Manufacturing of Lightweight Sandwich Composites with Bio-Based PU Foam Core and Cellulose Fiber Network Skin

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Materials Engineering, masters level
2016

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This project has received funding from the European Union Seventh Framework Programme (FP7/2007-2013) under grant agreement nº 608746
Acknowledgement

I would like to thank the European Union Seventh Framework Programme (FP7/2007-2013) under grant agreement n° 608746 for allowing me to perform this work. I would also like to thank Professor Mohini Sain for hosting me at the Faculty of Forestry at University of Toronto for three unforgettable months. Finally, I want to thank Professor Kristiina Oksman for providing me with this opportunity in the first place. I am very grateful for your trust and support in me throughout this past year. Thank you.

Luleå, August 2016

[Signature]
Abstract

The use of lightweight material are at an increasing demand in our society. Particularly, PU foams are used as lightweight insulation and support in sandwich composites for a wide range of products such as walls, packaging and car bumpers. This project aims to improve the mechanical properties of bio-PU foam by reinforcing the pre-resin with nanofibrillated cellulose (NFC). A lightweight sandwich panel was manufactured in a vacuum infusion process with carrot NFC-reinforced bio-PU foam core. Fine Kraft paper was used as skin and epoxy resin was used as adhesive. The sandwich was tested and compared to a non-reinforced PU foam core sandwich in terms of compressive, flexural and cellular properties. The result was a sandwich panel with improved mechanical properties, smaller cell size structure and lower open cell content. The summary table below shows the foam with largest difference to its reference foam, hence the highest performing foam.

Summary of NFC effect on material properties for foam density 35 kg/m$^3$

<table>
<thead>
<tr>
<th>Property</th>
<th>NFC effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell size</td>
<td>- 18 %</td>
</tr>
<tr>
<td>Open cell content</td>
<td>- 51 %</td>
</tr>
<tr>
<td>Compressive strength</td>
<td>+ 41 %</td>
</tr>
<tr>
<td>Compressive modulus</td>
<td>+ 46 %</td>
</tr>
<tr>
<td>Flexural strength</td>
<td>+ 167 %</td>
</tr>
<tr>
<td>Flexural modulus</td>
<td>+ 163 %</td>
</tr>
<tr>
<td>Facing strength</td>
<td>+ 88 %</td>
</tr>
</tbody>
</table>

Moreover, the results were evaluated in a material selection process by means of minimizing merit indices. A trend in the behavior of compressive properties of the foam and flexural properties of the sandwich panels could be established thanks to this evaluation method.

Overall, the results indicate that the mechanical properties of bio-PU foam can be improved by reinforcing the pre-resin with small amounts of wet carrot NFC. Ultimately, this work show a positive trend suggesting NFC- reinforced bio-PU foam has a great potential for use in commercialized products or structural components in future constructions.
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V. Nomenclature

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<th>Explanation</th>
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<tr>
<td>CNW</td>
<td>Cellulose nanowhiskers</td>
</tr>
<tr>
<td>NFC</td>
<td>Nanofibrillated cellulose</td>
</tr>
<tr>
<td>CO₂</td>
<td>Carbon dioxide</td>
</tr>
<tr>
<td>H₂O</td>
<td>Water</td>
</tr>
<tr>
<td>LTU</td>
<td>Luleå Technical University</td>
</tr>
<tr>
<td>OM</td>
<td>Optic microscope</td>
</tr>
<tr>
<td>MDI</td>
<td>Diphenylmethane diisocyanate</td>
</tr>
<tr>
<td>NCO</td>
<td>Isocyanate group</td>
</tr>
<tr>
<td>OH-value</td>
<td>Hydroxyl value</td>
</tr>
<tr>
<td>pphp</td>
<td>Parts per hundred polyol</td>
</tr>
<tr>
<td>PU</td>
<td>Polyurethane</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscope</td>
</tr>
<tr>
<td>U of T</td>
<td>University of Toronto</td>
</tr>
<tr>
<td>VI</td>
<td>Vacuum infusion</td>
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</table>
1. Introduction
This section covers the introduction and background to the project as well as the objectives, limitations and planning

1.1. General introduction
Lightweight materials are important in most industry sectors today; energy, construction and transportation only to mention a few. For the latter, including aeronautics and automotive industries, stricter rules on carbon dioxide emissions practically forces manufacturers to use more lightweight materials [1]. The industry market research group Freedonia, estimated that the use of lightweight materials in the automotive industry increased by 8.7% annually from 2008 to 2013 and that the use will have increased by another 5.2% annually by 2018 [2].

The increased tendency to use lightweight materials does not only depend on stricter emission norms but also on the many benefits they provide. The major advantages of using lightweight structures are decreasing costs, design flexibility, labor and transport efficiency as well as environmental factors [3]. Polymeric foams, such as PS, PET and PU foam, are lightweight materials often used due to their low density and excellent insulation properties. However, using neat foam as structural component is difficult since there often are constraints on mechanical properties such as impact resistance, stiffness or strength. One solution is to reinforce the foam with an additive, such as natural fibers. However dispersion can be an issue and this often increases the density of the foam composite more than it improves the mechanical properties [4], [5]. Another solution is to use foam as core material in sandwich composite structures. This way, the skin of the structure will provide for the constraints while a relatively low weight is maintained by the foam core [6]. In combining these solutions, a reinforced foam used as core in sandwich structure has great potential. Therefore, as a result of the many advantages these lightweight structures with improved mechanical properties provide, they are excellent candidates for structural components in automotive and other industries.

1.2. Background to project
The INCOM project is a European Union funded project for research and innovation concerning Industrial Production Processes for Nanoreinforced Composite Structures. The project started in 2013 with LTU as one of the consortium members. Among LTU’s contribution so far to the INCOM project is the extraction of nanofibrillated cellulose (NFC) from carrot residue [7], [8],
[9] and manufacturing of lightweight bio-PU foam reinforced with NFC [10], [11]. Furthermore, this foam has been used in a vacuum infusion process to produce a sandwich structure with Kraft paper skin [12]. The results were promising with a lightweight composite product however the data was inconclusive concerning what effect, if any, the NFC addition in the foam had on the final sandwich structure. This was mostly due to the many uncertainties introduced in the manufacturing process.

Based on the previous work within the INCOM project, it is already known that NFC reinforced bio-PU foam yields better mechanical properties compared to neat bio-PU foam [10]- [12]. What is not known, since the results were inconclusive, is if this improvement also would result in sandwich panels which are mechanically superior. Superior core materials that contribute to an overall increase in mechanical properties of the sandwich panel has great potential to replace core materials used today. They also have the potential to be used in a wider range of applications with higher mechanical demands or decrease the thickness of existing products.

1.3. Objective

This research project aims to compare NFC reinforced bio-PU foam with a non-reinforced bio-PU foam in manufacturing of sandwich composites. The goal is to obtain a bio-PU foam with higher mechanical performance and investigate whether the NFC addition in the foam has a positive effect on the final sandwich structure. This will mainly be evaluated through flexural properties of the sandwich panels and their cell structure.

The objectives will be achieved through three main tasks:

I. Manufacturing of two core materials; bio-PU foam (BPU) and NFC reinforced bio-PU foam (NFC); for four different controlled densities of:
   - 35 kg/m$^3$
   - 40 kg/m$^3$
   - 45 kg/m$^3$
   - 50 kg/m$^3$

II. Vacuum infusing the foams into sandwich composites with Kraft paper skin and epoxy resin
III. Characterizing and comparing properties
- Dispersion study of the NFC
- Microstructure: X-ray tomography, SEM, OM
- Open cell content
- Compressive properties
- Flexural properties

1.4. Project limitations
Due to a set timeline of 20 weeks covering the master thesis project of 30 ECTS, there are a few limitations to the project.

- The NFC is extracted from wet carrot residue and centrifuged to a concentration of 8%. The concentration is limited to this level due to the high increase in viscosity caused by the water removal. This means the NFC content in the foam is limited to a maximum of 0.17wt%, any higher will decrease the density below 35 kg/m³ (see Table 5 in section 3.2.1). NFC content is thus limited by the foam density.
- Only a few material properties will be investigated. A ranking list prioritizing the properties to investigate has been made should time prove to be limiting or in excess (see Appendix A).
- Skin thickness of sandwich structure is set to 1 Kraft paper for simplicity. The difference between 1 or more papers will not be investigated. This was tested briefly in previous work but might be interesting to investigate for future work [12].
- This project is also limited to using materials that could be bought/brought/found at U of T. Castor oil and carrot NFC was brought from LTU. Other ingredients were bought or borrowed from U of T.

