Optimization of *Typha* Fibre Extraction and Properties for Composite Applications Using Desirability Function Analysis

by

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Abstract

Recent studies on *Typha* spp. commonly-known as cattail has revealed its prospect of being used as a new source of natural fiber. An investigation was carried out to examine the influence of extraction time, temperature, and concentration of alkali on the physical and mechanical properties of cattail fibers for bio-composite applications. The fibres were obtained from the leaves of *Typha latifolia* L. using 3 levels of concentration (4, 7, 10%, w/v) of NaOH with 4 levels of temperature (70, 80, 90, and 95° C), and 5 levels of time (4, 6, 8, 10, and 12 h.) in a 3*4*5 factorial experimental design. The three-way factorial ANOVA revealed that yield (%), diameter, tensile strength, elongation at break (%), and moisture regain (%) of fibres were influenced significantly ($P < 0.05$) by time, temperature, concentration and the interaction between temperature and concentration. For modulus of elasticity of fibres, the effects of time, temperature, the interaction between time and concentration, and the interaction between temperature and concentration were found significant ($P < 0.05$).

After analyzing the estimated treatment means of response variables, it was determined that the mechanical properties of cattail fibres have similarities with sisal, and coir fibres and can be used in automobile and packaging applications. The extraction parameters were optimized using desirability function analysis (DFA) which determined the optimum extraction time, temperature and concentration of NaOH as 10 h., 90º C, and 7% for automobile applications and 6 h., 90º C, and 7% for packaging applications.

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Abbreviations

American Association of Textile Chemists and Colorists (AATCC)

American Society for Testing and Materials (ASTM)

Analysis of Variance (ANOVA)

Desirability Function Analysis (DFA)

International Organization for Standardization (ISO)

Sodium Tripolyphosphate (STPP)

Ethylenediaminetetraacetic Acid (EDTA)

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1. CHAPTER: INTRODUCTION

The term ‘textile’ refers to the materials made from fibres, filaments or thin threads which are natural or synthetic or a combination of these two. Example of textiles includes apparel, rugs, car seat upholstery, protective clothing, geotextiles, fibre-reinforced composites etc. Morton and Hearle (2008) defined that in order to be used for textile purposes, the fibres or filaments should possess a certain minimum strength, extensibility and an adequately high temperature stability.

A scheme for the classification of textile fibres is shown in Figure 1.1 (Chakma, 2018; M. R. I. Khan, 2016; Kozlowski, 2012; Robertson, Roux, & Wiggins, 1999) . The classification starts by dividing all fibres into two types i.e. natural fibres and man-made fibres. Nature offers a vast source of fibrous materials from which fibres can be extracted. Natural fibres can be subdivided based on the sources of fibres, and their compositions. Man-made fibres are subdivided based on the fibre forming polymers.

People from multiple early civilizations are known to use fibres obtained from nature to meet the basic requirements of clothing, building material, storage, and for accessories such as fishing nets and ropes (Kozlowski, 2012). Kozlowski (2012) reported that the use of cotton started in ancient Egypt at around 12000 BC, and in India the cotton spinning and weaving industry began as early as 1500 BC. Flax fibre was discovered at around 6500 BC and silk a natural protein fibre were discovered in China at around 3000 BC. Natural fibres have been used for clothing and other end uses for centuries until the first man-made fibre was produced commercially in 1885. However, during the early 1900s the use of man-made fibre was minimal and the demand started to grow rapidly since the mid-1900s due to the invention of nylon and polyester (Kozlowski, Muzyczek, & Mackiewicz-Talarczyk, 2013).

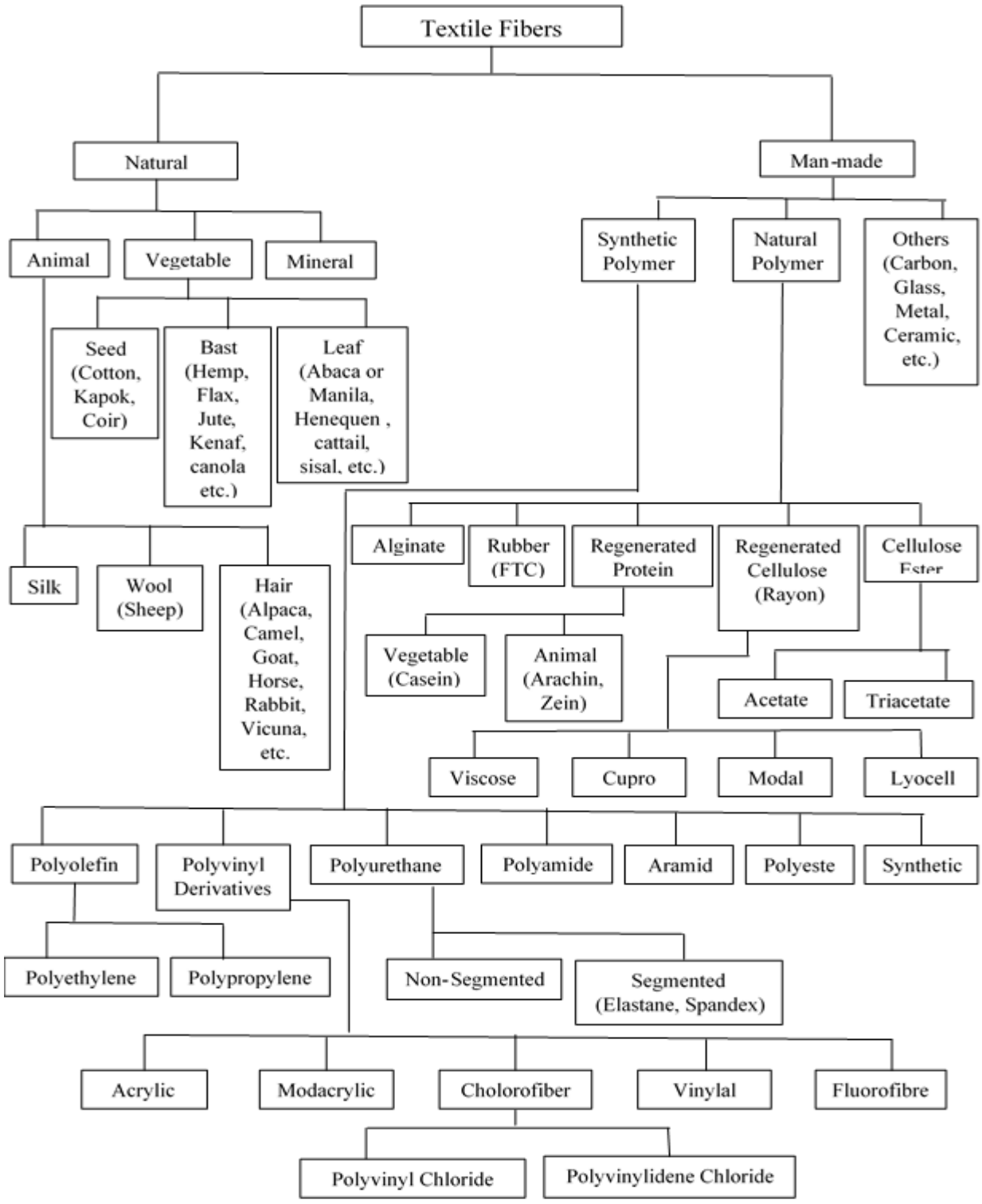

Figure 1.1: Classification of textile fibres 1 (Chakma, 2018; M. R. I. Khan, 2016; Kozlowski, 2012; Robertson, Roux, & Wiggins, 1999)

1.1 Current Trend in Textile Industry

At present, the textile industry is one of the largest global industries and is expected to grow continuously in the coming years. In 2017, the value of global apparel retail market alone consisting menswear, womenswear, and childrenswear totaled 1,414.1 billion USD and is predicted to reach $1,834 billion USD in value in 2022 (Lu, 2018).

Moreover, the market value of technical textiles representing non-aesthetic functional textile products was calculated as 147 billion USD based on raw material consumption in 2014, which was a 6% increase per annum in value between 2010 to 2014 (Delden & Aucouturier, 2016). The report also forecasted a growth rate of 5% per year in market value for this industry from 2014 to 2020.

This explosion of consumption of textiles worldwide has resulted a remarkable expansion of the usage of textile fibres (Collier, Bide, & Tortora, 2008; M. R. I. Khan, 2016). Textile Exchange (2019) reported the global fibre production surpassed 100 million metric ton in 2017, which is a tenfold increase from 1950 to 2017. Figure 1.2. shows the percentage of different types of fibres produced in 2017 for textile applications (Textile Exchange, 2019).From the figure (1.2) it can be seen that the synthetic fibres made up approximately 62% of the global fibre production in 2017. Synthetic fibres become dominant in the fibre market since the mid-1990s when their demand passed cotton (Carmichael, 2015). Carmichael also forecasted that the manufacture of synthetic fibres will exceed 70% of the total fibre production by the year 2030.

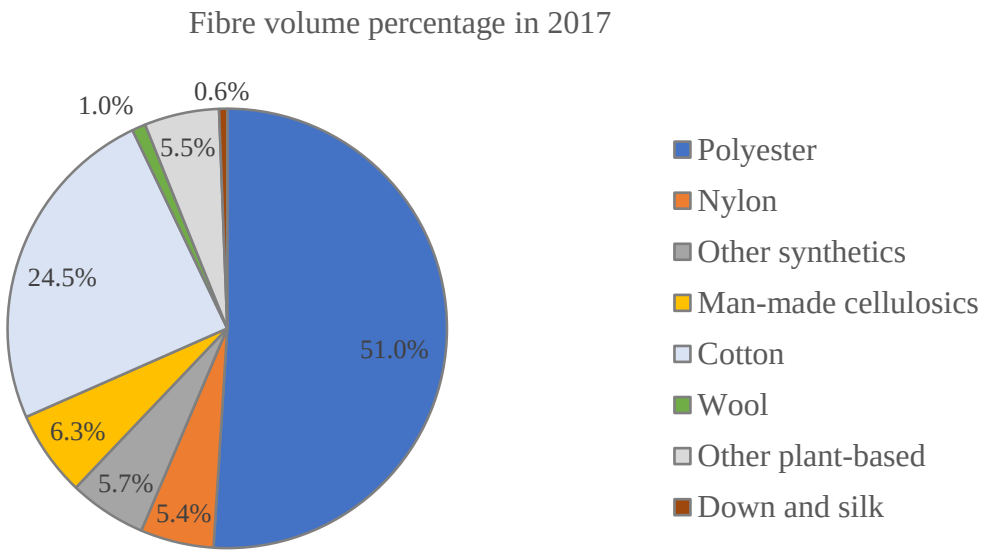

Figure 1.2: Volume percentages of different types of fibres produced in 2017 (Figure has been constructed by the author, % of fibre obtained from the Textile Exchange, 2019)..

1.2 Pollution from Synthetic Fibres

The rapid expansion of the consumption of synthetic fibre is causing increasing negative environmental impacts. Although synthetic fibres are more durable, elastic, higher stain resistant, less expensive, and easy to manufacture compared to natural fibres, they are non-biodegradable causing long-term pollution and their manufacturing processes contribute an enormous amount of chemical waste and carbon emissions (Collier et al., 2008; M. R. I. Khan, 2016). At present, polyester and nylon are the two most widely used synthetic fibres (as shown in Figure 1.2, section 1.1.1) which are manufactured from polymer solutions obtained from the biproduct of non-renewable petroleum resources (Deopura, Alagirusamy, Joshi, & Gupta, 2008). These fibres are nonbiodegradable and their manufacturing requires a huge amount of energy consumption (Barber & Pellow, 2006). Barber and Pellow (2006) reported that polyester, Nylon 6, and Nylon 6,6 require 125 MJ, 138.65 MJ, and 120.47 MJ of energy respectively to produce per Kg of fibre whereas

conventional cotton requires only 60 MJ of energy per Kg of fibre. During the manufacture of polyester, along with the large amount of waste gas, 1-4 dioxane, a human carcinogen is formed as a biproduct and the manufacture of nylon unleashes nitrous oxide, a substance partly accountable for diminishing earth's ozone layer (Chen & Burns, 2006; Deopura et al., 2008).

Synthetic fibres are considered as one of the primary sources of microplastics in marine and freshwater environments (Dris, Gasperi, Saad, Mirande, & Tassin, 2016). These microplastics are very small fragments of plastics with a diameter < 5 mm and considered a widespread singular contaminant in marine and freshwater ecosystems (Dris et al., 2016, 2015; Vegter et al., 2014). These microplastics are ingested not only by the vertebrates and other higher organisms in these aquatic environments, but also due to their smaller sizes, ingested by the low trophic organisms and therefore, incorporated in the food chain (Wright, Thompson, & Galloway, 2013). Oehlmann et al., (2009) and Talsness et al., (2009) reported that this accumulation of microplastics within organisms may result in physical harm such as internal abrasions and blockages. Furthermore, they found that due to the ingestion of microplastics, toxicity could arise from leaching constituent contaminants such as monomers and plastic additives, capable of causing carcinogenesis and endocrine disruption. These huge negative health and environmental impacts resulting from the manufacturing process and the usage of synthetic fibres have drawn attention on the development of fibres which are compatible with the environment.

1.3 Drawbacks of Natural Fibres

Natural fibres are bio-degradable and come from renewable resources, but their manufacturing processes are not free from negative environmental impacts (Chen & Burns, 2006; Richardson & Gorton, 2003). Muthu, Li, Hu, & Mok (2012) developed a unique model to rank textile fibres based on ecological sustainability which showed that organic cotton is the most preferred fibre

followed by flax, cotton, viscose, wool, polyester, and nylon. The authors concluded that although natural fibres are more preferable than synthetic fibres, certain negative environmental impacts are associated with each natural fibre affecting sustainability.

The most dominant natural fibre is cotton which held a total market share of 24.5% of the world textile fibre consumption in 2017 (Textile Exchange, 2019). The conventional cotton plant is very susceptible to attack by certain insects and fungi and requires a high volume of pesticides and fungicide applications for its growth (Chen & Burns, 2006). According to a report published by the Environmental Justice Foundation (2007), cotton accounted for 16% of the pesticides application worldwide although 2.5% of the arable land were cultivated for cotton. Also, Kalliala & Nousiainen (1999) reported that the manufacturing of cotton fibres necessitate the consumption of large quantity of water i.e. 22,000 Kg of water required per Kg of cotton fibre and therefore, generating enormous amount of effluents. The overall manufacturing process of conventional cotton fibre from cradle to gate emits more CO_2 (6 kg per Kg of fibre) than polyester (2.8 Kg per Kg of fibre) (Boustead, 2005; Kalliala & Nousiainen, 1999). Therefore, despite the 'natural' image, cotton production is associated with severe negative environmental impacts. Although few environmentally friendly cotton varieties have been introduced to the market i.e. organic cotton, their percentage in the total cotton production is nominal (Chen & Burns, 2006).

Wool is the second most important natural fibres obtained from the hair of animals, mostly sheep (Grayson, 1984; Textile Exchange, 2019). Although wool is easily recyclable and does not require the use of fertilizers or herbicides like cotton, the manufacturing of wool has some negative impacts on the environment (Chen & Burns, 2006). Overgrazing by sheep may cause soil erosion if not controlled properly, and runoff contamination can be created due to excessive sheep manure (Kadolph & Langford, 1998). Also, during the processing of wool, soap and alkaline solutions are

required to clean the fibres and remove grease and impurities (Collier, Bide, & Tortora, 2009). Furthermore, different chemicals are used on wool fabrics to prevent shrinkage, and ensure machine washability, therefore, creating significant amount of hazardous effluents (Chen & Burns, 2006).

Moreover, present natural fibres often lack the required properties necessary for specific biomedical and industrial applications. Biomedical applications such as artificial ligaments, and vascular grafts require the fibres to have long-term mechanical stability, higher elastic recovery, and abrasion resistance. The present natural fibres have significant deficiencies in those properties (Ratner, Hoffman, Schoen, & Lemons, 2004). However, natural fibres due to their inherent biocompatibility can regulate the host response resulting from the in vivo implanted biomaterial in a better way than synthetic fibres (Lee & Mooney, 2012; Ratner et al., 2004). Furthermore, in fibre reinforced composites for industrial applications, the main challenges of using natural fibre as a reinforcement are poor water resistance, low durability, and poor fibre-matrix interfacial bonding which cause a diminution in final properties of the composites and ultimately inhibit their industrial use (Milanese, Cioffi, Jacobus, & Voorwald, 2011; Mohanty, Misra, & Drzal, 2001). Nonetheless, despite the disadvantages of natural fibres, they are being investigated as a potential reinforcement for composites because of their low cost, high strength to weight ratio, low density, biodegradability, and recyclability (Van Voorn, Smit, Sinke, & De Klerk, 2001; Wallenberger & Weston, 2004).

1.4 New Sources of Natural Fibres

The present limitations of the existing natural fibres have accelerated the research activities to examine the feasibility of less common natural fibres such as okra fibres (De Rosa, Kenny, Puglia, Santulli, & Sarasini, 2010), bamboo fibres (Kushwaha & Kumar, 2011), palm fibres (Sghaier,

Zbidi, & Zidi, 2009), pineapple-leaf fibres (Kannojiya, Gaurav, Ranjan, Tiyer, & Pandey, 2013), and cattail fibres (Chakma, 2018; Sayed Majid Mortazavi & Moghadam, 2009) for textile applications and some of these fibres have shown potential of becoming a new source of natural fibres. This thesis focuses on cattail fibres.

1.4.1 Cattail Fibres

Typha spp. commonly known as cattail are perennial herbs growing abundantly in swamplands and near the edge of ponds and lakes and becoming increasingly dominant wetland plants in North America (Shih & Finkelstein, 2008). At present, three species of *Typha* are commonly found in this region. *Typha latifolia* L., also known as broad-leaf cattail which is the most common type ranging almost all of North America. *Typha angustifolia* L. also known as narrow-leaf cattail which is less widely distributed than the broad-leaved cattail but is rapidly increasing its range. And the hybrid between these two species *Typha x glauca* is common in the region where both the parent species are found (Selbo & Snow, 2004; Shih & Finkelstein, 2008). The chemical composition of cattail (*T. latifolia*) consists of primarily 63% cellulose, 8.7% hemi- cellulose, 9.6% lignin and pectin and other water soluble matters such as 1.4% wax and 2% ash (Vetayasuporn, 2007).

High productivity, abundance of cellulose present in the chemical composition, and physical, chemical, and thermal properties have made *Typha latifolia* L. (broad-leaf cattail) a potential source of new natural fibre for textile applications (Chakma, 2018; Sana, Mounir, & Slah, 2014). After analyzing the fibre characteristics, it was found that the cattail fibres obtained from the broad-leaved cattail possess similar characteristics to commonly used cellulosic fibres in terms of fibre diameter, moisture regain, mechanical properties, thermal properties, and burning behavior

(Bajwa, Sitz, Bajwa, & Barnick, 2015; Chakma, 2018). These fibre properties are essential for textile applications specially for fibre-reinforced composites. (Wallenberger & Weston, 2004).

Nonetheless, these properties change significantly with the change in fibre extraction conditions (Chakma, 2018; Sana et al., 2014) and no studies have been done so far to determine the effects of treatment temperature, concentrations of alkali, and the treatment duration on fibre characteristics and fibre yield (%). This knowledge is significant to set the production parameters to extract cattail fibres of desired characteristics for a specific textile application such as fibre-reinforced composites.

1.5 Scope of the Thesis

Hence, this thesis is focused on extracting fibres from broad-leaf cattail (*Typha latifolia* L.) using different extraction time, temperature, and concentrations of alkali to determine their effects on fibre yield (%), and fibre properties, and optimizing the extraction parameters to produce fibres of required characteristics for composite applications.

2. CHAPTER: LITERATURE REVIEW

2.1 Fibre Reinforced Composites

The term ‘composite’ can be defined as an assembly of different materials which when used together exhibit properties that is not attainable by either constituent material when applied individually (Abdussalam, 1999). Composites usually consist of one or more discontinuous phases bonded to a continuous phase (Agarwal & Broutman, 1990). This discontinuous phase which is usually harder and stronger, is called the reinforcement and the continuous phase is called the matrix. Figure 2.1. represents a frequently used classification for composite materials (Agarwal & Broutman, 1990) . With respect to this classification, particulates can be defined as materials nonfibrous in nature. They can be spherical, cubic, tetragonal or of other regular or irregular shapes but are approximately equiaxed. Composite laminates can be defined as an assembly of fibrous materials attached together to provide required engineering properties. The geometry of the reinforcements in composites strongly affects their strengthening mechanisms and therefore, it is common to classify composite materials based on the geometry of a representative unit of reinforcements (Agarwal & Broutman, 1990).

For the last few decades, the use of composite materials is spreading rapidly in engineering and medical applications and the desire for lightweight, yet stronger materials have accelerated the development of fibre-reinforced matrix composites (Wambua, Ivens, & Verpoest, 2003). In these composites, the fibres of high strength and modulus are embedded in or bonded to a matrix material maintaining definite interfaces. Although both the fibers and the matrix preserve their identities, they exhibit properties that are not attainable by either of the constituents acting alone.

.

Figure 2.1: Classification of composite materials (Adapted from Agarwal & Broutman, 1990, permission not obtained)

In general, the fibers are the prime constituents of the fibre-reinforced composites which provide strength and stiffness and carry majority of the applied load. They also occupy a substantial portion of the volume fraction in the laminate (Abdussalam, 1999). Most commonly used fibers for reinforcements are glass, carbon, and aramid (Wambua et al., 2003)

The matrix in fibre-reinforced composites serves as a binding material which uses its cohesive and adhesive properties to bind the fibres together in the structural unit. Moreover, in addition to contribute some required characteristics of the composites e.g. ductility, toughness or electrical insulation, the matrix distributes the applied loads to and between the fibers and protect them from

external damages (Gibson, 1994). Therefore, it is preferable that the matrix forms a mechanical or chemical bond with the fibres so that a strong interface strength between the fibers and the matrix can be developed (Agarwal & Broutman, 1990).

Due to the strong influence of the matrix on certain mechanical characteristics of composites such as modulus, strength, shear properties, and compressive properties, the selection of matrix is an important step in manufacturing fibre-reinforced composites (Abdussalam, 1999). There are generally four types of matrices used in composites i.e. polymeric, metallic, ceramic, and carbon. In fibre-reinforced composites, polymeric matrices are most commonly used which can be further classified into two types i.e. thermosets (epoxies. Polyimide and polyester), and thermoplastics (polyethylene, polystyrene, and nylons). Thermoset polymers are highly thermal resistant, and chemically stable but they require long fabrication time in the mold and have low strain to failures. On the other hand, thermoplastics have higher strains to failure and require shorter processing time, but they exhibit higher creep and stress relaxation than thermoset polymers (Abdussalam, 1999; Agarwal, Bhagwan D., Lawrence J. Broutman, 1990).

2.1.1 Natural Fibre Reinforced Composites

As stated earlier, synthetic fibers specially glass fibres due to their low manufacturing cost (compared to aramid, and carbon) and excellent mechanical properties are the most widely used to reinforce polymer composites. However, natural fibers as an alternative reinforcement in polymer composites have drawn attention in recent decades (Ku, Wang, Pattarachaiyakoop, & Trada, 2011). These natural fibers including flax, hemp, jute, sisal, kenaf, coir, kapok, and banana have distinct advantages over conventional glass and carbon fibres. Table 2.1 compares natural and glass fibres and shows areas in which natural fibres are superior in characteristics (Ku et al., 2011; Wambua et al., 2003).

Table 2.1: Comparison between natural fibers and glass fibers (Ku et al., 2011; Wambua et al., 2003).

Criteria	Natural Fibres	Glass Fibres
Cost	Low	Higher than natural fibres
Density	Low	Twice that of natural fibres
Disposal	Biodegradable	Not biodegradable
Recyclability	Yes	No
Energy consumption	Low	High
Renewability	Yes	No
Health risk when inhaled	No	Yes
Abrasion to machines	Non-abrasive	Abrasive

A comparison between the mechanical properties of natural fibres and E-glass fibres is shown in table 2.2. (Ku et al., 2011; Malkapuram, Kumar, & Singh Negi, 2009; Wallenberger & Weston, 2004; Wambua et al., 2003). Although natural fibres have lower mechanical properties, they are 50% lighter than glass fibres and generally cheaper (Wambua et al., 2003). These natural fibre reinforced composite materials are applicable for aerospace, leisure, construction, sport, packaging and especially automotive industries (Ku et al., 2011; Wallenberger & Weston, 2004).

