Investigation of using Typha latifolia fibers to enhance polyurethane foam's sound absorption properties.

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Abstract:

This study focused on measuring the sound absorption property of Typha latifolia fibers, and to investigate its use on enhancing polyurethanes' sound absorption properties. Typha latifolia (Broadleaf Cattail) is a common perennial marsh, which has been used in many applications. In Germany, Typha latifolia's insulation and acoustic capabilities have been titled as revolutionary in wall panel technology. Polyurethanes are one of the most versatile materials in the world. One of the applications where polyurethanes are being used, is sound insulation. Finding a way to lower the use of polyurethanes and replacing them with a greener alternative, which could potentially have the same sound absorption qualities, is always an attractive study subject. The fibers were extracted from Typha latifolia leaves by retting. The retting was done in a 100-degree water-sodium hydroxide-solution (98 % H2O, 2 % NaOH). Half of the fibers were separated by hand, and the other half was separated by using a carding tool. Hand separation kept the fibers longer and thicker compared to fibers that were separated with a carding tool. The sound absorption measurement was executed only for Typha latifolia fibers due to budget and schedule reasons. The sound absorption property of Typha latifolia fibers was good. The good result can be explained by material's porous, wool-like structure. Part of the fiber's environment friendliness is its biodegradability. Fiber's biodegradability was investigated by placing the fibers into a composter for four weeks. The experiment was done in winter circumstances, and despite of challenging conditions, it was proven that the fibers will decompose just in four weeks. Closed-cell MDI-polyurethane foam, provided by Kevra Oy, was used in this study. Polyurethane's cell structure was modified to be more porous, by using a water blow-method, which improved material's sound absorption property. Adding Typha latifolia fibers to a non-modified polyurethane did not open the cells of polyurethane, which meant that composite's sound absorption property stayed poor. Water-blown polyurethane's structure was ideal for sound absorption purposes. It was challenging to create a homogeneous mix from Typha latifolia fibers and water-blown polyurethane, and the enhancing effect of Typha latifolia fibers to material's sound absorption property was left as questionable. Typha latifolia fibers stood out from other natural fibers with their capability to create a strong bond with polyurethane. Next logical step would be to investigate if Typha latifolia fibers are suitable for making a textile.

Keywords:	Typha latifolia, Cattail, natural fiber, sound absorption, polyurethane composite
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Tiivistelmä:

Tämä tutkimus keskittyi Typha latifolia-kasvista tehdyn kuidun äänenabsorptio-ominaisuuksien mittaamiseen sekä kuidun käyttämiseen polyuretaanin äänenabsorptiokyvyn parantamiseksi. Typha latifolia (leveäosmankäämi) on yleinen monivuotinen kosteikkojen kasvi, jota on hyödynnetty jo pitkään eri käyttötarkoituksissa. Saksassa kasvista saatavien tuotteiden odotettaan mullistavan eriste- ja akustiikkalevymarkkinat. Polyuretaani on yksi monikäyttöisimpiä materiaaleja. Yksi polyuretaanin käyttökohteista on äänieristys, jossa vaahtopolyuretaanin rakennetta hyödynnetään kaiun poistamisessa. Polyuretaanin käytön vähentäminen lisäämällä materiaalin joukkoon ympäristöystävällisempää materiaalia, joka parhaimmillaan parantaa polyuretaanin ominaisuuksia, on aina kiinnostava tutkimuskohde. Kuitu valmistettiin Typha latifolia-kasvin lehdistä liuotusmenetelmällä. Liottaminen tapahtui 100 asteisessa vesi-natriumhydroksidi-seoksessa (98 % vesi, 2 % natriumhydroksidi). Puolet kuiduista erotettiin toisistaan käsin ja loppujen erottamiseen käytettiin käsikarstainta. Käsin erottaminen piti kuidut pidempinä ja paksumpina verrattuna karstaimella erotettuihin kuituihin. Äänenabsorptiomittaus toteutettiin vain Typha latifolia-kuiduille budjetti- sekä aikataulusyistä johtuen. Typha latifolia-kuitujen äänenabsorptiokyky oli odotetun hyvä. Hyvä tulos johtui materiaalin huokoisesta, villatyyppisestä rakenteesta. Osa kuidun ympäristöystävällisyyttä on sen kompostoitavuus. Typha latifolia-kuidun kompostoitavuutta testattiin pilottitason kokeella, jossa kuidut laitettiin kotikäyttöön suunniteltuun kompostoriin. Talviolosuhteissa tehty koe osoitti, että Typha latifolia-kuitu maatuu jo neljässä viikossa vaikeissakin oloissa. Polyuretaanina käytettiin Kevra Oy:n umpisoluista MDI-polyuretaanivaahtoa. Polyuretaanin solurakennetta muokattiin avoimemmaksi vesipuhallusmenetelmää käyttäen, jotta materiaali absorboisi ääntä. Pelkkä Typha latifolia-kuidun sekoittaminen muokkaamattomaan umpisoluiseen polyuretaaniin ei riittänyt tekemään komposiitista riittävän huokoista, joka on edellytys hyvään äänenabsorptioon. Vesipuhallettu polyuretaanivaahto oli rakenteeltaan sopiva hyvään äänenabsorptioon. Kuitujen sekoittaminen muokattuun polyuretaaniin oli haastavaa ja niiden vaikutus materiaalin äänenabsorptiokykyyn jäi kyseenalaiseksi. Monista luonnonkuiduista poiketen Typha-latifolia-kuitu sitoutui erinomaisesti polyuretaaniin. Seuraava luonnollinen askel tutkimukselle on selvittää, soveltuuko Typha latifolia-kuidut tekstiileihin.

Avainsanat:	Typha latifolia, leveäosmankäämi, äänenabsorptio, polyuretaani komposiitti
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LIST OF ABBREVIATIONS

MDI	Methylene diphenyl diisocyanate
PU	Polyurethane

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FOREWORD

I owe a great deal to many people for their help and support as I have worked on this thesis. Firstly, I want to thank my family and friends. Their support and presence have helped me to stay positive and energetic throughout the peculiar year of 2020.

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Juha Linjala

Helsinki

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1 INTRODUCTION

1.1 Background

Noise has a lowering effect on human health, the quality of living, potential land use, and the value of residential area (Ministry of the Environment, 2018). The growth in population and industrial advancements have resulted to new applications in noise cancelling industry (Karlinasari, Hermawan, Maddu, 2012). In recent years noise pollution has gained fair amount of attention, which has led to a rise in noise pollution cancelling solutions to increase welfare of environment and human health (Cai Xiaobing et al. 2014). Initiatives to minimize waste and reach environmental sustainability continuously push research and development in the field of derivatives such as green composites (Rissanen, 2016). A greener alternative, which could potentially have the same sound absorption qualities as the more established materials, is always an attractive subject to study.

Different parts of *Typha latifolia*, which belongs to the family of Typhaceae, have been utilized in many applications. Young sprouts and inflorescences were used as a substitute for vegetables, pillows were filled with pappuses due its insulation qualities, and old sprouts are excellent animal feed (Luontoportti, 2020). Typha latifolia (Commonly broadleaf cattail) is a common perennial marsh, or wetland plant in temperate, tropical, and subtropical climates throughout the Northern Hemisphere. Plants are typically 1.5-3 meters high, with 2-4 cm wide leaves (Plants for A Future). By using chemical retting, natural fibers can be obtained from Typha leaves (S.M. Mortazavi et al. 2009).

In Germany, Typha latifolia's insulation and acoustic capabilities have been titled as revolutionary in wall panel technology (Helsingin Sanomat, 16.3.2019). Typha fiber has been successfully introduced as a leaf fiber for development of new acoustic non-woven composite for lightweight structures (Moghaddam, et al. 2016). In this study, a composite will be made by mixing Typha fibers and polyurethane. Polyurethanes are, if not the most versatile class of materials, then certainly one of the most versatile polymer categories in existence (Sonnenschein, 2014). Low-density polyurethane foam is used as a sound absorbent e.g., in automotive industry (Szycher, 2012). The sound absorption properties of polyurethane can be improved by mixing it with natural fibers, and as an additional benefit, increasing the natural-fiber content in the polyurethane foam matrix reduces the amount of PU used, thus lessening the environmental impact, and reducing the cost of the developed material (Çelebi, et al. 2012).

1.2 Objectives

The main objective of this thesis was to study Typha latifolia's sound absorption properties and investigate its use and suitability as an enhancement, when creating a novel sound absorbing-composite. The experimental work focused on producing Typha fibers with a coarse surface, and removing impurities such as pectin, lignin, hemicellulose, wax, and fat materials.

The normal incidence sound absorption coefficient of Typha latifolia fibers was to bemeasured in the acoustic laboratory at Turku University of Applied Sciences. The biodegradability of Typha fibers was to be determined by performing a pilot-scale composting test. Microscope pictures was to be taken in Arcada University of Applied Sciences, to study the bonding between Typha fibers and polyurethane. The laboratory work was to be performed in the chemistry laboratory at Arcada.

The sound absorption ratio of polyurethane foam was planned to be increased by modifying its structure and mixing Typha latifolia fibers into it.

In summary, the objectives were:

- Produce Typha latifolia fibers.
- Determine the following fiber's properties: sound absorption coefficient, biodegradability, and fiber's ability to bond with polyurethane.
- Manufacturing polyurethane-Typha-composite based on our learnings.
- Analysing the polyurethane-Typha-composite samples and discussing the results.

1.3 Approach

A comprehensive study of published literature was conducted comparing the studies made from Typha's properties and the studies of sound absorption properties of polyurethane-natural fiber composite. Additional information relating to the subject was also gathered. This study is divided into seven chapters. The first chapter will focus on introducing this work, by stating the reason of study, the objectives, and the approach. The literature review gives the reader a holistic understanding of Typha latifolia properties and current trends of the use of natural fibers in sound absorbing applications. The manufacturing methods of Typha fibers and polyurethane will be covered, and the theory of determining the specific airflow and normal incidence sound absorption coefficient with an impedance tube. The literature review will also cover the biodegradability test. In the third chapter the experiments will be performed and obtained data will be presented in chapter 4. In the fifth chapter, the data that was obtained will be analysed. From the sixth chapter, the reader can find the discussion-section which also contains recommendations for future studies. The final chapter contains the conclusion.

2 LITERATURE REVIEW

2.1 Sound absorption and sound absorbing structures

Every material- and structure-layer absorbs and insulates sound. Easiest way to get to an excellent sound insulation is to have a massive and impenetrable layer of material. An excellent sound insulator reflects the sound waves back to its source (Hongisto, 2020).
In many instances this reflection of sound is not desired since it will disrupt original sound's orientation and the individual reflections are not resolved by the ear, and may perceived as echoes i.e., we hear the sound signal twice. Echo effects can disrupt music and speech quite considerably and should therefore be avoided (Mommertz, 2012).
Sound absorption is the measurement of the amount of energy removed from the sound wave as the wave passes through a given thickness of material (Anshuman, 2018). A good sound absorbent lets the sound in without almost any resistance and changes the acoustic energy to heat energy. After the sound has travelled through the material, the acoustic power has been reduced by an amount that is indicated by the sound absorption coefficient. The sound will be transformed to heat because the material pores cause friction to particle vibration in the material (Hongisto, 2020).

Good sounding absorbing structures are:

- Porous materials (like wools, cushions, and fabrics),
- Panel resonators, and
- Hemholtz resonator (Hongisto, 2020).

2.2 The use of polyurethanes in sound absorption

Flexible polyurethane foams are very versatile materials and are used in many applications such as in mattresses, cushions, sheets, padding, aircraft seats, and sound proofing (Defonseka, 2019). Egg-crate surfaced polyurethane foams are effective sound absorbers and are used e.g., in anechoic chambers.

Open-cell polyurethane foams in general have many factors that can affect to materials sound absorbing properties (Mills, 2007):

- Porosity
- Permeability
- Viscous characteristic length Λ that relates to the high frequency response.
- Polymer modulus and internal damping.
- Shape of the foam block related to the supporting panel or tube (Mills, 2007).

Closed-cell polyurethanes are used in sound absorbing applications where water ingress is unwanted. Polyurethane foams suffer from hydrolysis and can act as sponges (Mills, 2007).

The closed-cell structure obstructs the air flow through the subject which affects negatively to the polyurethanes sound absorbing properties, as can be seen from results of study made by Oliva et al. 2010 (figure 2).