1.5. Project plan
In the beginning of this project, a planning was made with all the main tasks and milestones. This planning, as well as a GANTT diagram can be found in Appendix A. The timeline of the project also takes into consideration the difficulties of planning experimental work, mainly due to unforeseen difficulties that may occur in both manufacturing and evaluation methods.
1.6. Thesis report outline

This report is divided up into nine chapters. Following the introduction is a literature study dealing mainly with the concept of lightweight sandwich materials, vacuum infusion processing and relevant characterization methods for this thesis. Chapter three deals with the methodology of the experimental work with material information and manufacturing methods. Following this is the result, discussion and conclusion as well as future work and references. Lastly, the appendices can be found under chapter nine.
2. Literary study

*This section covers the literature study which deals mainly with the concept of lightweight sandwich materials including components, vacuum infusion processing and then relevant characterization methods for this thesis*

2.1. Introduction to lightweight sandwich composites

A sandwich composite consists of two thin materials, the “skin”, separated by a much thicker lightweight material, the “core” (Figure 1). The principle is to increase the structure’s stiffness and strength without compromising on weight or cost. According to ASTM sub-committee D30-9, concerning common test methods for sandwich constructions, the ratio of the skin to core thickness should be equal to or less than 1/10\(^{th}\) (e.g. ASTM C393).

![Figure 1: Basic components of a sandwich composite](image)

There are numerous benefits to a material like this. The main advantage is that it has a considerably higher shear stiffness to weight ratio than an equivalent beam made of only the core material or the face-sheet material. The efficiency of a lightweight sandwich composite can be demonstrated by comparing it to a steel beam in bending. To have the same constraint on deflection, a standard sandwich panel can save almost 90% in weight compared to a steel beam although the sandwich is over three times thicker. [3]

Moreover, the weight that can be saved by using lightweight sandwich constructions compared to other materials of equivalent strength and stiffness leads to many more advantages. For example more efficient transporting and loading per shipment giving environmental advantages, and more efficient mobility and assembly saving both manpower and reducing safety concerns.
Many different types of sandwich panels can be accomplished by using different facings and core materials combined with varying geometries. As a result, optimum designs can be produced for specific applications. [13]

2.1.1. Life cycle aspect
As previously established, sandwich materials make structural designs lighter making the construction less energy consuming over its lifetime. This becomes very important to the transport industry for as long as a vehicle weights less, its fuel savings will be enormous.

Taking the whole life-cycle approach to materials mean that four stages have to be considered: raw material production, parts production, product usage and end-of-use disposal. From these categories, the product usage accounts for 85% of the total energy consumption for the whole life-cycle [1]. The environmental impact of material choice in the beginning and the end of a vehicle’s life cycle becomes minor in comparison to the impact of usage. Because of this, it becomes vital to reduce energy consumption in transportation with more lightweight materials.

2.2. Components and materials

2.2.1. Core material
The main task of a core material is to support the thin skins so that they do not buckle and keep their relative position to each other. Thus the core need to be stiff enough to keep a constant distance between the faces and rigid in shear so that the faces do not slide over each other. The shear rigidity forces the faces to cooperate with each other. If the core is weak in shear, the faces do not cooperate and the sandwich will lose its stiffness. [3]

The most important properties for core materials are low density and strength in different directions but there are also often demands on buckling, thermal or electrical insulation, moisture absorption, fire safety, ageing resistance and of course cost. Materials typically used as core in sandwich structures are wood, aluminum, foam and honeycomb structures. Foam has many of the advantage above but also the advantage of freedom in design. Foam materials are shapeable in almost any kind of geometry until the final stage of production when it gets its final shape. This makes it a popular material choice in structural design.
For the purpose of creating lightweight panels in the INCOM project, bio-PU foam has been agreed upon [14].

**PU foams**

Polyurethane foam was one of the first core material ever to be used in sandwich structures. It is particularly popular to use due to its low density, low moisture permeability, high strength to weight ratio and low thermal conductivity [5]. Having gone through decades of research and development, PU foam today has the unique potential to be tailored to a large variety of characteristics depending on desired application. In particular, PU foams can be divided into three groups based on degree of crosslinking or stiffness of the foam. They are flexible foam, semi-rigid foam, and rigid foam and are made using different formulations and compositions. The degree of crosslinking mainly depend on the side reactions taking place in the PU foam, primarily between isocyanate and other additives. [15]

Moreover, PU foams can also be divided into groups based on their open or closed cell structure. For open-cell foam, the cell walls are ruptured and air fills the hollow cells. In contrast, cell walls of closed-cell foam are essentially intact and cells are filled with gas. The gas is either remaining gas from those used to saturate the polymer or gas released by the blowing agent during the formation of the foam. In general, flexible foams have open-cell structure and are used for packaging and cushioning purposes, while rigid foams have a closed-cell structure and are used for building insulation. [15]

Evidently, the chemistry of polyurethanes is quite complex and is mainly influenced by the primarily used polyol and isocyanate, but also by additives like catalysts, surfactants and blowing agents. The choice of additives will strongly influence the properties and cell structure of the foam and so the selection of raw materials is essential [3]. The typical components of PU foam have been summarized in Table 1 with their specific task described.
Table 1: Components of PU foams and their specific task

<table>
<thead>
<tr>
<th>Component</th>
<th>Task</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyol</td>
<td>The resin base that provide the hydroxyl groups to the cross-linking reaction (Equation 1)</td>
</tr>
<tr>
<td>Isocyanate</td>
<td>The reactive pre-polymer. Provide the isocyanate groups to the cross-linking reaction and blowing reaction (Equation 2)</td>
</tr>
<tr>
<td>Blowing agent</td>
<td>React with isocyanate groups to initiate foaming</td>
</tr>
<tr>
<td>Surfactant</td>
<td>Reduce the surface tension, emulsify the incompatible ingredients, promote the bubble nucleation during mixing, stabilize the cell walls during foam expansion and reduce the de-foaming effect of any solids added</td>
</tr>
<tr>
<td>Catalyst</td>
<td>Balance the blowing and the cross-linking reaction</td>
</tr>
<tr>
<td>Other reinforcements/additives</td>
<td>UV-stabilizers, bacteriostats, flame retardants, pigments, plasticizers etc.</td>
</tr>
</tbody>
</table>

Since the beginning, polyols used in PU foam production have been derived from petrochemical sources. The polyols used are mainly hydroxyl-terminated polyethers but also hydroxyl-terminated polyesters. The presence of hydroxyl groups is a vital part of the polyol since they are to react with the cyanate functional groups to create urethane linkages. The choice of polyol, especially the functionality and the number of reactive hydroxyl groups per polyol molecule, ultimately control the degree of cross-linking between molecules. This has an important effect on the mechanical properties of the PU foams.

In recent years, scientists have been devoted to deriving polyol using vegetable oil. This is a rather complicated process since vegetable oil needs to be functionalized to create the necessary hydroxyl groups. The polyols successfully created from vegetable oil, so called bio-polyols are based on among others soy-bean oil [16], [17], [18], rape-seed oil [19], palm oil [20], [21] castor oil [22], sugar-cane bagasse [23] or canola oil [24]. Additionally, researchers have also successfully managed to replace part of the polyol content with lignin through an oxypropylation process [25], [26]. Nowadays, bio-polyols are increasingly used in the production of polyurethanes which means that at least part of the polymer is derived from renewable sources [27].
Moreover, life cycle assessments have shown that bio-polyols have less environmental impact than petroleum polyols. While for example 90% of energy is attributed to fossil fuels in the production of polyols, only 35% is attributed to castor oil polyols [15]. Also, the production of bio-polyol emits 1/5th the amount of acid gas compared to petroleum based polyol. Finally, since castor oil and other bio-polyols maintain an essentially zero carbon dioxide emission, it means bio-polyols provides a more environmental friendly route to produce PU foams.

Extensive research has been devoted to comparing polyol with bio-polyol in PU foam properties. This research has in many cases shown that bio-polyols improved the properties of the final PU foam compared to the use of petroleum based polyols. Especially, bio-polyols improve hydrophobicity and thermal stability [22], but also improve tensile strength and elongation with smaller and better distributed cell size [16]. Some research has suggested that it also improves the compressive strength [15], [23], while others contradict this result with lower compressive strength and modulus [16]. Whether replacing polyol with bio-polyol yields improved properties or not, all research concludes that vegetable oil derived polyols can successfully replace petroleum based polyol in fabrication of PU foam.

*Chemistry of PU foam*

PU foams can be produced using several different techniques: spray-applied technique, pour-in-place process, slabstock process and free-rise or restrained-rise continuous lamination process [28]. In the production of PU foam, liquid components are mixed together and react upon contact. There are two main reactions, the cross-linking reaction and the blowing reaction. The cross-linking reaction take place between the isocyanate groups in pMDI and hydroxyl groups in polyol and create urethane linkages (Equation 1). The blowing reaction depends on what kind of blowing agent is used, a chemical or a physical. In both cases, the exothermic cross-linking reaction volatiles the blowing agent into a gas which initiates the foaming. Cyclopentane is a physical blowing agent often used for industrial manufacturing [29] which volatiles when heated. More convenient for researching purposes is water, a chemical blowing agent that entrain air bubbles when stirred. These bubbles act as nucleation sites for the water to react with isocyanate and give a primary amine (polyurea) and carbon dioxide (Equation 2). The CO₂ gas then expand the bubbles and the foam starts rising [30].
Equation 1: The cross-linking reaction with polyurea group encircled

$$\text{OCN} - \text{NCO} + \text{OH-R-OH} \rightarrow \text{[NH-N=C-O-R-O]}$$

Equation 2: The blowing reaction

$$\text{OCN} + \text{H}_2\text{O} \rightarrow \text{[NH-C-OH]} \rightarrow \text{[NH}_2 + \text{CO}_2$$

The foaming reaction may be impeded by the cross-linking reaction. As cross-linking increases, the viscosity of the mixture increases, which hinders the foam expansion. Consequently it is important to control the relation between the blowing and cross-linking reaction to have a homogeneous cell structure [15]. The control of these two reactions is achieved through the additives.

