AALBORG UNIVERSITY

STUDENT REPORT

Study of Pelleting Process Using Design Of Experiment And Numerical Optimization

MSc Thesis

Aalborg University, Campus Esbjerg

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Titel page

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Abstract

In the transition from fossil-based to renewable energy sources, the use of fuel pellets created from leftover biomass from the industry for energy production has gained popularity.
The pelleting process is an established technology, where the main optimization focus in recent studies is based on the material used in the process. The focus in this study is the entire pelleting process, with an investigation of both material parameters and process parameters.
Design Of Experiment was used to determine the experimental variations to test at each run, from which an optimization equation with: total energy consumption of the process, maximum pressure used in the process and durability of the produced pellets as response variables.
A statistical analysis using significance was conducted on the design variables to determine the significance of the effect they have on the responses resulting in reducing the full model of terms.
Optimized settings for the pelleting process at the four different durability limits of EU regulations are present in the results along with a discussion and conclusion of the results.

Reading Guide

To distinguish between figures, equations, and citations, [] is used for referring to citations and numbers used for referring to figures and equations.

Contents

Nomenclature

Design Variables

A	Design variable: Temperature	$^{\circ}C$
B	Design variable: Moisture Content	$\%$
C	Design variable: Particle Size	mm
D	Design variable: Inlet Angle	$^{\circ}$
E	Design variable: Inlet Area Ratio	$-$
F	Design variable: Mass per Layer	g
G	Design variable: Roller Height	mm

Pelleting

μ	Friction coefficient	$-$
A	Area of the piston	mm^2
$c(x)$	Compression ratio	$-$
F	Force	N
m_{pellet}	Mass of the produced pellet	g
$m_{tumbler}$	Mass of the tumbled pellet	g
P	Pressure	MPa
P_x	Pelleting pressure	MPa
P_{NO}	Prestressing pressure	MPa
r	Radius of the pellet channel	mm
s	Extension of the piston	mm
v	Poisson's ratio	$-$
W	Work	J
x	Length of the pellet channel	mm

Design Of Experiment

β_n Effect of variable n on response

β_{nnn} Three-way interaction effect on response

β_{nn} Two-way interaction effect on response

x_n Design variable n

y Response variable

Statistics

α Significance level

$\bar{x}$ Mean of variable x

$\bar{y}$ Mean of response y

$\beta_{n,0}$ Null hypothesis

$\hat{\beta}_n$ Least Square Estimates of variable

$se(\hat{\beta}_n)$ Standard error of $\hat{\beta}_n$

t Number of standard deviations from mean

Chapter 1

Introduction

In the transition from fossil fuels to renewable energy sources, the use of biomass for backup to the solar and wind primary energy production is gaining popularity [1]. The main problem with biomass is the transportation [2] since the biomass often is produced far away from the power plants that generate electricity or heat from it. A solution to this is preprocessing and pelleting, which ensures a higher energy density of the biomass for transport. Other advantages of these processes include homogeneous conditions such as temperature, water content and heating value of the biomass [3].

The preprocessing of the material used in pelleting production follow three steps. The first preprocess is drying of the biomass followed by the second process, which is reduction of particle sizes using a hammer mill. The hammer mill grinds the raw biomass into an easier to handle particle size. The third preprocess is conditioning of the particle, meaning adding of moisture [4].

Figure 1.1: Showing the entire process from biomass to fuel pellets

The preprocessed material then enters the pelleting machine, which makes use of an eccentrically mounted roller within a ring die as seen in figure 1.2a. A plane illustration of the die can be seen in figure 1.2b, which also shows the pellet channels, where the biomass are compressed into pellets. The rollers compress the material and press it down through the pelleting channels, resulting in pellets being pushed out on the outside of the die.

Following the pelleting process, the pellets are then cooled using ambient cooling to reach homogeneous conditions, before being tested for pellet quality.

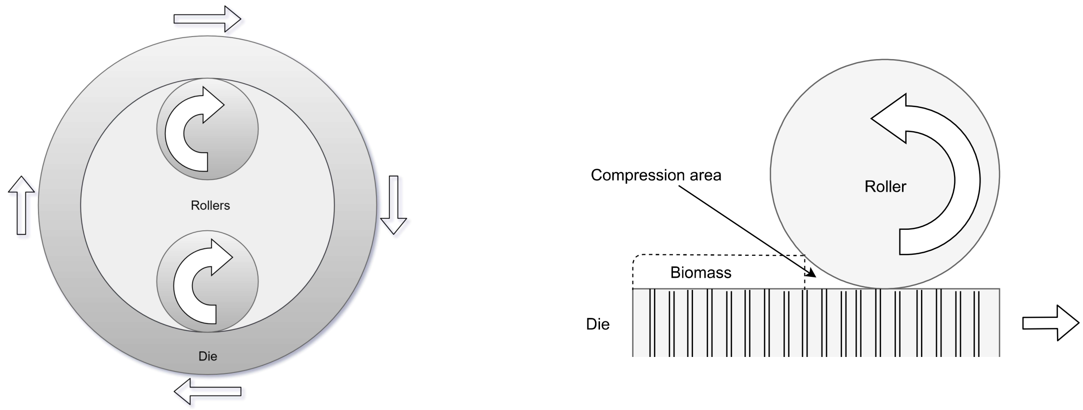

Figure 1.2: Showing the standard pelleting process

In 2016 Denmark used 2.6 Mio. tons [5] of wood pellets for energy purposes, this amount demonstrates a need for the pelleting process to be as effective as possible. This is done by experimenting with multiple variables to find the optimized settings that the process can be run at. The statistical analysis of the study will be done in the program Minitab.

This project included information about the experimental setup, design of experiment, statistical analysis, experimental work and a discussion followed by a conclusion.

Chapter 2

Project Definition

This MSc thesis aims to optimize the pelleting process; this is done by looking at the entire pelleting process, such as the geometry of the pelleting equipment, preprocessing conditions and the quality of the produced pellets. The goal is to decrease the amount of energy used per produced pellets while maintaining the regulation standards of quality. To this end, a statistical model is created using multivariate data analysis. The data required to create the model is obtained experimentally, by using a specially designed and manufactured modular single pellet piston unit at Aalborg University Esbjerg. From the experiment, the needed constraints for the optimization calculations are obtained. Furthermore, the statistical model will undergo a significance analysis to investigate the relevance of both the input and output variables.

The procedure of this study were to make an investigation into the relevant parameters of the pelleting process, followed by the theory of reducing experimental runs using Design Of Experiment. The chosen design from Design Of Experiment was then performed as experimental work, and the result were used to create an optimization model.

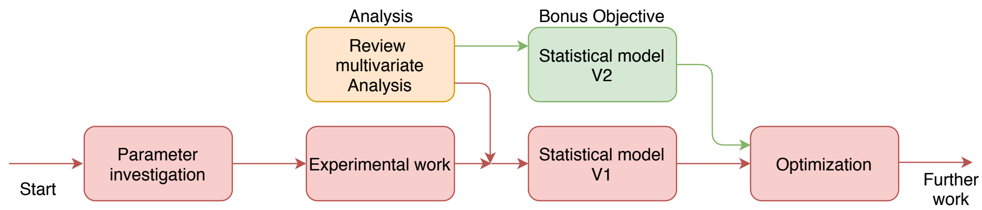

Figure 2.1: Flowchart of the project

Chapter 3

Pelleting Process Setup

In this chapter, the experimental setup commonly used in single pellet experiments was investigated in such a way that a new setup with increased interchangeability could be constructed. The chapter also includes a study of the relevant testing parameters and the limits for each parameter.

3.1 Modular Single Pellet

For experimental purposes, a testing setup needed to be constructed. This setup needed the ability to create pellets under different parameters and measure the relevant responses such as the pelleting pressure, energy consumption and the quality of the pellets. The basic knowledge needed for creating such a setup was obtained from a state of the art [6] study, that concluded that a single pellet setup which is shown in figure 3.1 was the standard for single pellet production created for experimental purposes.

Figure 3.1a shows the first compression of an experiment. This first compression uses a stop piston to create a material made stop piston for the following compressions. After the first compression as seen in figure 3.1b, the stop piston is then removed before the pellet production can be started as shown in figure 3.1c.

This setup is great for testing different species of wood and changes to the material such as; different moisture contents, particle sizes and amount of mass in the cylinder. The downside of this setup is the lack of options in changing the channel dimensions such as inlet angle and area ratios.

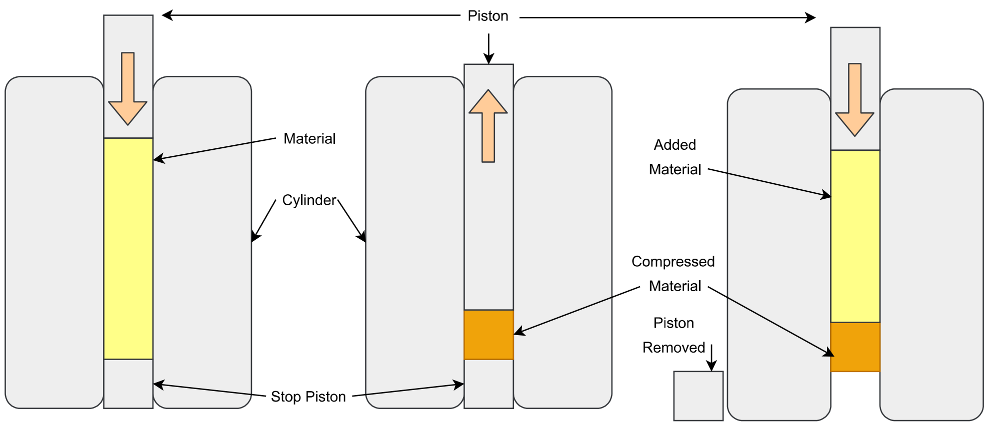

Figure 3.1: Pelleting process with circular die

To modify and improve the fixed single pellet setup, a modular single pellet setup was created. The setup can be seen in figure 3.2, which copies the use of a stop piston for creating the material plug. The figure shows a modular die as blue, which can be changed between experimental runs so that more parameters can be tested. Possible aditional parameters that can be tested with this setup is inlet angle, inlet area ratio and pellet diameter.

