



Norwegian University of Life Sciences  
Faculty of Environmental Science and  
Technology  
Department of Mathematical Sciences and  
Technology

Master Thesis 2014  
30 credits

# Effects of Oil Content, Pressure and Temperature on Physical and Chemical Properties of Spruce Pellets

Rajiv Balakrishna

**Effects of Oil Content, Pressure and Temperature on  
Physical and Chemical Properties of Spruce Pellets**

**RAJIV BALAKRISHNA**

**DEPARTMENT OF MATHEMATICAL SCIENCES AND  
TECHNOLOGY  
NORWEGIAN UNIVERSITY OF LIFE SCIENCES**

**SPRING 2014**

**Effects of Oil Content, Pressure and Temperature on  
Physical and Chemical Properties of Spruce Pellets**

**RAJIV BALAKRISHNA**

**A THESIS SUBMITTED**

**FOR THE DEGREE OF MASTER OF SCIENCE**

**DEPARTMENT OF MATHEMATICAL SCIENCES AND**

**TECHNOLOGY**

**NORWEGIAN UNIVERSITY OF LIFE SCIENCES**

## **ACKNOWLEDGEMENT**

The author would like to thank the following few people whom, without their help, this thesis would not have been completed successfully.

First and foremost, the author would like to thank and appreciate Professor John Mosbye and Graduate Student Nevena Misljenovic for all their constant guidance, support and encouragement during the course of this Master's Thesis.

The author would also like to express his gratitude towards research staff from the Norwegian University of Life Sciences Department of Mathematical Sciences and Technology, for their continuous technical support and wise advices.

The author would also like to thank Professor Carlos Salas Bringas for his kind assistance and guidance as well..

Last but not least, the author would like to thank his family, for their concern and understanding and his friends, Mr. Pratheesh Prasobhan and Mr Egambaram Arun who helped out in one way or another.

## TABLE OF CONTENTS

ACKNOWLEDGEMENT .....	I
TABLE OF CONTENTS .....	II
SUMMARY .....	V
LIST OF FIGURES .....	VI
LIST OF TABLES .....	VIII
INTRODUCTION.....	<b>Error! Bookmark not defined.</b>
1. Spruce Wood.....	<b>Error! Bookmark not defined.</b>
1.1 Type of Spruce .....	1
1.2 Norway Spruce.....	2
1.3 Uses of Spruce.....	3
1.4 Physical characteristics .....	3
1.5 Chemical Composition of Wood.....	4
2.0 Wood Pellets .....	6
2.1 Different types of Pallets.....	7
2.1.1 Advantages of Pallets .....	8
2.1.2 Disadvantages of Pallets.....	8
2.1.3 Table of comparisons between different Fuels .....	8
2.2 Pallets Production (Industrial).....	9
2.2.1 Energy Consumption by Pellets .....	9
2.2.2 Moisture Content in Pellets .....	9
3.0 Objectives for Paper .....	11
4. Literature Review on Earlier Studies .....	12
4.1 Heating values of wood pellets from different species .....	12
4.2 Torrefaction Influence on Pelletability and Pellet Quality of Norwegian Forest Residues .....	<b>Error! Bookmark not defined.</b>
4.3 The effects of kraft lignin additives on wood fuel pellet quality, energy use and shelf life .....	<b>Error! Bookmark not defined.</b>

4.4 Fuel pellets from biomass: The importance of the pelletizing pressure and its dependency on the processing conditions .....	<b>Error! Bookmark not defined.</b>
4.5 Wood Fuel Handbook .....	<b>Error! Bookmark not defined.</b>
5.0 Experimental Setup & Procedure .....	23
5.1 Introduction .....	23
5.2 Experimental Design .....	23
5.3 Preparation of the wood samples .....	24
5.4 Design Feature for Pellets .....	25
5.5 Production of wood pellets.....	26
5.6 Measurement of Pellet Strength and Compressibility.....	27
5.7 Measurement of Energy Content.....	28
5.8 Measurement of Bulk Density.....	29
5.9 Measurement of Particle Distribution .....	30
5.10 Measurement of Moisture Content.....	31
5.11 Measurement of Ash Content.....	32
5.13 Field Emission Scanning Electron Microscope (FE SEM).....	33
RESULTS AND DISCUSSION .....	34
6.0 Experimental Data – SEM/ FE SEM Images .....	34
6.1 S0 Powder .....	34
6.2 S5 Powder .....	35
6.3 S10 Powder .....	36
7.0 Experimental Data – EDX Analysis.....	37
7.1 EDX of the S0 Powders .....	37
7.2 EDX on the S5 and S10 Powders.....	<b>Error! Bookmark not defined.</b>
7.3 EDX on S0 (Small Fibres) .....	39
7.4 EDX on S10 (Small Fibres) .....	40
8.0 Pellet Strength and Compressibility.....	41
9.0 Pellet Strength and Compressibility .....	42
9.1 S0 at 60C, 120C and 180C .....	42
9.2 S0 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.....	44
9.3 S5 at 60C, 120C and 180C .....	46
9.4 S5 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.....	48
9.5 S10 at 60C, 120C and 180C .....	50
9.6 S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.....	52
9.7 Discussion of S0 vs S5 vs S10 (Strength) .....	54
9.8 Discussion of S0 vs S5 vs S10 (Compressibility) .....	58
9.8 Summary of Strength and Density of Pellets Produced .....	60

10.0 Bulk Density.....	61
11.0 Particle Distribution .....	62
12.0 Moisture Content.....	63
13.0 Water Activity.....	63
13.1 S0.....	63
13.2 S5.....	64
13.3 S10.....	64
13.4 S0 vs S5 vs S10 .....	64
14.0 Ash Content.....	65
15.0 Releasing Force .....	66
15.1 Temperature 60C.....	66
15.2 Temperature 120C.....	67
15.3 Temperature 180C.....	68
16.0 Conclusion.....	69
17.0 Reference.....	70
17.0 Appendix .....	75

## SUMMARY

This work addresses an investigation on and comparison of the physical and chemical properties of Spruce wood powder which is mixed with Oil and compressed in pellets. This work was carried out from January 2014 to May 2014. Variations in wood powder, moisture content, particle size or pre-treatment of powder have been investigated for many years. In recent years however, research has been focused on addition of different additives such as oil and lignin. This is because increasing the efficiency directly translates to saving cost and thus such studies have been primed as a key area for research.

Variation in calorific values, strength and compressibility studies, water activity level were the primary field of interest, since these results are affected by compacting pressure and temperature. Studies were also conducted for the bulk density, ash content, moisture content and SEM and EDX analysis to identify the physical and chemical properties of the product.

Studies indicated that the pellets with higher oil content had larger calorific values due to the addition of oil to it. Additional oil also significantly decreased the strength of the pellets, since additional oil reduced the interfacial forces and capillary pressure.

The water activity also reduced when oil was added to or compacting pressure was increased on the spruce powder.

The bulk density, ash content and moisture content remained significantly unaffected by the change of oil concentration.

Optimal pressure and temperature for the three pellets lied in the region of 150 MPa and 120°C, since any addition of pressure or temperature no longer changes the physical and chemical properties significantly. Studies were also done to investigate the quantity of oil lost during the mixing and the pelleting process to ensure that the correct level of oil is being investigated.



## LIST OF FIGURES

Figure 1a (left) and b (right): Cross section of a Spruce trunk and Names of different parts in a Spruce trunk .....	1
Figure 1-2 a (left) and b (right): Norway Spruce tree and European region where Norway Spruce tree is commonly found.....	2
Figure 2-1: Energy Consumption Scenario compared with energy potential of bio mass.....	6
Figure 2-3a: Diagram of Wood Pellets .....	7
Figure 2-3b: Diagram Spruce Wood Pellets .....	7
Figure 4-1-1 The Higher and Lower Heating Values of different type of trees in Portugal....	12
Figure 4-1-2 The average Higher or Lower Heating Values of Hardwoods and Softwoods...	13
Figure 4-2-1 Figure for HHV, moisture content and water activity for both raw and torrefied spruce; and birch .....	14
Figure 4-2-2 Strength and Density Graph against Pellets Compacting Pressure.....	14
Figure 4-2-2 Discharging Force for the Pellets.....	15
Figure 4-3-1 Durability vs wt% of kraft lignin .....	16
Figure 4-4-1 a) Compacting Pressure vs Compression Ratio (Left) and b) Compacting Pressure vs Temperature (Right).....	17
Figure 4-4-2 a) Compacting Pressure vs Moisture Content (Left) and b) Compacting Pressure vs Compression Ratio as a function of particle size (Right).....	17
Figure 4-4-2 Pellet Density vs Compacting Pressure.....	18
Figure 4-5-1 Figure of moisture and bulk density for different wood .....	19
Figure 4-5-2 Figure of Net Calorific Value and Ash Content in Wood.....	20
Figure 4-5-3 Chemical composition of elements in wood samples .....	21
Figure 4-5-4 Influence that moisture Content has on energy stored in the pellets.....	22
Figure 5-2: : P 1 - 6 – Diosna Mixer .....	24
Figure 5-3: Spruce Powder, 0% Oil, 11% Moisture .....	24
Figure 5-6: Lloyd LR5K (Left), and Diametral tests of feed pellets* (Right).....	27
Figure 5-7: Parr© Oxygen Bomb Setup (Left) and Drawing of Internal Components of Oxygen Bomb .....	28
Figure 5-8: Bulk Density Measurement Apparatus.....	29
Figure 5-9: Malvern Mastersizer 3000.....	30
Figure 5-10: Mettler Moisture Analyser .....	31
Figure 5-11: Nabertherm Oven .....	32
Figure 6-1 a (left) and b (right): SEM images of S0 Powders .....	34
Figure 6-2 a (left) and b (right): SEM images of B5 Powders .....	35
Figure 6-3 a (left) and b (right): SEM images of S10 Powders .....	36
Figure 7-1: EDX imaging for B0 Powder .....	37
Figure 7-2: EDX imaging for B5 and B10 Powders .....	38
Figure 7-3: EDX imaging for B0 Powders (Small Fibres).....	39
Figure 7-4: EDX imaging for B0 Powders (Small Fibres).....	40
Figure 8-1: Strength vs Density Curve.....	41
Figure 9-1a: Graph of Strength vs Pressure for S0 at 60C, 120C and 180C.....	42
Figure 9-1b: Graph of Compressibility vs Pressure for S0 at 60C, 120C and 180C. ....	43
Figure 9-2a: Graph of Strength vs Temperature for S0 at 75 MPa, 150 MPa, 225 MPa and 300 MPa. ....	44

Figure 9-2b: Graph of Compressibility vs Temperature for S0 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.....	45
Figure 9-3a: Graph of Strength vs Pressure for S5 at 60C, 120C and 180C.....	46
Figure 9-3b: Graph of Compressibility vs Pressure for S5 at 60C, 120C and 180C .....	47
Figure 9-4a: Graph of Strength vs Temperature for S5 at 75 MPa, 150 MPa, 225 MPa and 300 MPa. ....	48
Figure 9-4a: Graph of Compressibility vs Temperature for S5 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.....	49
Figure 9-5a: Graph of Strength vs Pressure for S10 at 60C, 120C and 180C.....	50
Figure 9-5b: Graph of Compressibility vs Pressure for S10 at 60C, 120C and 180C. ....	51
Figure 9-6a: Graph of Strength vs Temperature for S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa. ....	52
Figure 9-6b: Graph of Compressibility vs Temperature for S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa. ....	53
Figure 9-7a: Graph of Strength vs Pressure for S0, S5, S10 at 60C, 120C and 180C. ....	54
Figure 9-7b: Graph of Strength vs Temperature for S0, S5, S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa. ....	54
Figure 9-8a: Graph of Compressibility vs Pressure for S0, S5, S10 at 60C, 120C and 180C. ....	58
Figure 9-8b: Graph of Compressibility vs Temperature for S0, S5, S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa. ....	58
Figure 11-1: Figure of Particle Size Distribution for Powders. ....	62
Figure 15-1: Graph for Releasing Force for B0, B5 and B10 Pellets at 60C.....	66
Figure 15-2: Graph for Releasing Force for B0, B5 and B10 Pellets at 120C.....	67
Figure 15-3: Graph for Releasing Force for B0, B5 and B10 Pellets at 180C.....	68

## LIST OF TABLES

Table 1-4: Table for Physical characteristics for Spruce Wood.....	3
Table 1-5: Table for different composition make up of wood .....	4
Table 2-1-3: Table of comparisons between different Fuels [52, 53] .....	8
Table 10-1: Table for B0, B5 and S10 Powder Bulk Density.....	61
Table 13-1: Table for Water Activity in B0.....	63
Table 13-2: Table for Water Activity in B5.....	64
Table 13-3: Table for Water Activity in S10. ....	64
Table14-1: Table for Ash Content. ....	65

## **INTRODUCTION**

### **1. Spruce Wood**

The spruce wood originates from the coniferous evergreen spruce tree which comes from the Pinaceae family and the Picea genus. There are about 35 species in this genus which is mostly found in the northern regions in places such as Europe and North America [1, 2, 3]. They are large and fast growing trees that grow on average to 30 – 60 meters. These trees when fully grown also can have an average trunk diameter of 1 - 1.5 meters [1, 6]. They are made up of needle like leaves that are attached in a spiral manner to the branch to ensure maximum ecological efficiency from the tree during the winter months [1, 2, 3]. The xylem or heartwood in the tree trunk is pale in colour thus the spruce is also commonly referred to as white wood [9, 10].



Figure 1a (left) and b (right): Cross section of a Spruce trunk and Names of different parts in a Spruce trunk  
www.tradebit.com and www.aginclassroom.org-300

#### **1.1 Type of Spruce**

There are over 38 named species of Spruce. Their different species are named as the following [11]:

*Picea abies*, *Picea alcoquiana* , *Picea alpestris*, *Picea asperata*, *Picea brachytyla*, *Picea breweriana*, *Picea chihuahuana*, *Picea crassifolia*, *Picea engelmannii*, *Picea farreri*, *Picea glauca* var. *glauca*, *Picea glauca* var. *densata*, *Picea glauca* var. *albertiana*, *Picea glauca* var. *porsildii*, *Picea glehnii*, *Picea jezoensis*, *Picea koraiensis*, *Picea koyamae*, *Picea likiangensis*, *Picea mariana*, *Picea martinezii*, *Picea maximowiczii*, *Picea meyeri*, *Picea morrisonicola*, *Picea neveitchii*, *Picea obovata*, *Picea omorika*, *Picea orientalis*, *Picea pungens*, *Picea purpurea*, *Picea retroflexa*, *Picea rubens*, *Picea schrenkiana*, *Picea sitchensis*, *Picea smithiana*, *Picea spinulosa*, *Picea torano* and *Picea wilsonii* .

However the most commonly known species of Spruce in the Scandinavia and Northern Europe is the *Picea abies* which is also known as Norway Spruce.

### **1.2 Norway Spruce**

The *Picea abies* (Norway spruce or also commonly known as European spruce) is a type of spruce commonly found in Europe. It can grow rapidly in the first 25 years of its life to up to 20 meters before slowing down. These type of species are spread across Europe from Norway all the way across the Siberian desert through the Ural Mountains, while also spreading downwards to the European Alps [1, 2, 3, 11].



Figure 1-2 a (left) and b (right): Norway Spruce tree and European region where Norway Spruce tree is commonly found  
<http://en.wikipedia.org>

### **1.3 Uses of Spruce**

Spruce wood is commonly also called timber which primarily is used as a building wood. It is used for general purpose in the construction industry, used to making crates and used making musical instruments. These woods can also be used for a more specialized purpose such as to make indoor wall (Spruce has minimal insect and decay resistance and thus cannot be used outdoor as often).

It is also commonly used to make paper as its fibres are long and thus is able to bind together better, creating a better quality paper. These woods also can be easier bleached (Making the paper whiter) and thus used in mechanical pulping due to its excessive paleness in the heartwood.

Spruces' fresh shoot is a natural source of vitamin C and the essential oils found in its branches and needles has also been used to brew spruce beer. The spruce leaves are also an excellent source of water during survival situation.

But most importantly Spruce wood is commonly used to make pallets as it can be easily grinded down to its powder forms or it can be easily found as sawdust by-product in the manufacturing industry. Due to its abundance and relative easy growth Spruce wood is preferred for making pellets.

### **1.4 Physical characteristics**

Density (at 12 % moisture content)	441 kg/m <sup>3</sup>
Total longitudinal shrinkage	0.3 %
Total radial shrinkage	3.6 %
Total tangential shrinkage	7.8 %
Equilibrium moisture content	
(20° C/37 % rel. humidity)	7.0 %
(20° C/83 % rel. humidity)	16.4 %

Table 1-4: Table for Physical characteristics for Spruce Wood  
<http://jp.europeanwood.org/en/living-with-wood/selected-european-wood-species/spruce/>

### **1.5 Chemical Composition of Wood**

Wood is made up of cellulose, hemicelluloses, lignin, and extractives. These components can vary in different quantity in different type of species of wood. This in turn affects the physical and chemical properties of the wood as the fiber properties are altered if either if this four the compositions change [58].

Constituent	Scots Pine ( <i>Pinus sylvestris</i> )	Spruce ( <i>Picea glauca</i> )	Eucalyptus ( <i>Eucalyptus camaldulensis</i> )	Silver Birch ( <i>Betula verrucosa</i> )
Cellulose (%)	40	39.5	45.0	41.0
Hemicellulose				
-Glucomannan (%)	16.0	17.2	3.1	2.3
-Glucuronoxytan (%)	8.9	10.4	14.1	27.5
-Other polysaccharides (%)	3.6	3.0	2.0	2.6
Lignin (%)	27.7	27.5	31.3	22.0
Total extractives (%)	3.5	2.1	2.8	3.0

Table 1-5: Table for different composition make up of wood  
<http://www.ipst.gatech.edu>

Cellulose is the main compound found in wood contributing more than 40%-50% of the wood dry weight. Its chemical composition promotes intra and inter molecular bonding between molecules. It is made up of linear chains of D-glucose linked by  $\beta$ -1,4-glycosidic bonds which are mostly found on cell walls [58].

Hemicellulose has a random amorphous structure with little strength. Hemicelluloses are polysaccharides in plant cell walls that have  $\beta$ -1,4-linked backbones with an equatorial configuration. Hemicelluloses mainly include xylan, glucuronoxytan, arabinoxytan, glucomannan, and xyloglucan [58].

While lignin is a complex polymer of aromatic alcohols which are critical in cell wall in plants. Lignin is known to be fibrous, tasteless and insoluble in water and alcohol. Lignins are very useful and it can be used as an emulsifying, sequestering, binding, or dispersal agent. It also has a very higher burning efficiency[58]. In fact at its glass transition temperature lignin acts as a very efficient binder as it is able to bind two fibers together very efficiently once it cools down.



## 2.0 Wood Pellets

Pellet fuels are the new age renewable environmental friendly solution to energy production. It is currently used in over a million homes across the Americas and Europe. It is made up of renewable compressed biomass mainly wood. Wood used for pelletizing comes mainly from recycled wood waste. These wood pellets are made from the compacting sawdust or waste from industrial wood manufacturing [12, 13, 53, 60]. Wood biomass is also derived from coconut and palm kernel shell, unutilized branches from logging and also tree logging for pellet production. Pellets manufactured can be used for various functions, such as to fuel a power plant for electricity and heat up homes. Stoves, fireplace, boilers and industrial furnaces can be used to combust these pellets.

Pellets are produced under high pressure and low moisture thus is extremely dense and highly efficient when it is combusted. Moisture content (< 10 %) has to be kept low to ensure that the pellets are burned efficiently since higher moisture content reduces the calorific value significantly [43, 50, 53, 62]. The highly dense and compact pellets allow better transportation and better storage of the wood [12, 13, 53, 62].

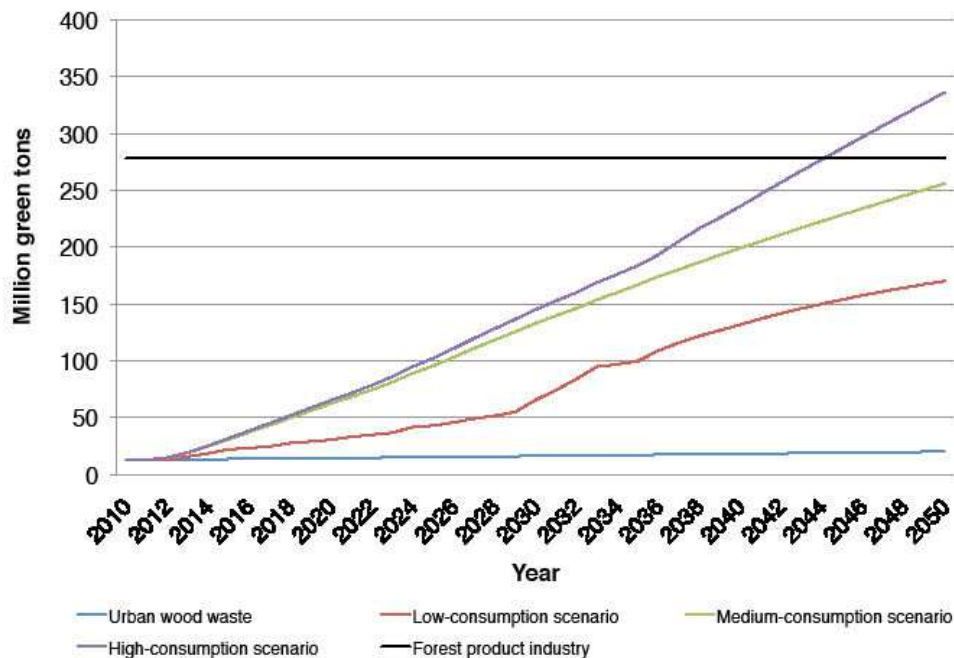


Figure 2-1: Energy Consumption Scenario compared with energy potential of bio mass  
\*Wood Pellet Handbook

The increase in price for fossil fuels has promoted the usage and increased the demand for pellets in the West. In 2010 pellets production accounted to 14 million tons which was double of what was produces in 2006 [5, 12, 13, 53, 62]. Figure 2-1 shows the increasing trend of energy consumption in the next 40 years with the forest product industry only able to co-support the world energy consumption only until 2042 if the high consumption scenario is taken into consideration. Pellet production is expected to double again in the next five years indicating a huge potential of research and new discoveries in this field. Sweden itself accounted for 1.9 million tons pellet produced which translate to 9TWh of energy [5, 12, 13, 53, 62, 63].

## **2.1 Different types of Pellets**

Pellets production is not only confined to wood powders. Non woody biomasses can be also used for the production of pellets. Distiller's dried grains can be used as a substitute to lignin to provide increased durability [62, 63]. Grass and rice-husk are also commonly used in fuel-pellets production. Grass when used as a feedstock is advantageous since it has s short growing time of 70 days and is easily cultivated. It also has about 96% of the wood powder energy content [62, 63].Rice husk pellets are made similarly from the by-products of rice cultivation and thus more environmental friendly than the other two pellets. They produce about 4.2 kcal/kg of energy, while wood pellets produce about 4.8 kcal/kg of energy [62, 63]. However rice husk pellets production is limited to countries which predominantly use rice has predominant food-crop.



Figure 2-3a: Diagram of Wood Pellets  
\* <http://www.pelletmillequipment.com>



Figure 2-3b: Diagram Spruce Wood Pellets

### **2.1.1 Advantages of Pallets**

Wood pellets ensure that the carbon usage net effect is neutral, since we are burning the organic carbon from the wood which can be replaced with proper management of woodlands. This is however not possible when we burn fossil fuels. Wood pellets also burn relatively cleaning releasing lesser environmental unfriendly by product during complete combustion. Combustion of wood pellets in the power plant also allows a constant amount of energy to be produced as the pellets with has similar energy content is being transport via a conveyer belt to be burned. Pellets are also less expensive than most fossil fuels.

### **2.1.2 Disadvantages of Pallets**

Wood pellets production itself will not be able to sustain the world energy and we will need to supplement it with other sources of renewable energy. Proper management of woodland is essential for palletisation, if not the environment will be badly damaged by greed.

### **2.1.3 Table of comparisons between different Fuels**

Commodity (% Moisture Content)	pence/ltr	£/tonne	Net Calorific Value		pence/kWh (Input)
			kWh/T	kWh/ltr	
<b>Electricity via Fossil</b>					12.00
<b>LPG</b>	55			6.6	9.80
<b>Heating Oil</b>	70			10.3	8.00
<b>Heating Oil</b>	60			10.3	6.85
<b>Wood Pellet Bagged</b>		250	4800		6.13
<b>Firewood (30%)</b>		150	3500		5.04
<b>Wood Pellet (10%)</b>		200	4800		4.90

Table 2-1-3: Table of comparisons between different Fuels [52, 53]

Wood pellets have the lowest input for the amount spend (4.90p/kWh) to produce 1 kWh of energy, while fossil fuel has the highest input for the amount spend (12.00/kWh) to produce 1 kWh of energy.

## **2.2 Pellets Production (Industrial)**

Wood powders are passed through hammer mill to ensure uniformity in the mass. These mass is then send to a press where it is squeezed through a die chamber with multiple holes and cut off to their respective sizes [48, 52, 53]. The typical size of a pellet varies from 6-8 mm in diameter. The high pressure in the chamber allows the high temperature to be transferred rapidly to the powders. The lignin in the powders thus plasticizes, thus acting as a natural organic binder that holds the pellets together as they cool down [48, 52, 53].