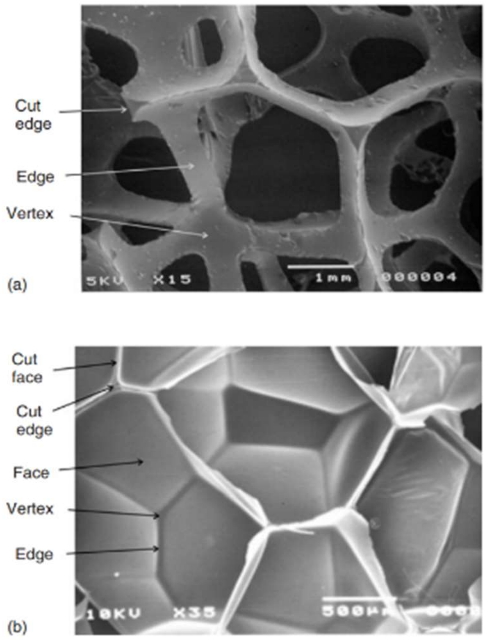


Figure 1:SEM-picture of open-cell (above) and closed-cell (below) structures of polyurethane (Mills, chapter 1.1, 2007)

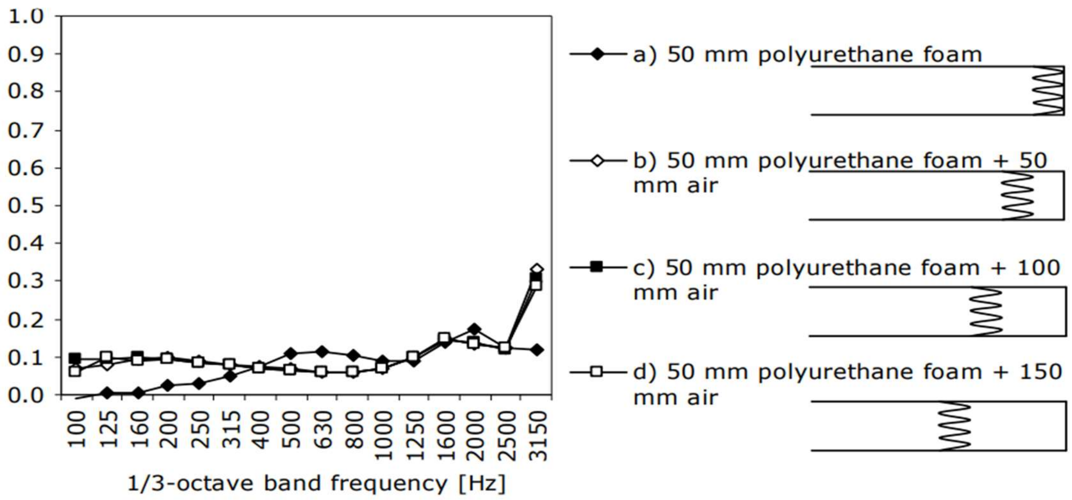


Figure 2:The absorption coefficient of Finnfoam FI300/50 (Oliva et al., page 36, 2010).

Addition to polyurethanes' amazing versatility, they are also attractively priced. Global growth and increasing prosperity have made plastics, including polyurethanes, more abundant, resulting in more abundant waste products that does not easily degrade in the environment. Compared to other plastics like polyethylene (PE) and polyethylene terephthalate (PET), polyurethanes are highly inhomogeneous materials. The large variety of polyurethanes causes that the recycling of them is extremely challenging and expensive. This is why most of the polyurethanes end up to landfills or to waste-to-energy facilities (Sonnenschein, 2020).

2.3 The use of fibers in polyurethanes to enhance sound absorption.

Natural fiber composites are proposed to substitute synthetic fibers composites due to several advantages such as renewability, less abrasiveness to equipment, biodegradability and low weight and cost (El-Shekeil et al. 2014). As an additional benefit the natural fibers will reduce the amount of polyurethane used, which reduces the environmental impact and might reduce the cost of developed material (Çelebi, et al. 2012).
Studies performed with micro-fabrics revealed that the sound absorption is superior to conventional fabrics of the same weight and thickness (Na, 2007). *Jayaraman* (2005) concluded in his study that the finer fibers presents higher absorption coefficients. *Nick* (2002) found that the fiber length does not show a significant influence on absorption.
The influence of the surface area or the relation between mass/area in sound absorption is reported in several works (Watanabe et al. 1999; Jayaraman, 2005).

Study made by Çelebi, et al. (2012), conclude that adding tea-leaf fibers to a soft polyurethane foam can enhance the polyurethane's sound absorption properties. The study also proved that it is extremely difficult to enhance rigid polyurethane foam's sound absorption properties with tea-leaf fibers if the structure remains as closed-cell.

2.4 Typha Latifolia

Typha latifolia, part of Typhaceae-family, is a common perennial marsh, or wetland plant in temperate, tropical, and subtropical climates throughout the Northern Hemisphere (Plants for A Future). Different parts of Typha latifolia, commonly known as Broadleaf Cattail, have been used as a replacement for vegetables, insulation material, medicinal purposes, and mechanical seal for wooden container's joints to prevent a leakage (Luontoportti).

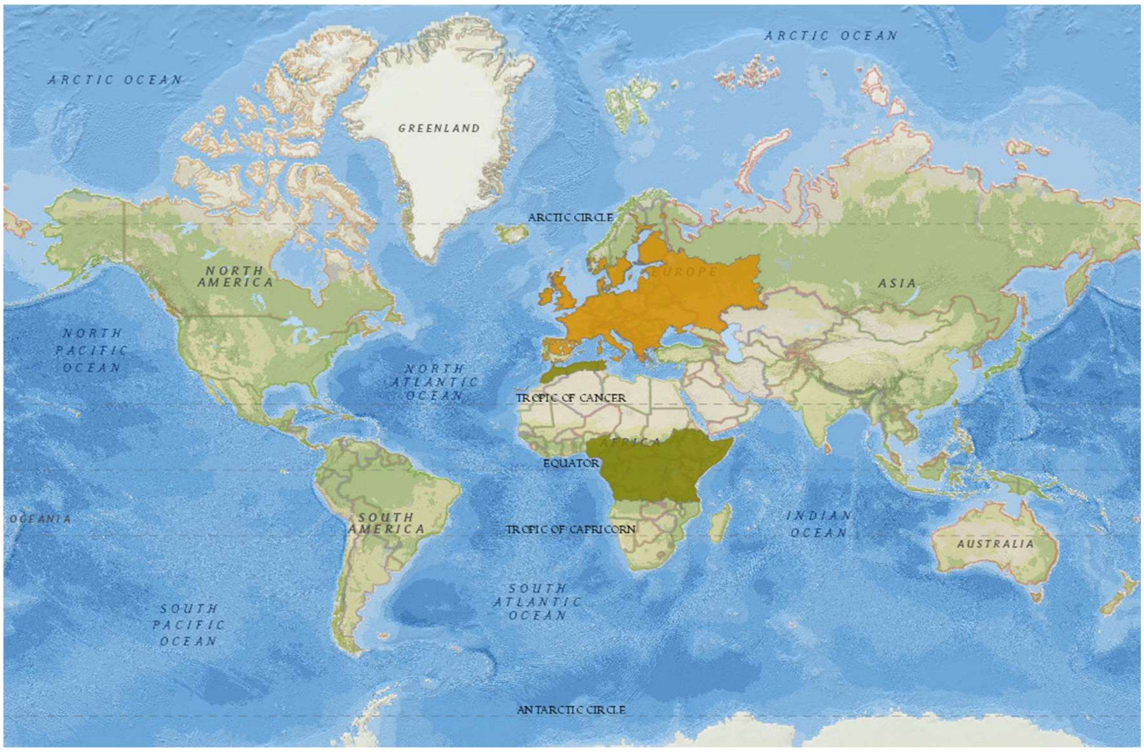


Figure 3:Typha latifolia's geographical range, where yellow indicates the area of extant, and green the possible area of extant (Lansdown, R.V., 2017).

All Typha spp. have aboveground leaves, an interconnected belowground rhizome system, and an easily recognizable inflorescence. Traits like yield potential, tolerance to weather and soil conditions, and resistant to insect and animal pests, may vary between species (Dubbe R. 1988). Typha latifolia is perennial and can grow up to 3 meters at fast rate. It can grow in acid, neutral or basic (alkaline) soils, which makes it an ideal plant for a manmade wetland (Plants for A Future, Paavilainen, 2005). Some studies of the use of Typha spp. in paludiculturing has been done (Wild U. et al. 2001).

Since Typha spp. grow in wetlands and marshes, it has a very high resistance to mold growth. The structure of Typha's leaf makes it tear and break resistant, yet flexible and able to keep its shape when dried. Typha spp. have remarkable load-bearing capacity and excellent insulation properties (Krus, 2014).

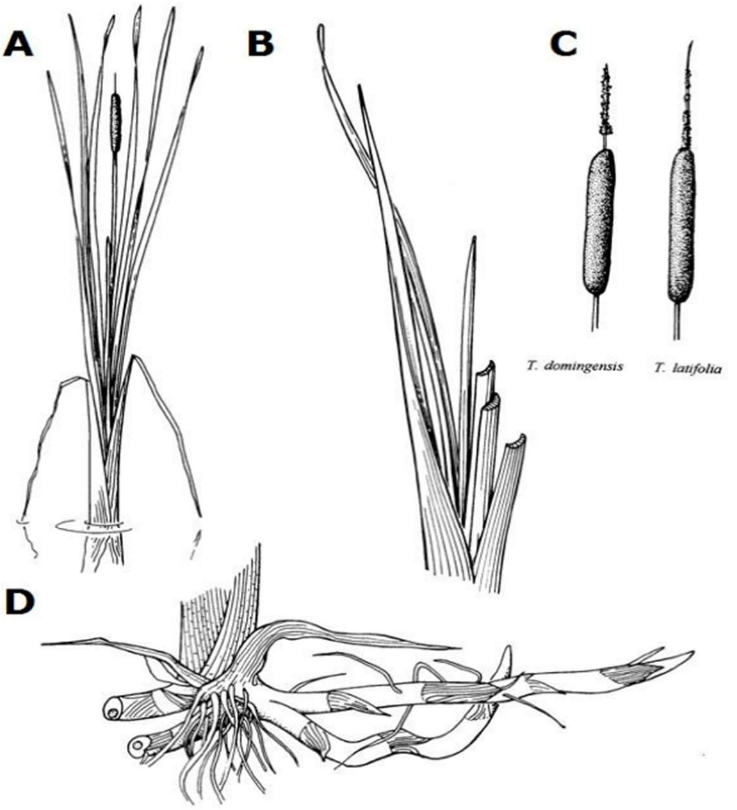


Figure 4:Different parts of Typha spp. Image A represent the whole plant, image B shows the leaf blades and image C is a representation of the seed heads of typha domingensis and typha latifolia (Colbers B, et al., page 3, 2017).

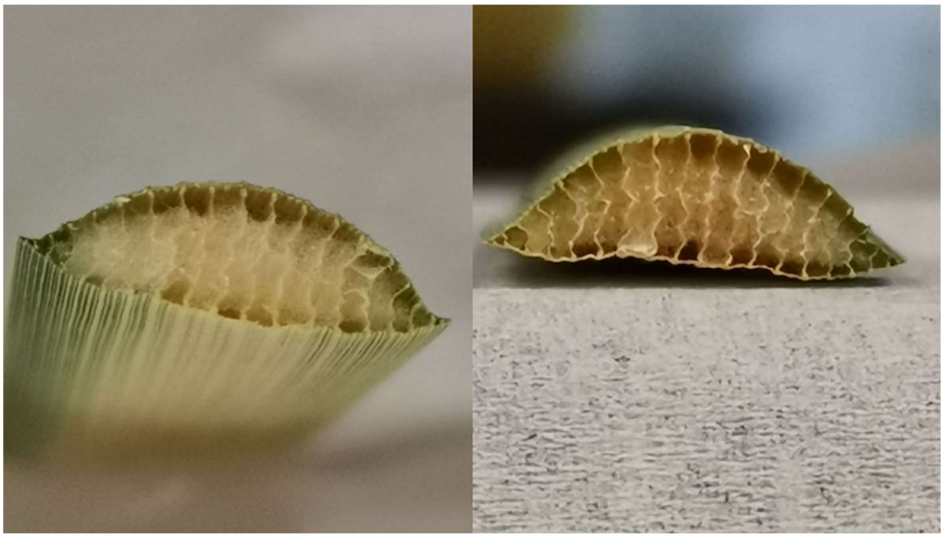
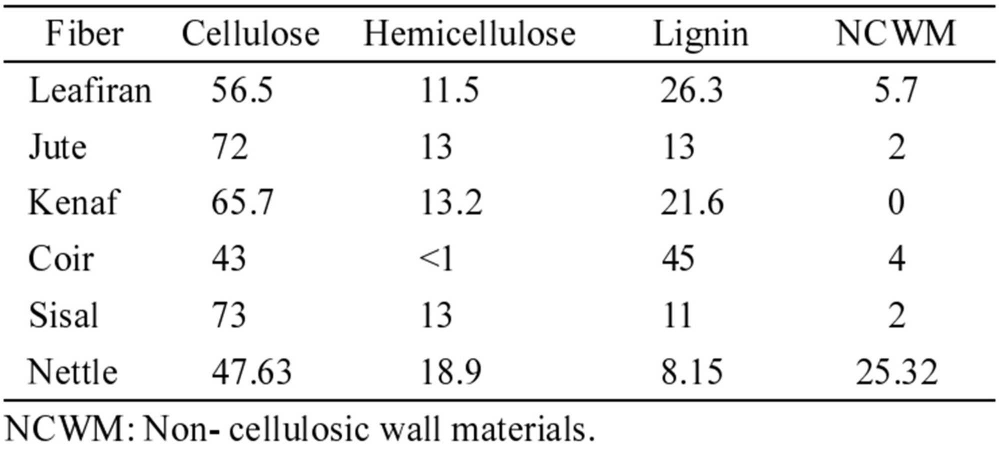


Figure 5: Typha latifolia's leaf structure.