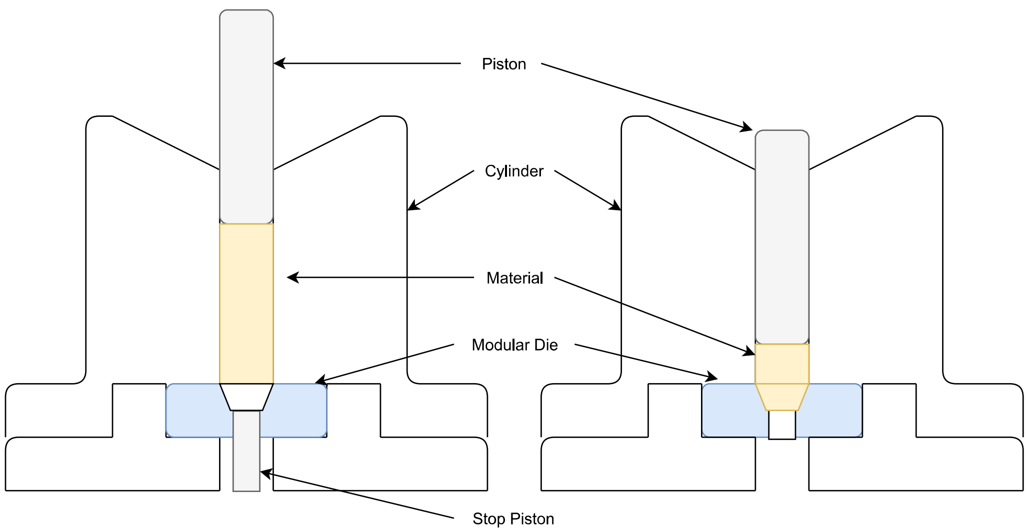

Figure 3.2: Pelleting process with modular die setup

3.2 Physics/Variable Analysis

For the experimental and statistical work, some important testing parameters had to be determined. The method for doing this is by creating a list of possible parameters by investigating the physics behind the pelleting process and the work done previously by others. This list of parameters can then be further investigated by doing a statistical analysis of the result from the chosen design of Design Of Experiment, which would result in the most important testing parameters.

Method One

The first method used for finding parameters is investigating previous works. The investigation into the pelleting pressure (Jens K. Holm et al. [4]) expressed the pressure needed to compress the pellet and to move it a distance of x through the cylinder. The expression they arrived at is shown in equation 3.1.

$$P_x = \frac{P_{NO}}{v}(e^{2\mu vc(x)} - 1) \tag{3.1}$$

The pelleting pressure equation 3.1 has these terms; P_{NO} is a prestressing pressure which is a term that takes added deformation pressure into account, v is the poison's ratio of the material, μ is the coefficient of friction between the pellets and the material of the die, c is the compression ratio shown in equation 3.2, which is the relation between the length and radius of the pellet channel.

$$c(x) = \frac{x}{r} \tag{3.2}$$

Relevant parameters:

- Poisson's ratio
- Coeffecient of friction
- Length of pellet
- Diameter of pellet

Method Two

Finding any relation as part of the leading physics.
Some of the same parameters will be found in the physics involved in the pelleting process, like those found in previous works.

The first concept to look at is the compression process. This is related to the material used and its composition along with the Poisson's ratio of the species. Furthermore, the Poisson's is dependent on the moisture content of the wood.

The next concept is the friction between the wood and the experimental setup, here the materials composition, moisture content [7] and internal friction comes into action. Furthermore, the internal friction depends on the temperature of the material [8].

The next is the flow rate, which is considered to have a low influence on the pressure due to it being a function of the speed of the pellet, which is very low.
The final parameters comes from the process itself, these parameters are such as; inlet angle of the die, inlet area of the die, how high the roller is mounted in the setup, how much material available per compression, the length and diameter of the channel.
Relevant parameters:

- Compression
  - Species
    - Composition
  - Poisson's Ratio
    - Moisture Content
- Friction
  - Internal Friction
    - Temperature
- Additional
  - Diameter of the cylinder
  - Length of the cylinder
  - Mass per layer
  - Roller height

3.3 Parameter study

The following section investigates the different parameters, to see if any can be removed to simplify the experimental testing process. Below is listed all the parameters from the parameter analysis, which all will be processed further in the next section.

- Species of wood
  - Composition
    - Lignin
    - Cellulose
    - Hemicellulose
    - Ash content

    Poisson's ratio Friction coefficient
  - Density
  - Moisture content
  - Temperature of the biomass
  - Particle size
- Process temperature
- Inlet angle
- Inlet area ratio
- Mass per compression/layer
- Roller height

3.3.1 Design and Response Variables

A requirement needed to do Design Of Experiment, which is later shown in chapter 4, is that the design variables are independent of each other. This requirement, along with possible design variables from the previous section, results in the parameters being divided into four groups, which are particle parameters, pellet parameters, die parameters and response variables.

The first design variable group contains the particle parameters, which is the relevant variables that are specific to the particles used in the pelleting process. These design variables include all parameters that differ between different species of wood and particle parameters such as composition (the content of Lignin, Cellulose, Hemicellulose, and Ash), friction coefficient, Poisson's ratio, density, particle size, temperature, and moisture content. As for composition, friction coefficient, Poisson's ratio, and density all are dependent on the species of wood used; these are all neglected in the setup of the experiment. The composition of the species used will be shown in the appendix if needed for any external source validation purposes.

The second design variable group is comprised of the pellet parameters such as length and diameter. These parameters are regulated by the EU [9], which makes these design variables not relevant for further study.

The third design variable group include the die parameters such as angle, temperature, and the ratio between the inlet area and piston area. All of these could have an impact on the response variables, so they are therefore included in further studies. Another interesting parameter to investigate is the placement of the rollers, in this case how high they are placed above the die. The last parameter in this group is the amount of material available per compression. Knowing that too much material can plug the setup this parameter is therefore considered for testing.

The last group is the response variables, which in this study include maximum pelleting pressure, total energy consumption, and quality control of the pellet. There are two different widely spread methods of testing for quality; these are the hardness tests and the durability test. Previous works using the two different quality methods have concluded that durability testing involving rotating the pellet a set number of times are superior to hardness testing which involves measuring the side force needed to break the pellets [10].

3.3.2 Range Of Variables

For use in the following chapters, a high and low limit along with the center value for each design variable is to be defined. This is done by reviewing previous works and international requirements.

Group one:
Starting with the particle parameters, the first design variables is the species of wood to be used in the experiments, to this end at least three different variants needs to be present, with more given additional information. It could be interesting and relevant to review and experiment on the often used species of wood for pelleting. A

number of seven species was therefore chosen for further investigation but was later rejected because of the time constraint of this project, resulting in only testing of one species of wood.
The particle size range was defined to be between zero and 3.15, which is the range set by the standard from ISO 17827-2:2016. From the overall range, a set of three sub-ranges was chosen to be from 0 to 1 as low limit, from 1 to 2 as center value and from 2 to 3.15 as the high limit.
Regulating the temperature of the wood, which in this case should be read as the biomass, is not a viable action due to the energy required for heating when it is already known that the pelleting process generates heat due to friction and compression. The temperature for the process will be discussed in group three. The only positive effect of preheating the biomass is when the process temperature is low, such as when the process is starting up.
Due to extensive testing done by other studies [11], the chosen range of the moisture content was from 10% to 20 %

Group two:
The length and diameter of the pellets have to be created according to ISO regulated 17831-1:2015.

Group three:
The inlet angle as seen in figure 3.3 was a variable with low to none research. It was therefore interesting to look at a wide range to get an understanding of the effect it would have on the process. The chosen range was from 60° to 140°, which is a wide range with no guaranty to have an optimum point within.

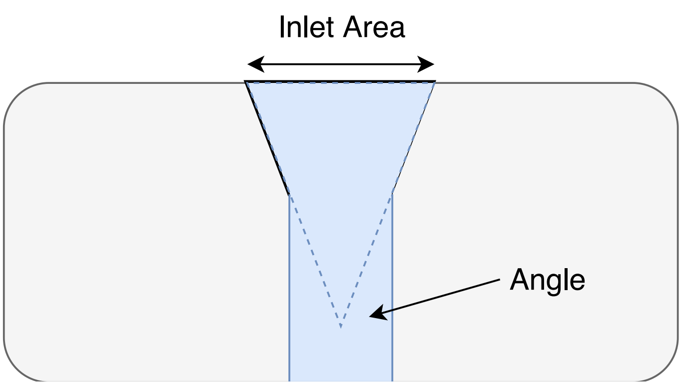

Figure 3.3: Showing the inlet angle and inlet area

The ranges of the process or die temperature was chosen from state of the art

investigation [6], that show a temperature range in the die from around $100°C$ to around $140°C$. Due to failed experiments and personal error, the used range was $80°C$ to $120°C$ on the first try and $100°C$ to $120°C$ on the second try. The lower limit of $80°C$ and $100°C$ was shown by experimental testing to be to low for the experimental setup, and therefore resulted in several failed experiments.
The inlet area ratio shown in figure 3.3 is the ratio between the inlet area of the die and the piston force area. Since it is a ratio, the high limit is 1, where a low limit was chosen to be 0.6 due to lack of information.

Response variables:
The maximum pelleting pressure needed to create pellets is required to be as low as possible. This is because the pressure influences the experimental setup, with lower max pressure possibly resulting in a motor change to a smaller and more efficient version. The compression machine used in the experiment measures the applied force, which has to be divided by the piston area, found from a diameter of $[9mm]$, to get pressure as seen in equation 3.3.

$$P = \frac{F}{A} \tag{3.3}$$

The total energy consumption is an important response variable because the whole pelleting process is about energy efficiency, so lowering the total consumption makes the whole pelleting process more efficient. The energy consumption is calculated using the midpoint rule of integration which takes a mean value of two force $[F]$ measurements and multiplies it by the extension $[s]$ of the piston. This is shown in equation 3.4.

$$W_{total} = \sum \left(\frac{F_{i+1} + F_i}{2} \cdot (s_{i+1} - s_i) \right) \tag{3.4}$$

The durability of a pellet is used to determine the quality of the pellets, with pellets produced with lower quality resulting in the manufacturer being subjected to fines. The durability is calculated from the mass of the produced pellet $[m_{pellet}]$ divided by the remaining mass after tumbling $[m_{tumbler}]$ as shown in equation 3.5. These fines are defined from which durability group that the sample of the pellets is within, these groups can be seen in bullet form below.

$$Durablility = \frac{m_{pellet}}{m_{tumbler}} \tag{3.5}$$

EU/ISO Regulations in percentage: [9]

- Durability group one: $\geq 97.5\%$
- Durability group two: $\geq 96.5\%$
- Durability group three: $\geq 95.0\%$
- Durability group four: $< 95.0\%$

3.3.3 Design Variables With Limits

Table 3.1 shows the different design variables, with both center point, low and high limits. From these variables, the different experiment is set up, such that each variable are tested the same amount of times for low and high limits.