Pellets made in Europe normally conforms to the DIN 51731 or Ö-Norm M-7135 standards, with less that 10% moisture content, uniform high density (ca.  $\approx 1 \text{ ton/m}^3$ ), good strength and low ash content [5, 12, 13, 53, 62, 63]. Wood fibres from different feed stock are well grinded in the hammer mill and pressed with different pressure, thus resulting in minimal difference in the finished pellets [48, 52, 53]. Norway, Sweden, Denmark and Finland are the main production centres for wood pellets production [5, 12, 13, 53, 62, 63]

### **2.2.1 Energy Consumption by Pellets**

Energy used to dry, pelletize and transport wood pellet made from industrial waste wood (pre-dried) makes up to about 11 % of energy content form the pellets [48, 52, 53]. While pellets made directly from logged wood uses up 26 % of energy content from the pellets [48, 52, 53]. Transportation of pellets across continents can add another 10-14 % to the total energy consumption content of the wood pellets [48, 52, 53].

### **2.2.2 Moisture Content in Pellets**

Pellets that have high moisture content produces more steam reducing energy content within it. Burning wet pellets causes tar build up, environment pollution and increases the risk of fire in the plant [43, 50, 57]. Therefore it is very important to dry and season the feedstock well before it is sent for hammer milling. Wood logged in winter can be dried and seasoned for a year, while wood logged in the summer has higher moisture content and needs to be season for two years before being milled and eventually burned [43, 50, 57]. While being seasoned the wood should

be stored in an open, dry and airy location. Ideally wood can be sent to the hammer mill once the moisture content is below 25% before it is further processed [43, 50, 57].

Hard wood (heavier wood) which is denser will thus have a higher calorific value than softwood [12,13,52] . Heavier wood is more difficult to burn as opposed to soft wood. Therefore it is common to burn soft wood with hardwood. However this makes very minimal difference if the wood is hammer milled and grinded to similar particle size [12,13,52].

### **3.0 Objectives for Paper**

The present work was focused on finding the physical and chemical characteristics of spruce wood pellets with added oil content. Three different temperatures and four different pressures were used to examine these characteristics.

Different studies have been conducted with varying lignin content, moisture content, wood quality, particle size and etc. to investigate its influence on the physical and chemical properties of the pellets. However addition of oil has to possibly increase the strength or the calorific values has not been readily studied upon. Therefore these paper aims to fulfil and bridge the gap in knowledge in this specialized area.

These were carried out at the materials laboratories of the Department of Mathematical Sciences and Technology at the Norwegian University of Life Sciences, with the following objectives:

- To understand the how the addition of different quantity of used vegetable oil influence the physical and chemical properties of the pellets.
- To understand the how varying the compacting pressure influences the physical and chemical properties of the pellets.
- To understand the how varying the compacting temperature influences the physical and chemical properties of the pellets.
- To identify the optimum level of compacting pressure and temperature for a given oil content needed to maximize the physical properties such as the strength and the chemical properties such as the calorific values.

## **4. Literature Review on Earlier Studies**

### **4.1 Heating values of wood pellets from different species[51].**

The higher heating values (HHV) for from trees of both softwood and hardwood were measured in Portugal where 38% of its land is covered by forest. These areas are occupied mainly by *Quercus suber*, *Pinus pinaster* and *Eucalyptus globulus*. The *Pinus pinaster* and the *Pseudotsuga menziesii* come Pinaceae family which is similar to *Picea abies*. Portugal is the fifth biggest producer of pulp and paper in Europe. Therefore it has huge potential to generate by products for the production of pellets to be used as energy. The HHV values are described in the figures below.

Mean  $\pm$  SD (CV %) of Higher and Low Heating values, moisture content ( $M_w$ ) of the samples.

Sample	Species	HHV (kJ/kg)	LHV (kJ/kg)	$M_w$ (%)
P4	<i>Pinus pinaster</i>	20237.89 $\pm$ 374.12 (1.849)	16935.72 $\pm$ 335.46 (1.981)	10.3
P8	<i>Pseudotsuga menziesii</i>	19660.02 $\pm$ 32.29 (0.164)	16704.30 $\pm$ 29.46 (0.176)	8.8
P12	<i>Cedrus atlantica</i>	20360.45 $\pm$ 187.30 (0.920)	15629.71 $\pm$ 154.00 (0.985)	17.8
P1	<i>Castanea sativa</i>	18754.86 $\pm$ 218.64 (1.166)	15468.56 $\pm$ 194.33 (1.256)	11.1
P2	<i>Eucalyptus globulus</i>	17631.66 $\pm$ 326.53 (1.852)	14411.54 $\pm$ 289.03 (2.006)	11.5
P3	<i>Fagus sylvatica</i>	19132.47 $\pm$ 231.97 (1.212)	15818.67 $\pm$ 206.36 (1.304)	11.0
P5	<i>Quercus robur</i>	18696.82 $\pm$ 47.00 (0.251)	15361.13 $\pm$ 41.63 (0.271)	11.4
P6	<i>Fraxinus angustifolia</i>	19090.90 $\pm$ 306.18 (1.604)	16450.82 $\pm$ 283.92 (1.726)	7.3
P7	<i>Prunus avium</i>	18256.48 $\pm$ 120.86 (0.662)	15552.33 $\pm$ 111.18 (0.715)	8.0
P9	<i>Salix babylonica</i>	18279.41 $\pm$ 348.08 (1.904)	15372.32 $\pm$ 316.07 (2.056)	9.2
P10	<i>Populus euro-americana.</i>	18791.20 $\pm$ 248.45 (1.322)	16130.08 $\pm$ 229.78 (1.424)	7.5
P11	<i>Acer pseudoplatanus</i>	18637.91 $\pm$ 152.15 (0.816)	15615.05 $\pm$ 137.43 (0.880)	9.7
P13	<i>Chlorophora excelsa</i>	20314.74 $\pm$ 378.88 (1.865)	17287.67 $\pm$ 345.38 (1.998)	8.8
P14	<i>Entandrophragma cylif.</i>	19053.87 $\pm$ 113.65 (0.596)	15691.61 $\pm$ 100.73 (0.642)	11.4
P15	<i>Gossweilerodendron b.</i>	20499.80 $\pm$ 338.74 (1.652)	17170.12 $\pm$ 303.73 (1.769)	10.3
P16	<i>Bowdichia nitida</i>	20809.47 $\pm$ 354.05 (1.701)	17907.85 $\pm$ 325.83 (1.819)	8.0
P17	<i>Hymenaea courbaril</i>	19296.38 $\pm$ 187.61 (0.972)	16183.69 $\pm$ 169.18 (1.045)	9.8

Figure 4-1-1 The Higher and Lower Heating Values of different type of trees in Portugal

Mean, SD in brackets, Tukey–Kramer test of calorific values and moisture contents (Mar) of wood.

Species	HHV (kJ/kg)	LHV (kJ/kg)	Mar (%)
Hardwoods (H)	19089.0 a	16030.1 a	9.6 a
	(889.9)	(919.6)	(1.5)
Softwoods (S)	20086.1 a	16423.2 a	12.3 a
	(374.1)	(696.9)	(4.8)
Hardwoods National (HN)	18585.7 a	15575.6 a	9.6 a
	(468.0)	(568.8)	(1.7)
Softwoods National (SN)	20086.1 b	16423.2 a	12.3 a
	(374.1)	(696.9)	(4.8)
Hardwoods National (HN)	18585.7 a	15575.6 a	9.6 a
	(468.0)	(568.8)	(1.7)
Hardwoods Tropical (HT)	19994.9 b	16848.2 b	9.7 a
	(773.7)	(894.3)	(1.3)

a: There are statistically no significant differences. b: There are statistically significant differences.

Fig 4-1-2 The average Higher or Lower Heating Values of Hardwoods and Softwoods

The figures above, shows the different calorific values of wood pellets from different species. Softwoods had HHV ranging from 19661 to 20361 kJ/kg. While, hardwoods had HHV ranging from 17631.66 to 20809.47 kJ/kg.

Pinus pinaster and the Pseudotsuga menziesi had a HHV of 20238 kJ/kg and 19660 kJ/kg respectively

Softwood trees generally has a higher heating value than hardwoods. However the only statistical differences were found in the case of the HHV for National wood.



#### 4.2 Torrefaction Influence on Pelletability and Pellet Quality of Norwegian Forest Residues [17].

The quality of the pellets for spruce and birch wood was measure while the torrefaction temperature used as a varying input. The wood were torrefied both 225 and 275 °C for 30 min before the higher heating value (HHV), moisture content, water activity and particle size distribution was examined. Pellets also was produced at four different temperature of 60, 120, and 180 °C; and at eight different compacting pressures 10, 20, 40, 80, 160, 240, and 300 MPa. Figure 4-2-1 shows these characteristics of the pellets produced.

	RS	TS225	TS275	RB	TB225	TB275
torrefaction temperature (°C)		225	275		225	275
residence time (min)	30	30	30	30	30	30
moisture content (%)	10.30 ± 0.06	7.98 ± 0.13	5.27 ± 0.01	9.74 ± 0.11	7.58 ± 0.10	5.34 ± 0.01
$a_w$	0.695 ± 0.002	0.698 ± 0.001	0.691 ± 0.005	0.705 ± 0.007	0.691 ± 0.003	0.687 ± 0.005
HHV (MJ/kg <sub>db</sub> <sup>a</sup> )	19.93 ± 0.28 a	20.27 ± 0.22 a,b	21.9 ± 0.66 c	19.14 ± 0.23 a	19.7 ± 0.37 a	21.33 ± 0.71 b,c
WL (% <sub>db</sub> <sup>a</sup> )		3.19	16.92		3.53	24.59
EL (%)		1.54	8.71		0.71	15.97
HHV <sub>i</sub> /HHV <sub>t</sub>	1	0.98	0.91	1	0.97	0.90

<sup>a</sup>db = dry basis. <sup>b</sup>Different letters indicate significant differences ( $p < 0.05$ ) for HHV according to post-hoc Tukey's test.

Fig 4-2-1 Figure for HHV, moisture content and water activity for both raw and torrefied spruce; and birch

Pure spruce had a moisture content of 10.3%, water activity level of 0.695 and a HHV of 19.93 MJ/kg. While the torrefied samples showed an increase in HHV values and also a decrease in moisture content.

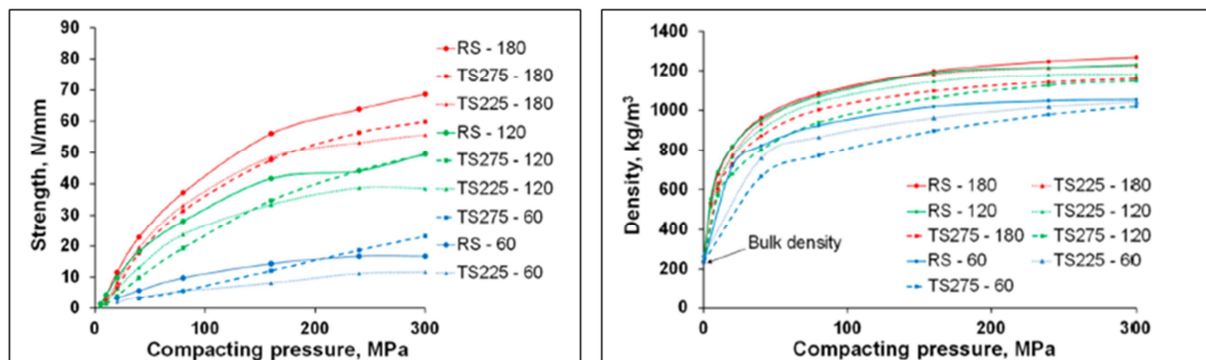


Fig 4-2-2 Strength and Density Graph against Pellets Compacting Pressure

The pure Spruce shows a increase in strength and density as the compacting pressure increases. However the increase tends to even out as the pressure gets higher. The maximum density is also almost reached after 275 MPa. The initial bulk densities for all the powders are  $\approx 200 \text{ kg/m}^3$ .

Figure 4-2-1 shows discharging force used to remove the pellets from the die after production.

	$P_{max}/length$ (MPa/mm)					
	RS	TS225	TS275	RB	TB225	TB275
Pellets Produced at 40 MPa						
60 °C	0.224 ± 0.021	0.259 ± 0.004	0.591 ± 0.016	0.147 ± 0.008	0.311 ± 0.0358	0.636 ± 0.0244
120 °C	0.125 ± 0.008	0.118 ± 0.011	0.494 ± 0.015	0.090 ± 0.005	0.225 ± 0.014	0.613 ± 0.0226
180 °C	0.077 ± 0.004	0.080 ± 0.006	0.305 ± 0.011	0.081 ± 0.001	0.163 ± 0.0198	0.517 ± 0.0215
Pellets Produced at 240 MPa						
60 °C	0.516 ± 0.036	0.789 ± 0.018	3.039 ± 0.141	0.314 ± 0.012	0.648 ± 0.034	2.549 ± 0.042
120 °C	0.174 ± 0.007	0.261 ± 0.007	1.338 ± 0.071	0.186 ± 0.032	0.430 ± 0.019	2.064 ± 0.145
180 °C	0.187 ± 0.037	0.188 ± 0.015	0.713 ± 0.017	0.142 ± 0.001	0.363 ± 0.030	1.633 ± 0.126

Fig 4-2-2 Discharging Force for the Pellets

The discharging force used to remove the pure spruce for pellets produced at 40 MPa ranges from 0.224 to 0.007 depending on the temperature. However it is clearly observed that increasing temperature allows the pellets to be removed easily from the die. A similar observation is seen for pellets produced at 240 MPa as the discharging force ranges from 0.516 to 0.187 here.

### **4.3 The effects of kraft lignin additives on wood fuel pellet quality, energy use and shelf life [46].**

Pellets are an important source for biomass energy production as it is used to supplement the other sources of energy production. 9 TWh of pellets energy was consumed in Sweden itself in 2011. Therefore research in kraft lignin is an important as it might be able to increase the efficiency and energy output of pellets being produced.

Kraft lignin was added to the pellets ranging from 1 to 4% and results indicated that it increase it mechanical durability and their lengths. However the energy content of the pellets remained unchanged due to this addition.

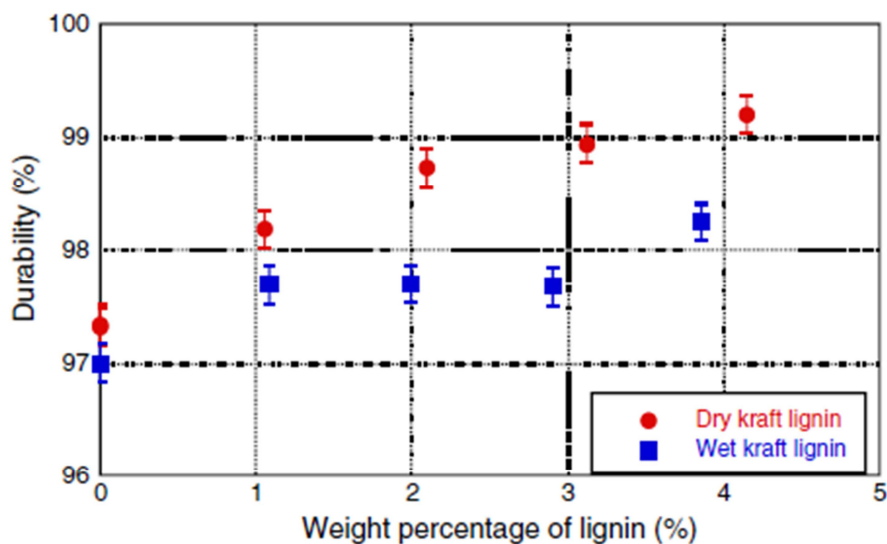


Fig 4-3-1 Durability vs wt% of kraft lignin

The dry kraft lignin also exhibited a larger durability as opposed wet kraft lignin. Fig 4-3-1 clearly illustrate that when 0% dry kraft lignin is used the mechanical durability is 97.2% while when 4% dry kraft lignin is used the mechanical durability rises to 99.2%.

#### 4.4 Fuel pellets from biomass: The importance of the pelletizing pressure and its dependency on the processing conditions [30].

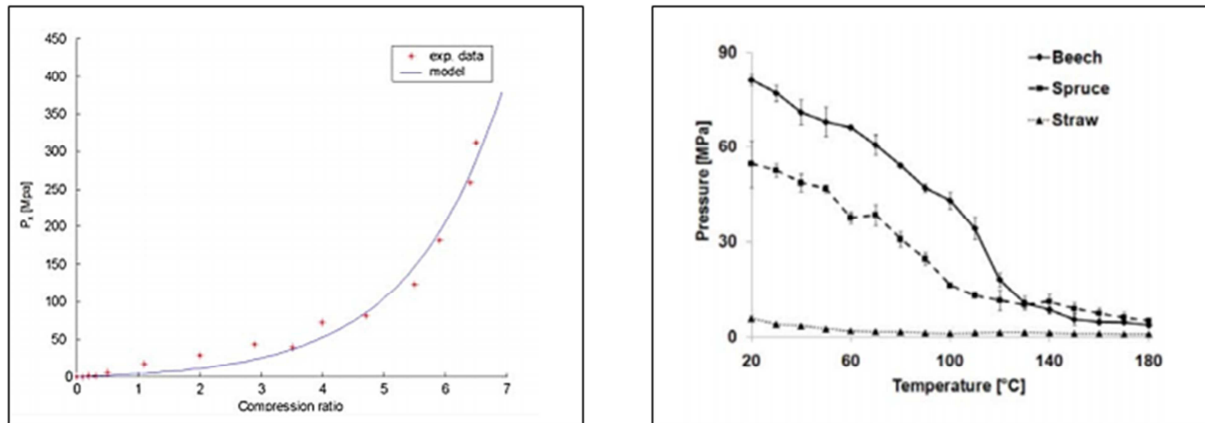


Fig 4-4-1 a) Compacting Pressure vs Compression Ratio (Left) and b) Compacting Pressure vs Temperature (Right)

This paper clearly indicates the relationship between the compacting pressure and its relationship to the processing condition used to make the pellets. It was also identified that an increase in temperature resulted in lesser compacting pressure used to achieve similar compression ratio of the pellets (Fig 4-4-1 b). The increase in length in the pellet also increased the compacting pressure exponentially and is dependent on the wood biomass species, temperature, moisture content and particle size (Fig 4-4-1 c).

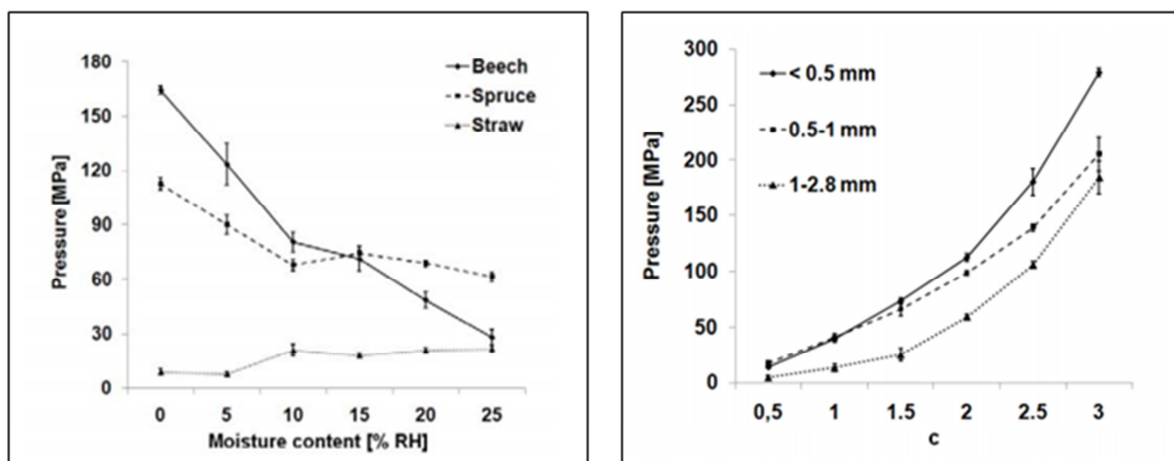


Fig 4-4-2 a) Compacting Pressure vs Moisture Content (Left) and b) Compacting Pressure vs Compression Ratio as a function of particle size (Right)

Figure 4-4-2 a also shows that the moisture content affects the compacting pressure of the wood as an increase in moisture contents correlates to a decrease in compacting pressure for the pellets. While Figure 4-4-2 b indicates that an increase in particle distribution size actually will decrease the compacting pressure required.

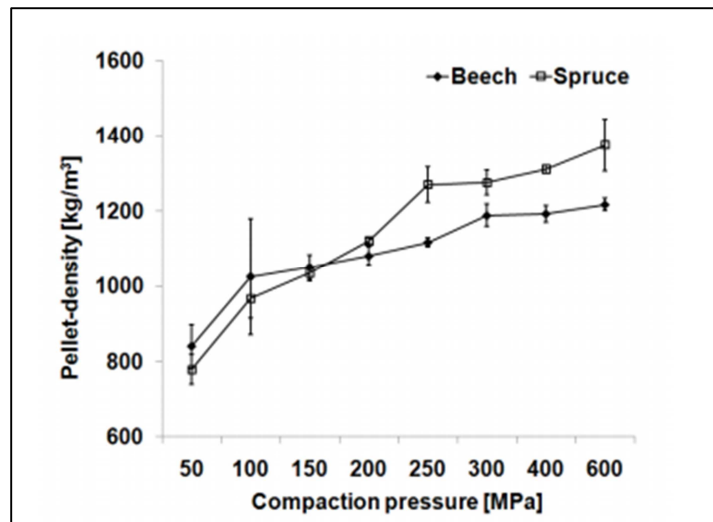


Fig 4-4-2 Pellet Density vs Compacting Pressure

Figure 4-4-2 show that an increase of compacting pressure is required to compress the wood fibres to give a higher final density. However there is also a maximum density which the pellets can attain where there is virtually no more empty space where the pellet can be compressed any further.

#### **4.5 Wood Fuel Handbook [53]**

Biomass is one of the most important renewable source of energy due to its high conversion efficiencies, competitiveness and sustainability. Biomasses can be used from small scale heating up to heating an industry. There are many physically and chemical properties with respect to wood pelleting which will be discussed with details below.

Wood fuels	M %	Specie	Bulk density (kg/bulked m <sup>3</sup> )
Log woods (33 cm piled)	15	Beech	445*
		Spruce and fir	304*
Wood chips	30	Beech	328
		Spruce and fir	223
Conifers' bark	15		180
Saw dusts			160
Shavings			90
Pellets	8		620-650
<b>Agriculture biomass</b>			
Bales	15	Miscanthus	140
Hog biomass		Miscanthus	110
Grain		Triticale	750

\* kg/stacked m<sup>3</sup>

Fig 4-5-1 Figure of moisture and bulk density for different wood

Figure 4-5-1 shows the typical industrial optimized moisture content for a wood pellet of 8% wt and its bulk density of 650 kg/m<sup>3</sup>. The Spruce wood chip has 30% moisture content and a bulk density of 223 kg/m<sup>3</sup>.

	NCV <sub>0</sub> MJ/kg	Ash (wt% d.b.)	Ash-melting point (°C)
Typical values for virgin wood materials Coniferous wood	19.2 (18.8-19.8)	0.3 (0.2-0.5)	
Typical values for virgin wood materials Deciduous wood	19 (18.5-19.2)	0.3 (0.2-0.5)	
Typical values for virgin bark materials	20 (19-21)	4-5 (2-10)	
Typical values for virgin wood materials Logging residues	19-20	1.5-2	
Typical values for virgin wood materials Short rotation coppice (SRC) (Willow and Poplar)	18.6-19.2	2	
Spruce (with bark)	18.8	0.6	1,426
Beech (with bark)	18.4	0.5	1,340
Poplar (SRC)	18.5	1.8	1,335
Willow (SRC)	18.4	2.0	1,283
Bark (coniferous trees)	19.2	3.8	1,440
Vine wood (chips)	19.8	3.4	1,450
Miscanthus	17.6	3.9	973
Wheat straw	17.2	5.7	998
Triticale (grains)	16.9	2.1	730
Rape cake	21.2	6.2	-

Fig 4-5-2 Figure of Net Calorific Value and Ash Content in Wood

Figure 4-5-2 shows that typical Lower Calorific Value or Net Calorific Value (NCV) of coniferous wood is 19.2 MJ/kg with a ash content ranging from 0.2~0.5% wt. Typically spruce wood with its bark intact has a NCV of 18.8 MJ/kg with a ash content of 0.6% wt.

	C	H	O	N	K	S	Cl
	wt% (d.b.)						
Spruce (with bark)	49.8	6.3	43.2	0.13	0.13	0.015	0.005
Beech (with bark)	47.9	6.2	43.3	0.22	0.22	0.015	0.006
Poplar SRC	47.5	6.2	44.1	0.42	0.35	0.031	0.004
Willow SRC	47.1	6.1	44.2	0.54	0.26	0.045	0.004
Bark (coniferous trees)	51.4	5.7	38.7	0.48	0.24	0.085	0.019
Typical values for virgin wood materials Coniferous wood*	47-54	5.6-7.0	40-44	<0.1-0.5		<0.01-0.05	<0.01-0.03
Typical values for virgin wood materials Deciduous wood*	48-52	5.9-6.5	41-45	<0.1-0.5		<0.01-0.05	<0.01-0.03
Typical values for virgin bark materials*	51-56	5.9-6.5	36-43	0.3-1.2		0.02-0.20	<0.01-0.05
Typical values for virgin wood materials Logging residues*	50-53	5.9-6.3	40-44	0.3-0.8		0.01-0.08	<0.01-0.04
Typical values for virgin wood materials Short rotation coppice*	47-51	5.8-6.7	40-46	0.2-0.8		0.02-0.10	<0.01-0.05
Miscanthus	47.5	6.2	41.7	0.73	0.70	0.150	0.220
Wheat straw	45.6	5.8	42.4	0.48	1.00	0.082	0.190
Triticale (grains)	43.5	6.4	46.4	1.68	0.60	0.110	0.070
Rape cake	51.5	7.4	30.1	4.97	1.60	0.550	0.019
<i>For comparison, fossil fuels</i>							
Coal	72.5	5.6	11.0	1.30	-	0.940	< 0,1
Lignite	65.9	4.6	23.0	0.70	-	0.390	< 0,1
Heating oil	85-86	11-13	1-4	-	-	-	-
Natural gas	75	25	-	-	-	-	-

\* CEN/TS 14961:2005 Solid biofuels - Fuel specifications and classes – Annex C

Fig 4-5-3 Chemical composition of elements in wood samples

Figure 4-5-3 shows that typically spruce wood with its bark intact has an element make up of C (49.8%), H (6.9%), O (43.2%) while the other element making up for the remaining percentages. While heating oil has element make up of C (85-86%), H (11-13%), O (1-4%).