In the INCOM project, the resin base will consist of a bio-polyol reinforced with NFC to optimize mechanical performance [14].

**Bio-PU foam reinforced with nanocellulose**

Bio-PU foam reinforced with nano-sized cellulose particles has showed remarkable improvements in mechanical properties. Zhou et al. [31] obtained an increase in compressive modulus and strength with 216% and 117% respectively using 1.6 wt% cellulose nanocrystals (CNC). Li et al. [32] reported as much as a 210% and 270% increase in compressive modulus and strength respectively on using 1 wt% freeze-dried cellulose nanowhiskers (CNW). Moreover Faruk et al. [33] showed that the open cell content was significantly influenced as they incorporated micro/nanofiber cellulose and lignin to a soy-bean based PU foam. The open cell content reduced from 90 to 12%. Also the compressive properties improved with the cellulosic nanofiber enhancement and the isocyanate content reduced to 7% in the foam formulation.

Furthermore the moisture content of nano-sized cellulose is very important to the foaming process and properties of the foam. Zhou et al. [10] compared using both freeze-dried nanofibrillated cellulose (NFC) and wet NFC in 10 wt% concentration as reinforcement in bio-PU foams. They successfully added dried fibers, NFC$_D$, up to 2 wt% in the polyol mixture before viscosity became too high for mixing to be efficient. For wet fibers only minimal
amounts of NFC_w, maximum 1 wt%, were added. Otherwise the water content became too high which lead to a heterogeneous foam with much lower performance. On a side note, they suggested that water removal in the dry NFC might have affected the dispersion of NFC in the polyol, leading to less dispersed NFC in the foam compared to the wet NFC foams.

2.2.2. Skin material

The main task of a skin material is to carry the tensile and compressive stress in the sandwich structure. The skin must also be able to carry local pressure [3]. The most common skin materials to use are steel, aluminum, glass-fiber or carbon-fiber reinforced plastics. In the INCOM project, the objective is to manufacture more environmental friendly panel structures and so cellulose fiber networks will be used, namely eco Kraft paper.

Kraft paper (Figure 2a) is a strong and resistant material typically used for packaging products with high demands on strength and durability. Kraft paper is produced from chemical pulp in the Kraft Process. The raw material in this process is normally softwood pulp which can originate from a wider range of fiber sources than most other pulping processes. All types of wood, including very resinous types like southern pine, and non-wood species like bamboo and kenaf can be used in the Kraft process. The long fibers provides the paper its strength although wet strength chemicals are added to the pulp to even further improve the strength [34]. The chemical pulp is transferred to paper machines where the paper is produced. The success of the final paper product often depends on the relative abundance of hemicellulose. Paper’s tear strength is directly related to the level of hemicellulos. Because of this, manufacturers of Kraft pulp for papermaking work hard to remove lignin and maintaining the hemicellulose to make high strength paper structures [35].

![Figure 2: a) Kraft paper roll and b) used as lining in particle boards (Kotkamills Oy, Finland [36])](image-url)
The qualities of Kraft paper include high elasticity, high tear resistance, high tensile strength and high permeability and it has for this reason been used in various composite applications [37]. For example, Kraft paper can be used as a cheap material for lining particle boards (Figure 2b). Moreover, manufacturers of counter-top laminates impregnate highly porous “saturating Kraft paper” with phenolic or melamine resins. The dimensional stability achieved by combining the cellulosic fiber matrix with the resin, filling the void spaces, is far in excess of what can be achieved by either component [38].

2.2.3. Adhesive material
To attach the skin to the core material, an adhesive is generally used. The main task of an adhesive is to transfer shear and tensile forces between skin and core. As a general rule, the adhesive should be able to take up the same shear stress as the core. It must also be compatible with the core and the skin. Depending on what skin material is used, the adhesive must also be able to impregnate the skin completely. Particularly when using skin based on cellulose networks, a compatible resin should be used to impregnate the skin. The level of impregnation of the skin has a huge impact on mechanical properties as non-impregnated Kraft paper has much lower properties compared to impregnated Kraft paper.

Some common resins are polyesters, vinylester, epoxies, urethane and silicones [39]. The type of resin used depend on application. Typically, epoxy is preferred for vacuum infusion because it has the advantage of low viscosity and can be cured in room temperature. The low viscosity ensures uniform and fast wetting of the platform. Epoxy also has advantageous mechanical properties and a wider temperature range in comparison to many other polymeric resins. [40]

2.3. Manufacturing
Sandwich panels are manufactured by attaching the two thin, strong and stiff skins to the lightweight and relatively thick core using the adhesive. The process may be hand lay-up, RTM, compression or vacuum guided. For the purpose of this project, a simple vacuum infusion process will be used. The theory behind the process is described below.
2.3.1. Vacuum infusion processing

The vacuum infusion (VI) process is a technique that uses vacuum pressure to drive resin into a laminate. It is a popular technique to create medium to large high-tech composite parts for the boating or automotive industry, often with very complicated geometry [41]. In sandwich structures, it has the advantage of attaching the skin to the core at the same time as impregnating the skin layer.

Figure 3 demonstrates the basic set-up of a vacuum infusion process. Materials are laid dry onto the mold under a vacuum bag and when vacuum is applied, the air is sucked out from the bag, compressing the part. Once a complete vacuum is achieved, resin is sucked into the laminate through carefully placed tubing. The resin saturates the part from input to output. Once resin has saturated the whole part, vacuum is switched off and the part is left to cure.

![Figure 3: Illustration of the set-up for a basic VI](image)

**Advantages and disadvantages of VI**

The advantages of VI are multiple. Vacuum bagging greatly improves the fiber-to-resin ratio, and results in a stronger and lighter product. VI uses less resin than typical lamination or hand lay-up procedures. This is favorable as resin alone is very brittle and if in excess, will weaken the component. Vacuum infusion offers unlimited set-up time, which is good for complicated parts. It is also a very clean process as there are no brushes or rollers, and therefore no splashing or spattering. Also, there are no fumes to worry about. [42]
However, VI also has the drawback of being a rather complicated process since errors cannot be corrected after infusion starts. For example, if a leak occurs in the vacuum bag, it could result in resin pooling, undersaturation or flow stoppage. Even the smallest amount of air can destroy a component completely. Because of this, it is very important to be sure the bag is intact before initiation. [42]

**Problems in manufacturing**

One of the biggest difficulties in vacuum infusing lightweight sandwich structures is the resin impregnation of the skin. This becomes an even bigger problem when using thicker core materials since the top and bottom skin are further apart. A higher pressure is needed to drive the resin though the vacuum bag and completely impregnate both the top and bottom skin. Dweib et al. [43] manufactured structural panels based on soy-bean oil resin and natural fibers through a VI process. They reported that recycled paper was poorly infused on the bottom side which was attributed to the high density and low permeability of the paper. This problem they managed to surpass using a combination of recycled paper and fluffy fiber as reinforcement around the foam. Another way to overcome the problem of poor impregnation could be to use a more permeable paper. Kraft paper has the advantage of a very high permeability, significantly decreasing the resin fill time as shown by Aitomäki et al. [44]. This makes Kraft paper a good candidate for VI.

Another problem with the VI process is the consumption of additional resin in the core-laminate interface. The extent of consumption depends on the porosity of the core material. The higher the open cell content, where air flows freely in and out, the more consumption. The uptake will also be influenced by the cutting/sawing process. Eventual closed cells will be ruptured and resin can enter during the infusion (Figure 4). It becomes very important to minimize the resin consumption in all designs to reduce weight and cost as well as brittleness of the structure. [45]
2.4. Characterization of important properties

ASTM International has appointed a subcommittee D30.09 for standard test methods concerning sandwich constructions. Relevant test methods concern among others mechanical behavior (compression, tensile, flexural) and thermal behavior (insulation, water absorption) [46]. For the purpose of this literary study, only characterization methods relevant to this thesis work will be discussed; open cell content and compressive properties for the foam and flexural properties for the sandwich composite.

Open cell content is a very important property in foams. The importance of determining the level of open to closed cells in a foam lies in the strong relationship cellular structure has to mechanical performance. In particular, a closed cell foam has much higher compressive strength than an open cell foam structure which is illustrated in Figure 5 [47].
The typical open cell content for rigid PU foams lies between 2-10% [28]. For closed cell PU foam classification, the closed cell content should be higher than 90%. For open cell classification, the level of closed cells should generally be less than 20% [48].