Typha spp. leaves (Typha australis) contain gummy material (pectin) which holds the fibers in bundle. Typha fibers compose from materials like cellulose, lignin, pectin, wax, and fat materials (Mortazavi, S. M., 2010).

Table 1: The fiber composition of Typha australis (Leafiran) compared to other natural fibers (Mortazavi, S. M., 2010)



Fiber	Cellulose	Hemicellulose	Lignin	NCWM
Leafiran	56.5	11.5	26.3	5.7
Jute	72	13	13	2
Kenaf	65.7	13.2	21.6	0
Coir	43	<1	45	4
Sisal	73	13	11	2
Nettle	47.63	18.9	8.15	25.32

NCWM: Non- cellulosic wall materials.

2.4.1 Producing Typha fibers

Mortazavi, S. M et al. (2010) carried several experiments to find ideal conditions to extract fibers from Typha australis leaves. The optimized method of extracting fibers from the leaves was to treat them in a solution containing sodium hydroxide with ratio of 0.5-6 % w/v, ethylenediaminetetraacetic acid (EDTA) with ratio of 0.1 % w/v, and sodium tripolyphosphate (STPP) with ratio of 3 % w/v for 4 hours at 100° C with L: R=100:1. The retting process was done in Ahiba Polymat Dryer. Sodium hydroxide was the main component used for degumming and releasing the fibers from Typha leaves. STTP was used as a chelating agent to improve the extraction of fibers. After the extraction, the fibers were thoroughly washed with warm water to remove dissolved substances, and neutralized with acetic acid with the ratio of 0.2 % w/v. Finally, the fibers were rinsed with water and dried under ambient conditions.

The fiber fineness is dependant of process parameters such as alkali concentration and treatment temperature and duration. With the methods described above, the fiber fineness was high. This can be explained by a rather long 4-hour retting process, which resulted a thin fiber with low number of impurities and gummy materials.

Table 2:The fineness of fibers extracted at 100°C, with different retting times, where the CV is coefficient of variation (Mortazavi, S. M., 2010).

	Concentration (w/v)			2 h		4 h	
	NaOH	EDTA	STPP	Fineness (tex)	CV%	Fineness (tex)	CV%
A	0.5	0.1	-	7.3	21.3	3.92	13.5
B	1.5	0.1	-	5.47	16.9	3.46	11.2
C	3	0.1	-	5.07	19.8	4.61	9.7
D	4.5	-	3	6.27	12.5	4.38	16.8
E	6	-	3	4.6	23.1	5.84	6.8

2.4.2 Manufacturing polyurethane with an open-cell structure

Industrial production of polyurethane foam is highly optimized and reproducible operation (Sonnenschein, 2014). For this study, two-step (shot) PU-foam with rigid structure was used as it was easily accessible for consumers. The two-step PU-foam, provided by Kevra Oy, was stored in two containers. The first container contained the first component, MDI-catalyser, and the other container contained the second component, the resin. When the PU-foam is manufactured correctly, by following manufacturer's instructions, the timeline for the physical and chemical processes that occurs in polyurethane foaming will follow the steps seen at figure 6.

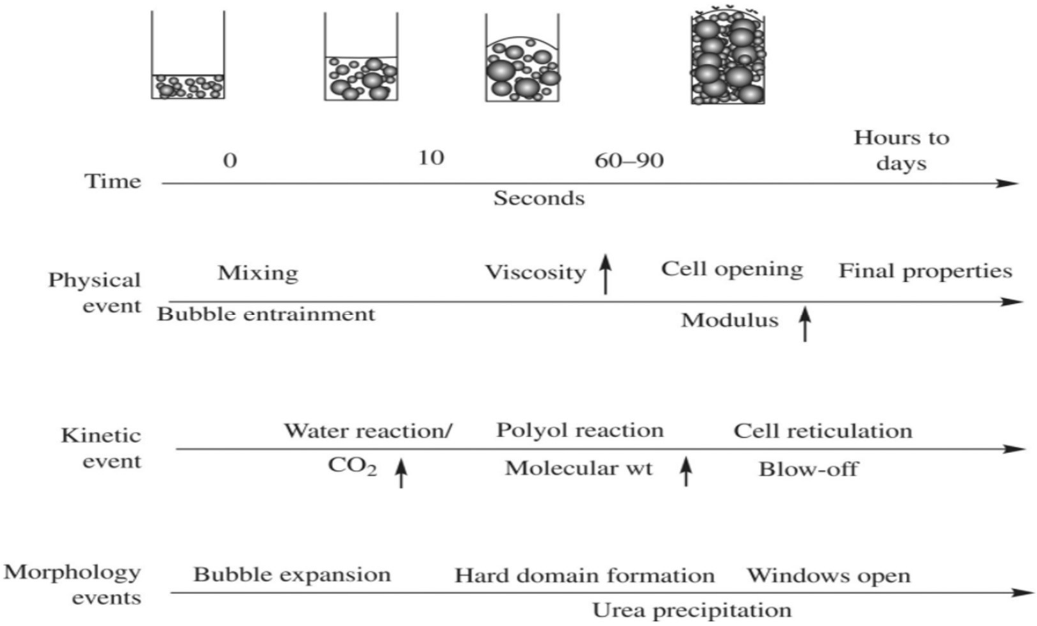


Figure 6: Timeline for the physical and chemical processes occurring in polyurethane foaming (Sonnenschein, chapter 6.2, 2014).

PU-foam, provided by Kevra, has a closed-cell structure which means its sound absorption properties are poor, as explained in Chapter 2.2. One way of creating an open-cell structure is to increase the intensity of blowing reaction with water. Adding water reduces the foam density as more carbon dioxide (CO_2) and heat are generated. Water also increases foam's hardness, decreases shrinkage, and increases aging degradation. The art of polyurethane foam formulation is finding the right balance between components to get the wanted structure and properties (Sonnenschein, 2014).

2.5 The bonding between natural fibers and rigid polyurethane

Incompatibility between polyurethane and natural fibers are one of the obstacles that is faced when a composite is being manufactured. Incompatibility is caused by hydrophobicity of most polymers used in making polyurethanes versus hydrophilicity of natural fibers (Venkateshwaran et al. 2013). This causes the lack of adhesion and wettability between fibers and polymer. Compatibilizers are used to enhance the adhesion and compatibility. Enhancing the interface between fibers and matrix can be done by treating the fibers with agents, e.g., an alkali treatment, that react with the polymer's hydroxyl group. Alkali treatment is a common method that is used to remove natural fiber's hemicellulose, lignin and waxes. Alkali treatment increases fiber's surface roughness and helps the interlocking of fiber and matrix (Mwaikambo, 2002).

Isocyanate active group (NCO) is eager to react with the hydroxide group of cellulose. Isocyanate has been used to improve the interfacial bonding and tensile strength of kenaf fiber reinforced thermoplastic polyurethane (El-Shekeil et al., 2014). As can be seen from the study by Mortazavi, S. M et al. (2010), figure 6, 65,7 percent of kenaf is cellulose and for Typha australis the cellulose percentage decreases to 56,5 percent. Çelebi, et al. (2012) observed that for both hard and soft structures, the tea-leaf fibers will form clusters on the walls of the cellular structures.

2.6 Characterization and measurements.

Preparation and characterization of materials form a crucial and vital aspect of materials research. Materials testing is done to classify material's performance and attributes. Quotative data is gathered from the performed tests to support the research and future development (Zhang et al. 2008). Some essential methods for analysing Typha latifolia's sound absorption properties and characterising its suitability with polyurethane will be discussed in this section.

2.6.1 Determining the normal incidence sound absorption coefficient with an impedance tube

Sound is created by the vibration of substance and is spread by sound wave produced through the sympathetic vibration of the medium. When sound spreads, part of it is gradually diffused and part is weakened due to the absorption of air molecules. In indoors sound diffuses less than in open air and is mainly absorbed by the surface of materials. When sound wave meets the surface of material, part of it is reflected, part of it passes through the material, and the rest of it is transferred to the material. Sound wave that enters the material, causes friction and viscosity resistance between the air molecules and the wall of pores, which leads to that a certain part of the sound energy is converted into heat energy and is absorbed. Sound absorption coefficient ($\propto$) is an index that enables the evaluation the sound absorbing performance of a material. It is the ratio of the sound energy absorbed by a material (E) to the overall sound energy previously spread and reaching the surface of the material (E_0) (Li, Y. 2011).

Mathematically this can be expressed as:

$$\propto = \frac{E}{E_0} \qquad \text{(Li, Y. 2011)}$$

, where $\propto$ is the sound absorption coefficient of material, E is the sound energy absorbed by material, and E_0 is the overall sound energy previously spread and reaching the surface of material. (Li, Y. 2011)

Any material has certain ability of sound absorption. What varies, is the sound absorption capacity. Generally, hard, smooth, and heavy materials in dense structure have weaker sound absorption quality but are strong in reflecting power (Li, Y. 2011).

For large samples, reverberant room method (ISO 354) is used to determine the sound absorption coefficient (Oliva et al. 2010). In product development, a commonly used method to determine the absorption coefficient is the standing wave tube method (impedance tube method, ISO 10534-2) (Liu, 2014, Oliva et al. 2010). The impedance tube method is not a valid method to test structures with large surface pattern. Also, the sound absorption coefficient acquired by impedance tube method, will be lower than the results obtained by the reverberant room method (Oliva et al. 2010).

The impedance tube method measures the vertical incident sound absorption coefficient of the materials or structure with sound absorbability (Liu, 2014). Sound absorption coefficient can be determined by the difference of p_{max} and p_{min}:

$$\alpha_{vertical} = \frac{4 \times p_{max} \div p_{min}}{(1 + p_{max} \div p_{min})^2} \qquad \text{(Liu, 2014)}$$

, where p_{max} is the first maximum sound pressure and p_{min} is the first minimum sound pressure to the sample surface.

The standing wave ratio is defined as the sound pressure ratio, as follows:

$$S = p_{max} \div p_{min} \qquad \text{(Liu, 2014)}$$

So, the vertical incidence sound coefficient can be written as follows:

$$\alpha_{vertical} = 4 \times S \div (1 + S)^2 \qquad \text{(Liu, 2014)}$$

The impedance tube facility is composed of an impedance tube, sound source system, detector, and output indicator. During the test, the disk-shaped sample is put to the front end of the tube, and the surface is the vertical axis of the tube. The maximum and minimum sound pressures of the standing wave are measured, and the sound absorption coefficient is obtained by using the vertical incidence sound coefficient equation from page 23 (Liu, 2014).

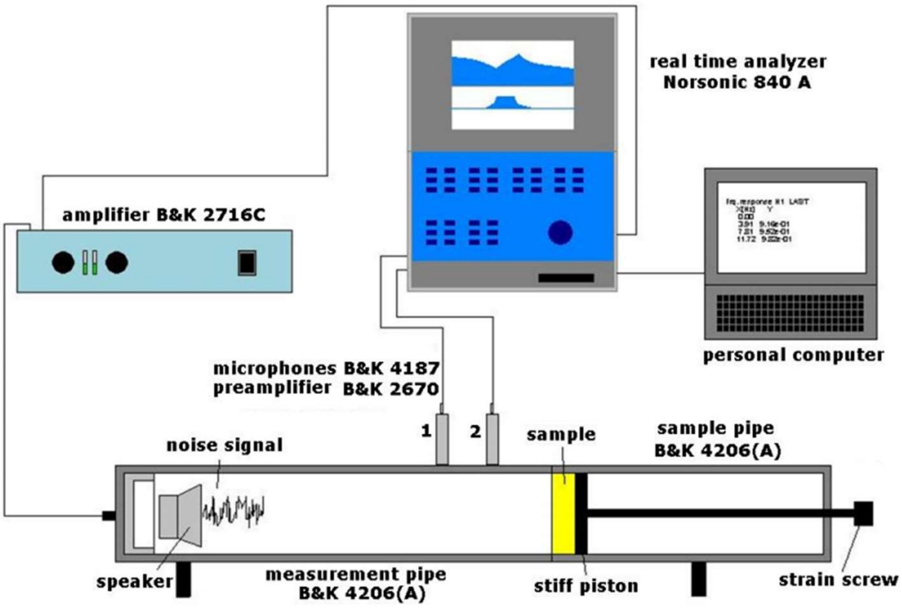


Figure 7:Layout of the impedance tube (Oliva et al., page 56, 2010).

2.6.2 Biodegradability and composting test of Typha latifolia

New ambitious recycling and circular economy targets has been set, which will shift the focus more on how the materials and resources are maintained in the economy as long as possible (European Parliament). Composting has been used for many years to stabilize organic residue. Composting a relatively easy and cost-effective method of treating organic waste (L.F. Diaz et al. 2011).

Multiple standards for different materials have been published to test the biodegradability in a controlled composting condition e.g., SFS-EN 13432 and ISO 14855-1:2012.

