in hot press method (wet pressing) for *Typha* fibres.

Wet pressing temperature (°C) / time (seconds)									
	110 /15			150/15			200/15		
Sample ID	**Rating for staining**		**Rating for CC**	**Rating for staining**		**Rating for CC**	**Rating for staining**		**Rating for CC**
	Dry undyed Cotton fabric	**Wet undyed Cotton fabric**		**Dry undyed Cotton fabric**	**Wet undyed Cotton fabric**		**Dry undyed Cotton fabric**	**Wet undyed Cotton fabric**	
S10-L2-MS (1.A)	4.75±0.26	4.02±0.48 (1.H)	3.64±1.14 (1.I)	4.5±0.23	4.03±0.47 (1.J)	4.01±0.95 (1.K)	4.65±0.34	3.85±0.89 (1.L)	3.59±0.74 (1.M)
S10-L1-MS (2.A)	4.5±0.23	3.75±0.40 (2.H)	4.5±0.62 (2.I)	4.08±0.71	3.04±0.99 (2.J)	4.33±0.62 (2.K)	4.39±0.48	3.87±0.75 (2.L)	3.97±0.96 (2.M)
S8-K3-MS (3.A)	5±0	4.29±0.37 (3.H)	4.44±0.51 (3.I)	4.45±0.37	3.25±0.87 (3.J)	3.46±0.93 (3.K)	4.39±0.48	3.78±0.72 (3.L)	3.94±0.65 (3.M)
S9-K5-MS (4.A)	4.95±0.15	4.14±0.36 (4.H)	4.15±0.67 (4.I)	4.9±0.21	4.28±0.95 (4.J)	4.28±0.57 (4.K)	4.6±0.31	3.93±0.61 (4.L)	3.72±0.56 (4.M)
S8-K2-MS (5.A)	4.49±0.43	3.81±0.38 (5.H)	4.5±0.40 (5.I)	3.88±0.63	3.08±0.83 (5.J)	3.5±0.92 (5.K)	4.5±0.23	3.51±0.60 (5.L)	4.09±0.70 (5.M)
S5-NaOH-MS (6.A)	4.8±0.26	3.91±0.70 (6.H)	3.68±0.86 (6.I)	4.59±0.54	3.4±0.80 (6.J)	3.95±0.81 (6.K)	4.23±0.52	3.34±0.81 (6.L)	3.76±0.72 (6.M)
Cotton (7.A)	5±0	3.87±0.54 (7.H)	4.65±0.41 (7.I)	4.8±0.25	3.62±0.60 (7.J)	4.14±0.60 (7.K)	4.75±0.35	4.24±0.44 (7.L)	3.77±0.83 (7.M)

Table 5.11(b): Two-way ANOVA results tests on the effect of temperature and treatments on colour fastness for damp pressing.

	Independent Variable (Temperature)			Independent variable (Treatment)		
Dependent Variable	Temperature	Mean	Std. Error	Treatment	Mean	Std. Error
Dry undyed Cotton fabric	110°C	4.784	.046	S8-K2	4.290	0.070
	150°C	4.464	.045	S8-K3	4.630	0.070
	200°C	4.501	.046	S10-L1	4.323	0.070
				S10-L2	4.633	0.070
				S9-K5	4.817	0.070
				S5-NaOH	4.540	0.070
				Cotton	4.850	0.070
Wet undyed Cotton fabric	110°C	3.967	.081	S8-K2	3.467	0.123
	150°C	3.545	.080	S8-K3	3.803	0.124
	200°C	3.789	.081	S10-L1	3.553	0.123
				S10-L2	3.967	0.123
				S9-K5	4.117	0.123
				S5-NaOH	3.550	0.123
				Cotton	3.910	0.123
Rating for staining	110°C	4.230	.090	S8-K2	4.030	0.137
	150°C	3.960	.089	S8-K3	3.979	0.137
	200°C	3.834	.090	S10-L1	4.267	0.137
				S10-L2	3.747	0.137
				S9-K5	4.050	0.137
				S5-NaOH	3.797	0.137
				Cotton	4.187	0.137

Treatments	Dry undyed Cotton fabric for staining[n]	$F_{calculated}$ (0.634)[n]	P=(0.703 > α =0.05)[n]
	Wet undyed Cotton fabric for staining[s]	$F_{calculated}$ (2.388)[s]	P=(0.030 < α =0.05)[s]
	Rating for colour change [s]	$F_{calculated}$ (3.088) [s]	P=(0.007 < α =0.05)[s]
Temperature	Dry undyed Cotton fabric for staining[n]	$F_{calculated}$(0.367) [n]	P=(0.693 > α =0.05)[n]
	Wet undyed Cotton fabric for staining[s]	$F_{calculated}$ (5.184) [s]	P=(0.006 < α =0.05)[s]
	Rating for colour change [s]	$F_{calculated}$ (3.184) [s]	P=(0.044 < α =0.05)[s]
Treatment * Temperature	Dry undyed Cotton fabric for staining[n]	$F_{calculated}$(0.776) [n]	P=(0.675> α =0.05)[n]
	Wet undyed Cotton fabric for staining[s]	$F_{calculated}$ (1.585)[s]	P=(0.099< α =0.05)[s]
	Rating for colour change[s]	$F_{calculated}$ (3.237)[s]	P=(0.0005< α =0.05)[s]

s: Significant, n: Not Significant,

Table 5.12(b): Two-way ANOVA results tests on the effect of temperature and treatments on colour fastness for wet pressing.

		Independent Variable (Temperature)			Independent variable (Treatment)		
Dependent Variable		Temperature	Mean	Std. Error	Treatment	Mean	Std. Error
Dry undyed Cotton fabric		110°C	4.784	.046	S8-K2	4.290	0.070
		150°C	4.464	.045	S8-K3	4.630	0.070
		200°C	4.501	.046	S10-L1	4.323	0.070
					S10-L2	4.633	0.070
					S9K5	4.817	0.070
					S5-NaOH	4.540	0.070
					Cotton	4.850	0.070
Wet undyed Cotton fabric		110°C	3.967	.081	S8-K2	3.467	0.123
		150°C	3.545	.080	S8-K3	3.803	0.124
		200°C	3.789	.081	S10-L1	3.553	0.123
					S10-L2	3.967	0.123
					S9K5	4.117	0.123
					S5-NaOH	3.550	0.123
					Cotton	3.910	0.123
Rating for staining		110°C	4.230	.090	S8-K2	4.030	0.137
		150°C	3.960	.089	S8-K3	3.979	0.137
		200°C	3.834	.090	S10-L1	4.267	0.137
					S10-L2	3.747	0.137
					S9K5	4.050	0.137
					S5-NaOH	3.797	0.137
					Cotton	4.187	0.137
Treatments	Dry undyed Cotton fabric for staining^{s}				$F_{calculated}$ (9.780)^{s}	P=(0.0005 > α =0.05)^{s}	
	Wet undyed Cotton fabric for staining^{s}				$F_{calculated}$ (4.030)^{s}	P=(0.001 < α =0.05)^{s}	
	Rating for colour change^{s}				$F_{calculated}$ (1.915)^{s}	P=(0.080 > α =0.05)^{s}	
Temperature	Dry undyed Cotton fabric for staining^{s}				$F_{calculated}$(14.47)^{s}	P=(0.0005 > α =0.05)^{s}	
	Wet undyed Cotton fabric for staining^{s}				$F_{calculated}$ (6.899) ^{s}	P=(0.001 < α =0.05)^{s}	
	Rating for colour change ^{s}				$F_{calculated}$ (5.051) ^{s}	P=(0.007 < α =0.05)^{s}	
Treatment * Temperature	Dry undyed Cotton fabric for staining^{s}				$F_{calculated}$(2.460) ^{s}	P=(0.05 < α =0.05)^{s}	
	Wet undyed Cotton fabric for staining^{n}				$F_{calculated}$ (1.689)^{n}	P=(0.072 > α =0.05)^{n}	
	Rating for colour change^{s}				$F_{calculated}$ (1.895)^{s}	P=(0.037< α =0.05)^{s}	

s: Significant, n: Not Significant,

Table 5.13: Results of Post Hoc tests for the effect of hot pressing to colour fastness.

Damp hot pressing			Wet hot pressing		
	Post Hoc multi comparison	**Sig. D**		**Post Hoc multi comparison**	**Sig. D**
Dry undyed Cotton fabric (staining)	N/A	N/A	Dry undyed Cotton fabric for stating	S8-K2 vs. S8-K3 S8-K2 vs. S10-L2 S8-K2- vs. S9-K5 S8-K2 vs. Cotton S8-K3 vs. S8-K2 S10-L1 vs. S10-L2	(p= .02) (p= .011) (p= .000) (p= .000) (p= .021) (p= .032)
Wet undyed Cotton fabric (staining)	S9K5 vs. Cotton	(p=.039)	Wet undyed Cotton fabric	S8-K2 vs. S9K5 S10L1 vs. S9K5 S9K5 vs. S5-NaOH	(p=.005) (p=.024) (p=.023)
Rating for colour change	S8-K3 vs. Cotton S9K5 vs. Cotton	(p=.005) (p=.027)	Rating for colour change	N/A	N/A

Two-way multivariate ANOVA was conducted for each dependent variable. The results showed. in Table (5.11(b)), that treatment and temperature during damp testing, for wet undyed Cotton fabric staining and colour change ratings are significantly different with $P=0.030<\alpha=0.05$ and $p=0.007<\alpha=0.05$ for treatments and $P=0.030<\alpha=0.05$ and $P=0.007<\alpha=0.05$), respectively. For dry undyed Cotton fabric for staining, the treatment and temperature effects are not significant (treatment: $p=0.703>\alpha=0.05$; temperature: $P=0.693>\alpha=0.05$). Similar results were obtained for interaction between treatment*temperature (Table 5.12(b)).

Post-hoc comparisons using the Tukey HSD test, given in Table 5.13, indicated that there was a significant pairwise difference of mean temperature for damp hot pressing wet undyed Cotton fabric (staining) for Cotton and S9K5 ($p < .039$), while for colour change the significant pairwise differences were obtained for S8-K3 and Cotton ($p < .005$), and S9K5 and Cotton ($p < .027$),

During wet pressing test, no significant difference was obtained for colour change. However, for staining, it was noticed that dependant variable dry undyed Cotton fabric for staining was shown

significant differences between S8-K2 vs. S8-K3 (p= .02), S8-K2 vs. S10-L2 (p= .011), S8-K2-vs. S9-K5 (p= .000), S8-K3 vs. S8-K2 (p= .021), and S10-L1 vs. S10-L2 (p= .032). For wet undyed Cotton fabric had only three significant differences which were S8-K2 vs. S9K5 (p=.005), S10L1 vs. S9K5 (p=.024), S9K5 vs. S5-NaOH (p=.023).

5.5.1.1 Summary

Dyeing is a modification process which is an essential part of apparel that gives an identity and exclusive appearance to a material. *Typha* fibre has good dye absorbency due to their crenelated structure encapsulate reactive dye paticles that assist increase the dye absorbency. *Typha* fibre (S9-K5-MS) dye absorption (%) before the washing treatment was 92.54 and dye exhaustion (%) was 79.29. The standared rating for colour fastness to hot pressing is 3 to 4.8 which is acceptable in apperal performance specification standards. Statistical results conclude that there were no significant differences obtained for Dry undyed Cotton fabric detaining during damp test, which is expected due to the state of the test fabric. The differences in colourfastness ratings (both colour change and staining) were observed between Cotton and *Typha* samples and among *Typha* samples.

5.5.2 Colour differences of dyed *Typha* fibre

It was difficult to determine how much dye was absorbed by each sample. The colour measuring machine is able to measure only the colour of fabric samples. Therefore, in order to measure the absorbed dye, a method has been adopted and the results are given in Table 5.14. In this method, at first, the – (ve) b value (blue colour) of the control solution was measured as18.79. Then, after the completion of dyeing of each sample, the – (ve) b value of the leftover solution was measured. This value was different for different samples. For example, the – (ve) b value for sample S8-K2-MS was 1.81 and for Cotton, this value was 1.91 in Table 5.14. The dye absorbency was found for sample S8-K2-MS 90.3% and Cotton was 89.83%. The – (ve) of absorbed dye in

the sample S8-K2-MS was determined by subtracting the – (ve) b value of leftover dye solution for sample S8-K2-MS (1.81) from the – (ve) b value of control sample (18.79) leftover dye solution is (- 16.98). The percent of absorbed dye was calculated by – (ve) b value dye absorbed by sample S8-K2-MS of *100/-(ve) b value of control dye solution.

In order to calculate the dye exhaustion, the dyed sample was washed in hydrochloric acid for 15 minutes at 60 °C in 100 ml water. Then the – (ve) b value of this solution was determined as 2.42 for S8-K2-MS. This value (2.42) is subtracted from the – (ve) b value for the dyed sample before washing (16.98 - 2.42) which gives the – (ve) b value for absorbed dye in the washed fibre sample S8-K2-MS which is 14.56. Finally, the dye exhaustion (%) of S8-K2-MS was found 77.5% calculated by – (ve) b value for absorbed dye in the washed * 100 / – (ve) b value of control sample. The dye exhaustion (%) for all *Typha* samples together with Cotton is calculated and given in Table 5.15. It can be seen from this table that the dye exhaustion (%) for *Typha* fibres lies between 72% and 79%, while the value for Cotton is 74.5%. It is known that the exhaustion (%) of reactive dye by Cotton is between 50-90% (Cai et al., 1999; Hamdaoui et al., 2013; Acharya et al., 2014).