The compressive strength is also an important property in a foam. In particular for NFC reinforced foams, it can give an idea of dispersion of the fibers. The variation in strength throughout the sample also gives an idea of this. [49]

Furthermore, flexural properties are often used to evaluate the mechanical performance of sandwich composites, mainly because of their application as structural components. Consequently, a third of the standards on committee D30.09 concern flexural properties. [46]. A flexural test is very effective as it does not only give quantitative information about flexural properties but also qualitative information about failure mode and failure location. The flexural properties can be determined by a variety of different standards depending on the specimen geometry and desired property. For the purpose of evaluating the laminates in this work, a “standard” 3-point bending method will be used.
2.5. Material selection considerations

Another way to characterize the results is to take material selection into account. In material selection, the goal is to decide which material option has the best performance by deriving appropriate material indices. These indices are derived from basic material properties which are relevant for the function of the component. In most cases, the objective is to minimize mass and so materials with lower index values will maximize that aspect of the performance of the component.

Material selection applied to this case means that the goal is to find which of the sandwich panels has the minimal density without compromising on mechanical performance, hence compressive (Figure 6) and flexural properties (Figure 7). The derivations for the material indices in compression and bending are shown in Table 2-3 below with derivation method applied from Ashby’s Material Selection in Mechanical Design. [49].

Figure 6: Mechanical properties of foam in compression

Figure 7: Mechanical properties of sandwich in 3-point bending
Table 2: Material index derivation for foams loaded in compression [49]

<table>
<thead>
<tr>
<th>Case study</th>
<th>Foams in compression, length and shape specified</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Free variable</strong></td>
<td>Foam material choice</td>
</tr>
<tr>
<td><strong>Objective</strong></td>
<td>Minimize mass: $m = V\rho = AL$ (1)</td>
</tr>
<tr>
<td><strong>Constraint 1:</strong> Compressive strength</td>
<td></td>
</tr>
<tr>
<td>Compressive force: $F_c = \sigma_c A$ (2)</td>
<td>$\Rightarrow A = \frac{F_c}{\sigma_c}$</td>
</tr>
<tr>
<td>(2) in (1) $\Rightarrow m_1 = F_c L \left(\frac{\rho}{\sigma_c}\right)$ (3)</td>
<td></td>
</tr>
<tr>
<td><strong>Equation 3: Material index 1</strong></td>
<td>$M_1 = \frac{\rho}{\sigma_c}$ (minimize)</td>
</tr>
<tr>
<td><strong>Constraint 2:</strong> Compressive modulus</td>
<td></td>
</tr>
<tr>
<td>Critical compressive force: $F_{crit} = \frac{n^2\pi^2E_c L}{L}$ (4)</td>
<td>Where $n=$ constant</td>
</tr>
<tr>
<td>I-moment ($m^2$): $I = \frac{bh^3}{12} = \frac{(bh)^2}{12} = \frac{A^2}{12}$ (5)</td>
<td></td>
</tr>
<tr>
<td>(5) in (4) $\Rightarrow F_{crit} = \frac{n^2\pi^2E_c A^2}{12L} \rightarrow A = \left(\frac{12LF_{crit}}{n^2\pi^2E_c}\right)^{\frac{1}{2}}$ (6)</td>
<td></td>
</tr>
<tr>
<td>(6) in (1) $\Rightarrow m_2 = \left(\frac{12LF_{crit}}{n^2\pi^2E_c}\right)^{\frac{1}{2}} L \left(\frac{\rho}{E_c^{1/2}}\right)$ (7)</td>
<td></td>
</tr>
<tr>
<td><strong>Equation 4: Material index 2</strong></td>
<td>$M_2 = \frac{\rho}{E_c^{1/2}}$ (minimize)</td>
</tr>
</tbody>
</table>
### Table 3: Material index derivation for sandwich loaded in bending [49]

<table>
<thead>
<tr>
<th>Case study</th>
<th>Sandwich in bending, length and shape specified</th>
</tr>
</thead>
<tbody>
<tr>
<td>Free variable</td>
<td>Sandwich material choice</td>
</tr>
<tr>
<td>Objective</td>
<td>Minimize mass: ( m = V \rho = AL ) (8)</td>
</tr>
</tbody>
</table>

#### Constraint 3: Flexural strength

Flexural force: \( F_f = \frac{CZ \sigma_f}{L} \)  
Where \( C = \) constant

\[
Z\text{-moment (m}^3\text{)}: Z = \frac{bh^2}{6} \simeq \frac{(bh)^{3/2}}{6} = \frac{A^{3/2}}{6} 
\]  
(10) in (9) \( \rightarrow \) \( F_f = \frac{CA^{3/2} \sigma_f}{6L} \rightarrow A = (\frac{6F_fL}{C\sigma_f})^{2/3} \) (11)

(11) in (8) \( \rightarrow \) \( m_3 = (\frac{6F_fL}{C})^{2/3} L \left( \frac{\rho}{\sigma_f^{2/3}} \right) \) (12)

**Equation 5: Material index 3**

\[ M_3 = \frac{\rho}{\sigma_f^{2/3}} \text{ (minimize)} \]

#### Constraint 4: Flexural modulus

Flexural stiffness: \( S = \frac{F}{\delta} = \frac{C_1E_fI}{L^3} \)  
Where \( C_1 = \) constant

\[
I\text{-moment (m}^2\text{):} \quad I = \frac{bh^3}{12} \simeq \frac{(bh)^2}{12} = \frac{A^2}{12} 
\]  
(14) in (13) \( \rightarrow \) \( S = \frac{C_1E_fI}{L^3} = \frac{C_1E_fA^2}{12L^3} \rightarrow A = (\frac{12SL^3}{C_1E_f})^{1/2} \) (15)

(15) in (8) \( \rightarrow \) \( m_4 = (\frac{12SL^3}{C_1})^{1/2} L \left( \frac{\rho}{E_f^{1/2}} \right) \) (16)

**Equation 6: Material index 4**

\[ M_4 = \frac{\rho}{E_f^{1/2}} \text{ (minimize)} \]
Note that the moment in constraint 2-4 assumes a near square cross-section \((b \approx h)\) which is not always the case. However, since all beams have the same geometry in this case, the shape factor can be ignored. This makes the indices very general, but rather useful [49].

2.6. Summary of literary study

The following can be concluded from the literature study:

- Bio-PU foam can be comparable in properties to traditional petro-oil PU foam and is preferred because of its renewable source. Bio-PU foam reinforced with NFC has even more potential as a core material because it has shown superior mechanical properties compared to neat bio-PU foam of similar density.
- Kraft paper is a preferable skin material mainly because of its high permeability and renewable source.
- Epoxy is a preferred resin to use as adhesive between the core and the skin since it has low viscosity, high mechanical properties, wide temperature range and room temperature curing.
- Vacuum infusion can successfully be used to manufacture the sandwich composite. It also has the ultimate advantage of attaching the skin to the core at the same time as impregnation the skin.
- Relevant methods to evaluate the sandwich structure are by; open cell content, compressive properties and flexural properties through 3-point bending test.
- Material selection indices provide a mean to characterize the results based on most efficient performance per mass. In this case, the highest compressive and bending strength and modulus per weight.
- Before foaming it is important to control the moisture content and dispersion of NFC.
- Before and during VI, there are some important variables to keep in mind namely: cellular structure of the core (open or closed cells), compatibility of the resin with skin and core, complete vacuum to avoid air spill or resin pooling, and full impregnation of the skin.
3. Methodology

This section covers a detailed description of the materials used and the formulation of the foams. The manufacturing method for the foam and the sandwich panel are also described and finally the characterization methods explained.

3.1. Material

The sandwich composite was manufactured with a bio-PU foam core, Kraft paper skin and epoxy resin as matrix. The Kraft paper was an Absorbex Eco Kraft paper (Kotkamills Oy, Finland, TI352_040_0) made from sawdust and recycled fibers with a weight of 30 g/m² [36]. The resin was a mixture of low viscosity epoxy with a slow hardener (Plastic World, Canada, West System 105/206). It had a mixed density of 1180 kg/m³ and viscosity of 725cps at 22°C [50]. The materials used in the bio-PU foam are summarized in Table 4.

Table 4: Foam constituents, role and properties

<table>
<thead>
<tr>
<th>Bio-foam material</th>
<th>Role</th>
</tr>
</thead>
<tbody>
<tr>
<td>Castor oil (Jagropol-400). Jayant Agro-Organics LTD, India.</td>
<td>Resin Base</td>
</tr>
<tr>
<td>Functionality: 3, OH-value: 0.35 gKOH/g</td>
<td></td>
</tr>
<tr>
<td>Polymeric methane diphenyl isocyanate, pMDI (Rubinate M). Huntsman Corporation,</td>
<td>Reactive pre-polymer</td>
</tr>
<tr>
<td>West Point, GA, USA</td>
<td></td>
</tr>
<tr>
<td>Functionality: 2.7, NCO: 31%</td>
<td></td>
</tr>
<tr>
<td>De-ionized water. Functionality: 2</td>
<td>Blowing agent</td>
</tr>
<tr>
<td>Polysiloxane (DABCO DC 5357). Air Products, Canada</td>
<td>Surfactant</td>
</tr>
<tr>
<td>N,N-Bis[3-(dimethylamino)propyl]-N,N-dimethylpropane-1,3-diamine (Polycat 9)</td>
<td>Reaction catalyst</td>
</tr>
<tr>
<td>Air Products, Canada</td>
<td></td>
</tr>
<tr>
<td>Dipropylene glycol- Triethylene diamine (DABCO 33 LV). Air Products, Canada</td>
<td>Gelling catalyst</td>
</tr>
<tr>
<td>Wet NFC. Wood and bionanocomposite division, LTU, Sweden. 8 wt% fiber in water</td>
<td>Reinforcement</td>
</tr>
</tbody>
</table>

The NFC was extracted from carrot residue supplied by Brännhults AB by a grinding method developed previously by the group at LTU [7], [8] (Figure 8). The residue was washed and treated in an alkali treatment before bleached and washed to a neutral pH. Grinding was
performed with a supermass collider, (Masuko Sansuko, Japan, MKCA6-2) until a gel was formed with a NFC content of 1.4 % (98.6% H₂O). The wet fibers were then centrifuged to a concentration of 8 wt% NFC (Beckman Coulter J25i). Higher concentrations were limited due to the increasing viscosity of the gel.