In this study home composting was used to put Typha fiber's degradation properties to a test, as the equipment and time of the year was favourable for this experiment. As stated by Klauβ M. (2004), the degradation of certain materials will reduce considerably compared to industrial level composting. At present (2021), there are no European Norm Standards (EN-standards) for home compostable fibers and bio-packaging (Vikman, M et al. 2015)

In home composting, the decomposition is primarily done by bacteria, fungi, ray fungi, and number of micro-organisms (Biolan). These organisms produce heat as their by-product of enzymatic catabolism of substrates and synthesis of cell material. The amount of heat generated by bacteria depends on the growth substrate, growth rate and growth stage. In general, heat production is inversely proportional to growth rate: the faster the growth, the lower the rate of heat production per unit weight (Rosenberg et al. 2016). As a result of the activity of the micro-organisms, water, carbon dioxide, nutrients and heat are released. The biowaste will transform to a fertile soil that is excellent for plants. The micro-organism activity will generate heat, and the temperature in the compost can rise as high as 70-80°Celsius. The outdoor temperature naturally has an effect to the temperature in compost. Decomposing continues slower in a colder weather and stops completely when the mass in the compost freezes. The compost mass freeze when the temperature in the compost is near -5°Celsius (Biolan).

There are three stages in composting: Warm phase, hot phase, and cooling phase. In the warm phase decomposers break down dead or decaying organisms, and their activity raises the composter temperature. After warm phase, the compost will be occupied by bacteria and ray fungi, that uses easily biodegradable materials like sugars and proteins for their nutrition. In this stage the temperature in composter will rise to its highest and the compost will become sour i.e., its pH value decreases. At its final stage, in cooling phase, the slowly biodegrading materials, like wood chips and eggshells, will be decomposed by new decomposers. The cooling phase can last several months. The pH of compost that has completed its degradation process is around 7 (Biolan).

2.6.3 Air flow resistance measurement

Air flow resistivity is the ratio of gas pressure differences at both sides of the porous material with unit thickness to the air flow rate. To achieve the best sound absorption, an optimal value of airflow resistivity must be found. (Liu, 2014). Sound absorption by porous materials is partly described by their ability to resist airflow. The parameters that quantify this property are: Air flow resistance, specific air flow and air flow resistivity (Hopkins, 2007).

Air flow resistance, R_f $(Pa.s/m^3)$ is defined as

$$R_f = \frac{\Delta_p}{q_v} \qquad \text{(Fangueiro, 2011)}$$

, where Δp is the air pressure difference across the test specimen with the respect of atmosphere, and q_v is the volumetric air flow rating passing through the test specimen (m^3/s).

The volumetric air flow rate is:

$$q_v = uS \qquad \text{(Fangueiro, 2011)}$$

, where u is the linear air velocity (m/s), and S is the cross-section area of porous material perpendicular to the direction of air flow (m^2).

Specific air flow resistance, R_S $(Pa.s/m^3)$ applies to specific resistance (Fangueiro, 2011).

2.6.4 Light optical microscopy

Light optical microscopy is one of the simplest and low-cost methods for analysing polymers and the surface of natural fibers (Suprakas, 2013; Ryszard M., 2012). The fine structures and morphology can be studied by using microscope's transmitted beam or reflecting beam. In transmitted beam, a fixed beam light will penetrate through a transparent or very thin specimen and provides a much-telling look on the specimen's structure. Reflecting beam will be used to study the specimen's surface (Girkin John, 2019). For this study, magnification from 1 to 50x will be sufficient to provide enough information on the structure of the specimen and on the possible bonding with polyurethane and Typha latifolia fibers.

3 EXPERIMENTAL WORK

3.1 Introduction

The aim of this experimental work was to produce Typha latifolia fibers and investigate if they could be a green alternative for enhancing polyurethane's sound absorbing property. As mentioned in section 2.2, polyurethane with a closed-cell structure will have low sound absorption property. This chapter will cover the manufacturing of both Typha latifolia fibers and an open-cell polyurethane foam. Biodegradability test, sound absorption test, and light optical microscopy-analysis of Typha latifolia will be also covered in this chapter. The experimental work will follow the following path:

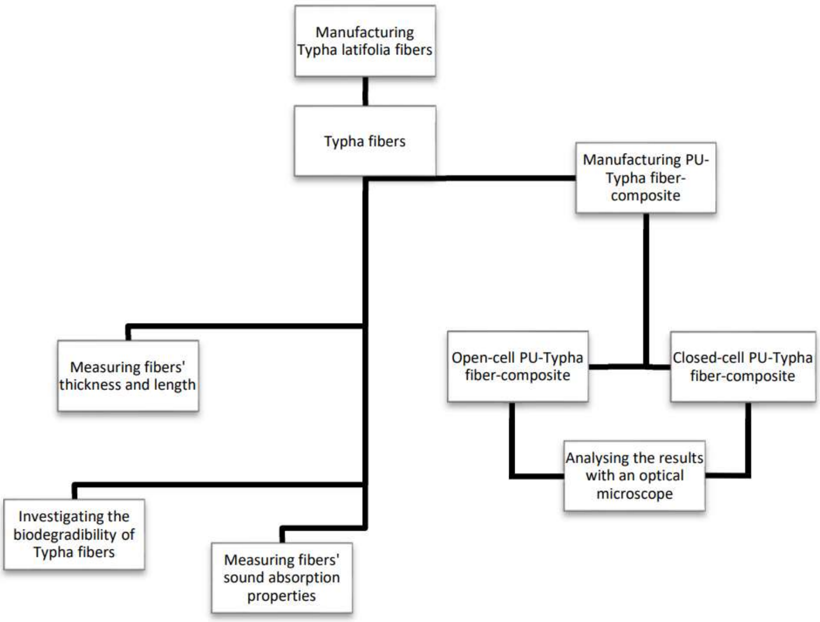


Figure 8: Experimental work stages and relations

3.2 Materials and equipment

3.2.1 Manufacturing Typha fibers

Typha latifolia leaves were picked in Lohja. Scissors were used to cut the leaves to fit a laboratory grade glass beaker. 20 grams of sodium hydroxide pellets (Merck, pure pellets) were added to 980 grams of distilled water (Merck, Direct-Q). Polytetrafluoroethylene-coated magnetic stirrer was used for mixing and laboratory hot plate for heating the retting solution. Finnsonic ultrasonic bath was used to separate the fibers. The fibers were neutralized by using acetic acid (2% w/v), thoroughly rinsed with deionized water, spread on a steel-mesh, and removed by using a scalpel. The final fiber separation was done with a carding tool.

3.2.2 Manufacturing PU-foam with an open-cell structure

Two component MDI-polyurethane was bought from Kevra Oy. A cardboard mould was used as it will be easy to break. Deionized water was used at manufacturing of water blown PU-foam. Dosing of components was done with a plastic syringe. Mixing was done with a plastic spoon. For composites, the fibers were cut with scissors.

3.2.3 Experiment equipment

The fiber thickness was measured with a Helios Presser dial indicator. Pincers were used to place the fiber under the dial indicator's contact point. The length of fibers was measured with digital caliper. The sound absorption property of Typha latifolia fibers' was measured with an impedance tube method at Turku University of Applied Science. Researcher's family-owned composter (Biolan Ltd. Model 220) was used in pilot scale composting test.

3.3 Preparation of Typha fibers

Typha leaves were collected from the shore of Lohjanjärvi, Uusimaa. The leaves were picked on middle of October, before the ambient air reached sub-zero temperatures.

Figure 9:Left: Typha latifolia plant in its natural habitat in Lohja. Right: Typha latifolia leaf.

After the picking, the leaves were washed with water, and dried in a room temperature for 24 hours.

In Arcada's laboratory, the leaves were cut to 5 cm pieces so that they will fit and fill a glass beaker. Retting was done in solution that contained 98% purified water and 2% sodium hydroxide (Merck, pellets pure) (w/v). The solution covered the leaves completely. The temperature of solution was 100°Celsius and the retting process took approximately 4 hours. Before the neutralization of fibers, Finnsonic ultrasonic bath was used for 30 minutes to prevent the fibers forming a lump. The temperature of water in the bath was set to 100°Celsius. The fibers were neutralized with 2% acetic acid.

Figure 10:Left: Typha leaves in sodium hydroxide. Right: The solution in an ultrasonic bath.

After the neutralization, the fibers were washed thoroughly with distilled water, spread on a stainless-steel mesh, and left to dry for 10-24 hours. The dried fibers were removed from the mesh and possible fiber lumps were separated manually by using a carding tool.

Figure 11:Left: Wet Typha fibers spread on steel-meshes. Right: Dried damp-removed Typha fibers.

10 hour drying time caused that the fibers were damp when they were removed. Damp fibers were easier to remove from the steel mesh by hand. The length of the fibers can be kept greater by damp removing the fibers. Removing dried Typha fibers from the steel mesh requires force which damages the fibers. Damp fibers turned out to be too soft to be separated by a carding tool. The damp-removed fibers were left to dry after the removal.

3.4 Measuring the length and thickness of Typha fibers

The fiber length was measured with a digital calliper. As mention in the section 3.3, the leaves were cut to 5 cm pieces, so that they could fit in the beaker. This measurement was executed to collect data on how the retting affected to the fiber length. It is worth noting, that the retting process was not optimized to produce as long fibers as possible, since the main focus was in fiber’s sound absorption properties. The measurement was also done to compare how much the fiber length differs between damped removed fibers and dry removed fibers. From both batches, five of the longest and five of the shortest fibers were selected for the measurement. Fibers with length less than 1 mm were neglected as the measuring with calliper would become too unreliable.

The fiber thickness was measured using a dial indicator. Before measuring, the dial indicator was calibrated and tested to confirm that the results would be accurate. The measurement was performed to fibers that had a length greater than 20 mm. Longer fibers were easier to handle and place under the dial indicator's contact point.

3.5 Determining the normal incidence sound absorption coefficient of Typha latifolia fibers

Since the research budget allowed only one impedance tube measurement to be performed, the measurement was done nothing but to Typha latifolia fibers. The reason behind this was to ensure that the data collected can be compared to other natural fibers and wools and could be used as a reference point in future works. A homogenous 50/50 mix was done from the dry removed fibers and damp removed fibers. This procedure guaranteed that a compact insulation-wool-like-structure was achieved. It is worth noting that to make a sample seen in figure 12, approximately two batches of fibers were needed to form a wanted shape and size. In this case, one batch of fibers meant that approximately 600 ml of Typha leaves were used in retting process.

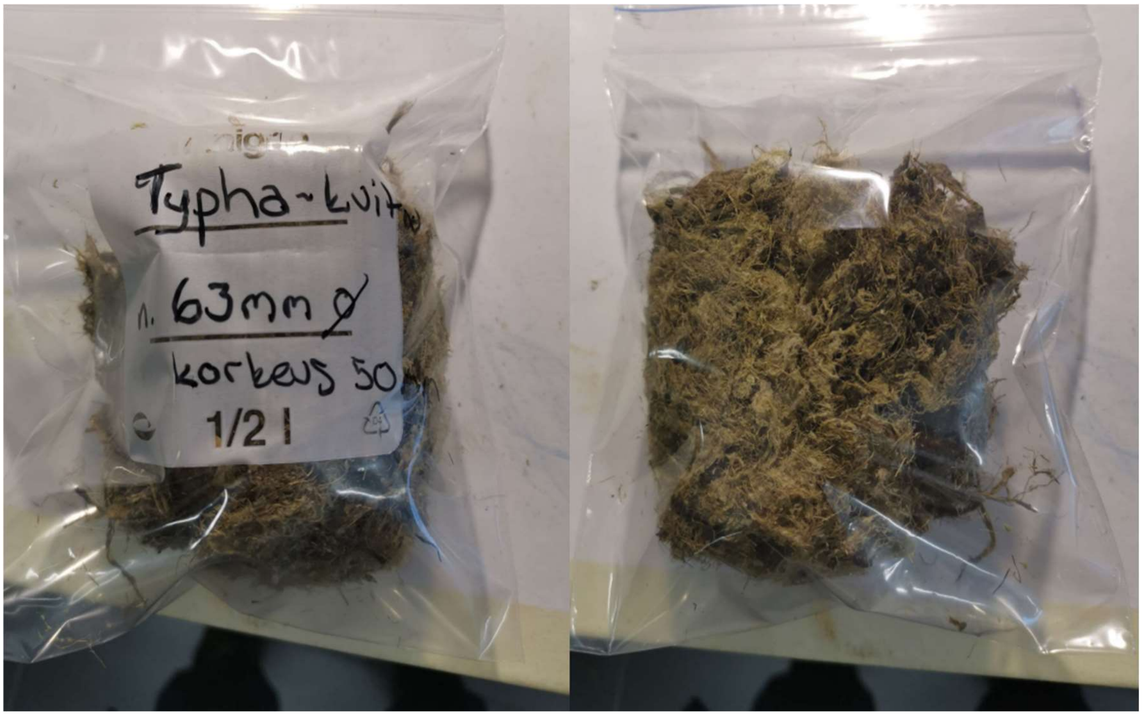


Figure 12:The Typha latifolia sample that was sent to Turku for an impedance tube measurement.

A sample, with a diameter of 63mm and height of 60mm, was shaped, packaged properly, and sent to the acoustic laboratory of Turku Applied Sciences for an impedance tube measurement.