M (%)	MWh/t	GJ/t	M (%)	MWh/t	GJ/t
15	4.27	15.36	38	2.93	10.54
16	4.21	15.15	39	2.87	10.33
17	4.15	14.94	40	2.81	10.12
18	4.10	14.73	41	2.76	9.91
19	4.04	14.52	42	2.70	9.71
20	3.98	14.31	43	2.64	9.50
21	3.92	14.10	44	2.58	9.29
22	3.86	13.89	45	2.52	9.08
23	3.80	13.68	46	2.47	8.87
24	3.75	13.47	47	2.41	8.66
25	3.69	13.27	48	2.35	8.45
26	3.63	13.06	49	2.29	8.24
27	3.57	12.85	50	2.23	8.03
28	3.51	12.64	51	2.17	7.82
29	3.45	12.43	52	2.12	7.61
30	3.40	12.22	53	2.06	7.40
31	3.34	12.01	54	2.00	7.19
32	3.28	11.80	55	1.94	6.98
33	3.22	11.59	56	1.88	6.77
34	3.16	11.38	57	1.82	6.56
35	3.11	11.17	58	1.77	6.35
36	3.05	10.96	59	1.71	6.15
37	2.99	10.75	60	1.65	5.94

Fig 4-5-4 Influence that moisture Content has on energy stored in the pellets

Figure 4-5-4 shows clearly that the increasing the moisture content will decrease the energy content and efficiency of the wood pellets. Therefore it is highly recommended that pellet moisture content is kept between 8 - 12% wt.

## **5.0 Experimental Setup & Procedure**

### **5.1 Introduction**

Spruce wood was cut, treated and grinded down and then the powder was dried before moisture of about 11% was added to it. These spruce fibres were then added with waste vegetable oil, which was obtained from MacDonald's in Vestby, Akershus, Norway, of 3 varying compositions of 0%, 5% and 10%. The mechanical and physical properties of the powder were measured as described in the following experimental setup and procedures.

### **5.2 Experimental Design**

Spruce with 11 % moisture content was mixed with oil and prepared in three different compositions of 0%, 5% and 10%. It was then compacted in three different temperatures of 60 C, 120 C and 180 C; and four different pressures of 75 MPa, 150 MPa, 225MPa and 300 MPa.

The moisture content was chosen to be 11 % because it was the most common moisture content used in commercial wood pelleting [14, 32, 43, 50]. While similarly the temperature and pressure ranges encompass with the most common pellet production range [14, 32, 43, 50]. Higher moisture content will lead to a problem in producing pellets on the ring type of die pellet presses [14, 32, 43, 50].

S0-T : Spruce (11 wt % moisture) + 0 wt % Oil

S5-T : Spruce (11 wt % moisture) + 5 wt % Oil

S10-T : Spruce (11 wt % moisture) + 10 wt % Oil

\*T : Temperature of compacted pellets in degree Celsius

### **5.3 Preparation of the wood samples**



Figure 5-2: : P 1 - 6 – Diosna Mixer



Figure 5-3: Spruce Powder, 0% Oil, 11% Moisture

Spruce wood used was taken from stumps in spruce trees (It is meant to represent similar stumps from fallen tree during logging) that are found in the region of Ås in Akershus in the Southern Eastern regions of Norway ( $59.6603^{\circ}$  N,  $10.7836^{\circ}$  E) at 95 m in altitude. The barks were removed to ensure a homogenous composition. The stumps were cut into smaller pieces with a thickness ranging from 3-4 mm. These smaller pieces were dried for 48 hours at  $105^{\circ}\text{C}$  before its mass were measured and noted as the initial mass.

These wood samples were grinded with a sieve size of 1.5 mm using a, Brabender, Duisburg, Germany, cutting mill. The grinded wood was then dried for 24 hours in an oven, Termaks, Norway, at  $105^{\circ}\text{C}$  to remove all moisture. It was then re-moisturized using a mixer, Mixer Granulator P 1 - 6 – Diosna, to 10-12 wt% water. The mixer speed was pre-set to 250 rpm and the chopper speed was set to 500 rpm.

The moisture content in the fibre were then rechecked by spreading and weighing it on the surface of a Mettler Moisture Analyser and then reweighing the dried fibres once the weigh had stabilized.

The moist fibres were now mixed with 5% or 10% wt Oil using the same mixer, Mixer Granulator P 1 - 6 – Diosna. Similarly the mixer speed was pre-set to 250 rpm and the chopper speed was set to 500 rpm. Pellets were then produced using these fibres. Any unused sample was vacuumed and kept in a climactic chamber to maintain the homogenous moisture content distribution is not lost (At 20C and 50% Relative Humidity).

#### **5.4 Design Feature for Pellets**

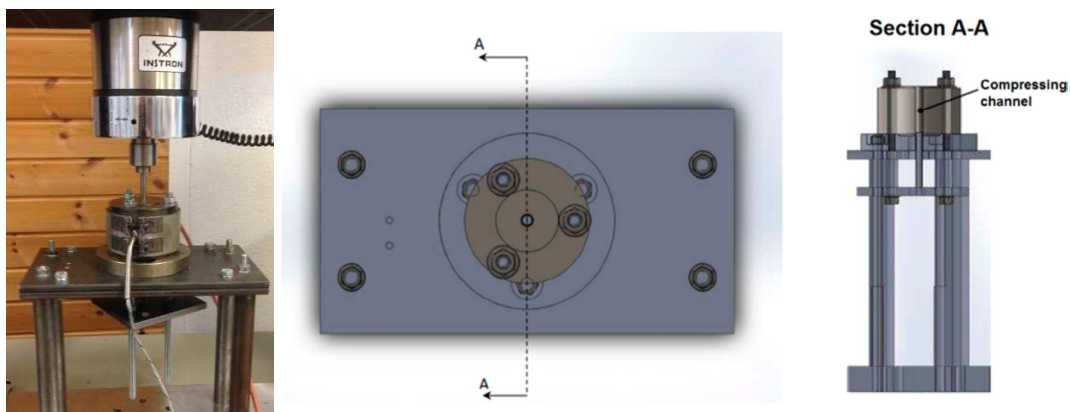


Figure 5-4: Single Pellet Press Unit (Left), Drawing of Single Press Unit: Top View\* (Centre) and Section A-A view\* (Right)  
\* Diagram from Mišljenović et al [11]

The wood pellet production was done using a pre-assembled die pelleting rig (see Fig. 5-1) as a fixture that was as described and used by Mišljenović et al [17]. The setup was made up of a compressing chamber of diameter 8 mm where the powders were compressed using a tungsten carbide compressing rod; and a thermocouple connected to a proportional integral derivative controlled heater with a power of 450 W which controls the production temperature of the pellets. The compacting pressures in the chamber were controlled using a Instron 5800R 100 kN texture analyser which was connected to the assembly. The detailed design for the working principle and the pellet production process is described in Salas-Bringas et al [20].

### **5.5 Production of wood pellets**

The Spruce powder sample were added into a cylindrical compressing chamber until all the powder were inside it. The Spruce power were then compressed at varying tempreature and pressure. Three tempreature (60C, 120C and 180C) and four pressure (75 MPa, 150MPa,225MPa and 300MPa) were used in compressing and producing the pellets. All the Spruce powder were compressed in the normal direction and 5 pellets were produced under each respective pressure as the tempreature was held\_constant. The speed of the compression was set to 2 mm/s with a short retension time of 1s. Through literiture studies we can show that different pressure can be used to produce pellets for different wood powders. Spruce sawdust pellets can be produced from pressures starting from 50MPa [20], while other different wood powder starts from 70MPa up to 300MPa [20]. These difference can be attributed to the tempreature used, die properties and; the physical and chemical properties of the wood powders such as particle size distribution, mositure content and other organice products added in such as oil [14, 32, 43, 50]. The compression rod was released once the the required pressure was attained. The compression rod was then again used to remove the pellets from the chmaber channel with a releasing force which is the force required to move the pellet from the inside of the chamber channel.

Pellets produced using a compression chamber has a density gradient which is a source of error . The friction between the wood poweder and the sides of the chamber rudeuces in stress from the top to the bottom [18, 19, 20]. This is therefore the cause of the grdiant in density, with the highest density away from the blank die. The oil used also will have the highest density away from the blank die for this same reason, with most of the oil pushed and retained towards the bottom. However since the pellets were produced was short and had a relatively small diameter around 8mm, the density gradient can be safely be neglected.

The pellet diameter was determined using the weight of each pellets divided by the volume of the pellets by measuring the length and the diameter of each pellets produced.

$$\text{Density of Pellets} = \frac{\text{mass of pellet}}{\text{volume of pellet}}$$

Material binded better at higher tempreature since the the amorphous thermoplastic material lignin can act as a binder, when its tempreature is higher than the glass transition temperature

and lower than its melting point[39]. This explains the higher strength due to improved particle bonding.

### **5.6 Measurement of Pellet Strength and Compressibility**



Figure 5-6: Lloyd LR5K (Left), and Diametral tests of feed pellets\* (Right)  
\*Diagram from Salas-Bringas et al [11]

The pellet strength and compressibility were experimentally conducted by measuring the maximum peak force during a diametrical compression. The test indicated a plastic and ductile nature of the wood pellets. The shear strength per unit length ( $\text{kNmm}^{-1}$ ) was measured as we divided the maximum yield load with the pellet length. The speed of the test was pre-set to 1 mm/min and the test ended when the probe reached 2.2 mm below the diameter of the pellet (strain  $\sim 0.23$ ). The analysis of pellet strength and compressibility was similar to the procedure used by Rhén et al [57].

## 5.7 Measurement of Energy Content



Figure 5-7: Parr® Oxygen Bomb Setup (Left) and Drawing of Internal Components of Oxygen Bomb

The heat of combustion is the amount of energy given out when the reactants burn completely with oxygen under standard conditions. Typical reaction involves the combustion of organic products to produce water and carbon dioxide as by products.

The heating value is now the energy of the reacted according to the amount of substance used. It is usually defined in mass or mol. The higher heating value (HHV) is a measure of the amount of energy produced when the one unit of reactants is completely combusted. The HHV value is determined when the combustion is complete as all material are burned and as temperature is cooled down and stabilized to a constant pre combustion temperature.

The higher heating value measurement is done using an oxygen bomb calorimeter. The reactants in an oxygen explosive-proof container are emerged in water. The reactants are then ignited and the change in temperature is measure with a highly sensitive thermometer.

The Parr® Oxygen Bomb user manual was used to calculate the higher heating value of the pellets.

## 5.8 Measurement of Bulk Density



Figure 5-8: Bulk Density Measurement Apparatus

Bulk density (BD) is the measure of wood mass per unit volume of wood. BD of the wood is the degree of compaction of the wood since the BD is mainly depended on the empty air spaces, porosity and minerals compositions. Wood has varying sizes (this will be looked at in particle distribution) while the remaining are made up of generally pore space. The pore space can contain either gas or liquid space.

The spruce powder was funnelled into a measure cylinder in which it was weighed in using a weighing machine. The volume in which the fibre occupied was also recorded directly reading off the measuring cylinder. The bulk density was then found using the equation below.

$$\text{Dry bulk density} = \frac{\text{mass of birch wood}}{\text{volume of cylinder}}$$



## **5.9 Measurement of Particle Distribution**



Figure 5-9: Malvern Mastersizer 3000  
[www.malvern.com](http://www.malvern.com)

The particle size distribution (PSD) of a powder defines the relative quantity typically in mass or volume of particles sizes present in the powder. The Malvern Mastersizer 3000 laser diffraction particle size analyser was used to analyse our results. These values are expressed typically in percentages [64]. PSD is also known as grain size distribution. The PSD can be very important in analysing and understanding the physical and chemical characteristic of the powder. It can also affect the reactivity of solids which participate in a chemical reaction. PSD is normally done through sieving of powder by its size.

The Malvern Mastersizer 3000 user manual was used to measure the particle distribution. The powders were drained in a sieved container better separated the fibres into its respective diameter for examination. It then uses a laser diffraction to measure the size of particles. The intensity of light is scattered as a laser beam passes through the spruce fibres. These data is then computed in the computer to calculate the size of the particles based on the scattering pattern.

### **5.10 Measurement of Moisture Content**



Figure 5-10: Mettler Moisture Analyser

The moisture content of wood can rise up to 10%-15% even when left untouched or undisturbed since the absorption of moisture from air humidity itself occurs rapidly. The hygroscopicity of the wood also changes if it is stored, transported or burned in unfavourable conditions. The calorific or heating value of the wood is reduced due to presence of moisture thus making it an adulterant when sold wrongly to consumers.

Moisture content is experimentally determined by drying and measuring a weighted sample of the powdered wood in a Mettler Moisture Analyser. The moisture content percentage is then expressed against its initial wet weight.

$$\text{Moisture Content (\%)} = \frac{\text{Dry Sample Weight}}{\text{Wet Sample Weight}} \times 100$$

### **5.11 Measurement of Ash Content**



Figure 5-11: Nabertherm Oven

Ash normally constitutes mainly of inorganic materials such as silica and magnesium oxides after combustion [56]. These materials are present from the original wood compound or come from containments during processing. Ash contents can vary from 0.5% to 5 % depending on the wood and process used [56]. The bark of the wood or the earth sand included during processing can also affect the ash content quantity. Good quality wood can have ash content of about 3% while fine wood can have a higher ash content value [56].

Ash is experimentally determined by combusting a weighed sample in air with thus burning all the organic combustible material away.

Measurements for ash content were conducted following the EN 14775 Standard Method instruction manual.

$$\text{Ash Content (\%)} = \frac{\text{Incombustible Sample Weight}}{\text{Total Sample Weight}} \times 100$$

### **5.12 Field Emission Scanning Electron Microscope (FE SEM)**



Figure 5-12: FE SEM/ FEI Quanta 200F Environmental SEM with EDAX

The field-emission scanning electron microscope/energy-dispersive X-ray spectroscopy (FE SEM/EDAX) modelled FEI Quanta 200F Environmental SEM with EDAX Microanalysis was used to examine the morphology of the fibres and to investigate the percentage of element that were present in the Spruce fibres (See Figure 5-13). The FEI Quanta 200F user manual was used for the FE SEM/EDAX Analysis.

## **RESULTS AND DISCUSSION**

### **6.0 Experimental Data – SEM/ FE SEM Images**

The SEM images from FE-SEM (FEI Quanta 200F); of the grinded spruce powder with different oil concentration are shown in the Figures below. The wood fibres were placed on a carbon film and Low Vacuum and High Voltage electron beam of 20kV was used for this investigation. The FEI Quanta 200F user manual was used for the FE SEM Analysis. The measured average diameter and diameter range is only with respect to the recorded images.

#### **6.1 S0 Powder**

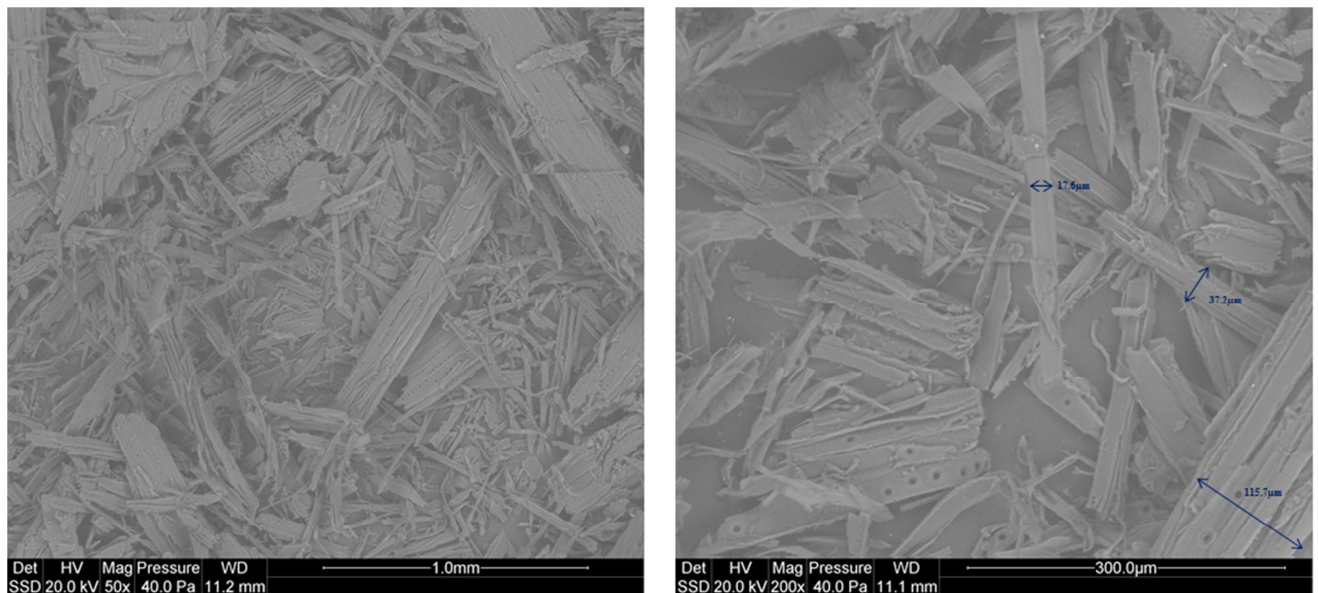


Figure 6-1 a (left) and b (right): SEM images of S0 Powders

Figure 6-1 a and b shows the Spruce wood which was grinded into fibre powders and stabilize at 110 °C before being examined. The S0 fibres had an average diameter of about 57 µm while its diameter ranged from 1 µm to 120 µm. The fibres surface was generally rough through most of the fibres.

## **6.2 S5 Powder**

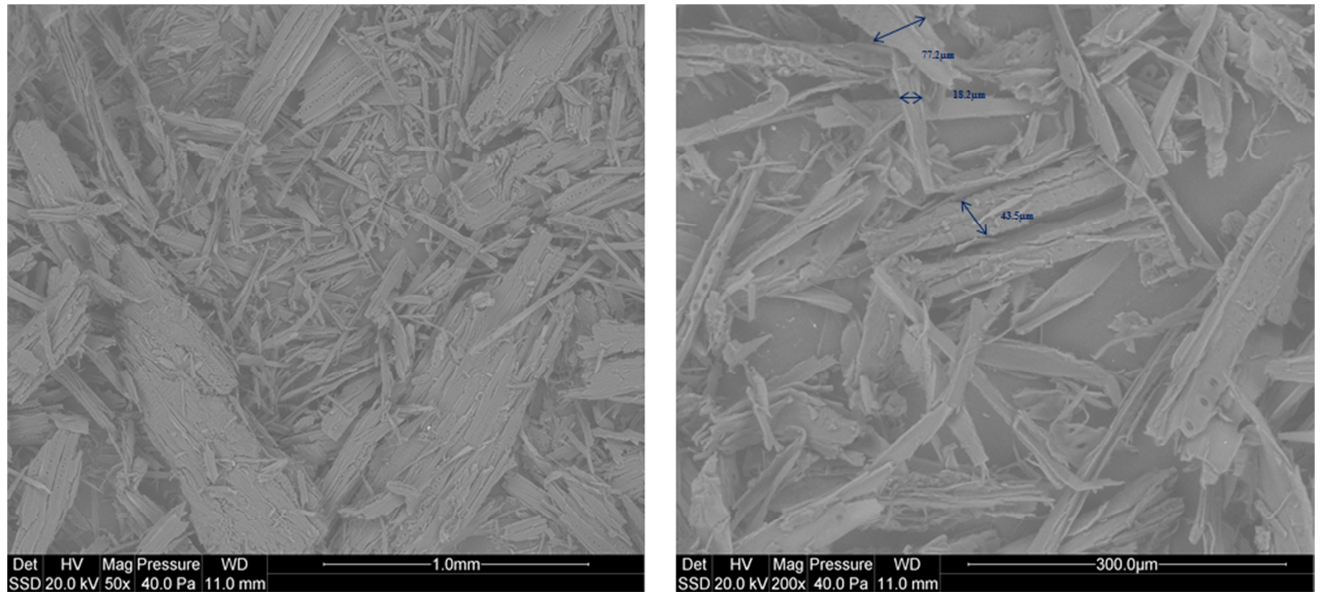


Figure 6-2 a (left) and b (right): SEM images of B5 Powders

Figure 6-2 a and b shows the Spruce wood which was grinded into fibre powders mixed with 5% oil and then stabilize at 110 °C before being examined. The S5 fibres had an average diameter of about 47 µm while its diameter ranged from 0.5 µm to 90 µm. The fibres surface was generally rough through most of the fibres.

### **6.3 S10 Powder**

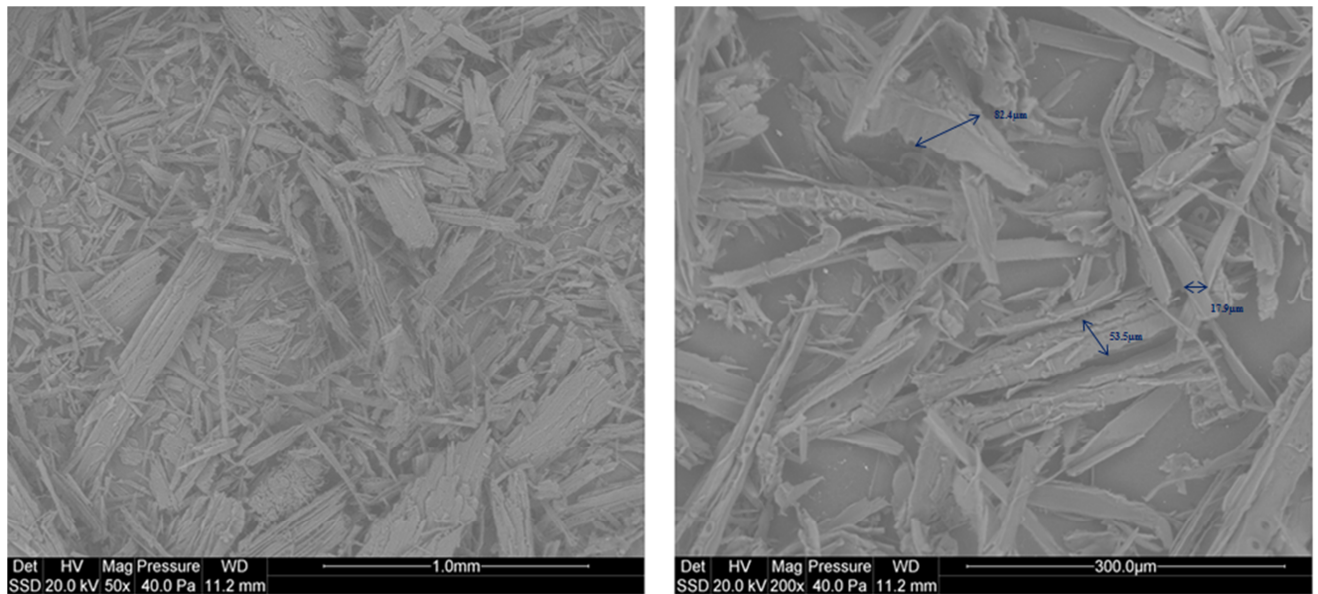


Figure 6-3 a (left) and b (right): SEM images of S10 Powders

Figure 6-3 a and b shows the Spruce wood which was grinded into fibre powders mixed with 10% oil and then stabilize at 110 °C before being examined. The S10 fibres had an average diameter of about 51 µm while its diameter ranged from 1.5 µm to 90 µm. The fibres surface was generally rough through most of the fibres.

## **7.0 Experimental Data – EDX Analysis**

The energy-dispersive X-ray spectroscopy (FE SEM/EDX) modelled FEI Quanta 200F was used to investigate the element composition that were present in the fibres. The FEI Quanta 200F user manual was used for the EDX Analysis. The measured element composition is only with respect to different points in the recorded images.

### **7.1 EDX of the S0 Powders**

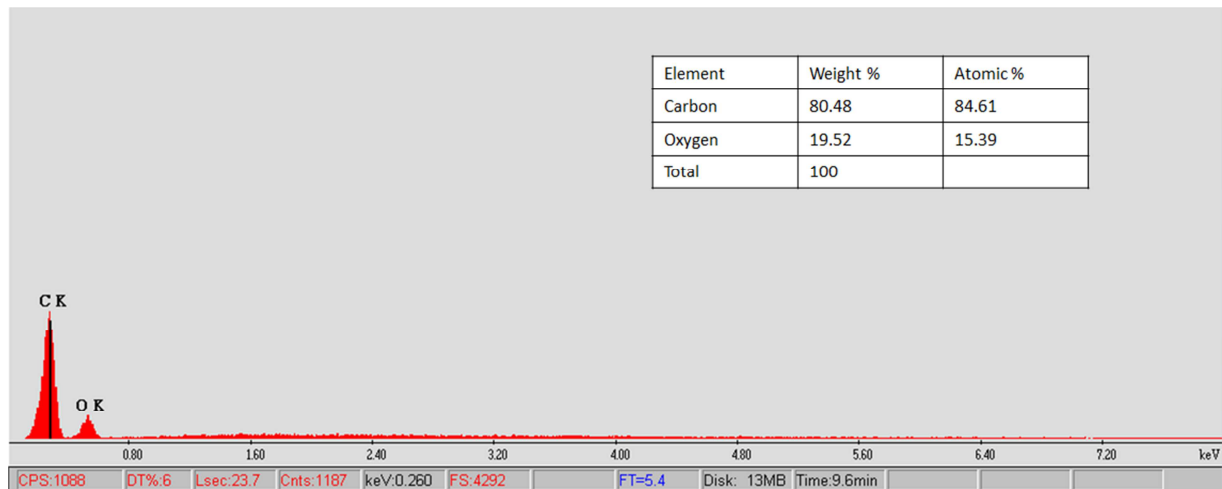


Figure 7-1: EDX imaging for B0 Powder

Figure 7-1 shows that at the medium and bigger S0 fibres have a more absolute quantity of Carbon and Oxygen in them. It has C:O ratio of 85:15 respectively.