Table 3.1: Table showing the design variables at low, centerpoint and high limit

		Inputs		
Factor		Low	Center point	High
Die Temperature (First Try)	A	$80°C$	$100°C$	$120°C$
Die Temperature (Second Try)	A	$100°C$	$110°C$	$120°C$
Moisture Content	B	10%	15%	20%
Particle Size	C	$0 \geq 1$	$1 > 2$	$2 \geq 3.15$
Inlet Angle	D	60°	100°	140°
Inlet Area Ratio	E	0.6	0.8	1.0
Mass per Layer	F	0.1 g	0.2 g	0.3 g
Roller Height	G	1.00m	1.25 mm	1.50 mm

The first try of the temperature indicates that the first 15-20 experiments with $80°C$ as the low limit had multiple failed runs, this resulted in a retry with the lower limit increased to $100°C$

Chapter 4

Design Of Experiment

Design of experiments (DOE) is a statistical method of experimental planning with multiple usages with the main one focussing on minimizing the required number of runs of an experiment needed to achieve the desired data.
Compared to the traditional approach of experimenting, which includes experimenting with varying one variable at a time, using DOE varies several variables at a time. Using DOE results in a decreased amount of runs and therefore reduces experimental cost. These benefits of DOE fits the purpose of experimental design, which is efficiency and focus.

4.1 DOE in practice

4.1.1 How to design the experiment

The steps for building the design are presented in bullet points below.

First Step

The first step is determining the experimental responses for measurements and design variable to adjust the different experimental runs. For this study, an example is given in figure 4.1.

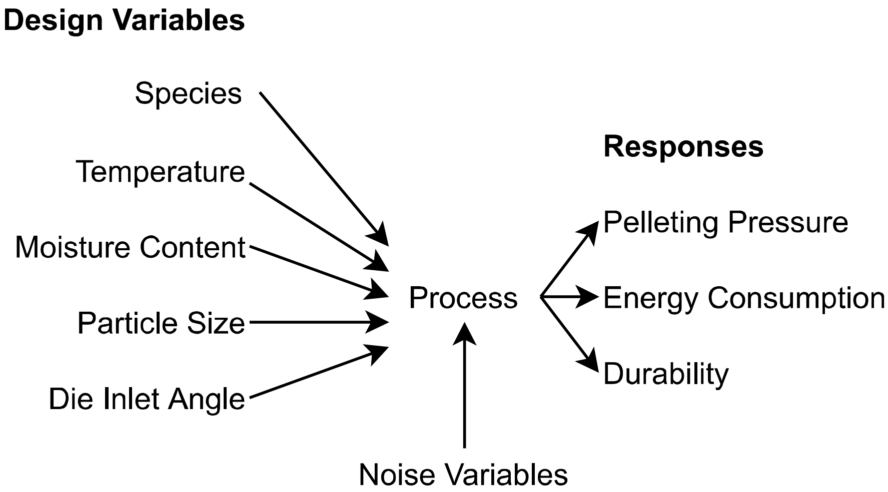

Figure 4.1: DOE: Design variables and responses

The noise variable is a collection of overlooked important design variables and other sources that influence the response variables. Examples of these sources are noise from instruments and measurement errors.

Second Step

The second step is defining levels to each of the design variables. The standard number of levels are based on the assumption that there is a linear relationship between the change in a design variable and the effect it has on a response variable. This linear relation requires two values, a low and a high value of each design variable. In cases with a relation that are nonlinear, another point can easily be added for increased accuracy. This is normally done by adding a center point for each design variable, such that an experimental test of the mean value of all design variables can be obtained. Notation of levels are commonly done with [-1] for low values, [+1] for high values and [0] for center points.

Table 4.1: Example of a two-level, two-factor DOE with notations

	Factor	
Runs	A	B
1	-1	-1
2	+1	-1
3	-1	+1
4	+1	+1
5	0	0

Third Step

The third step is defining the required information, such as an investigation of design variable, factor, and interaction effects or finding optimum values of the design variables. From understanding what is to be investigated, the type of design used is then chosen. These designs can be classified into three model groups, as described later in this chapter.

4.2 Types Of DOE

The first type investigated is the full factorial design. This is due to the fact that these designs are the most extensive in terms of runs and therefore a good starting point for understanding other less extensive designs.

4.2.1 Full Factorial Designs

The full factorial designs include all the information that can be obtained from experimental testing. This is because all combinations of the design variables are tested and investigated. In most cases this results in a high number of experimental runs, with a low yield of additional information. The number of runs needed for full factorial designs are calculated with 4.1. An example of a two-level, two-factor full factorial design with one center point was used previously in table 4.1, showing all of the possible different runs with the two design variables. For the experimental results, columns are added on the right side with the measured values of each run.

$$Runs = Levels^{Factors} \tag{4.1}$$

Table 4.2: Table showing two-level full factorial design

Factor	2	3	4	5	6	7	8	9
Runs	4	8	16	32	64	128	256	512

Table 4.2 shows the number of runs in full factorial design for a different number of design variables. From the table, it is clear that designs with several design variables ranging from 4-5 and onwards requires an uneconomical amount of runs. Therefore introducing Fractional Factorial Designs, which are based on the concept of Full Factorial Design but with a decreased amount of runs.

Effects

The main effect is the variation in a response variable when varying design variables low and high level. The significance of the main effect can be found statistically.

Another important effect is the interaction effect, which indicates the interaction between two or more design variables. The same methods for finding the main effect can also be used to find the interaction effects, which will be explained in the next chapter.

Important variables

Important variables are the design variables that are shown by statistical calculation to have a main effect on a response variable. A design variable without a main effect on a response variable can still be important if it has statistically significant interaction with another design variable. All non-important variables should be excluded from any modeling work because they are without effect on the response variables.

4.2.2 Fractional Factorial Designs

The concept of fractional factorial designs are running a full factorial design but ignoring interaction terms, such as a design with a focus on main effect and two-way interaction but assuming that three-way or more interactions can be neglected. This results in a reduction in the number of runs required to obtain the required information. The number of interaction available in a fractional factorial design is shown by its resolution, these are indicated with Roman numerals, and an example of how they work are shown later in this chapter. Table 4.3 shows different factorial designs, including full and fractional designs. The "factors" in the table indicate the number of independent design variables to investigate, whereas "run" indicates the minimum number of experiments needed to get a proper understanding of the independent variables significance and the significance of any interactions.

Table 4.3: Resolution of available factorial designs in Minitab [12]

	Factors									
Run	2	3	4	5	6	7	8	9	10	11
4	Full	III								
8		Full	IV	III	III	III				
16			Full	V	IV	IV	IV	III	III	III
32				Full	VI	VI	IV	IV	IV	IV
64					Full	VII	V	V	IV	IV
128						Full	VIII	VI	V	V

Resolution

The drawback of using fractional design is the possibility of losing significant data, therefore introducing the resolution of a chosen design. These are indicated with Roman numerals, and they show the amount of significant information that can be obtained from the chosen design.
The resolution indicates the level of the designs, which are ranging from full factorial designs $[Full]$ to fractional factorial designs $[III, IV, V, VI...]$. The full factorial design includes significant information for the variables, but also the information for all interactions such as two, three, four...-way interactions. The fractional factorial designs maintain the number of factors but use fewer runs to achieve the relevant pieces of information. This is where the resolution comes into play. To understand the resolution, two examples are made:

Resolution [III]

At this resolution, the basic variables are confounded with the two-way interaction terms, meaning that the two-way interaction term and higher are inconclusive and therefore irrelevant. The lack of interaction terms make this resolution a great tool for basic order investigations, but a bad tool for overall investigations.

Resolution [V]

For this resolution the confounded terms are: $[V] = 1 + 4 = 2 + 3$, showing that the confounded terms are the three-way and four-way interactions. This leaves the first order and second order to be available for further processing, which in most cases are more than enough.

The recommended resolution for a higher number of factors, where three-way interactions or higher are neglected, is [V].

4.3 Choosing the model

4.3.1 Screening

The screening process is usually done at the start of a project with a large number of design variables. This is done by minimum testing of the design variables where the insignificant basic design variables are removed from further modelling. The screening model usually consists of a linear model, only containing the main effects. The most used design for screening is a resolution [III] fractional design with a focus on the main effects only; this way, the important design variables remain. There is still the possibility that an important interaction term is removed.

Plackett-Burman Designs

Other designs often used in screening are the Plackett-Burman designs. These designs are primarily used when dealing with a high number of design variables while doing a reduced number of experimental runs. As with the [III] design, Plackett-Burman designs only focusses on the main effects. The lowest number of runs needed for these designs are 12. Equation 4.2 show the model parameter obtained from screening, with β_0 being the intercept value for the response and β_n is the response change with increasing or decreasing x_n.
Linear Model:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 \tag{4.2}$$

4.3.2 Prediction Model

When a linear model fails to explain the response accurately, a prediction model can be used. The difference between the linear model and the prediction model is

the addition of interaction terms. Equation 4.3 shows the added terms compared to equation 4.2. These terms are the so-called interaction terms, with x_1x_2 being a two-way interaction and $x_1x_2x_3$ being a three-way interaction. Higher than three-way interaction is normally neglected because of the lack of information gained with increased calculations.
Prediction Model:

$$y = Linear + \beta_{12}x_1x_2 + \beta_{13}x_1x_3 + \beta_{23}x_2x_3 + \beta_{123}x_1x_2x_3 \tag{4.3}$$

Figure 4.2 shows a two-factor version of the prediction model, the so-called cube display. The blue points indicates the low and high value of each of the factors, whereas the green point indicates the center point value.