Table 5.14: T*ypha* fibres colour differences.

Sample Type (Dye liquor)	**After Dyeing Colorimetric values**				**After treatment Colorimetric values**				**Difference** ΔE*
	L*	Δa*	Δb*	ΔE*	L*	Δa*	Δb*	ΔE*	
Control	65	-0.57	-18.79	67.70					
S10-L1-MS	92.36	1.04	-1.55	92.37	91.75	-1.13	-2.38	91.78	0.59
S10-L2-MS	91.85	-1.13	-2.14	91.88	88.53	0.18	-3.08	88.58	3.3
S8-K2-MS	91.67	0.91	-1.81	91.69	92.79	0.88	-2.42	92.82	-1.13
S8-K3-MS	92.31	1.0	-1.43	92.30	91.55	0.61	-2.49	91.58	0.72
S9-K5-MS	93.35	1.03	-1.40	93.36	92.67	0.87	-2.49	92.70	0.66
S5-NaOH-MS	93.33	1.13	-1.44	93.34	91.88	0.72	-2.56	91.91	1.43
Cotton	92.08	1.0	-1.91	92.10	92.47	0.80	-2.89	92.51	0.41

L = lightness of colour (0 = black, 100 = white), a = +ve value indicates red and –ve value indicated green b = +ve value indicates yellow and –ve value indicates blue.

Table 5.15: Dye Exhaustion by *Typha* fibre.

Sample ID	(-ve) b value of left over dye solution	(-ve) b value of dyed fibre	Dye absorb (%) by sample	(-ve) b value of dye solution from washed fibre	(-ve) b value of dyed fibre after washing	Exhaustion (%) after washing treatment
Control	18.79	0.00	0.00	0.00	0.00	0.00
S10-L1-MS	1.55	17.24	91.75	2.38	14.86	79.08
S10-L2-MS	2.14	16.65	88.61	3.08	13.57	72.21
S8-K2-MS	1.81	16.98	90.3	2.42	14.56	77.5
S8-K3-MS	1.43	17.36	92.38	2.49	14.87	79.13
S9-K5-MS	1.40	17.39	92.54	2.49	14.90	79.29
S5-NaOH-MS	1.44	17.35	92.33	2.56	14.79	78.71
Cotton	1.91	16.88	89.83	2.89	13.99	74.45

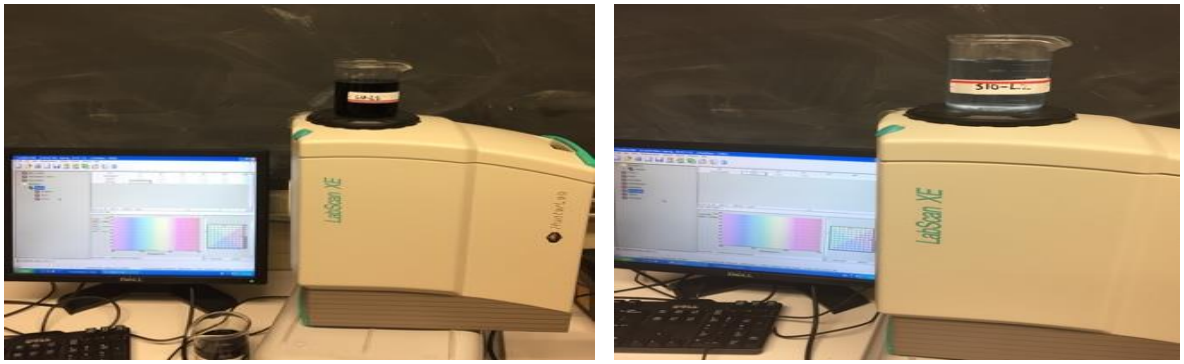


Figure 5.47: Dye liquor evaluation by Hunter lab for sample S10-L2-MS (Before dyeing and after washing).

5.5.2.1 Summary

The acceptable range of dye fixation is 50 - 90 (%) for cellulose fibre in reactive dyeing (Cai et al., 1999). It was found that the *Typha* fibre dye absorbency before the washing treatment was above 85% and after the washing treatment was around 70%, which is well within the acceptable range. Taking these dye uptake and colour fastness properties into account, it can be concluded that *Typha* fibre can be used in textile application.

5.6 Hand Properties for *Typha* fibres

The oldest and most common process of transforming fibres into yarn is ring spinning. Twist is inserted as the fibres leave the drafting unit. Fibre softness influences not only the fibre mechanical properties such as coefficient of fabric surface friction, shearing properties, bending, and draping properties, but also the yarn structure (twist). The finer the fibres, the greater the twist of the yarn (Chattopadhyay & Sinha, 2007). A soft hand (feel of the fabric) is a desirable characteristic of fabrics used in the apparel industry as it is the fabric feel which can influence a customer's purchase decision.

The hand property (softness) was measured using a standard AATCC evaluation procedure 5 (AATCC, 2010). In this method softness is evaluated using a rating scale of 1 (Least Soft) to 5 (Softest) and the evaluators were asked to evaluate based on compression, bending and surface. The details of the evaluation method are given in Section 3.3.3. This rating may be used whether or not the fibre can be processed in Cotton spinning machinery for yarn transformation.

At the beginning of the evaluation, evaluators were given eight standard fibre samples: Cotton, wool, nylon, acrylic, silk, polyester, cannabis (virgin), and brassica (virgin). They were then asked to rate the samples from 1 to 5 by touching, feeling, pressing and bending using thumb and fourth finger. Once the evaluators had evaluated the standard samples, they were provided with the four *Typha* samples: *Typha* leaf plant and three *Typha* fibre samples obtained by NaOH treatment (S5-NaOH-MS), KOH treatment (S8-K3-MS) and LiOH treatment (S10-L3-MS). The results of rating for standard and *Typha* samples are shown in Table 5.16(a).

It can be seen from Table 5.16(a) that silk fibre obtained the highest rating (4.8) while polyester's rating was 4.6. The ratings for Cotton, acrylic, wool and nylon were 4.0, 3.8, 3.0 and 2.0 respectively. The rating for virgin cannabis and virgin brassica were 1.0, and physical

examination showed that these two samples were very stiff. While the ranking was consistent for the seven standard samples (the standard deviation ranged between 0.44 and 0.89), the rating for acrylic fibre varied widely with a standard deviation of 0.81.

Among the *Typha* samples, the rating for *Typha* leaf plant was 1.0 while the ratings for NaOH, KOH and LiOH treated fibre were 1.8, 2.8 and 1.6 respectively. All three ratings for *Typha* fibres were lower than widely used textile fibres Cotton, wool, and polyester, however, the rating of 2.8 (S8-K3-MS) was higher than nylon. A statistical analysis was employed to establish whether these all fibres have significant difference in Table 5.16(a).

Table 5.16(a): *Typha* fibre softness grading with other common fibres.

Fibre Type	Mean	Std. Deviation	Std. Error
Cotton	4.00	.000	.000
Wool	3.00	.000	.000
Nylon	2.00	.000	.000
Acrylic	3.80	.837	.374
Silk	4.80	.447	.200
Polyester	4.60	.548	.245
Cannabis (virgin)	1.00	.000	.000
Brassica (virgin)	1.00	.000	.000
Typha leaf (virgin)	1.00	.000	.000
Typha Fibre(S5-NaOH-MS)	1.80	.447	.200
Typha Fibre (S8-K3-MS)	2.80	.837	.374
Typha Fibre (S10-L3-MS)	1.60	.894	.400
$F_{calculated}$	40.18		
P-value	0.0005< (α =0.05)		
Partial Eta squared (η)	0.902		
Sample size (N)	5		

A one-way between groups ANOVA was performed to compare the impact different types of fibre had on hand feel properties rating. Twelve selected fibre with rating observations were taken from observers (N=5) (in Appendix IX). The actual difference in the mean softness rating

between fibres was big effect size, (η =.902). There was a statistically difference between groups as determined by one-way ANOVA (F (11, 59) = 40.176, p=.0005) in Table 5.23(a). A tukey post hoc test revealed in pyramid shape that fibre hand feel properties were measured statistically significance among twelve fibres shown in Table 5.16(b).

Cotton versus Typha hand feel properties exhibited S5-NaOH-MS (.0005<p), S10-L3-MS (.0005<p) are statistically significant and S8-K3-MS (0.16<p) is not significant. Whereas Wool vs. *Typha* showed S10-L3-MS, (.0005<p) was statistically significant, however, S8-K3-MS (1.0<p) and S5-NaOH-MS, (0.16>p) were not significant. In Nylon vs. Typha no significant difference was observed such as S5-NaOH-MS (1.0>p), S8-K3-MS (.323 >p) and S10-L3-MS (.977>p). When comparing with *Typha* and Acrylic fibre, results showed a similar trend to Cotton; for example S5-NaOH-MS (.0005<p) and S10-L3-MS (.0005<p) were significantly different in their hand feel characteristics but S8-K3-MS (.086 >p) did not show any significant difference. The evidence to reject the null hypothesis and conclude that all three *Typha* fibres were statistically significant compared to polyester (S5-NaOH-MS (.0005 <p), S8-K3-MS (.0005<p), S10-L3-MS (.0005<p) and silk (S5-NaOH-MS (.0005<p), S8-K3-MS (.0005<p), S10-L3-MS (.0005<p), respectively. Virgin fibres including Cannabis, Brassica and Typha leaf plant possessed homogeneous and have almost similar softness properties for S5-NaOH-MS (0.323 >p) and S10-L3-M (0.735>p) were not significant, however, only S8-K3-MS (.0005<p) was significant (Table 5.16 (b)).

Table 5.16(b): Multi-comparisons using Tukey post hoc test results for the effect of hand feel properties on common fibres in pyramid shape.

				S8-K3-MS) vs. S10-L3-MS (.016 >p)						
				S5-NaOH-MS vs. S8-K3-MS (.086 >p)	S5-NaOH-MS vs. S10-L3-MS (1.0 >p)					
				Typha leaf plant (v) (v) vs.S5-NaOH-MS (.323>p)	*Typha* leaf plant (v) vs.S8-K3-MS (.0005<p)	*Typha* leaf plant (v) vs.S10-L3-MS (.735 >p)				
			Brassica (v) vs. Typha Fibre (v) (1.0 >p)	Brassica (v) vs. S5-NaOH-MS (.323 >p)	Brassica (v) vs. S8-K3-MS (.0005 <p)	Brassica (v) vs. S10-L3-MS (.735 >p)				
		Cannabis (v) vs Brassica (v) (1.0 >p)	Cannabis (v) vs *Typha* Fibre (v) (1.0 >p)	Cannabis (v) vs S5-NaOH-MS (.323 >p)	Cannabis (v) vs S8-K3-MS (.0005 < p)	Cannabis (v) vs S10-L3-MS (.735 >p)				
	Polyester vs. Cannabis (v) (.0005 <p)	Polyester vs. Brassica (v) (.0005 <p)	Polyester vs. Typha leaf plant (v) (.0005 <p)	Polyester vs. S5-NaOH-MS (.0005 <p)	Polyester vs. S8-K3-MS (.0005<p)	Polyester vs. S10-L3-MS (.0005 <p)				
	Silk vs. Polyester (.977 >p)	Silk vs. Cannabis (v) (.0005 <p)	Silk vs. Brassica (v) (.0005 <p)	Silk vs. *Typ*ha leaf plant (v) (.0005 <p)	Silk vs. S5-NaOH-MS (.0005 <p)	Silk vs. S8-K3-MS (.0005 <p)	Silk vs. S10-L3-MS (.0005 <p)			
	Acrylic vs. Silk (.735 >p)	Acrylic vs. Polyester (.086 >p)	Acrylic vs. Cannabis (virgin) (.0005 <p)	Acrylic vs. Brassica (virgin) (.0005 <p)	Acrylic vs. Typha leaf *plant* (v) (.0005 <p)	Acrylic vs. S5-NaOH-MS (.0005 <p)	Acrylic vs. S8-K3-MS (.086 >p)	Acrylic vs. S10-L3-MS (.0005 <p)		
	Nylon vs. Acrylic (.0005<p)	Nylon vs. Silk (.0005<p)	Nylon vs. Polyester (.0005<p)	Nylon vs. Cannabis (v) (.086 >p)	Nylon vs. Brassica (v) (.086 >p)	Nylon vs. Typha Fibre (v) (.086 >p)	Nylon vs. S5-NaOH-MS (1.0>p)	Nylon vs. S8-K3-MS (.323 >p)	Nylon vs. S10-L3-MS (.977 >p)	
	Wool vs. Nylon (.086>p)	Wool vs. Acrylic (.323>p)	Wool vs. Silk (.002<p)	Wool vs. Polyester (.0005<p)	Wool vs. Cannabis (v) (.0005<p)	Wool vs. Brassica (v) (.0005<p)	Wool vs. Typha leaf *plant* (v) (.0005<p)	Wool vs. S5-NaOH-MS (0.16>p)	Wool vs. S8-K3-MS (1.0>p)	Wool vs. S10-L3-MS (.0005<p)
Cotton vs. Wool (.086 > p)	Cotton vs. Nylon (.0005<p)	Cotton vs. Acrylic (1.0 >p)	Cotton vs. Silk (.977>p)	Cotton vs. Polyester (0.323>p)	Cotton vs. Cannabis (v) (.0005<p)	Cotton vs. Brassica (v) (.0005<p)	Cotton vs. *Typha* Leaf plant (v) (.0005<p)	Cotton vs. S5-NaOH-MS (.0005<p)	Cotton vs. S8-K3-MS (0.16>p)	Cotton vs. S10-L3-MS (.0005<p)

5.6.1 Summary

The statistical result shows that polyester and silk were not significant compared to *Typha* fibres, which indicates *Typha* fibre cannot be processed on spinning machine alone. It appears that *Typha* fibre sample S8-K3-MS is the best among *Typha* fibres because this sample is statistically similar to Cotton, wool, nylon, acrylic, and better than cannabis and brassica. Considering this softness grade of *Typha*, it could be possible to produce fibre blending with Cotton or wool or nylon or acrylic with various blending ratio (for example 70:30, 60:40, Liu etal., 2011) to make suitable for fibre spinning.