## 7.2 EDX on the S5 and S10 Powders

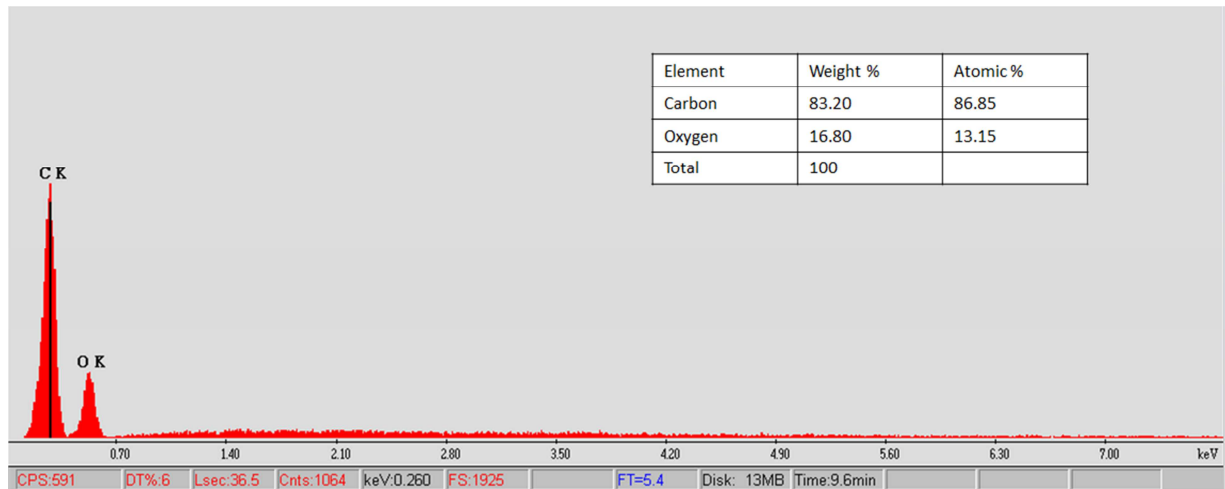


Figure 7-2: EDX imaging for B5 and B10 Powders

Figure 7-2 shows that at the medium and bigger S5/S10 fibres have a more absolute quantity of Carbon and Oxygen in them. It has C:O ratio of 87:13 respectively. Its carbon content is higher than that in the S0 powder.

### **7.3 EDX on S0 (Small Fibres)**

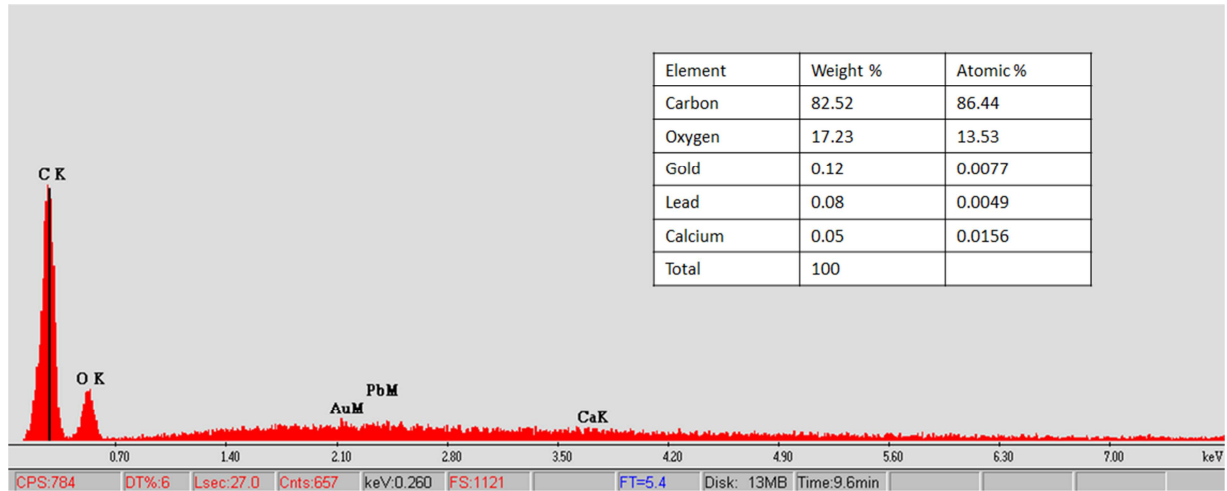


Figure 7-3: EDX imaging for B0 Powders (Small Fibres)

Figure 7-3 shows that the small S0 fibres have a mixed quantity of Carbon, Oxygen, Gold, Lead and Calcium in them. Only Carbon and Oxygen is in high quantity while the other elements are in very minimal in quantity as seen in the Table with in Figure.

### 7.4 EDX on S10 (Small Fibres)

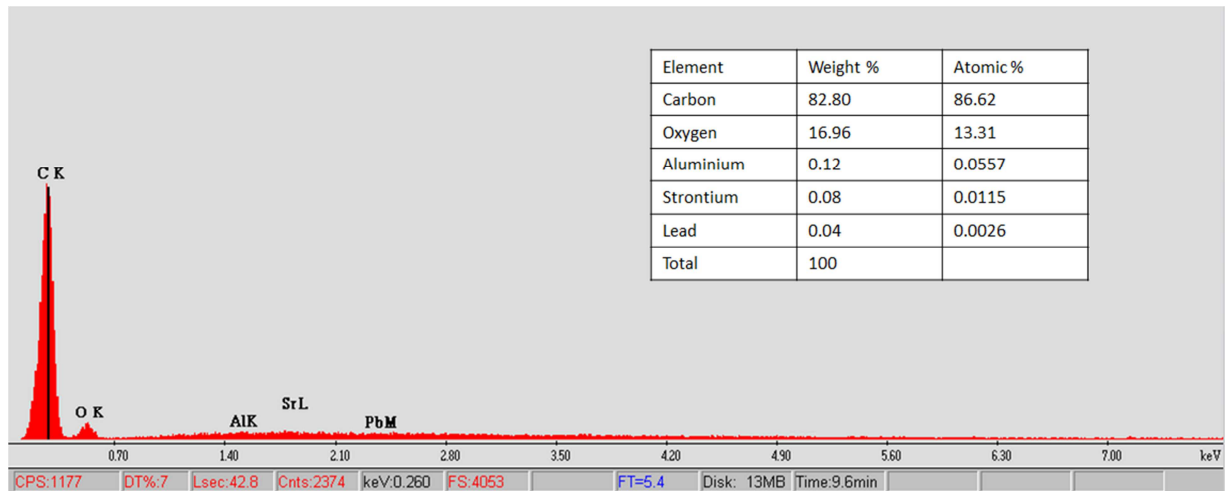


Figure 7-4: EDX imaging for B0 Powders (Small Fibres)

Figure 7-4 shows that the small S0 fibres have a mixed quantity of Carbon, Oxygen, Aluminium, Strontium and Lead in them. Only Carbon and Oxygen is in high quantity while the other elements are in very minimal in quantity. Its carbon content is higher than that in the S0 powder.

## 8.0 Pellet Strength and Compressibility

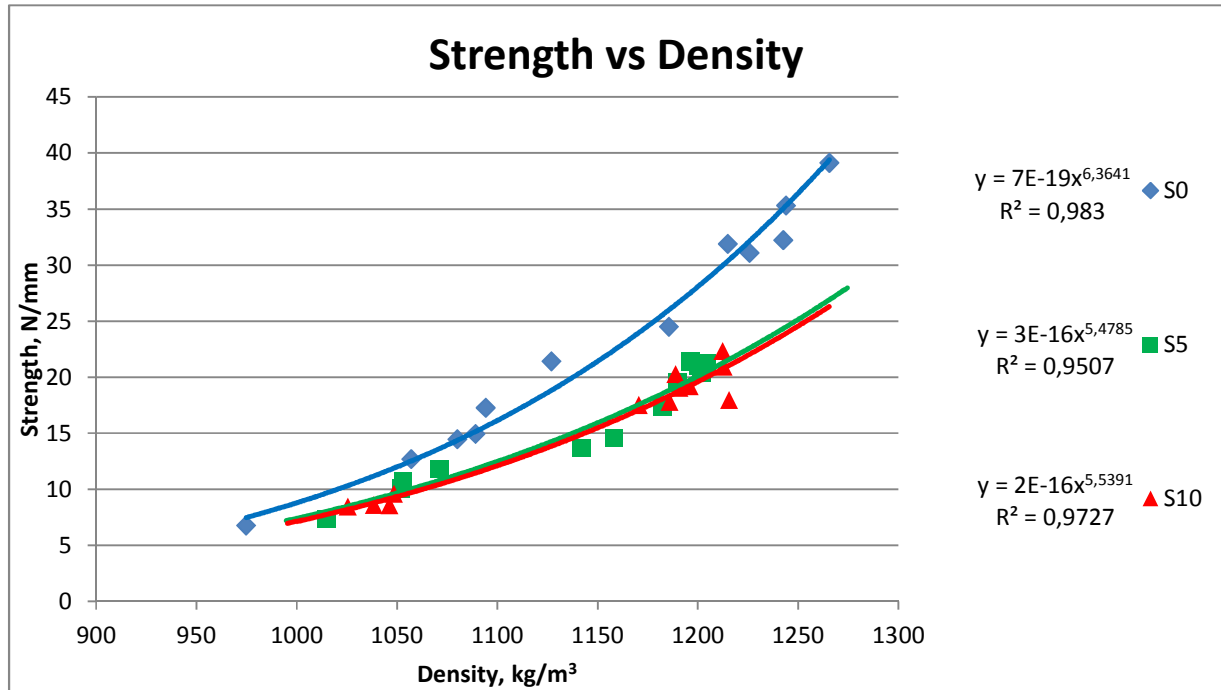


Figure 8-1: Strength vs Density Curve

Figure 8-1 above shows the graph for strength vs density which was conducted for the S0, S5 and S10 pellets at 60C, 120C and 180C and 75 MPa, 150 MPa, 225 MPa and 300 MPa (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

There is a increasing trend of an increase in strength when the density increase. This is expected as an increase in density would indicate a more compact structure which stronger bonding and forces acting on them. Addition of oil also shows the decrease in strength due to the reduction of possible hydrogen bonding sites and the interfacial forces and capillary pressure. The reasoning for this expected result is discussed below in Section 9.7 and 9.8.

## 9.0 Pellet Strength and Compressibility

Pellet Strength and Compressibility for the three S0, S5 and S10 pellets; was done using diametrical compression. The speed of the strength test was pre-set to 1 mm/min with the test ending when the probe is 2.2 mm below the pellet.

### 9.1 S0 at 60C, 120C and 180C

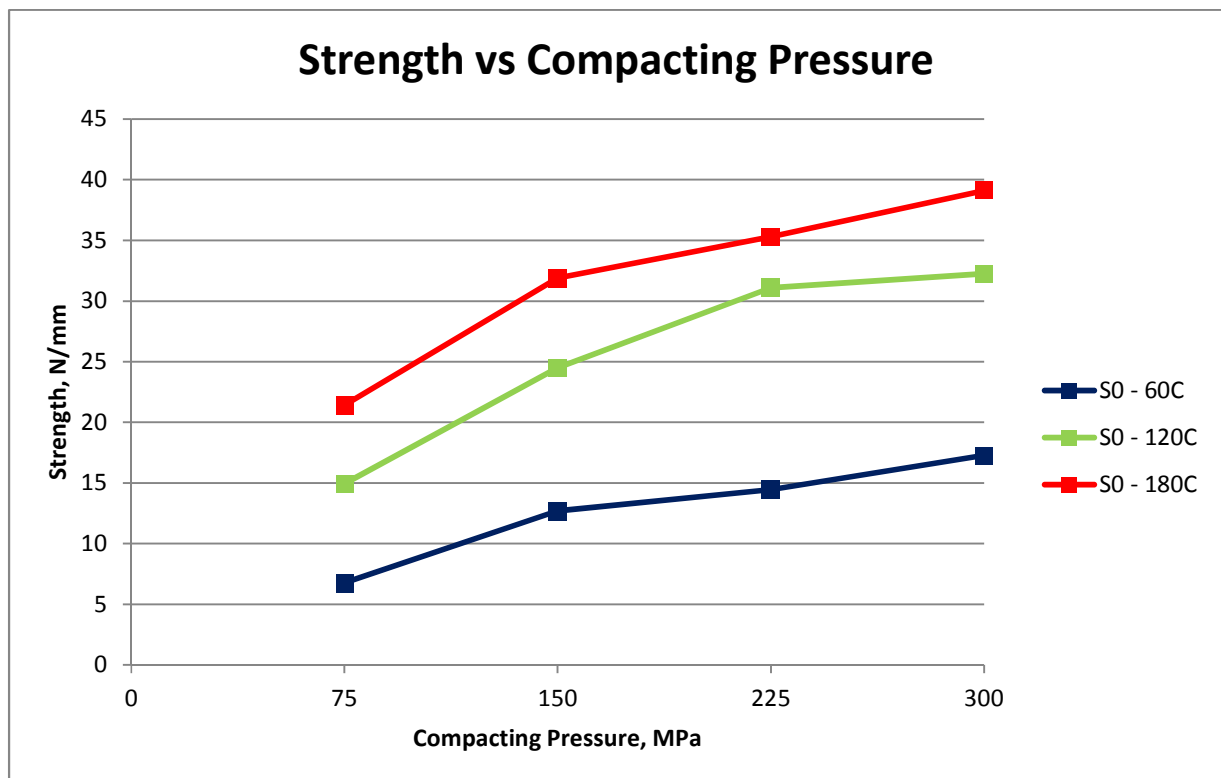


Figure 9-1a: Graph of Strength vs Pressure for S0 at 60C, 120C and 180C.

The figure 9-1a above shows the graph for strength test conducted for the S0 pellets at 60C, 120C and 180C (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The strength of the S0 pellets increased as the compacting pressure of the pellets increased.

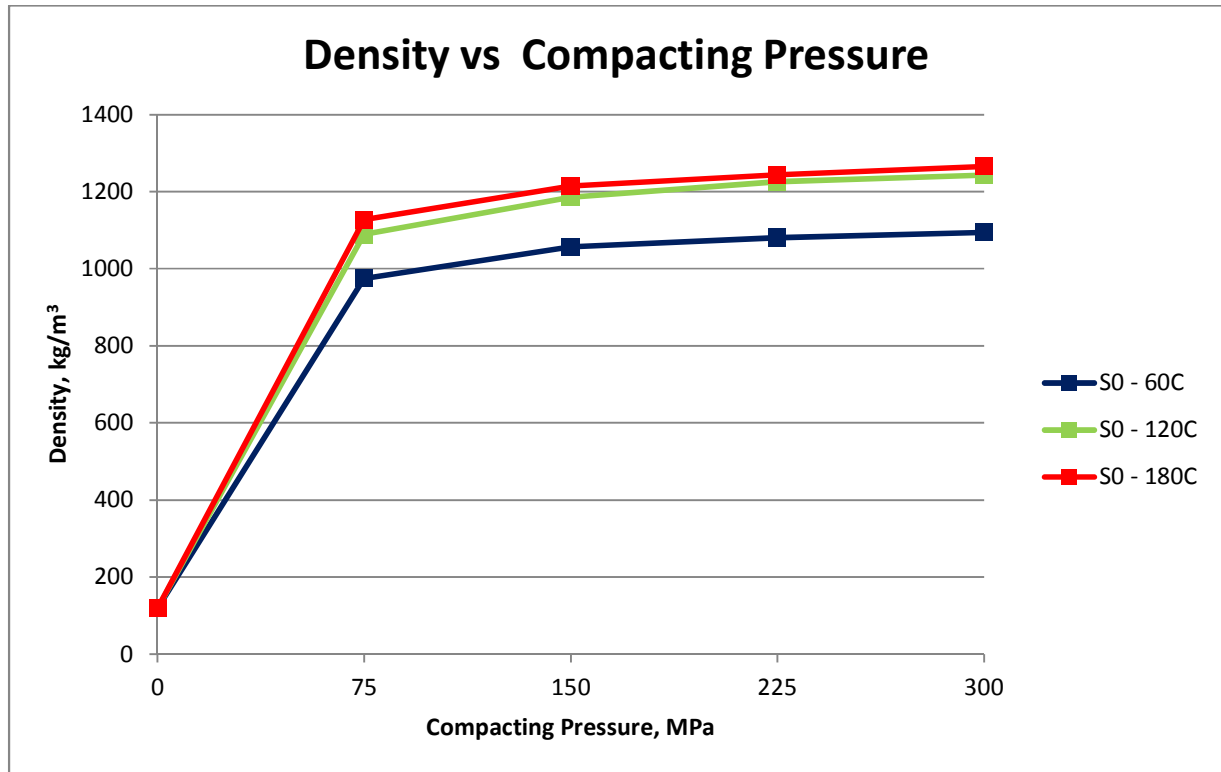


Figure 9-1b: Graph of Compressibility vs Pressure for S0 at 60C, 120C and 180C.

The figure 9-1b above shows the compressibility graph, for the S0 pellets at 60C, 120C and 180C (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The density of the S0 pellets increased as the compacting pressure of the pellets increased.

The maximum attainable density of all the S0 pellets would plateau at the same maximum compressibility if extrapolated to a higher pressure (Possibly at 1000 MPa).

S0 pellets produced after 150 MPa exhibited only minimal differences in the change in density for the any extra pressure exerted.

### 9.2 S0 at 75 MPa, 150 MPa, 225 MPa and 300 MPa

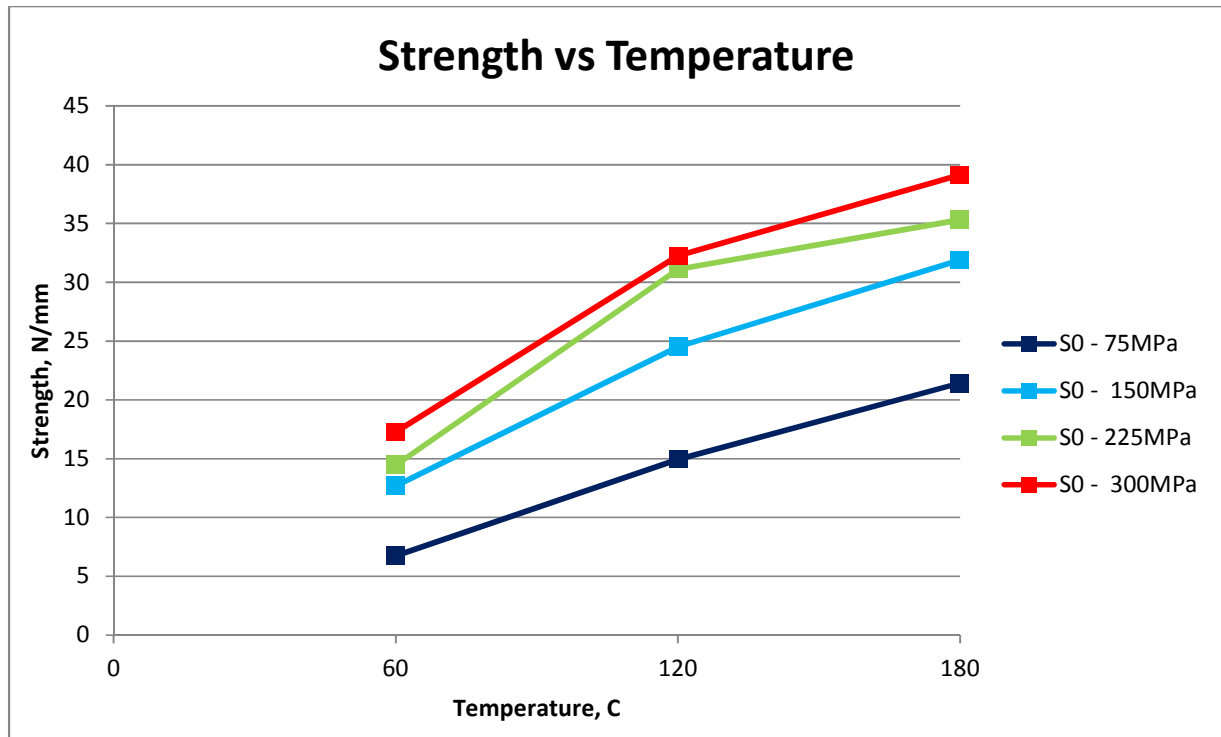


Figure 9-2a: Graph of Strength vs Temperature for S0 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.

The figure 8-2a above shows the graph for strength test conducted, for the S0 pellets at 75 MPa, 150 MPa, 225 MPa and 300 MPa (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The strength of the S0 pellets increased as the compacting temperature of the pellets increased.

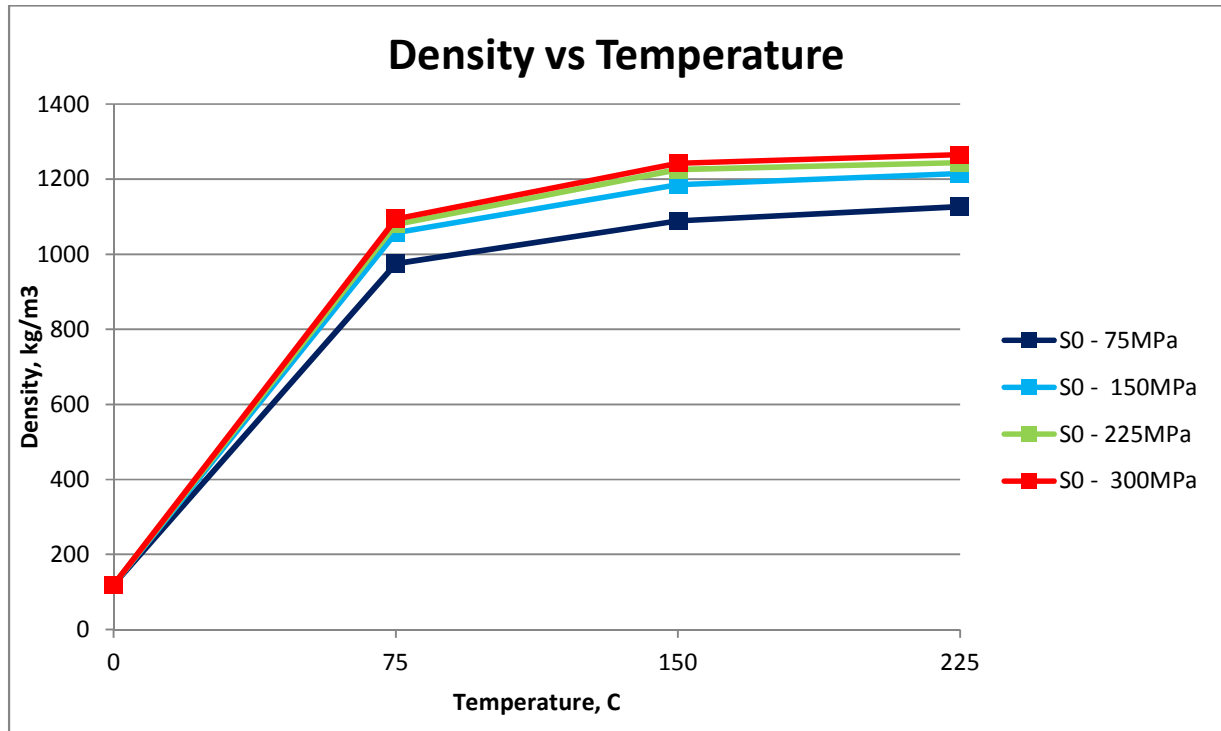


Figure 9-2b: Graph of Compressibility vs Temperature for S0 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.

The density of the S0 pellets increased as the compacting temperature of the pellets increased.

The maximum attainable density of all the S0 pellets would plateau at the same maximum compressibility if extrapolated to a higher temperature (Possibly at 500C).

S0 pellets produced after 120C exhibited only minimal differences in the change in density for the any extra temperature exerted. In fact the S0 – 150 MPa, S0 – 225 MPa and S0 – 300 MPa has almost reached maximum compressibility after 150C.

S0 pellets produced after 150C exhibited only minimal differences in the change in density for the any extra temperature exerted.