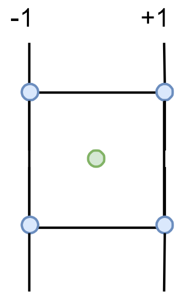

Figure 4.2: The cube display of a two factor design

4.3.3 Optimization Model

An optimization model is based on a quadratic method, where the linear relation of the basic variables from screening are combined with the two-way interactions of the prediction model and the square effects to study the curvature of the response surface.
Quadratic :

$$y = Prediction_{model} + \beta_{11}x_1^2 + \beta_{22}x_2^2 + \beta_{33}x_3^2 \tag{4.4}$$

Cubic:

$$\begin{aligned} y = Quadratic &+ \beta_{123}x_1x_2x_3 + \beta_{112}x_1^2x_2 + \beta_{113}x_1^2x_3 + \beta_{122}x_1x_2^2 \\ &+\beta_{133}x_1x_3^2 + \beta_{223}x_2^2x_3 + \beta_{233}x_2x_3^2 + \beta_{111}x_1^3 + \beta_{222}x_2^3 + \beta_{333}x_3^3 \end{aligned} \tag{4.5}$$

Central Composite Designs

The most used design for optimization is the Central Composite Designs. The Central Composite Designs can be categorized into three methods; these are the

Circumscribed, Inscribed, and Face-centered designs, which will be described next. For some Central Composite designs, it is possible to reuse some of the experimental tests and results obtained from either the full or fractional factorial designs used as screening or prediction.

Central Composite Circumscribed [CCC]

The original design for Central Composite is the circumscribed version, which includes some additional points called star points. These star points are located alpha [α] from the center and are referred to as new extremes of both the high and low level. The needed number of levels for CCC is the original three levels adding the two new extreme points, leaving a need for five levels.
Alpha is used to make of design rotatable, which means that all points at a distance of alpha from the center point are available as a response surface. Alpha in CCC would be the distance from the center to the limit points (blue points), which shows that the four new red points emerge.
From figure 4.3, it can be seen that the new star points, which is marked by the color red, are placed outside of the -1 to +1 range showing the new extreme levels needed for doing CCC.

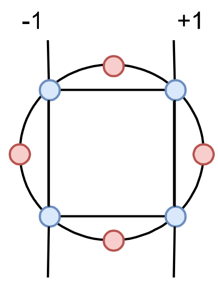

Figure 4.3: Showing the concept of CCC

Central Composite Inscribed [CCI]

The Central Composite Inscribed method uses the same principle as CCC to become rotatable. This is done by using the low and high limits as the star points. This is relevant when extreme limits are impossible to achieve. The concept can be seen in figure 4.4, which shows the CCC concept but restricted to the low and high limits.

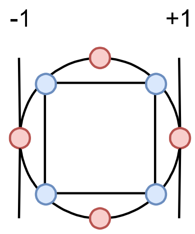

Figure 4.4: Showing the concept of CCI

Central Composite Face-Centered [CCF]

Another design is the Central Composite Face-centered, which is a nonrotatable design. Like the CCI, this design uses the information available from the low, high, and center point of each factor. The method uses a α value of zero, resulting in the star points being located on the center of the cube. The information available from the design is close to same as a full factorial design.

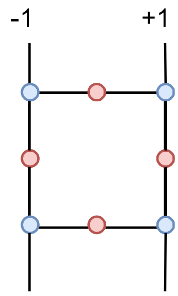

Figure 4.5: Showing the concept of CCF

Half Designs

The rules that apply to higher factor factorial design also apply to central composite designs, meaning that for higher factor designs a lower run with high enough information is available. These designs are called half designs because they usually require half as many runs as the full Central Composite designs. Table 4.4 shows the number of runs needed for both full Central Composite designs, half Central Composite designs and the Box-Behnken designs, which are explained below the table.

Table 4.4: Design methods of runs for statistical model

Design	Continuous Factors						
	2	3	4	5	6	7	8
Central Composite Full	13	20	31	52	90	152	-
Central Composite Half	-	-	-	32	53	88	154
Box-Behnken	-	15	27	46	54	62	-

Box-Behnken Designs

The Box-Behnken designs utilize the center point values of each factor to create a rotatable representation of the optimization area. This can be seen in figure 4.6, which shows the center point of all factors in a three-factor design. The center point on the back of the cube is also used, along with the center of the cube. By having the same distance to all points, this method is classified as a rotatable design.
The upside of using Box-Behnken is that only the low and high limits along with center points are used. Whereas one of the downsides is the overuse of center points, resulting in no reuse of results from previously created factorial designs.

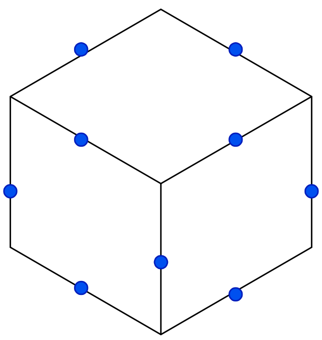

Figure 4.6: Showing the concept of Box-Behnken

Methods for increasing accuracy of design

There are two general methods of increasing accuracy; these are increasing runs and improved notation or grouping.
The first method can be achieved by adding more center points along with having replicates of the experiment, which is a certified way of increasing the accuracy of the model.
The second method to increase accuracy is to decrease human interaction related noise variables, such as performing the experiments in a random order removing

any bias from the person running the experiment. This can also be done by the use of blocks, which is a way of checking for noise variables between two sets of experimental data, an example of this is if the experimental runs are done on different days, the block system could show if there are any noise variables as a function of day change.

4.4 Model Selection

To get an overview of the different designs and the needed runs between them, a table with runs and designs can be seen below 4.5. The screening design chosen for comparison was with 12 runs, whereas the prediction designs chosen were the Full Factorial Designs and Fractional Factorial Design where the minimum resolution is five.

Table 4.5: Listing the needed runs for the different designs

Designs:	4	5	6	7	8
Screening	-	-	-	-	12
Fractional Factorial Designs [V-VI]	16	32	32	64	64
Full Factorial Designs	16	32	64	128	256
Central Composite [CCC,CCI,CCF]	31	52	90	152	304
Central Composite HALF	-	32	53	88	176
Box-Behnken	27	46	54	62	124

Experimental constraints:

- Fewer Center Points Materials
- Time - Prefer Fewer Experiments
- No Extreme Limits/ Points

The time constraint makes all of the experiments with more than 100 runs impossible. The lack of extreme limits makes the CCC impossible to do, while the lower amount of center point material makes the CCI and Box-Benkhen more undesirable. All of these constraints leave the CCF and factorial designs to be compared, with the Full Factorial designs and CCF having more information and the [V-VI] Fractional Factorial designs being shorter on runs. Getting the Full Factorial designs down on runs require doing the screening process, which has no guarantee for lowering the number of design variables and also has the chance of losing some important interactions, this leaves these designs as lacking compared to that of CCF designs. Furthermore, the extra information gained from CCF compared to the [V-VI] Fractional Factorial Designs, results in the CCF being chosen as the preferred designs for the experimental setup.

The last problem addressed to decrease the number of runs of the CCF is removing species as a design variable. This is done simply because of extensive research done by others on these variables and also that harder woods would increase the needed energy, but all other relations should remain the same when going from softwoods to hardwoods.

This leaves CCF as the preferred design with seven design variables, meaning the required number of runs needed are 88. A table 4.6 showing the first 15 runs describes which settings to use in the different experimental runs. To save space, the design variables are designated by their assigned letters from table 3.1. The full table with the 88 runs can be seen in the appendix A.1.

Table 4.6: Showing the first 15 of the 88 experimental runs

StdOrder	RunOrder	A	B	C	D	E	F	G
	Random	[$°C$]	[%]	[mm]	[°]	[-]	[g]	[mm]
1	81	100	10	1	60	0.6	0.1	1.5
2	79	120	10	1	60	0.6	0.1	1
3	39	100	20	1	60	0.6	0.1	1
4	9	120	20	1	60	0.6	0.1	1.5
5	46	100	10	3	60	0.6	0.1	1
6	88	120	10	3	60	0.6	0.1	1.5
7	75	100	20	3	60	0.6	0.1	1.5
8	78	120	20	3	60	0.6	0.1	1
9	62	100	10	1	140	0.6	0.1	1
10	24	120	10	1	140	0.6	0.1	1.5
11	17	100	20	1	140	0.6	0.1	1.5
12	27	120	20	1	140	0.6	0.1	1
13	87	100	10	3	140	0.6	0.1	1.5
14	77	120	10	3	140	0.6	0.1	1
15	47	100	20	3	140	0.6	0.1	1

Chapter 5

Statistical Analysis

The statistical objective of this chapter is reviewing how to find the effect of all of the design variables on the responses, along with how to determine and select the significance of the design variables.

5.1 Effect And Significance

5.1.1 Effect Calculation

Finding the effect of the design variables is done by the least square estimate method, which is shown in equation 5.1, where $\hat{\beta}_n$ is an estimation of design variables slope on the response, $\bar{x}$ and $\bar{y}$ is the mean of x and y.

$$\hat{\beta}_n = \frac{\sum x_n y - n\bar{x}\bar{y}}{\sum x^2 - n\bar{x}^2} \tag{5.1}$$

The intercept value β_0 is then found from a linear relation, as seen in equation 5.2.

$$\hat{\beta}_0 = \bar{y} - \hat{\beta}_1 \bar{x} \tag{5.2}$$

5.1.2 Significance Calculation

For finding the significance of the design variables, a couple of statistical concepts has to be reviewed. These concepts follow the route of doing a t-test to find a probability value p, which is then compared to a significance level α.

The first concept is the t-test, which is found from equation 5.3. In this equation $\beta_{n,0}$ is the hypothesis value, which is typically zero, $se(\hat{\beta}_n)$ is the standard error of $\hat{\beta}_n$.

$$t = \frac{\hat{\beta}_n - \beta_{n,0}}{se(\hat{\beta}_n)} \tag{5.3}$$

The results of the t-test are then shown in a normal distribution 5.1, where the t value is the standard deviation from zero. The following figure shows the normal distribution with a t-value and the p-value, which is the next concept. As seen in the figure, the p-value is the area from the t-value point to the right tail.

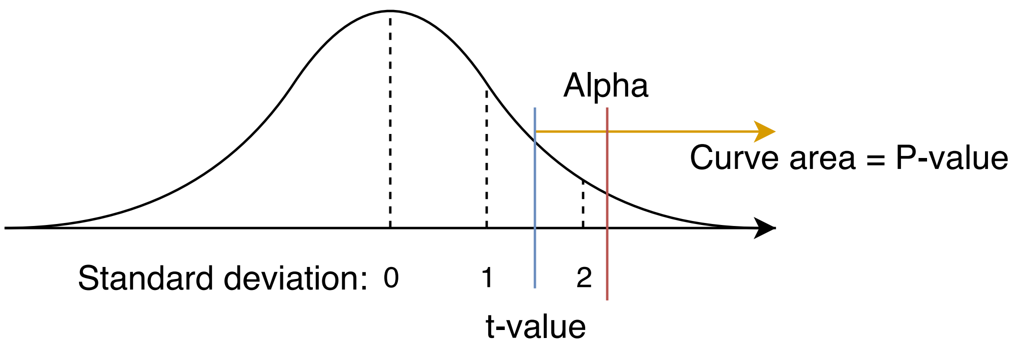

Figure 5.1: Showing a normal distribution with a t-value, α and p area

The p-value is a level of trust in the null hypothesis, in such a way, that the lower the p-value is, the more accepting the alternative hypothesis becomes. The significance level α is to point where you switch from the null hypothesis to the alternative one, this is often set to be either 0.05 or 0.01, where 0.05 is the one used in this study. In conclusion, if the p-value is below the significance level of 0.05, then the investigated design variable has a slope not equal to zero; therefore, it is relevant for the model.