CHAPTER 6: CONCLUSION AND FUTURE WORK

Bast fibres, such as linum, brassica, cannabis and corchorus, are usually extracted using retting methods, most commonly water, enzymes, or acid. Different treatments have been employed to extract the fibres. Since the *Typha* plant grows in marshland areas, it demonstrated good resistance to the extraction of starch, and it was hard to break the *Typha* cell walls through microbial attack for retting. In this experiment, a water extraction method was applied for 90 days; however, it was not suitable for isolating fibre from *Typha* stems. It has been observed that several attempts have been made to extract fibres using enzymes, acid, and alcohol. None of these methods worked. In our tests, the only extraction method that worked used alkali.

The fibres obtained from alkali retting were stiff and clustered together. During chemical retting with LiOH, a higher yield of 66% was found compare to other bast fibres, but more fibre protruding fibres were produced. The alkali method using KOH was more suitable than LiOH, because of price and quality. It was noticed that alkali retting at low temperatures yields a fibre with a gummy substance; better fibres are produced at higher temperatures of 80°C to 95°C. A summary table (Table 6.1) has been prepared that indicates the fibre extraction process and suitability of Typha fibres for textile and apparel applications.

Table 6.1: Summary for fibre extraction, yield (%), textile properties of Typha fibre and their suitability in textiles and apparel applications

Parameters	Results	Comments
Retting		
Water retting	X	Up to 100°C and three months of treatment did not produce any fibre.
Chemical retting		
Acid retting	X	
Alcohol retting	X	
Enzyme retting	X	
Alkali	√	
Fibre yield (%)	√	Yield (%) higher than all other bast fibres
Physical properties		
Length	Yes	Fibre length is shorter than plant cut length
Diameter	Yes	Larger diameter variation
Microscopic nature	Yes	Round, rough, cells are polygonal, have unique crenelated structures.
Chemical properties		
Chemical compositions	Cellulosic	Wavenumber of 3433 cm^{-1} and 2920 cm^{-1} contain cellulose peaks.
Moisture regain (%)	Yes	Average MR% is 10.9
Thermal properties		
Thermal properties	Yes	Burning behavior similar to cotton, heat resistance is 268.7 °C.
Chemical properties		
Dyeing with reactive dye	Yes	80% dye exhaustion
Colourfastness to hot pressing	Yes	Performance rating: Dry state: 3 to 4.5 Wet state: 2.5 to 4
Physical properties		
Hand feel properties	(*)	Poor (2.8 out of 5), better than nylon, however, poorer than cotton and polyester, need surface modifications.

X – no fibre obtained; √: fibre obtained; (*) – suitable for textiles applications; Yes – properties comparable with cotton and wool;

Typha fibres possess some potential textile properties with a diameter in the range of 10 to 14 µm, and the average cut length of 39 to 54 mm is similar to Cotton and wool fibre. Fourier transform infrared spectrometer (FTIR) test values characterize that most important components at the wave 3452.24 cm^{-1} for S10-L1-MS, are cellulose and hemicellulose. The wave length 1733.66 cm^{-1} for *Typha* plant indicates lignin and pectin content; the band for wax content at 2849.75 cm^{-1} was found to be a cellulosic fibre. As with Cotton and other cellulosic materials, this group is dominant in *Typha* fibre.

There was no clear evidence that time or temperature of retting affects *Typha* fibre yield (%), but it can be seen that the fibre yield (%) decreases with the increase in treatment time. On the other hand, using similar treatment conditions in KOH at 95°C, the yield (%) is much lower than the yield (%) at 80°C for most treatment hours, except for the 8 hours treated sample. It is worth noting here that for the equivalent treatment conditions, the yield (%) using LiOH is much higher than the KOH, particularly after 6 and 8 hours' treatment. For example, after 6 hours' treatment at 80°C, KOH treatment produced 35% fibre [S8-K3-MS, Figure 4.17(a)], whereas the LiOH produced 56% fibre [S10-L3-MS, Figure 4.25 (a)], and after 8 hours' treatment, KOH treatment produced 15% fibre [S8-K4-MS, Figure 4.18 (a)] compared to 53% fibre [S10-L4-MS, Figure 4.26 (a)] produced in LiOH, but the LiOH treatment is more costly.

While investigating visual characterization, it was discovered that the *Typha* plant consists of different segments. There are some barriers for fibre extraction; one of them is that the *Typha* inner core, hard woody stems increases fibre flakes (%). Plant type and seasonal cultivated plants also affect extraction. Research found that fibre yield (%) may also be affected by plant morphology. The flakes are fibre-like materials found in the fibre bundle from which fibres can be separated by hand after immersing the stems in water, whereas, bark is a part of the hard/soft

woody stems. Fibre cannot be separated by hand from these stems after immersing in water, so the fibre yield (%) varies widely in an otherwise good fibre bundle. It was found that, although initially not known, the *Typha* plant can be categorized into three types: plants with a hard wood stem, plants with a soft stem, and plants with no stem (only leaves). A suggestion to improve fibre yield (%) and quality is to consider using no stem plants, as no leaf loss is required to get quality fibres.

Typha fibres represent a huge natural source of fibre with unique physical properties. If these naturally abundant plants are used for textile and industrial applications, it will not only benefit the grower or the farmer, but also increase overall economic activity.

Textile properties such as softness, individualization, moisture regain, thermal properties, decomposition temperature, dyeability and colour fastness properties were evaluated. A textile fibre should be soft for better acceptance in spinning systems; though bast fibres generally exhibit a higher stiffness compared to other fibres. *Typha* demonstrated a softness rating of 2.8, softer than brassica and cannabis, which had a softness rating of 1, and the synthetic fibre nylon, which had a softness rating of 2. Moisture regain refers to wear comfort. *Typha* fibre exhibits an acceptable range of moisture regain between 9.4 (%) and 12.7 (%). A textile fibre needs dye absorbability. *Typha* fibre showed good dyeing properties in reactive blue dye, having dye exhaustion (%) of 72 to 79%, while the value for Cotton is 74.5%. During the burn test, *Typha* fibre displayed thermal properties similar to Cotton fibre; *Typha* decomposition temperature was 268.7°C. The colour fastness properties to hot pressing demonstrated good bearing power at different temperatures in wet and dry conditions.

To make a quality fibre or yarn, one important condition is the softness of the fibre for ease of spinning on the Cotton spinning system. However, *Typha* fibres obtained from the chemical retting process are very stiff. Future research should focus on producing fine, flexible fibres. Bast

fibres are usually stiff because they consist of millions of microfibrils angled inside the cellulosic fibres that are inversely proportional to stiffness (Tanushree & Chanana, 2016). Therefore, it is necessary to further investigate mechanical properties to improve the knowledge of single fibre tensile strength and flexural strength of *Typha* fibre. Though the softness grading of *Typha* fibre was relatively low, it can be used as a blending fibre with other common fibres. Further study to develop standard chemical retting parameters and establish the effect of chemical treatment on fibre quality will be important. During *Typha* fibre extraction, different quality fibres (visual appearance) were produced from different types (Hard stems, Soft stems and No stems) of plants. Plant types / species or cultivars and the season of collection need to be investigated because the level of fertility, temperature, plant density and irrigation could improve the fibre quality (Tanushree & Chanana, 2016). Recently, bast fibres have received increased attention for their unique characteristics. A suitable word for the new fibre could be “eco-bast” or “eco-leaf” fibre, which could improve the sustainability of the textile industry by moving away from high input crops to natural, low input feedstock.

REFERENCES

AATCC Test Method 133. (2009)**.** Colorfastness to heat: hot pressing. In *AATCC Technical Manual* NC, USA: American Association of Textile Chemists and Colorists, 215–216.

AATCC. (2010). Fabric hand: Guidelines for the subjective evaluation of. In *AATCC Technical Manual*. NC, USA: American Association of Textile Chemists and Colorists, *85*, 371–373.

Abdullah, M., Rahmah, A.U., & Man, Z. (2010). Physicochemical and sorption characteristics of Malaysian Ceiba pentandra (L.) Gaertn as a natural oil sorbent. *Journal of Hazardous Materials*, *177*, 683–691.

Academic Consultancy Training (ACT) project. (2013). Cattail an innovative solution for our water challenges? *Cattail Biorefinery*. Project 1482

Acharya, S., Abidi, N., Rajbhandari, R., Meulewaeter, F. (2014). Chemical cationization of Cotton fabric for improved dye uptake. *Cellulose,* 21, 4693-4706

Agrawal, B. K. (1988). *Introduction to Engineering Materials*. New Delhi: Tata McGraw-Hill, 52.

Aizenshtein, E. M. (1996). Current production and consumption of polyester fibres and yarns, *Fibre Chemistry*, *28*(5).

Akin, D.E., Condon, B., Sohn, M., Foulk, J.A., Dodd, R.B., & Rigsby, L.L. (2007). Optimization for enzyme-retting of flax with pectate lyase. *Industrial Crops and Products*, *25*, 136-146.

Alawar, A., Hamed, A.M., & Al-Kaabi, K. (2009). Characterization of treated date palm tree fiber as composite reinforcement. *Composites Part B Engineering*, *40*, 601–606.

Ali, A. (2013). *Surface modification of hemp fiber to improve spinning properties*. Masters

thesis, University of Manitoba, 1-101.

Anderson, J. P., & Craig, W. J. (1984). *Growing energy crops on Minnesota's wetlands: The land use perspective.* University of Minnesota Center for Urban and Regional Affairs, 84-3.

ASTM International. (2008). *Standard Practice for Conditioning and Testing Textiles*. West Conshohocken, United States. http://doi.org/10.1520/D1776-08E01.2

ASTM International. (2012). *ASTM D2495-07 Standard Test Method for Moisture in Cotton by Oven-Drying*. Retrieved from https://doi-org.uml.idm.oclc.org/10.1520/D2495-07R12

ASTM International. (2016). *ASTM D1776/D1776M-16 Standard Practice for Conditioning and Testing Textiles.* Retrieved from https://doiorg.uml.idm.oclc.org/10.1520/D1776_D1776M-16

bacterial pectinolytic strains for improvement of hemp water retting. *Euphytica, 140*.47–54

Bednarz, C.W., Bridges, D.C., & Brown. S.M. (2000). Analysis of Cotton yield stability across population densities. *Agronomy Journal*, *92*,128-135.

Bidin, N., Zakaria, M.H., Bujang, J.S., & Aziz, N.A.A. (2015). Suitability of Aquatic plant Fibres for Handmade Papermaking. *International Journal of Polymer Science, 9.* http://dx.doi.org/10.1155/2015/165868

Blackburn, R.S. (Ed). (2005). *Biodegradable and sustainable fibres*. Cambridge: Woodhead. The Textile Institute. 47.

Booth, J. E. (1968). *Principles of textile testing: an introduction to physical methods of testing textile fibres, yarns and fabrics*. London, UK, Heywood Books, 111.

Brauer, D.S., Russel, C., Vogt, S., Weisser, J., & Schnabelrauch, M. (2008). Degradable phosphate glass fiber reinforced polymer matrices: mechanical properties and cell

response. *Journal of Materials Science: Materials in Medicine*, *19*,121–127. doi 10.1007/s10856-007-3147-x

Bridge, R.R., Meredith, W.R. Jr., & Chism, J.F. (1973). Influence of planting method and plant population on Cotton (Gossypium hirsutum L.). *Agronomy Journal, 65*,104-109.

Cai, Y., Pailthorpe, M.T., & David, S.K. (1999). A new method for improving the dyeability of Cotton with reactive dyes. *Textile Research Journal*, *69*(6), 440- 446.

Cao, S., Dong, T., Xu, G., & Wang, F. (2016). Study on structure and wetting characteristic of cattail fibers as natural materials for oil sorption. *Environmental Technology, 37*(24), 3193-3199, doi: 10.1080/09593330.2016.1181111

Cesar, N.R., Pereira-da-Silva, M.A., Botaro, V.R., & Menezes, A.J.D. (2015). Cellulose nanocrystals from natural fiber of the macrophyte Typha domingensis: extraction and characterization. *Cellulose*, 22:449–460.

Chattopadhyay, R., & Sinha, S. K. (2007). A study on spinning limits and yarn properties with progressive change in yarn count in friction spinning. *AUTEX Research Journal*, *7*(1).

Collier, B. J., & Epps, H. H. (1999). *Textile testing and analysis*. Upper Saddle River, New Jersey: Prentice Hall.

Collier, B. J., Bide, M. J., & Tortora, P. G. (2009). *Understanding textiles* (7th ed.). Upper Saddle River, New Jersey: Pearson Prentice Hall.

Cook, J.G. (2001). *Handbook of textile fibres.* Cambridge England: Woodhead. 2, 192 -193.