However, the main drawback of using natural fibre reinforced polymer composites is the incompatibility between the hydrophobic polymer matrices and the hydrophilic natural fibres which leads to weak bonding between the fibre and the matrix (Ku et al., 2011; Malkapuram et al., 2009). Therefore, it is necessary to modify the surface of the fibre to enhance the adhesion between the fibre and the matrix (Malkapuram et al., 2009; Wambua et al., 2003). Fibre content or amount of fillers is another important factor which significantly affects the tensile properties of composites. It was observed that the increase in fibre content in composites leads to an increase tensile properties (Ahmad, Baharum, & Abdullah, 2006). Furthermore, processing parameters

significantly influence composite characteristics and therefore, it is necessary to carefully choose processing parameters for optimum composite properties (Ku et al., 2011).

Table 2.2: Comparison between the mechanical properties of natural fibres and E-glass fibres (Ku et al., 2011; Malkapuram et al., 2009; Wallenberger & Weston, 2004; Wambua et al., 2003).

	Fibres					
Properties	Glass	Hemp	Flax	Jute	Sisal	Coir
Density	2.55	1.48	1.4	1.46	1.5	1.25
Tensile strength (MPa)	2400	690	600 - 1200	400 - 800	350 - 370	100 - 175
Elastic modulus (GPa)	73	70	27.6	10 -30	9.4 - 19	6
Elongation at break (%)	3	1.6 – 4.5	1.2 – 3	1.8	1.9 - 3	15 - 20
Moisture absorption (%)	-	8	7	12	11	10

2.2 Important Fibre Properties for Composite Applications

In most cases, the mechanical properties of fibres are significantly higher than the un-reinforced resin systems used to manufacture composites. Therefore, the mechanical properties of the fibre-reinforced composites are dominated by the contribution of fibres in the composites. The main factors that control fibres' contribution in the composites are discussed below.

2.2.1 The Basic Mechanical Properties of the Fibres

The basic mechanical properties of the fibres i.e. tensile strength, modulus of elasticity, and elongation at break significantly influence the mechanical properties of composites. The tensile strength measures the force require to pull the fibre to the point where it breaks. However, in most physical and engineering applications, the force is replaced by stress which is defined by the following equation.

$$\text{Stress} = \frac{\text{Load}}{\text{Area of cross-section}} \ldots\ldots\ldots\ldots\ldots\ldots (2.1)$$

Therefore, the tensile strength of fibres can be defined as the maximum amount of stress it can endure before failure. The modulus of elasticity is a mechanical property which measures the stiffness of a material. It is the ratio of stress to strain (proportional deformation) as shown in equation (2.2) in a material within the elastic limit of a uniaxial deformation.

$$\text{Modulus of elasticity} = \frac{\text{Uniaxial stress}}{\text{Strain or proportional deformation within the elastic limit}} \ldots\ldots\ldots (2.2)$$

The elongation at break also known as fracture strain is related to the ability of a material to resist deformation without breaking. It can be defined as the ratio between increase in length and initial length of the material after breakage as shown in equation (3). Elongation at break is usually measured in percentage.

$$\text{Elongation at break (\%)} = \frac{\text{Increase in length}}{\text{Initial Length}} \times 100 \ldots\ldots\ldots\ldots\ldots\ldots (2.3)$$

In general, higher mechanical properties of the reinforcing fibres will produce composites having higher mechanical characteristics (Agarwal & Broutman, 1990; Wallenberger & Weston, 2004). For applications such as in aircraft and aerospace industries require high performance composites with excellent mechanical properties and carbon and glass fibres are commonly used as a reinforcing fiber because of their higher mechanical strength, and modulus. At present, the strength of the natural fibres are considerably weaker than the conventional synthetic reinforcing fibres (glass, carbon, and aramid) and this is one of the primary reasons why conventional synthetic fibers cannot be fully replaced by natural fibres as reinforcements (Hamidon, Sultan, Ariffin, & Shah, 2019).

2.2.2 The Amount of Fibres in the Composite

The amount of fibre in the composite, also known as the fibre volume fraction (V_f), is the percentage of the fibre volume in the total volume of a fibre-reinforced composite material. It is an important composite property which significantly affects the ultimate strength, stiffness, toughness, and failure mode of a composite (Abdussalam, 1999; Ciprian, Radu, & Ioana, 2015). In general, by increasing the fibre volume fraction, the mechanical properties of a fibre-reinforced composite can be improved (Ciprian et al., 2015; Rejab, Theng, Rachman, Noor, & Rose, 2008).

In most cases, the amount of fibre in a composite is primarily governed by the manufacturing process used. However, the reinforcing fabrics used in the composite is also an important factor. In the composite laminate, reinforcing fabrics consist of closely packed fibres will have higher fibre volume fraction than fabrics consist of coarser fibres or which have wide gaps between the fibre bundles (Ciprian et al., 2015). The fibre diameter is another important factor here as smaller diameter fibres provide higher surface areas and distribute the fibre-matrix interfacial loads more properly which contribute to the higher mechanical properties of the composites (Agarwal & Broutman, 1990; Kim & Mai, 1998).

Generally, the increase in the fibre volume fraction will improve the stiffness and strength of the composite. However, if the amount of fibre in the composite is too large, it will degrade the ultimate strength of the composite. When the fibre volume fraction in a composite is higher than 60-70%, the amount of matrix used will not be sufficient to hold the fibres together properly. Therefore, the laminate's ultimate strength, after reaching a peak, will start to decrease (Ciprian et al., 2015; Kim & Mai, 1998).

2.2.3 The Orientation of Fibres in the Composite

The mechanical properties of the composites are highly dependent on the orientation of the reinforcing fibres. Composites are usually designed to carry the load along the length of the fibres and a change in the direction of load will significantly reduce the strength of the composites (Abdussalam, 1999; Agarwal & Broutman, 1990). This 'anisotropic behavior' of the composite materials necessitate the majority of the fibres being placed parallel to the direction of the main loads. This important feature can be utilized to design more efficient composites by minimizing the amount of reinforcing material put in the directions where there is little or no load (Thomas & Pham, 2018).

2.2.4 Moisture Absorption of Fibres in the Composite

When a composite is exposed to a moist environment, it may absorb or lose moisture depending on the environment and the conditions of the structural components. The percent moisture content or the percent weight gain can be calculated using the following equation.

$$\text{Moisture Regain (\%)} = \frac{\text{Weight of Moist Material - Weight of Dry Material}}{\text{Weight of Dry Material}} \times 100 \ldots\ldots (2.4)$$

In fibre-reinforced polymer composites, the absorbed water not only produces changes to the physical and chemical nature but also degrades the mechanical properties of the materials (Wang & Chang, 1983). This degradation of strength and stiffness is caused by the weakening of three composite phases i.e. the fibre, the matrix, and the interphase (Ishai, 1975; Wang & Chang, 1983). The degree of degradation is governed by the moisture content in the composite material and also depends on the constituents and the quality of the composite (Wang & Chang, 1983).

Currently, the fibre-reinforced composites have a wide range of applications in industries such as aerospace, marine, transportation etc. Therefore, the degradation of mechanical properties in composites due to moisture absorption cannot be ignored in the design of the composite structures specially for the applications where they will be subjected to the humid environment for a long period of time. In general, natural fibres are more susceptible to moisture absorption than synthetic fibres when exposed to external conditions (temperature and humidity) and therefore, various surface modification treatments such as plasma treatment, alkali treatment, and silane coupling agents treatment have been reported in the literature to reduce the moisture absorption, and improve the fibre-matrix interface bonding of natural fibres for composite applications (Mokhothu & John, 2017; Xie, Hill, Xiao, Militz, & Mai, 2010).

2.3 Applications of Fibre-reinforced Composites

2.3.1 Synthetic Fibres

The major characteristics and applications of the widely used synthetic fibres used in reinforcing composites are briefly described below.

2.3.1.1 Carbon Fibres

Carbon fibres are dominant high strength, high modulus, and unidirectional reinforcements used to manufacture high performance polymer matrix composites. It is predominantly used in sporting goods, aerospace, and automobiles due to its high strength and modulus, low density, and high temperature resistance (Ali Munawar, Taj, & ullah Khan, 2007).

2.3.1.2 Glass Fibres

Glass fibres are the most widely used reinforcing fibres for polymer matrix composites. The major advantages of glass fibres are low cost (compared to carbon, and aramid), high tensile strength,

high chemical resistance, and excellent insulation properties (Ali Munawar et al., 2007). It is widely used in electronics, home and furniture, automobiles, boats and marine applications, medical applications, and aerospace applications (Sathishkumar, Satheeshkumar, & Naveen, 2014).

2.3.1.3 Aramid Fibres

Aramid fibres have lowest specific gravity, and highest tensile strength to weight ratio among the current reinforcing fibres but exhibit poor characteristics in compression, and relatively expensive (Agarwal & Broutman, 1990). They are primarily used in marine and aerospace applications (Ali Munawar et al., 2007).

2.3.2 Natural Fibres

The major characteristics and applications of the widely used natural fibres used in reinforcing composites are briefly described below.

2.3.2.1 Hemp Fibres

Hemp fibres obtained from the *Hibiscus cannabinus* L. is fully biodegradable, nontoxic, and can be recycled. They also have high modulus and strength among the natural fibres. Major applications include ropes, toys, shoes, mobile phone casing, door panel in automobiles (Peças, Carvalho, Salman, & Leite, 2018).

2.3.2.2 Flax Fibres

Flax fibres (*Linum usitatissimum)* are non-abrasive, cost effective, eco-friendly, and possess good mechanical properties. Flax fibre reinforced composites have increasing demand in automotive, building and appliance industries. Specially in automotive industries, it is widely used to manufacture door panels, door bolsters, headliners, side and back walls, seat backs, rear deck trays,

pillars, center consoles, load floors, trunk trim etc. (Witayakran, Smitthipong, Wangpradid, Chollakup, & Clouston, 2016).

2.3.2.3 Jute Fibres

Jute (*Corchorus sp*.) is a lignocellulosic fibre obtained from the ribbon of the plant stem. It has good tensile strength, high heat resistance, and high rotting resistance. It is mainly used in packaging materials, ropes, carpet baking, and wall decoration purposes (Sen & Jagannatha Reddy, 2011)

2.3.2.4 Sisal Fibres

Sisal fibres *(Agave sisalana)* possess good moisture and heat resistance. Sisal fibres combined with flax and hemp are used as a reinforcement to manufacture interior door linings and panels, door panels, side and back walls in automotive industries (Peças et al., 2018). Other applications include ropes, mats, carpets. and cement reinforcement (Sen & Jagannatha Reddy, 2011).

2.3.2.5 Coir Fibres

Coir fibre *(Cocos nucifera)* is obtained from the husk of the fruit of the coconut palm. In addition of being cheap, these fibres are strong, and can easily withstand heat and saltwater. These fibres are mainly used to manufacture containers, boxes, trays, packaging, ropes, and fishing nets

2.4 Typha spp. or Cattail

Typha spp. commonly known as cattail, water torch, or bulrush is an aquatic, erect herb having perennial creeping. The stoloniferous rootstocks of these herbs form an irregular networks of 8-10 cm under the surface of the soil (Claassen, 1919). The leaves are erect, grass-like, flat, and tapered at the apex with a length ranging up to 3.5 m and a width ranging from 3 to 23 mm (J. F. Morton, 1975). J. F. Morton (1975) also reported that the flowering stem is stiff having a cylindrical or oval

shape which may be shorter or taller than the leaves, unjointed and carries a terminal set of 2 or sometimes 3. *Typha* spp. are emerging wetland plants that are becoming plentiful in the wetlands of North America (Hager, 2004).

2.4.1 Species of Cattail in North America

Mainly three species of *Typha* are found in this region i.e. *Typha latifolia* L., *Typha angustifolia* L., and the hybrid of these two *Typha x glauca*. A comparison between the physical properties of these three species are shown in table 2.3 (J. F. Morton, 1975; Smith, 1967).

Table 2.3. Comparison between the three species of Typha spp. primarily found in North America (J. F. Morton, 1975; Smith, 1967).

	Species		
Properties	*Typha latifolia* L.	*Typha angustifolia* L.	*Typha x glauca*
Plant height	1.2-2.5 m	1.5-2.5 m	2-3.5 m
No. of leaves	12-16	7-13	Usually less than 10
Leaf-width	5-30 mm	3-15 mm	6-15 mm
Leaf description	Light green, flat, taller than the spikes.	Dark green, sometimes reddish, concave above, convex beneath, much taller than the spikes.	Blue-green, flat, slightly convex beneath, taller than the spikes.
Leaf sheaths	Articulate	Not articulate	Usually articulate
Spikes	Male spike is yellow-brown with black markings, female spike is dark brown	Male spike is yellow-brown without black markings, female spike is dark brown	Male spike is yellow-brown, female spike is reddish brown

Typha latifolia L. also known as the broad-leaf cattail, or native cattail primarily grows in freshwater situations. It can withstand acid soils and inhabits almost all of North America (J. F. Morton, 1975; Smith, 1967). *Typha angustifolia* L. also known as the narrow-leaf cattail inhabits primarily in temperate to temperate to subtropical regions of North America extending from Nova Scotia west to Ontario, down the east coast and around the Great Lakes, and throughout the southern United States. They often populate in water bodies which are too saline for the *Typha latifolia* L. Finally, the hybrid between these two *Typha x glauca* is found in the regions where both the parent species are found i.e. the Atlantic Coast of North America (Virginia, North Carolina, South Carolina, Georgia, Alabama, Florida), and around the Great Lakes, and San Francisco Bay (J. F. Morton, 1975; Smith, 1967).

Cattail plants are often considered undesirable, and invasive because of their wind-dispersed seeds and the expeditious growth of the rhizomes which enable them to compete with the wetland native herbs and supersede them by inhabiting germination (Frieswyk & Zedler, 2006; Hager, 2004). The studies reported that these phenomena seriously affect the wetland biodiversity. Furthermore, at the end of the lifecycle, the leaves and the stems of these plants remain unused for any immediate application and is returned to the soil for decomposition producing a huge amount of waste biomass. In 2017, measures were taken to remove excessive amount of invasive cattail plants at the Voyageurs National Park at Minnesota, near Canadian border to restore natural wetlands and native ecosystems (Johnson, 2017).

2.4.2 Common Uses of *Typha*

Despite the invasive characteristics, all species of *Typha* have been considered as a potential crop for many years because of their productivity, abundance, and feed value (Dubbe, Garver, & Pratt, 1988). Cattail provides protective covering to the wildlife and plays an important role for water

filtration, and flood protection (Houlahan, Keddy, Makkay, & Findlay, 2006; J. F. Morton, 1975). In addition of being used as a source of food, various native populations have extensively used almost all parts of cattail to meet many of their needs (J. F. Morton, 1975). The stems and leaves of cattail were used to make carpets, sandals, room portioners, ropes, coarse mats etc. (Dalziel, 1937; Yacovleff & Herrero, 1934 as mentioned in J. F. Morton, 1975). Cattail floss and pollen was used as a filler in pillows and mattresses (Yacovleff & Herrero, 1934 as mentioned in J. F. Morton, 1975). In World War II, several million pounds of cattail floss were used in life jackets as a replacement of kapok manufactured in Chicago (Marsh, 1959 as mentioned in J. F. Morton, 1975). These flosses have also been used as a filling in baseballs and sleeping bags (Timmons et al., 1963). Furthermore, cattail flowers were used to treat wounds and burn injuries (Maiden, 1889 as mentioned in J. F. Morton, 1975). Therefore, cattail had exceedingly diverse use by men and are employed to some extent commercially even today.

2.4.3 Prospect of *Typha* in Industrial Applications

Typha spp. possesses some unique characteristics- it is cosmopolitan and available locally, populates rapidly, is recyclable, hydroscopic, biodegradable, non-abrasive, reactive, and easily available in many forms (Othman, Ruzaidi, Khalisanni, & Nazarudin, 2012). All these characteristics have drawn attention of the researchers and the recent investigations on *Typha* spp. have shown its prospect of being used in various industrial applications.

Krus et al. (2014) found that the leaves of *Typha angustifolia* L. plants have higher load bearing capability and thermal insulation properties due to the presence of high strength stem fiber and spongy like tissues. These characteristics have made *Typha* an ideal option for manufacturing new sustainable building materials. Cao et al. (2018) examined the structured *Typha* fiber assemblies for cyclic filtration behavior to remove spilled oil contamination from the water and the results

showed that the *Typha* fibers are highly oleophilic and hydrophobic removing 70%-90% of the oil contamination.

Also, studies have revealed that *Typha latifolia* L. fibres blended with wheat straw exhibited acceptable mechanical properties for composite panels (Bajwa et al., 2015). Wuzella et al. (2011) developed novel, biobased, and binder-free fibre-board composites using *Typha latifolia* L. fibres which showed higher mechanical properties than fibre-boards made from common natural fibres using phenolic binder upto 15%. Moreover, the cellulose in the *Typha latifolia* L. can be extracted to produce cellulose nanofibres which has potential applications in packaging, fibre reinforced composites and, electronic display purposes (Sundari & Ramesh, 2012). All these findings revealed that cattails fibres specially extracted from the *Typha latifolia* L. have great potential for being used as reinforcements in 'novel' fibre-reinforced bio-composites and in other industrial applications.

2.4.4 Extraction of fibres from *Typha latifolia* L.

So far, different retting methods have been applied to extract fibers from *Typha latifolia* L. such as water retting, alkali retting, enzyme retting, acid retting, and alcohol retting (Chakma, 2018; Sana et al., 2014). Out of these methods, alkali treatment particularly treated with NaOH was found to be the simplest, most effective and economic, and possesses least environmental impacts (Chakma, 2018; Sana et al., 2014; Xiao, Sun, & Sun, 2001). Until now, different concentrations of NaOH (1-5%), were evaluated to extract fibers from these plants using various treatment durations (1-8 hrs.) and treatment temperatures (60°-120° C) (Chakma, 2018; Ruangmee & Sangwichien, 2013; Sana et al., 2014).

The studies have revealed that the extracted fibers obtained from the broad-leaf cattail possess similar characteristics to commonly used cellulosic fibers such as hemp, jute, flax in terms of mechanical properties, fiber diameter, moisture regain, and thermal properties (Chakma, 2018; Sana et al., 2014). All these properties are prerequisites in order to be used as reinforcements in fibre-reinforced composites. Moreover, the fibre yield (%) of broad-leaf cattail was found to be significantly higher than other common natural fibres (Chakma, 2018). Therefore, fibres obtained from the broad-leaf cattails can be an ideal source to produce 'novel' fibre-reinforced bio-composites which may be used in automotive industries, packaging industries, construction materials, furniture and home décor materials. However, no studies have been done so far to validate the properties of these fibres to identify its usage as a fibre-reinforced composite.

Furthermore, literature has shown that the properties of fibres are affected significantly by varying the alkali concentrations, the treatment time, and the treatment temperature (Sana et al., 2014; Xiao et al., 2001). Therefore, it is significant to know these effects of processing parameters (time, temperature, and alkali concentration) on fibres properties and fibre yield (%) in order to determine the optimum processing parameters that would produce fibres of desired characteristics for a specific composite application. Nonetheless, no research has been done so far which would define the effects of these processing parameters on the physical, and mechanical properties of cattail fibres obtained from *Typha latifolia* L.

2.5 Objectives

Therefore, the objectives of this thesis are:

1. Evaluating the properties of cattail fibres extracted from *Typha latifolia* L. using different concentrations of NaOH, treatment time, and treatment temperature to validate their usability in a specific composite application i.e. automobiles, packaging materials, and aerospace applications.
2. Determining the effects of processing parameters (time, temperature, and NaOH concentration) on composite properties of the fibres.
3. Optimizing the fibre manufacturing process using 'Desirability Function Analysis' to assist manufacturers to set production parameters (time, temperature, and NaOH concentration) which would produce fibres of desired characteristics for a specific composite application.

3. CHAPTER: MATERIALS AND METHODS

This chapter discusses the extraction method used to obtain cattail fibres from the leaves of *Typha latifolia* L., the composite properties of the cattail fibres, and the optimization of the processing parameters (time, temperature, concentration of alkali) to improve the physical and mechanical properties of the fibres for composite applications.

The main material, *Typha latifolia* L. as shown in Figure 3.1 was collected from the Assiniboine Forest, Winnipeg, Manitoba (Canada) and the plants were harvested by simply using a knife. The plants were collected during the late October 2017, and the color of the leaves were greenish to brownish. Some plants had a fully developed stem with a flower having cylindrical shape and dark brown color. The branches had approximately 5-10 leaves per plant and the leaves were approximately 2-3 m long and 15-30 mm wide. The stems are light green to brown in color having a diameter of approximately 6-12 mm (Figure 3.1).

Figure 3.1: *Typha Latifolia* L. collected from the Assiniboine Forest

3.1 Fibre Extraction

For cellulosic fibres, various extraction methods have been assessed such as water, chemical, microbe and microbe-chemical retting and how this methods influence fiber properties are reported (Xiao et al., 2001; Yu & Yu, 2010). However, unlike other cellulosic fibers, water retting did not produce any fibers from cattail plants and amidst the chemical rettings, only alkali retting produced fibers at 60° C or higher temperatures (Chakma, 2018). Different alkali solutions were examined to produce fibers such as NaOH, KOH, and LiOH and it has been observed that out of these alkalis, treating with NaOH most efficient with least environmental impacts (Chakma, 2018; Sana et al., 2014; Xiao et al., 2001). The chemicals used to extract fibres in this study are listed in table 3.1.

Table 3.1: Chemicals used for cattail fibre extraction

Chemical	Supplier	Purity
Sodium hydroxide	Sigma Aldrich-USA	≥ 97%
Acetic acid	Kimax-35 USA	5g/l

In this study, 3 levels of concentration (4, 7, 10%, w/v) of NaOH solution were used with 5 levels of time (4, 6, 8, 10, and 12 h.) and 4 levels of temperature (70, 80, 90 and 95º C) in a 3*4*5 factorial experimental setup to observe the effects of different levels of concentration of NaOH, treatment time, and temperature on fiber yield (%) and fiber properties. A total of 60 treatments were performed and each treatment was classified by the latter ‘T’ with a subscript starting from 1 to 60. The experimental design is illustrated in table 3.2.

Table 3.2: Experimental Design (3*4*5 factorial setup)

Concentration of NaOH: 4%						
		Time levels				
		4 Hrs.	6 Hrs.	8 Hrs.	10 Hrs.	12 Hrs.
Temp. levels	70° C	T_1	T_2	T_3	T_4	T_5
	80° C	T_6	T_7	T_8	T_9	T_{10}
	90° C	T_{11}	T_{12}	T_{13}	T_{14}	T_{15}
	95° C	T_{16}	T_{17}	T_{18}	T_{19}	T_{20}
Concentration of NaOH: 7%						
		Time levels				
		4 Hrs.	6 Hrs.	8 Hrs.	10 Hrs.	12 Hrs.
Temp. levels	70° C	T_{21}	T_{22}	T_{23}	T_{24}	T_{25}
	80° C	T_{26}	T_{27}	T_{28}	T_{29}	T_{30}
	90° C	T_{31}	T_{32}	T_{33}	T_{34}	T_{35}
	95° C	T_{36}	T_{37}	T_{38}	T_{39}	T_{40}
Concentration of NaOH: 10%						
		Time levels				
		4 Hrs.	6 Hrs.	8 Hrs.	10 Hrs.	12 Hrs.
Temp. levels	70° C	T_{41}	T_{42}	T_{43}	T_{44}	T_{45}
	80° C	T_{46}	T_{47}	T_{48}	T_{49}	T_{50}
	90° C	T_{51}	T_{52}	T_{53}	T_{54}	T_{55}
	95° C	T_{56}	T_{57}	T_{58}	T_{59}	T_{60}

3.1.1 Sample Preparation

The leaves of *Typha Latifolia* L. were cut to 4 cm length as shown in Figure 3.2. and then dried in the oven at 105° C. The oven was equipped with a ventilator and a thermostat, and the dehydration by heating was carried out for 8 hours until a constant dry weight was achieved. Desiccation was considered complete when the difference in weights was obtained less than 0.05% of the mass in

a sample when weighed two successive times at 15 minutes interval. The weighing was carried out using electronic balance.

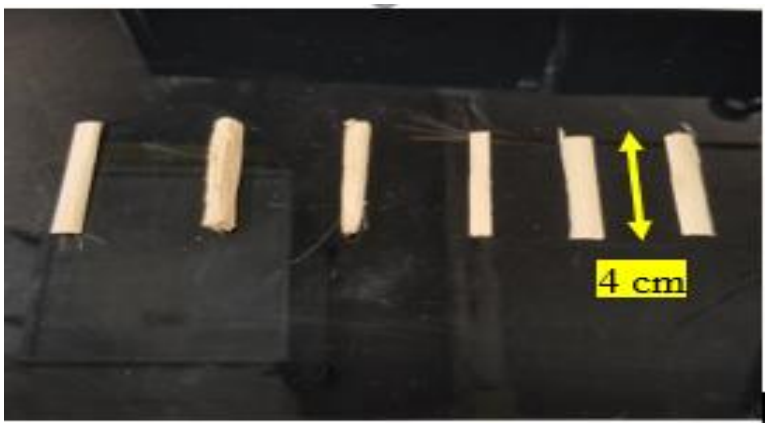

Figure 3.2: Leaves cut in 5 cm length for fiber extraction.