- Null hypothesis
  - Slope is not significantly different from zero. $b_n = 0$
- Alternative hypothesis
  - Slope is significantly different from zero. $b_n \neq 0$

5.2 Model Reduction

5.2.1 R-Squared

The R^2 value as seen in equation 5.4 is used to explain the difference between the fitted regression model and the measurements. This gives that a high R^2 value indicates a well-fitted model. The downsides of using standard R^2 is the inability to determine if added model terms increase the effectiveness of the model. The R^2 value will always go up when adding terms, so a model with fewer terms will always be less accurate than a model with more terms, this is not true given that adding insignificant terms does nothing for the accuracy of the model.

$$R^2 = \frac{ExplainedVariation}{TotalVariation} \tag{5.4}$$

To address the problems with standard R^2, other methods were created, the first being the adjusted R^2 and the last being the predicted R^2.

The adjusted R^2 was created to compare models with a different number of design variables or terms. The adjusted part is adjusting the models according to the number of terms so that the problem of standard R^2 is removed.

The predicted R^2 explains how well a model is to predict new measurement values. This is done by removing a runs measurements and then calculating these measurements using the fitted prediction model. The difference between the measured point and the predicted point is then the predicted R^2.

5.2.2 Reduction Methods

For reducing the terms included in the model, the following method is used. These methods include forward selection, backward elimination, and stepwise reduction. These methods will be compared to the full model from each of the models R^2, adjusted R^2, and predicted R^2.
The full model includes all parts of the quadratic equation, such as linear, two-way interaction, and square relations.

Forward selection uses the pr-value of each effect to remove any insignificant design variables. It does so by looking at all of the design variables, then selecting and adding the most significant one or the one with the lowest p-value to the model. It continues to add to the model until no significant variables remain.

Backward elimination uses a reversed method of forward selection, namely elimination instead. This is done by also looking at all design variables, but instead of adding from a pool of variables to the model, it considered all variables as part of the model from the beginning. It then finds and removes the most insignificant or the one with the highest p-value from the model. This continues until only significant design variables remain in the model.

The stepwise reduction makes use of both forward selection and backward elimination. This is done by removing insignificant and adding significant variables. This is possible because the p-value of the design variables can change with removing or adding terms. If the reduction parameters are correct, then the resulting model should include all of the significant variables.

Chapter 6

Experiment

This chapter describes the experimental testing steps such as equipment used, the accuracy of equipment, the material used and where it is from, the preprocesses used on the material, execution of the experiment, result section and ending with possible sources of error.

6.1 Experimental Equipment

This section includes a list of used equipment in the experiments. The industrial machines used are listed with manufacturer and model, so that replication from an outside source is possible.

- Compression Machine With Heating Chamber
  - Manufacturer And Model: AMETEK - LLOYD LR50K
  - Load Cell Accuracy $< 0.5\%$
  - Extension Resolution < 0.05 microns
- Modular Single Pellet Unit
  - Nine Different Dies - Appendix A.2
- Preheat Oven
- Precision Scale
  - Manufacturer And Model: OHAUS - Pioneer Precision - PA214C
  - Uncertainty Of The Instrument: ± 0.110554 [mg]
- Computer
- Tumbler
  - Regulation Approved
- Laboratory Test Sieve
  - Mesh Size Of 3.15 mm, According To Regulations: ISO 17831-1:2015

6.2 Material

The species of wood used in the experiment were Pine, which is obtained from Sweden around the fall or winter of 2018.

6.2.1 Preprocessing

The material underwent the preprocessing as described in the introduction, starting with a drying process, which was done in a $100°C$ oven for one day, followed by a particle size reduction using an industrial hammer mill. The resulting particles from the hammer mill was then sieved so that a particle distribution could be obtained. The particle distribution can be seen in figure 6.1.

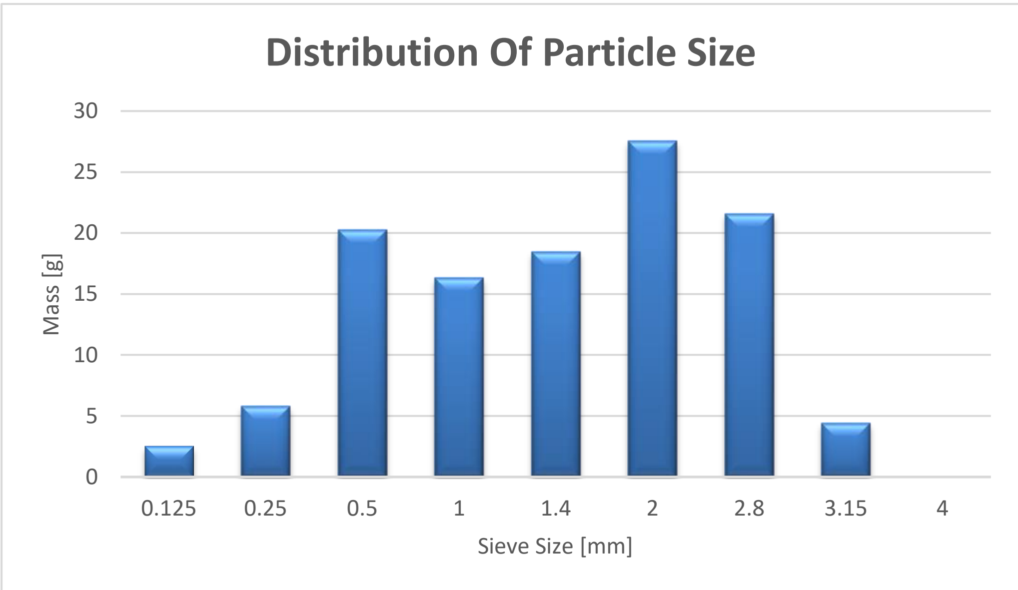

Figure 6.1: Distribution of particle size

With the distribution of particles, it was then important for the limits of the experiment to divide them into three groups or ranges. These ranges can be seen in figure 6.2 and are also defined in bullet form below the figure.

Figure 6.2: Showing the three particle ranges in cm

- Particle Range Distribution
  - Range 1: From 0 To 1 mm
  - Range 2: From 1 To 2 mm
  - Range 3: From 2 To 3.15 mm

The last step of the preprocessing was conditioning, which consists of adding the required moisture, as stated in chapter 3, to the different particle ranges. Each of the three particle ranges was added three different moisture contents, which resulted in 9 different testing variations for the experimental runs. The material was then placed in storage for a few days to insure that the moisture was uniform in the sample. The method for adding moisture was in accordance with regulations: ISO 18134-1:2015.

6.3 Execution Of Experiments

The procedure of the experiment can be seen as a sketch in figure 6.3. The first step of the experiment was moving the setup from the preheating oven to the heated compression chamber. Next step was to assemble the setup with the correct roller height; this was done in settings on the computer attached to the compression machine. After this the setup was complete, only the material preparation was needed.

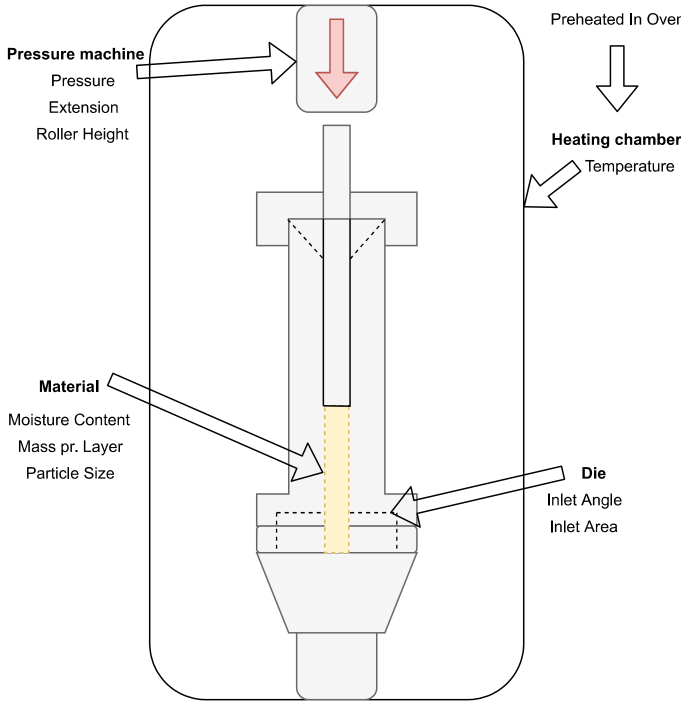

Figure 6.3: Showing the experimental pelleting setup

The material preparations included picking the correct particle range along with the correct moisture content. The loss of moisture in the material is considered to be negligible, due to the fact that the material was stored in an airtight container which was only opened during weighing of materials. After that, only the layer mass size design variable was missing to achieve the same amount of material per pellet, different number of layers had to be added depending on the mass per layer. This is shown in graph 6.4, which has the three different layer masses 0.3, 0.2 and 0.1 as pillars with the colors showing the different pellets produced.

As the graph shows, then the first pellet, the red section uses more material than the rest. This is because extra material is needed to be added compared to the layer mass, to ensure that the cylinder was filled adequately before removing the stop piston. Given that this makes the first pellet different in compression conditions than the following pellets, the first pellet was discarded for each variation experiment. After discarding the first pellet, it was easy to create multiple pellets under the same conditions; therefore, three pellets were created for all variables making averaged values possible. These extra pellets were added to the DOE as replicates. Due to the replicates being done in sequence the same testing conditions across the three produced pellets are not guaranteed, but a change in conditions such as temperature of the process is considered negligible.

To ensure that the first layer had sufficient mass to plug the cylinder, a mass was calculated based on the volume of cylinders and of a frustum. The resulting values can be found in appendix A.2.