Cookson, P.G., & Slota, I.J. (1993). Hysteresis effects associated with the adsorption and desorption of water by woven wool fabrics, *Textile Research Journal, 63*(9), 495-503.

Cotton fabric for improved dye uptake. *Cellulose, 21*, 4693–4706.

Curtis, S. (publ.) (1828). *Curtis's Botanical Magazine, Illustration of Corchorus olitorius*, 55 (2), Tab. 2810, Image courtesy of Missouri Botanical Garden © 1995 -2009, http//: www.botanicus.org

Dave, H., Lalita, L., Chandwani, N., Chauhan, N., & Nema, S.K. (2013). The removal of impurities from gray Cotton fabric by atmospheric pressure plasma treatment and its characterization using ATR-FTIR spectroscopy. *The Journal of The Textile Institute*, 105 (6), 586–596.

Dubbe, D. R., Garver, E. G., & Pratt, D. C. (1988). Production of cattail (*Typha spp*.) biomass in Minnesota, USA. *Biomass*, *17*(2), 79–104. https://doi.org/10.1016/0144-4565(88)90073X.

Dubrovski, P. D. (Ed.) (2010). *Woven fabric engineering*, Rijeka, Croatia: Sciyo, 25.

Dungani, R., Karina, M., Subyakto, S. A., Hermawan, D., & Hadiyane, A., (2016). Agricultural waste fibres towards sustainability and advanced utilization: A review. *Asian Journal of Plant Sciences, 15*, 42-55. doi: 10.3923/ajps.2016.

Environment Canada. (2017). *Environment Canada's weather* - Environment and natural resources - Weather information. Retrieved September 19, 2017, from http://climate.weather.gc.ca/climate_data/hourly_data_e.html?StationID=27174&month=6&day=11&timeframe=1&Year=2017&Month=6&Day=11&StartYear=1840&EndYear=2017

Fahlgren, D. (2017). *Genetic variation in common cattail (Typha latifolia) in southern Sweden*. M.Sc. Thesis, Swedish University of Agricultural Sciences.

Franck. R.R. (2005). Ramie. In*: Bast and other plant fibres*, Franck. R.R. (Ed.). Cambridge, UK: Woodhead, 8-9.

Fu, J. (Ed.). (2013). *Dyeing: Processes, techniques and applications,* New York: Nova Science ISBN: 978-1-62808-847-2, 1-18.

Gordon, S., & Hsieh, Y. L. (Eds.). (2007). *Cotton: Science and technology* (1st ed). Boca Raton, USA: Woodhead, 68–102.

Grace, J. B., & Harrison, J. S. (1986). The biology of Canadian weeds: 73. Typha latifolia L., Typha angustifolia L. and Typha xglauca Godr. *Canadian Journal of Plant Science*, *66*(2), 361-379.

Grosshans, R. E. (2014). *Cattail (Typha spp.) biomass harvesting for nutrient capture and sustainable bioenergy for integrated watershed management*. PhD thesis, University of Manitoba, 274.

Hamdaoui, M., Turki, S., Romdhani, Z., & Halaoua, S. (2013). Effect of reactive dye mixtures on exhaustion values. *Indian Journal of Fibre & Textile Research*, *38*, 405-409.

Hatch, K. L. (1993). *Textile science*. Saint Paul, Minneapolis: West.

Hearle, J.W.S., & Peters, R.H. (1963). *Fibre structure*. London: The Textile Institute Butterworths.

Hoffmann, C. (1884). *Botanischer Bilder- Atlas nach de Candolle's Naturlichem Pflanzensystem,* with over 500 full-scale picture of plants on 85 fine colour plates and explanatory text, *Cocos nucifera*, Plate 75, 431 a/b, J. Haffmann, Stuttgart, Germany, online resource, Kurt Stueber (2003) http://caliban.mpiz-koeln.mpg.de/stueber/hoffmann/index.html

Houlahan, J. E., Keddy, P. A., Makkay, K., & Findlay, C. S. (2006). The effects of adjacent land use on wetland species richness and community composition. *Wetlands*, *26*, (1), 79–96.

Hybl, J. (1975). *Comparison of ring and open-end spinning*. New York: Elsevier.

Jackowski, T., Chylewska, B., & Cyniak, D. (1999). *Long staple Cotton – Technology of spinning*, GCA Cotton conference, Gdynia, Poland.

Judkins, G. (2008). Declining Cotton cultivation in Maricopa country, Arizona: An examination of macro-and micro-scale driving forces. *Yearbook of the Association of Pacific Coast Geographers*, *70*(1), 70–95.

Judl, J., Hildén, M., Antikainen, S. R., Temmes, A., Kuisma, A.M., & Peck, P. (2016). *The renewal of forest-based industries needs to focus on environmental opportunities and challenges*. Conference paper, (10).

Kaczmar, J.W., Pach, J., & Burgstaller, C. (2011). The chemically treated hemp fibres to reinforce polymers. *Polimery,* 56, 1112.

Kalia, S., Kaith, B. S., & Kaur, I. (Eds.). (2011). *Cellulose fibres: Bio-and nano-polymer composites*, Berlin, Germany: Springer, ISBN: 978-3-642-17369-1, 3-42.

Kaushik, R. C. D., Tyagi, G. K., & Chatterjee, K. N. (1990). Contribution of fibre profile and twist factor to flexural rigidity and elastic recovery of polyester fibre yarns, *Indian Journal Fibre & Textile Research*, 200–201.

Khalid, R.H., Jawaid, M., Umer, R. (Eds.). (2014). *Processing and properties of date palm fibers and Its composites* (1st ed). Springer-Verlag, 1–25. doi 10.1007/978-3-319-07641-6_1

Khan, M. R. I. (2016). *Evaluation of brassica fibre for textile and spinning properties*. Unpublished manuscript, Department of Biosystems Engineering, University of Manitoba, Winnipeg, Manitoba, Canada.

Kim, J. H., Rodgers, J., Delhom, C., & Cui, X. (2014). Comparisons of methods measuring fibre maturity and fineness of upland Cotton fibres containing different degrees of fibre cell wall development. *Textile Research Journal*, *84*(15), 1622–1633.

King, D. (2007). Dyeing Cotton and Cotton products. In S. Gordon, & Y. L. Hsieh (Eds.), *Cotton: Science and Technology* (1st ed). Boca Raton, USA: Woodhead, 353–380.

Kopke, V., & Lindberg, J. (1966). Steam-pressing of wool farbric: the influence of temperature and moisture regain on the efficiency of pressing. *Journal of the Textile Institute (Transactions), 57*(12), 551-573.

Kozłowski, R. M. (Ed.). (2012). *Handbook of natural fibres*. Cambridge, UK: Woodhead. *2,* 219–253. http://doi.org/10.1533/9780857095510.2.219

Kozlowski, R. M. (2012a). *Handbook of natural fibres* (1st ed). Toronto, Canada: Woodhead, *2,* 221–223. https://doi.org/10.1021/jf0617723

Kozlowski, R. M. (2012b). *Handbook of natural fibres* (1st ed), Cambridge, UK: Woodhead. *1,* 64–65.

Kozlowski, R. M. (Ed.). (2012c). *Handbook of natural fibres* (1st ed), Cambridge, UK: Woodhead . *1,* 12.

Kozlowski, R. M. and Mackiewicz-Talarczyk, M. (2012). *Handbook of natural fibres*, (1st ed). Cambridge,UK: Woodhead, 64-65.

Krässig, H. A. (1993). *Cellulose, Structure, Accessibility and Reactivity*. Philadelphia, USA: Gordon and Breach Science.

Krowicki, R.S., Hemstreet, J.M., & Duckett, K.E. (1997). Determination of the 50% and 2.5% span lengths and the uniformity ratio with the fibrograph, *Journal of Textile Institute, 88* (Part I), 167-173.

Krus, M., Theuerkorn, W., Großkinsky, T., & Künzel, H. (2014). New sustainable and insulating building material made of cattail. *Nordic Symposium on Building Physics (NSB),10,* (156), 1252–1260. Retrieved from http://publica.fraunhofer.de/documents/N-

323927.html

Kumar, R.S. (2015). *Process management in spinning*. New York, Taylor & Francis Group.

Lawrence, C. A. (2003). *Fundamental of spun yarn technology*. Washington, New York: CRC Press.

Lewin, S. M., & Pearce, E.M. (Eds.). (1998). *Handbook of fibre chemistry*. New York: Marcel Dekker, 505-571.

Liggett, R.W., Hoffman, H. L., Tanquary, A.C., & Vail, S.L. (1968). Cross-linked cotton of high moisture regain.*Textile Research Journal*, *38*(4), 375-387.https://doi.org/10.1177/004051756803800408

Li, X., Tabil, L.G., & Panigrahi,S. (2007). Chemical treatment of natural fibre for use in natural fibre-reinforced composits. *Journal of Polymer and the Environment*, *15*(1), 25-33.

Liu, L., Han, Y., & Zhang, X. (2011). Research on the spinnability of the renewable cattail fiber. *Advanced Material Research*, 332- 334, 192-195.

Lord, P.R. (2003). *Handbook of yarn production*. Cambridge, England: Woodhead, 201.

Luamkanchanaphan, T., Chotikaprakhan, S., & Jarusombati, S. (2012). *A Study of physical, mechanical and thermal properties for thermal insulation from narrow-leaved cattail fibres.* APCBEE Procedia 1, 46 – 52.

Maizatul, O., Ruzaidi, G., Khalisanni, K., & Nazarudin, Z. (2012). Morphology and water absorption analysis on single fibre and leaf of typha latifolia. *Advanced Materials Research, 576,* 492-495.

Marques, G., Rencoret, J., Gutiérrez, A., & Del Río, J. C. (2010). Evaluation of the chemical composition of different non-woody plant fibres used for pulp and paper manufacturing. *The Open Agriculture Journal, 4,* 93-101.

Mohammed, L., Ansari, M.N.M., Pua, G., Jawaid, M., & Islam M.S.(2015). A review on natural fibre reinforced polymer composite and its applications. International Journal of Polymer Science, 2015(243947), 15, http://dx.doi.org/10.1155/2015/243947

Mather, R. R., & Wardman, R. H. (2015). *The Chemistry of textile fibres* (2nd ed). Cambridge, UK: The Royal Society of Chemistry.

Meyers (1906).*Großes Konversations-Lexikon*. Band 6. *Faserpflanzen I und Faserpflanzen II.* Bibliographisches Institut, Wien/Leipzing, Austria/Germany, online resource, Editura Gesellschaft för Verlagsdienstleistungen mbH (2009), http://www.zeno.org.

Minotte, S. J., & Franck. R.R. (2005). Flax. In*: Bast and other plant fibres*, R. R. Franck (Ed.), Cambridge, England: Woodhead, 159-160.

Mohanty, A.K., Manjusri, M., & Drzal, L.T. (2005). (Eds.). *Natural fibres*, Biopolymers and Biocomposites. Florida, USA: CRC Press, 45 -50.

Moncrieff, R.W. (1963). *Man-Made fibre* (4th eds). London: Heywood. 19.

Montalvo, J., & G, Jr. (2005). Relationships between micronaire, fineness, and maturity. Part I. Fundamentals. *The Journal of Cotton Science, 9*, 81–88.

Morton, W. E., & Hearle, J.W. S. (2008). *Physical properties of textile fibres.* Cambridge, England: Woodhead, 134.

Muthu, S. (2018). *Sustainable innovations in textile chemical processes.* Singapore: Springer. https://doi.org/10.1007/978-981-10-8491-1

Mussig, J. (Ed.). (2010). Function in nature. In: *Industrial applications of natural fibre*s, West Sussex, UK: John Wiley & Sons, *29*.

Mwaikambo, Y. L., & Ansell, P. M. (2002). Chemical modification of hemp, sisal, jute, and kapok fibers by alkalization. *Journal of Applied Polymer Science*. *84*, 2222–2234.

Naveen, E., Venkatachalam, N., & Maheswaran, N. (2015). Alkali chemical treatment on the surface of natural fiber. *International Journal of Innovative Research in Science, Engineering and Technology*. 4 (4), 172-178.

Pabst, G. (ed.) (1887). *Kohler's Medizinal-Pflanzen in naturgetreuen Abbildungen mit kurz erlauterndem Texte*, Band I, Cannabis sativa L., Tafel 13, Organalzeichnungen von Walther Muller in Gera, Gera- Untermhaus, Germany, Verlag von Fr. Eugen Kohler, Image courtesy of Missouri Botanical Garden © 1995 -2009, http;//www.botanicus.org

Pandey, A. & Verma, R. K. (2018). Taxonomical and pharmacological status of Typha: A Review. *Annals of Plant Sciences*, 7.3, 2101-2106.

Pandey, S.N. (2007). Ramie fibre: part II. Physical fibre properties. A critical appreciation of recent developments. *Textile progress.* 19(4), 241.

Rashid, B., Yousaf, I., Haider, T., Rasheed, Z., Ali, Q., Javed, F., & Husnain, T. (2016). Roadmap to sustainable Cotton production. *Life Science Journal*, *13*(11), 41-48.

Rohlena, V. & Plisek, L. (1975). *Open-end spinning*. Elsevier, Amsterdam.

Rowland, S.P., Nelson, M. L., Welch, C.M., & Hebert, J.J. (1976). Cotton fiber morphology and textile performance properties. *Textile Research Journal*, *46*(3), 194- 214.

Roy, S., & Lutfar, L. B. (2012a). Bast fibres: ramie. In R. M. Kozlowski (Ed.), *Handbook of natural fibres: Properties and factors affecting breeding and cultivation*. Cambridge, UK: Woodhead, *1,* 47–55.