3.1.2 Water-bath Extraction

For each treatment, 4 weighted samples were placed in 4 different Erlenmeyer flasks . The sample weight was kept between .400-.420 gm. In each flask, 200 mL of either 4/7/10% (w/v) concentrated sodium hydroxide solution was added. The ceiling of the flasks were sealed using parafilm tape and foil paper. Then the treatment was carried out in water-bath, which has a temperature control system, for a fixed set of time and temperature as shown in Figure 3.3. Throughout the treatment, the level of water in the water bath was kept consistent by adding 200 mL of hot water (heated until the temperature is equal to the treatment temperature) every 4 hours. After the treatment, the treated fibres were thoroughly washed with distilled cold water to remove the alkali and dissolved substances. The waste chemicals were stored in a waste-container and later disposed after adding sufficient amount of water and acetic acid to bring the pH to 7.5. The fibers were then dried using the same procedures mentioned above to determine the fiber yield (%), and other physical, and mechanical properties.

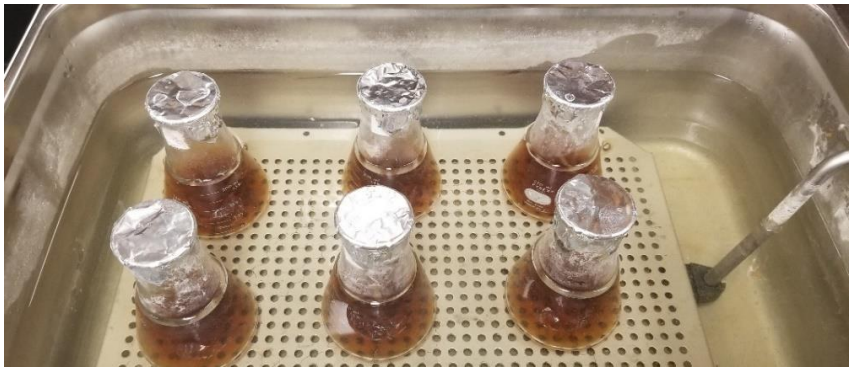

Figure 3.3: Cattail fibre extraction using water bath

3.2 Determining the Properties of Typha Fibre for Composite Reinforcement

The machines and methods used to determine the composite reinforcement properties of *Typha* fibres are listed in Table 3.3.

Table 3.3: The machines and methods used to determine the composite properties of Typha fibres

Composite properties	Machine/Method	Standard test method
Yield (%)	Thermo scientific oven and weighting machine	(Gravimetric method) standard: NF G 08- 001
Length (mm)	Scissor and ruler	
Diameter (μm)	Bioquant life science image analyzer	France standard NF G 07- 004 (1983)
Tensile strength, modulus of elasticity and elongation at break (%)	Instron Tensile Tester	ASTM D3822
Moisture regain (%)	Humidity chamber and Thermo scientific oven	ASTM D2564

3.2.1 Yield Measurement

The yield (y %) of the fibers in each treatment is measured as the percentage of the ratio of the oven dried mass of the fibers after chemical treatment (M_a) and the oven dried mass of the *Typha* leaves before chemical treatment (M_b) which is expressed in equation 3.1. The gravimetric method was used with the following standard: NF G 08- 001, to calculate fibre yield (%).

$$\text{Yield (\%)} = \frac{M_a}{M_b} \times 100 \text{.....(3.1)}$$

3.2.2 Fibre Diameter Measurement

Before measuring the fiber diameter and other mechanical properties, first 10 single fibers from each treatment, a total of 600 fibers were randomly separated. Then each single fibre was cut approximately 30 mm in length and was attached to a rectangular frame using adhesives as shown in Figure 3.4. The inside length of the frame was 25 mm, therefore the length of the fibre attached inside the frame was approximately 25 mm. Then the frame is placed on a glass slide and the fibre was observed using 10x magnification using Bioquant Analyzer which was connected with a projection microscope and camera. The diameter of the single fiber attached inside the frame was measured following France standard NF G 07- 004 (1983) and the unit of measurement was in micrometer (μm). Due to the variations in diameter across the length of the single fibre which is a common phenomenon in natural fibres(Morton & Hearle, 2008), the diameters were measured in five different places across the length and the average diameter was reported.

3.2.3 Mechanical Properties Measurement

The mechanical properties i.e. tensile strength, modulus of elasticity, and elongation at break (%), were measured using the Instron Tensile Tester. Before the measurements, all the fibre samples

were conditioned at 21° C temperature and 50% relative humidity for 48 hours. After the conditioning is done, the frame holding the single fibre is placed between the jaws of the machine in a way (as shown in fig. 3.5) that the inside length of the frame acted as a gauge length. Then the vertical stands of the frame were cut with a scissor and the length of the fiber inside the frame acted as a gauge length which is approximately 25 mm.

Figure 3.4: Fibres attached to the frames by glue

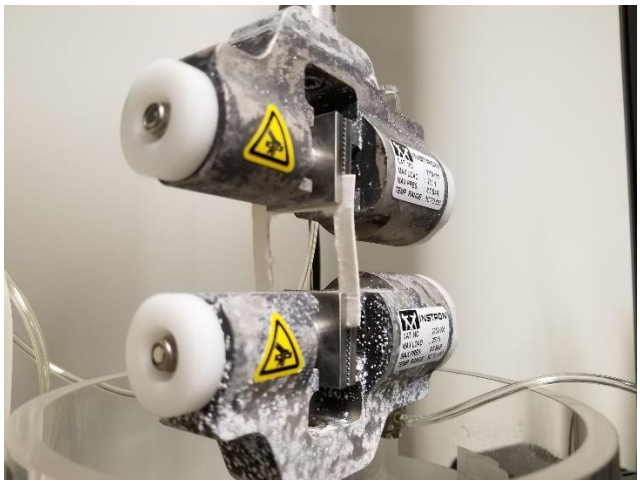

Figure 3.5: Fibre-frame placed between the jaws

However, the fibres acquired some crimp during the fibre extraction and washing, and it was necessary to remove the crimp to determine the actual length of the fibre attached inside the frame. For this reason, 'Pretest' and 'Auto-balance' functions in the 'Instron Bluehill 3' software were used. The 'Pretest' function allows the machine to extend the fibre, but no data is reported until a small amount of load is experienced by the load cell (which was chosen as 0.3 N for this experiment). When the load cell experiences the specified amount of load, the extension up to this point is considered due to the crimp and the 'Auto-balance' function adds this length with the initial length of the fibre and then the original test begins. All the 600 fibre samples were tested using this procedure. For this experiment, the crosshead moved at a speed of 20mm/min, and a 5 N load cell was used to conduct the tensile strength tests. All the results (tensile strength, modulus of elasticity, and elongation at break) and graphs for each fibre was obtained directly from the 'Instron Bluehill 3' software.

3.2.4 Moisture Regain (%) Measurement

For measuring the moisture regain, 3 fibre bundles from each treatment, a total of 180 samples from 60 treatments were randomly taken. The oven dry weight of the samples were measured following the same procedure mentioned in section 3.1.1. Then the fibres were conditioned according to ASTM D 1776- 4 (2008) in a humidity chamber at the grain storage of the University of Manitoba for 1 week. The temperature and relative humidity of the chamber was maintained 21±1° C and 65±3%. respectively. The moisture regain (%) was measured as a percentage of the ratio of the weight of water absorbed by the sample to the oven dry weight of the sample as expressed in equation 3.2. After measuring moisture regain (%) for each sample, the moisture regain (%) of each treatment was reported as an average of the three samples taken from that treatment.

$$\text{Moisture Regain (\%)} = \frac{M_w - M_o}{M_o} \times 100 \text{.....(3.2)}$$

Where, M_w = Weight of the samples after conditioning, and M_o = Oven dry weight of the samples.

3.3 Statistical Analysis

The experimental design was a full factorial design that is all levels of each factor (time, temperature, and concentration of NaOH) were present in combinations of all levels of other factors (Box, Hunter, & Hunter, 1978). A full factorial analysis of variance were performed to determine the effects of time, temperature, concentration of NaOH, and their interactions on fiber yield (%), and fibre properties. In this analysis, treatment time (t), temperature (T), and concentration of NaOH (C) were used as input variables and fiber yield (%), diameter, moisture regain (%), and mechanical properties (tensile strength, extension at break, and modulus of elasticity) were considered as output variables. The number of replications per treatment, and the total number of prepared samples are illustrated in Table 3.4. All statistical analysis has been performed using the SAS® University Edition software and JMP® 14.1. software.

Table 3.4: Number of replications per treatment, and total number of samples for each response variable.

Response Variables	No. of Replications Per Treatment	Total No. of Treatments	Total No. of Samples
Yield (%)	4	60	240
Diameter (μm)	10		600
Tensile strength (MPa)	10		600
Modulus of elasticity (GPa)	10		600
Elongation at break (%)	10		600
Moisture regain (%)	3		180

The model used for analysis was a fixed model as all the levels of time, temperature, and concentrations of NaOH were predetermined. The model that has been used to perform the three-way factorial ANOVA is shown below:

$$Y_{ijkl} = \mu + T_i + t_j + C_k + tT_{ij} + tC_{jk} + TC_{ik} + tTC_{ijk} + e_{ijkl} \quad \text{.....(3.3)}$$

Here,

Y_{ijkl} = l'th response treated with i'th temperature, j'th treatment time, and k'th concentration of NaOH.

μ = population mean

T_i = effect of i'th treatment temperature on l'th response.

t_j = effect of j'th treatment time on l'th response.

C_k = effect of k'th concentration of NaOH on l'th response.

tT_{ij} = effects of interaction of time and temperature on l'th response.

tC_{jk} = effects of interaction of time and concentration on l'th response.

TC_{ik} = effects of interaction of temperature and concentration on l'th response.

tTC_{ijk} = effects of interaction of time, temperature, and concentration on l'th response.

$eijk_l$ = error variations.

Range-

J = 70 to 95º C

i = 4 to 12 h.

k = 4 to 7%

I = 1 to 4 for yield (%)

= 1 to 10 for diameter, tensile strength, modulus of elasticity, elongation at break (%)

= 1 to 3 for moisture regain (%)

After performing ANOVA, the non-significant effects were removed to simplify the model. The simplified model for each response variable is shown in the following chapter. Estimated treatment mean for each response variable for each treatment was calculated using the simplified models.

3.3.1 Desirability Function Analysis

Desirability function analysis (DFA) popularized by Derringer and Suich (1980) is one of the most widely used methods for process optimization having multiple responses. Desirability function transforms each estimated response variables "$\hat{Y}_i$" to a desirability value d_i, where $0 \leq d_i \leq 1$. The value of d_i increases as the "desirability" of the corresponding response increases. The individual desirabilities are then combined using the geometric mean. In this study, DFA was used to optimize the fibre extraction parameters (time, temperature, and concentration of NaOH) which would produce fibres of desired characteristics for a specific composite application.

Here two types of desirability functions "d_i" were used i.e. desirability function to maximize, and desirability function to minimize. For maximizing a property "Y_i" , the desirability function (d_i) was calculated using the following formulas-

$$d_i = 0 \text{ if } \hat{Y}_i < Y_{min} \text{.....(3.4)}$$

$$d_i = \left[\frac{\hat{Y}_i - Y_{min}}{C - Y_{min}}\right]^S \text{ if } Y_{min} \leq \hat{Y}_i < C \text{.....(3.5)}$$

$$d_i = 1 \text{ if } \hat{Y}_i \geq C \text{.....(3.6)}$$

Where C is the upper criteria value or the requirement, Y_{min} is the lower tolerance value, and s represents weight. When $\hat{Y}_i$ equals or exceeds the upper criteria value which is the requirement, the desirability function equals to 1. When $\hat{Y}_i$ is less than the lower tolerance value which is unacceptable, the desirability function equals to 0. For minimizing a property "Y_i", the desirability function (d_i) was calculated using the following formulas-

$$d_i = 1 \text{ if } \hat{Y}_i \leq C \text{.....(3.7)}$$

$$d_i = \left[\frac{\hat{Y}_i - Y_{max}}{C - Y_{max}}\right]^t \text{ if } C < \hat{Y}_i \leq Y_{max} \text{.....(3.8)}$$

$$d_i = 0 \text{ if } \hat{Y}_i > Y_{max} \text{.....(3.9)}$$

Where C is the lower criteria value or the requirement, Y_{max} is the upper tolerance value, and t represents weight. When Y_i is equal or less than the lower criteria value which is the requirement, the desirability function equals to 1. When Y_i exceeds the upper tolerance value which is unacceptable, the desirability function equals to 0. Therefore, before calculating the individual desirability function, the objective of each property, the criteria value, the tolerance value, and the weights were fixed. The values of r, and t is specified by the user, and Derringer & Suich (1980) suggested that a large value of weights would be specified if the property (Y_i) is very desirable. After calculating individual property's desirability index (d_i), the composite desirability, "d_G" which combines all the desirability functions to form a single value was calculated using the following formula-

$$d_G = \sqrt[w]{{d_1}^{w_1} \times {d_2}^{w_2} \times {d_3}^{w_3} \times \ldots \times {d_n}^{w_n}} \text{.....(3.10)}$$

Where $i \in [1 \ldots n]$, d_i is the individual desirability of the property Y_i, w_i is the relative importance of the property "Y_i" in the composite desirability (d_G), w is the sum of the individual importance (w_i), and n is the number of properties. Therefore, this single value of composite desirability represents the overall assessment of the desirability of the combined response levels (Derringer & Suich, 1980). The value of d_G falls within the range of 0 to 1, and the value of d_G increases as the combination of properties becomes more favorable. Moreover, if any $d_i = 0$, that is one of the response variables is unacceptable, the value of d_G becomes 0 which implies that the overall product is unacceptable.

3.3.2 Sensitivity Analysis for Desirability Functions

At present there is no standard which can be used to select or determine the parameters of desirability functions. The subjective parameters such as, the weight of each response used to determine the shape of desirability, the range of individual response, and the importance coefficient of each response in the composite desirability are so called 'user specified'. (Derringer & Suich, 1980; Jaouadi, M'sahli, & Sakli, 2009; Malenović et al., 2011). Therefore, the selection of these parameters are susceptible to biased or arbitrary choices (Malenović et al., 2011). In general, these parameters are determined based on applications and manufacturing cost (Derringer & Suich, 1980; Jaouadi et al., 2009). However, from design and quality perspective, these selection of parameters should have some statistical basis so that the optimization results can be analyzed further (Malenović et al., 2011). This analysis should examine the robustness of overall desirability to changes in these parameters.

In this study, a sensitivity analysis of desirability functions was conducted by following the approach proposed by Aksezer (2008). This analysis is performed by varying the parameters to

certain levels and analyzing their effects on the overall desirability by treating it now as the response of interest. This procedure has the following steps-

1. Assigning the upper and lower edges for each parameter.
2. Determining the appropriate experimental design and defining the experimental set-up.
3. Obtaining the overall desirability value by solving the multi response problem in each run that will be used as response value in the corresponding sensitivity analysis.
4. Finding the most significant model, model terms, and calculating prediction equation considering the overall desirability as the response variable.
5. The coefficients, and signs of factors in the prediction equation would identify the sensitivity of each parameter.

The positive valued factors would improve the overall desirability while the negative factors will decrease it. However, coefficients close to zero could be marked as insensitive regardless of the sign.

4. CHAPTER: FACTORIAL ANALYSIS OF RESPONSE VARIABLES

Before conducting the three-way factorial ANOVA of fibre yield (%), and fibre properties, the distribution of each response variable was examined. Fiber yield (%) are proportions that vary continuously from the range 0 to 1 (0 to 100 when expressed as a percentage). By definition, it falls under the category of beta distribution which is used to represent the variability over a fixed range, and predict the random behavior of percentages and fractions (Mun, 2015). Furthermore, for fibre properties i.e. diameter, tensile strength, modulus of elasticity, elongation at break (%), and moisture regain (%) the distribution of each response variable was examined. The values of skewness and kurtosis between ± 1, and ± 2 respectively were considered acceptable for being considered as a normal distribution because, if the sample size is large enough (>30 or 40), minor deviation from the normality assumption should not cause major problems. (Altman & Bland, 1995; Elliot & Woodward, 2006). The skewness and kurtosis values of the data of each response variable, and the distribution used for analysis is summarized in Table 4.1. The main effects, and the interaction effects of treatment time, temperature, and concentration of NaOH on fibre yield (%), and fibre properties are described in the following sections.

4.1 Fibre Yield (%)

For fiber yield (%), a beta-logistic model was used with the 'Glimmix' procedure in SAS which fitted the model using maximum likelihood estimation, and the logit link function was used to ensure that the predicted means stayed within the bounds (0 to 1) (SAS Institute Inc., n.d.).

Table 4.1: Summary of data, and the distribution used for each response variable

Response Variables	Description of Data	Distribution Used
Yield (%)	Proportions, ranged between 0 to 1 (0 to 100 when expressed as a percentage).	Beta distribution (Mun, 2015)
Diameter (μm)	Highly skewed to the right with skewness = 1.19 and kurtosis = 2.52	Lognormal distribution (Mun, 2015)
Tensile strength (MPa)	Heavily skewed to the right with skewness = 1.43 and kurtosis = 4.66.	Lognormal distribution (Mun, 2015)
Modulus of elasticity (GPa)	Heavily skewed to the right with skewness = 1.57 and kurtosis = 4.18.	Lognormal distribution (Mun, 2015)
Elongation at break (%)	Approximately followed the normal distribution with skewness = 0.66 and Kurtosis = 0.79	Normal distribution (Altman & Bland, 1995)
Moisture Regain (%)	Approximately followed a normal distribution with skewness = 0.88 and kurtosis = 0.49	Normal distribution (Altman & Bland, 1995)

A three-way factorial ANOVA of fibre yield (%) is shown in Table 4.2. It was observed that the main effects of time, temperature, and concentration of NaOH, and the interaction between temperature and concentration had significant influences ($p < 0.05$) on fibre yield (%). For pairwise comparison of the treatment means, Tukey-Kramer post-hoc procedure was followed at $\alpha = 0.05$.

Table 4.2: Three-way factorial ANOVA of fibre yield (%)

Effects	Probability	Significance
Time	< 0.001	**
Temperature	< 0.001	**
Concentration	< 0.001	**
Time*Temperature	0.768	NS
Time*Concentration	0.611	NS
Temperature*Concentration	0.005	*
Time*Temperature*Concentration	0.114	NS

P < 0.05 = significant (*); P < 0.001 = Highly Significant (**); P > 0.05 = Not Significant (NS).

The main effects of temperature, time and concentration of NaOH on fibre yield (%) are shown in Figure 4.1-4.3 respectively. In these figures, columns with common letters (A, B, C, D) indicate that there are no significant differences (P > 0.05) between the means. From Figure 4.1, it can be observed that with increased treatment time, the fibre yield (%) continuously decreased. This decrease became significant (P < 0.05) between 4 and 8 h. (P = 0.002), 4 and 10 h. (P < 0.001), 4 and 12 h. (P < 0.001), 6 and 10 h. (P < 0.001), 6 and 12 h. (P < 0.001), and 8 and 12 h. (P < 0.001). Similarly, from Figure 4.2 and 4.3, it can be observed that with increased treatment temperature, and concentration of NaOH, the fibre yield (%) decreased. For treatment temperature (Figure 4.2), this decrease was significant (P < 0.05) between 70° and 80° C (P = 0.015), 70° and 90° C (P < 0.001), 70° and 95° C (P < 0.001), and 80° and 95° C (P < 0.001). For concentration of NaOH (Figure 4.3), this decrease was significant (P < 0.05) between 4 and 7% (P < 0.001), and 4 and 10% (P < 0.001).

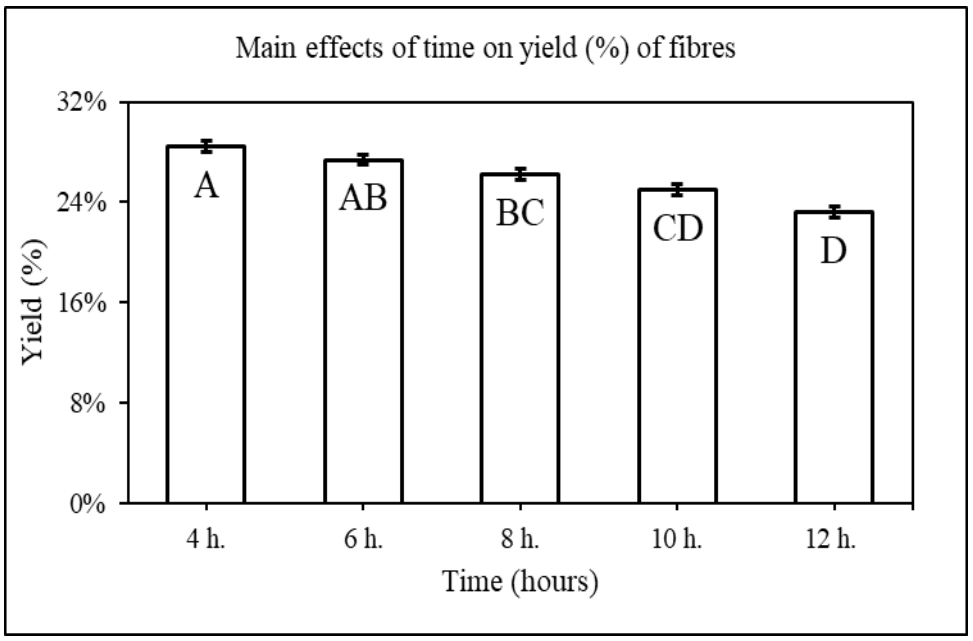

Figure 4.1: Main effects of treatment time on fibre yield (%)

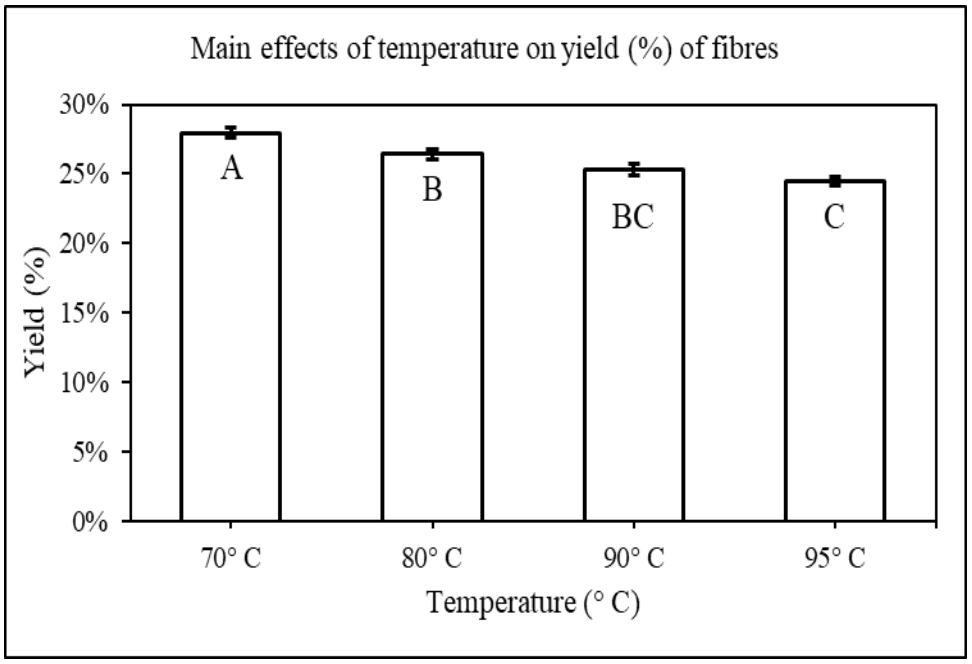

Figure 4.2: Main effects of temperature on fibre yield (%).

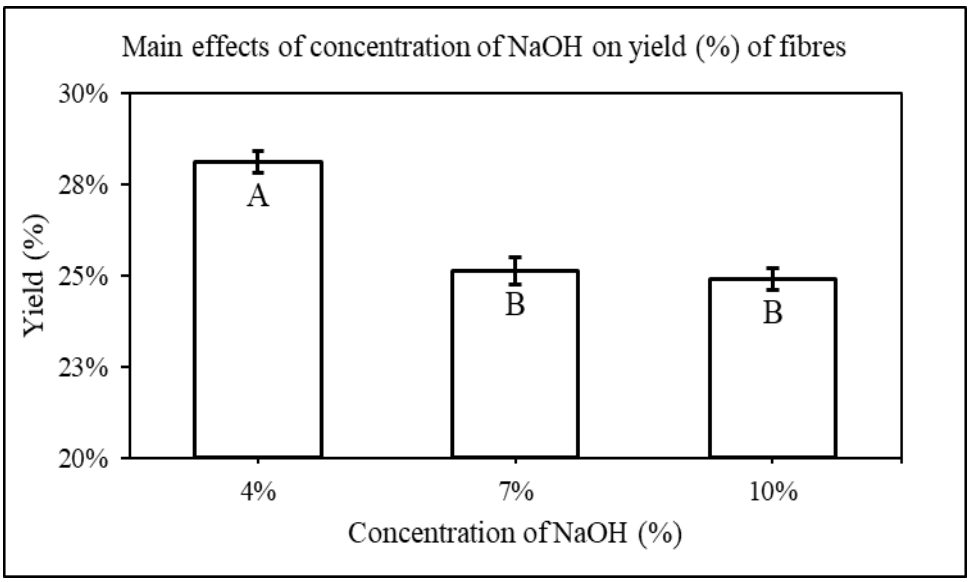

Figure 4.3: Main effects of concentration of NaOH (%) on fibre yield (%).