In Turku, the specimen was mounted directly against the rigid back plate (figure 13). No adhesives were used to attach the specimen to the back plate. The diameter of the impedance tube (Brüel & Kjaer 4206A) was 63.5 mm, and the spacing of the microphones was 31.8 mm. This results a measurement frequency in range of 100 - 3200 Hz. Sound pressure in the tube was measured with a real time analyser (Norsonic RTA 840) and two pressure microphones (1/4" Brüel & Kjaer 4187 with preamplifier Brüel & Kjaer 2670) mounted to the sidewall of the tube. White noise from the analyser was used as the measurement signal. The signal was amplified with a power amplifier (Brüel & Kjaer 2716C). The transfer function was measured with the analyser using a fast Fourier transform (FFT). The frequency resolution was 3.9 Hz. The calibration of the microphones and the cables were checked before and after the measurements with a sound level calibrator (Brüel & Kjaer 4231). The results were averaged into one-third octave band and octave band results. The acoustical measurement equipment fulfils the following IEC standards and grades of accuracy:

- IEC 60651 Sound level meters, type 1
- IEC 60804 Integrating sound level meters, type 1
- IEC 61260 Octave band and fractional octave band filters, class 1
- IEC 60942 Sound level calibrators, class 1

ISO 10534-2: 1998(E) Acoustics Determination of sound absorption coefficient and impedance in impedance tubes (International Organization for Standardization, 1998, Genève, Switzerland), was used as a test standard.

Figure 13: Left: Typha latifolia sample. Right: Typha latifolia sample mounted on the rigid backplate. Below: Layout of the measurement equipment in the acoustic laboratory of Turku Applied Sciences.

3.6 Biodegradability of Typha latifolia fibers

The biodegradability of Typha latifolia fibers were studied on a pilot scale level, by using a 200-litre year-round outdoor composter bin (Model 220, manufactured by Biolan Ltd, Finland). Four samples, two samples from damp-removed fibers-batch, and two samples from dry-removed fibers-batch, were taken. Reason behind this was to study if damp-removed fibers and dry-removed fibers had a different decomposing time. The weight of samples was determined by weighting them with an analytical balance. The weight of the samples can be found from table 5, at page 43. The biodegradability test was performed in Lohja. The test samples (figure 14) were put into plastic seed sowing pots, which were marked with different coloured tapes to make the sample identification easier in latter stages (figure 20). The samples were put into a middle of a soil that was a mix between easily decomposing material and slowly decomposing material (figure 14). 50/50 mix was done to speed up the normal decomposing circulation between the first bacterial decomposer-step and the second, fungi- and actinomycete-step. The temperature in compost was 32° Celsius when the samples were placed inside it. Composter was used rather normally after the samples were placed, apart from not using a hand cultivator on testing area as to avoid on breaking the samples. Compost litter (Biolan Ltd, Komposti- ja Huussikuivike) were added alongside biowaste to keep the composter's soil fluffier and at correct moisture ratio.

Figure 14: (Left): The composter bin, (in middle): The samples put into sowing pots, (right): Picture of the 50/50 mixed soil that were used to speed up the decaying process.

Merck pH-indicators were used to measure the pH-value of the compost that had reached the end of hot phase. The result followed the theory that was explained on section 2.6.2 as the pH of the compost was close to 7. As mentioned at page 25, in the hot phase the pH-value will go lower, and the soil will be acidic.

Figure 15:The pH of finished compost from Biolan 202 composter.

In the first two weeks, the temperature in composter varied between 32-23° Celsius and the outdoor temperature varied between 4 and -2° Celsius. On the third week, the outdoor temperature dropped and varied between -7 and -20° Celsius. The temperature in composter dropped to 5° Celsius. On the final – fourth – week, the outdoor temperature rose back to same temperatures that were reported on week one and two. However, the temperature in composter stayed at 5° Celsius since the decomposers need time to restart their activity.

The samples were taken out from the composter exactly after one month (31 days). The fibers were carefully taken out from the sowing pots and gently brushed with a small paint brush to remove compost soil. After that, the samples were put into a mini grip bag and brought to Arcada Chemistry lab where they were studied, and their weight was measured.

3.7 Manufacturing an open-cell polyurethane-Typha fiber-composite

As explained in Chapter 2.4.2, an open-cell structure can be created by adding water as a blowing agent to the mix of PU-foam components. For this experiment, a rigid polyurethane foam, bought from Kevra Oy, was used and distilled water acted as a blowing agent. Experiments were made to find the optimal ratio of compounds, and it was noticed, that 5/16 of resin, 5/16 of water and 6/16 of catalyser gave the desired result.

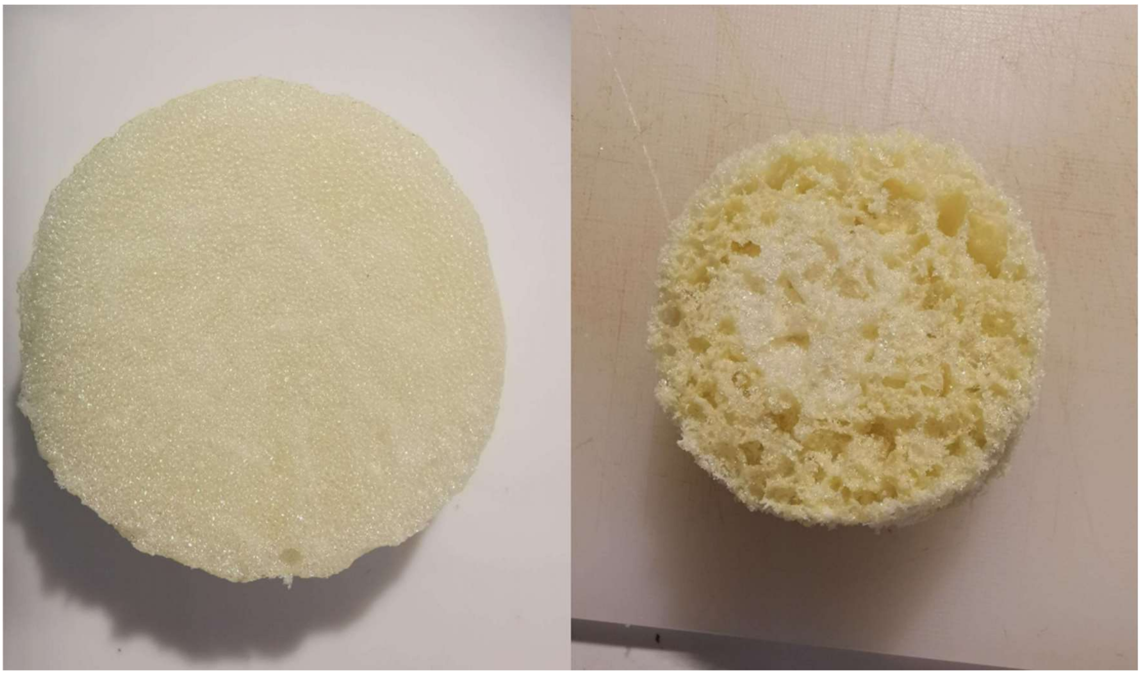


Figure 16:A closed-cell polyurethane foam with 50/50 ratio of resin and catalyser (left). An open-cell water blown rigid polyurethane foam with ratio of 5/16 of resin, 5/16 of water and 6/16 of catalyser (right).

After the optimal ratio was found for producing an open-cell structure, the next step was to manufacture Typha latifolia-PU-composite. As stated in section 2.5, the quality of bond between fibers and polyurethane will be decided with surface treatment and selecting a polyurethane that has an isocyanate active group (NCO) which is eager to react with the hydroxide group of cellulose. Polyurethane used in this experiment, provided by Kevra, was MDI-isocyanate based, and as covered in section 3.3, Typha latifolia fibers were manufactured by retting them in an alkali solution. A 50/50 mix was made from damp-removed fibers-batch and dry-removed fibers-batch as there should be no detectable difference in bonding quality that would be caused by fibers' thicknesses.

Fibers were cut to approximately 1 mm length pieces to make the mixing process easier. Once the fibers were cut, a cardboard mould was filled a quarter full of a mix of 5/16 of resin, 5/16 of water and 6/16 of catalyser. Half of the volume of the mould of fibers were mixed to polyurethane mixture before it reached the bubble expansion stage. The mixing was done with a simple plastic spoon. After the mixing, a reaction, that can be seen in figure 6, occurred and resulted a Typha latifolia-polyurethane foam with an open cell structure. The Typha latifolia-polyurethane foam was left to cure for 24 hours before removing it from the mould. Another sample was made from flax wool to provide a baseline. The fibers were separated from the flax wool and cut to 1 mm pieces. After that, exactly same procedure was followed as with manufacturing Typha latifolia-polyurethane foam.

After manufacturing the open-cell-composites, one Typha latifolia-polyurethane foam with closed-cell structure and one flax-polyurethane foam with closed-cell structure were manufactured. A quarter of the mould was filled with 50/50 mix of resin and catalyser. To this mixture, half of the volume of the mould of 1 mm Typha fibers were added, and carefully mixed with the resin-catalyser mixture before it reached bubble expansion stage. A plastic spoon was used for mixing. The Typha latifolia-polyurethane foam with closed-cell structure was left to cure for 24 hours before removing it from the mould. Exactly same procedure was executed with flax fibers.

3.8 Analysing manufactured composites with an optical microscope

The manufactured PU-Typha latifolia- and PU-linen-composites, and open-cell- and closed-cell-PU foam, were cut with a sharp scalpel to a disk shape with a thickness of 25 mm. Zeiss optical microscope was used to analyse the cut composites. 5x objective was observed to be the best for analysing the composites. Because of the porous surface of the samples, getting clear images required moving the sample up and down to find the correct depth of focus. The pictures were taken with Canon system camera and transferred to computer for more thorough analysis.

4 RESULTS

4.1 Typha latifolia fibers

The Typha latifolia fibers that were manufacture by retting are shown in figures 17 and 18.

Figure 17:Dry-removed Typha latifolia fibers.

Figure 18:Damp-removed Typha latifolia fibers

The retting process that was used, was identical for both fibers. The scent that Typha leaves released on retting process was rather pleasant and resembled the scent of hibiscus flower hand soap. Identifying the dry removed fibers (figure 17), that were separated with carding tool, from the damp-removed fibers (figure 18), that were separated by hand, was easy as the dry-removed fibers had greater diverseness in fiber thickness and length. As can be seen from the figures 17 and 18, the amount of short fibers is much greater among dry-removed fibers than damp-removed fibers.

The length of the fibers were measured with a digital caliber. The results can be seen from the table 3.

Table 3: Typha latifolia fiber length

Typha latifolia fiber length			
Damp-removed fibers [mm]		Dry-removed fibers [mm]	
Longest	Shortest	Longest	Shortest
40.38 mm	15.31 mm	42.68 mm	6.25 mm
36.17 mm	8.72 mm	29.62 mm	3.80 mm
33.72 mm	5.07 mm	29.34 mm	2.53 mm
32.22 mm	3.69 mm	25.82 mm	1.24 mm
32.03 mm	1.68 mm	11.57 mm	1.23 mm
Average [mm]		Average [mm]	
34.904 mm	6.894 mm	27.806 mm	3.010 mm

The thicknesses of the fibers were measured with a dial indicator. The results can be seen from the table 4.

Table 4: Typha latifolia fiber thicknesses

Typha latifolia fiber thicknesses			
Damp-removed fibers [mm]		Dry-removed fibers [mm]	
Fiber 1	0.245 mm	Fiber 6	0.153 mm
Fiber 2	0.230 mm	Fiber 7	0.100 mm
Fiber 3	0.187 mm	Fiber 8	0.085 mm
Fiber 4	0.079 mm	Fiber 9	0.071 mm
Fiber 5	0.056 mm	Fiber 10	0.062 mm
Average	0.1594 mm	Average	0.0942

4.2 Biodegradability of Typha latifolia fibers

The biodegradability test was done to damp-removed fibers and to dry-removed fibers. After four weeks of composting, the plastic sowing pots were taken out from the compost. The remaining fibers were collected and weighted. All most all of the fibers were decomposed completely, and only a thin delicate layer was observed when fibers were collected. The fibers that were not decomposed, were very fragile and broke immediately when handled.