Sana, R., Jaouadi, M., Khoffi, F., & Durand, B. (2016). Optimization of processing parameters for extraction of typha stem fibers. International Journal of Applied Research on Textile, *4*(2), 53-64.

Salit, M.S. (2014). Tropical natural fibre composites. *Engineering Materials Singapore*:

Springer. doi 10.1007/978-981-287-155-8_1

Sana, R., Mounir, J., & Slah, M. (2014). Study of structure and properties of tunisian typha leaf fibres. *International Journal of Engineering Research & Technology, 3*(3), 539–546.

Sgriccia, N., Hawley, M. C., & Misra, M. (2008). Characterization of natural fibre surfaces and natural fibre composites. *Composites Part A: Applied Science and Manufacturing*, *39*(10), 1632–1637. https://doi.org/10.1016/j.compositesa.2008.07.007

Sharp, J. L. (B.S.). (2002). *Managing cattail (Typha latifolia) growth in wetland systems*. Masters thesis, University of North Texas, 1-53.

Shore, J. (Eds.). (1995). *Cellulosics dyeing*, Manchester, UK: Society of Dyers and Colourists, 223-224.

Smith, C.W., & Coyle, G.G. (1997). Association of fibre quality parameters and within-boll yield components in upland Cotton. *Crop Science*, *37*, 1775-1779.

Smole, S.M., Hribernik, S., Kleinschek, S.K., & Kreže, T. (2013). Plant fibres for textile and technical applications. *Advances in Agrophysical Research*, 369–397. http://dx.doi.org/10.5772/52372

Sojda, R. S., & Solberg, K. L. (1993). *Management and control of cattails.* U.S. Fish and Wildlife Leaflet. Waterfowl Management Handbook. 33. http://digitalcommons.unl.edu/icwdmwfm/33

Sopit, V. (2007). Using cattails (*Typha Latifolia*) as a substrate for pleurotus ostreatus (Fr.) *kummer cultivation*, *7*(1), 218–221.

Sridach, W., & Paladsongkhram, R. (2014). Improvement of hardwood kraft paper with narrow-leaved Typha latifolia L. fibre, cationic starch and asa. *Cellulose Chemistry and Technology*, *48* (3-4), 375-383.

Stevulova, N., Cigasova, J., Estokova, A., Terpakova, E., Geffert, A., Kacik, F., Singovszka, E., & Holub, M. (2014). Properties characterization of chemically modified hemp hurds. *Materials,* 7, 8131-8150; doi:10.3390/ma7128131.

Stout, E. E. (1960). *Introduction to textiles*. New York, USA: John Wiley & Sons, 26.

Sun, R.C., Lu, Q., & Sun, X.F. (2001). Physico-chemical and thermal characterization of lignins from Caligonum monogoliacum and Tamarix spp. *Polymer Degradation. Stability, 72,* 229–238.

Tahir, P.M., Amel B. Ahmed, A.B., Saifulazry, S. O. A., & Ahmed, Z. (2011). Retting process of some bast plant fibres and its effect on fibre quality: Review of bast fiber retting, *BioResources, 6*(4), 5260-5281.

Tamburini, E., Gordillo, L. A., Perito, B, Candilo, M. D. (2004). Exploitation of bacteria pectinolytic strains for improvement of hemp water retting. *Euphytica, 140*. 47-54

Tanushree, & Chanana, B. (2016). Characterization and mechanical properties of bast fibre. *International Journal of Home Science, 2*(2), 291-295.

Taylor, M. A. (1972). *Technology of textile properties*. London: Forbes Publications, 7-10.

Thakur, V. K. (2015). *Lignocellulosic polymer composites: Processing, characterization, and properties*. Massachusetts, USA: Scrivener.

Thomas, L. H., Forsyth, V. T., Šturcová, A., Kennedy, C.J., May, R.P., Altaner, C.M., Apperley, D.C., Wess, T.J., & Jarvis, M.C. (2013). Structure of cellulose microfibrils in primary cell walls from collenchyma. *Plant Physiology, 161,* 465–476,

Van der Werf, H. M. G., & Turunen, L. (2008). The environmental impacts of the production of hemp and flax textile yarns. *Industrial Crops and Products, 27,* 1–10.

Vėjelienė, J., Gailius, A., Vėjelis, S., Vaitkus, S., & Balciunas, G. (2011). Evaluation of structure

influence on thermal conductivity of thermal insulating materials from renewable resources. *Materials Science*, *17*(2).

Wang, J.T., Zheng, Y.A., & Wang, A.Q. (2012). Effect of kapok fiber treated with various solvents on oil absorbency. *Industrial Crops and Products*, *40*, 178–184.

Wang, Y. (2006). *Textile recycling.* Cambridge UK: Woodhead.

Weatherall, I. L., & Coombs, B. D. (1991). Skin color measurements in terms of *CIELAB* color space values. *Invest Dermatol*, *99*(4), 468 - 473.

Williams, T., Hosur, M., Theodore, M., Netravali, A., Rangari, V., & Jeelani, S. (2011). Time effects on morphology and bonding ability in mercerized natural fibers for composite reinforcement. *International Journal of Polymer Science*, *2011*, 1 – 9.

Wool, R. P., & Sun, X. S. (Eds.). (2005). *Bio-Based Polymers and Composites*. Amsterdam: Elsevier.1–14. http://doi.org/10.1016/B978-012763952-9/50002-2

Wuzellaa, G., Mahendrana, A. R., Batgeb, T., Jurya, & Kandelbauerb. (2011). Novel, binder-free fibre reinforced composites based on a renewable resource from the reed-like plant Typha sp.ll, *Industrial Crops and Products*, *33*, 683 – 689.

WWF Global (n.d.). *Cotton farming, Cotton: A water wasting crop*. Retrieved from http://wwf.panda.org

Xiao, B., Sun, X. F., & Sun, R. (2001). Chemical, structural, and thermal characterizations of alkali-soluble lignins and hemicelluloses, and cellulose from maize stems, rye straw, and rice straw. *Polymer Degradation and Stability*, *74*(2), 307–319. https://doi.org/10.1016/S0141-3910(01)00163-X

Zeng, Y., Himmel, M.E., & Ding, S.Y. (2017).Visualizing chemical functionality in plant cell walls. *Biotechnol Biofuels*, 10: 263,1 -16. doi 10.1186/s13068-017-0953-3

Zawani, Z., Abdullah, L. C., & Abdan, K. (2015). Characterization of kenaf fibre-retting wastewater. *International Journal of Science and Research, 4*(6), 314–317.

Zhang, Y., Yang, S., Wu, J.Q., Yuan, T.Q., & Sun, R.C. (2014). Preparation and characterization of lignocellulosic oil sorbent by hydrothermal treatment of Populus fiber. *Materials, 7*, 6733-6747; doi: 10.3390/ma7096733

Zheng, D. F., Qiu, X.Q., & Lou, H. M. (2005). The structure of lignin and its chemical modification. *Fine Chemicals*, *22*(4), 249–52.

Appendix I

Figure 4.15 (b): *Typha* fibre treated with alkali (Sample ID: S8-K1-HS) for 2h/3%/80°C

Figure 4.15 (c): *Typha* fibre treated with alkali (Sample: S8-K1-SS) for 2h/3%/80°C

Figure 4.15 (d): *Typha* fibre treated with alkali (Sample: S8-K1-NS) for 2h/3%/80°C

Figure 4.16 (b): *Typha* fibre treated with alkali (Sample: S8-K2-HS) for 4h/3%/80°C

Figure 4.16 (c): *Typha* fibre treated with alkali (Sample: S8-K2-SS) for 4h/3%/80°C

Figure 4.16 (d): *Typha* fibre treated with alkali (Sample: S8-K2-NS) for 4h/3%/80°C

Figure 4.17 (b): *Typha* fibre treated with alkali (Sample: S8-K3-HS) for 6h/3%/80°C

Figure 4.17 (c): *Typha* fibre treated with alkali (Sample: S8-K3-SS) for 6h/3%/80°C

Figure 4.17 (d): *Typha* fibre treated with alkali (Sample: S8-K3-NS) for 6h/3%/80°C

Figure 4.18 (b): *Typha* fibre treated with alkali (Sample: S8-K4-HS) for 8h/3%/80°C

Figure 4.18 (c): *Typha* fibre treated with alkali (Sample: S9-K4-SS) for 8h/3%/80°C

Figure 4.18 (d): *Typha* fibre treated with alkali (Sample: S9-K4-NS) for 8h/3%/80°C

Figure 4.19 (b): *Typha* fibre treated with alkali (Sample: S9-K5-HS) for 2h/3%/95°C

Figure 4.19 (c): *Typha* fibre treated with alkali (Sample: S9-K5-SS) for 2h/3%/95°C

Figure 4.19 (d): *Typha* fibre treated with alkali (Sample: S9-K5-NS) for 2h/3%/95°C

Figure 4.20 (b): *Typha* fibre treated with alkali (Sample: S9-K6-HS) for 4h/3%/95°C

Figure 4.20 (c): *Typha* fibre treated with alkali (Sample: S9-K6-SS) for 4h/3%/95°C

Figure 4.20 (d): *Typha* fibre treated with alkali (Sample: S9-K6-NS) for 4h/3%/95°C

Figure 4.21 (b): *Typha* fibre treated with alkali (Sample: S9-K7-HS) for 6h/3%/95 °C

Figure 4.21 (c): *Typha* fibre treated with alkali (Sample: S9-K7-SS) for 6h/3%/95 °C

Figure 4.21 (d): *Typha* fibre treated with alkali (Sample: S9-K7-NS) for 6h/3%/95°C

Figure 4.22 (b): *Typha* fibre treated with KOH (Sample: S9-K8-HS) for 8h/3%/80°C

Figure 4.22 (c): *Typha* fibre treated with with KOH (Sample: S9-K8-SS) for 8h/3%/80°C

Figure 4.22 (d): *Typha* fibre treated with with KOH (Sample: S9-K8-NS) for 8h/3%/80°C

Figure 4.23 (b): *Typha* fibre treated with LiOH (Sample: S10-L1-SS) for 2h/3%/80°C

Figure 4.23 (c): *Typha* fibre treated with LiOH (Sample: S10-L1-HS) for 2h/3%/80°C

Figure 4.23 (d): *Typha* fibre treated with LiOH (Sample: S10-L2-NS) for 2h/3%/80°C

Figure 4.24 (b): *Typha* fibre treated with LiOH (Sample: S10-L2-HS) for 4h/3%/80°C

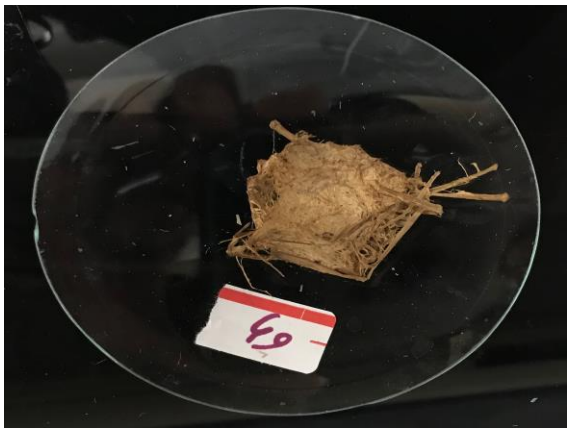


Figure 4.24 (c): *Typha* fibre treated with LiOH (Sample: S10-L2-SS) for 4h/3%/80°C

Figure 4.24 (d): *Typha* fibre treated with LiOH (Sample ID: S10-L2-NS) for 4h/3%/80°C

Figure 4.25 (b): *Typha* fibre treated with LiOH (Sample: S10-L3-HS) for 6h/3%/80°C

Figure 4.25 (c): Typha latifolia L. fibre treated with LiOH (Sample ID: S10-L3-SS) for 6h/3%/80°C

Figure 4.25 (d): *Typha* fibre treated with LiOH (Sample: S10-L3-NS) for 6h/3%/80°C

Figure 4.26 (b): Typha latifolia L. fibre treated with LiOH (Sample ID: S10-L4-HS) for 8h/3%/80°C

Figure 4.26 (c): *Typha* fibre treated with LiOH (Sample: S10-L4-SS) for 8h/3%/80°C

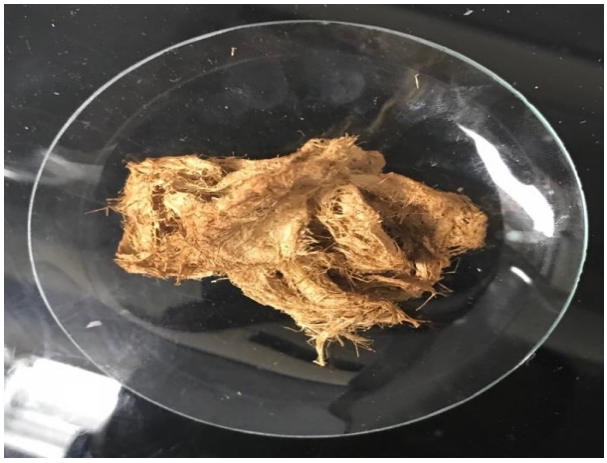


Figure 4.26 (d): Typha latifolia L. fibre treated with LiOH (Sample ID: S10-L4-NS) for 8h/3%/80°C