Figure 8: NFC processing by ultrafine grinding

3.2. Method

3.2.1. Manufacturing methods

Free-rise foaming method
The foam was prepared using a free-rise foaming method with a formulation determined by the isocyanate index and moisture content of NFC. The isocyanate index is the mole ratio of isocyanate groups to hydroxyl groups in the reactant mixture (Equation 7). The ratio was set to equal one in order to complete cross-linking of the foam. The quantity of the constituents are calculated from parts per hundred polyol (php) after a pre-determined polyol mass has been fixed. Due to the presence of moisture in the NFC, which acts as the blowing agent, the amount of the other constituents could be calculated (Table 5). The additives were kept constant while the NFC/H₂O and pMDI was varied for different densities. For the exact formulation of the different foams, see Appendix B.

Equation 7: Isocyanate index

\[
\frac{\text{Moles of isocyanate groups}}{\text{Moles of hydroxyl groups}} = \frac{n(NCO)}{n(OH)}
\]
### Table 5: Foam formulation

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Short</th>
<th>Parts per hundred polyol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resin base</td>
<td>C.O</td>
<td>100</td>
</tr>
<tr>
<td>Reactive pre-polymer</td>
<td>pMDI</td>
<td>Varied</td>
</tr>
<tr>
<td>Surfactant</td>
<td>S</td>
<td>2</td>
</tr>
<tr>
<td>Reaction Catalyst</td>
<td>R.C</td>
<td>1</td>
</tr>
<tr>
<td>Gelling Catalyst</td>
<td>G.C</td>
<td>0.2</td>
</tr>
<tr>
<td>Wet NFC</td>
<td>NFC/H\textsubscript{2}O,*</td>
<td>0, 4, 4.5, 5, 6</td>
</tr>
</tbody>
</table>

*NFC content in foam (wt %)*

| NFC/H\textsubscript{2}O\,* | 0 | 0.13 | 0.14 | 0.15 | 0.17 |

* NFC concentration 8 % in water

The foam was produced in a similar procedure to the method used by Zhou et al. [31], seen in Figure 9. The wet NFC was mixed with C.O in a cup for 5 min with an electrical stirrer (kitchen whiskers, speed level 5 of 5). The additives were then introduced and the solution mixed for another minute. Finally, the pMDI was added and mixed in for 15 seconds before the whole solution could be poured into a mold and foaming was initiated. The foam was poured into a wide rectangular mold of base area 140x200 mm then left to cure in room temperature for 4 days.

*Figure 9: Illustration of the free-rise foaming method [31]*
Vacuum infusion
The laminates were prepared using an experimental vacuum infusion process. The set-up can be seen in Figure 10. A vacuum pump was connected to a resin trap to avoid any resin contact with the pump. The resin trap was then connected to the mold through an outlet tube on the mold. The part was covered by a vacuum bag which in turn was well sealed to the mold. The intactness of the system was tested as vacuum was applied. When the process started, the resin was sucked in through the inlet and through the part to finally exit through the outlet.

After the epoxy was infused, the whole system was left to cure under the bag in room temperature for 24h. The sandwich could then be extracted from the vacuum bag and left to post cure in room temperature for additional 3 days to maximize working strength [50].
Figure 10: Experimental set-up of the vacuum infusion
3.2.2. Sample preparation

The foams were sawed into rectangular plates of thickness 25 mm. The bottom plate was used for foam characterization while remaining plates were further machined to 10 mm thickness and used as core material in the vacuum infusion (Figure 11). The sandwich was further sawed and grinded into test samples (Figure 12). Due to the risk of inhomogeneity in the foam, hence the lower part having a different structure to the upper part, sampling for a specific test was made from the same location in all foams. Also, the thickness of all foam samples was parallel to the foam rising direction.

![Figure 11: Illustration of foam sampling](image1)

![Figure 12: Illustration of sandwich sampling (a) and before and after grinding (b)](image2)
3.2.3. Characterization methods

**Microstructure**

3-D imaging of sandwiches was done to determine resin penetration level and foam structure. An X-ray microtomography of model 1172 X-ray SkyScan was used. Resin penetration was measured by applying an eight line grid to the captured X-ray image and measuring the penetration depth at each line. This gave sixteen measurements per image. The procedure was repeated for five random images to give the average resin penetration.

**Optical microscope** (OM) was used to characterize dispersion in polyol mixture and in the foam structure. A red colorant was added to display higher contrast between fibers and matrix. OM was also used to calculate the cell size of the foam structure. Cell size was determined by *Equation 8* in reference to ASTM D3576.

*Equation 8: Cell size increase*

\[ d = 1.623 \frac{l}{n} \]

*Table 6: Symbols for cell size determination*

<table>
<thead>
<tr>
<th>L</th>
<th>Length of reference line [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>n</td>
<td>Number of cell wall intersections with the reference line [-]</td>
</tr>
</tbody>
</table>

**Scanning electron microscope** (SEM) was used to confirm results of 3-D imaging and OM in determining dispersion, cell size and resin penetration. A Hitachi SU 3500VPSEM was used with a 5 kV acceleration voltage. Sandwich samples were sawed and grinded to a smaller dimension and smoother surface, approximately 10x20 mm cross-section and 10 mm depth. Sample surfaces were sputtering coated with gold to avoid charging.

**Open cell content**

Open cell content was measured by gas pycnometry according to ASTM D6226-15/ ISO 4590. An AccuPyc 1330 Pycnometer was used with helium as an insertion gas. The test samples were of dimension 25x25x25 mm and a minimum of three samples were tested. The procedure yields
a volume of the sample chamber displaced by the specimen. The difference between this volume and the geometric volume of the specimen is a measure of the open-cell volume which gives the open cell content (Equation 9).

Notably, the apparent volume percentage of open cells includes the volume of the cells that were cut open during the preparation of the test specimen. This depend on the nature of the cellular plastic and on the surface to volume ratio of the test specimen. Therefore it is important to keep in mind that Equation 9 is not the actual open cell content calculated, but rather an approximation of it.

\[ O_c = \frac{V_g - V_i}{V_g} \times 100 \]

Table 7: Symbols for open cell content determination

<table>
<thead>
<tr>
<th>( O_c )</th>
<th>Open cell content [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>( V_g )</td>
<td>Geometric volume of specimen([\text{cm}^3])</td>
</tr>
<tr>
<td>( V_i )</td>
<td>Specimen displacement volume ([\text{cm}^3])</td>
</tr>
</tbody>
</table>

**Compressive properties**

The compressive properties were tested with an Instron 4411 universal machine with a 3 kN load cell and at a crosshead rate of 2.5 mm/min according to ASTM D1621 (in room temperature and 50% relative humidity). The specimen were of dimension 25x50x50 mm and a minimum of five specimen were tested for each sample. Compressive strength and modulus were determined by Equation 10-11.

\[ \sigma_c = \frac{F_{\text{max}}}{A} \]
Equation 11: Compressive modulus

\[ E_c = \frac{F l_0}{A \Delta l} \]

Table 8: Symbols for determining compressive properties

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>F&lt;sub&gt;max&lt;/sub&gt;</td>
<td>Maximum force [N]</td>
</tr>
<tr>
<td>A</td>
<td>Area of specimen [m&lt;sup&gt;2&lt;/sup&gt;]</td>
</tr>
<tr>
<td>F</td>
<td>Force [N]</td>
</tr>
<tr>
<td>l&lt;sub&gt;0&lt;/sub&gt;</td>
<td>Initial thickness of specimen [m]</td>
</tr>
<tr>
<td>Δl</td>
<td>New thickness of specimen [m]</td>
</tr>
</tbody>
</table>

**Flexural properties**

The flexural properties were tested through a 3-point bending test with an Instron 4411 universal machine (in room temperature and 50% relative humidity). The set-up of the test is illustrated in Figure 13. The deflection and force is measured as the pressure of load cell 3 kN is applied at constant rate 2.5 mm/min and from this data the bending strength (Equation 12) and modulus (Equation 13) of the sandwich panels were calculated. Five specimen of dimension 12x30x100 mm were tested for each sample. Additionally, the facing strength was calculated from Equation 14 according to ASTM C393 [46].