Figure 19: Typha fibers before biodegradability test

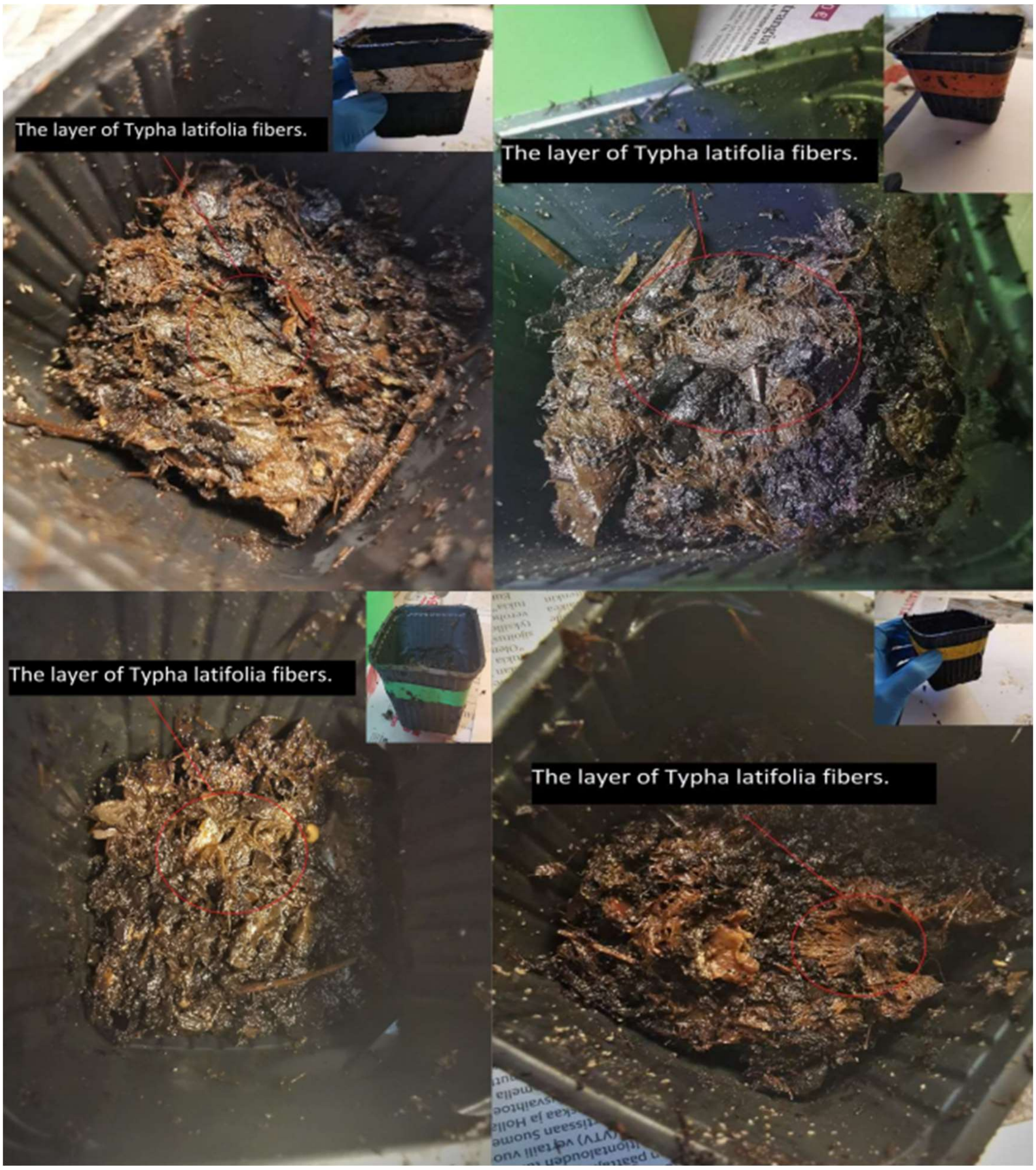


Figure 20: Typha latifolia fibers after 4 weeks of decomposing in a Biolan composter.

Only two fiber specimens, from sample 2 (yellow) and sample 3 (green), were obtained for the weighting. Fibers of sample 1 (white) and sample 4 (orange) were too decomposed and broke down immediately to small pieces when handled.

Table 5:The weight of Typha fibers before biodegradability test and after it.

The weight of Typha fibers before and after biodegradability test			
	The weight of damp-removed fibers [g]	The weight of damp-removed fibers [g]	Weight loss [%]
Sample 1 (white)	1.6371	-	100
Sample 2 (yellow)	1.5133	0.0111	99.983
	The weight of dry-removed fibers [g]	The weight of dry-removed fibers [g]	
Sample 3 (green)	0.5204	0.0508	99.974
Sample 4 (orange)	0.7494	-	100

4.3 The impedance tube measurement

The sound absorption measurement was done to a sample that had a diameter of 63 mm and height of approximately 60 mm (uneven surface). ISO 10534-2-standard was used in measurement.

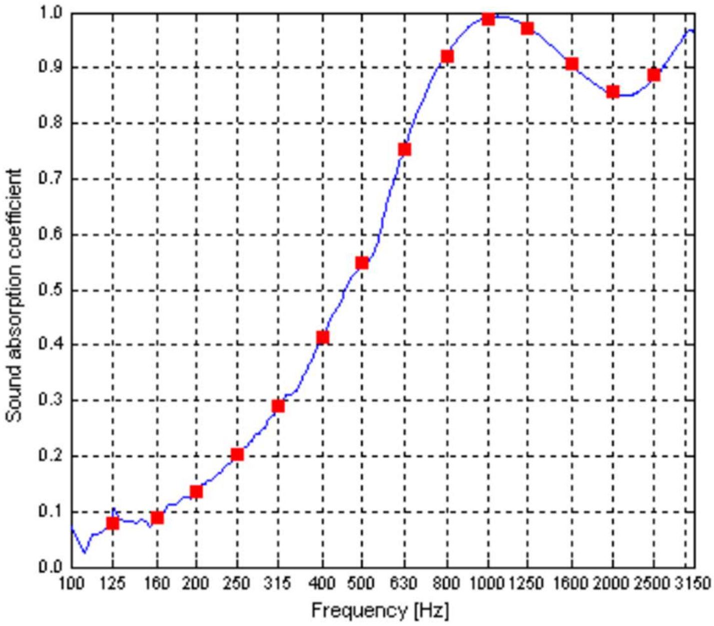


Figure 21: Result of sound absorption measurement (ISO 10534-2) done to Typha latifolia fibers with height of ~60 mm. Measurement was done in acoustic laboratory of Turku Applied Sciences.

Table 6: Table of the sound absorption coefficient of Typha latifolia fibers

Frequency f [Hz]	Sound absorption coefficient $\propto$
125	0.08
160	0.09
200	0.14
250	0.20
315	0.29
400	0.41
500	0.55
630	0.75
800	0.92
1000	0.99
1250	0.97
1600	0.91
2000	0.86
2500	0.89

4.4 The bonding between Typha latifolia fibers and rigid polyurethane

As stated in section 3.7, six samples were manufactured in total. Open-cell polyurethane foam and closed-cell polyurethane foam can be seen in figure 16, at section 3.7. Composites with Typha and linen fibers can be seen in figures 22 and 23. As can be seen from figure 22, the mixing process between Typha fibers and non-modified polyurethane components was not as successful as with water blown polyurethane. It was observed that both fibers were easier to mix to water blown polyurethane.

Figure 22: Typha latifolia-PU foam with open-cell structure (left). Typha latifolia-PU foam with closed-cell structure (right).

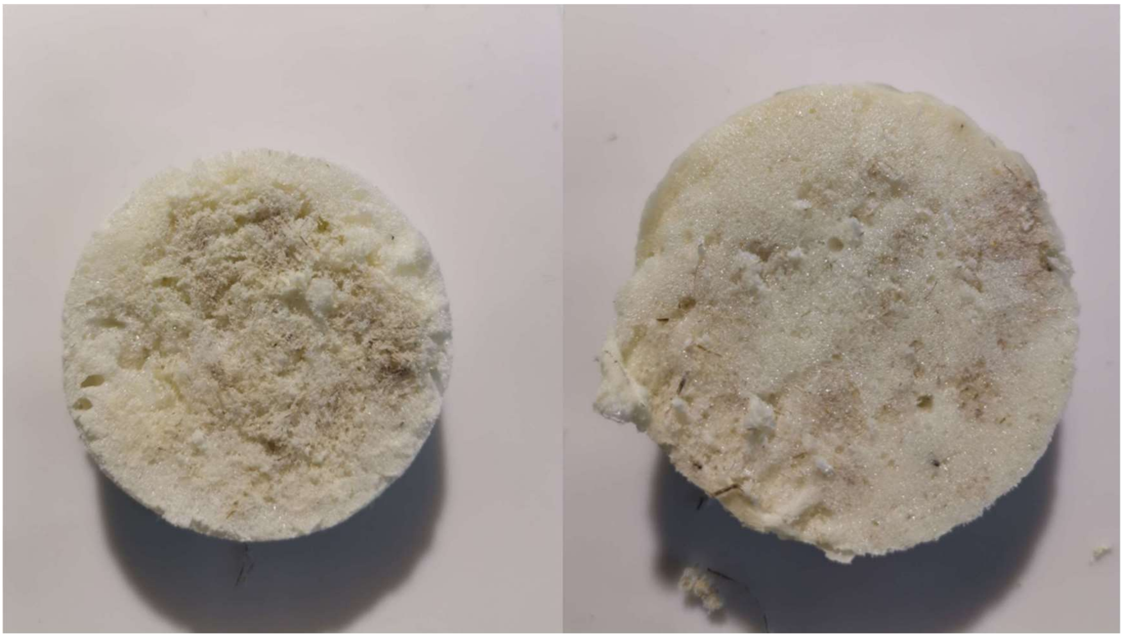


Figure 23:Linen-PU foam with open-cell structure (left). Linen-PU foam with closed-cell structure (right).

Microscope pictures were taken from the surface of the samples.

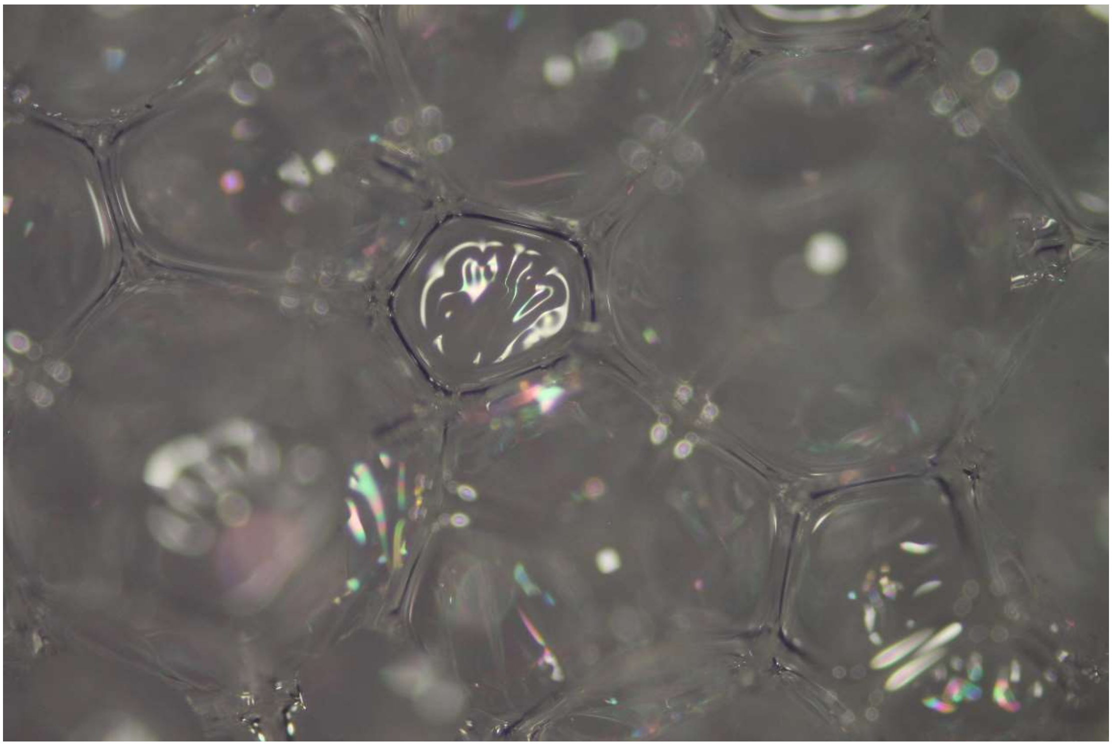


Figure 24:The structure of closed-cell polyurethane foam, seen in Figure 16 at section 3.7, under a microscope.

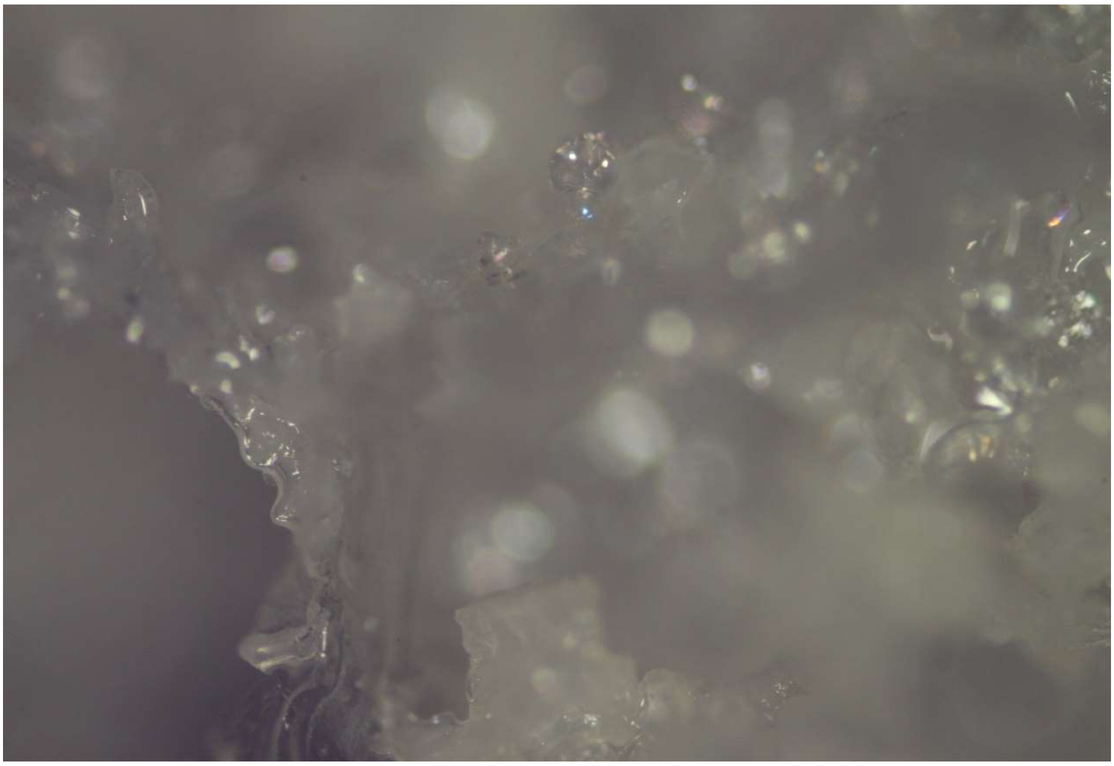


Figure 25:The structure of open-cell polyurethane foam, seen in Figure 16 at section 3.7, under a microscope.