Appendix II

Fibre diameters from various *Typha* samples

	Fibre Type	**Diameter of *Typha* fibre**				
	S5-NaOH	D1	D2	D3	D4	D5
		100	140	38	150	40
		95	145	54	145	120
		85	170	60	155	115
Mean:		93.33	151.67	50.67	150.00	91.67
SD			43.10			
CV%			40.13			
	S8K1					
		126	90	175	80	68
		128	70	162	45	30
		124	65	159	48	35
Mean:		126.00	75.00	165.33	57.67	44.33
SD			50.65			
CV%			54.07			
	S8K2					
		50	50	27	15	55
		40	43	30	20	45
		53	70	20	12	35
Mean		47.67	54.33	25.67	15.67	45.00
SD			16.28			
CV%			43.21			
	S8K3					
		75	63	80	30	95
		45	51	45	20	110
		38	40	41	44	100
Mean		52.67	51.33	55.33	31.33	101.67
SD			25.97			
CV%			44.41			
	S8K4					
		110	58	60	95	30
		115	45	85	100	40
		135	50	90	80	60
Mean		120.00	51.00	78.33	91.67	43.33
SD			31.13			
CV%			40.49			

	S9K5					
		60	38	80	43	242
		70	45	75	55	220
		20	25	95	68	95
Mean		50.00	36.00	83.33	55.33	185.67
SD			60.41			
CV%			73.62			
	S9K6					
		42	40	38	20	48
		50	55	43	46	60
		35	60	20	27	110
Mean		42.33	51.67	33.67	31.00	72.67
SD			16.83			
CV%			36.38			
	S9K7					
		35	55	33	200	40
		52	42	26	70	70
		38	35	19	175	89
Mean		41.67	44.00	26.00	148.33	66.33
SD			48.61			
CV%			74.48			
	S9K8					
		50	36	105	150	70
		37	41	94	122	109
		85	17	88	90	66
Mean		57.33	31.33	95.67	120.67	81.67
SD			34.46			
CV%			44.55			
	S10L1					
		105	210	85	72	64
		150	170	115	85	30
		50	140	35	50	45
Mean		101.67	173.33	78.33	69.00	46.33
SD			48.72			
CV%			51.98			
	S10L2					
		112	80	35	43	148

		93	65	55	48	135
		144	120	30	37	96
Mean		116.33	88.33	40.00	42.67	126.33
SD			40.29			
CV%			48.70			
	S10L3					
		160	120	54	33	80
		140	107	96	30	50
		125	70	39	20	46
Mean		141.67	99.00	63.00	27.67	58.67
SD			43.67			
CV%			55.98			
	S10L4					
		21	130	33	12	17
		19	150	16	17	26
		45	111	22	25	35
Mean		28.33	130.33	23.67	18.00	26.00
SD			47.71			
CV%			105.39			

Appendix III

Obs	fibre	diameter	Obs	fibre	diameter	Obs	fibre	diameter
1	*Typha*	12	**41**	*Typha.*	13	**81**	*Typha*	12
2	*Typha*	14	**42**	*Typha*	13	**82**	*Typha*	10
3	*Typha*	14	**43**	*Typha*	13	**83**	*Typha*	8
4	*Typha*	12	**44**	*Typha*	13	**84**	*Typha*	8
5	*Typha*	14	**45**	*Typha*	13	**85**	*Typha*	8
6	*Typha*	12	**46**	*Typha*	13	**86**	*Typha*	7
7	*Typha*	12	**47**	*Typha*	11	**87**	*Typha*	8
8	*Typha*	14	**48**	*Typha*	11	**88**	*Typha*	8
9	*Typha*	10	**49**	*Typha*	11	**89**	*Typha*	7
10	*Typha*	10	**50**	*Typha*	11	**90**	*Typha*	8
11	*Typha*	16	**51**	*Typha*	16	**91**	*Typha*	10
12	*Typha*	16	**52**	*Typha*	16	**92**	*Typha*	10
13	*Typha*	16	**53**	*Typha*	14	**93**	*Typha*	11
14	*Typha*	15	**54**	*Typha*	12	**94**	*Typha.*	11
15	*Typha*	15	**55**	*Typha*	12	**95**	*Typha*	10
16	*Typha*	14	**56**	*Typha*	8	**96**	*Typha*	13
17	*Typha*	15	**57**	*Typha*	8	**97**	*Typha*	13
18	*Typha*	15	**58**	*Typha*	11	**98**	*Typha*	7
19	*Typha*	16	**59**	*Typha*	15	**99**	*Typha*	7
20	*Typha*	16	**60**	*Typha*	15	**100**	*Typha*	10
21	*Typha*	15	**61**	*Typha*	13	**101**	Cotton	13
22	*Typha*	15	**62**	*Typha*	15	**102**	Cotton	13
23	*Typha*	15	**63**	*Typha*	15	**103**	Cotton	13
24	*Typha*	15	**64**	*Typha*	15	**104**	Cotton	13
25	*Typha*	15	**65**	*Typha*	13	**105**	Cotton	13
26	*Typha*	15	**66**	*Typha*	13	**106**	Cotton	13
27	*Typha*	15	**67**	*Typha*	15	**107**	Cotton	10
28	*Typha*	13	**68**	*Typha*	15	**108**	Cotton	8
29	*Typha*	13	**69**	*Typha*	11	**109**	Cotton	8
30	*Typha*	13	**70**	*Typha*	15	**110**	Cotton	8
31	*Typha*	13	**71**	*Typha*	18	**111**	Cotton	8
32	*Typha*	13	**72**	*Typha*	16	**112**	Cotton	12
33	*Typha*	13	**73**	*Typha*	14	**113**	Cotton	12
34	*Typha*	13	**74**	*Typha*	14	**114**	Cotton	13
35	*Typha*	13	**75**	*Typha*	14	**115**	Cotton	8
36	*Typha*	13	**76**	*Typha*	14	**116**	Cotton	13
37	*Typha*	13	**77**	*Typha*	16	**117**	Cotton	12
38	*Typha*	13	**78**	*Typha*	14	**118**	Cotton	12
39	*Typha*	13	**79**	*Typha*	14	**119**	Cotton	12
40	*Typha*	11	**80**	*Typha*	12	**120**	Cotton	8

Obs	fibre	diameter	Obs	fibre	diameter
121	Cotton	10	**161**	Cotton	14
122	Cotton	13	**162**	Cotton	15
123	Cotton	13	**163**	Cotton	15
124	Cotton	13	**164**	Cotton	13
125	Cotton	13	**165**	Cotton	15
126	Cotton	10	**166**	Cotton	15
127	Cotton	10	**167**	Cotton	13
128	Cotton	10	**168**	Cotton	13
129	Cotton	8	**169**	Cotton	15
130	Cotton	8	**170**	Cotton	13
131	Cotton	14	**171**	Cotton	10
132	Cotton	14	**172**	Cotton	12
133	Cotton	10	**173**	Cotton	13
134	Cotton	10	**174**	Cotton	14
135	Cotton	12	**175**	Cotton	13
136	Cotton	12	**176**	Cotton	13
137	Cotton	12	**177**	Cotton	12
138	Cotton	12	**178**	Cotton	10
139	Cotton	14	**179**	Cotton	10
140	Cotton	14	**180**	Cotton	10
141	Cotton	15	**181**	Cotton	12
142	Cotton	12	**182**	Cotton	12
143	Cotton	15	**183**	Cotton	14
144	Cotton	15	**184**	Cotton	14
145	Cotton	15	**185**	Cotton	12
146	Cotton	15	**186**	Cotton	14
147	Cotton	12	**187**	Cotton	12
148	Cotton	12	**188**	Cotton	14
149	Cotton	12	**189**	Cotton	9
150	Cotton	10	**190**	Cotton	12
151	Cotton	10	**191**	Cotton	13
152	Cotton	10	**192**	Cotton	13
153	Cotton	12	**193**	Cotton	15
154	Cotton	12	**194**	Cotton	15
155	Cotton	12	**195**	Cotton	15
156	Cotton	12	**196**	Cotton	13
157	Cotton	12	**197**	Cotton	13
158	Cotton	12	**198**	Cotton	13
159	Cotton	12	**199**	Cotton	13
160	Cotton	12	**200**	Cotton	13

Obs	fibre	diameter	Obs	fibre	diameter	Obs	fibre	diameter
1	Typha	12	**41**	Typha	13	**81**	Typha	12
2	Typha	14	**42**	Typha	13	**82**	Typha	10
3	Typha	14	**43**	Typha	13	**83**	Typha	8
4	Typha	12	**44**	Typha	13	**84**	Typha	8
5	Typha	14	**45**	Typha	13	**85**	Typha	8
6	Typha	12	**46**	Typha	13	**86**	Typha	7
7	Typha	12	**47**	Typha	11	**87**	Typha	8
8	Typha	14	**48**	Typha	11	**88**	Typha	8
9	Typha	10	**49**	Typha	11	**89**	Typha	7
10	Typha	10	**50**	Typha	11	**90**	Typha	8
11	Typha	16	**51**	Typha	16	**91**	Typha	10
12	Typha	16	**52**	Typha	16	**92**	Typha	10
13	Typha	16	**53**	Typha	14	**93**	Typha	11
14	Typha	15	**54**	Typha	12	**94**	Typha	11
15	Typha	15	**55**	Typha	12	**95**	Typha	10
16	Typha	14	**56**	Typha	8	**96**	Typha	13
17	Typha	15	**57**	Typha	8	**97**	Typha	13
18	Typha	15	**58**	Typha	11	**98**	Typha	7
19	Typha	16	**59**	Typha	15	**99**	Typha	7
20	Typha	16	**60**	Typha	15	**100**	Typha	10
21	Typha	15	**61**	Typha	13	**101**	Wool	15
22	Typha	15	**62**	Typha	15	**102**	Wool	15
23	*Typha.*	15	**63**	*Typha*	15	**103**	Wool	15
24	*Typha*	15	**64**	*Typha*	15	**104**	Wool	15
25	*Typha*	15	**65**	*Typha*	13	**105**	Wool	15
26	*Typha*	15	**66**	*Typha*	13	**106**	Wool	15
27	*Typha*	15	**67**	*Typha*	15	**107**	Wool	15
28	*Typha*	13	**68**	*Typha*	15	**108**	Wool	15
29	*Typha*	13	**69**	*Typha*	11	**109**	Wool	15
30	*Typha*	13	**70**	*Typha*	15	**110**	Wool	15
31	*Typha*	13	**71**	*Typha*	18	**111**	Wool	18
32	*Typha*	13	**72**	*Typha*	16	**112**	Wool	18
33	*Typha*	13	**73**	*Typha*	14	**113**	Wool	18
34	*Typha*	13	**74**	*Typha*	14	**114**	Wool	16
35	*Typha*	13	**75**	*Typha*	14	**115**	Wool	15
36	*Typha*	13	**76**	*Typha*	14	**116**	Wool	14
37	*Typha*	13	**77**	*Typha*	16	**117**	Wool	14
38	*Typha*	13	**78**	*Typha*	14	**118**	Wool	14
39	*Typha*	13	**79**	*Typha*	14	**119**	Wool	14
40	*Typha*	11	**80**	*Typha*	12	**120**	Wool	14

Obs	fibre	diameter	Obs	fibre	diameter
121	Wool	15	**161**	Wool	12
122	Wool	14	**162**	Wool	12
123	Wool	14	**163**	Wool	12
124	Wool	14	**164**	Wool	12
125	Wool	14	**165**	Wool	12
126	Wool	14	**166**	Wool	11
127	Wool	14	**167**	Wool	11
128	Wool	14	**168**	Wool	12
129	Wool	14	**169**	Wool	12
130	Wool	15	**170**	Wool	12
131	Wool	9	**171**	Wool	11
132	Wool	8	**172**	Wool	12
133	Wool	8	**173**	Wool	12
134	Wool	8	**174**	Wool	12
135	Wool	8	**175**	Wool	12
136	Wool	8	**176**	Wool	12
137	Wool	8	**177**	Wool	12
138	Wool	8	**178**	Wool	12
139	Wool	7	**179**	Wool	12
140	Wool	7	**180**	Wool	14
141	Wool	12	**181**	Wool	13
142	Wool	12	**182**	Wool	14
143	Wool	12	**183**	Wool	14
144	Wool	12	**184**	Wool	14
145	Wool	12	**185**	Wool	14
146	Wool	12	**186**	Wool	11
147	Wool	12	**187**	Wool	11
148	Wool	12	**188**	Wool	11
149	Wool	12	**189**	Wool	14
150	Wool	12	**190**	Wool	14
151	Wool	14	**191**	Wool	15
152	Wool	14	**192**	Wool	15
153	Wool	14	**193**	Wool	15
154	Wool	14	**194**	Wool	14
155	Wool	14	**195**	Wool	14
156	Wool	14	**196**	Wool	13
157	Wool	14	**197**	Wool	13
158	Wool	12	**198**	Wool	14
159	Wool	12	**199**	Wool	13
160	Wool	10	**200**	Wool	13