![Set up of a 3-point bending test](image_url)

*Figure 13: Set up of a 3-point bending test*
Equation 12: Bending strength

\[ \sigma_b = \frac{3F_{max}S}{2bd^2} \]

Equation 13: Bending modulus

\[ E_b = \frac{FS^3}{4bd^3D} \]

Equation 14: Facing strength

\[ \sigma_f = \frac{F_{max}S}{2t(d - c)b} \]

Table 9: Symbols for determining flexural properties

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>b</td>
<td>Width of specimen [m]</td>
</tr>
<tr>
<td>c</td>
<td>Core thickness [m]</td>
</tr>
<tr>
<td>d</td>
<td>Total thickness of sample [m]</td>
</tr>
<tr>
<td>D</td>
<td>Deflection due to a force F in bending [m]</td>
</tr>
<tr>
<td>F</td>
<td>Force [N]</td>
</tr>
<tr>
<td>( F_{max} )</td>
<td>Maximum force during compression/bending [N]</td>
</tr>
<tr>
<td>S</td>
<td>Support span length [m]</td>
</tr>
<tr>
<td>t</td>
<td>Facing thickness [m]</td>
</tr>
</tbody>
</table>
Statistics

All results were averaged and given standard deviation according to Equation 15-16.

Equation 15: Average

\[ \mu = \sum_{i=1}^{N} p_i x_i \]

Equation 16: Standard deviation

\[ \sigma = \sqrt{\frac{1}{N} \sum_{i=1}^{N} p_i (x_i - \mu)^2} \]
4. Results
This section covers the main results from foam and sandwich characterization including dispersion of the NFC, bio-foam cellular properties, sandwich general properties and mechanical properties

4.1. Dispersion of NFC
Figure 14A-F, shows the dispersion of the NFC carrot fibers in the water (A), the resin premix (B) and in the foam (C-F).

Figure 14: Dispersion of NFC: in water (A), castor oil (B) and in the foam at different scales (C-F). All images show mixtures based on 4.5 pphp
Figures A-C were taken with OM and a red coloring agent has been added to the polyol to enhance the contrast of the fibers in the matrix. Figures D-F were taken with SEM and shows the fibers on the surface of the foam.

The overall dispersion is worse in the polyol (Figure 14B) compared to in the water (Figure 14A). Many nanofibers are clustered together to form bigger micro-sized agglomerates in order of 100 µm in length. The diameter of the fibers is in the order of 4-10 nm yet appear as much thicker in the agglomerated clusters. Furthermore, these fiber aggregates appear in similar size in the foam (Figure 14D-F). The NFC is located in the cell strut area and cell wall and the SEM images show fiber clusters in different sizes from 10-100 µm while the diameter is much less.

4.2. Sandwich composite
The vacuum infused sandwich composite can be seen in Figure 15. There was no problem with permeability of the Kraft paper and the foam was stiff enough to remain stable under the vacuum pressure. The thickness of core was however limited by the vacuum pump pressure to drive the resin through the vacuum bag. If this process become industrialized through for example a RTM process, the thickness could be extensively increased.

![Manufactured sandwich composite](image-url)
4.3. General characteristics

Figure 16 shows some general characteristics of the foams and the sandwich composites. Cell size is clearly decreased in the NFC foams. Also open cell content tends to decrease with addition of NFC reinforcement. Notable there is a very large difference in the density group 35 kg/m$^3$ while the difference is very small in density group 40 kg/m$^3$. The resin penetration and sandwich density are closely related but hard to see any trend between different density groups. Generally, is seems a higher cells size and open cell content leads to a higher resin penetration and sandwich density. On a side note, the standard deviation is very high in Figure 16D. This is to be expected as a consequence of the large variation in penetration depth of the resin.

Figure 16: General properties of the foam and sandwich. NFC in light yellow, BPU in dark purple
Moreover, the images from x-ray tomography shows the resin penetration varied a lot throughout the surface of the samples. Figure 17 shows the resin penetration in yellow contrast throughout the cross-section of higher and lower density groups 35/45.

![Figure 17: Resin penetration images from x-ray tomography. Epoxy in light yellow contrast](image)

4.4. Mechanical properties
The mechanical properties from compression of the foams and 3-point bending of the sandwich panels are presented in Table 10. For all density groups, the NFC foams exhibit higher properties than their reference foam. The only exception is the compressive modulus for 50 kg/m³ which is not improved. The sometimes large standard deviation is mostly seen in the
NFC foams. This can probably be attributed to poor dispersion with pockets of higher and lower concentration of NFC.

Table 10: Compressive properties of the foams and flexural properties of the sandwich composites

<table>
<thead>
<tr>
<th>Density [kg/m³]</th>
<th>Type</th>
<th>Compressive Properties [MPa]</th>
<th>Flexural Properties [MPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Strength</td>
<td>Modulus</td>
</tr>
<tr>
<td>35</td>
<td>NFC</td>
<td>0.252 ± 0.007</td>
<td>5.00 ± 0.33</td>
</tr>
<tr>
<td></td>
<td>BPU</td>
<td>0.179 ± 0.007</td>
<td>3.42 ± 0.06</td>
</tr>
<tr>
<td>40</td>
<td>NFC</td>
<td>0.242 ± 0.010</td>
<td>5.72 ± 0.46</td>
</tr>
<tr>
<td></td>
<td>BPU</td>
<td>0.225 ± 0.006</td>
<td>4.74 ± 0.31</td>
</tr>
<tr>
<td>45</td>
<td>NFC</td>
<td>0.282 ± 0.016</td>
<td>6.59 ± 0.11</td>
</tr>
<tr>
<td></td>
<td>BPU</td>
<td>0.234 ± 0.019</td>
<td>5.03 ± 0.51</td>
</tr>
<tr>
<td>50</td>
<td>NFC</td>
<td>0.303 ± 0.021</td>
<td>6.09 ± 0.19</td>
</tr>
<tr>
<td></td>
<td>BPU</td>
<td>0.266 ± 0.017</td>
<td>6.26 ± 0.43</td>
</tr>
</tbody>
</table>

Figure 18 shows a comparison of the compressive and flexural strength (A), and compressive and flexural modulus (B). It is clear that there is no strong relation between the increase in compressive and flexural properties. On the other hand, there is a strong similarity between compressive strength and modulus, and flexural strength and modulus.
Figure 18: Comparison of compressive and flexural strength (A) and modulus (B)

Noteworthy is that the relationship between the properties and increasing density does not have to be linear. In this case, they are only plotted together to see the general trend and the impact of NFC on the foam material.

**Facing strength**

The facing strength of the sandwich composite is seen in Table 11. The results show a large improvement in facing strength of the impregnated Kraft paper with the NFC foam cores.

*Table 11: Facing strength of impregnated Kraft paper*

<table>
<thead>
<tr>
<th>Density [kg/m³]</th>
<th>BPU</th>
<th>NFC</th>
</tr>
</thead>
<tbody>
<tr>
<td>35</td>
<td>6.07 ± 0.96</td>
<td>11.40 ± 0.31</td>
</tr>
<tr>
<td>40</td>
<td>6.42 ± 0.82</td>
<td>8.82 ± 0.67</td>
</tr>
<tr>
<td>45</td>
<td>6.05 ± 0.54</td>
<td>9.27 ± 0.50</td>
</tr>
<tr>
<td>50</td>
<td>8.81 ± 0.64</td>
<td>12.52 ± 0.70</td>
</tr>
</tbody>
</table>
5. Discussion

This section discusses the results and general trends while presenting an alternative evaluation method through material indices by a material selection evaluation process.

The addition of NFC had important effects on the properties of the foam. However, to be able to rely on these effects a proper dispersion of the nanofibers has to be achieved. The diameter of the carrot nanofibers has reportedly been between 3-36 nm with length up to a few μm [8]. Based on the microscopy images of the foam (Figure 14), the fibers found were in bundles of much higher magnitude, up to 100 μm, and thus agglomerates of multiple fiber strands. This suggests that the dispersion is rather poor and the foam properties should not be affected in a significant manner.

Conversely, the main results from the foam properties suggests otherwise. The NFC reinforced foams show a clear decline in cell size and an important increase in compressive properties. Cell size decreased between 18-19% (Figure 16A) while compressive strength and modulus increased up to 41% and 46% respectively (for density group 35, Table 10). These facts suggest the dispersion is rather good and that the NFC has a notable reinforcing effect. Alternatively, it is possible that a side reaction between the OH-groups on the nanocellulose and the isocyanate has taken place (Figure 19). This reaction would create inter penetrating networks which are known to improve mechanical properties [51].

![Chemical formula of cellulose](image)

*Figure 19: Chemical formula of cellulose*

Furthermore the NFC addition shows a small decline in open cell content ranging from insignificant up to a 51% decline, Figure 16B. This, together with decreased cell size suggests the resin has less potential to penetrate into the core, giving a thinner resin penetration and lighter sandwich composite. However, this was not noticeable in the results which instead showed a more random distribution of resin penetration and sandwich density (Figure 16C-D).
The reason why NFC-35 has a much higher density and resin penetration can possibly be attributed to a more sensitive foam with thinner cell walls, in which cells were opened and crushed during cutting. This would have allowed epoxy to enter deeper into the foam during infusion and brought on the irregularities.