The effects of interaction of temperature and concentration is illustrated in Figure 4.4. At 4% concentration level of NaOH, the yield (%) of fibres progressively decreased with increase in temperature. This decrease was highly significant ($P < 0.001$) from 70° to 80° C. However, from 80° C, the reduction in yield (%) was not significant ($P > 0.05$) due to further increase in temperature up to 95° C. At 7% concentration level of NaOH, from 70° up to 90° C, no significant change ($P > 0.05$) in yield (%) was observed. However, when the temperature was further increased to 95° C, the decrease in fibre yield (%) was found significant between 70° and 95° C ($P = 0.044$). Finally, at 10% concentration level, no significant change ($P > 0.05$) in fibre yield (%) was observed for all levels of temperature (70°, 80°, 90°, and 95° C).

Previous studies have revealed that *Typha* fibre has a composite structure where cellulosic fibre bundles of different sizes and numbers are linked together by gummy and waxy substances composed of pectin (mostly), lignin, hemicellulose, wax, and fat materials (S. M. Mortazavi & Moghaddam, 2010; Sana et al., 2014). Fibre extraction at high temperature, concentration, and

longer treatment times facilitates the separation of fibre bundles by removing the gummy and waxy substances and therefore, decreasing the yield (%) (S. M. Mortazavi & Moghaddam, 2010). Moreover, the hydrolysis of pectin takes place at high temperature by sodium hydroxide with the formation of sodium pectate (Bhattacharya & Das, 2001) which contributes to the decrease in yield (%) at high temperature. As a result, for both 4 and 7% concentration levels, the decrease in yield (%) was significant ($P < 0.05$) between 70° and 95° C (Figure 4.4). However, at 10% concentration level, no significant difference ($P > 0.05$) in yield (%) was observed between 70° and 95° C which could be due to the fact that, high level of concentration (10%) of NaOH might accelerated the hydrolysis of pectin at 70° C and therefore, minimizing the effects of increase in temperature.

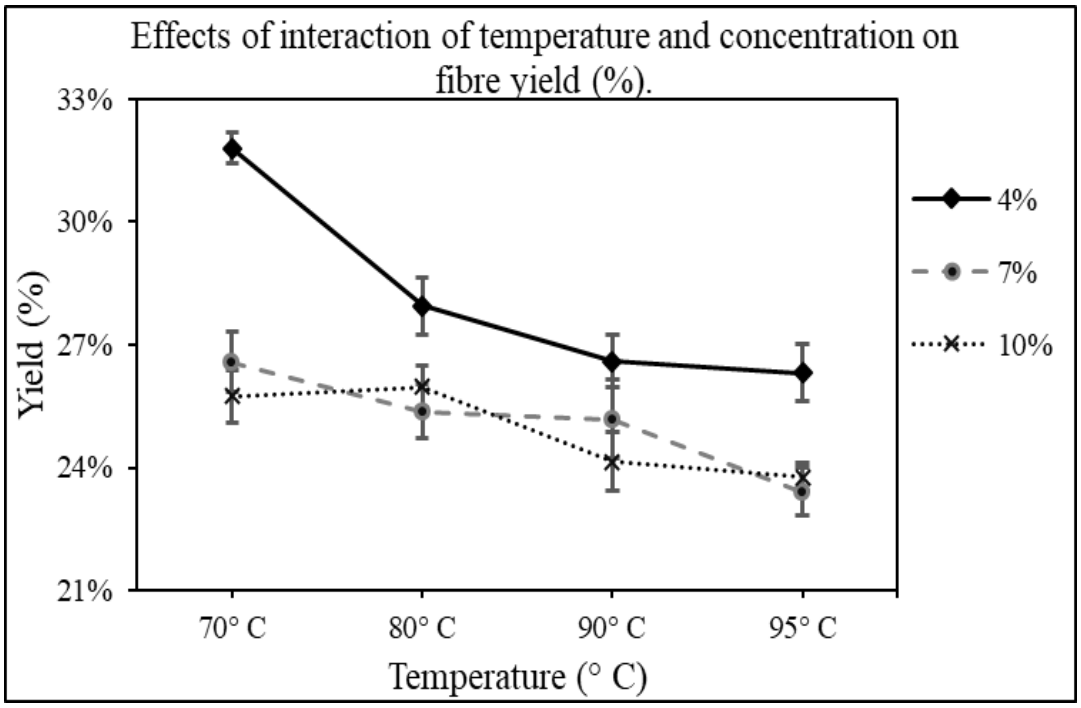

Figure 4.4: Effects of interaction of temperature and concentration of NaOH on fibre yield(%).

4.2 Fibre Diameter

The distribution of fibre diameter (μm) was highly skewed to the right with skewness and kurtosis values of 1.19 and 2.52 respectively. The data was analyzed considering as a lognormal distribution which had the skewness = 0.11 and kurtosis = 0.15. Also, due to the heterogeneity of the variances, Satterthwaite approximation was used to determine the effective degrees of freedom.

The three-way factorial ANOVA of fibre diameter is shown in Table 4.3. The main effects of time, temperature, and concentration of NaOH, and the interaction between temperature and concentration had highly significant influences ($p < 0.001$) on fibre diameter. For pairwise comparison of the treatment means, Tukey-Kramer post-hoc procedure was followed at $\alpha = 0.05$.

Table 4.3: Three-way factorial ANOVA of fibre diameter.

Effects	Probability	Significance
Time	< 0.001	**
Temperature	< 0.001	**
Concentration	< 0.001	**
Time*Temperature	0.832	NS
Time*Concentration	0.646	NS
Temperature*Concentration	< 0.001	**
Time*Temperature*Concentration	0.615	NS

P < 0.05 = significant (*); P < 0.001 = Highly Significant (**); P > 0.05 = Not Significant (NS).

Figure 4.5-4.7 represent the main effects of treatment time, temperature, and concentration of NaOH on fibre diameter respectively. In these figures, the columns having common letters indicate that there are no significant differences (P > 0.05) between the means. From Figure 4.5, it can be observed that with increased treatment time, the fibre diameter continuously decreased. This decrease became significant (P < 0.05) between 4 and 8 h. (P = 0.011), 4 and 10 h. (P < 0.001), 4

and 12 h. (P < 0.001), 6 and 12 h. (P < 0.001), and 8 and 12 h. (P = 0.004). Similarly, from Figure 4.2 and 4.3, it can be observed that with increased treatment temperature, and concentration of NaOH, the diameter of the fibres decreased. For treatment temperature (Figure 4.6), this decrease was significant (p < 0.05) between 70° and 90° C (P < 0.001), 70° and 95° C (P < 0.001), and 80° and 95° C (P = 0.036). For concentration of NaOH (Figure 4.7), this decrease was significant (P < 0.05) between 4 and 7% (P < 0.001), and 4 and 10% (P < 0.001).

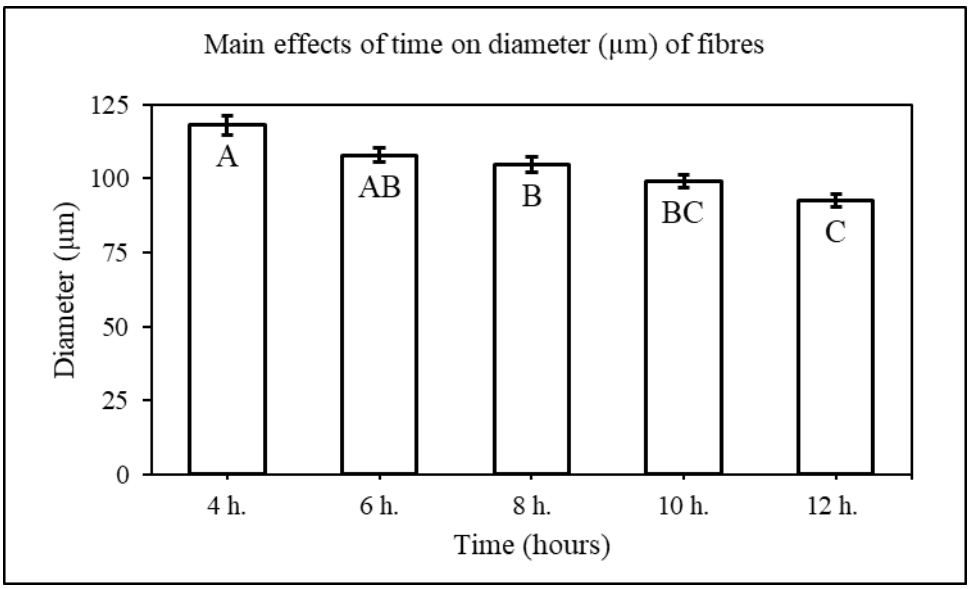

Figure 4.5: Main effects of treatment time on fibre diameter.

The effects of interaction of temperature and concentration of NaOH on diameter of cattail fibres are illustrated in Figure 4.8. At 4% concentration level of NaOH, the diameter of fibres steadily decreased with increase in temperature. This decrease was significant (P = 0.041) from 70° to 80° C. However, from 80° C, due to further increase in temperature up to 95° C, the reduction in diameter was not significant (P > 0.05). At 7% concentration level of NaOH, from 70° up to 90° C the fibre diameter progressively decreased which was significant between 70° and 90° C (P < 0.001) and 80° and 90° C (P < 0.001). However, a small not significant increase (P > 0.05) in

diameter was observed from 90° to 95° C. And finally, at 10% concentration level of NaOH, no significant changes ($P > 0.05$) in fibre diameter was observed from 70° to 95° C.

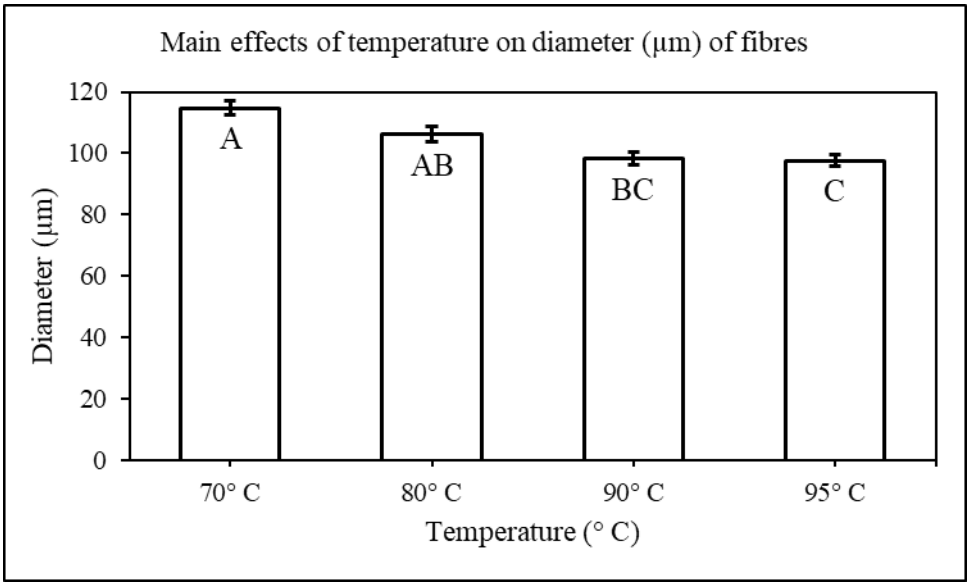

Figure 4.6: Main effects of temperature on fibre diameter.

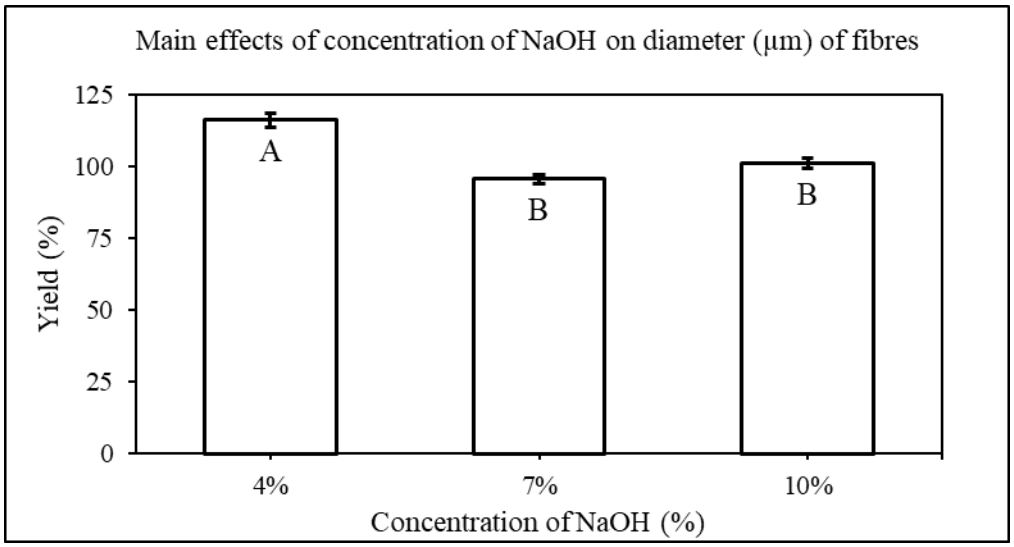

Figure 4.7: Main effects of concentration of NaOH on fibre diameter.

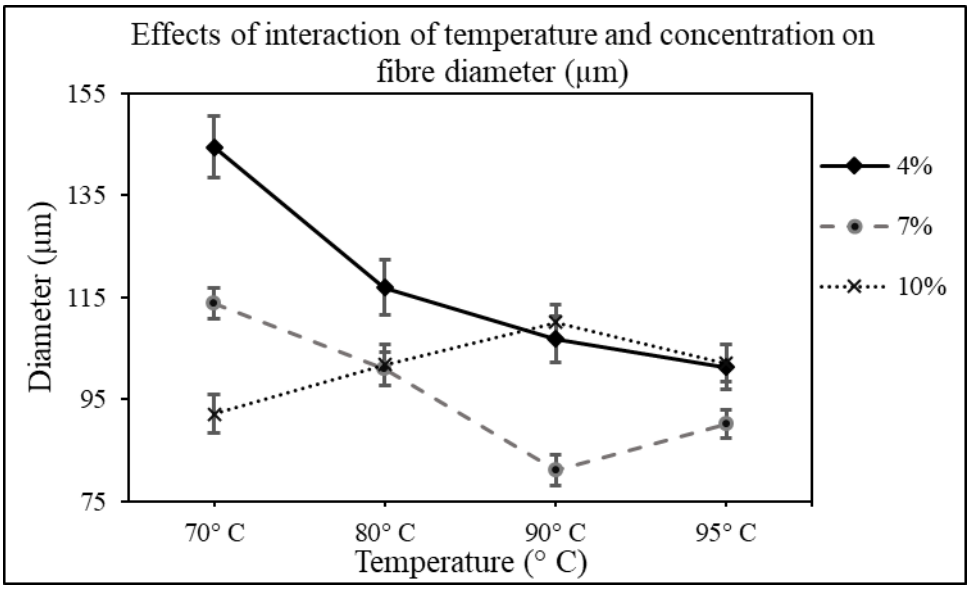

Figure 4.8: Effects of interaction of temperature and concentration of NaOH on fibre diameter.

Sana et al. (2014) observed that the alkali treatment removed the wax and fatty substances from the surface of cattail fibres layer by layer and therefore, decreasing the fibre diameter. They reported that high treatment duration, concentration of NaOH, and treatment temperature favor the separation of cattail fibres and therefore, in order to obtain a thin fiber, the extraction conditions need to be severe. However, when considering the effects of interaction of temperature and concentration (Figure 4.8) it was found that at 4% concentration level, the decrease in fibre diameter was highly significant from 70 to 95° C ($P < 0.001$); but at 10% concentration level, the change in diameter was not significant from 70 to 95° C ($P = 0.745$). This may result due to the probable fact that, at low level of concentration (4%), the removal of impurities was accelerated when the temperature was increased and therefore, yielding thinner fibres. However, at high level of concentration (10%), the removal of impurities was already higher at 70° C and the increase in temperature (up to 95°C) did not have any significant effect. Furthermore, the increase in diameter

at 90° C as the concentration was increased from 7 to 10% may be resulted due to the presence of inherent variability of materials sourced directly from nature (Kamat, 2000).

4.3 Fibre Tensile Strength

The distribution of tensile strength (MPa) of fibres was heavily skewed to the right with skewness = 1.43 and kurtosis = 4.66. The data was analyzed as a lognormal distribution with skewness = -.56, and kurtosis = .84. Also, due to the heterogeneity of the variances, Satterthwaite approximation was used to determine the effective degrees of freedom.

The three-way factorial ANOVA of tensile strength of fibres is shown in Table 4.4. From the ANOVA table, it can be observed that the main effects of time, temperature, and concentration of NaOH, and the interaction between temperature and concentration had significant influences ($P < 0.05$) on tensile strength of fibres.

Table 4.4: Three-way factorial ANOVA of tensile strength (MPa) of fibres

Effects	Probability	Significance
Time	0.034	*
Temperature	0.003	*
Concentration	< 0.001	**
Time*Temperature	0.990	NS
Time*Concentration	0.440	NS
Temperature*Concentration	< 0.001	**
Time*Temperature*Concentration	0.697	NS

$P < 0.05$ = significant (*); $P < 0.001$ = Highly Significant (**); $P > 0.05$ = Not Significant (NS).

However, after conducting the Tukey-Kramer post hoc analysis at $\alpha = 0.05$, no significant differences between the means were found for all levels of time (4, 6, 8, 10, and 12 h.) which is illustrated in Figure 4.9, The bars having common letters in the figure indicate that the differences

in means are not significant (P > 0.05). Figure 4.10-4.11 represent the main effects of treatment temperature, and concentration of NaOH respectively. Again, in these figures, the bars having common letters indicate that the differences in means are not significant (P > 0.05). For the main effects of temperature (Figure 4.10), a significant increase (P = 0.017) in tensile strength was observed from 70° C to 80° C although, no significant changes (P > 0.05) in tensile strength was found with increase in temperature from 80° C up to 95° C. Similarly, for the main effects of concentration, a highly significant increase (P < 0.001) in tensile strength of fibres was observed from 4 to 7%. And no significant change (P = 0.085) in tensile strength was found due to further increase in concentration from 7 to 10%.

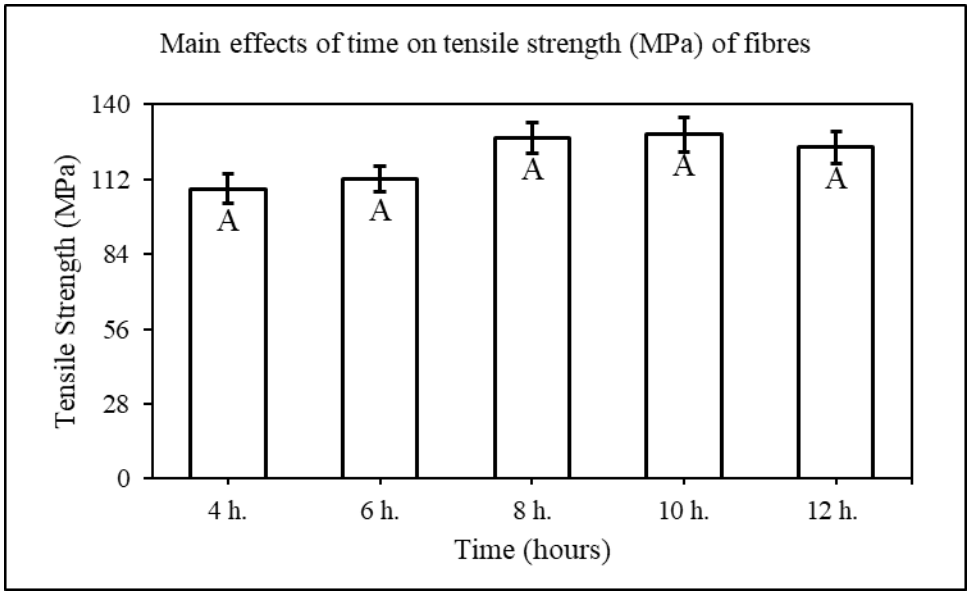

Figure 4.9: Main effects of time on fibre tensile strength.

The effects of interaction of temperature and concentration is illustrated in Figure 4.12. At 4% concentration level of NaOH, the tensile strength of fibres gradually increased with increase in temperature which became highly significant (P < 0.001) between 70° and 95° C. At 7%

concentration level of NaOH, with increase in temperature from 70° up to 90° C, a small progressive increase in strength was observed. However, further increase in temperature from 90° to 95° C, a minor decrease in tensile strength was observed. Overall, the change in tensile strength of fibres at this level of concentration was not significant (P > 0.05) for all levels of temperature. Similarly, at 10% concentration level of NaOH, the change in tensile strength was not significant (P > 0.05) for all levels of temperature. However, minor increase in tensile strength from 70° to 80° C, and then a small gradual decrease in tensile strength from 80° to 95° was observed.

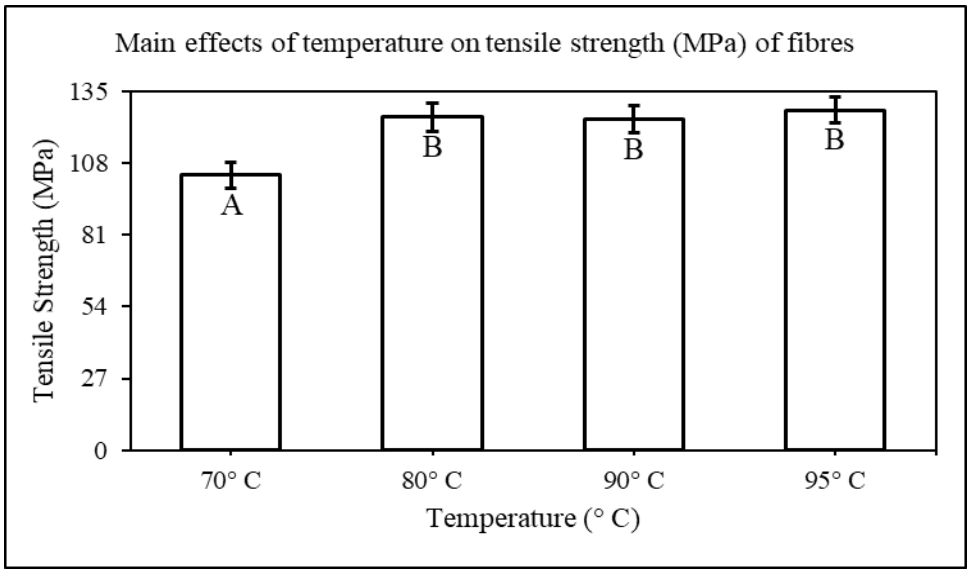

Figure 4.10: Main effects of temperature on fibre tensile strength.

Previous studies have revealed that the tensile properties (strength, fineness) of cellulosic fibres increase with the concentration of NaOH due the removal of impurities and the rearrangement of cellulosic chains (S. M. Mortazavi & Moghaddam, 2010; Sana et al., 2014). Bhattacharya and Shah (2004) explained that in the extraction bath, NaOH removes impurities and gummy materials that hold the fibers together, causing chelators (EDTA and STPP) to combine with calcium ions available in the plant leaves and therefore, extracting them from the pectin structure. Furthermore, it was found that at higher temperature, the removal of non-cellulosic components in

the alkali treatment occurred at a faster rate (Zhang, Okubayashi, & Bechtold, 2005). This may be the reason for the highly significant increase in tensile strength from 70° to 95° C at 4% concentration level. However, both at 7 and 10% concentration level, no significant differences ($P > 0.05$) in tensile strength were found from 70 to 95° C. This may be due to the fact that high concentration of NaOH (7 and 10%) caused greater removal of impurities at 70° C, and at these levels of concentrations, the increase in temperature up to 95° C did not cause any significant effects. Moreover, from Figure 4.12 a significant decrease ($P = 0.045$) in tensile strength was observed at 90° C with increase in concentration of NaOH from 7 to 10% indicating the possible destruction of cellulosic structure due to high temperature, and concentration of NaOH.