Appendix IV

Obs	Treatment	Fibre length	Obs	Treatment	Fibre length
1	S8-K3-MS	4.6	**27**	S10-L4-MS	2.7
2	S8-K3-MS	4.1	**28**	S10-L4-MS	1.1
3	S8-K3-MS	3.9	**29**	S10-L4-MS	2.8
4	S8-K3-MS	4.5	**30**	S10-L4-MS	2.1
5	S8-K3-MS	4.5			
6	S8-K3-MS	3.5			
7	S8-K3-MS	4.9			
8	S8-K3-MS	3.6			
9	S8-K3-MS	2.7			
10	S8-K3-MS	2.6			
11	S8-K3-MS	4.6			
12	S8-K3-MS	4.2			
13	S8-K3-MS	4.1			
14	S8-K3-MS	4.6			
15	S8-K3-MS	4			
16	S10-L4-MS	4.1			
17	S10-L4-MS	2			
18	S10-L4-MS	2.3			
19	S10-L4-MS	1.7			
20	S10-L4-MS	1.8			
21	S10-L4-MS	4.8			
22	S10-L4-MS	1.2			
23	S10-L4-MS	2.1			
24	S10-L4-MS	1.6			
25	S10-L4-MS	1.8			
26	S10-L4-MS	2.7			

Appendix V

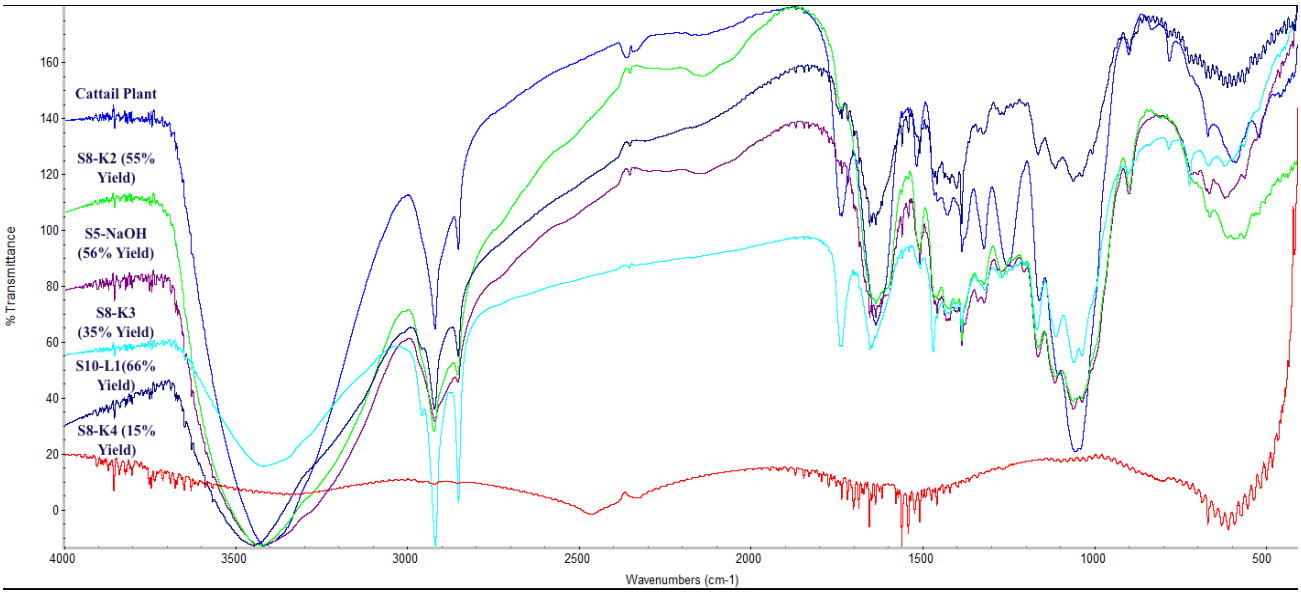


Figure 5.13: FTIR spectrum of extracted fibre from (a) *Typha* plant and (b) S8-K2; (c) S5-NaOH; (d) S8-K3; (e) S10-L1; (f) S8-K4;

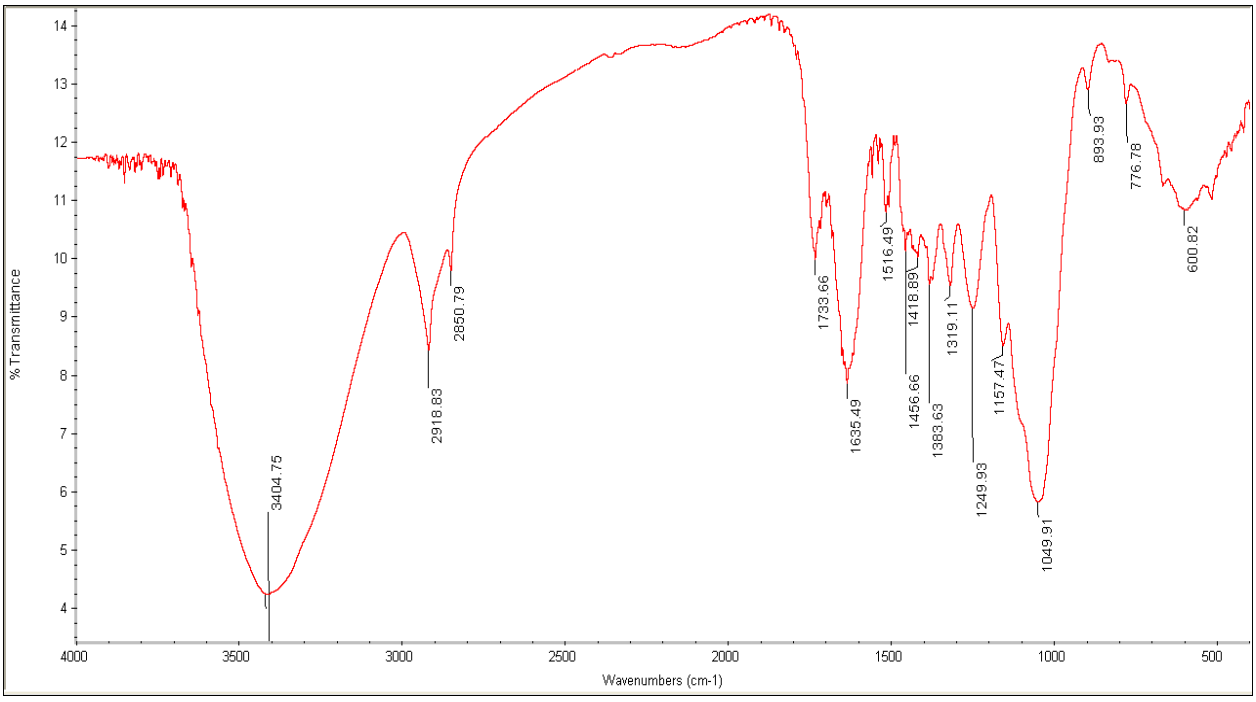


Figure 5.14(a): FTIR spectrum of *Typha* plant.

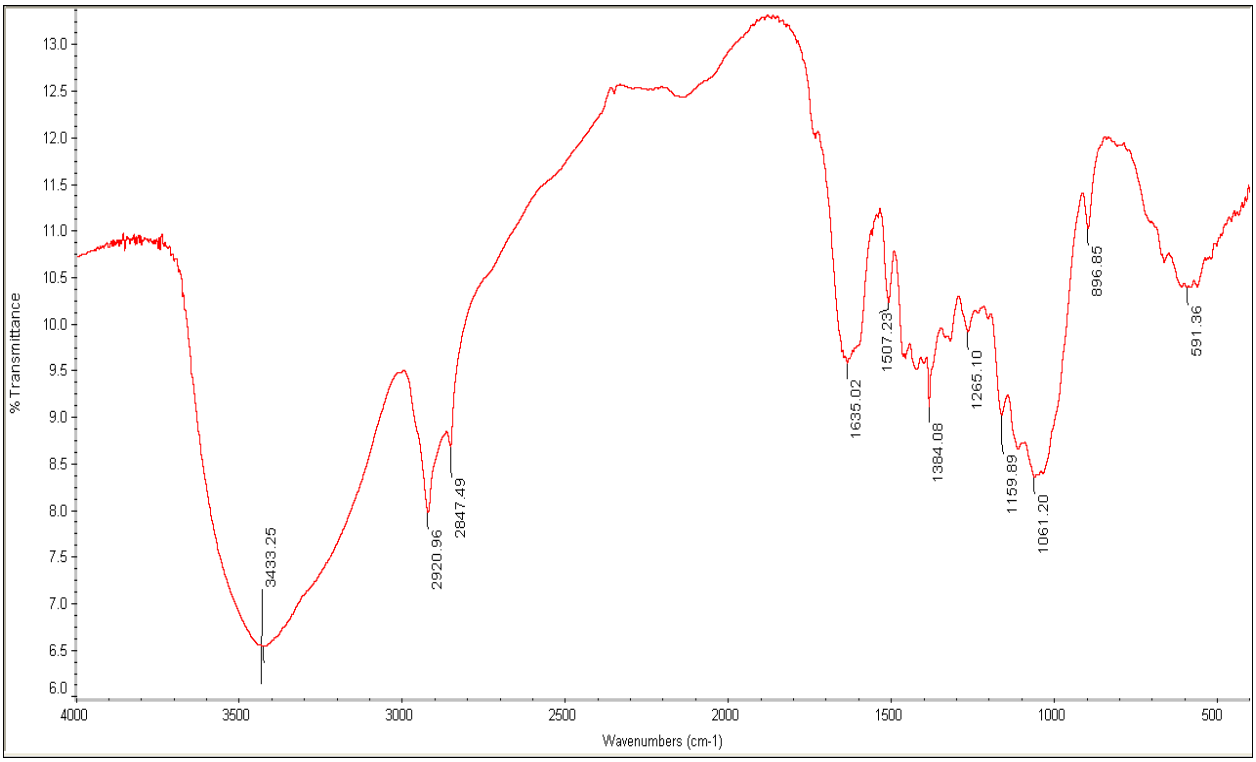


Figure 5.14(b): FTIR spectrum of S8-K2-MS *Typha* fibre.

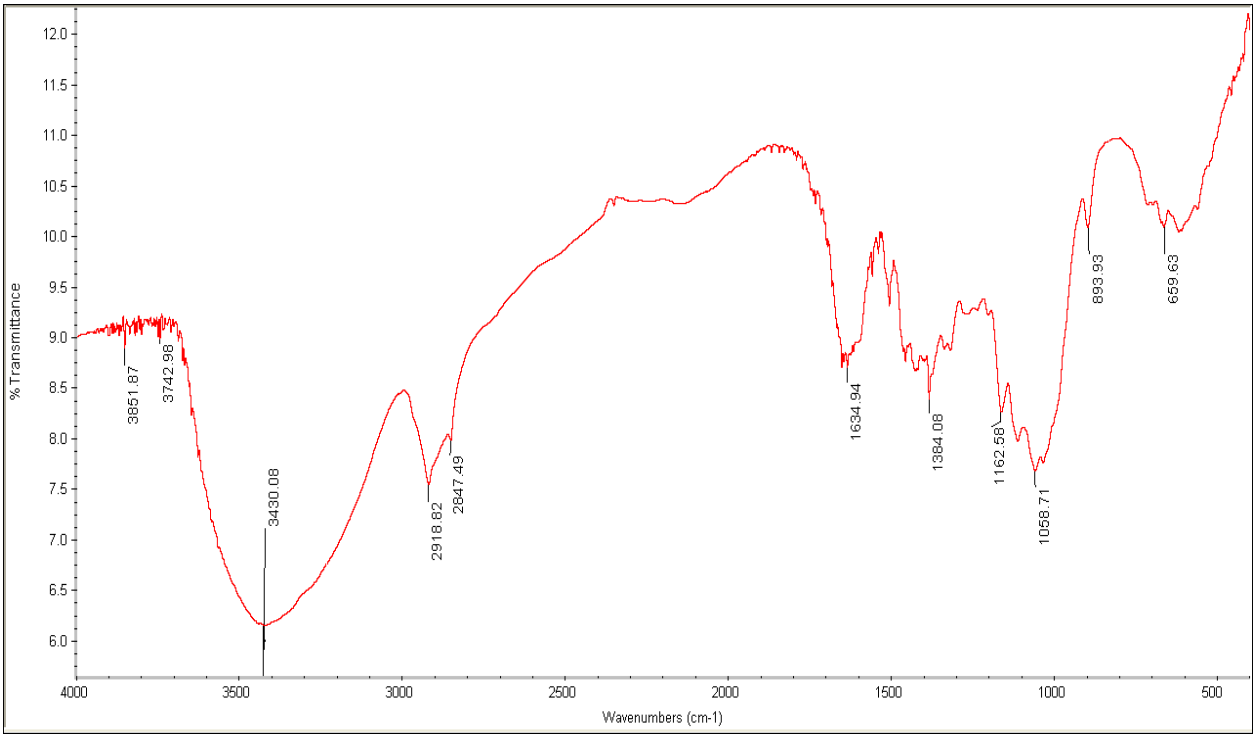


Figure 5.14(c): FTIR spectrum of S5-NaOH-MS *Typha* fibre.