Similarly to the compressive properties, the flexural properties were also superior in the NFC reinforced foams. The flexural strength increased in a wide range between 2% (p=40) and 167% (p=35) and the modulus between 3% (p=40) and 163% (p=35). Table 10. Both values are extreme and most probably depend on the resin penetration, not on the open cell content as the differences in these were too insignificant to be noticeable in the resin penetration. Instead, the penetration depended more on the number of closed cells that were cut open during sample preparation, and the size of them as noted in the method section and ISO 4590.

Moreover, the problem with uneven resin penetration, as portrayed by Figure 17, becomes evident in pockets where resin penetration is deep. Here, the stiffness of the epoxy resin causes a brittle fracture of the sandwich in flexural load, which gives a very high strength and modulus yet a quick time to break. Similarly, pockets with very thin resin penetration shows the opposite (low modulus and strength with long time to break or no break at all). This means that the flexural results are very dependent on resin penetration which in turn explains the wide range in flexural properties.

Finally, the facing strength was clearly improved by 37-88 %, Table 11. Looking at the equation for facing strength (Equation 14), the difference must involve the relation between core and skin thickness. The skin thickness is based on the average resin penetration, which in turn is higher for two NFC foams and lower for the other two (Figure 16D) making deductions difficult. Instead, the improved facing strength must be a positive effect of the NFC addition, similarly to the trend with lower cell size, which improved the adhesion between skin and core and enhanced the strength of the interphase.

Hypothetically, a good dispersion of the NFC should give the following effects on the foam:
The actual results were similar to this but with slightly less evident results:

- Smaller cell size
- Higher compressive properties
- **Random** sandwich weight
- **Lower*/unchanged** open cell content
- **Random** resin penetration
- Higher flexural properties

From these results it can be deduced that, even though various irregularities during manufacturing and processing brought on uneven results, the mechanical properties were increased for sandwich panels with NFC reinforced cores.
5.1. Material selection evaluation

A material selection evaluation through merit indices was done in order to get a clear overview of the results on mechanical properties. The indices give a ranking of the foams in terms of best performance. Four different indices were investigated, Table 12, derived in section 2.5. The results are displayed in Table 13. Note that all indices should be minimized for best performance.

Table 12: Summary of the merit indices considered in the evaluation process

| Compressive strength Index: | \( M_1 = \frac{\rho}{\sigma_c} \) |
| Compressive modulus Index: | \( M_2 = \frac{\rho}{E_c^{1/2}} \) |
| Flexural strength Index: | \( M_3 = \frac{\rho}{\sigma_f^{2/3}} \) |
| Flexural modulus Index: | \( M_4 = \frac{\rho}{E_f^{1/2}} \) |

Table 13: Values of material indices for compressive and flexural strength and modulus

<table>
<thead>
<tr>
<th>Density [kg/m³]</th>
<th>M₁</th>
<th>M₂</th>
<th>M₃</th>
<th>M₄</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>BPU</td>
<td>NFC</td>
<td>BPU</td>
<td>NFC</td>
</tr>
<tr>
<td>35</td>
<td>19.6</td>
<td>13.9</td>
<td>125.1</td>
<td>86.0</td>
</tr>
<tr>
<td>40</td>
<td>17.8</td>
<td>16.5</td>
<td>106.6</td>
<td>97.4</td>
</tr>
<tr>
<td>45</td>
<td>19.2</td>
<td>15.9</td>
<td>116.7</td>
<td>97.7</td>
</tr>
<tr>
<td>50</td>
<td>18.8</td>
<td>16.5</td>
<td>98.7</td>
<td>82.5</td>
</tr>
</tbody>
</table>

Noteworthy from the results in Table 13 is that all NFC foams have lower index than their reference foam proving they all have better performance. There is but one exception, \( M_4 \) for density group 45, where the NFC is not higher ranked.

Furthermore, to see if there is a relation between different indices, the change in index from BPU and NFC was calculated and standardized, see example in Equation 17. This yielded four different comparisons, plotted in Figure 20A-D.
Equation 17: Example of standardized $\Delta M$

$$\Delta \overline{M}_n = \frac{M_{n}^{BPU} - M_{n}^{NFC}}{\Delta M_{n}^{max}}$$

Standardized $\Delta M$ comparison ($M_{n}^{BPU} - M_{n}^{NFC}$)

Figure 20: Comparison of material indices
Although there are some irregularities, the graphs show that there is indeed a relation between the indices. This is seen most clearly in Figure 20B where the compressive and flexural strength indices follow the almost exact same path when taking the difference between $M^{\text{BPU}}$ and $M^{\text{NFC}}$.

Finally, the best foam out of these four density groups was $\text{NFC}_{\rho=35}$ which had the overall highest performance (Table 13) and the largest improvement to its reference foam (Figure 18).

As a final material selection evaluation, the manufactured NFC and BPU bio-foams were placed in bubble graphs in reference to commercial foams on the market today. These are petroleum based PU foams of different grades. The bio-foams have very low density with high compressive strength (Figure 21) and the bio-sandwich panels are showing competiveness with the higher density PU foams (Figure 22).
Figure 21: Compressive strength of bio-PU foam in comparison to commercial PU foams

Figure 22: Flexural strength of bio-PU foam in comparison to commercial PU foams
6. Conclusion
A lightweight sandwich panel with carrot NFC-reinforced bio-PU foam core was manufactured in a vacuum infusion process with Kraft paper skin and epoxy resin. The sandwich was tested and compared to a non-reinforced bio-PU foam core sandwich in terms of compressive, flexural and cellular properties. The result was a sandwich panel with improved mechanical properties, smaller cell size structure and lower open cell content. Furthermore, a trend in the behavior of compressive properties of the foam and flexural properties of the sandwich panels could be established thanks to the merit indices. Table 14 shows a summary of the main results. Foam group ρ=35 kg/m³ had the best improvement in properties, partially due to a higher epoxy penetration into the core, followed by ρ=45 kg/m³.

<table>
<thead>
<tr>
<th>Property</th>
<th>NFC influence in foam</th>
<th>ρ=35 kg/m³</th>
<th>ρ=40 kg/m³</th>
<th>ρ=45 kg/m³</th>
<th>ρ=50 kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cell size</td>
<td>- 18 %</td>
<td>- 19 %</td>
<td>- 19 %</td>
<td>- 18 %</td>
<td>- 18 %</td>
</tr>
<tr>
<td>Open cell content</td>
<td>- 51 %</td>
<td>0 %</td>
<td>- 18 %</td>
<td>- 7 %</td>
<td>- 7 %</td>
</tr>
<tr>
<td>Compressive strength</td>
<td>+ 41 %</td>
<td>+ 8 %</td>
<td>+ 21 %</td>
<td>+ 14 %</td>
<td>+ 14 %</td>
</tr>
<tr>
<td>Compressive modulus</td>
<td>+ 46 %</td>
<td>+ 21 %</td>
<td>+ 31 %</td>
<td>0 %</td>
<td>0 %</td>
</tr>
<tr>
<td>Flexural strength</td>
<td>+ 167 %</td>
<td>+ 2 %</td>
<td>+ 72 %</td>
<td>+ 14 %</td>
<td>+ 14 %</td>
</tr>
<tr>
<td>Flexural modulus</td>
<td>+ 163 %</td>
<td>+ 3 %</td>
<td>+ 38 %</td>
<td>+ 21 %</td>
<td>+ 21 %</td>
</tr>
<tr>
<td>Facing strength</td>
<td>+ 88 %</td>
<td>+ 37 %</td>
<td>+ 53 %</td>
<td>+ 42 %</td>
<td>+ 42 %</td>
</tr>
</tbody>
</table>

Unfortunately, proper dispersion of the nanofibers was difficult to achieve which caused incoherent non-homogeneous properties in the foams. Even though the fibers were not well dispersed in the matrix, they were dispersed enough to achieve a notable improvement on mechanical properties. On a final note, dispersion could always be improved to increase the material properties.

There are still a lot of uncertainties during manufacturing to determine the reinforcing factor of the NFC in the sandwich composite. Nevertheless, the results indicate that the mechanical properties of bio-PU foam can be improved by reinforcing the bio-polyol with small amounts
of wet carrot NFC. This work shows a positive trend suggesting NFC-reinforced bio-PU foams has a great potential for use in commercialized products (Figure 23) or structural components in future constructions.

Figure 23: Potential use of material in for example gear knob or steering wheel.
7. Future work

The dispersion of NFC need to be improved for better efficiency and homogeneity in the foam. It could be interesting to look into the grinding of the cellulose and see if polyol could be introduced already here. Excess water can then be removed beforehand while remaining water will be removed by evaporation during the grinding process. This could perhaps give better dispersion of the NFC and the polyol mixture could be used directly in the foam making.

Another important factor to consider is the economical perspective. A thorough analysis, from grinding of NFC to manufacturing of the sandwich panel, has to be made in order to establish if the extra energy required in manufacturing is worth the improvement in mechanical properties.