### 9.3 S5 at 60C, 120C and 180C

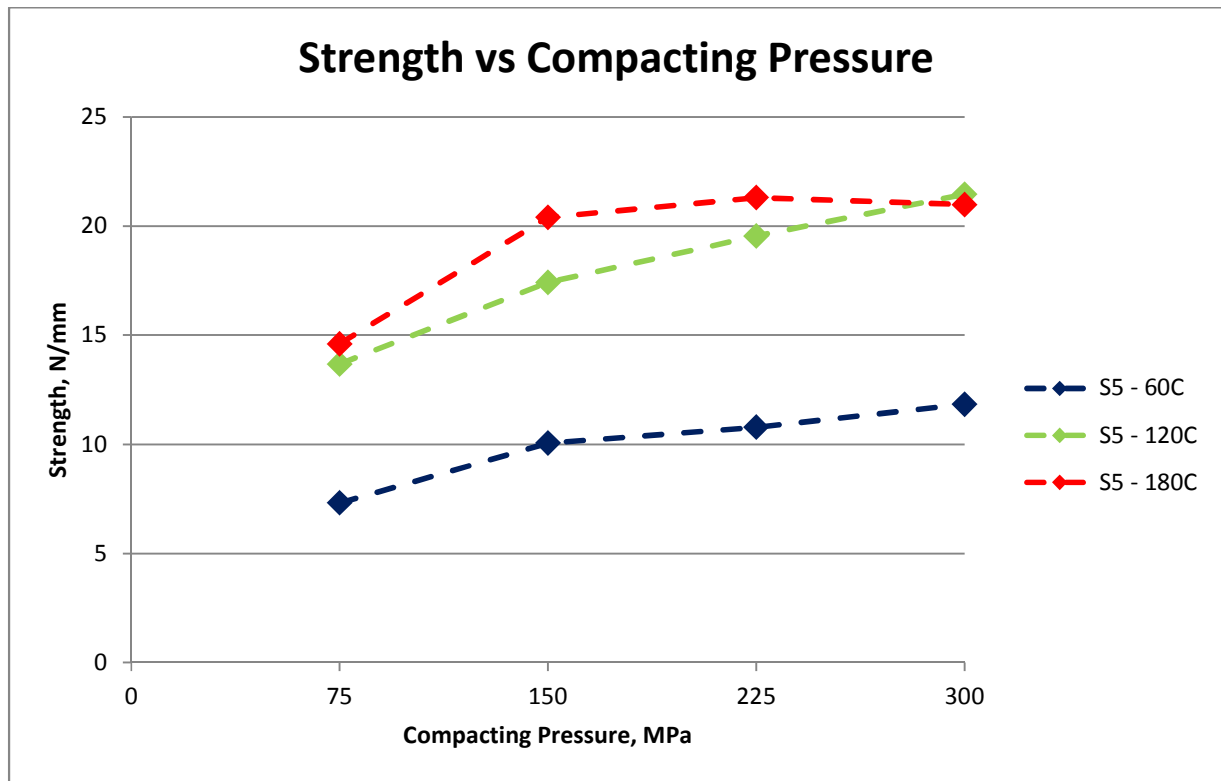


Figure 9-3a: Graph of Strength vs Pressure for S5 at 60C, 120C and 180C

The figure 9-3a above shows the graph for strength test conducted, for the S5 pellets at 60C, 120C and 180C (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The strength of the S5 pellets increased as the compacting pressure of the pellets increased. There is a discrepancy in strength for the S5 – 180 C at 300 MPa where is strength drops below the strength of the S5 – 120 C at 300 MPa at 180C.

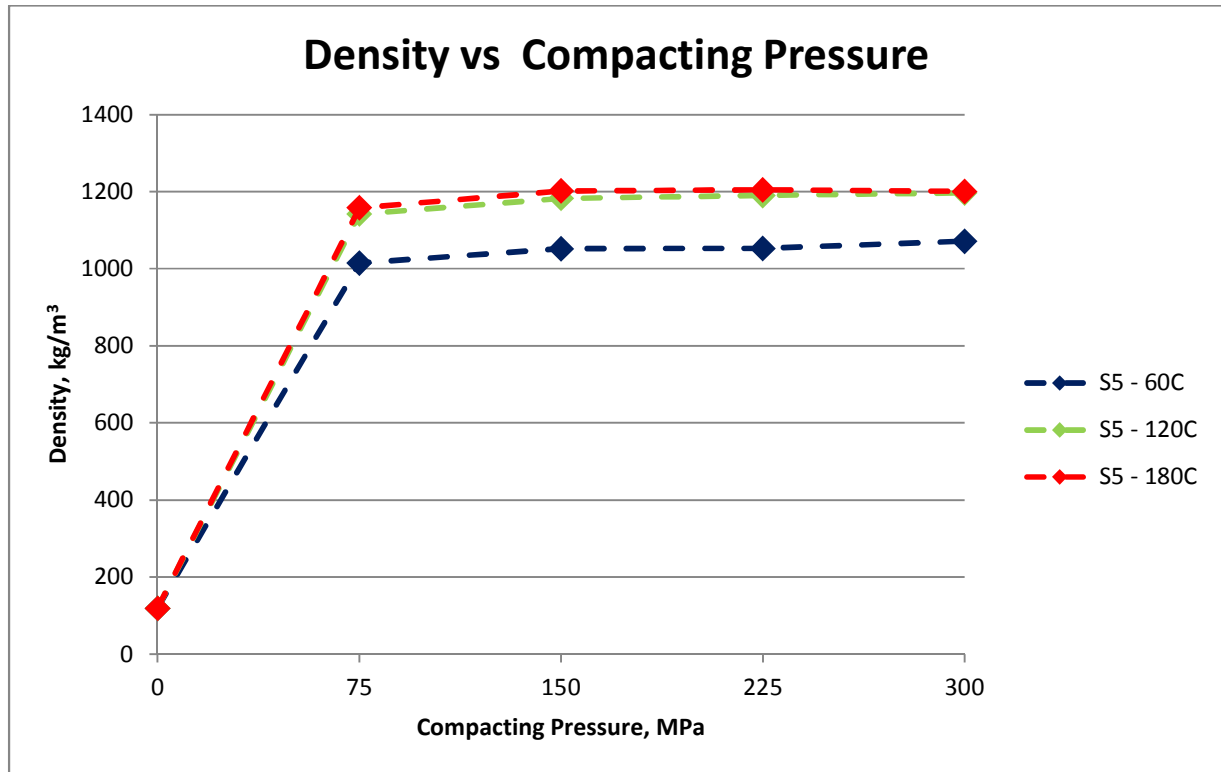


Figure 9-3b: Graph of Compressibility vs Pressure for S5 at 60C, 120C and 180C

The figure 8-3b above shows the compressibility graph, for the S5 pellets at 60C, 120C and 180C (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The density of the S5 pellets increased as the compacting pressure of the pellets increased.

The maximum attainable density of all the S5 pellets would plateau at the same maximum compressibility if extrapolated to a higher pressure (Possibly at 1000 MPa).

S5 pellets produced after 150 MPa exhibited only minimal differences in the change in density for the any extra pressure exerted. The S5 – 120 and S5 – 180 has already reached its maximum compressibility.

**9.4 S5 at 75 MPa, 150 MPa, 225 MPa and 300 MPa**

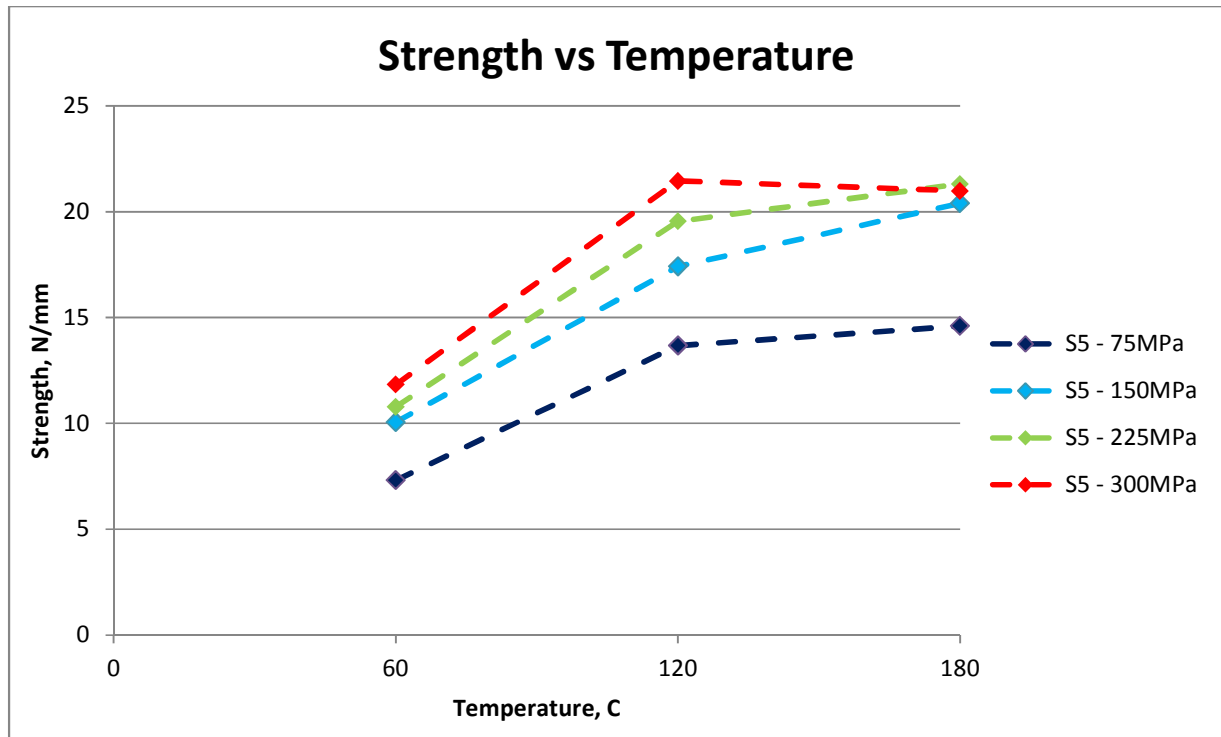


Figure 9-4a: Graph of Strength vs Temperature for S5 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.

The figure 9-4a above shows the graph for strength test conducted, for the S5 pellets at 75 MPa, 150 MPa, 225 MPa and 300 MPa (diameter ≈ 8 mm, mass ≈ 0,7~0,8g and length ≈ 12~14 mm).

The strength of the S5 pellets increased as the compacting temperature of the pellets increased. There is a discrepancy in strength for the S5 – 300 MPa at 180C where is strength drops below the strength of the S5 – 225 MPa at 180C.

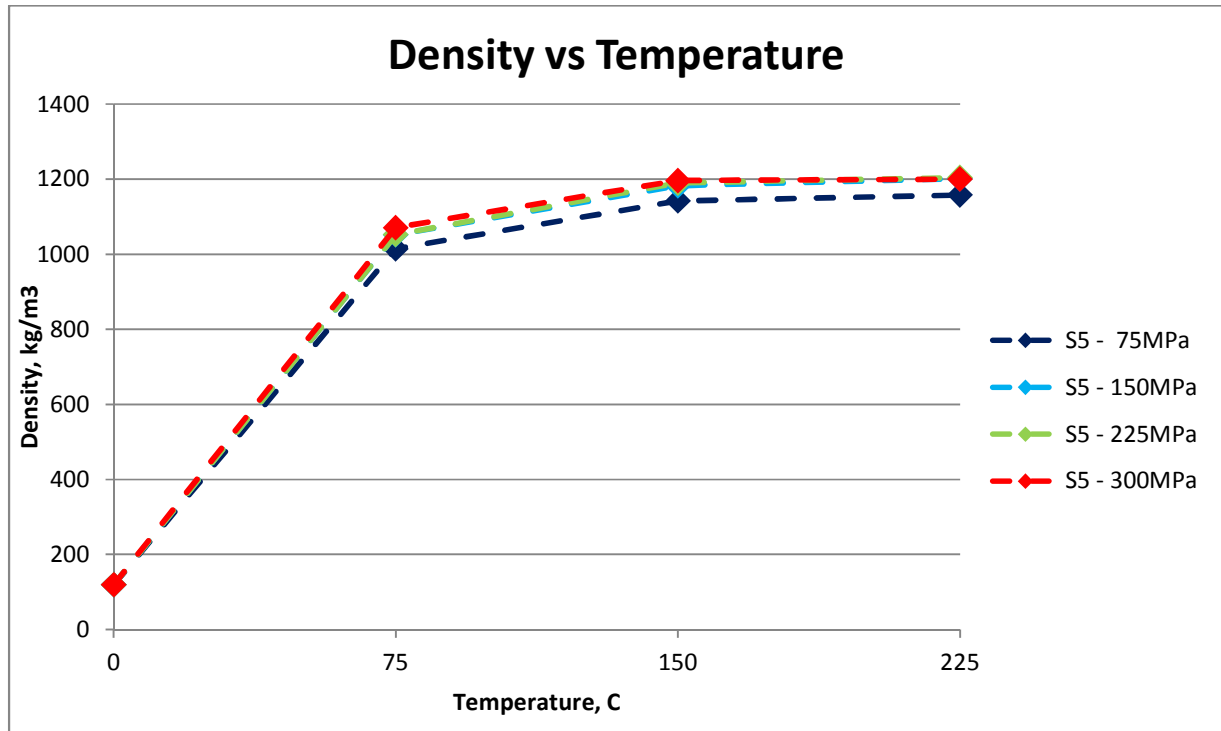


Figure 9-4a: Graph of Compressibility vs Temperature for S5 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.

The figure 9-4b above shows the compressibility graph, for the S5 pellets at 75 MPa, 150 MPa, 225 MPa and 300 MPa (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The density of the S5 pellets increased as the compacting temperature of the pellets increased.

The maximum attainable density of all the S5 pellets would plateau at the same maximum compressibility if extrapolated to a higher temperature (Possibly at 500C).

S5 pellets produced after 120C exhibited only minimal differences in the change in density for the any extra temperature exerted. In fact the S5 – 150 MPa, S5 – 225 MPa and S5 – 300 MPa has almost reached maximum compressibility after 150C.

S5 pellets produced after 150C exhibited only minimal differences in the change in density for the any extra temperature exerted.

### 9.5 S10 at 60C, 120C and 180C

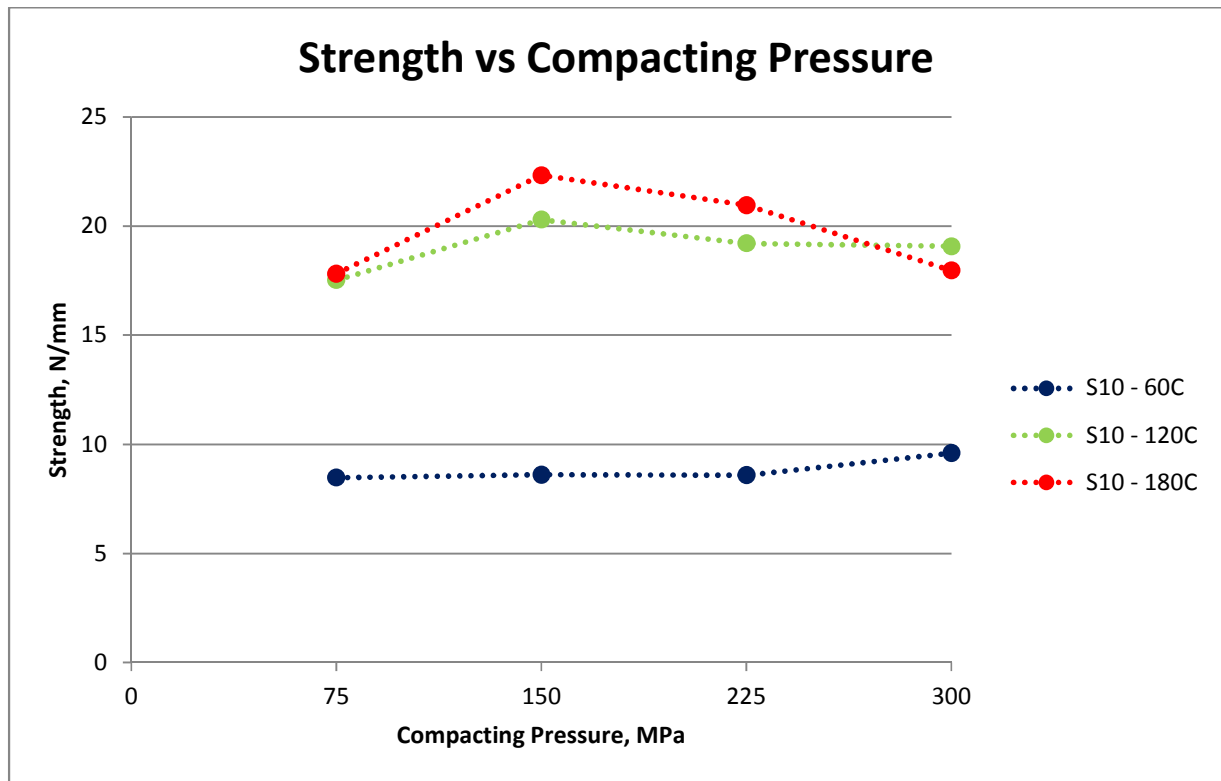


Figure 9-5a: Graph of Strength vs Pressure for S10 at 60C, 120C and 180C

The figure 9-5a above shows the graph for strength test conducted, for the S10 pellets at 60C, 120C and 180C (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The strength of the S10 pellets increased as the compacting pressure of the pellets increased. There is a discrepancy in strength for the S10 – 180 C at 300 MPa where is strength drops below the strength of the S10 – 120 C at 300 MPa at 180C.

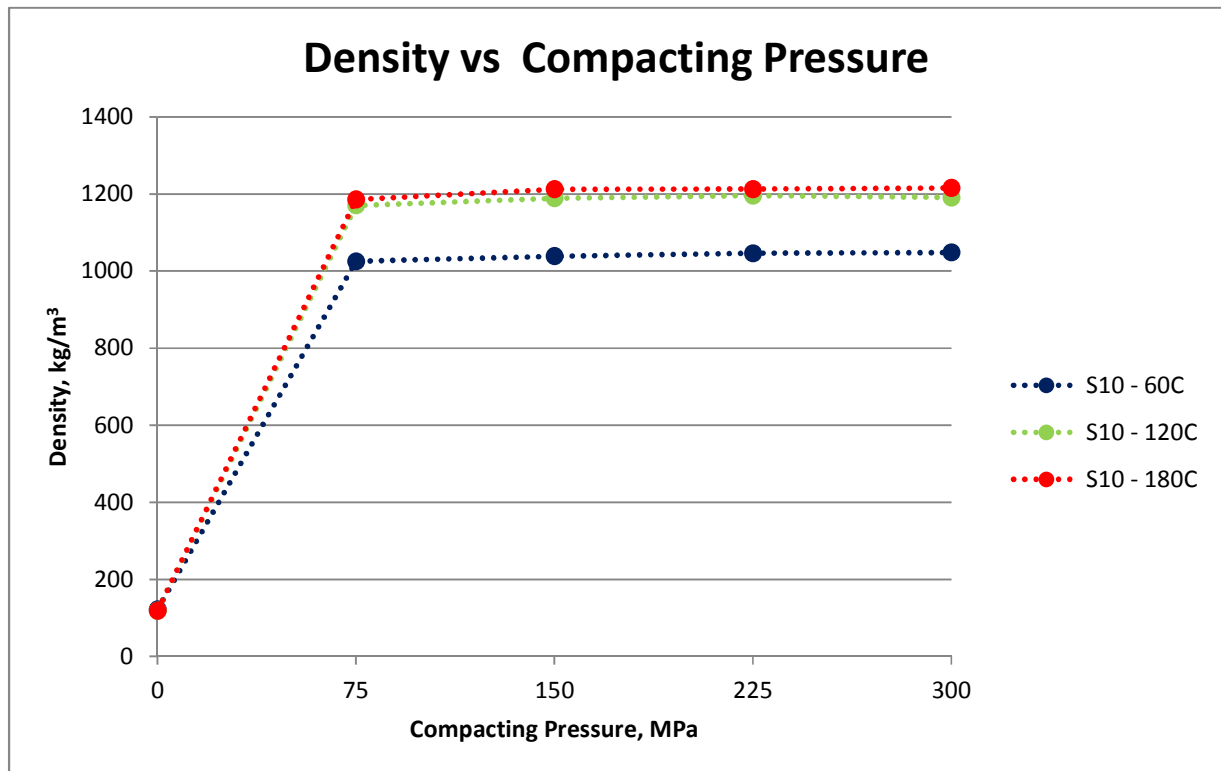


Figure 9-5b: Graph of Compressibility vs Pressure for S10 at 60C, 120C and 180C.

The figure 9-5b above shows the compressibility graph, for the S10 pellets at 60C, 120C and 180C (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The density of the S10 pellets increased as the compacting pressure of the pellets increased.

The maximum attainable density of all the S10 pellets would plateau at the same maximum compressibility if extrapolated to a higher pressure (Possibly at 1000 MPa).

S10 pellets produced after 150 MPa exhibited only minimal differences in the change in density for the any extra pressure exerted. The S5 – 120 and S5 – 180 has already reached its maximum compressibility.

### 9.6 S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa

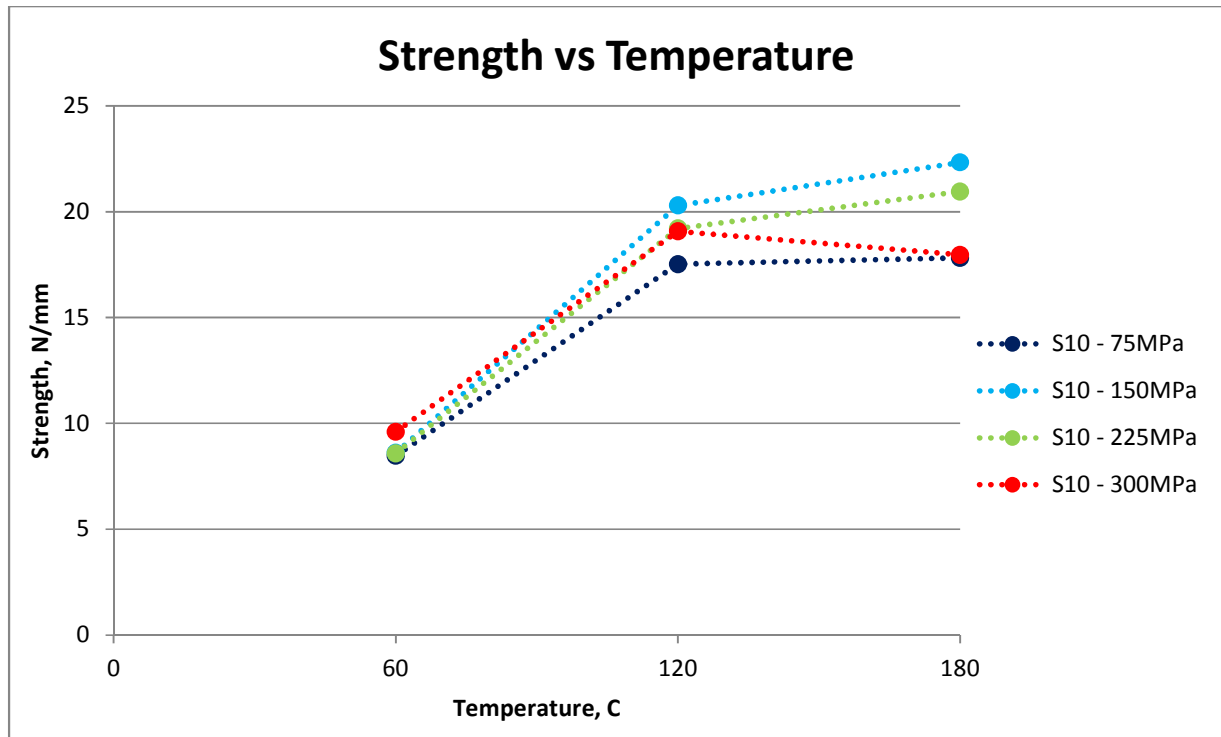


Figure 9-6a: Graph of Strength vs Temperature for S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.

The figure 9-6a above shows the graph for strength test conducted, for the S10 pellets at 75 MPa, 150 MPa, 225 MPa and 300 MPa (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The strength of the S10 pellets increased as the compacting temperature of the pellets increased. There is a discrepancy in strength for the S10 – 300 MPa at 180C where is strength drops below the strength of the S10 – 225 MPa and S10 – 225 MPa at 180C.

In fact the S10 – 150 MPa has the highest strength in the temperatures of 120C and above.

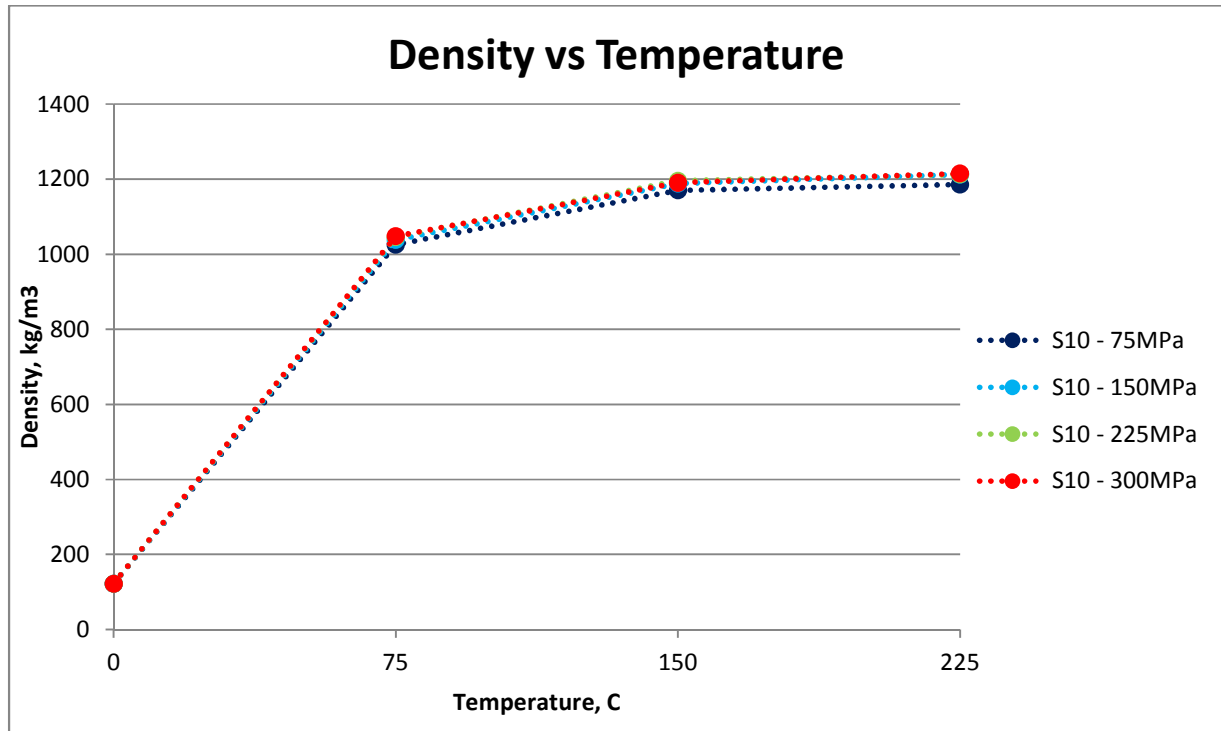


Figure 9-6b: Graph of Compressibility vs Temperature for S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.