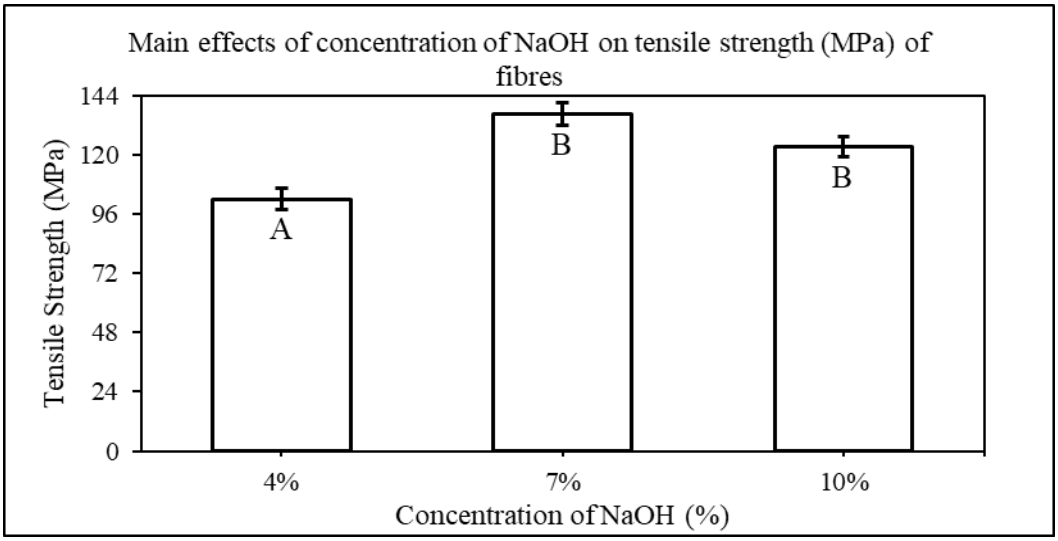

Figure 4.11: Main effects of concentration of NaOH on fibre tensile strength.

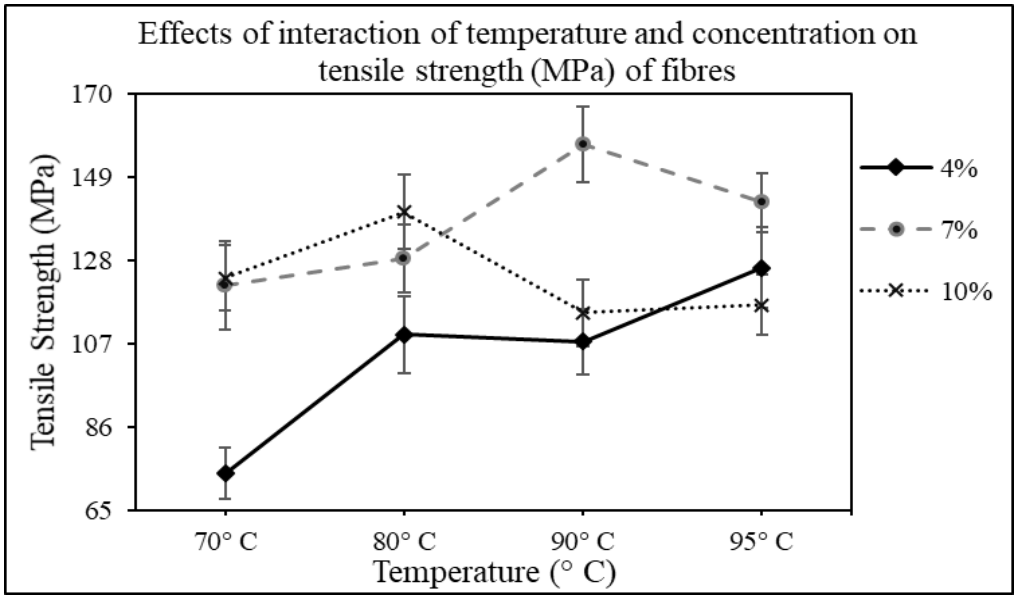

Figure 4.12: Effects of interaction of temperature and concentration on fibre tensile strength.

4.4 Fibre Modulus of Elasticity

The distribution of modulus of elasticity (GPa) of the fibres was also heavily skewed to the right with skewness = 1.57 and kurtosis = 4.18. The data was analyzed as a lognormal distribution with skewness = -0.11 and kurtosis = 0.31. The three-way factorial ANOVA of modulus of elasticity of the fibres is shown in Table 4.5. For pairwise comparison of the treatment means, the Tukey-Kramer post hoc analysis were conducted at $\alpha = 0.05$. From the ANOVA table (Table 4.4) it can be observed that the main effects of time, temperature, the interaction between time and concentration, and the interaction between temperature and concentration had significant influences on modulus of elasticity of fibres.

Table 4.5: Three-way factorial ANOVA of modulus of elasticity (GPa) of fibres.

Effects	Probability	Significance
Time	< 0.001	**
Temperature	< 0.001	**
Concentration	0.172	NS
Time*Temperature	0.912	NS
Time*Concentration	0.002	*
Temperature*Concentration	< 0.001	**
Time*Temperature*Concentration	0.423	NS

P < 0.05 = significant (*); P < 0.001 = Highly Significant (**); P > 0.05 = Not Significant (NS).

The main effects of time and temperature is shown in Figure 4.13 and 4.14 respectively. In these figures, bars with common letters indicate that the difference between the means are not significant (P > 0.05). For the main effects of time (Figure 4.13), the modulus of elasticity increased with increase in treatment time. This increase was significant (P < 0.05) between 4 and 10 h. (P < 0.001), 4 to 12 h. (P < 0.001), 6 to 10 h. (P = 0.039), and 6 to 12 h. (P = 0.0381). Similarly, for the main effects of temperature (Figure 4.14), the modulus of elasticity of fibres increased with increase in temperature. This increase was significant (P < 0.05) between 70 and 90° C (P = 0.014), 70 and 95° C (P < 0.001), and 80 and 95° C (P = 0.042).

The effects of interaction of time and concentration is shown in Figure 4.15. At 4% concentration level of NaOH, minor progressive increase in the modulus of elasticity of fibres were observed with increase in treatment time which was not significant (P > 0.05). Similarly, at 7% concentration level of NaOH, no significant changes in modulus of elasticity were observed for all levels of time. However, at 10% concentration level of NaOH, the modulus of elasticity increased

with increase in time which became significant ($P < 0.05$) between 4 and 10 h. ($P = 0.012$), 4 and 12 h. ($P = 0.002$), and 6 to 12 h. ($P = 0.009$).

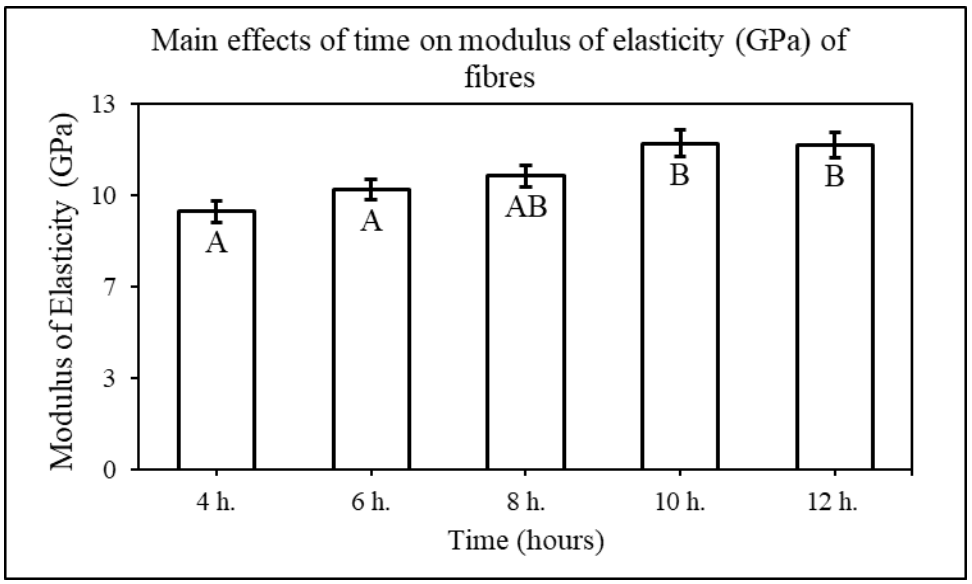

Figure 4.14: Main effects of time on fibre modulus of elasticity.

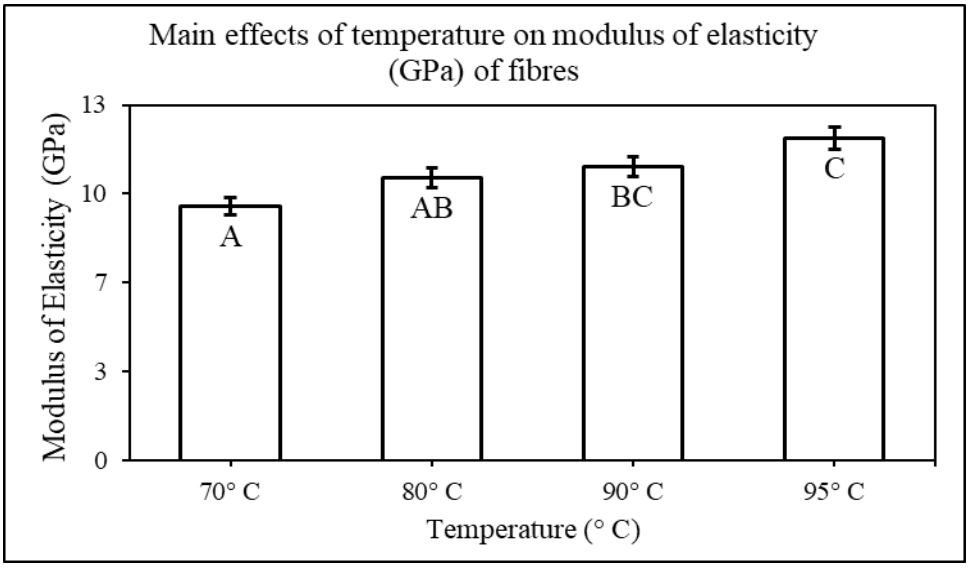

Figure 4.13: Main effects of temperature on fibre modulus of elasticity.

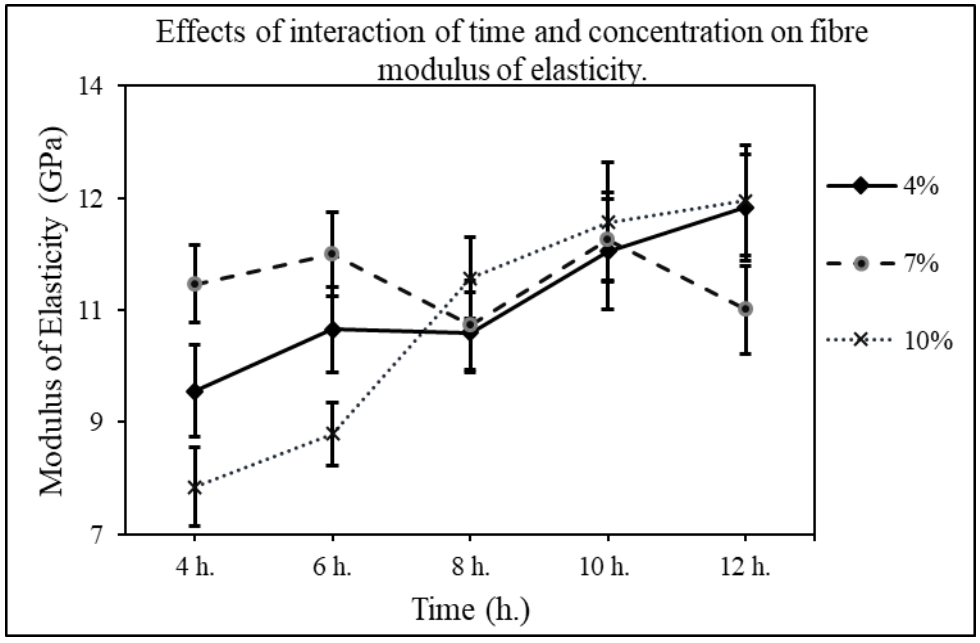

Figure 4.15: Effects of interaction of time and concentration on fibre modulus of elasticity.

Rong et al. (2001) reported that the alkali treatment of cellulosic fibres improves the molecular orientation of cellulose, and removes the impurities (primarily lignin, and hemi-cellulose) and therefore, increasing the crystallinity of the fibres . These treated fibres can result in higher fibre stiffness due to the increased crystallinity of hard cellulose and as a result, significant improvements in the modulus of elasticity of these fibres can be observed (Rong et al., 2001). Similar observations were found for cattail fibres as higher concentration of NaOH, and treatment duration increased the modulus of elasticity of the fibres (Figure 4.15). The mean of modulus of elasticity was maximum (12.21 GPa) at 10% concentration of NaOH when the fibres were treated for 12 hours.

Figure 4.16 illustrates the effects of interaction of temperature and concentration on the modulus of elasticity of fibres. It can be observed that, at 4% concentration level of NaOH, for all levels of temperature (70, 80, 90, and 95° C), the changes in modulus were not significant ($P > 0.05$). At

7% concentration level, a significant increase ($P < 0.05$) in the modulus was observed from 70° to 90° C ($P = 0.004$), 80° to 90° C ($P < 0.001$), and 80° to 95° C ($P = 0.009$). At 10% concentration level, from 70° up to 90° C, no significant differences ($P > 0.05$) in the modulus of fibres were observed. However, a significant increase in the modulus was observed ($P = 0.02$) when the temperature was increased from 90° to 95° C. Therefore, high concentration of NaOH treated at high temperature improved the modulus of fibres. As discussed above, removal of impurities, and improvements in molecular orientation increases the crystallinity of the alkali treated fibers (Rong, Zhang, Liu, Yang, & Zeng, 2001). At high temperature and concentration of NaOH, this process becomes faster and therefore, yielding coarser fibres with improved modulus of elasticity.

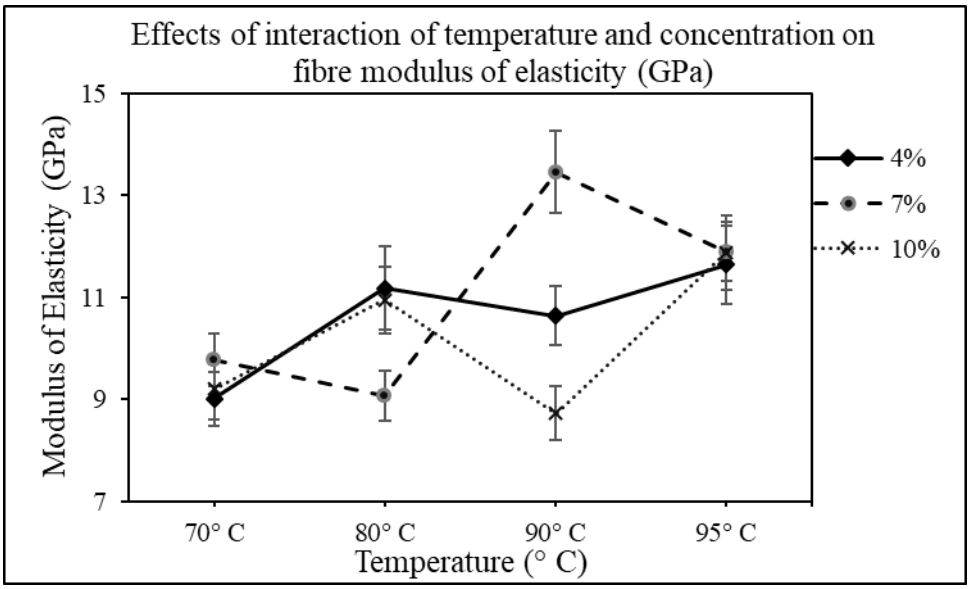

Figure 4.16: Effects of interaction of temperature and concentration on fibre modulus of elasticity.

4.5 Elongation at Break (%)

The distribution of elongation at break (%) of the fibres approximately followed the normal distribution with skewness = 0.66 and Kurtosis = 0.79. The three-way factorial ANOVA of elongation at break (%) is shown in Table 4.6. Due to the heterogeneity of the variances,

Satterthwaite approximation was used to calculate the effective degrees of freedom. From the Table 4.5 it can be seen that the elongation at break (%) of the fibres is influenced significantly ($p < .05$) by the main effects of time, temperature, and concentration, and the interaction between temperature and concentration. In order to compare the treatment means, Tukey-Kramer post hoc analysis was followed at $\alpha = 0.05$.

Table 4.6: Three-way factorial ANOVA of elongation at break (%) of fibres

Effects	Probability	Significance
Time	0.017	*
Temperature	0.002	*
Concentration	< 0.001	**
Time*Temperature	0.925	NS
Time*Concentration	0.282	NS
Temperature*Concentration	< 0.001	**
Time*Temperature*Concentration	0.071	NS

P < 0.05 = significant (*); P < 0.001 = Highly Significant (**); P > 0.05 = Not Significant (NS).

The main effects of time, temperature and concentration on fibre elongation at break (%) is shown in Figures 4.17-4.19 respectively. In these figures, bars with common letters indicate that the changes in means are not significant (P > 0.05). For the main effects of time (Figure 4.17), it can be observed that from 4 to 8 h., the elongation at break (%) slightly increased which was not significant (P > 0.05). Although, a significant decrease (P = 0.02) in the elongation at break (%) was observed from 8 to 12 h. For the main effects of temperature (Figure 4.18), a small increase in the elongation at break (%) was noticed from 70° to 80° C which is not significant (P > 0.05). However, from 80°C, the elongation at break (%) of the fibres started to decrease and between 80° and 95° C this decrease in elongation was found significant (P = 0.003). For the main effects of

concentration of NaOH (Figure 4.19), the elongation at break (%) increased with increase in concentration which was significant ($P < 0.05$) from 4 to 7% ($P < 0.001$) and 4 to 10% ($P < 0.001$).

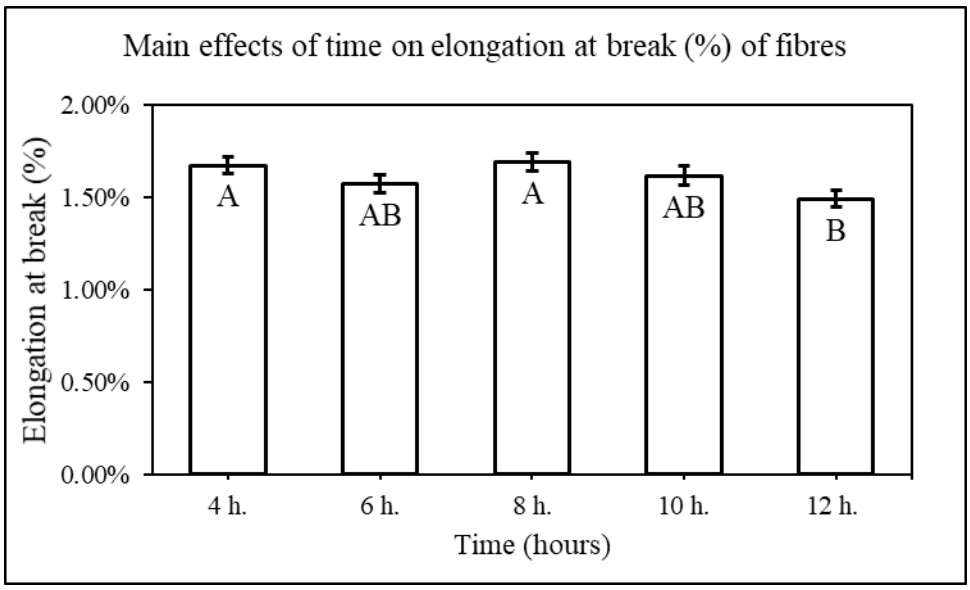

Figure 4.17: Main effects of time on fibre elongation at break (%).

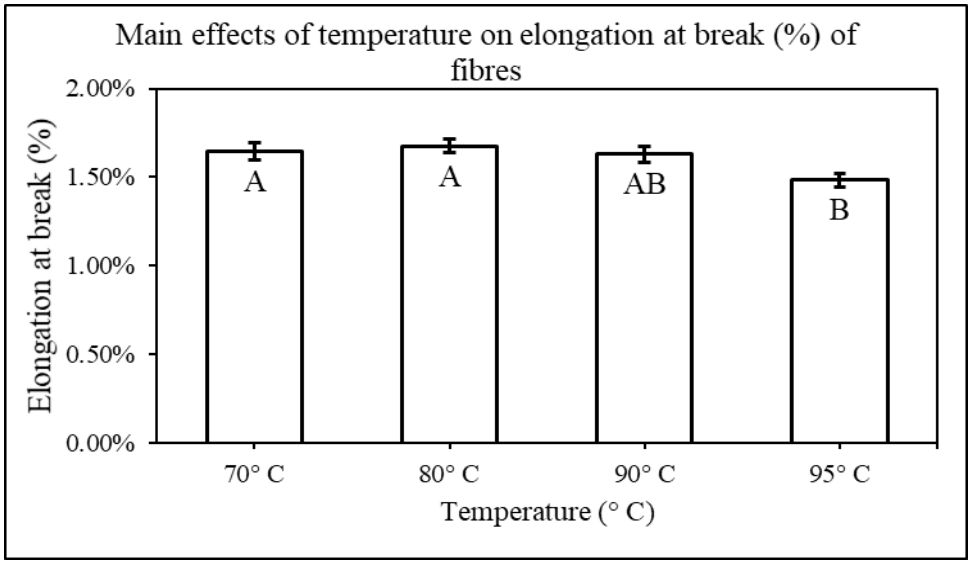

Figure 4.18: Main effects of temperature on fibre elongation at break (%).

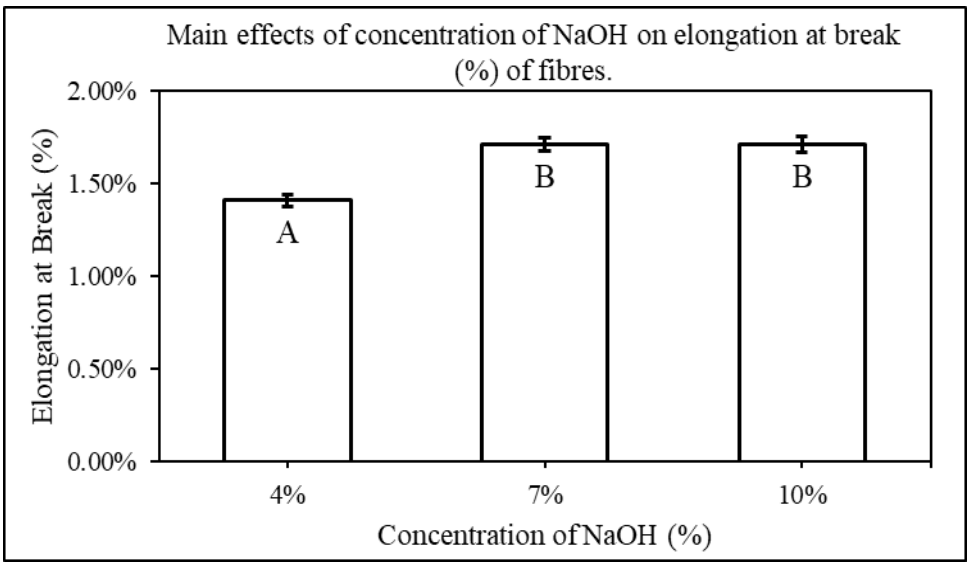

Figure 4.19: Main effects of concentration of NaOH on fibre elongation at break (%).

The effects of interaction of temperature and concentration on fibre elongation at break (%) is shown in Figure 4.20. At 4% concentration level of NaOH, with increase in temperature, continuous small increase in elongation at break (%) were observed. However, these increase in elongation was not significant ($P > 0.05$) for all levels of temperature. At 7% concentration level, the elongation at break (%) of the fibres slightly increased from 70° to 80° C which was not significant ($P > 0.05$). However, from 80° to 90° C the elongation at break (%) decreased significantly ($P < 0.001$). Further increase in temperature from 90° to 95° C did not cause any significant change ($P > 0.05$) in the elongation at break (%). Finally, at 10% concentration level of NaOH, an initial small increase in elongation at break (%) was observed from 70° to 90° C which was not significant ($P > 0.05$). However, when the temperature was increased from 90° to 95° C, the elongation at break (%) of the fibres reduced significantly ($P < 0.001$).