Finally, other important properties such as fatigue, ageing and stability, are necessary to investigate before NFC should be introduced in PU foam products.
8. List of references


9. Appendix

A. Project Plan

The project consists of 20 weeks, 30 ECTS, beginning on February 1st 2016 and finishing June 17th 2016. The planning is based on three different modules consisting of preliminary work, experimental work and post-experimental work. A detailed view of the project plan can be seen in Table 16 and Figure 24.

Module 1

The preliminary work goes on for five weeks starting February 1st, 2016 at LTU. During this period the planning and organization has to be made as well as a literary study. The first module is finished with a dispersion study of wet CNF in castor oil. There are three milestones during this module; the project plan to be submitted on February 12th, the raw material to be sent to U of T by February 19th and the literary study to be submitted on February 26th.

Module 2

The second module starts on March 7th at U of T and includes the experimental work. The foam core and the sandwich laminate will be manufactured and evaluated for relevant properties. A breakdown of the properties to be evaluated can be seen in the Gantt diagram (Figure 24). Additionally, sample preparation is included in the duration for property evaluation. A milestone for this part is to present a progress of the project including results and conclusions so far. This should be done by April 15th.

The timeline for the experimental part is rather difficult to estimate given the unforeseen difficulties that may occur in both manufacturing and evaluation methods. The manufacturing will take place in an unfamiliar environment to the user as well as some new evaluation methods. To allow for unforeseen difficulties, an additional 5+10 days have been added under the name “eventual retrials”. If this is not needed, the time can instead be used for data analyzing and report writing.

Should it prove difficult to evaluate all desired properties, a ranking system has been made so that the most important properties will be investigated (Table 15). For instance, should the foam
preparation be postponed by one week, the electrical insulation can be left out of the evaluation. Likewise, if there is more time, a few more evaluation methods can be considered at the bottom of the ranking list. The ranking is primarily based on important properties of the foam in order to be vacuum infused successfully (Rank 1). Then follows the VI evaluation (Rank 2-4) and finally some relevant properties for foam in insulation.

Table 15: Ranking of evaluation properties made from most to least important

<table>
<thead>
<tr>
<th>Rank</th>
<th>Property</th>
<th>Rank</th>
<th>Property</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Open &amp; closed cell content</td>
<td>7</td>
<td>Electrical &amp; thermal insulation</td>
</tr>
<tr>
<td>2</td>
<td>Impregnation of skin</td>
<td>8</td>
<td>(Impact toughness)</td>
</tr>
<tr>
<td>3</td>
<td>Bonding skin-to-core</td>
<td>9</td>
<td>(Fire properties)</td>
</tr>
<tr>
<td>4</td>
<td>Flexural properties</td>
<td>10</td>
<td>(Water absorption)</td>
</tr>
<tr>
<td>5</td>
<td>Compressive properties</td>
<td>11</td>
<td>(Acoustic insulation)</td>
</tr>
<tr>
<td>6</td>
<td>3D Tomography</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Module 3
Lastly, the third module concerns the post-experimental work the last five weeks of the project. During this time, the data will be analyzed and the oral and written presentation will be undertaken. There are four milestones for this last module; the first draft on May 23rd, poster presentation at ISBBB Guelph 2016 conference on May 31st, final presentation ready by June 17th and final report ready by June 24th. Noteworthy is that the final presentation probably will be postponed to the next presentation opportunity at LTU, in the beginning of September. Additionally, the poster presentation at the ISBBB conference is not an obligation but rather optional if there is time.
Table 16: List of tasks in the project

<table>
<thead>
<tr>
<th>Module</th>
<th>Task Number</th>
<th>Task Name</th>
<th>Days</th>
<th>Start</th>
<th>Finish</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1</td>
<td>Planning and organization</td>
<td>10</td>
<td>Mon 16-02-01</td>
<td>Fri 16-02-12</td>
<td>LTU</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>Submit project plan</td>
<td>0</td>
<td>Fri 16-02-12</td>
<td>Fri 16-02-12</td>
<td>LTU</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>Send material to U of T</td>
<td>0</td>
<td>Fri 16-02-19</td>
<td>Fri 16-02-19</td>
<td>LTU</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>Literary study</td>
<td>21</td>
<td>Mon 16-02-01</td>
<td>Fri 16-02-26</td>
<td>LTU</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>Submit literary study</td>
<td>0</td>
<td>Fri 16-02-26</td>
<td>Fri 16-02-26</td>
<td>LTU</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>Dispersion study</td>
<td>14</td>
<td>Mon 16-02-15</td>
<td>Thu 16-03-03</td>
<td>LTU</td>
</tr>
<tr>
<td></td>
<td>7</td>
<td>Travel days LTU to U of T</td>
<td>1</td>
<td>Fri 16-03-04</td>
<td>Sun 16-03-06</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>8</td>
<td>Foam preparation</td>
<td>15</td>
<td>Mon 16-03-07</td>
<td>Sun 16-03-27</td>
<td>U of T</td>
</tr>
<tr>
<td></td>
<td>9</td>
<td>Present result progress</td>
<td>0</td>
<td>Fri 16-04-15</td>
<td>Fri 16-04-15</td>
<td>U of T</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>Vacuum infusion</td>
<td>25</td>
<td>Mon 16-03-21</td>
<td>Fri 16-04-22</td>
<td>U of T</td>
</tr>
<tr>
<td></td>
<td>11*</td>
<td>Evaluation of properties</td>
<td>40</td>
<td>Mon 16-03-28</td>
<td>Mon 16-05-23</td>
<td>U of T</td>
</tr>
<tr>
<td>3</td>
<td>12</td>
<td>First draft on report</td>
<td>0</td>
<td>Mon 16-05-23</td>
<td>Mon 16-05-23</td>
<td></td>
</tr>
<tr>
<td></td>
<td>13</td>
<td>Oral presentation</td>
<td>35</td>
<td>Mon 16-05-02</td>
<td>Fri 16-06-17</td>
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</tr>
<tr>
<td></td>
<td>14</td>
<td>ISBBB Guelph poster presentation</td>
<td>0</td>
<td>Tue 16-05-31</td>
<td>Tue 16-05-31</td>
<td>Guelph</td>
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<tr>
<td></td>
<td>15</td>
<td>Final presentation</td>
<td>0</td>
<td>Fri 16-06-17</td>
<td>Fri 16-06-17</td>
<td>LTU</td>
</tr>
<tr>
<td></td>
<td>16</td>
<td>Written report</td>
<td>105</td>
<td>Mon 16-02-01</td>
<td>Fri 16-06-24</td>
<td></td>
</tr>
<tr>
<td></td>
<td>17</td>
<td>Final report</td>
<td>0</td>
<td>Fri 16-06-24</td>
<td>Fri 16-06-24</td>
<td></td>
</tr>
</tbody>
</table>

* See more detailed on which properties will be evaluated in Figure 20

**NB:** Final presentation postponed to September 2\textsuperscript{nd}, and final report due August 31\textsuperscript{st}
Figure 24: Gantt diagram of the project plan

- Task Name
  - Planning and organization
  - Submit plan
  - Send material to U of T
  - Literary study
  - Submit literary study
  - Dispersion study
  - Travel days
  - Foam preparation
  - Present result progress
  - Vacuum infusion
  - Evaluation of properties
    - Open & closed cell
    - Compressive properties
    - Electrical & thermal-insulation
    - 3D Tomography
    - Eventual retrials
      - SEM
      - Impregnation skin/core
      - Flexural properties
      - Eventual retrials
  - First draft on report
  - Oral presentation
  - ISEBB Guelph poster presentation
  - Final presentation
  - Written report
  - Final report
B. Bio-foam formulations

Table 17: Bio-foam formulation

<table>
<thead>
<tr>
<th>Constituent</th>
<th>Short</th>
<th>Parts per hundred polyol [BPU/BPU-NFC]</th>
<th>ρ=35 kg/m³</th>
<th>ρ=40 kg/m³</th>
<th>ρ=45 kg/m³</th>
<th>ρ=50 kg/m³</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resin base</td>
<td>C.O</td>
<td></td>
<td>100</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Reactive pre-polymer</td>
<td>pMDI</td>
<td></td>
<td>167.6</td>
<td>153.8</td>
<td>146.8</td>
<td>139.9</td>
</tr>
<tr>
<td>Blowing agent</td>
<td>H₂O</td>
<td></td>
<td>5.52/0</td>
<td>4.6/0</td>
<td>4.14/0</td>
<td>3.68/0</td>
</tr>
<tr>
<td>Surfactant</td>
<td>S</td>
<td></td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Reaction Catalyst</td>
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<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Gelling Catalyst</td>
<td>G.C</td>
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<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td>Wet NFC</td>
<td>NFC</td>
<td></td>
<td>0/6</td>
<td>0/5</td>
<td>0/4.5</td>
<td>0/4</td>
</tr>
<tr>
<td>Total NFC content</td>
<td></td>
<td></td>
<td>0.17</td>
<td>0.15</td>
<td>0.14</td>
<td>0.13</td>
</tr>
</tbody>
</table>