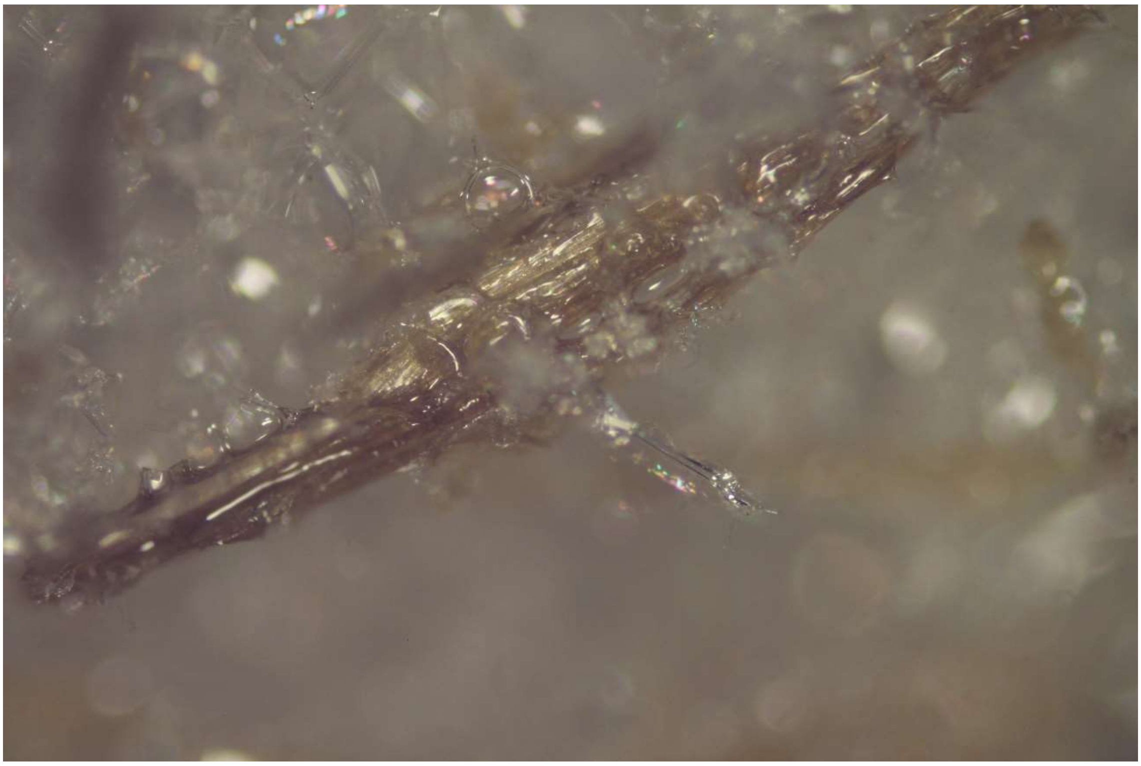


Figure 26:Typha latifolia-PU foam with closed-cell structure, seen in figure 22 at section 4.3, under a microscope.

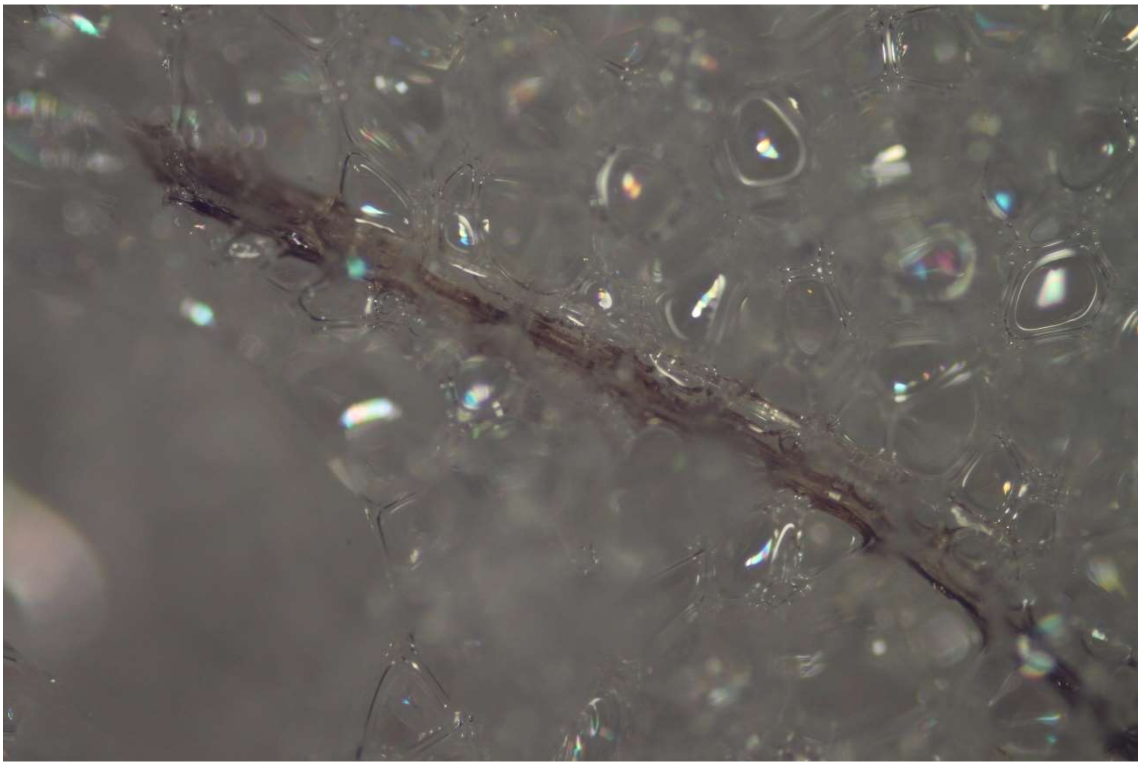


Figure 27:Linen-PU foam with closed-cell structure, seen in Figure 23 at section 4.3, under a microscope.

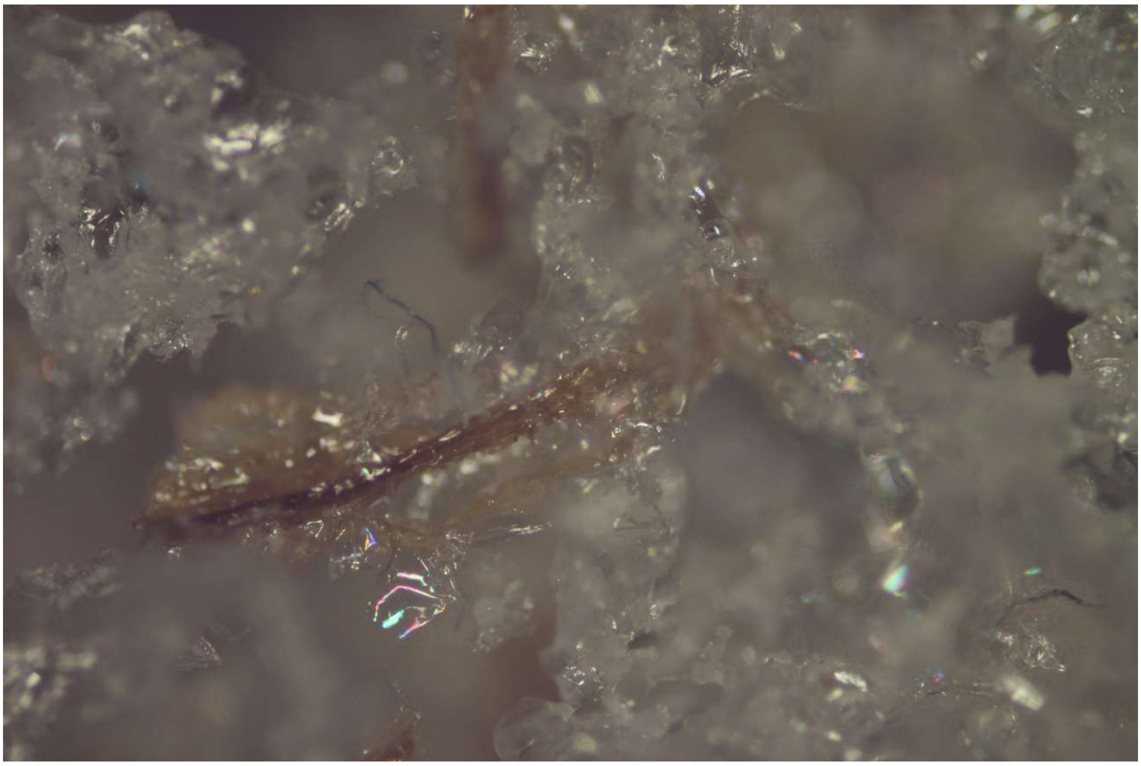


Figure 28:Typha latifolia-PU foam with open-cell structure, seen in Figure 22 at section 4.3, under a microscope.

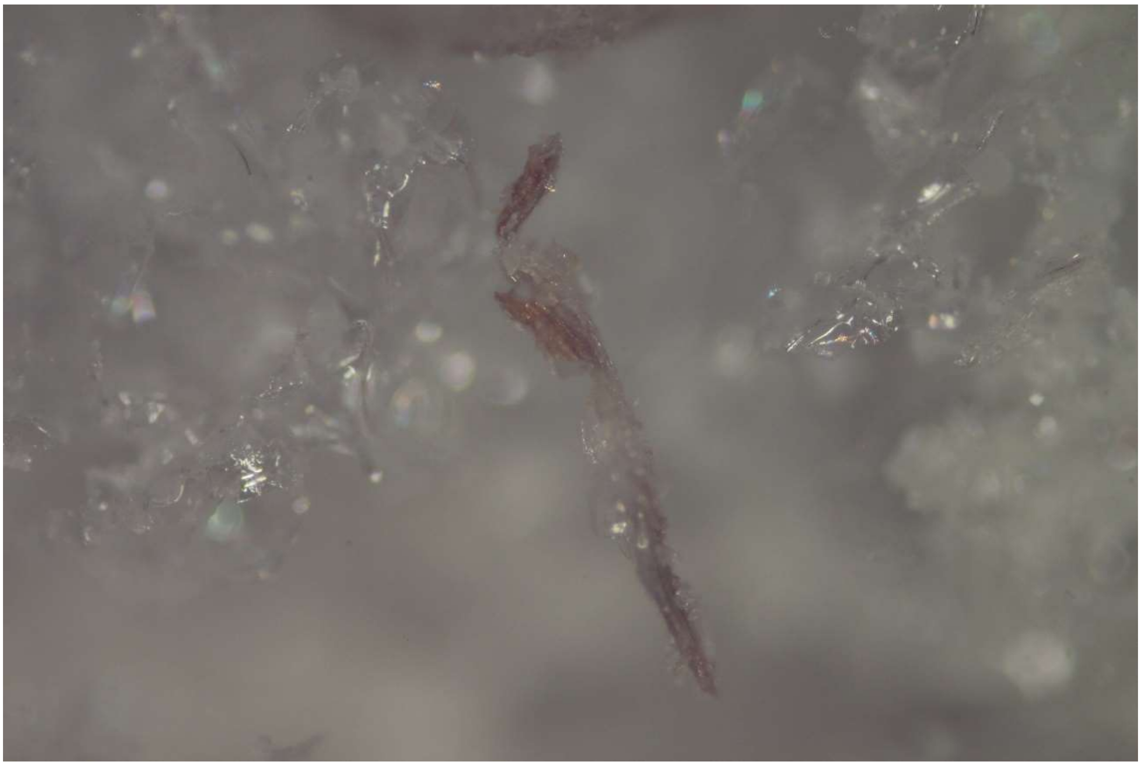


Figure 29:Linen-PU foam with open-cell structure, seen in Figure 23 at section 4.3, under a microscope.

5 ANALYSING THE RESULTS

5.1 Analysing manufactured Typha latifolia fibers

A manufacturing process was successfully developed to produce consistent fiber replication. It was noticed that magnetic mixing rod should be kept at low speed to ensure longer fiber length. Also, ultrasonic bath was found to be helpful, but not necessary, on separating the fibers. The biggest factor that effects to the fiber length and thickness is the fiber separation process. Greater fiber length and thickness can be achieved by carefully separating the fibers by hand while they are still damp. If the fibers are left to dry on a steel mesh, they will create a card-board-like layer which can be separated back to fibers by using a carding tool. This process will put the fibers under a stress, which leads to shorter fibers and smaller fiber thickness. Damp-removed fibers were thicker than jute fibers, but not as coarse. Dry-removed fibers were softer than damp-removed, which can be explained by the lower fiber thickness, and thus resembled more of linen fibers. Dry Typha fibers release a scent that is close to dry tea leaves.