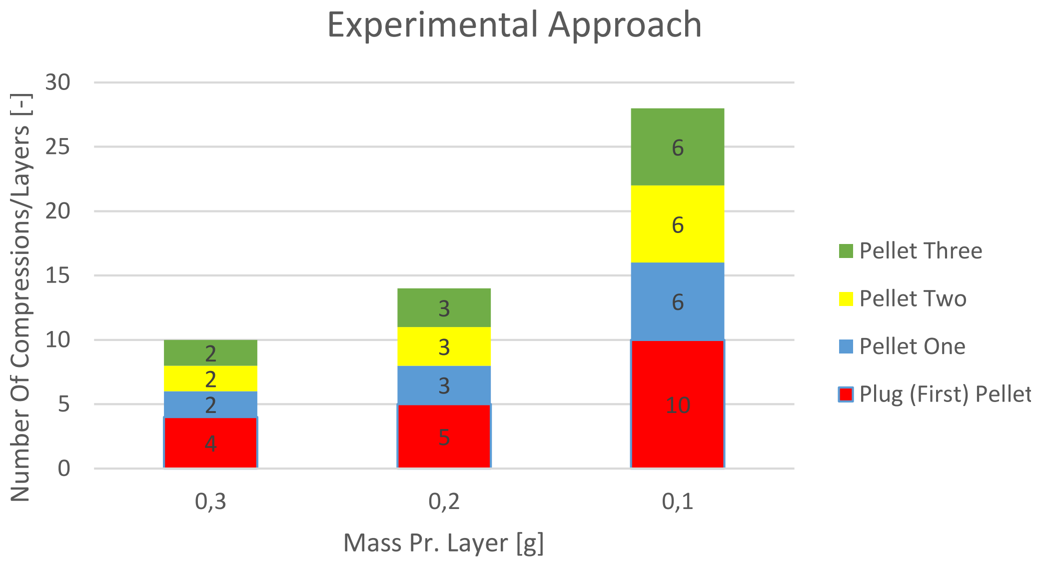

Figure 6.4: Showing the number of layers per experiment

6.3.1 Durability tests

The last experiment to run was the durability tests; these were done by measuring the weight of the pellets before and after going through the tumbler. Inside the tumbler, the pellets were subjected to 500 rotations. The last step before the last measuring was sieving the pellets to separate approved sizes above 3.15 mm from the discarded sizes below 3.15 mm according to regulation ISO 17831-1:2015.

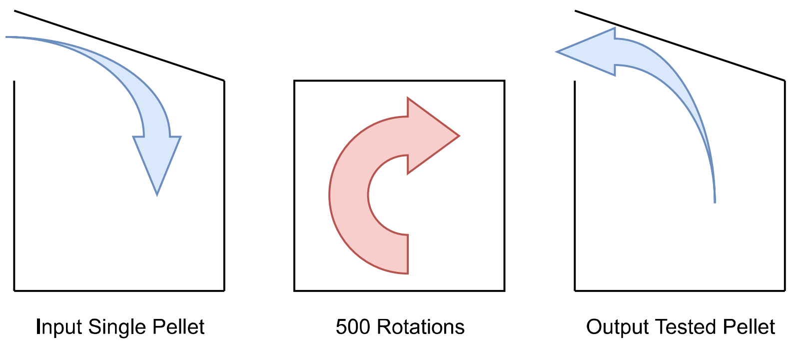

Figure 6.5: Showing the experimental durability setup

6.4 Results

6.4.1 Covariance Analysis

Relations between the response variables could simplify the optimization process in that it could decrease the number of variables in the optimization settings and calculations. Plotting the covariance between the response variables 6.6 shows if there are any relation. From the scatterplot, the response total energy consumption can be seen in the first row and column with the unit of [J], where the second row and column show the durability as a ratio, the third row and column show the maximum pressure in [MPa].

Looking at the relations it is shown in row one and column 3 that there are some relation between the total energy consumption and the maximum pressure, which is an information that can help reduce the setup of the optimization calculations. For the relation between durability and the two other response variables, it can be seen that no apparent relation is present.

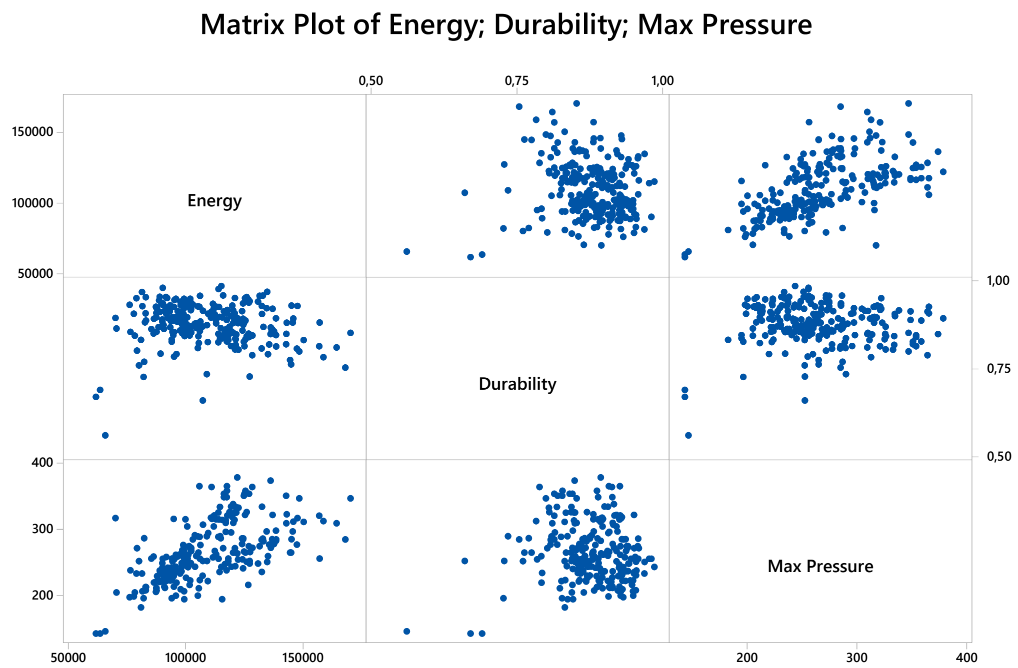

Figure 6.6: Showing the covariance between response variables

6.5 Model Reduction

Using the statistical p-value it is interesting to see the significance of the terms of the optimization mode. The following table 6.1 shows the R^2 along with the adjusted value and the predicted value of R^2 for each of the three reduction methods which was detailed in chapter 5. All of the reduction methods used a significance [α] of 0.05.

Table 6.1: Showing the accuracy of the different model reductions

	R^2	Full model	Forward	Backward	Stepwise
Energy	-	72.26	66.67	70.55	66.43
Pressure	-	74.56	68.06	72.04	67.85
Durablility	-	55.80	45.35	51.85	44.78
Average	-	67.54	60.03	64.81	59.69
Energy	Adjusted	67.75	64.70	68.40	64.74
Pressure	Adjusted	70.42	66.16	69.87	66.09
Durablility	Adjusted	48.63	41.88	48.57	42.25
Average	Adjusted	62.27	57.58	62.28	57.69
Energy	Predicted	61.53	61.80	65.37	62.23
Pressure	Predicted	65.47	63.62	67.21	63.78
Durablility	Predicted	38.47	36.81	43.40	38.26
Average	Predicted	55.16	54.08	58.66	54.76

From the model, it can be concluded that the full and the backward models are superior to the forward and stepwise models. This is determined by comparing the averaged adjusted R^2 and the averaged predicted R^2 of the different models. For optimization calculation, both the full model and the backward model will be included.

6.6 Optimization

For the optimization calculation some constraints are required, these includes which response variable that is of most importance. The logical response constraint is the durability levels as stated by the regulations, which stated that a penalty/fine system is in place for the durability ranges detailed in chapter 3. It is therefore considered important to test the two models at the four different levels. Furthermore the relation between total energy consumption and maximum pressure results in a reduction in both responses when lowering one of them. From this is can be concluded that only one of these responses could be set at the dominant variable to minimize.

6.6.1 Optimized Settings

Full Model

Using the durability limits set as part of regulation ISO 17831-1:2015, four different results were calculated with the full model. A summary of the settings being shown in table 6.2 and one visual representation can be seen in figure 6.7 and the rest in appendix A.5.

Figure 6.7 shows the relation between all of the design variables and the three response variables. The composite desirability curve is a optimization tool to compare the responses from set values of importance and weight.

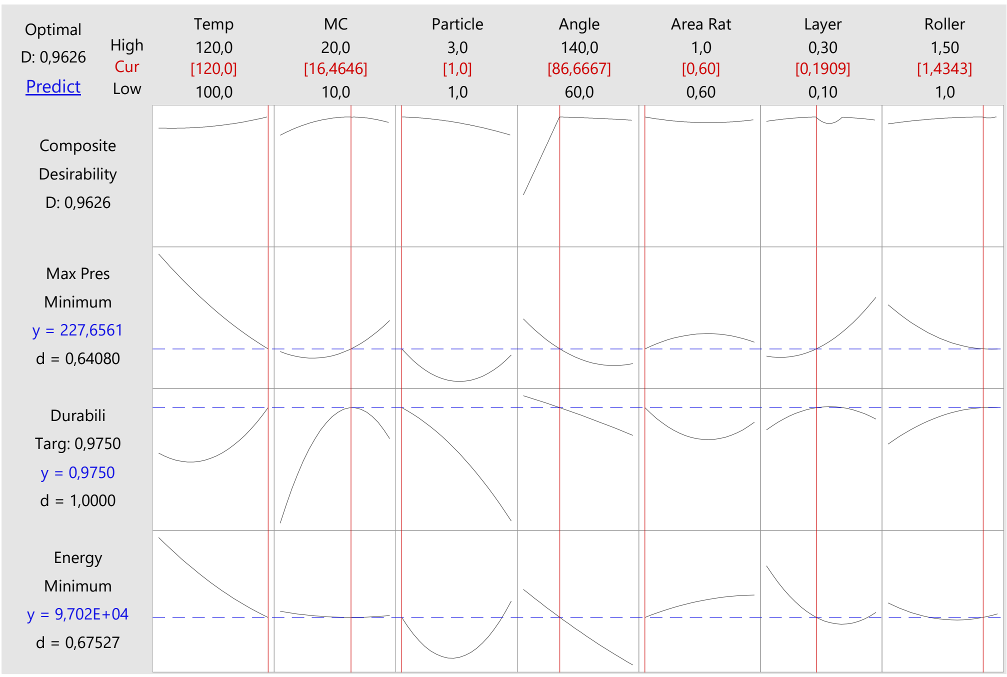

Figure 6.7: Showing the optimized conditions for 97.5% durability

Figure 6.7 and the table 6.2 shows that the more focus there is on durability, the higher the total energy consumption becomes. The first three columns, with durability 97.5%, 96.5% and 95.0% are calculated with the important response variable set as the durability, whereas the last column had the energy consumption as the most important response.