The figure 9-6b above shows the compressibility graph, for the S10 pellets at 75 MPa, 150 MPa, 225 MPa and 300 MPa (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The density of the S10 pellets increased as the compacting temperature of the pellets increased.

The maximum attainable density of all the S10 pellets would plateau at the same maximum compressibility if extrapolated to a higher temperature (Possibly at 500C).

S10 pellets produced after 120C exhibited only minimal differences in the change in density for the any extra temperature exerted. In fact the S5 – 150 MPa, S5 – 225 MPa and S5 – 300 MPa has almost reached maximum compressibility after 150C.

S10 pellets produced after 150C exhibited only minimal differences in the change in density for the any extra temperature exerted.



**9.7 Discussion of S0 vs S5 vs S10 (Strength)**

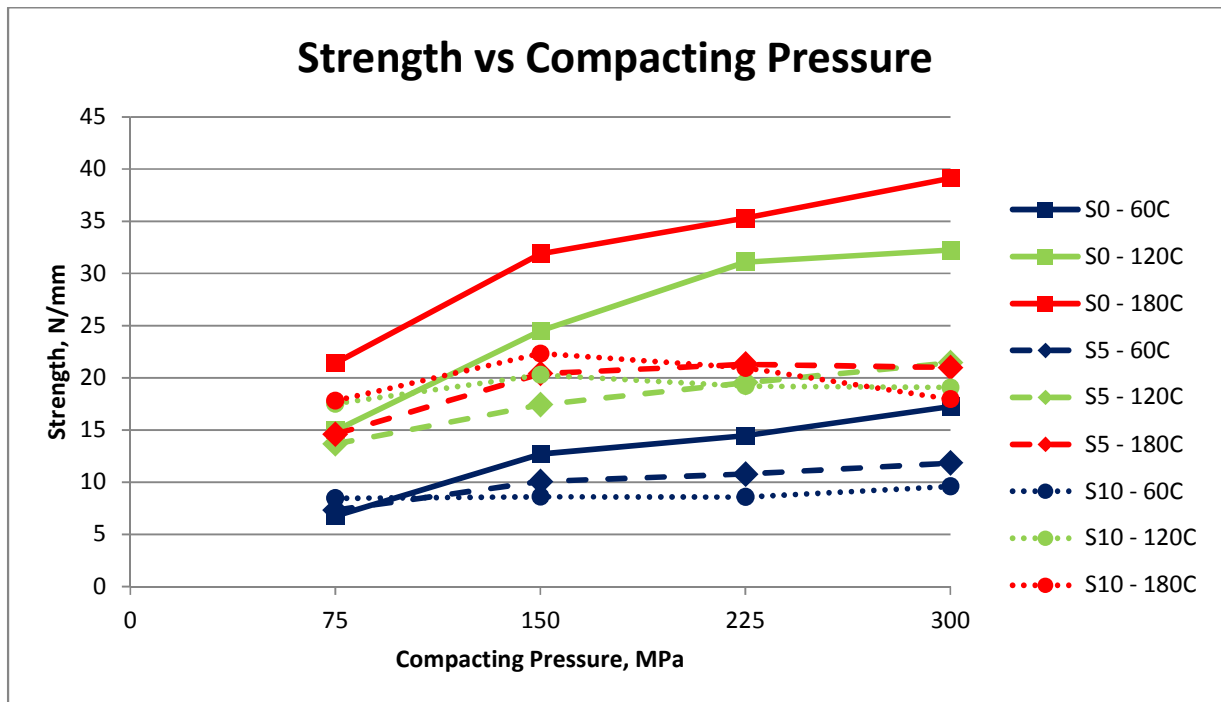


Figure 9-7a: Graph of Strength vs Pressure for S0, S5, S10 at 60C, 120C and 180C.

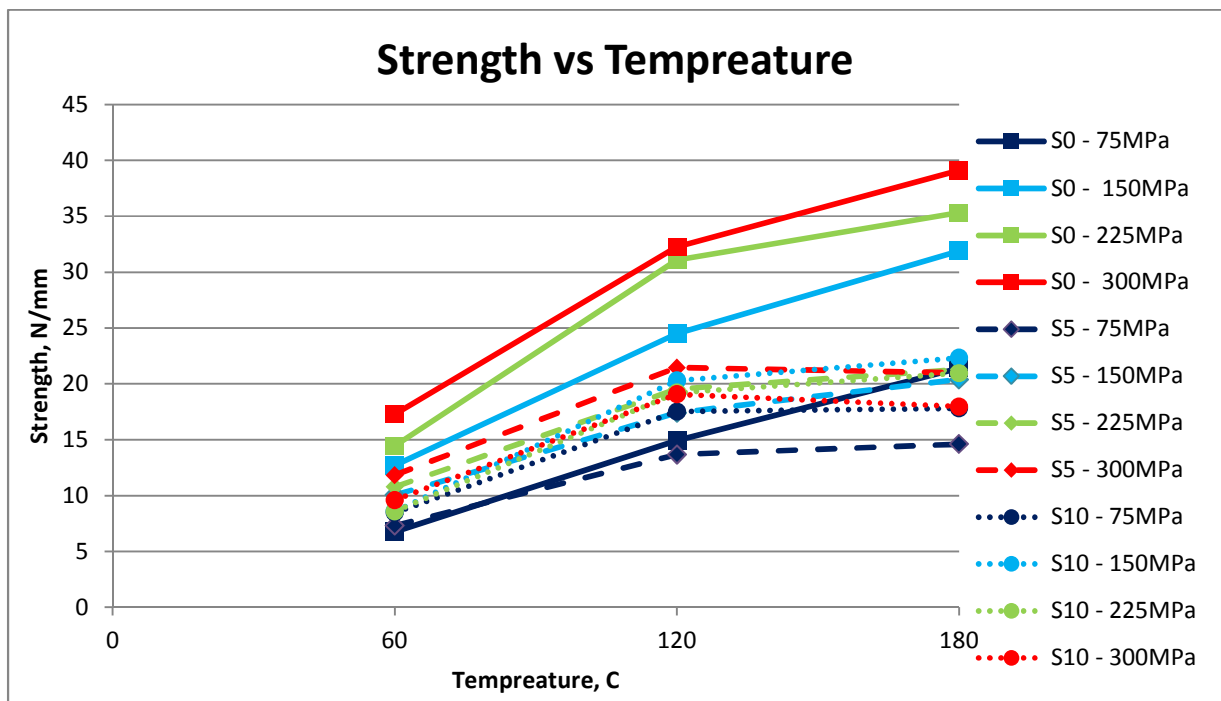


Figure 9-7b: Graph of Strength vs Temperature for S0, S5, S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.

The figure 9-7a above shows the graph for strength test conducted, for the S0, S5, S10 pellets at 60C, 120C and 180C (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The figure 9-6a above shows the graph for strength test conducted, for the S0, S5, S10 pellets at 75 MPa, 150 MPa, 225 MPa and 300 MPa (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

Figure 9-7a shows an increasing trend for strength as the compacting pressure increases for the pellets manufactured at similar oil concentration but varying temperature. The strength increases from S0-60C  $\rightarrow$  S0-120C  $\rightarrow$  S0-180C.

The result is explained as the lignin in the wood powders shows plasticization effect at higher temperatures [16]. The lignin is activated at temperature more than 60C, causing the wood fibre to form solid bridges. Heating also results plastic deformation of the fibres as compacting pressure is exerted on it, thus increasing particle contact as the distance between the particles is decreased. This increases the number of intermolecular attraction sites allowing the pellets to be strongly binded to one another [20, 21,23]. The pellets forms better mechanical interlocking after it cools down due to some plastic recovery. The plasticization effect is better at higher temperatures thus accounting to greater strength at higher temperatures [16, 20,23].

The increase in strength when compacting pressure increase is explained the powders are better compacted, the fibres distance is reduced and thus the intermolecular forces within the fibres are better held together to form a stronger and tighter bond which require a higher strength to break. The intermolecular forces include valance attractions, hydrogen bonding and van der Waals forces (VDW). VDW forces are assumed to be the most effective type of attraction in this case [20,23].

Mechanical interlocking between the fibres also can cause an increase in strength when the fibres are pressed against one another. Elastic recovery is also reduced after compression due to this type of mechanical interlocking [21,23].

Similar strength increases pattern is also observed for S5-60C  $\rightarrow$  S5-120C  $\rightarrow$  S5-180C, or for S10-60C  $\rightarrow$  S10-120C  $\rightarrow$  S10-180C with similar explanation.

Figure 9-7a also shows an increasing trend for strength as the compacting pressure increases for the pellets manufactured at similar temperature but varying oil concentration. The strength increases from S0-60C → S5-60C → S10-60C.

The addition of oil affects the interfacial forces and capillary pressure at the fibres. There are three states that represent this densification process. The first state, called the pendular state, is a state where the fibres acquire surface tension and capillary suction when the oil fills up the spaces previously occupied by the air. The second state, called an intermediate state, is a state where the quantity of the oil affects the agglomeration of particles. If too much oil is present at this state it will adversely affect the agglomeration of particles since liquids are incompressible while too little oil will not allow this state to fully develop. The third state, called the capillary state, is a state where the interfacial attraction between the fibres and the oil is dominant [16,23]. The oil content that is in: B10 > B5 > B0. Since oil is incompressible and its content is higher in B10 and B5 than in B0, the affected agglomeration of particles from the intermediate state, outweigh the increase of attractive forces by the small addition of oil. The agglomeration is affected since the oil prevents hydrogen bonding and dipole bonding to be formed between fibres [23, 24, 25, 26]. This leads to the drop in strength for the pellets with a larger quantity of oil content in them. Therefore the strength of the pellets is: S0 > S5 > S10.

The addition of oil may be also another source for the reduced strength because oil has a lower viscosity and this allows the fibres to easier slip if they are connect through mechanical interlocking and not chemical bonding [23, 26].

Similar strength increases pattern is also observed for S0-120C → S5-120C → S10-120C, or for S0-180C → S5-180C → S10-180C with similar explanation.

There are also some irregularities that can be identified from the graph. One such irregularity is the drop in strength for the S5 – 180 from 225 MPa to 300 MPa (Figure 9-3a). This can be accounted due to the loss of oil in the pellets due to higher pressure as discussed in Section 8 of this report. It can be observed that from Figure 9-3b that maximum density for the S5 – 180 has been attained between 150 and 225 MPa. Therefore any remaining oil will be forced out of the S5 – 180 pellets due to the increase in the compacting pressure. This oil however cannot be recovered during the recovery period when the compacting pressure is released since it has already left the pellet. Therefore there is lesser net quantity of oil in the S5 – 180 pellets at 300 MPa than the S5 – 180 pellets at 225 MPa. Thus the intermolecular forces acting on the S5 – 180, 300 MPa pellets is lesser than that which is acting on the S5 – 180, 225 MPa pellets, since removing oil will reduce the interfacial forces and capillary pressure [16, 23]. The drop in strength for the S10 – 180 from 225 MPa to 300 MPa (Figure 9-5a) can be explained similarly.

While another interesting irregularities lies with the drop in strength for the S10 – 300 MPa as opposed to the S10 – 150 MPa or S10 – 225 MPa at temperatures of 120C and above (Figure 9-6a). Similarly this can be accounted through the oil leakage as the pellets are being compressed with compacting pressure in the pellets as described above. This has led to a reduction in strength because of the reduction of the interfacial forces and capillary pressure [16, 23]. The S5 – 300 MPa possibly has lesser oil content than S10 – 150 MPa or S10 – 225 MPa, thus accounting to the lesser intermolecular bonding between the molecules [16, 20, 21]. In fact the S10 – 150 MPa has the highest strength in the temperatures of 120C and above. It probably indicates that after oil leakage the net oil that is left behind in the S10 – 150 MPa at 120C is the maximum saturation of oil in this range.

**9.8 Discussion of S0 vs S5 vs S10 (Compressibility)**

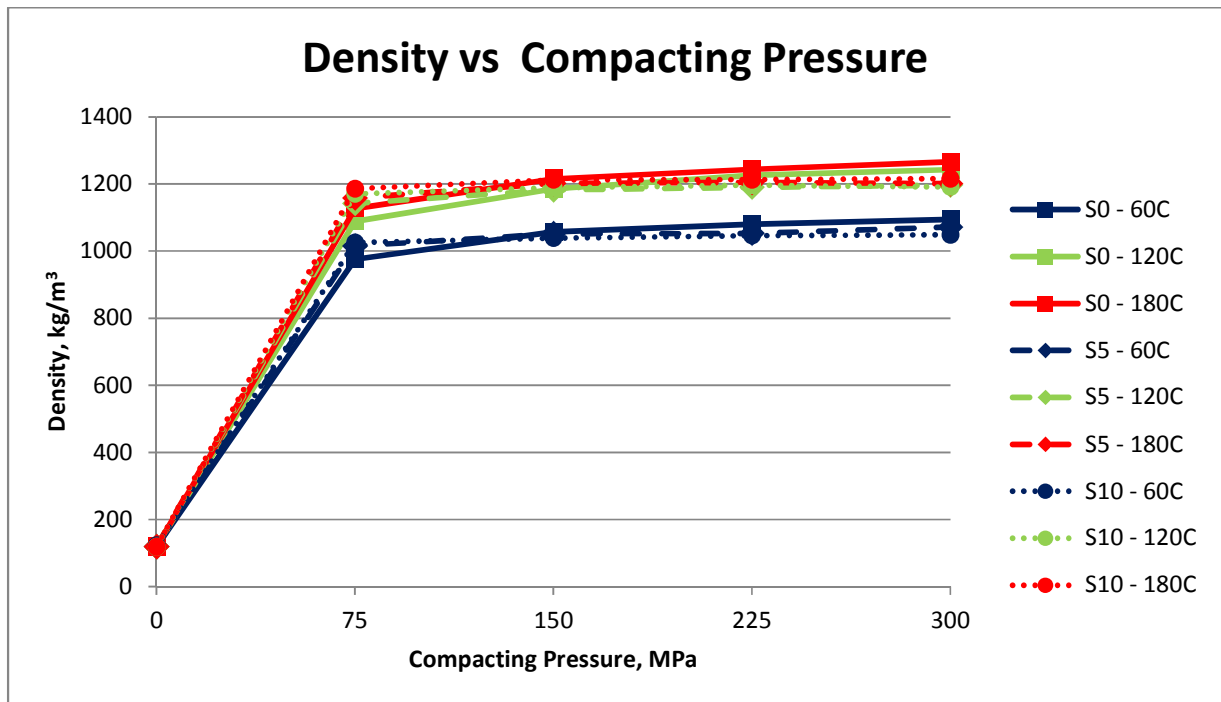


Figure 9-8a: Graph of Compressibility vs Pressure for S0, S5, S10 at 60C, 120C and 180C.

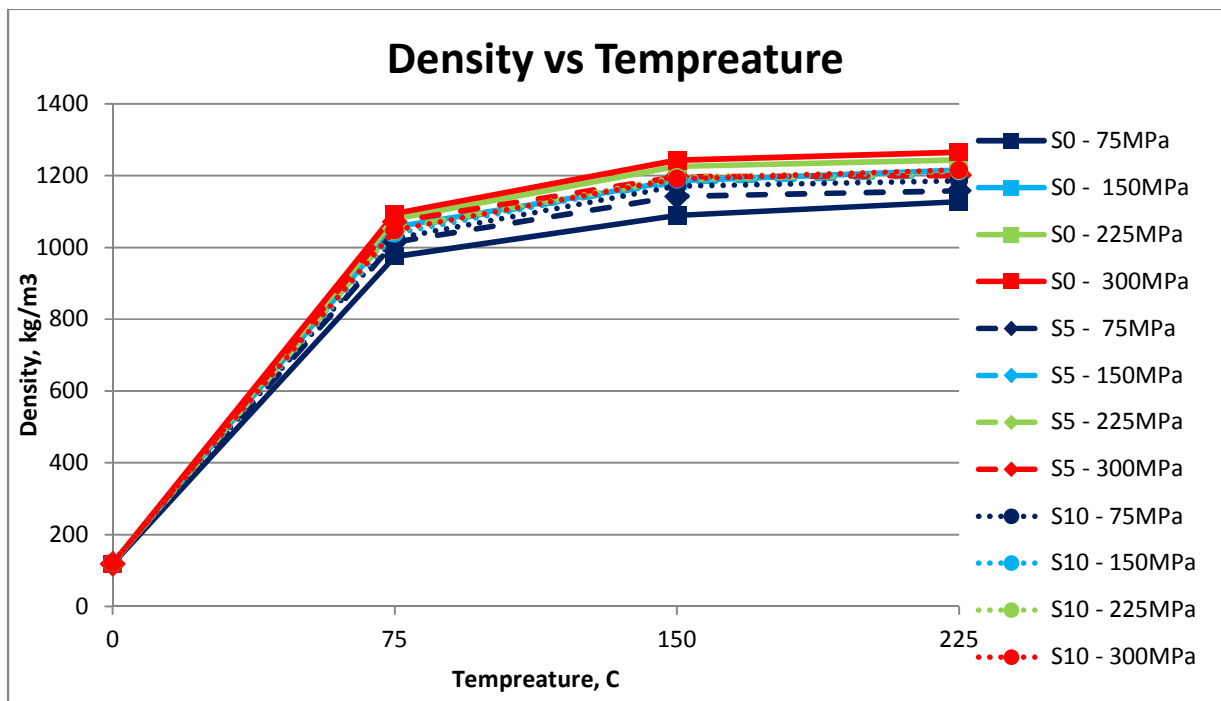


Figure 9-8b: Graph of Compressibility vs Temperature for S0, S5, S10 at 75 MPa, 150 MPa, 225 MPa and 300 MPa.

The figure 9-8a above shows the compressibility graph for the density studies, for the S0, S5, S10 pellets at 60C, 120C and 180C (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

The figure 9-8b above shows the compressibility graph for the density studies, for the S0, S5, S10 pellets at 75 MPa, 150 MPa, 225 MPa and 300 MPa (diameter  $\approx$  8 mm, mass  $\approx$  0,7~0,8g and length  $\approx$  12~14 mm).

Figure 9-7a shows an increasing trend for density as the compacting pressure increases for the pellets manufactured at similar oil concentration but varying temperature. The strength increases from S0-60C  $\rightarrow$  S0-120C  $\rightarrow$  S0-180C.

When the compacting pressure increases, the powders are better compacted and the fibres are closer to one another and this would result in an increase in intermolecular attraction between the fibres [20, 21, 23].

While pellets compressed at 60C are the least compressible, while pellets compressed at the higher temperatures are the most compressible which is accounted through the plasticization effect of lignin [16, 23].

In fact the S0 – 120 and S0 – 180 has already reached maximum compressibility, since the oil has occupied all the spaces in between the fibres and there is negligible space left to be compressed. The S0 – 60 is still has lower density because its lignin has not been properly plasticized since the temperature is lower than the glass transition temperature and therefore the solid bridges between the fibres have not been formed [16, 20, 21]. The fibres at this temperature are not properly binded to one another accounting for the reduced density in the pellets.

The fibres that were plasticized at the higher pressure (eg. 150 MPa, 225MPa and 300 MPa) will exhibit a plastic recovery, which thus will not change the density [20, 21].

There is also stronger binding due to plasticization effect at higher temperatures when solid bridges between the fibres are formed, stronger intermolecular forces and mechanical locking, when the wood particle bind closer to one another [16, 20, 21].

Similar density increases pattern is also observed for S5-60C → S5-120C → S5-180C, or for S10-60C → S10-120C → S10-180C with similar explanation.

### **9.9 Summary of Strength and Density of Pellets Produced**

It can also be concluded that maximum strength has been achieved at pressure of 150 MPa and 120 C for the S5 and S10 pellets, since we have compressed all the oil to the space between the pores and have conclusively binded almost all the fibres together. Any increase in pressure or temperatures does not significantly increase the strength. In fact increasing the pressure is counterproductive as it forces out the oil from the pellets and thus reduces the strength.

While it can also be concluded that the maximum compressibility has been achieved for most pellets at the temperature 150 MPa and 120C. Since increasing the pressure or temperatures beyond this point does not significantly alter the density of the pellets. In fact increasing the pressure is counterproductive as it forces out the oil from the pellets and thus reduces the density of the pellets, which in turn will affect its calorimetric values as show in Section 8.

## **10.0 Bulk Density**

Bulk density test was done simply using a 100 mL cylinder by weighing and computing the density of the S0, S5 and S10 powders.

	Average Bulk Density (kg/m <sup>3</sup> )	Standard Deviation
S0	119.44	1.54
S5	119.35	1.95
S10	122.05	1.81

Table 10-1: Table for B0, B5 and S10 Powder Bulk Density.

$$\text{Bulk Density, } \frac{g}{m^3} = \frac{\text{Mass of Powder}}{\text{Volume Occupied by Powder}}$$

The bulk density in the S0, S5 and S10 powders ranges from 119~122 kg/m<sup>3</sup> indicating the high porosity and low density existent between the particle.



## **11.0 Particle Distribution**

Particle distribution test was using a Malvern Mastersizer 3000 particle size analyser which computed the distribution of the particles in the powder. Particle distribution analysis was done in 25 cycles and the average if the 25 cycles is shown in the particle size distribution curve below(Figure 11-1).

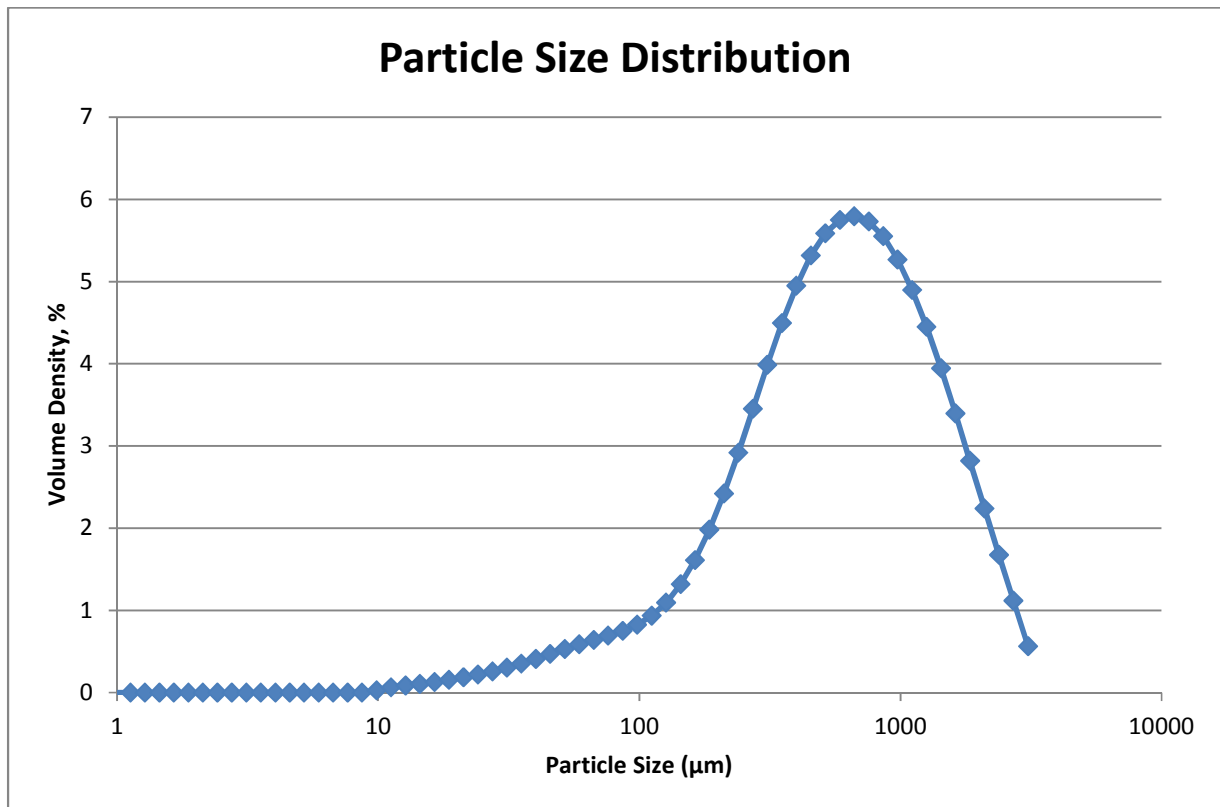


Figure 11-1: Figure of Particle Size Distribution for Powders.

Particle sized 600 ~ 1000 µm makes up the majority of the powder at ca 5-6% volume density, while there were almost negligible particle sized lesser than 10 µm and 5000 µm.

## **12.0 Moisture Content**

Moisture content analysis was done using a heater weighing and evaporating away the moisture content in existent in the S0 powder. The measurement was measured after the weight of the dry powder stabilized thus indicating maximum removal of water.

*Average Moisture Content*  $\approx$  10.72 %

Moisture content which are  $\geq$  12% are extremely efficient when combusted as they have lesser water content which reduces the combustion and thus the higher heating value [14, 32, 43].

## **13.0 Water Activity**

Water activity test was done for the S0, S5 and S10 powders and pellets by using a hygrometer and dew-point sensors.