5.2 Analysing the results of the normal incidence sound absorption measurement

The impedance tube measurement that was done for Typha latifolia fibers, section 4.3, gave a promising result on fiber's sound absorbing potential. The result itself was not a surprise as covered in section 2.1, wools and wool-like materials are good sound absorbers. When Typha latifolia results are compared to other wool-like materials, the similarities at the curve can be seen quite effortlessly. The author wishes to emphasize that Typha latifolia's sample's thickness is approximately 60 mm when wool's and glass wool's thicknesses, in page 50, are 50 mm. The Typha latifolia's extra 10 mm in thickness will likely cause that the material will perform slightly better at lower frequencies, as can be seen from figure 32.

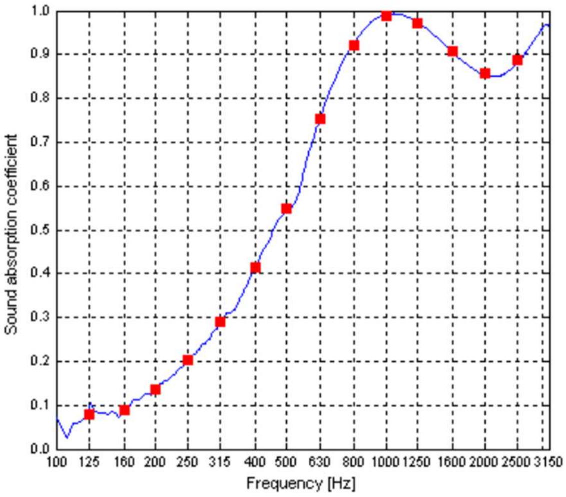


Figure 30: Result of sound absorption measurement (ISO 10534-2) done to Typha latifolia fibers with thickness of ~60 mm with no air gap between the sample and the rigid back plate.

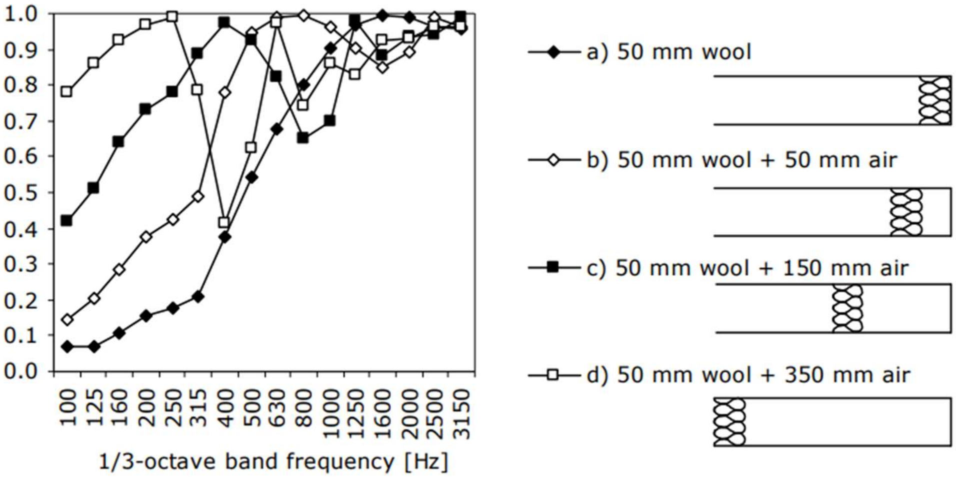


Figure 31: The absorption coefficient of wool (ISO 10534-2) (Oliva et al., page 1, 2010).

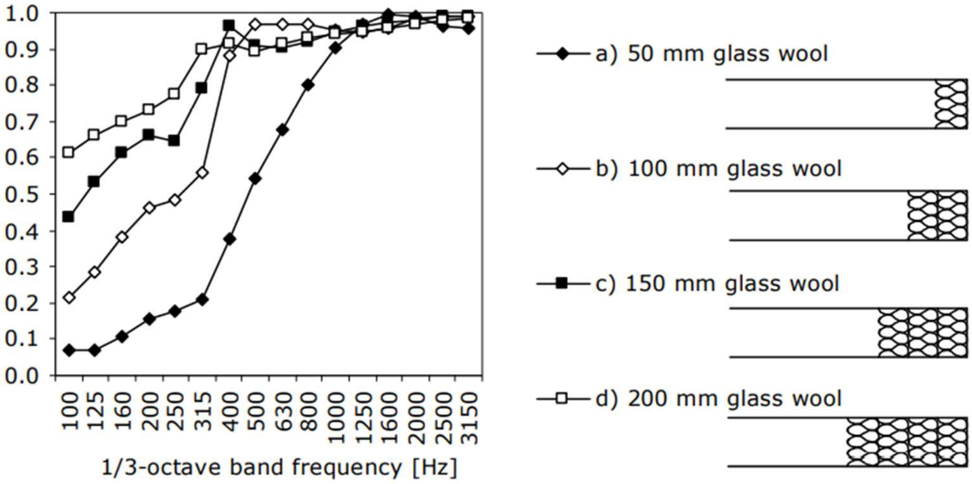


Figure 32: The absorption coefficient of KL-35 Saint Gobain Ecophon Glass wool, with density of 18 kg/m^3 (ISO 10534-2) (Oliva et al., page 15, 2010).

5.3 Analysing the results of biodegradability test of Typha latifolia

The results obtained from biodegradability test can be considered excellent since as stated by Klauβ M. (2004) the home composting is not as fast nor effective as industrial level composting. Also, the outdoor temperatures were really challenging for home composting as the temperature in composter fell to 5 °Celsius for the last two weeks. The decreased temperature in composter was eventually a positive event as the biodegrading process continued slower for the last two weeks of the experiment, and the samples did not decay completely. Interestingly, no difference was noticed between the damp-removed and dry-removed fibers. Both fibers seemed to decay at the same rate and broke down identically when handled after four weeks of biodegrading. As measured in section 4.1 (table 4), the difference between damp-removed and dry-removed fibers' thicknesses is not as big as it would affect to decaying rate.

Next step would be to perform a biodegradability test which follows standards for a controlled composting condition e.g., SFS-EN 13432 and ISO 14855-1:2012. Obviously, some changes must be made to the fibers so that they will fit to these standards.

5.4 Analysing the bonding between rigid polyurethane and Typha latifolia

In figure 24, the polyurethanes closed-cell structure can be observed, and as explained in sections 2.2 and 2.6, this structure lowers materials sound absorbing properties as material resistance to airflow is significant. The modified polyurethane with an open-cell structure, figure 25, is more porous and has a smaller airflow resistance and thus has the properties that enable good sound absorbance. As stated in section 2.5, creating a fine bonding between polyurethanes and natural fibers can become an obstacle. The bonding between non-modified PU and the fibers was successful as both Typha and linen fibers were fully coated by PU. The image of water blown PU-Typha foam, figure 28, clearly shows that polyurethane has coated Typha fiber. In figure 29, it can be observed that water blown PU has not coated linen fiber evenly, but only partly. Also, the PU has formed several clumps on the surface of linen fiber. This would indicate that it is harder to create a bond between water blown PU and natural fibers, and how the alkaline treatment for Typha fibers likely improved the bonding. One factor which likely helped the bonding between the polyurethane and selected natural fibers was that the polyurethane, provided by Kevra, was isocyanate based, and as stated in section 2.5, isocyanate active group (NCO) is eager to react with the hydroxide group of cellulose. According to Zommere G. et al. (2013) cellulose constituents of linen fibers varies between 64 percent to 70 percent. Typha latifolia's cellulose constituents will most probably be close to Typha australis' ones (table 1), which would indicate that even though linen fibers have more cellulose, the alkali treatment of Typha latifolia fibers can be a difference maker when creating a successful bonding between the fiber and water blown polyurethane.

6 DISCUSSION

This study offers a descriptive and unique reporting on manufacturing Typha latifolia fibers. Even though one of the main objectives were just to manufacture Typha fibers, the author wanted to ensure that especially the experimental work was clear, detailed, and well documented for future researchers.

As already stated in section 5.1, Typha latifolia fibers were successfully manufactured. It is left for future works to study the possibility of optimizing the fiber properties. It is possible to state that Typha latifolia fibers can be considered as a green option. Typha latifolia could be used in wetland cultivation when cleared peatlands are being replaced to lower the emissions (Helsingin Sanomat, 23.2.2021). Sodium hydroxide, that was needed in retting process of manufacturing Typha latifolia, is already used in Finnish forest industry when they manufacture cellulose (UPM). The energy that will be used in retting should be produced with emission-free way. Part of the ecology of the Typha fibers is that it biodegrades even in a composter that is meant for ordinary home-use, as studied in chapter 3.6.

In section 2.1 and 2.2 the ideal polyurethane structure was covered, and the earlier studies clearly indicated that open-cell structure should be targeted to achieve a sound absorbing test sample. Çelebi, et al. (2012) research indicates that the structure of polyurethane will be deciding factor for material's sound absorbing properties. The verification of Typha's enhancing effect to polyurethane's sound absorption properties would have needed multiple impedance tube tests. Collecting this amount of data was not possible in schedule-nor budget-wise. This explains why the sound absorption measurement was done only for Typha latifolia fibers, and the focus was centred on the bonding between Typha fibers and polyurethane, since it is one of the biggest challenges when manufacturing a natural fiber-PU-composite, as explained in section 2.5. The sound absorption properties of Typha fibers were promising as stated in section 4.3 and 5.2. This knowledge can be useful e.g., in production of insulation wool or sound absorbing wools from Typha latifolia fibers. More research is needed to produce Typha latifolia wool that would be as homogeneous as current insulation wools.

The promising bonding ability between polyurethane and Typha latifolia fibers may be an interesting study subject. The following questions would be 1) Does Typha fibers bond with other polyurethanes - especially, with polyurethanes without an isocyanate active group? 2) And how much the alkali treatment helped Typha fiber to bond with polyurethane? Even though the actual sound-absorbing property of manufactured Typha-polyurethane-composite was not measured, it can be stated based on our learnings that, at least in theory, it should have better sound-absorbing properties than non-modified polyurethane.

The author would suggest focusing next on producing textile from Typha latifolia fibers. As can be seen from table 3 (page 40), the Typha fiber length is rather short, less than 50 mm, which indicates that short-staple spinning machine could be the best option to process the fibers (Lord P. 2003). Staple Typha latifolia fibers can be tested by using ASTM D3822 – A Standard Test Method for Tensile Properties of Single Textile Fibers. Typha latifolia fibers' thermal transition were planned to be studied, but after a thorough conversation with many experts of the field, credited in Other sources-section, it was noticed that the best way to study thermal transitions is to manufacture a textile from Typha latifolia and use it as a specimen for fire testing. Manufacturing a textile from Typha latifolia enables the use of standards like SFS-EN ISO 6940:2004 and SFS-EN ISO 6941:2004 and fire testing can be done in textile laboratory of Tampere University of Applied Science. Decomposition of the fiber can be measured with a TGA, which can be found from Aalto University and VTT. LOI-value can be measured in Tampere by VTT. Typha latifolia's thermal transition results can be compared to commonly used textile fibers' thermal transition properties that can be found from book by A. Richard Horrocks, D. Price; *Fire Retardant Materials*, table 4.3 (2001) and *Palosuojatut tekstiilit: Ominaisuudet ja käyttö*, page 24 (2001).

7 CONCLUSION

One of the key ideas of this thesis was to complement earlier Typha-studies and create a better starting point for future researchers to build their own ideas on top of this research. That is why the author made a choice to focus especially on the quality of presentation by including sufficient number of pictures and manufacturing details.

As already stated in section 5.1, Typha latifolia fibers were successfully manufactured and useful information was gained from the manufacturing process. It was noticed that the biggest factor to control the fiber length and thickness is to choose the correct fiber separation process. Damp fibers that were separated by hand had greater thickness and length compared to fibers that were separated with carding tool.

Typha latifolia fibers' sound absorption properties were as good as expected. The fibers' good sound absorbing properties can be explained by its wool-like structure. Even though Typha latifolia fibers created a visually strong bond with polyurethane, the challenges with the mixing and the assumed small impact to the composite's sound absorbing property will leave a fair doubt in the rationality of manufacturing this kind of composite for sound absorption applications.

The wool manufactured from Typha latifolia fibers has potential to rise as one of the sustainable sound insulation materials. What supports this, is that Typha latifolia cultivation does not take land from food production and cultivating it can help to lower the emissions (Helsingin Sanomat, 23.2.2021). Also, the manufacturing process is fairly simple and can be optimized to be more sustainable as explained in discussion-section. Typha latifolia fibers decompose easily, as was proved in the section 4.2.

The next step would be producing textile from Typha latifolia fibers. This step would open new ways to study Typha latifolia fiber's properties, as explained in discussion-section. The biggest challenge of manufacturing Typha latifolia fibers in greater scale will most likely be that a relatively small amount of fibers can be extracted from Typha latifolia leaves, as stated in section 3.5. This leaves a question open if enough of Typha latifolia fibers can be cultivated for production needs. The author recommends an investigation where the focus will be on this subject.

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