Table 6.2: Showing a summation of the different optimization settings

Full Model:		DU 97.5%	DU 96.5%	DU 95.0%	Min: Energy DU 94.7%
Temperature	[°C]	120	120	120	120
Moisture Content	[%]	16.46	16.67	15.05	18.08
Particle Size	[mm]	1.0	1.10	1.36	1.83
Inlet Angle	[°]	86.67	133.54	123.43	140
Area Ratio	[-]	0.60	0.60	0.60	0.60
Mass Per Layer	[g]	0.19	0.22	0.19	0.22
Roller Height	[mm]	1.43	1.49	1.32	1.32
Maximum Pressure	[MPa]	227.7	222.5	208.8	216.9
	[±]	1.138	1.112	1.044	1.085
Energy Consumption	[J]	97016	84455	82421	75505
	[±]	485.08	422.28	412.11	377.53

From the table 6.2, it can be concluded that the high limit of temperature is used in all the calculations, which means that a higher temperature than 120°C should improve the process. Likewise the low limit of inlet area ratio is used, which suggest that a lower area ratio should also improve the process.
The parameters that reach the limit on only some of the equations, could be further investigated if that durability level is the required one.

Backward Elimination Model

Like the full model summary, a version for the backward Elimination model can be seen in table 6.3 and visual representation can be seen in figure 6.8 appendix A.7.

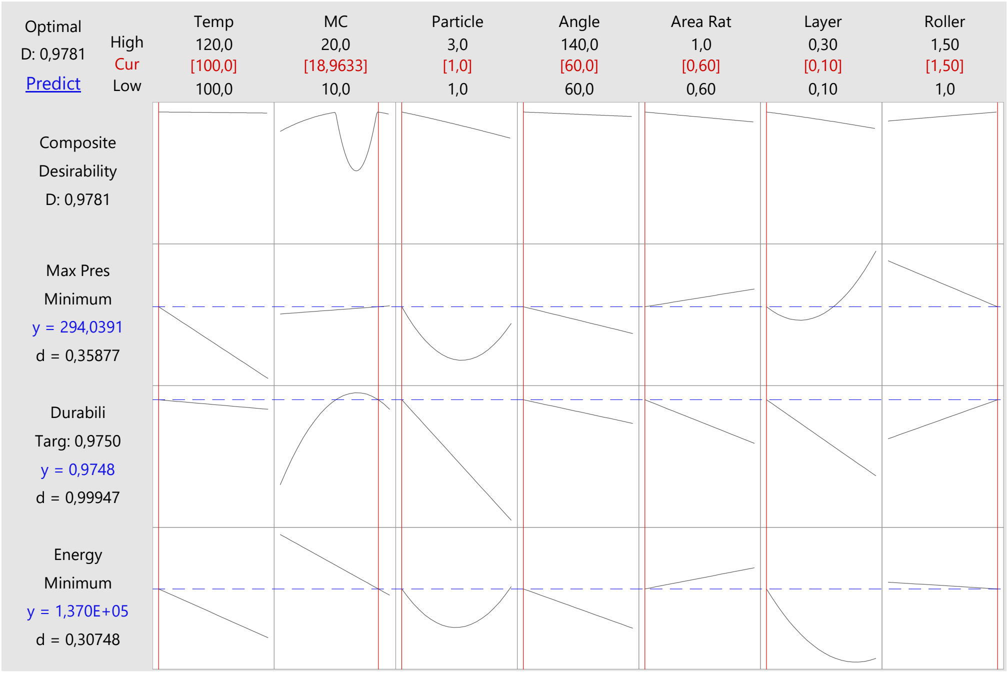

Figure 6.8: Showing the optimized conditions for 97.5% durability

The optimization calculations for the backward model shows the same trend as the full model when it comes to higher energy consumption with higher durability. The huge difference lies in the changed design variables along with a great increase in energy consumption.

Table 6.3: Showing a summation of the different optimization settings

Backward Model:		DU 97.5%	DU 96.5%	DU 95.0%	Min: Energy DU 92.5%
Temperature	[°C]	100	118.99	120	120
Moisture Content	[%]	18.96	14.55	16.36	20.00
Particle Size	[mm]	1.00	1.00	1.00	1.83
Inlet Angle	[°]	60.0	60.0	140	140
Area Ratio	[-]	0.60	0.60	0.60	0.60
Mass Per Layer	[g]	0.10	0.10	0.21	0.21
Roller Height	[mm]	1.50	1.50	1.44	1.50
Maximum Pressure	[MPa]	294.04	253.15	228.76	202.50
	[±]	1.470	1.266	1.144	1.013
Energy Consumption	[J]	136951	119922	84756	72118
	[±]	684.76	599.61	423.78	360.59

From the table 6.3, it is shown that some parameters such as; temperature, particle size, inlet angle, inlet area ratio, mass per layer and roller height reach limits at some of the durability levels, which suggest that further testing could be relevant for those levels.

Chapter 7

Summary And Conclusion

In this study, an investigation into relevant parameters that affect a pelleting process was performed. To investigate the parameters an experiment was set up using Design Of Experiment as the method of designing the experimental runs and a modular single pellet processing unit to produce the pellets with the variables defined by Design Of Experiment.

The design variable temperature was for the first experimental runs set with limits of $80°C$ and $120°C$, which resulted in several failed experiments at the lower limit. The following experiments were then executed with a lower limit of $100°C$, which later showed to also be too low to avoid failed runs. The conclusion of this compared with the trend of temperatures effect on the responses is that the optimal area for pelleting of the process temperature is higher than $110°C$, with increasing effectiveness with higher temperatures.

The selected design for DOE was the Central Composite Face-Centered half design, which included all the terms needed to create an optimization equation. The downside that a halfed design only get half the information is concluded irrelevant due to the fact that the resulting relations follow the expected trends.

An investigation into the covariance between the different responses showed that there is no apparent way to lower the total energy consumption while raising the durability since the durability has high and low measurements at both the high and low point of total energy consumption and maximum pressure.

Assuming that pelleting process manufacturers wants to construct a setup that produces the highest possible durability or at least reaching the limits of 97.5 %, then it can be concluded from the full model that the only design variables that would decrease the total energy consumption while maintaining the high durability level are a higher temperature and lower inlet area ratio. The same investigation into the backward elimination model shows that there is no apparent design variable that can be optimized without lowering the durability.

As for the accuracy of the models, it can be concluded that the backward model is a better predictor than the full model, this is due to the removal of unwanted terms. Another conclusion to the backward model is the lack of complete explanation, with an adjusted R^2 of roughly 62.28, indicating that there are some important variables that were not included in the Design Of Experiment part of the study.

As a final note, the most important information gained in the study is the relations between the design variables and the responses, which all show the different optimum points for the variables. Another important conclusion is that all of the design variables is significant either as a basic variable or as an interaction.

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Appendix A

Appendix

A.1 The DOE Results

Table A.1: Showing the result of the DOE with 88 runs

StdOrder	RunOrder	A	B	C	D	E	F	G
		[$^{\circ}C$]	[%]	[mm]	[°]	[-]	[g]	[mm]
1	81	100	10	1	60	0.6	0.1	1.5
2	79	120	10	1	60	0.6	0.1	1
3	39	100	20	1	60	0.6	0.1	1
4	9	120	20	1	60	0.6	0.1	1.5
5	46	100	10	3	60	0.6	0.1	1
6	88	120	10	3	60	0.6	0.1	1.5
7	75	100	20	3	60	0.6	0.1	1.5
8	78	120	20	3	60	0.6	0.1	1
9	62	100	10	1	140	0.6	0.1	1
10	24	120	10	1	140	0.6	0.1	1.5
11	17	100	20	1	140	0.6	0.1	1.5
12	27	120	20	1	140	0.6	0.1	1
13	87	100	10	3	140	0.6	0.1	1.5
14	77	120	10	3	140	0.6	0.1	1
15	47	100	20	3	140	0.6	0.1	1
16	14	120	20	3	140	0.6	0.1	1.5
17	64	100	10	1	60	1	0.1	1
18	12	120	10	1	60	1	0.1	1.5
19	76	100	20	1	60	1	0.1	1.5
20	45	120	20	1	60	1	0.1	1
21	82	100	10	3	60	1	0.1	1.5
22	10	120	10	3	60	1	0.1	1
23	34	100	20	3	60	1	0.1	1
24	43	120	20	3	60	1	0.1	1.5

25	57	100	10	1	140	1	0.1	1.5
26	85	120	10	1	140	1	0.1	1
27	26	100	20	1	140	1	0.1	1
28	42	120	20	1	140	1	0.1	1.5
29	48	100	10	3	140	1	0.1	1
30	65	120	10	3	140	1	0.1	1.5
31	28	100	20	3	140	1	0.1	1.5
32	69	120	20	3	140	1	0.1	1
33	32	100	10	1	60	0.6	0.3	1
34	29	120	10	1	60	0.6	0.3	1.5
35	55	100	20	1	60	0.6	0.3	1.5
36	54	120	20	1	60	0.6	0.3	1
37	66	100	10	3	60	0.6	0.3	1.5
38	18	120	10	3	60	0.6	0.3	1
39	59	100	20	3	60	0.6	0.3	1
40	11	120	20	3	60	0.6	0.3	1.5
41	44	100	10	1	140	0.6	0.3	1.5
42	60	120	10	1	140	0.6	0.3	1
43	19	100	20	1	140	0.6	0.3	1
44	5	120	20	1	140	0.6	0.3	1.5
45	13	100	10	3	140	0.6	0.3	1
46	7	120	10	3	140	0.6	0.3	1.5
47	1	100	20	3	140	0.6	0.3	1.5
48	36	120	20	3	140	0.6	0.3	1
49	23	100	10	1	60	1	0.3	1.5
50	68	120	10	1	60	1	0.3	1
51	3	100	20	1	60	1	0.3	1
52	33	120	20	1	60	1	0.3	1.5
53	52	100	10	3	60	1	0.3	1
54	41	120	10	3	60	1	0.3	1.5
55	31	100	20	3	60	1	0.3	1.5
56	84	120	20	3	60	1	0.3	1
57	70	100	10	1	140	1	0.3	1
58	71	120	10	1	140	1	0.3	1.5
59	25	100	20	1	140	1	0.3	1.5