### **13.1 S0**

	$a_w$	Standard Deviation
Powder	0.585	0.0083
S0 - 60 Pellets	0.504	0.0114
S0 - 120 Pellets	0.088	0.0112
S0 - 180 Pellets	0.021	0.0270

Table 13-1: Table for Water Activity in B0.

The S0 powder has  $a_w$  of 0.585, while the S0-180 has the lowest water activity level ( $a_w = 0.02$ ) and the S0-60 progressively having the highest water activity level ( $a_w = 0.51$ ) amongst the pellets. Water activity that is  $\geq 0.50$  has no microbial proliferation, indicating that the pellets will not have a microbial proliferation [54].

**13.2 S5**

	$a_w$	Standard Deviation
Powder	0.540	0.00656
S5 - 60 Pellets	0.452	0.00574
S5 - 120 Pellets	0.106	0.00265
S5 - 180 Pellets	0.0123	0.00287

Table 13-2: Table for Water Activity in B5.

The S5 powder has  $a_w$  of 0.54, while the S5-180 has the lowest water activity level ( $a_w = 0.01$ ) and the S5-60 progressively having the highest water activity level ( $a_w = 0.45$ ) amongst the pellets. Water activity that is  $\geq 0.50$  has no microbial proliferation, indicating that the pellets will not have a microbial proliferation [54].

**13.3 S10**

	$a_w$	Standard Deviation
Powder	0.461	0.01761
S10 - 60 Pellets	0.388	0.00896
S10 - 120 Pellets	0.136	0.00854
S10 - 180 Pellets	0.00375	0.00404

Table 13-3: Table for Water Activity in S10.

The S10 powder has  $a_w$  of 0.46, while the S10-180 has the lowest water activity level ( $a_w = 0.03$ ) and the S10-60 progressively having the highest water activity level ( $a_w = 0.39$ ) amongst the pellets. Water activity that is  $\geq 0.50$  has no microbial proliferation, indicating that the pellets will not have a microbial proliferation [54].

**13.4 S0 vs S5 vs S10**

The powders clearly have the highest water activity and this can be attributed to the higher surface area (“attack sites”) present in the powder to expose the moisture to the air. However the pellets which are made at higher temperature have the lowest water activity since some of the

moisture left the pellets during the compressing process leading to lesser site where the moisture can reside. The enhance binding of the lignin also reduces the moisture site microscopically in the pellets. The presence of oil also reduces the water activity as the sites are now occupied with oil rather than moisture.

#### **14.0 Ash Content**

Ash content test was done simply by heating the S0, S5 and S10 powders at high temperature (500 ~ 600°C) in a muffle furnace in the presence of oxidizing agents such as oxygen in the air.

	Ash Content	Standard Deviation
S0 - 60 Pellets	0.283	0.0193
S0 - 120 Pellets	0.229	0.00963
S0 - 180 Pellets	0.0211	0.0231

Table14-1: Table for Ash Content.

$$\text{Ash Content, \%} = \frac{\text{Mass of Dry Ash}}{\text{Mass of Powder Used}} \times 100$$

Low ash content ( $\geq 0.21$ ) indicates that the powder are mostly made up of organic compound and thus making it a very good combustible reactant [55].

## **15.0 Releasing Force**

The average force needed to release the pellet from the pelleting die after production is called the releasing force. It is important to investigate the releasing force because it will give a better picture of the total energy need to produce a pellet during pellet production.

### **15.1 Temperature 60C**

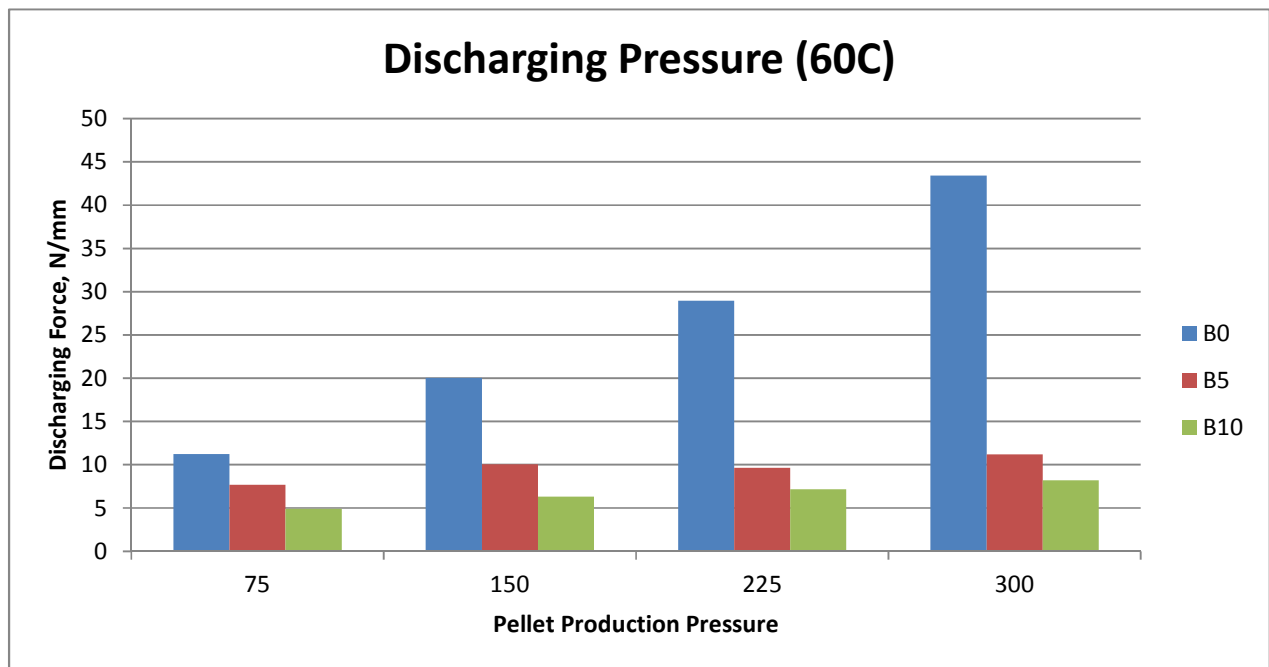


Figure 15-1: Graph for Releasing Force for B0, B5 and B10 Pellets at 60C.

Figure 15-1b shows a decreasing trend for the releasing force from S0 to S10.

### 15.2 Temperature 120C

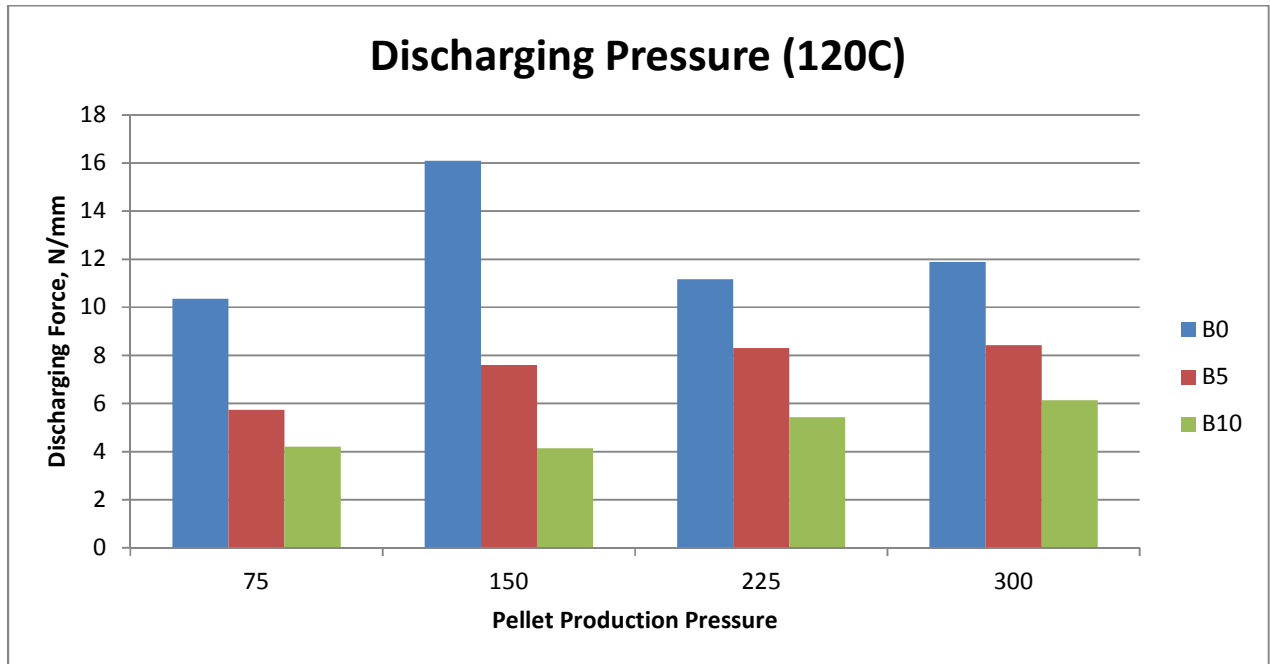


Figure 15-2: Graph for Releasing Force for B0, B5 and B10 Pellets at 120C.

Figure 15-2b shows a decreasing trend for the releasing force from S0 to S10.

### 15.3 Temperature 180C

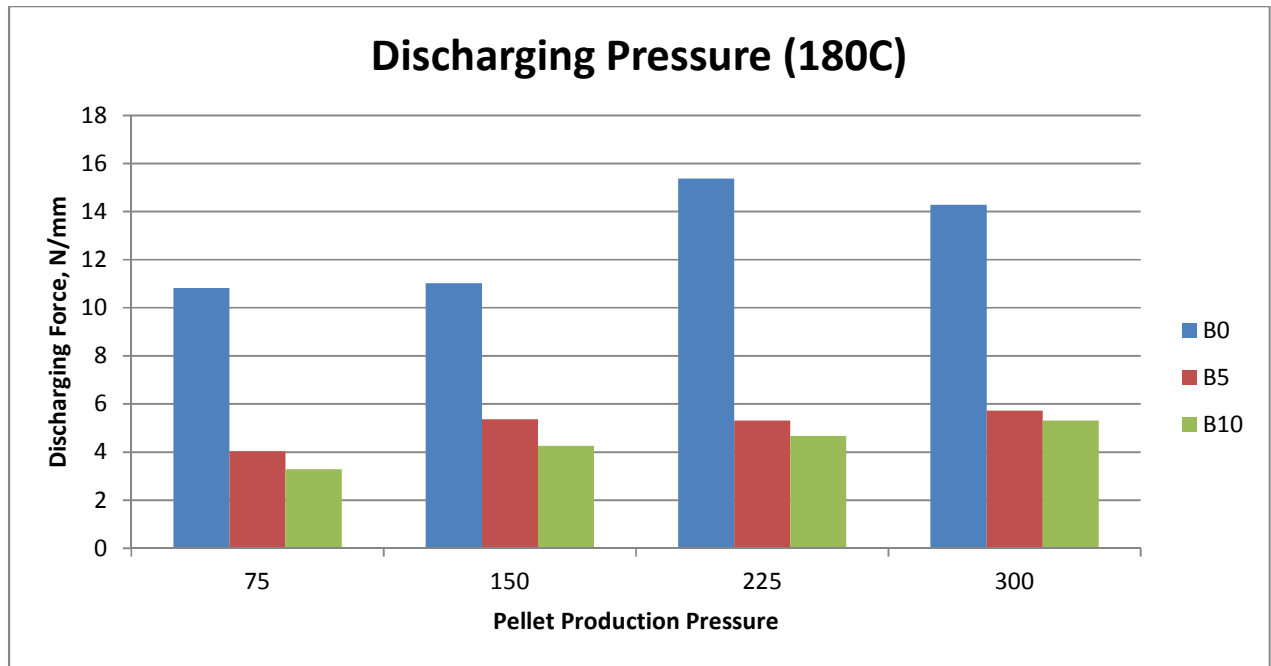


Figure 15-3: Graph for Releasing Force for B0, B5 and B10 Pellets at 180C.

Figure 15-3b shows a decreasing trend for the releasing force from S0 to S10.

## **16.0 Conclusion**

The physical and chemical properties for the S0, S5 and S10 pellets were analysed. Three different temperatures and four different pressures were used to examine these characteristics.

The calorific values for the S5 and S10 pellets were larger than the S0 pellets due to the addition of oil to it. While the strength of the S0 pellets were the largest followed by the S5 and the S10. Oil addition reduced the strength of the pellets significantly. Optimal pressure and temperature for any of the three pellets lied in the region of 150 MPa and 120C, since any addition of pressure or temperatures no longer changes the physical and chemical properties significantly.

The water activity result identifies that the pellets created at the highest pressure had significantly low water activity level while the powder has the highest water activity levels. Addition of oil also significantly reduced the water activity level.

The other values calculated such as the bulk density, particle distribution, moisture content and ash content showed generally similar trends for all the three S0, S5 and S10.



## **17.0 Reference**

1. Tjoelker, M. G. (2007). *Biology and ecology of Norway spruce*. Dordrecht: Springer.
2. Wang, X. Molecular phylogeny and biogeography of *Picea* (Pinaceae): Implications for phylogeographical studies using cytoplasmic haplotypes. *Molecular Phylogenetics and Evolution*, 405-419.
3. Peterson, E. B. (1997). *Ecology and management of Sitka spruce, emphasizing its natural range in British Columbia*. Vancouver: UBC Press.
4. Brackley, A. M., & Parrent, D. J. Production of wood pellets from Alaska-grown white spruce and hemlock. *U.S. Department of Agriculture, Forest Service, Pacific Northwest Research Station, J.*
5. *Handbook for Certification of Wood Pellets*. (2014). : European Pellet Council.
6. 12 Species of Spruce Trees and Shrubs. (n.d.). *About.com Trees & Shrubs*. Retrieved May 1
7. Spruce Tree, 2014, from <http://treesandshrubs.about.com/od/selection/tp/Spruce-Trees-and-Shrubs.htm>
8. Fromm, J.H. (2001). Xylem Water Content and Wood Density in Spruce and Oak Trees Detected by High-Resolution Computed Tomography
9. Gardiner, B. The regulation of sapwood area, water transport and heartwood formation in Sitka spruce. *Plant Ecology & Diversity*, 45-56.
10. . *NC Forestry*. Retrieved May 1, 2014, from <http://www.ncforestry.org>
11. Spruce Tree Names and types of *Picea* Species. (n.d.). *with pictures*. Retrieved May 1, 2014, from <http://www.treenames.net/ti/picea/picea.html>
12. Obernberger, I., & Thek, G. (2010). *The pellet handbook: the production and thermal utilisation of pellets*. London, UK: Earthscan.
13. Obernberger, I., & Thek, G. (2010). *The Pellet Handbook the Production and Thermal Utilization of Biomass Pellets.*. London: Earthscan.
14. Portero, H. Effect of moisture content, particle size and pine addition on quality parameters of barley straw pellets. *Fuel Processing Technology*, 699-706.
15. Felby, C. Effect of extractives and storage on the pelletizing process of sawdust. *Fuel*, 94-98.

16. Wilson, T. O., & Buffington, D. E. (2010). *Factors affecting wood pellet durability*. University Park, Pa.: Pennsylvania State University.
17. Mišljenović, N. Torrefaction Influence on Pelletability and Pellet Quality of Norwegian Forest Residues. *Energy & Fuels*, 140319151937005.
18. Salas-Bringas, C. Comparison between diametral and uniaxial compression tests of pelleted feed. *ANNUAL TRANSACTIONS OF THE NORDIC RHEOLOGY SOCIETY*, 19.
19. Salas-Bringas, C. Assessment of new laboratory die palleting rig attached to a texture analyzer to predict process ability of wood pellet.. *ANNUAL TRANSACTIONS OF THE NORDIC RHEOLOGY SOCIETY*, 18.
20. Salas-Bringas, C. Compression rheology and physical quality of wood pellets pre-handled with four different conditions. *ANNUAL TRANSACTIONS OF THE NORDIC RHEOLOGY SOCIETY*, 18.
21. Morey, R. V. Factors affecting strength and durability of densified biomass products. *Biomass and Bioenergy*, 337-359.
22. Henriksen, U. B. Pelletizing properties of torrefied spruce. *Biomass and Bioenergy*, 4690-4698.
23. Lestander, T. The influence of raw material characteristics on the industrial pelletizing process and pellet quality. *Fuel Processing Technology*, 1442-1447.
24. Guan, Y. Aromatic and Hydrophobic Surfaces of Wood-derived Biochar Enhance Perchlorate Adsorption via Hydrogen Bonding to Oxygen-containing Organic Groups. *Environmental Science & Technology*, 279-288.
25. Schneider, M. H. Hygroscopicity of wood impregnated with linseed oil. *Wood Science and Technology*, 107-114.
26. Selbo, M. L. (1975). *Adhesive bonding of wood*. Washington: U.S. Dept. of Agriculture.
27. Millett, M. A., & Gillespie, R. H. (1977). *Evaluating wood adhesives and adhesive bonds: performance requirements, bonding variables, bond evaluation, procedural recommendations*. Madison, Wis.: Forest Products Laboratory, Forest Service, U.S. Dept. of Agriculture.
28. Agriculture, U. D. (2011). *The Encyclopedia of Wood*. New York: Skyhorse Publishing, Inc..

29. Blomquist, R. F. (1983). *Adhesive bonding of wood and other structural materials*. University Park, Pa.: Educational Modules for Materials Science and Engineering (EMMSE) Project Materials Research Laboratory, the Pennsylvania State University.
30. Henriksen, U. B. Fuel pellets from biomass: The importance of the pelletizing pressure and its dependency on the processing conditions. *Fuel*, 3285-3290.
31. Rosillo-Calle F, Hemstock S, de Groot, P., Woods J. *The Biomass Assessment Handbook* London: Earthscan; 2007.
32. Simpson W, TenWolde A. Physical properties and moisture relations of wood. In: *Wood handbook - Wood as an engineering material*. Madison: United states department of agriculture - Forest Service - Forest products laboratory; 1999
33. Robbins WC. Density of wood chips. *J Forest* 1982;80:56.
34. Holm JK, Henriksen UB, Hustad JE, Sorensen LH. Toward an understanding of controlling parameters in softwood and hardwood pellets production. *Energy Fuels* 2006;20:2686-94.
35. Goring DAI. Thermal softening of lignin, hemicellulose and cellulose. *Pulp Paper Mag Canada* 1971;64:512-27.
36. Holm JK, Henriksen UB, Wand K, Hustad JE, Posselt D. Experimental verification of novel pellet model using a single pelleter unit. *Energy Fuels* 2007;21:2446-9.
37. Nielsen NPK, Holm JK, Felby C. Effect of fiber orientation on compression and frictional properties of sawdust particles in fuel pellet production. *Energy Fuels* 2009;23:3211-6.
38. Olsson AM, Salmen L. Viscoelasticity of insitu lignin as affected by structure – softwood vs hardwood. In: Glasser, W. editor. *Viscoelasticity of Biomaterials*. ACS Symposium Series 489. Washington: American Chemical Society 1992.p.133-43.
39. Irvine GM. The glass transitions of lignin and hemicellulose and their measurement by differential thermal-analysis. *Tappi J* 1984;67:118-21.
40. Nielsen NPK, Nørgaard L, Strobel BW, Felby C. Effect of storage on extractives from particle surfaces of softwood and hardwood raw materials for wood pellets. *Eur J Wood Prod* 2009;67:19-26.
41. Salmen L, Olsson AM. Interaction between hemicelluloses, lignin and cellulose: Structureproperty relationships. *J Pulp Paper Sci* 1998;24:99-103

42. Hatakeyama H, Hatakeyama T. Thermal properties of isolated and in situ lignin. In: Heitner C, Dimmel D, Schmidt J, editors. *Lignin and Lignans: Advances in Chemistry*. New York: CRC Press; 2010
43. Nielsen NPK, Gardner DJ, Poulsen T, Felby C. Importance of temperature, moisture content, and species for the conversion process of wood into fuel pellets. *Wood Fiber Sci* 2009;41:414-25.
44. Nielsen NPK, Gardner DJ, Felby C. Effect of extractives and storage on the pelletizing process of sawdust. *Fuel* 2010;89:94-8.
45. Salas-Bringas, C. Use of Strain Gauges to Estimate Pressure in Capillary Rheometers. *ANNUAL TRANSACTIONS OF THE NORDIC RHEOLOGY SOCIETY, 21*.
46. Tomani, P. The effects of kraft lignin additives on wood fuel pellet quality, energy use and shelf life. *Fuel Processing Technology*, 64-69.
47. Sanadi, A. R. Thermal transitions of the amorphous polymers in wheat straw. *Industrial Crops and Products*, 1053-1056.
48. Rowell, R. M. (2005). *Handbook of wood chemistry and wood composites*. Boca Raton, Fla.: CRC Press.
49. Moore, J. (2011). *Wood properties and uses of Sitka spruce in Britain*. Edinburgh: Forestry Commission.
50. Salas-Bringas, C. Strength of Wheat Gluten Pellets Made a Different Temperature, Moisture Content and Compacting Stress. *ANNUAL TRANSACTIONS OF THE NORDIC RHEOLOGY SOCIETY, 19*.
51. Lousada, J. Heating values of wood pellets from different species. *Biomass and Bioenergy*, 2634-2639.
52. Speight, J. G. (2011). *The biofuels handbook*. Cambridge: RSC Pub..
53. Francescato, V. (2009). *Wood Fuel Handbook* . : AEBIOM.
54. Water activity for product safety and quality.. (n.d.). *Water activity for product safety and quality*.. Retrieved May 1, 2014, from <http://www.aqualab.com/education/water-activity-for-product-safety-and-quality/>
55. *Evaluation of Gas, Oil and Wood Pellet Fueled Residential Heating System Emissions Characteristics*. (2009). Upton, N.Y.: Brookhaven National Laboratory.

56. Hubbard, W. C., & Partnership, I. (2007). *Sustainable forestry for bioenergy and bio-based products*. Athens, GA: Southern Forest Research Partnership, Inc.
57. Rhén, C., R. Gref, M. Sjöström, and I. Wästerlund, (2005), "Effects of raw material moisture content, densification pressure and temperature on some properties of Norway spruce pellets", *Fuel Processing Technology*, 87: p. 11-16.
58. Chemical Composition of Wood. (n.d.). Retrieved May 1, 2014, from [http://www.ipst.gatech.edu/faculty/ragauskas\\_art/technical\\_reviews/Chemical%20Overview%20of%20Wood.pdf](http://www.ipst.gatech.edu/faculty/ragauskas_art/technical_reviews/Chemical%20Overview%20of%20Wood.pdf)
59. Pellet Fuels Institute - What are Pellets?. (n.d.). *Pellet Fuels Institute - What are Pellets?*. Retrieved May 1, 2014, from <http://pelletheat.org/pellets/what-are-pellets/>
60. Hardwood VS Softwood. (n.d.). *Ferguson's Energy Systems*. Retrieved May 15, 2014, from <http://www.fergusonsenergysystems.com/FAQ.html>
61. *Biomass sustainability and carbon policy study*. (2010). Brunswick, Me.: Manomet Center for Conservation Sciences.
62. Walker, T. T. *Biomass Sustainability and Carbon Policy Study. Commonwealth of Massachusetts*.
63. Cocchi, M. M. *Global Wood Pellet Industry Market and Trade Study. IEA Bio Engineering*.
64. Malvern Mastersizer User Manual. (n.d.). Retrieved May 1, 2014, from <http://www.ceic.unsw.edu.au/centers/partcat/facilities/Mastersizer.pdf>

## **17.0 Appendix**

### **17.1 Pellet Strength and Compressibility**

Pellet Name			S0	
Compacting Pressure (MPa)	Temperature (C)		Strength (N/mm)	Standard Deviation
75	60		6.760634675	0.357208033
150	60		12.69023398	2.322714501
225	60		14.46046519	2.048655035
300	60		17.26515859	3.002474215
75	120		14.94276468	2.314278248
150	120		24.49991654	2.974815196
225	120		31.08419299	2.914981873
300	120		32.23987914	1.039480779
75	180		21.41225425	4.017509665
150	180		31.88885493	1.639796942
225	180		35.30838094	0.590091707
300	180		39.11282642	1.678799691

Table 17-1a: Table for S0 Pellets Strength

Pellet Name			S5	
Compacting Pressure (MPa)	Temperature (C)		Strength (N/mm)	Standard Deviation
75	60		7.317797987	1.001117548
150	60		10.05612832	0.823533196
225	60		10.78235042	0.158040509
300	60		11.8327147	0.71687447
75	120		13.6748813	1.012331583
150	120		17.41226291	1.364587959
225	120		19.54212141	2.137736883
300	120		21.4513275	1.155119973
75	180		14.60078031	2.035213767
150	180		20.39484798	1.273938387
225	180		21.29960832	1.485729898
300	180		20.98099591	1.208243977

Table 17-1b: Table for S5 Pellets Strength

Pellet Name			S10	
Compacting Pressure (MPa)	Temperature (C)		Strength (N/mm)	Standard Deviation
75	60		8.469462192	0.989376646
150	60		8.608540768	0.960054697
225	60		8.589184427	0.601296027
300	60		9.600527822	1.954513555
75	120		17.51814964	1.507288683
150	120		20.28966479	1.538374954
225	120		19.20207079	0.52830539
300	120		19.06960488	0.783231861
75	180		17.81139627	1.197920716
150	180		22.3261755	1.919503415
225	180		20.94946414	1.206649434
300	180		17.96121524	8.896334783

Table 17-1c: Table for S10 Pellets Strength

Pellet Name			S0	
Compacting Pressure (MPa)	Temperature (C)		Compressibility (kg/m <sup>3</sup> )	Standard Deviation
75	60		974.873909	16.64058244
150	60		1057.065394	11.35091082
225	60		1080.029785	14.78094783
300	60		1094.247853	15.01119604
75	120		1089.035168	10.7193626
150	120		1185.54612	20.82415226
225	120		1225.835003	14.4783415
300	120		1242.500675	4.533669338
75	180		1127.059672	19.94120403
150	180		1214.864625	10.16246938
225	180		1243.934222	8.54473318
300	180		1265.601392	2.125138893

Table 17-1d: Table for S0 Pellets Compressibility

Pellet Name			S5	
Compacting Pressure (MPa)	Temperature (C)		Compressibility (kg/m <sup>3</sup> )	Standard Deviation
75	60		1014.834679	22.65216983
150	60		1051.898174	15.96582158
225	60		1052.780855	7.087938364
300	60		1071.438938	2.318943792
75	120		1142.060248	5.35591281
150	120		1182.344455	4.243344298
225	120		1189.967105	8.852936904
300	120		1196.45486	4.076062424
75	180		1158.136556	5.921345966
150	180		1201.702793	5.823198989
225	180		1204.541533	9.693776174
300	180		1200.523152	9.631907296

Table 17-1e: Table for S5 Pellets Compressibility

Pellet Name			S10	
Compacting Pressure (MPa)	Temperature (C)		Compressibility (kg/m <sup>3</sup> )	Standard Deviation
75	60		1025.386593	23.41903708
150	60		1038.542987	12.72920138
225	60		1046.217446	10.16305544
300	60		1048.424881	29.46368548
75	120		1170.498511	4.535377327
150	120		1188.885902	13.63607492
225	120		1195.950827	8.243199637
300	120		1190.899553	3.849835867
75	180		1185.82389	22.37160946
150	180		1212.296612	7.480738971
225	180		1212.734553	5.817268956
300	180		1215.500683	7.210164978

Table 17-1f: Table for S10 Pellets Compressibility



**17.2 Energy Content****17.2.1 S0**

mass			0.9122	0.903	0.932 g
length before			10.6	10.3	10.4 cm
length after			2.2	2	2.1 cm
length used			8.4	8.3	8.3 cm
	time(min)	time(s)	temperature	temperature	temperature
	0	0	23.378	23.685	23.804
	1	60	23.369	23.652	23.798
	2	120	23.367	23.648	23.792
	3	180	23.363	23.642	23.784
	4	240	23.358	23.636	23.778
	5	300	23.354	23.632	23.774
	545	345	23.825	24.047	24.147
	6	360	24.082	24.347	24.479
	615	375	24.325	24.555	24.647
	630	390	24.469	24.628	24.841
	645	405	24.591	24.821	24.976
	7	420	24.73	24.935	25.12
	730	450	24.853	25.086	25.271
	8	480	24.965	25.196	25.393
	9	540	25.065	25.319	25.503
	10	600	25	25.365	25.554
	11	660	25	25.38	25.573
	12	720	25.135	25.388	25.582
	13	780	25.134	25.387	25.58
	14	840	25.13	25.385	25.578
	15	900	25.125	25.383	25.572
	16	960	25	25.378	25.567
	17	1020	25.112	25.373	25.558
	18	1080	25.105	25.367	25.55
$t = t_c - t_a - r_1 (b-a) - r_2 (c-b)$					
$H_g = (t W - e_1 - e_2 - e_3) / m$					
t			1.824981597	1.790967047	1.854
H <sub>g</sub>			4690.321927	4649.653815	4664.248927
Average H <sub>g</sub>		4668.075			
Standard Deviation H <sub>g</sub>		20.60224			

Figure 17-2-1: Figure for S0 Pellets Energy Content

Figure 17-2-1 shows calorimetric value (Higher Heating Value) of,

Higher Heating Value, H<sub>g</sub>,(cal/g) : 4668 cal/g and a standard deviation of ± 20.6 cal/g.