The small increase in elongation at break (%) may be due to the fact that, when cellulosic fibres are treated with alkali, the impurities i.e. pectin, lignin, hemi-cellulose, and fatty substances are removed which acted as a cementing material and therefore, making the fibres relatively ductile

(K, Reddy, & Gowda, 2015; Rong et al., 2001). However, increase in temperature and concentration of alkali (NaOH) significantly reduced the elongation at break (%) of the fibres. Which may be because high temperature and concentration of alkali cause higher removal of impurities, and improvements in molecular orientation and therefore, increase the crystallinity of the alkali treated fibers ((Rong et al., 2001). As a result, fibres become stiff with hard crystalline

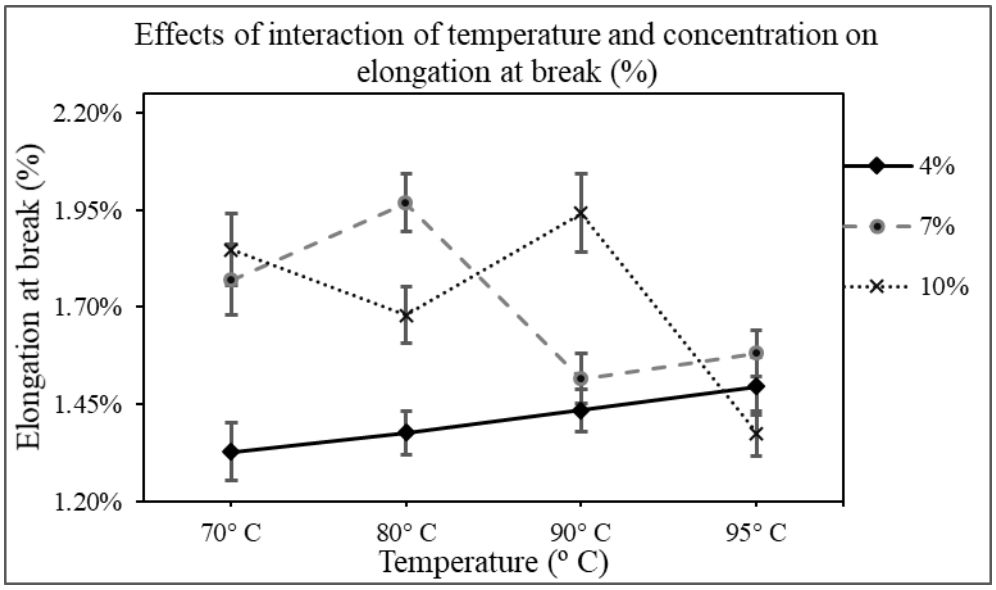

Figure 4.20: Effects of interaction of temperature and concentration on fibre elongation at break (%)

structure.

4.6 Moisture Regain (%)

The distribution of moisture regain (%) of fibres approximately followed a normal distribution with skewness = 0.88 and kurtosis = 0.49. The three-way factorial ANOVA for moisture regain is shown in Table 4.7. For post hoc analysis, Tukey-Kramer procedure was followed at α =0.05. Also, due to the heterogeneity of the variances, Satterthwaite approximation was used to determine the effective degrees of freedom. From Table 4.6, it can be found that the main effects of time, temperature, and concentration; and the interaction between temperature and concentration had

significant influences ($P < 0.05$) on moisture regain (%) of the cattail fibres. However, the post hoc analysis of the main effects of time as illustrated in Figure 4.21 revealed that there are no

Table 4.7: Three-way factorial ANOVA of moisture regain (%) of cattail fibres

Effects	Probability	Significance
Time	0.035	*
Temperature	< 0.001	**
Concentration	< 0.001	**
Time*Temperature	0.476	NS
Time*Concentration	0.576	NS
Temperature*Concentration	< 0.001	**
Time*Temperature*Concentration	0.067	NS

$P < 0.05$ = significant (*); $P < 0.001$ = Highly Significant (**); $P > 0.05$ = Not Significant (NS).

significant differences($p > 0.05$) between the treatment means for all levels of time.

Figure 4.22-4.23 shows the main effects of temperature, and concentration of NaOH on moisture regain (%) of fibres respectively. From Figure 4.22, it can be observed that with increase in treatment temperature, the moisture regain (%) of the fibres decreased which was significant ($p < 0.05$) between 70° and 80° C ($P < 0.001$), 70° and 90° C ($P < 0.001$), 70° and 95° C ($P < 0.001$), and 80° and 95° C ($P = 0.04$). Similarly, for the main effects of concentration of NaOH, it can be observed from Figure 4.23 that increase in concentration of NaOH decreased the moisture regain (%) which was significant ($p < 0.05$) from 4 to 7% ($P < 0.001$), and 4 to 10% ($P < 0.001$).

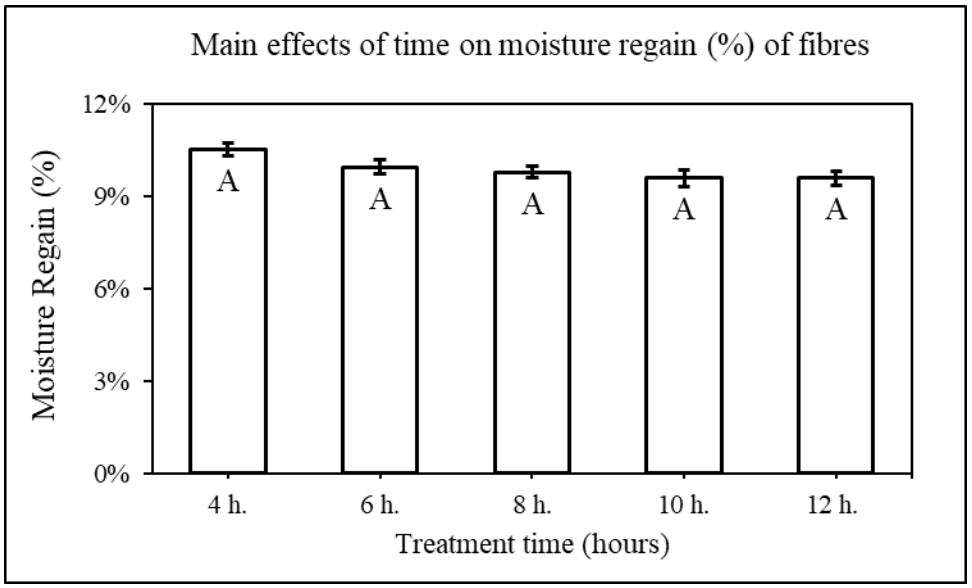

Figure 4.21: Main effects of time on fibre moisture regain (%).

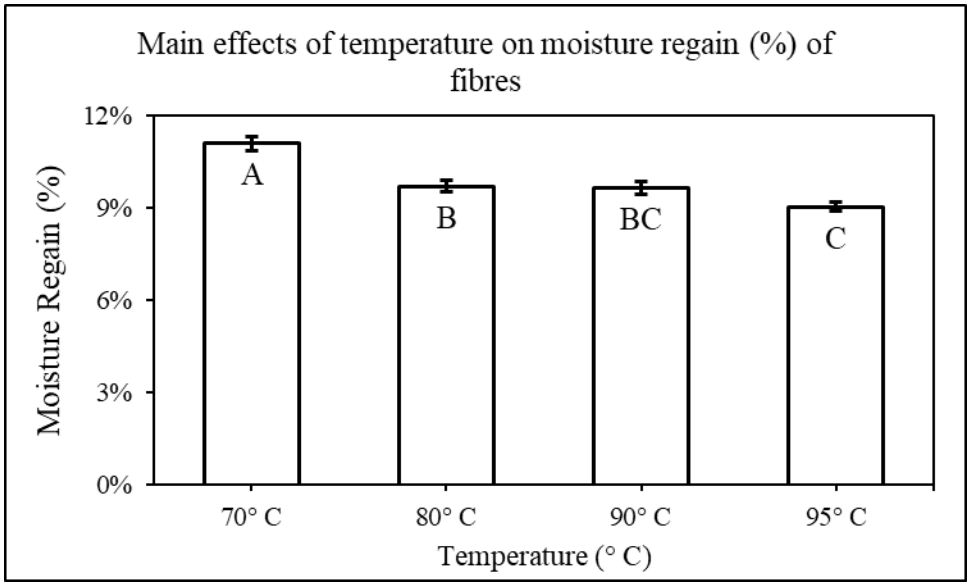

Figure 4.22: Main effects of temperature on fibre moisture regain (%)

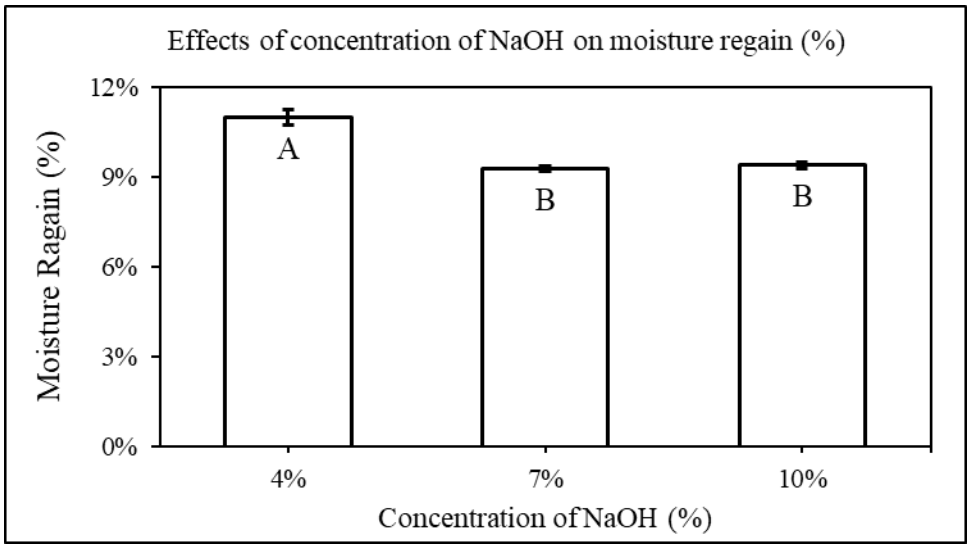

Figure 4.23: Main effects of concentration of NaOH on fibre moisture regain (%).

The effects of interaction of temperature and concentration on moisture regain (%) is shown in Figure 4.24. It can be observed that at 4% concentration level of NaOH, between 70°-90° C, the decrease in moisture regain (%) was not significant ($P > 0.05$). However, a significant decrease ($P = 0.001$) in moisture regain (%) was observed between 70° and 95° C at this level. At 7% concentration level of NaOH, a significant decrease ($P = 0.002$) in moisture regain (%) was observed from 70° to 80° C. However, when the temperature was further increased from 80° up to 95° C, small decrease in moisture regain (%) were observed which was not significant ($P > 0.05$). Similarly, at 10% concentration level, the moisture regain (%) decreased with increase in temperature which was significant ($P = 0.001$) between 70° and 90° C. The change in moisture regain (%) was not significant ($P = 0.083$) when the temperature was further increased from 90° to 95° C.

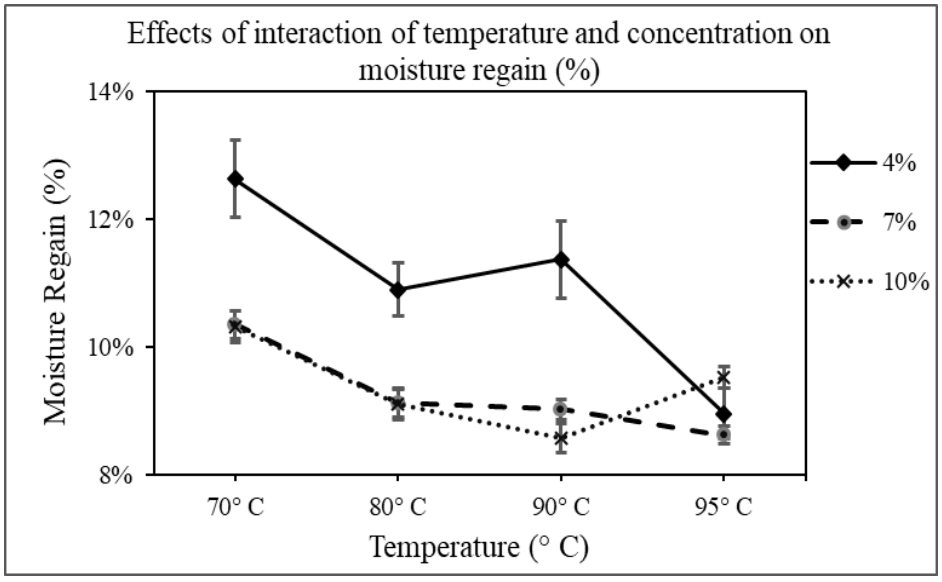

Figure 4.24: Effects of interaction of temperature and concentration of NaOH on fibre moisture regain (%)

In natural cellulosic fibres, moisture is attracted by the cell wall polymers due to the presence of hydroxyl and other oxygen containing groups which form hydrogen bond with a water molecule or a cluster of water (Pejic, Kostic, Skundric, & Praskalo, 2008; Sampathkumar, Punyamurthy, Bennehalli, & Venkateshappa, 2012). The hemicellulose present in the polymeric composite structure of natural fibres are primarily responsible for moisture absorption although, accessible cellulose, nanocrystalline cellulose, lignin and surface of crystalline cellulose also play major roles (Sampathkumar et al., 2012). Previous studies have revealed that, alkali treatment of natural cellulosic fibres lowers the moisture absorption due to the removal of lignin, and hemicellulose components from the composite structure (Pejic et al., 2008; Sampathkumar et al., 2012; Venkateshappa, Bennehalli, Kenchappa, & Ranganagowda, 2010). Similar finding were observed for cattail fibres. With increased temperature and concentration of NaOH, the removal of impurities were higher which may contribute to the decrease in moisture regain (%). From Figure

4.24, it can be found that the mean moisture regain (%) of fibres treated at 70° C and 4% concentration of NaOH was 12.63% which significantly reduced (P < 0.001) to 8.58% when treated at 90°C and 10% NaOH. However, the small not-significant (P = 0.083) increase in moisture regain (%) from 90° to 95° C at 10% NaOH may arise due to the inherent variability of naturally sourced materials.

4.7 Estimated Treatment Means for Each Response Variable

For each response variable i.e. yield (%), diameter, tensile strength, modulus of elasticity, elongation at break (%), and moisture regain (%), the model was simplified by removing the non-significant terms obtained from the ANOVA. The revised models of fibre yield (%), and fibre properties are shown in Table 4.8. The estimated treatment means ± standard error for all response variables obtained from each treatment are shown in Table 4.9-4.11 for 4, 7, and 10% concentration level of NaOH respectively.

Table 4.8: Simplified models of fibre yield (%) and fibre properties.

Response	Simplified Models	Definitions
Yield (%)	$Y_{ijkl} = \mu + T_i + t_j + C_k + TC_{ik} + e_{ijkl}$ …(4.1)	μ = population mean, T_i = effect of i'th treatment temperature on l'th response, t_j = effect of j'th treatment time on l'th response, C_k= effect of k'th concentration of NaOH on l'th response, tC_{jk}= effects of interaction of time and concentration on l'th response. TC_{ik}= effects of interaction of temperature and concentration on l'th response. e_{ijkl} = error variations.
Diameter (µm)	$Y_{ijkl} = \mu + T_i + t_j + C_k + TC_{ik} + e_{ijkl}$ …(4.2)	
Tensile strength (MPa)	$Y_{ijkl} = \mu + T_i + t_j + C_k + TC_{ik} + e_{ijkl}$ …(4.3)	
Modulus of elasticity (GPa)	$Y_{ijkl} = \mu + T_i + t_j + tC_{jk} + TC_{ik} + e_{ijkl}$ …(4.4)	
Elongation at break (%)	$Y_{ijkl} = \mu + T_i + t_j + C_k + TC_{ik} + e_{ijkl}$ …(4.5)	
Moisture regain (%)	$Y_{ijkl} = \mu + T_i + t_j + C_k + TC_{ik} + e_{ijkl}$ …(4.6)	

Table 4.9: Estimated treatment means of response variables at 4% concentration level of NaOH

4% concentration level of NaOH						
Trt.	Yield (%)	Diameter (μm)	Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Elongation at break (%)	Moisture Regain (%)
T_1	34.57 ± 0.79	163.1 ± 6.75	67.98 ± 6.07	8.023 ± 0.73	1.347 ± 0.07	13.40 ± 0.16
T_2	33.38 ± 0.78	150.3 ± 6.33	70.25 ± 6.13	8.977 ± 0.67	1.205 ± 0.07	12.62 ± 0.16
T_3	32.07 ± 0.76	144.3 ± 6.21	80.21 ± 6.68	8.841 ± 0.64	1.339 ± 0.07	12.66 ± 0.06
T_4	30.68 ± 0.75	136.7 ± 5.75	80.10 ± 7.06	9.865 ± 0.86	1.296 ± 0.08	12.51 ± 0.19
T_5	28.73 ± 0.73	127.8 ± 5.37	81.53 ± 7.30	10.30 ± 0.87	1.159 ± 0.07	12.15 ± 0.15
T_6	30.45 ± 0.76	132.3 ± 6.36	102.3 ± 9.33	9.694 ± 0.95	1.432 ± 0.06	12.27 ± 0.41
T_7	29.33 ± 0.74	121.9 ± 5.55	105.6 ± 8.98	10.85 ± 0.92	1.290 ± 0.07	11.49 ± 0.42
T_8	28.12 ± 0.73	117.0 ± 5.40	120.7 ± 10.6	10.69 ± 0.93	1.423 ± 0.07	11.53 ± 0.43
T_9	26.83 ± 0.71	110.9 ± 5.14	120.5 ± 10.9	11.92 ± 1.18	1.380 ± 0.07	11.37 ± 0.44
T_{10}	25.04 ± 0.69	103.6 ± 4.79	122.6 ± 10.8	12.45 ± 1.14	1.244 ± 0.06	11.02 ± 0.42
T_{11}	29.07 ± 0.75	122.1 ± 5.18	97.26 ± 8.21	9.352 ± 0.78	1.526 ± 0.06	12.30 ± 0.25
T_{12}	27.99 ± 0.73	112.5 ± 4.70	100.5 ± 8.30	10.47 ± 0.79	1.384 ± 0.07	11.52 ± 0.28
T_{13}	26.80 ± 0.72	108.0 ± 4.61	114.8 ± 9.48	10.31 ± 0.75	1.517 ± 0.07	11.56 ± 0.29
T_{14}	25.56 ± 0.70	102.3 ± 4.15	114.6 ± 9.69	11.50 ± 0.97	1.474 ± 0.07	11.40 ± 0.31
T_{15}	23.83 ± 0.67	95.68 ± 4.01	116.7 ± 9.80	12.01 ± 0.98	1.338 ± 0.07	11.05 ± 0.28
T_{16}	28.77 ± 0.74	114.1 ± 4.98	112.1 ± 9.73	10.06 ± 0.95	1.470 ± 0.07	9.577 ± 0.27
T_{17}	27.69 ± 0.73	105.2 ± 4.62	115.8 ± 9.73	11.26 ± 0.97	1.328 ± 0.07	8.778 ± 0.26
T_{18}	26.51 ± 0.71	100.9 ± 4.43	132.3 ± 11.2	11.09 ± 0.88	1.461 ± 0.08	8.818 ± 0.27
T_{19}	25.28 ± 0.69	95.65 ± 4.05	132.0 ± 11.1	12.37 ± 1.13	1.418 ± 0.07	8.665 ± 0.28
T_{20}	23.56 ± 0.67	89.42 ± 3.94	134.4 ± 11.3	12.91 ± 1.02	1.282 ± 0.07	8.310 ± 0.25

Table 4.10: Estimated treatment means of response variables at 7% concentration level of NaOH

7% concentration level of NaOH						
Trt.	Yield (%)	Diameter (μm)	Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Elongation at break (%)	Moisture Regain (%)
T_{21}	28.99 ± 0.74	126.9 ± 3.50	112.8 ± 10.5	9.843 ± 0.65	1.839 ± 0.09	11.27 ± 0.15
T_{22}	27.90 ± 0.73	116.9 ± 3.71	116.5 ± 9.91	9.953 ± 0.64	1.697 ± 0.09	10.49 ± 0.17
T_{23}	26.72 ± 0.71	112.2 ± 3.47	133.1 ± 11.9	9.589 ± 0.76	1.830 ± 0.09	10.54 ± 0.14
T_{24}	25.48 ± 0.70	106.4 ± 3.22	132.9 ± 12.0	10.57 ± 0.72	1.787 ± 0.09	10.38 ± 0.20
T_{25}	23.75 ± 0.67	99.45 ± 3.22	135.2 ± 12.3	9.874 ± 0.61	1.651 ± 0.09	10.03 ± 0.16
T_{26}	27.73 ± 0.73	111.7 ± 4.21	123.6 ± 8.79	9.429 ± 0.56	1.967 ± 0.08	9.686 ± 0.26
T_{27}	26.67 ± 0.72	102.9 ± 3.85	127.8 ± 9.25	9.542 ± 0.66	1.824 ± 0.07	8.908 ± 0.25
T_{28}	25.53 ± 0.70	98.77 ± 3.50	146.0 ± 10.3	9.186 ± 0.68	1.958 ± 0.08	8.948 ± 0.26
T_{29}	24.32 ± 0.68	93.61 ± 3.53	145.7 ± 10.2	10.12 ± 0.54	1.915 ± 0.08	8.794 ± 0.27
T_{30}	22.65 ± 0.65	87.52 ± 3.34	148.3 ± 11.2	9.471 ± 0.71	1.778 ± 0.08	8.440 ± 0.25
T_{31}	27.47 ± 0.73	95.24 ± 3.70	140.5 ± 10.2	13.04 ± 0.98	1.584 ± 0.07	9.839 ± 0.17
T_{32}	26.42 ± 0.72	87.76 ± 3.27	145.2 ± 10.2	13.20 ± 1.04	1.442 ± 0.07	9.060 ± 0.13
T_{33}	25.28 ± 0.70	84.24 ± 2.92	165.9 ± 11.4	12.70 ± 1.04	1.575 ± 0.07	9.101 ± 0.17
T_{34}	24.08 ±0.68	79.83 ± 2.78	165.6 ± 11.6	14.01 ± 1.03	1.532 ± 0.07	8.947 ± 0.19
T_{35}	22.42 ± 0.65	74.64 ± 2.79	168.5 ± 12.1	13.09 ± 0.96	1.395 ± 0.07	8.592 ± 0.14
T_{36}	25.81 ± 0.72	100.2 ± 3.37	127.6 ± 8.27	11.55 ± 0.75	1.647 ± 0.07	9.280 ± 0.13
T_{37}	24.80 ± 0.70	92.30 ± 2.86	131.8 ± 7.82	11.69 ± 0.80	1.505 ± 0.07	8.502 ± 0.11
T_{38}	23.71 ± 0.68	88.60 ± 2.66	150.5 ± 8.62	11.25 ± 0.80	1.638 ± 0.06	8.542 ± 0.13
T_{39}	22.56 ± 0.66	83.96 ± 2.35	150.3 ± 9.25	12.40 ± 0.75	1.595 ± 0.07	8.388 ± 0.17
T_{40}	20.97 ± 0.63	78.50 ± 2.62	153.0 ± 9.85	11.60 ± 0.85	1.459 ± 0.07	8.033 ± 0.07

Table 4.11: Estimated treatment means of response variables at 10% concentration level of NaOH

10% concentration level of NaOH						
Trt.	Yield (%)	Diameter (μm)	Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Elongation at break (%)	Moisture Regain (%)
T_{41}	28.15 ± 0.74	100.5 ± 4.11	116.5 ± 8.29	6.850 ± 0.64	1.814 ± 0.06	10.87 ± 0.17
T_{42}	27.09 ± 0.72	92.62 ± 3.32	120.4 ± 8.34	7.862 ± 0.61	1.671 ± 0.07	10.09 ± 0.17
T_{43}	25.93 ± 0.71	88.92 ± 3.49	137.5 ± 8.47	9.741 ± 0.62	1.805 ± 0.07	10.13 ± 0.11
T_{44}	24.71 ± 0.69	84.27 ± 3.31	137.3 ± 8.92	10.78 ± 0.99	1.762 ± 0.08	9.980 ± 0.20
T_{45}	23.02 ± 0.66	78.77 ± 3.02	139.7 ± 9.04	11.26 ± 0.94	1.625 ± 0.07	9.625 ± 0.16
T_{46}	28.48 ± 0.74	116.0 ± 4.85	124.5 ± 9.06	8.261 ± 0.69	1.739 ± 0.07	10.13 ± 0.12
T_{47}	27.40 ± 0.73	106.9 ± 4.22	128.7 ± 9.35	9.488 ± 0.72	1.596 ± 0.07	9.349 ± 0.14
T_{48}	26.24 ± 0.71	102.6 ± 4.18	147.0 ± 10.3	11.77 ± 0.89	1.730 ± 0.08	9.390 ± 0.15
T_{49}	25.01 ± 0.69	97.21 ± 4.04	146.7 ± 10.8	13.01 ± 1.17	1.686 ± 0.08	9.236 ± 0.18
T_{50}	23.30 ± 0.66	90.88 ± 3.77	149.3 ± 10.0	13.60 ± 1.17	1.550 ± 0.07	8.881 ± 0.10
T_{51}	26.47 ± 0.72	124.5 ± 4.42	105.5 ± 8.54	6.798 ± 0.59	1.988 ± 0.10	9.072 ± 0.18
T_{52}	25.45 ± 0.71	114.7 ± 3.73	109.0 ± 8.51	7.685 ± 0.51	1.846 ± 0.10	8.293 ± 0.18
T_{53}	24.33 ± 0.69	110.1 ± 3.68	124.5 ± 9.81	9.538 ± 0.73	1.979 ± 0.10	8.334 ± 0.18
T_{54}	23.17 ± 0.67	104.3 ± 3.57	124.3 ± 9.88	10.55 ± 0.97	1.936 ± 0.10	8.180 ± 0.22
T_{55}	21.55 ± 0.64	97.51 ± 2.94	126.4 ± 9.33	11.02 ± 0.87	1.799 ± 0.09	7.825 ± 0.16
T_{56}	26.16 ± 0.72	113.4 ± 4.17	107.8 ± 7.54	8.996 ± 0.79	1.462 ± 0.07	9.902 ± 0.17
T_{57}	25.15 ± 0.70	104.5 ± 3.32	111.4 ± 7.37	10.31 ± 0.51	1.319 ± 0.07	9.123 ± 0.11
T_{58}	24.04 ± 0.69	100.3 ± 3.34	127.3 ± 8.28	12.81 ± 0.93	1.453 ± 0.07	9.164 ± 0.17
T_{59}	22.88 ± 0.66	95.04 ± 3.39	127.1 ± 8.86	14.16 ± 1.23	1.410 ± 0.08	9.010 ± 0.18
T_{60}	21.28 ± 0.63	88.85 ± 3.26	129.4 ± 9.32	14.80 ± 1.26	1.273 ± 0.07	8.655 ± 0.15

4.8 Potential Composite Applications of Cattail Fibres

A comparison of properties of cattail fibres with other commonly used natural fibres i.e. flax, hemp, sisal, and coir used for composite applications are shown in Table 4.12 (Ku et al., 2011; Malkapuram et al., 2009; Wallenberger & Weston, 2004). From the table, it is evident that cattail fibres showed lower tensile strength and modulus of elasticity compared to flax, and hemp fibres. However, it showed similar tensile strength as coir fibres and the modulus of elasticity was found higher than coir fibres and almost similar to sisal fibres. Therefore, similar to sisal and coir fibre composites, the cattail fibre composites have potential applications in automotive and packaging industry.