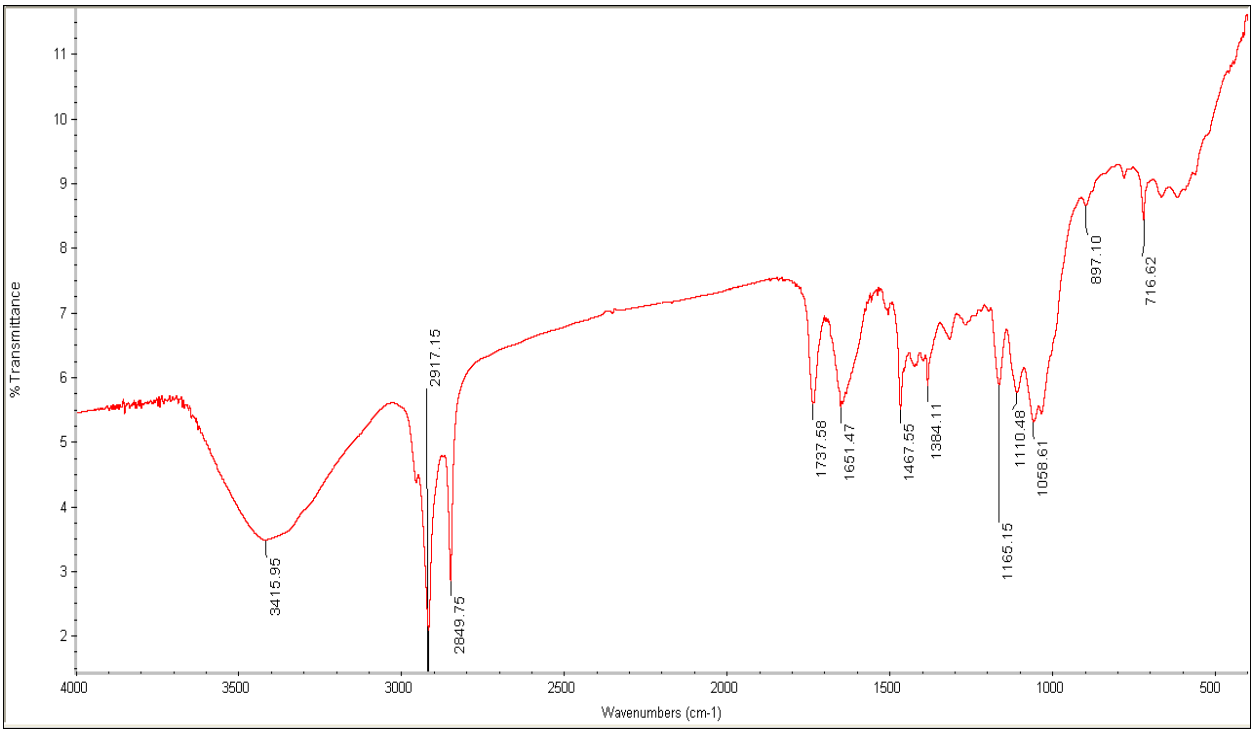


Figure 5.14(d): FTIR spectrum of S8-K3-MS *Typha* fibre.

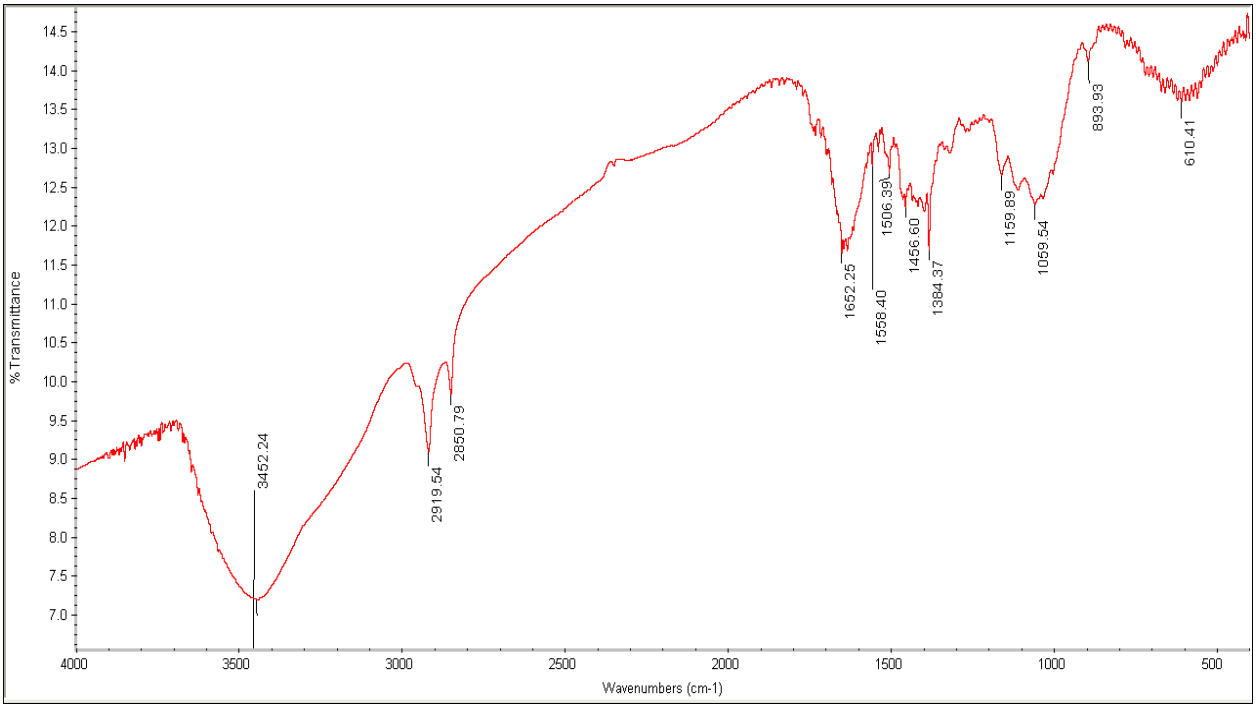


Figure 5.14(e): FTIR spectrum of S10-L1-MS *Typha* fibre.

Appendix VI

Moisture regain (%) of fibres obtain from different sources

Test No	***Typha***	**Wool**	**Cotton**
1	6.03	11.1	7.3
2	8.1	8.8	8.5
3	10.9	11	6.3
4	3.69	15	6.5
5	7.47	7.5	7
6	7.43	10.5	6
7	9.03	9.6	6.1
Avg	7.52	10.5	6.81
SD	2.28	2.37	0.89

Avg: Average, SD: Standard Deviation, Ref: for Cotton (Liggett et al., 1968,)
For Wool (Kopke & Lindberg, (1966), and for *Typha* (experimental data).

Appendix VII

Figure # 5.19 – 5.32 (a)
Holding time (10 minutes)

Figure 5.19 (a)

Figure 5.20 (a)

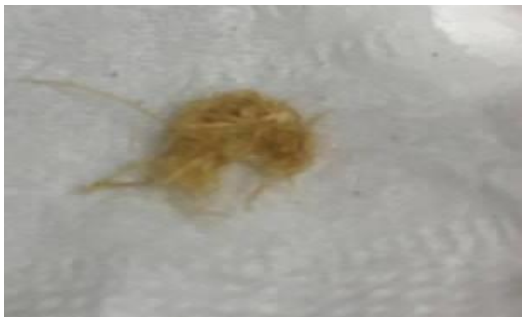


Figure 5.21 (a)

Figure 5.22 (a)

Figure # 5.19 – 5.32 (b)
Holding time (20 minutes)

Figure 5.19 (b)

Figure 5.20 (b)

Figure 5.21 (b)

Figure 5.22 (b)

Figure 5.23 (a)

Figure 5.23 (b)

Figure 5.24 (a)

Figure 5.24 (b)

Figure 5.25 (a)

Figure 5.25 (b)

Figure 5.26 (a)

Figure 5.26 (b)

Figure 5.27 (a)

Figure 5.27 (b)

Figure 5.28 (a)

Figure 5.28 (b)

Figure 5.29 (a)

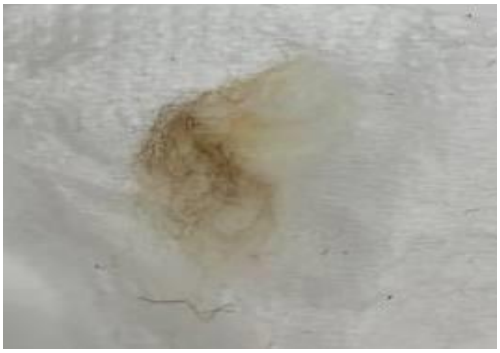


Figure 5.29 (b)

Figure 5.30 (a)

Figure 5.30 (b)

Figure 5.31 (a)

Figure 5.31 (b)

Figure 5.32 (a)

Figure 5.32 (b)

Appendix VIII

Sample Name	RGB	HEX	HSL	Picture
Virgin *Typha* plant	148, 132, 106	#94846a	37, 16%, 49%	
Virgin *Typha* decomposed plant	75, 63, 51	#4b3f33	30, 19%, 24%	
Typha fibre	142, 125, 78	#8e7d4e	44, 29%, 43%	
Typha decomposed fibre	93, 71, 43	#5d472b	33, 36%, 26%	
Virgin Cotton	152, 152, 141	#98988d	60, 5%, 57%	
Decomposed Cotton	78, 63, 44	#4e3f2c	33, 27%, 23%	
TCF1(10)	186, 173, 144	#baad90	41, 23%, 64%	
TCF1(20)	162, 133, 77	#a2854d	39, 35%, 46%	
TCF2(10)	143, 112, 62	#8f703e	37, 39%, 40%	
TCF2(20)	137, 126, 97	#897e61	43, 17%, 45%	
TCF3(10)	121, 92, 44	#795c2c	37, 46%, 32%	
TCF3(20)	87, 65, 38	#574126	33, 39%, 24%	
TCF4(10)	140, 109, 59	#8c6d3b	37, 40%, 39%	
TCF4(20)	82, 60, 37	#523c25	30, 37%, 23%	
TCF5(10)	51, 31, 18	#331f12	23, 47%, 13%	
TCF5(20)	79, 58, 36	#4f3a24	30, 37%, 22%	
TCF6(10)	46, 26, 10	#2e1a0a	26, 64%, 10%	

TCF6(20)	43, 27, 18	#2b1b12	21, 40%, 11%	
TCF7(10)	19, 13, 13	#130d0d	0, 18%, 6%	
TCF7(20)	21, 16, 17	#151011	348, 13%, 7%	
TCO1(10)	164, 164, 156	#a4a49c	60, 4%, 62%	
TCO1(20)	129, 130, 125	#81827d	71, 1%, 50%	
TCO2(10)	164, 164, 155	#a4a49b	60, 4%, 62%	
TCO2(20)	130, 132, 123	#82847b	73, 3%, 50%	
TCO3(10)	156, 156, 142	#9c9c8e	60, 6%, 58%	
TCO3(20)	131, 132, 126	#83847e	70, 2%, 50%	
TCO4(10)	108, 93, 71	#6c5d47	35, 20%, 35%	
TCO4(20)	124, 106, 76	#7c6a4c	37, 24%, 39%	
TCO5(10)	94, 66, 41	#5e4229	28, 39%, 26%	
TCO5(20)	103, 90, 68	#675a44	37, 20%, 33%	
TCO6(10)	60, 44, 29	#3c2c1d	29, 34%, 17%	
TCO6(20)	54, 40, 30	#36281e	25, 28%, 16%	
TCO7(10)	41, 29, 19	#291d13	27, 36%, 11%	
TCO7(20)	30, 19, 16	#1e1310	12, 30%, 9%	

Appendix IX

Evaluation of softness grading with *Typha* and other common fibres.

	Evaluators No																			
	1				**2**				**3**				**4**				**5**						
Name of Fibres																			**Avg**	**Std**	**Yield (%)**
Cotton	4				4				4				4				4		4	0.0	N/A
Wool	3				3				3				3				3		3	0.0	N/A
Nylon	2				2				2				2				2		2	0.0	N/A
Acrylic	4				4				5				3				3		3.8	0.81	N/A
Silk	4				5				5				5				5		4.8	0.44	N/A
Polyester	5				5				5				4				4		4.6	0.54	N/A
Cannabis (virgin)	1				1				1				1				1		1	0.0	12-15
Brassica (virgin)	1				1				1				1				1		1	0.0	10-15
Typha latifolia L. (Virgin)	1				1				1				1				1		1	0.0	N/A
Typha latifolia L. (S5-NaOH-MS)	2				2				2				1				2		1.8	0.44	56
Typha latifolia L. Fibre (S8-K3-MS)	3				3				4				2				2		2.8	0.83	35
Typha latifolia L. (S10-L3-MS)	2				1				3				1				1		1.6	0.89	56

Appendix X

FORMULA

The pooled estimate of the sample standard deviation

$$S = \sqrt{\left[\frac{(n_1-1)S.D.1^2+(n_2-1)S.D.2^2}{n_1+n_2-2}\right]}$$

Calculate the value t from the equation

$$t = \left|\frac{X_1-X_2}{s\sqrt{(\frac{1}{n_1}+\frac{1}{n_2})}}\right| = 2.40 \text{ (Approx.)}$$

Standard error of mean difference (1-2) = $\sqrt{(\frac{S_1^2}{n_1} + \frac{S_{12}^2}{n_2})}$

Pooled Variance $(S_p)^2 = \frac{(n-1)S_1^2+(n-1)S_2^2}{n_1+n_2-2}$

Pooled Standard Deviation $\sqrt{(S_p)^2}$

Degree of freedom for Satterthwaite adjustment: $d_f = \frac{(\frac{S_1^2}{n_1}+\frac{S_2^2}{n_2})^2}{\frac{1}{n_1-1}(\frac{S_1^2}{n_1})^2+\frac{1}{n_2-2}(\frac{S_2^2}{n_2})^2}$

T-test formula for unequal variances: $t_{cal} = \frac{(\overline{X_1}-\overline{X_2})}{\sqrt{\frac{S_1^2}{n_1}+\sqrt{\frac{S_2^2}{n_1}}}}$

T-test formula pooled method: $t_{cal} = \frac{(\overline{X_1}_- \overline{X_2})}{\sqrt{\frac{2S_p^2}{n}}}$