Higher Heating Value, H<sub>g</sub>, (MJ/Kg) : 117.154 MJ/Kg and a standard deviation of ± 0.087 MJ/Kg.

### 17.2.2 S5

mass			0.8659	0.6637	0.8969 g
length before			10.1	10.2	10.2 cm
length after			2	2.3	2.3 cm
length used			8.1	7.9	7.9 cm
	time(min)	time(s)	temperature	temperature	temperature
	0	0	19.228	20.965	21.558
	1	60	19.232	20.964	21.544
	2	120	19.235	20.964	21.535
	3	180	19.237	20.962	21.528
	4	240	19.238	20.96	21.522
	5	300	19.239	20.958	21.518
	545	345	19.652	21.221	21.892
	6	360	19.982	21.435	22.188
	615	375	20.115	21.582	22.407
	630	390	20.302	21.719	22.597
	645	405	20.441	21.826	22.717
	7	420	20.534	21.877	22.851
	730	450	20.68	22.03	23.002
	8	480	20.785	22.117	23.103
	9	540	20.901	22.213	23.226
	10	600	20.96	22.254	23.277
	11	660	20.988	22.276	23.296
	12	720	21.002	22.285	23.305
	13	780	21.009	22.287	23.305
	14	840	21.011	22.286	23.301
	15	900	21.01	22.284	23.297
	16	960	21.009	22.28	23.29
	17	1020	21.007	22.278	23.283
	18	1080	21.005	22.273	
$t = t_c - t_a - r_1(b-a) - r_2(c-b)$					
$H_g = (t W - e_1 - e_2 - e_3) / m$					
t			1.771904011	1.349180935	1.827567789
Hg			4797.556237	4759.908243	4778.40578
Average Hg		4778.623			
Standard Deviation Hg		18.82494			

Figure 17-2-2: Figure for B5 Pellets Energy Content

Figure 17-2-2 shows calorimetric value (Higher Heating Value) of,

Higher Heating Value, Hg, (cal/g) : 4778 cal/g and a standard deviation of  $\pm 18.8$  cal/g.

Higher Heating Value, Hg, (MJ/Kg) : 20.00 MJ/Kg and a standard deviation of  $\pm 0.079$  MJ/Kg.

**17.2.3 S10**

mass			0.9504	0.8887	0.9395 g
length before			10.6	10.7	10.2 cm
length after			2.5	1.8	2.1 cm
length used			8.1	8.9	8.1 cm
	time(min)	time(s)	temperature	temperature	temperature
	0	0	22.886	24.034	25.115
	1	60	22.887	24.029	25.105
	2	120	22.889	24.025	25.099
	3	180	22.89	24.023	25.095
	4	240	22.887	24.019	25.085
	5	300	22.885	24.015	25.08
	545	345	23.391	24.422	25.576
	6	360	23.693	24.73	25.833
	615	375	23.941	25.025	26.103
	630	390	24.132	25.123	26.229
	645	405	24.259	25.271	26.414
	7	420	24.348	25.392	26.527
	730	450	24.56	25.553	26.701
	8	480	24.633	25.655	26.809
	9	540	24.784	25.77	26.93
	10	600	24.842	25.82	26.97
	11	660	24.863	25.845	26.993
	12	720	24.873	25.85	26.997
	13	780	24.875	25.85	26.995
	14	840	24.872	25.849	26.99
	15	900	24.871	25.845	26.985
	16	960	24.867	25.84	26.977
	17	1020	24.863	25.835	26.97
	18	1080	24.856		
$t = t_c - t_a - r_1(b-a) - r_2(c-b)$					
$H_g = (t W - e_1 - e_2 - e_3) / m$					
t			2.015249738	1.860185714	1.96060381
H <sub>g</sub>			4973.993196	4906.343375	4894.722694
Average H <sub>g</sub>		4925.02			
Standard Deviation H <sub>g</sub>		42.80839			

Figure 17-2-3: Figure for B10 Pellets Energy Content

Figure 17-2-3 shows calorimetric value (Higher Heating Value) of,

Higher Heating Value, H<sub>g</sub>,(cal/g) : 4925 cal/g and a standard deviation of ± 42.8 cal/g.

Higher Heating Value, H<sub>g</sub>, (MJ/Kg) : 20.61 MJ/Kg and a standard deviation of ± 0.18 MJ/Kg.

**17.2.4 S5 Cold Pressed (CP)**

mass			0.9482	1.0032	1.013 g
length before			10	9.9	10.3 cm
length after			3	1.6	3.2 cm
length used			7	8.3	7.1 cm
	time(min)	time(s)	temperature	temperature	temperature
	0	0	20.65	22.108	23.942
	1	60	20.658	22.104	23.939
	2	120	20.663	22.1	23.937
	3	180	20.665	22.102	23.934
	4	240	20.669	22.099	23.932
	5	300	20.672	22.096	23.928
	545	345	21.104	22.423	24.282
	6	360	21.399	22.89	24.734
	615	375	21.657	23.095	24.955
	630	390	21.804	23.285	25.127
	645	405	21.952	23.469	25.319
	7	420	22.103	23.599	25.471
	730	450	22.269	23.757	25.596
	8	480	22.395	23.897	25.731
	9	540	22.514	24.025	25.864
	10	600	22.576	24.075	25.934
	11	660	22.605	24.105	25.959
	12	720	22.62	24.115	25.979
	13	780	22.627	24.12	25.986
	14	840	22.629	24.118	25.983
	15	900	22.629	24.115	25.981
	16	960	22.627	24.11	25.978
	17	1020	22.626	24.105	25.974
	18	1080	22.624	24.101	25.972
t = tc - ta - r1 (b-a) - r2 (c-b)					
Hg = (t W - e1 - e2 - e3) / m					
t			1.951953716	2.054051685	2.0804
Hg			4830.996627	4802.832653	4820.347483
Average Hg		4818.059			
Standard Deviation Hg		14.22078			

Figure 17-2-4: Figure for B5 Cold Pressed Pellets Energy Content

Figure 17-2-4 shows calorimetric value (Higher Heating Value) of,

Higher Heating Value, Hg, (cal/g) : 4818 cal/g and a standard deviation of  $\pm 14.2$  cal/g.

Higher Heating Value, Hg, (MJ/Kg) : 20.17 MJ/Kg and a standard deviation of  $\pm 0.06$  MJ/Kg.

**17.2.5 S10 Cold Pressed (CP)**

mass			0.6134	0.7804	0.7752 g
length before			10.3	10.2	10.1 cm
length after			2.2	3.7	2.3 cm
length used			8.1	6.5	7.8 cm
	time(min)	time(s)	temperature	temperature	temperature
	0	0	20.853	21.845	23.852
	1	60	20.855	21.84	23.853
	2	120	20.857	21.837	23.854
	3	180	20.86	21.836	23.856
	4	240	20.862	21.835	23.857
	5	300	20.864	21.835	23.857
	545	345	21.202	22.229	24.252
	6	360	21.37	22.525	24.556
	615	375	21.504	22.67	24.691
	630	390	21.618	22.845	24.866
	645	405	21.716	22.956	24.977
	7	420	21.804	22.054	25.073
	730	450	21.889	23.2	25.221
	8	480	21.98	23.278	25.301
	830	510	22.038	23.34	25.36
	9	540	22.073	23.382	25.402
	10	600	22.116	23.426	25.448
	11	660	22.139	23.448	25.472
	12	720	22.151	23.46	25.48
	13	780	22.158	23.463	25.485
	14	840	22.161	23.465	25.487
	15	900	22.162	23.464	25.486
	16	960	22.162	23.463	25.485
	17	1020	22.163	23.46	25.481
	18	1080	22.162	23.458	25.479
	19	1140	22.162	23.457	25.478
	$t = t_c - t_a - r_1(b-a) - r_2(c-b)$				
	$Hg = (t W - e_1 - e_2 - e_3) / m$				
t			1.292076531	1.644981714	1.642124
Hg			4930.241652	4944.876906	4965.508282
Average Hg		4946.876			
Standard Deviation		17.71807			

Figure 17-2-5: Figure for B10 Cold Pressed Pellets Energy Content

Figure 17-2-5 shows calorimetric value (Higher Heating Value) of,

Higher Heating Value, Hg,(cal/g) : 4986 cal/g and a standard deviation of  $\pm 17.71$  cal/g.

Higher Heating Value, Hg, (MJ/Kg) : 20.88 MJ/Kg and a standard deviation of  $\pm 0.074$  MJ/Kg.

**17.2.6 S5 150 MPa/ 120C**

mass			0.6134	0.7804	0.7752 g
length before			10.3	10.2	10.1 cm
length after			2.2	3.7	2.3 cm
length used			8.1	6.5	7.8 cm
	time(min)	time(s)	temperature	temperature	temperature
	0	0	20.853	21.845	23.852
	1	60	20.855	21.84	23.853
	2	120	20.857	21.837	23.854
	3	180	20.86	21.836	23.856
	4	240	20.862	21.835	23.857
	5	300	20.864	21.835	23.857
	545	345	21.202	22.229	24.252
	6	360	21.37	22.525	24.556
	615	375	21.504	22.67	24.691
	630	390	21.618	22.845	24.866
	645	405	21.716	22.956	24.977
	7	420	21.804	22.054	25.073
	730	450	21.889	23.2	25.221
	8	480	21.98	23.278	25.301
	830	510	22.038	23.34	25.36
	9	540	22.073	23.382	25.402
	10	600	22.116	23.426	25.448
	11	660	22.139	23.448	25.472
	12	720	22.151	23.46	25.48
	13	780	22.158	23.463	25.485
	14	840	22.161	23.465	25.487
	15	900	22.162	23.464	25.486
	16	960	22.162	23.463	25.485
	17	1020	22.163	23.46	25.481
	18	1080	22.162	23.458	25.479
	19	1140	22.162	23.457	25.478
	t = tc - ta - r1 (b-a) - r2 (c-b)				
	Hg = (t W - e1 - e2 - e3) / m				
t			1.292076531	1.644981714	1.642124
Hg			4930.241652	4944.876906	4965.508282
Average Hg	4946.876				
Standard Deviation	17.71807				

Figure 17-2-6: Figure for B5 150 MPa/ 120C Pellets Energy Content

Figure 17-2-6 shows calorimetric value (Higher Heating Value) of,

Higher Heating Value, Hg, (cal/g) : 4769 cal/g and a standard deviation of ± 15.48 cal/g.  
 Higher Heating Value, Hg, (MJ/Kg) : 117.197 MJ/Kg and a standard deviation of ± 0.065 MJ/Kg.

**17.2.7 S10 150 MPa/ 120C**

mass			1.044	0.9562	0.9926 g
length before			10.4	10.4	9.9 cm
length after			3.5	5.9	2.3 cm
length used			6.9	4.5	7.6 cm
	time(min)	time(s)	temperature	temperature	temperature
	0	0	23.547	25.086	26.634
	1	60	23.545	25.084	26.629
	2	120	23.544	25.076	26.627
	3	180	23.543	25.071	26.626
	4	240	23.541	25.064	26.624
	5	300	23.537	25.057	26.62
	545	345	24.046	25.54	27.141
	6	360	24.301	25.822	27.442
	615	375	24.595	26.095	27.727
	630	390	24.847	26.27	27.906
	645	405	24.987	26.433	28.059
	7	420	25.139	26.558	28.191
	730	450	25.349	26.719	28.342
	8	480	25.46	26.817	28.427
	9	540	25.602	26.93	28.502
	10	600	25.649	26.973	28.554
	11	660	25.674	26.899	28.586
	12	720	25.682	26.991	28.659
	13	780	25.683	26.987	28.65
	14	840	25.679	26.978	28.643
	15	900	25.673	26.97	28.638
	16	960	25.664	26.959	28.631
	17	1020	25.657	26.948	28.624
	18	1080	25.648		
t = tc - ta - r1 (b-a) - r2 (c-b)					
Hg = (t W - e1 - e2 - e3) / m					
t			2.194611111	1.9902104	2.08212352
Hg			4935.286558	4890.813106	4922.346251
Average Hg		4916.149			
Standard Deviation Hg		22.87531			

Figure 17-2-7: Figure for B10 150 MPa/ 120C Pellets Energy Content

Figure 17-2-7 shows calorimetric value (Higher Heating Value) of,

Higher Heating Value, Hg, (cal/g) : 4916 cal/g and a standard deviation of  $\pm 22.88$  cal/g.

Higher Heating Value, Hg, (MJ/Kg) : 20.56 MJ/Kg and a standard deviation of  $\pm 0.096$  MJ/Kg.

**17.2.8 Oil**

mass			1.3682	1.02255 g
lenght before			10.6	10.2 cm
lenght after			6	3.4 cm
lenghtused			4.6	6.8 cm
	time(min)	time(s)	tempreature	tempreature
	0	0	23.184	25.031
	1	60	23.178	25.017
	2	120	23.169	25.003
	3	180	23.163	25.001
	4	240	23.155	24.977
	5	300	23.148	24.966
	545	345	23.71	25.772
	6	360	23.774	26.305
	615	375	24.779	26.791
	630	390	25.26	27.186
	645	405	25.648	27.56
	7	420	26.811	27.87
	730	450	27.478	28.256
	8	480	27.845	28.453
	9	540	28.133	28.664
	10	600	28.249	28.741
	11	660	28.282	28.773
	12	720	28.294	28.774
	13	780	28.287	28.767
	14	840	28.274	28.755
	15	900	28.259	28.737
	16	960	28.242	28.721
	17	1020	28.221	
t = tc - ta - r1 (b-a) - r2 (c-b)				
Hg = (t W - e1 - e2 - e3) / m				
t			5.234315297	3.873411578
Hg			9004.676008	8908.296206
Average Hg		8980.61		
Standard Deviation Hg		68.15081		

Figure 17-2-8: Figure for Oil Energy Content

Figure 17-2-8 shows calorimetric value (Higher Heating Value) of,

Higher Heating Value, Hg,(cal/g) : 8980.6 cal/g and a standard deviation of ± 68.15 cal/g.

Higher Heating Value, Hg, (MJ/Kg) : 37.60 MJ/Kg and a standard deviation of ±0.285MJ/Kg.



**17.3 Bulk Density**

Bulk density test was done simply using a 100 mL cylinder by weighing and computing the density of the S0, S5 and S10 powders.

S0				
Empty Container	Full Container	Difference	Volume	Bulk Density
108.11	117.67	9.56	81	118.0246914
108.11	118.16	10.05	83	121.0843373
108.11	118.6	10.49	88	119.2045455
		Average Bulk Density		119.4378581
		Standard Deviation		1.543108705
S5				
Empty Container	Full Container	Difference	Volume	Bulk Density
108.1	117.67	9.57	79	121.1392405
108.1	118.49	10.39	86	120.8139535
108.1	118.99	10.89	92	118.3695652
108.1	118.99	10.89	93	117.0967742
		Average Bulk Density		119.3548834
		Standard Deviation		1.94788132
S10				
Empty Container	Full Container	Difference	Volume	Bulk Density
108.1	118.39	10.29	85	121.0588235
108.1	120.02	11.92	99	120.4040404
108.1	119.1	11	90	122.2222222
108.1	118.31	10.21	82	124.5121951
		Average Bulk Density		122.0493203
		Standard Deviation		1.805886417

Figure 17-3: Figure for B0, B5 and B10 Powder Bulk Density

### **17.4 Moisture Content**

Moisture Content of Conditioned Sawdust				
	1	2	3	
Mass of Empty Crucible	21.2148	22.941	22.6368	
Mass of Crucible with Moist Powder	22.5324	24.2048	23.9365	
Mass of Crucible with Dry Powder	22.3981	24.0656	23.7941	
% of moisture	10.19277	11.0144	10.95637	
	Average Moisture Content, %			10.72118

Figure 17-4: Figure of Moisture Content in Powders

## 17.4 Water Activity

Water activity test was done for the S0, S5 and S10 powders and pellets by using a hygrometer and dew-point sensors.

### 17.4.1 S0

S0 Powder			B0 - 60		
	aw	Temperature (C)		aw	Temperature (C)
	0.578	20.01	75	0.496	19.07
	0.594	20.01	150	0.492	19.08
	0.582	20.01	225	0.514	19.05
			300	0.513	19.06
Average aw		0.584666667	Average aw		0.50375
Standard Deviation		0.008326664	Standard Deviation		0.011383468
B0 - 120			B0 - 180		
	aw	Temperature (C)		aw	Temperature (C)
75	0.079	19.5	75	0.016	19.57
150	0.078	19.38	150	0.004	19.61
225	0.095	19.58	225	0.002	19.53
300	0.1	19.48	300	0.06	19.56
Average aw		0.088	Average aw		0.0205
Standard Deviation		0.011165423	Standard Deviation		0.027049338

Figure 17-4-1: Figure for Water Activity in B0

**17.4.2 S5**

S5 Powder			B5 - 60		
	aw	Temperature (C)		aw	Temperature (C)
	0.534	20.03	75	0.445	19.47
	0.539	20.09	150	0.453	19.78
	0.547	20.14	225	0.459	19.8
			300	0.452	19.98
Average aw		0.54	Average aw		0.45225
Standard Deviation		0.006557439	Standard Deviation		0.005737305
B5 - 120			B5 - 180		
	aw	Temperature (C)		aw	Temperature (C)
75	0.109	19.66	75	0.016	20.08
150	0.103	19.83	150	0.012	20.18
225	0.106	19.92	225	0.012	20.28
300	0.104	19.99	300	0.009	20.37
Average aw		0.1055	Average aw		0.01225
Standard Deviation		0.002645751	Standard Deviation		0.002872281

Figure 17-4-2: Figure for Water Activity in B5

**17.4.3 S10**

S10 Powder			B10 - 60		
	aw	Temperature (C)		aw	Temperature (C)
	0.445	20.12	75	0.387	20.01
	0.48	20.12	150	0.384	20.08
	0.459	20.14	225	0.379	20.11
			300	0.4	20.13
Average aw		0.461333	Average aw		0.3875
Standard Deviation		0.017616	Standard Deviation		0.008963
B10 - 120			B10 - 180		
	aw	Temperature (C)		aw	Temperature (C)
75	0.131	18.1	75	0.035	17.4
150	0.137	18.2	150	0.043	17.56
225	0.129	18.27	225	0.038	17.63
300	0.148	18.36	300	0.034	17.7
Average aw		0.13625	Average aw		0.0375
Standard Deviation		0.008539	Standard Deviation		0.004041

Figure 17-4-3: Figure for Water Activity in B10

**17.5 Ash Content**

Ash content test was done simply by heating the S0, S5 and S10 powders at high temperature (500 ~ 600°C) in a muffle furnace in the presence of oxidizing agents such as oxygen in the air.

S0					
	1	2	3	4	5
Mass of Empty Crucible	22.5681	23.1582	22.5681	24.884	23.1583
Mass of Crucible with Powder	23.699	24.3769	23.397	25.7332	24.019
Mass of Crucible with Ash	22.571	23.1615	22.5705	24.8866	23.1608
Ash Content, %	0.256433	0.27078	0.28954	0.306171	0.290461
			Average Ash Content, %		0.282677
			Standard Deviation		0.0193
S5					
	1	2	3		
Mass of Empty Crucible	22.5687	24.8846	22.9095		
Mass of Crucible with Powder	23.5219	26.0624	23.9154		
Mass of Crucible with Ash	22.5709	24.8874	22.9117		
Ash Content, %	0.230802	0.237731	0.21871		
			Average Ash Content, %		0.229081
			Standard Deviation		0.009627
S10					
	1	2	3		
Mass of Empty Crucible	23.1589	22.0301	23.222		
Mass of Crucible with Powder	24.3995	23.1686	24.3059		
Mass of Crucible with Ash	23.1614	22.0328	23.2241		
Ash Content, %	0.201515	0.237154	0.193745		
			Average Ash Content, %		0.210805
			Standard Deviation		0.023148

Figure 17-5: Figure for Ash Content

## **17.6 Releasing Force**

### **17.6.1 Temperature 60C**

Temperature 60 C			
Pressure(MPa)	Pellets Name	Discharging Force(N/mm)	Standard Deviation
75	B0	11.2453328	4.124383729
150	B0	20.0459532	6.756336939
225	B0	28.94632627	5.472043834
300	B0	43.39551617	10.52455265
75	B5	7.706188316	1.243951549
150	B5	10.03384467	3.238305573
225	B5	9.658071305	1.155883697
300	B5	11.18783513	1.761318838
75	B10	4.957440118	0.463085945
150	B10	6.33680533	0.571654249
225	B10	7.155348615	0.619994484
300	B10	8.203731454	1.498086004

Figure 17-6-1: Figure for Releasing Force for B0, B5 and B10 Pellets at 60C

### **17.6.2 Temperature 120C**

Temperature 120 C			
Pressure(MPa)	Pellets Name	Discharging Force(N/mm)	Standard Deviation
75	B0	10.35649688	1.762309564
150	B0	16.0872851	5.613174874
225	B0	11.17219018	1.788846849
300	B0	11.88141083	0.81868148
75	B5	5.743644472	1.244180199
150	B5	7.60793007	1.018409471
225	B5	8.301378137	1.395122814
300	B5	8.426106161	0.89274608
75	B10	4.212026454	0.614854056
150	B10	4.134369655	1.368717892
225	B10	5.432005277	0.580032397
300	B10	6.142707391	0.267652525

Figure 17-6-2: Figure for Releasing Force for B0, B5 and B10 Pellets at 120C

### **17.6.3 Temperature 180C**

Temperature 180 C			
Pressure(MPa)	Pellets Name	Discharging Force(N/mm)	Standard Deviation
75	B0	10.81758116	3.15416639
150	B0	11.02610677	2.360590716
225	B0	15.36433465	1.736744512
300	B0	14.28114191	2.137483039
75	B5	4.02906572	0.543878669
150	B5	5.362902173	0.498846314
225	B5	5.320708485	0.844256751
300	B5	5.725668683	0.904617376
75	B10	3.300454979	0.317725415
150	B10	4.260463009	0.370110897
225	B10	4.663994036	0.626963164
300	B10	5.309276148	0.463432514

Figure 17-6-3: Figure for Releasing Force for B0, B5 and B10 Pellets at 180C





Norwegian University  
of Life Sciences

Postboks 5003  
NO-1432 Ås, Norway  
+47 67 23 00 00  
[www.nmbu.no](http://www.nmbu.no)