In automotive industry, due to its similarity with sisal fibres, cattail fibre composites can be used to manufacture carrier for hard and soft armrests, seat back panels, door panels, door bolsters, headliners, side and back walls, seat backs, rear deck trays, pillars, center consoles, load floors, trunk trim etc. (Peças et al., 2018). However, these applications require high strength, and modulus, and the fibres extraction process needs to be optimized to obtain maximum strength, and modulus of fibres. Furthermore, in packaging industry, cattail fibre composites can be used to manufacture containers, boxes, trays, and packaging which require moderate strength and modulus similar to coir fibre composites (Ngo, 2018). Therefore, it is necessary to optimize the fibre extraction processes according to applications to obtain fibres of desired characteristics.

Table 4.12: Comparison of cattail fibres with other natural fibres commonly used for composites (Ku et al., 2011; Malkapuram et al., 2009; Wallenberger & Weston, 2004).

	Properties			
	Strength (MPa)	Modulus of elasticity (GPa)	Elongation at Break (%)	Moisture Regain (%)
Flax	600 - 1200	27.6	1.2 - 3	7
Hemp	690	70	1.6 – 4.5	8
Sisal	350 - 370	9.4 - 19	1.9 - 3	11
Coir	100 - 175	6	15 - 20	10
Cattail (Current Research)	68 - 169	6.8 - 15	1.16 - 2	8 - 13

5. CHAPTER: OPTIMIZING FIBRE EXTRACTION PROCESS

As discussed in chapter 4, section 4.8; cattail fibres obtained from *Typha latifolia* L. have the petential to be used as reinforcements in composites for automobile and packaging applications. However, the fibre extraction parameters (treatment time, temperature, and concentration of NaOH) need to be optimized, which would produce fibre characteristics i.e. yield (%), diamter, tensile strength, modulus of elasticity, elongation at break (%), and moisture regain (%) that are optimum for a specific composite application. In this study, the desirability function analysis (DFA) was adopted to optimize the fibre extarction process. In the following sections, the DFA approach for both automobilie and packaging applications to determine the optimum extraction parameters (concentration of NaOH, treatment time, and treatment temperature) and a sensitivity analysis on desirability functions for both applications are discussed.

5.1 Desirability Function Analysis for Automobile Applications

Before performing DFA to optimize the alkali extraction process of cattail fibres for automobile applications, the objectives of each property, the criteria/target value, and the upper/lower tolerance values need to be fixed. The fibre-reinforced automobile composites require high tenacity fibres i.e. high tensile strength, and high modulus which would act as a reinforcement of the matrix materials (Rana & Fangueiro, 2016). Fiber diameter is also an important factor because higher fiber volume fraction can be achieved from smaller diameter fibers which would better spread the fiber-matrix interfacial loads and increase the mechanical properties (Rana & Fangueiro, 2016). Moreover, in natural fibre-reinforced polymer composites, the moisture regain (%) needs to be lower as absorbed water changes the physical and chemical nature and degrades the mechanical properties of the materials (Wang & Chang, 1983).

The optimum values of fibre yield (%), and fibre properties of extracted cattail fibres obtained from the estimated treatment means are shown in Table 5.1. The cattail fibres showed a relatively low elongation at break (%) and the estimated means ranged between 1.16 – 2% (Table 4.10). It confirms the property of natural fibres having relatively low elongation. Due to the small range, this property was not included in the list of optimized properties. Finally, although the fibre yield (%) is expected to be higher to attain economic benefits, it is not directly related to the performance of automobile composites and therefore, omitted from the list of properties required to be optimized.

Table 5.1: Optimum values of fibre yield (%), and fibre properties of extracted cattail fibres

Response Variables	Optimum Values
Yield (%)	34.57 ± 0.79
Diameter (μm)	74.64 ± 2.79
Tensile strength (MPa)	168.5 ± 12.09
Modulus of elasticity (GPa)	14.80 ± 1.26
Elongation at break (%)	1.988 ± 0.10
Moisture regain (%)	7.825 ± 0.16

Therefore, the objectives tensile strength and modulus of elasticity of the fibres are to maximize; and the objectives of fibre diameter, and moisture regain (%) are to minimize. The objectives of fibre properties, the criteria/target values, the tolerance values, weight of individual property, the importance co-efficient of each property compared to others, and the reference values are listed in

Table 5.1. The reference values were taken from the properties of commonly used natural fibres used in composite applications (Peças et al., 2018; Wallenberger & Weston, 2004).

Table 5.2: The objectives of different properties, their relative weights, target values and tolerance values for automobile applications.

Fibre Properties	Objectives	Criteria or Target Value	Upper or Lower Tolerance Value	Weights	Importance coefficients	Reference Values
Moisture regain (%)	Minimize	8	11	1	1	Moisture regain (%) of sisal is 11 (Peças et al., 2018)
Diameter (μm)	Minimize	75	120	1	2	-----
Tensile strength (MPa)	Maximize	168	100	2	3	Tensile strength (MPa) of coir fibre is 100 – 200 (Gurunathan, Mohanty, & Nayak, 2015)
Modulus of elasticity (GPa)	Maximize	15	10	2	3	Modulus of elasticity (GPa) of sisal is 9.4 – 19 (Gurunathan et al., 2015)

For automobile applications, it is desirable for tensile strength, and modulus of elasticity to attain the optimum values and therefore, higher weights are given to these properties. Furthermore, higher importance coefficients are given to fibre diameter, tensile strength, and modulus as these are primary characteristics affecting the overall performance of composites (Peças et al., 2018).

After finalizing the objectives, criteria/target values, tolerance values, weights, and importance coefficients, the desirability index of individual property (d_i) was calculated by selecting the

appropriate formula listed from 3.4-3.9. Finally, the composite desirability (d_G) for each treatment by combining all the individual desirabilities were calculated using the formula 3.10. The individual desirability index of each property and the composite desirability for each treatment is listed in Table 5.3-5.5 for 4, 7 and 10% concentration of NaOH respectively. The individual desirability index (d_i) became 0 when the response variable in any treatment had a value outside of the tolerance resulting the composite desirability (d_G) equal to 0. Therefore, the treatment is rejected. By comparing these tables, it can be found that the highest composite desirability value was obtained from treatment 'T_{34}' (highlighted in Table 5.3). For this treatment, the concentration of NaOH, treatment duration, and treatment temperature is 7%, 10 h., and 90° C respectively (Table 3.2). Therefore, the optimum extraction parameters are: 7% (w/v) Concentration of NaOH, 10 hours treatment duration, and 90° C treatment temperature and the cattail fibres obtained from this treatment would be most suitable for automobile applications.

5.1.1 Validation of Optimum Treatment Conditions

The optimum values or the estimated treatment means of diameter (µm), tensile strength (MPa), modulus of elasticity (GPa), and moisture regain (%) for treatment 'T_{34}' are 79.83, 165.56, 14.01, and 8.73 respectively (Table 4.8). In order to validate the optimal treatment parameters obtained by the DFA, three trial measurements on fibre properties i.e. diameter, tensile strength, modulus of elasticity, and moisture regain (%) of cattail fibers treated with 7% (w/v) NaOH at 90° C for 10 h. were taken. Error (%) between the optimum values and the actual values were calculated using the following formula-

$$\text{Error (\%)} = \left[\frac{\text{Optimum Value} - \text{Actual Value}}{\text{Optimum Value}}\right] \times 100 \quad \text{.....(5.1)}$$

Table 5.3: Individual desirability indexes, and composite desirability for each treatment at 7% concentration level of NaOH for automobile applications.

4% concentration level of NaOH					
Trt.	Diameter (μm)	Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Moisture Regain (%)	Composite desirability (d_G)
T_1	0.000	0.000	0.000	0.000	0.000
T_2	0.000	0.000	0.000	0.000	0.000
T_3	0.000	0.000	0.000	0.000	0.000
T_4	0.000	0.000	0.000	0.000	0.000
T_5	0.000	0.000	0.004	0.000	0.000
T_6	0.000	0.001	0.000	0.000	0.000
T_7	0.000	0.007	0.029	0.013	0.000
T_8	0.067	0.093	0.019	0.070	0.049
T_9	0.203	0.091	0.147	0.133	0.133
T_{10}	0.364	0.110	0.240	0.130	0.190
T_{11}	0.000	0.000	0.000	0.000	0.000
T_{12}	0.167	0.000	0.009	0.000	0.000
T_{13}	0.267	0.047	0.004	0.000	0.000
T_{14}	0.392	0.046	0.090	0.000	0.000
T_{15}	0.540	0.060	0.162	0.000	0.000
T_{16}	0.131	0.032	0.0001	0.470	0.010
T_{17}	0.330	0.054	0.064	0.660	0.113
T_{18}	0.424	0.225	0.048	0.717	0.176
T_{19}	0.541	0.222	0.225	0.780	0.312
T_{20}	0.680	0.255	0.339	0.777	0.395

Table 5.4: Individual desirability indexes, and composite desirability for each treatment at 7% concentration level of NaOH for automobile applications.

7% concentration level of NaOH					
Trt.	Diameter (µm)	Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Moisture Regain (%)	Composite desirability *(d_G)*
T_{21}	0.000	0.035	0.000	0.007	0.000
T_{22}	0.068	0.059	0.000	0.197	0.000
T_{23}	0.172	0.237	0.000	0.253	0.000
T_{24}	0.303	0.234	0.013	0.317	0.098
T_{25}	0.457	0.268	0.000	0.313	0.000
T_{26}	0.185	0.121	0.000	0.417	0.000
T_{27}	0.380	0.167	0.000	0.603	0.000
T_{28}	0.472	0.457	0.000	0.663	0.000
T_{29}	0.586	0.452	0.001	0.723	0.055
T_{30}	0.722	0.505	0.000	0.720	0.000
T_{31}	0.550	0.355	0.370	0.447	0.407
T_{32}	0.716	0.442	0.410	0.637	0.500
T_{33}	0.795	0.938	0.292	0.693	0.592
T_{34}	0.893	0.930	0.643	0.757	0.796
T_{35}	1.000	1.000	0.382	0.753	0.703
T_{36}	0.441	0.164	0.096	0.580	0.197
T_{37}	0.616	0.219	0.114	0.770	0.255
T_{38}	0.698	0.552	0.063	0.827	0.294
T_{39}	0.801	0.547	0.230	0.890	0.471
T_{40}	0.922	0.607	0.102	0.887	0.384

Table 5.5:Individual desirability indexes, and composite desirability for each treatment at 7% concentration level of NaOH for automobile applications.

10% concentration level of NaOH					
Trt.	Diameter (µm)	Tensile Strength (MPa)	Modulus of Elasticity (GPa)	Moisture Regain (%)	Composite desirability *(d_G)*
T_{41}	0.433	0.059	0.000	0.020	0.000
T_{42}	0.608	0.090	0.000	0.210	0.000
T_{43}	0.691	0.304	0.000	0.267	0.000
T_{44}	0.794	0.301	0.024	0.330	0.163
T_{45}	0.916	0.341	0.064	0.327	0.241
T_{46}	0.090	0.130	0.000	0.420	0.000
T_{47}	0.292	0.178	0.000	0.610	0.000
T_{48}	0.387	0.477	0.125	0.667	0.303
T_{49}	0.506	0.472	0.362	0.730	0.461
T_{50}	0.647	0.525	0.518	0.727	0.568
T_{51}	0.000	0.006	0.000	0.597	0.000
T_{52}	0.118	0.017	0.000	0.787	0.000
T_{53}	0.220	0.130	0.000	0.843	0.000
T_{54}	0.348	0.127	0.012	0.907	0.090
T_{55}	0.500	0.151	0.042	0.903	0.156
T_{56}	0.147	0.013	0.00000	0.280	0.000
T_{57}	0.346	0.028	0.004	0.467	0.035
T_{58}	0.438	0.161	0.316	0.527	0.287
T_{59}	0.555	0.159	0.692	0.587	0.396
T_{60}	0.692	0.186	0.922	0.583	0.482

The results are listed in Table. 5.5. From the table it can be observed that the maximum error (%) is 13.78 and it was obtained for the modulus of elasticity (GPa) of the fibers. For other properties of fibers, the error (%) remained below 5. Therefore, it can be concluded that the optimum treatment conditions are valid (Zannen, Ghali, Halimi, & Hassen, 2016).

Table 5.6: Validation of optimum treatment conditions for automobile application.

Properties	Trial	Actual values	Mean actual values	Optimum values	|Error%|
Diameter (µm)	1	100.6	81.97	79.83	2.680
	2	67.78			
	3	77.50			
Tensile strength (MPa)	1	177.0	168.6	165.6	1.812
	2	179.7			
	3	149.1			
E-modulus (GPa)	1	10.40	12.18	14.01	13.78
	2	14.83			
	3	11.31			
Moisture regain (%)	1	9.212	8.957	8.947	0.112
	2	9.046			
	3	8.613			

5.1.2 Sensitivity Test of DFA for Automobile Applications

In order to conduct the sensitivity analysis of desirability functions as discussed in Chapter 3, section 3.3.2, the upper and lower edges of each parameter were assigned as shown in Table 5.7. Each parameter was designated by a letter A to L. For this particular sensitivity analysis, the number of parameters (factors) required to be investigated is significantly high i.e. 12 factors as importance coefficients, weights, and ranges of 4 responses are needed to be analyzed. The selected factors should be investigated by a 2-level design. For this kind of experiments, where large number of factors are involved, fractional-factorial design or Plackett-Burman design can be selected (Malenović et al., 2011). Finally, the Plackett-Burman design was selected as for this

sensitivity analysis, only the main effects are required to examine to draw relevant conclusions (Aksezer, 2008). Furthermore, this design is very helpful for detecting the main effects with a smaller number of experiments as this design can examine up to N-1 number of factors for N experiments.

Table 5.7: Parameter settings with upper and lower edges (automobile).

Parameters	Levels		
	Upper (+1)	Lower (-1)	Designation
Range for diameter (μm)	75 – 120	75 – 100	A
Range for tensile strength (MPa)	100 – 168	100 – 148	B
Range for modulus of elasticity (GPa)	10 – 15	10 – 13	C
Range for moisture regain (%)	8 – 11	8 – 10	D
Weight for diameter	4	1	E
Weight for tensile strength	4	1	F
Weight for modulus of elasticity	4	1	G
Weight for moisture regain (%)	4	1	H
Importance coefficient for diameter	5	1	I
Importance coefficient for tensile strength	5	1	J
Importance coefficient for modulus of elasticity	5	1	K
Importance coefficient for moisture regain (%)	5	1	L

For this analysis, the Plackett-Burman design with 20 runs was selected. The experimental set-up is shown in Table 5.8. The resulting values for overall desirability, as well as the corresponding input variables and the responses are shown in Table 5.9. All the appropriate calculations were conducted using the JMP® 14.1. software.

Table 5.8: The 20 run Plackett-Burman experimental design for 12 factors (automobile)

Run	Coded values for selected factors											
	A	B	C	D	E	F	G	H	I	J	K	L
1	-1	+1	+1	-1	+1	-1	-1	-1	+1	-1	+1	+1
2	+1	-1	+1	-1	+1	+1	+1	+1	+1	-1	-1	-1
3	-1	-1	+1	+1	-1	-1	+1	-1	-1	+1	-1	+1
4	-1	-1	+1	+1	+1	-1	-1	+1	-1	-1	-1	-1
5	+1	-1	-1	-1	+1	+1	-1	-1	-1	-1	-1	+1
6	-1	+1	-1	+1	+1	+1	-1	+1	-1	-1	+1	+1
7	+1	+1	-1	+1	-1	-1	+1	+1	+1	-1	-1	+1
8	-1	-1	-1	-1	+1	-1	+1	-1	+1	+1	+1	+1
9	+1	-1	+1	+1	-1	+1	+1	-1	-1	-1	+1	+1
10	+1	+1	-1	-1	-1	-1	-1	+1	-1	+1	-1	+1
11	-1	+1	-1	+1	+1	+1	1	-1	-1	+1	-1	-1
12	+1	+1	+1	+1	+1	-1	-1	-1	+1	+1	-1	-1
13	-1	-1	+1	-1	-1	+1	-1	+1	+1	+1	-1	+1
14	+1	+1	+1	-1	-1	+1	-1	-1	-1	+1	+1	-1
15	-1	+1	+1	-1	-1	-1	+1	+1	-1	-1	+1	-1
16	+1	-1	-1	-1	+1	-1	+1	+1	-1	+1	+1	-1
17	-1	-1	-1	+1	-1	+1	-1	+1	+1	+1	+1	-1
18	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1
19	-1	+1	-1	-1	-1	+1	+1	-1	+1	-1	-1	-1
20	+1	-1	-1	+1	-1	-1	-1	-1	+1	-1	+1	-1

Table 5.9: Overall desirability obtained from each run and corresponding input variable (automobile).

Run	Overall Desirability	Treatment	Corresponding input variables		
			Time	Temperature	Concentration
1	0.745	T_{35}	12 h.	90º C	7%
2	0.624	T_{35}	12 h.	90º C	7%
3	0.813	T_{34}	10 h.	90º C	7%
4	0.668	T_{35}	12 h.	90º C	7%
5	0.790	T_{40}	12 h.	95º C	7%
6	0.624	T_{35}	12 h.	90º C	7%
7	0.680	T_{39}	10 h.	95º C	7%
8	0.891	T_{35}	12 h.	90º C	7%
9	0.614	T_{60}	12 h.	95º C	10%
10	0.629	T_{39}	10 h.	95º C	7%
11	0.965	T_{35}	12 h.	90º C	7%
12	0.938	T_{35}	12 h.	90º C	7%
13	0.704	T_{40}	12 h.	95º C	7%
14	0.819	T_{34}	10 h.	90º C	7%
15	0.450	T_{60}	12 h.	95º C	10%
16	0.857	T_{35}	12 h.	90º C	7%
17	0.932	T_{35}	12 h.	90º C	7%
18	0.522	T_{34}	10 h.	90º C	7%
19	0.944	T_{35}	12 h.	90º C	7%
20	0.977	T_{35}	12 h.	90º C	7%

The resulting ANOVA is given in Table 5.10. Overall desirability model is found to be significant with a p-value < 0.05 and an acceptable adjusted R-square value of 0.8557. The prediction equation on the overall desirability is found as following-

Table 5.10: ANOVA for Plackett-Burman experiment (automobile)

Source	DF	Sum of Squares	Mean Sum of Squares	F-Ratio	Probability
Model	12	0.4397	0.0366	10.39	0.0024
Error	7	0.0247	0.0035		
Total	19	0.4644			

$$\text{Overall desirability } = 0.7593 - 0.0143(\text{A}) - 0.0277(\text{B}) - 0.0696(\text{C}) + 0.014(\text{D})$$

$$+ 0.0031(\text{E}) - 0.0055(\text{F}) - 0.0233(\text{G}) - 0.0903(\text{H}) + 0.0364(\text{I}) + 0.0477(\text{J})$$

$$- 0.0162(\text{K}) - 0.0581(\text{L})$$

After analyzing the parameter estimates, it can be concluded that the weight for moisture regain (H) is the most sensitive parameter. The negative sign indicates that any increase in weight for moisture regain will decrease the overall desirability rapidly. Therefore, selecting the weight for moisture regain to 1 while determining the optimum parameters for automobile applications was a prudent decision. Furthermore, the weight for tensile strength (F), and the weight for diameter proved to be least sensitive. The proposed procedure allowed the sensitivity of the important characteristic parameters of the desirability function and their impact on optimal solution to be analyzed. However, it should be noted that optimum levels of input variables (time, temperature, and concentration of NaOH) obtained from different design points ended up with equal settings

with different overall desirability levels, which proved that the higher overall desirability does not necessarily mean a better solution.

5.2 Desirability Function Analysis for Packaging Applications

As shown in Table 4.10, cattail fibres showed similar tensile strength and higher modulus of elasticity compared to coir fibres. Therefore, similar to coir fibres, cattail fibre composites can be used in packaging industry to manufacture containers, boxes, and trays (Ngo, 2018). Before performing DFA to optimize the alkali extraction process of cattail fibres for packaging applications, the objectives of the properties, the criteria/target values, and the upper/lower tolerance values need to be fixed. The modulus of elasticity of cattail fibres are found higher than coir fibres for all treatments (Table 4.10). Therefore, this property was not optimized. Furthermore, as cattail fibres showed a relatively low elongation at break (%) and the estimated means ranged between 1.16 – 2% (Table 4.10), this property was not included in the list of optimized properties. Finally, although good mechanical performance is also a prerequisite for packaging applications (Ludueña, Vázquez, & Alvarez, 2012), the requirement is not as high as automobile applications (For example, the tensile strength of modulus of elasticity of coir fibre is 100 – 175 MPa and 6 GPa respectively). Therefore, the fibre yield (%) was added to the list of properties to be optimized to attain higher economic benefits.

The objectives of fibre properties, the criteria/target values, the tolerance values, weight of individual property, the importance co-efficient of each property compared to others, and the reference values are listed in Table 5.11. The reference values were taken from the properties of commonly used natural fibres used in composite applications (Peças et al., 2018; Wallenberger & Weston, 2004).

Table 5.11: The objectives of different properties, their relative weights, target values and tolerance values for packaging applications.

Fibre Properties	Objectives	Criteria or Target Value	Upper or Lower Tolerance Value	Weights	Importance coefficients	Reference Values
Moisture regain (%)	Minimize	8	11	1	1	Moisture regain (%) of sisal is 11 (Peças et al., 2018)
Diameter (μm)	Minimize	75	120	1	1	-----
Tensile strength (MPa)	Maximize	168	100	1	1	Tensile strength (MPa) of coir fibre is 100 – 200 (Gurunathan et al., 2015)
Yield (%)	Maximize	25	35	1	1	Fibre content (%) in flax is 25 (Subramanian, 2017)

For packaging applications, equal weights and importance coefficients are given to all properties. The desirability index of individual property (d_i) was calculated by selecting the appropriate formula listed from 3.4-3.9 after finalizing the objectives, criteria/target, and tolerance values. Finally, by combining all the individual desirabilities, the composite desirability (d_G) for each treatment was calculated using the formula 3.10. The individual desirability index of each property and the composite desirability for each treatment is listed in Table 5.12-5.14 for 4, 7 and 10% concentration of NaOH respectively. The individual desirability index (d_i) became 0 when the response variable in any treatment had a value outside of the tolerance resulting the composite desirability (d_G) equal to 0. Therefore, the treatment is rejected. By comparing these tables, it can be found that the highest composite desirability value was obtained from treatment 'T_{32}'

(highlighted in Table 5.8). For this treatment, the concentration of NaOH, treatment duration, and treatment temperature is 7%, 6 h., and 90° C respectively (Table 3.2). Therefore, the optimum extraction parameters are: 7% (w/v) Concentration of NaOH, 6 hours treatment duration, and 90° C treatment temperature and the cattail fibres obtained from this treatment would be most suitable for packaging applications.

5.2.1 Validation of Optimum Treatment Conditions

The optimum values or the estimated treatment means of fibre yield (%), diameter (µm), tensile strength (MPa), and moisture regain (%) for treatment 'T_{32}' are 26.42, 87.76, 145.21, and 9.09 respectively (Table 4.8). In order to validate the optimal treatment parameters obtained by the DFA, three trial measurements on fibre yield (%), and fibre properties i.e. diameter, tensile strength, and moisture regain (%) of cattail fibers treated with 7% (w/v) NaOH at 90° C for 6 h. were taken. Error (%) between the optimum values and the actual values were calculated using the formula 5.1.

The results of error (%) are listed in Table 5.15. From the table it can be observed that the maximum error (%) is 14.46 and it was obtained for the tensile strength (MPa) of the fibers. For other properties of fibers, the error (%) remained below 10. Therefore, it can be concluded that the optimum treatment conditions are valid (Zannen et al., 2016).

Table 5.12: Individual desirability indexes, and composite desirability for each treatment at 4% concentration level of NaOH for packaging application.

4% concentration level of NaOH					
Trt.	Diameter (µm)	Tensile Strength (MPa)	Yield (%)	Moisture Regain (%)	Composite desirability *(d_G)*
T_1	0.000	0.000	0.957	0.000	0.000
T_2	0.000	0.000	0.838	0.000	0.000
T_3	0.000	0.000	0.707	0.000	0.000
T_4	0.000	0.000	0.568	0.000	0.000
T_5	0.000	0.000	0.373	0.000	0.000
T_6	0.000	0.033	0.545	0.000	0.000
T_7	0.000	0.083	0.433	0.013	0.000
T_8	0.067	0.304	0.312	0.070	0.145
T_9	0.203	0.301	0.183	0.133	0.197
T_{10}	0.364	0.332	0.004	0.130	0.089
T_{11}	0.000	0.000	0.407	0.000	0.000
T_{12}	0.167	0.008	0.299	0.000	0.000
T_{13}	0.267	0.218	0.180	0.000	0.000
T_{14}	0.392	0.215	0.056	0.000	0.000
T_{15}	0.540	0.245	0.000	0.000	0.000
T_{16}	0.131	0.178	0.377	0.470	0.253
T_{17}	0.330	0.232	0.269	0.660	0.342
T_{18}	0.424	0.475	0.151	0.717	0.384
T_{19}	0.541	0.471	0.028	0.780	0.273
T_{20}	0.680	0.505	0.000	0.777	0.000

Table 5.13: Individual desirability indexes, and composite desirability for each treatment at 7% concentration level of NaOH for packaging applications.

7% concentration level of NaOH					
Trt.	Diameter (µm)	Tensile Strength (MPa)	Yield (%)	Moisture Regain (%)	Composite desirability (d_G)
T_{21}	0.000	0.188	0.399	0.007	0.000
T_{22}	0.068	0.242	0.290	0.197	0.175
T_{23}	0.172	0.487	0.172	0.253	0.246
T_{24}	0.303	0.483	0.048	0.317	0.217
T_{25}	0.457	0.518	0.000	0.313	0.000
T_{26}	0.185	0.348	0.273	0.417	0.293
T_{27}	0.380	0.409	0.167	0.603	0.354
T_{28}	0.472	0.676	0.053	0.663	0.325
T_{29}	0.586	0.672	0.000	0.723	0.000
T_{30}	0.722	0.711	0.000	0.720	0.000
T_{31}	0.550	0.596	0.247	0.447	0.436
T_{32}	0.716	0.665	0.142	0.637	0.456
T_{33}	0.795	0.968	0.028	0.693	0.350
T_{34}	0.893	0.964	0.000	0.757	0.000
T_{35}	1.000	1.000	0.000	0.753	0.000
T_{36}	0.441	0.405	0.081	0.580	0.303
T_{37}	0.616	0.468	0.000	0.770	0.000
T_{38}	0.698	0.743	0.000	0.827	0.000
T_{39}	0.801	0.739	0.000	0.890	0.000
T_{40}	0.922	0.779	0.000	0.887	0.000

Table 5.14: Individual desirability indexes, and composite desirability for each treatment at 10% concentration level of NaOH for packaging applications.

10% concentration level of NaOH					
Trt.	Diameter (µm)	Tensile Strength (MPa)	Yield (%)	Moisture Regain (%)	Composite desirability (d_G)
T_{41}	0.433	0.243	0.315	0.020	0.160
T_{42}	0.608	0.300	0.209	0.210	0.299
T_{43}	0.691	0.551	0.093	0.267	0.312
T_{44}	0.794	0.548	0.000	0.330	0.000
T_{45}	0.916	0.584	0.000	0.327	0.000
T_{46}	0.090	0.360	0.348	0.420	0.262
T_{47}	0.292	0.422	0.240	0.610	0.366
T_{48}	0.387	0.690	0.124	0.667	0.386
T_{49}	0.506	0.687	0.001	0.730	0.126
T_{50}	0.647	0.724	0.000	0.727	0.000
T_{51}	0.000	0.080	0.147	0.597	0.000
T_{52}	0.118	0.132	0.045	0.787	0.153
T_{53}	0.220	0.360	0.000	0.843	0.000
T_{54}	0.348	0.357	0.000	0.907	0.000
T_{55}	0.500	0.389	0.000	0.903	0.000
T_{56}	0.147	0.115	0.116	0.280	0.153
T_{57}	0.346	0.168	0.015	0.467	0.142
T_{58}	0.438	0.401	0.000	0.527	0.000
T_{59}	0.555	0.398	0.000	0.587	0.000
T_{60}	0.692	0.432	0.000	0.583	0.000

Table 5.15: Validation of optimum treatment conditions for packaging application.

Properties	Trial	Actual values	Mean actual values	Optimum values	|Error%|
Yield (%)	1	29.24	28.84	26.42	9.160
	2	28.91			
	3	28.37			
Diameter (μm)	1	80.82	86.84	87.76	1.048
	2	91.15			
	3	88.56			
Tensile strength (MPa)	1	170.0	166.2	145.2	14.46
	2	179.7			
	3	149.0			
Moisture regain (%)	1	9.613	9.233	9.060	1.909
	2	9.144			
	3	8.942			

5.2.2 Sensitivity Test of DFA for Packaging Applications

The same procedure was followed to perform the sensitivity test of desirability functions for packaging applications as followed for automobile applications. First, the upper and lower edges of each parameter were assigned as shown in Table 5.16. Each parameter was designated by a letter A to L. Similar to automobile application, the number of parameters (factors) required to be investigated for this sensitivity test is significantly high i.e. 12 factors as importance coefficients, weights, and ranges of 4 responses are needed to be analyzed. The Plackett-Burman design was selected for this sensitivity analysis, as only the main effects are required to examine to draw relevant conclusions (Aksezer, 2008).

For this analysis, the Plackett-Burman design with 20 runs was selected. The experimental set-up is shown in Table 5.17. The resulting values for overall desirability, as well as the corresponding

input variables and the responses are shown in Table 5.18. All the appropriate calculations were conducted using the JMP® 14.1. software.

Table 5.16: Parameter settings with upper and lower edges (packaging).

Parameters	Levels		
	Upper (+1)	Lower (-1)	Designation
Range for yield (%)	25 – 35	25 – 30	A
Range for diameter (µm)	75 – 120	75 – 100	B
Range for tensile strength (MPa)	100 – 168	100 – 148	C
Range for moisture regain (%)	8 – 11	8 – 10	D
Weight for yield (%)	2	0.5	E
Weight for diameter	2	0.5	F
Weight for tensile strength	2	0.5	G
Weight for moisture regain (%)	2	0.5	H
Importance coefficient for yield (%)	3	1	I
Importance coefficient for diameter	3	1	J
Importance coefficient for tensile strength	3	1	K
Importance coefficient for moisture regain (%)	3	1	L

The resulting ANOVA is given in Table 5.19. Overall desirability model is found to be significant with a p-value << 0.05 and an acceptable adjusted R-square value of 0.9229. The prediction equation on the overall desirability is shown below.

Table 5.17: The 20 run Plackett-Burman experimental design for 12 factors (packaging)

Run	Coded values for selected factors											
	A	B	C	D	E	F	G	H	I	J	K	L
1	-1	-1	+1	+1	-1	-1	1	-1	-1	+1	+1	+1
2	-1	+1	+1	-1	-1	+1	-1	-1	+1	+1	+1	+1
3	+1	-1	+1	-1	-1	-1	-1	+1	+1	-1	-1	+1
4	-1	-1	+1	+1	+1	+1	-1	+1	-1	+1	-1	-1
5	-1	-1	+1	-1	-1	+1	1	+1	+1	-1	+1	-1
6	+1	-1	-1	-1	-1	+1	1	-1	-1	+1	-1	-1
7	-1	-1	-1	+1	+1	-1	-1	+1	-1	-1	+1	+1
8	-1	+1	-1	+1	-1	-1	-1	-1	+1	+1	-1	-1
9	+1	+1	-1	+1	-1	+1	-1	-1	-1	-1	+1	+1
10	+1	-1	-1	+1	-1	-1	+1	+1	+1	+1	-1	+1
11	-1	+1	-1	-1	-1	-1	+1	+1	-1	-1	+1	-1
12	+1	+1	+1	-1	+1	-1	+1	-1	-1	-1	-1	+1
13	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1
14	-1	+1	-1	-1	+1	+1	+1	+1	-1	+1	-1	+1
15	+1	-1	-1	+1	+1	+1	+1	-1	+1	-1	+1	-1
16	+1	+1	+1	+1	-1	+1	-1	+1	-1	-1	-1	-1
17	+1	+1	-1	-1	+1	-1	-1	+1	+1	+1	+1	-1
18	-1	+1	-1	-1	+1	+1	-1	-1	+1	-1	-1	+1
19	-1	+1	+1	+1	+1	-1	+1	-1	+1	-1	-1	-1
20	+1	-1	+1	-1	+1	-1	-1	-1	-1	+1	+1	-1

Table 5.18: Overall desirability obtained from each run and corresponding input variable (packaging)

Run	Overall Desirability	Treatment	Corresponding input variables		
			Time	Temperature	Concentration
1	0.750	T_{33}	8 h.	90º C	7%
2	0.623	T_{32}	6 h.	90º C	7%
3	0.358	T_{32}	6 h.	90º C	7%
4	0.268	T_{32}	6 h.	90º C	7%
5	0.434	T_{33}	8 h.	95º C	7%
6	0.445	T_{33}	8 h.	90º C	7%
7	0.492	T_{32}	6 h.	90º C	7%
8	0.737	T_{31}	12 h.	90º C	7%
9	0.740	T_{32}	8 h.	90º C	7%
10	0.505	T_{32}	6 h.	90º C	7%
11	0.639	T_{48}	8 h.	80º C	10%
12	0.364	T_{32}	6 h.	90º C	7%
13	0.208	T_{32}	6 h.	90º C	7%
14	0.310	T_{32}	6 h.	90º C	7%
15	0.194	T_{31}	4 h.	90º C	7%
16	0.503	T_{32}	6 h.	90º C	7%
17	0.283	T_{17}	6 h.	95º C	4%
18	0.280	T_{32}	6 h.	90º C	7%
19	0.377	T_{46}	4 h.	80º C	10%
20	0.473	T_{32}	6 h.	90º C	7%

Table 5.19: ANOVA for Plackett-Burman experiment (packaging)

Source	DF	Sum of Squares	Mean Sum of Squares	F-Ratio	Probability
Model	12	0.5687	0.0474	19.97	0.0003
Error	7	0.0166	0.0024		
Total	19	0.5853			

$$\text{Overall desirability } = 0.4492 - 0.0419(\text{A}) + 0.0293(\text{B}) - 0.0134(\text{C}) + 0.0283(\text{D})$$

$$- 0.1243(\text{E}) - 0.0487(\text{F}) - 0.0266(\text{G}) - 0.0492(\text{H}) - 0.0493(\text{I}) + 0.0111(\text{J})$$

$$+ 0.0345(\text{K}) + 0.0139(\text{L})$$

After analyzing the parameter estimates, it can be concluded that the weight for yield (%) (E) is the most sensitive parameter. The negative sign indicates that any increase in weight for yield (%) will decrease the overall desirability rapidly. Therefore, selecting the weight for yield (%) to 1 while determining the optimum parameters for packaging applications was a wise decision. Furthermore, the range for tensile strength (C), and the importance coefficient for diameter (J) proved to be least sensitive. The proposed procedure allowed the sensitivity of the important characteristic parameters of the desirability function and their impact on optimal solution to be analyzed. However, similar to the sensitivity test of desirability for automobile applications, optimum levels of input variables (time, temperature, and concentration of NaOH) obtained from different design points ended up with equal settings with different overall desirability levels, which proved that the higher overall desirability does not necessarily mean a better solution.

6. CHAPTER: SUMMARY, CONCLUSION, AND FUTURE WORK

Due to the high productivity of *Typha* plants, presence of high amount of cellulose in the chemical structure, and similarity in textile characteristics to commonly used cellulosic fibres such as flax, hemp, sisal, and coir, cattail fibres can be a potential substitute to synthetic fibres such as glass and carbon for applications, such as automobiles, aerospace, and packaging industries where a high load carrying capacity is not required (Bajwa et al., 2015; Chakma, 2018). In this study, cattail fibres obtained from the leaves of *Typha latifolia* L. (broad-leaf cattail) were examined as a new source of natural fibre for bio-composite applications.

6.1 Summary of Findings

Previous studies have revealed that, alkali extraction, particularly treated with NaOH at high temperature is the simplest, and effective way to extract fibres from cattail plants with least environmental impacts (Chakma, 2018; Sana et al., 2014; Xiao et al., 2001). However, change in extraction parameters (treatment time, temperature, and concentration of NaOH) influences the fibre yield (%), and fibre characteristics (Sana et al., 2014; Xiao et al., 2001). Therefore, in order to determine the effects of treatment time, temperature, and concentration of NaOH on fibre yield (%), and important composite characteristics of cattail fibres i.e. diameter, tensile strength, modulus of elasticity, elongation at break (%), and moisture regain (%), a 3*4*5 factorial experimental design with 3 levels of concentration of NaOH (4, 7, 10% w/v), 4 levels of temperature (70, 80, 90, and 95º C), and 5 levels of treatment time (4, 6, 8, 10, and 12 h.) was used to extract fibres. A three-way factorial ANOVA of fibre yield (%), and fibre properties were conducted to determine the significance of the main effects (time, temperature, and concentration of NaOH) and their interactions.

The three-way factorial ANOVA revealed that, for both fibre yield (%) and fibre diameter, the main effects of time, temperature, and concentration of NaOH, and the interaction between temperature and concentration had significant influences ($p < .05$). When analyzing the effect of interaction of temperature and concentration on fibre yield (%) and diameter, it was observed that at both 4 and 7% concentration level of NaOH, the fibre yield (%) and diameter decreased significantly ($P < 0.05$) when the temperature was increased from 70º to 95º C. However, at 10% concentration level, no significant difference ($P > 0.05$) in yield (%) and diameter was observed between 70° and 95° C.

This could be due to the probable fact that at 4 and 7% concentration level, the hydrolysis of pectin took place at high temperature by sodium hydroxide with the formation of sodium pectate (Bhattacharya & Das, 2001). Therefore, the removal of impurities was accelerated when the temperature was increased. However, at high level of concentration (10%), the removal of impurities was already higher at 70° C and the increase in temperature (up to 95°C) did not have any significant effect.

For the mechanical properties of cattail fibres i.e. tensile strength, modulus of elasticity, and elongation at break (%), the three-way factorial ANOVA revealed that all three main effects (time, temperature, and concentration of NaOH) have significant influences ($P < 0.05$) on tensile strength and elongation at break (%). However, the modulus of elasticity of the fibres were only influenced by time and temperature ($P < 0.05$). When analyzing the effects of interaction on the mechanical properties of fibres, it was found that both tensile strength, and elongation and break (%) was significantly influenced ($P < 0.05$) by the interaction between temperature and concentration of NaOH. However, for modulus of elasticity of fibres, interaction between time and concentration, and interaction between temperature and concentration had significant influences ($P < 0.05$).

For tensile strength, the interaction between temperature and concentration showed that at 4% concentration level, the strength of the fibres increased significantly ($P < 0.001$) from 70º to 90º C. However, at 7 and 10% concentration level, the increase in temperature did not have any significant effect ($P > 0.05$). This may be due to the fact that high concentration of NaOH (7 and 10%) caused greater removal of impurities at 70° C which minimized effects of the increase in temperature up to 95° C.

For elongation at break (%), the interaction between temperature and concentration showed that at 4% concentration level, the change in elongation of the fibres were not significant. However, the elongation of the fibres decreased significantly from 80º C to 95º C ($P = 0.004$) at 7% concentration level, and from 70º C to 95º C ($P < 0.001$) at 10% concentration level. This may be because high temperature and concentration of alkali caused higher removal of impurities and improved the molecular orientation of the fibres. and therefore, increasing the crystallinity of the alkali treated fibers ((Rong et al., 2001). As a result, fibres become stiff with hard crystalline structure.

For modulus of elasticity of the fibres, the interaction between time and concentration showed that at both 4 and 7% concentration level, the change in modulus of the fibres were not significant ($P > 0.05$) with increase in treatment time. However, at 10% concentration level, with increase in treatment time from 4 to 12 h., the modulus of elasticity of the fibres increased significantly ($P = 0.002$). Furthermore, the interaction between temperature and concentration showed that at 4% concentration level, the increase in temperature from 70º C up to 95º did not have any significant effect on the modulus of fibres. However, the modulus of fibres increased significantly from 80º to 95º C ($P < 0.001$) at 7% concentration level, and from 90° to 95° C ($P = 0.002$) at 10% concentration level. Therefore, high concentration, high temperature, and longer treatment time improved the modulus of elasticity of cattail fibres. As discussed earlier, alkali treatment of

cellulosic fibres improves the molecular orientation of cellulose, and removes the impurities and therefore, increasing the crystallinity of the fibres (Rong et al., 2001). At high temperature, high concentration, and longer treatment time this process becomes faster and therefore, yielding thinner fibres with improved modulus of elasticity.

Finally, the three-way factorial ANOVA of moisture regain (%) of fibres showed that the main effects of time, temperature, and concentration, and the interaction between temperature and concentration had significant influences ($P < 0.05$). For the main effects, it was observed that the moisture regain (%) decreased with increase in treatment time, temperature, and concentration of NaOH. When analyzing the effects of interaction of temperature and concentration, it was found that at 4% concentration level, the moisture regain (%) decreased significantly ($P = 0.001$) with increase in temperature from 70º to 95º C. Similarly, a significant decrease in moisture regain (%) was observed from 70º to 80º ($P = 0.001$) at 7% concentration level, and from 70º to 90º C ($P = 0.002$) at 10% concentration level. Therefore, high temperature, longer treatment duration, and high concentration of NaOH decreased the moisture regain (%) of cattail fibres. These findings match with previous results which revealed that alkali treatment of natural cellulosic fibres lowers the moisture absorption due to the removal of lignin, and hemicellulose components from the composite structure (Pejic et al., 2008; Sampathkumar et al., 2012; Venkateshappa et al., 2010). With increased temperature, time and concentration of NaOH, the removal of impurities (lignin, pectin, hemicellulose) were higher which may contribute to the decrease in moisture regain (%).

The non-significant terms of the factorial models obtained from the ANOVA were removed to calculate the estimated treatment means for all response variables for each treatment. The maximum and minimum treatment means of response variables i.e. fibre yield (%), and fibre properties with standard error are summarized in Table 6.1.

Table 6.1: Maximum and minimum estimated treatment means of response variables.

Response Variables	Maximum	Minimum
Yield (%)	34.57 ± 0.79	20.97 ± 0.63
Diameter (μm)	163.1 ± 6.75	74.64 ± 2.79
Tensile strength (MPa)	168.5 ± 12.09	67.98 ± 6.07
Modulus of elasticity (GPa)	14.80 ± 1.26	6.70 ± 0.59
Elongation at break (%)	1.988 ± 0.10	1.159 ± 0.07
Moisture regain (%)	13.40 ± 0.16	7.825 ± 0.16

6.2 Conclusion

The estimated treatment means were compared with the commonly used natural fibre for composite applications and it was found that the cattail fibres has tensile strength similar to coir fibre and modulus of elasticity similar to sisal fibre and higher than coir fibre. Therefore, similar to sisal fibre reinforced composites, cattail fibre reinforced bio-composites can be used in automobile industry to manufacture carrier for hard and soft armrests, seat back panels, door panels, door bolsters, headliners, side and back walls, seat backs, rear deck trays, pillars, center consoles, load floors, trunk trim etc. (Peças et al., 2018). Moreover, due to its similarity with coir fibres, cattail fibre reinforced composites can be used in packaging industry to manufacture containers, boxes, and trays (Ngo, 2018).

Therefore, in order to produce fibre characteristics i.e. yield (%), diamter, tensile strength, modulus of elasticity, elongation at break (%), and moisture regain (%) that are optimum for a specific composite application i.e. automobile or packaging, the fibre extraction parameters (treatment time, temperature, and concentration of NaOH) were optimized using DFA.

The DFA calculated the optimum extraction parameters for automobile applications as: 7% (w/v) concentration of NaOH, 10 hours treatment duration, and 90° C treatment temperature and the cattail fibres obtained from this treatment would be most suitable for automobile applications. Similarly, the DFA calculated the optimum extraction parameters for packaging applications as: 7% (w/v) concentration of NaOH, 6 hours treatment duration, and 90° C treatment temperature and the cattail fibres obtained from this treatment would be most suitable for packaging applications. The optimum values with standard errors of the response variables are listed in Table 6.2.

Table 6.2: Optimum values of response variables for automobile and packaging applications.

Response Variables	Optimum Values for Automobile Application	Optimum Values for Packaging Application
Yield (%)	24.08 ± 0.68	26.42 ± 0.72
Diameter (μm)	79.83 ± 2.78	87.76 ± 3.27
Tensile strength (MPa)	165.6 ± 11.59	145.2 ± 10.21
Modulus of elasticity (GPa)	14.01 ± 1.03	13.20 ± 1.04
Elongation at break (%)	1.532 ± 0.07	1.442 ± 0.07
Moisture regain (%)	8.947 ± 0.19	9.060 ± 0.13

The optimum treatment parameters (time, temperature, and concentration of NaOH) obtained by the DFA were validated by taking three trial measurements on fibre yield (%), and fibre properties, and calculating the error (%). For automobile application, the maximum error (%) was 13.78 which was obtained for the modulus of elasticity (GPa) of the fibers. For other properties of fibers, the error (%) remained below 5. For packaging application, the maximum error (%) was 14.46 which

was obtained for the tensile strength (MPa) of fibers. For other properties of fibers, the error (%) remained below 10. Therefore, it was be concluded that the optimum treatment conditions were valid (Zannen et al., 2016).

A sensitivity analysis of desirability functions for both automobile and packaging applications were performed following the procedure described by Aksezer (2008). This test allowed the effects of range of individual properties, the weights of the properties, and the importance coefficient of each property in the composite desirability to be analyzed. The analysis showed that the optimum treatment conditions can be changed by changing the range, weight and importance coefficient of individual property. Therefore, it should be noted that higher composite desirability does not necessarily mean the better treatment. However, the analysis showed that majority of optimum settings for time, temperature, and concentration of NaOH found in the sensitivity analysis matched with the optimum conditions determined for both automobile and packaging applications which confirmed the validity of the optimum treatment conditions.

6.3 Future Work

This study has determined the application of cattail fibres reinforced bio-composites in automobile and packaging industries. The optimum extraction parameters (time, temperature, and concentration of NaOH) obtained by the DFA would help manufacturers to set production parameters that would produce fibre characteristics that are most desirable for the specific composite application. However, one of the important factors that controls the fibres' contribution in the composites is the fibre-matrix interfacial bonding (Khan, 2016). High wetting of the fibers by the matrix is an indication and pre-requisite of perfect bonding between the fibers and the matrix. The degree of wetting can be measure by the contact angle (θ) between the liquids and the

fibers with higher contact angle generally indicates lower wettability. Therefore, the future work would focus on determining the contact angle between cattail fibres and polymer matrices to select the suitable polymer matrix for a specific composite application. Also, bio-composites, reinforced by cattail fibres optimized for automobile and packaging applications, would be manufactured and their mechanical properties would be evaluated.

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