60	50	120	20	1	140	1	0.3	1
61	2	100	10	3	140	1	0.3	1.5
62	8	120	10	3	140	1	0.3	1
63	15	100	20	3	140	1	0.3	1
64	6	120	20	3	140	1	0.3	1.5
65	21	100	15	2	100	0.8	0.2	1.25
66	58	120	15	2	100	0.8	0.2	1.25
67	53	110	10	2	100	0.8	0.2	1.25
68	83	110	20	2	100	0.8	0.2	1.25
69	49	110	15	1	100	0.8	0.2	1.25
70	51	110	15	3	100	0.8	0.2	1.25
71	63	110	15	2	60	0.8	0.2	1.25
72	22	110	15	2	140	0.8	0.2	1.25
73	74	110	15	2	100	0.6	0.2	1.25
74	72	110	15	2	100	1	0.2	1.25
75	16	110	15	2	100	0.8	0.1	1.25
76	35	110	15	2	100	0.8	0.3	1.25
77	38	110	15	2	100	0.8	0.2	1
78	80	110	15	2	100	0.8	0.2	1.5
79	61	110	15	2	100	0.8	0.2	1.25
80	73	110	15	2	100	0.8	0.2	1.25
81	86	110	15	2	100	0.8	0.2	1.25
82	67	110	15	2	100	0.8	0.2	1.25
83	56	110	15	2	100	0.8	0.2	1.25
84	4	110	15	2	100	0.8	0.2	1.25
85	20	110	15	2	100	0.8	0.2	1.25
86	30	110	15	2	100	0.8	0.2	1.25
87	40	110	15	2	100	0.8	0.2	1.25
88	37	110	15	2	100	0.8	0.2	1.25

A.2 Different dies and first layers

Table A.2: Different dies with first layer mass

Number	Angle	Area	First layer mass at roller height in [g]		
			1.00 mm	1.25 mm	1.50 mm
6	60	0.6	0.260	0.282	0.304
7	60	0.8	0.290	0.312	0.334
8	60	1.0	0.332	0.354	0.376
9	100	0.6	0.252	0.274	0.296
10	100	0.8	0.266	0.288	0.310
11	100	1.0	0.286	0.308	0.330
12	140	0.6	0.247	0.269	0.291
13	140	0.8	0.253	0.275	0.297
14	140	1.0	0.262	0.284	0.306

A.3 Experimental Pictures

Tumbler:

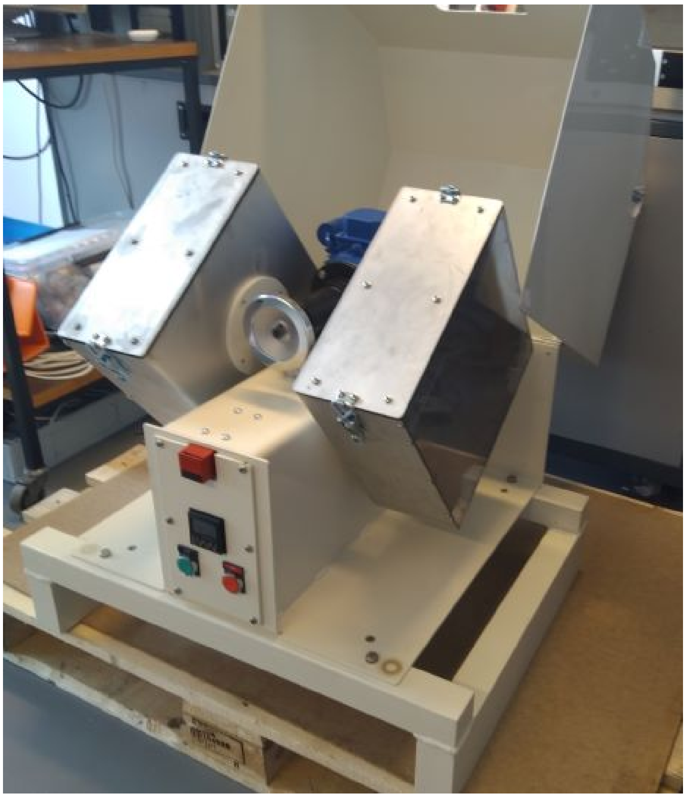

Figure A.1: Showing the tumbler machine

Laboratory Test Sieve - 3.15 mm:

Figure A.2: Showing the 3.15 sieve for durability testing

A.4 FULL MODEL

$Energy = 884034 - 6670Temp - 13811MC - 35650Particle + 201Angle - 74162AreaRatio - 682463Layer - 173405Roller + 17.7Temp \cdot Temp + 37MC \cdot MC + 12207Particle \cdot Particle + 0.51Angle \cdot Angle - 32263AreaRatio \cdot AreaRatio + 771129Layer \cdot Layer + 43811Roller \cdot Roller + 79.8Temp \cdot MC - 33.8Temp \cdot Particle - 5.46Temp \cdot Angle + 311Temp \cdot AreaRatio + 2699Temp \cdot Layer + 187Temp \cdot Roller - 312MC \cdot Particle + 1.62MC \cdot Angle + 3234MC \cdot AreaRatio - 1634MC \cdot Layer + 1096MC \cdot Roller + 54.8Particle \cdot Angle - 13456Particle \cdot AreaRatio - 12911Particle \cdot Layer + 2681Particle \cdot Roller - 30Angle \cdot AreaRatio + 596Angle \cdot Layer - 43.7Angle \cdot Roller + 35544AreaRatio \cdot Layer + 40810AreaRatio \cdot Roller - 28993Layer \cdot Roller$

$MaxPressure = 130000 - 975Temp - 1885MC - 3932Particle - 128.2Angle - 13527AreaRatio + 23643Layer - 36577Roller + 2.69Temp \cdot Temp + 27.7MC \cdot MC + 1070Particle \cdot Particle + 0.386Angle \cdot Angle - 10618AreaRatio \cdot AreaRatio + 71354Layer \cdot Layer + 7383Roller \cdot Roller + 7.45Temp \cdot MC + 4.0Temp \cdot Particle - 0.109Temp \cdot Angle + 79.6Temp \cdot AreaRatio - 127Temp \cdot Layer + 48.0Temp \cdot Roller - 105.4MC \cdot Particle + 0.176MC \cdot Angle + 399MC \cdot AreaRatio - 800MC \cdot Layer + 192.2MC \cdot Roller + 10.83Particle \cdot Angle - 1470Particle \cdot AreaRatio - 3309Particle \cdot Layer + 954Particle \cdot Roller + 26.9Angle \cdot AreaRatio + 27.2Angle \cdot Layer + 6.2Angle \cdot Roller - 1256AreaRatio \cdot Layer + 10025AreaRatio \cdot Roller - 7960Layer \cdot Roller$

$Durability = 2.34 - 0.0334Temp + 0.0598MC + 0.1376Particle - 0.00132Angle - 0.621AreaRatio - 0.459Layer + 0.218Roller + 0.000156Temp \cdot Temp - 0.001547MC \cdot MC - 0.0080Particle \cdot Particle - 0.000001Angle \cdot Angle + 0.350AreaRatio \cdot AreaRatio - 1.02Layer \cdot Layer - 0.099Roller \cdot Roller - 0.000020Temp \cdot MC - 0.000490Temp \cdot Particle - 0.000011Temp \cdot Angle - 0.00092Temp \cdot AreaRatio + 0.00736Temp \cdot Layer + 0.00086Temp \cdot Roller - 0.001434MC \cdot Particle + 0.000064MC \cdot Angle - 0.00114MC \cdot AreaRatio - 0.02170MC \cdot Layer - 0.00371MC \cdot Roller + 0.000148Particle \cdot Angle - 0.0255Particle \cdot AreaRatio - 0.0525Particle \cdot Layer - 0.0303Particle \cdot Roller + 0.000495Angle \cdot AreaRatio + 0.002238Angle \cdot Layer + 0.000396Angle \cdot Roller + 0.437AreaRatio \cdot Layer + 0.0472AreaRatio \cdot Roller - 0.024Layer \cdot Roller$

A.5 Optimization [FULL MODEL] Results

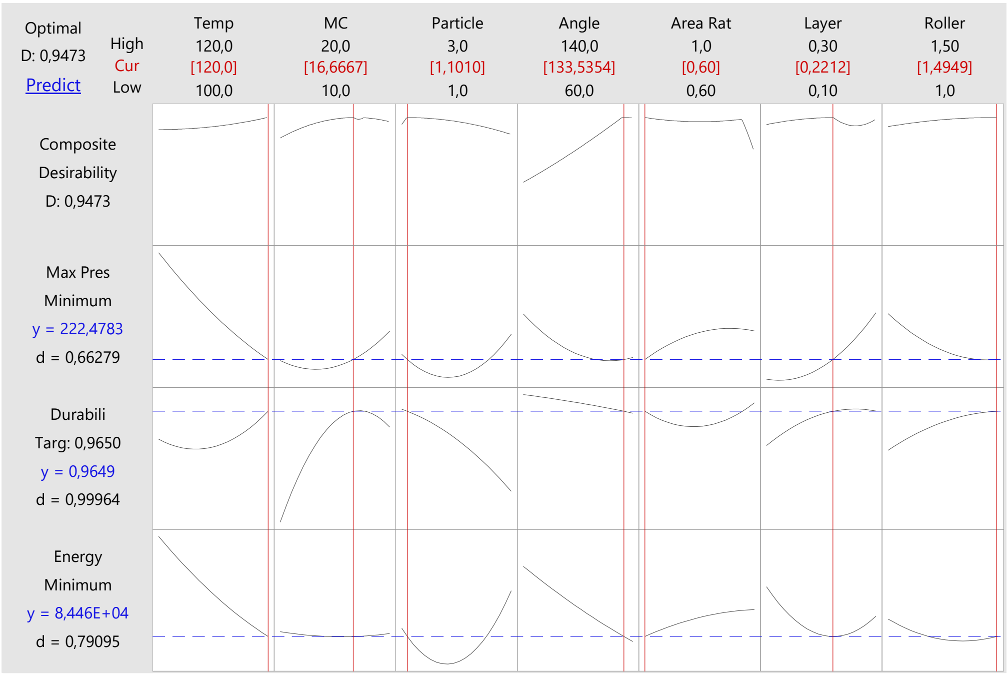

Figure A.3: Showing the optimized conditions for 96.5% durability

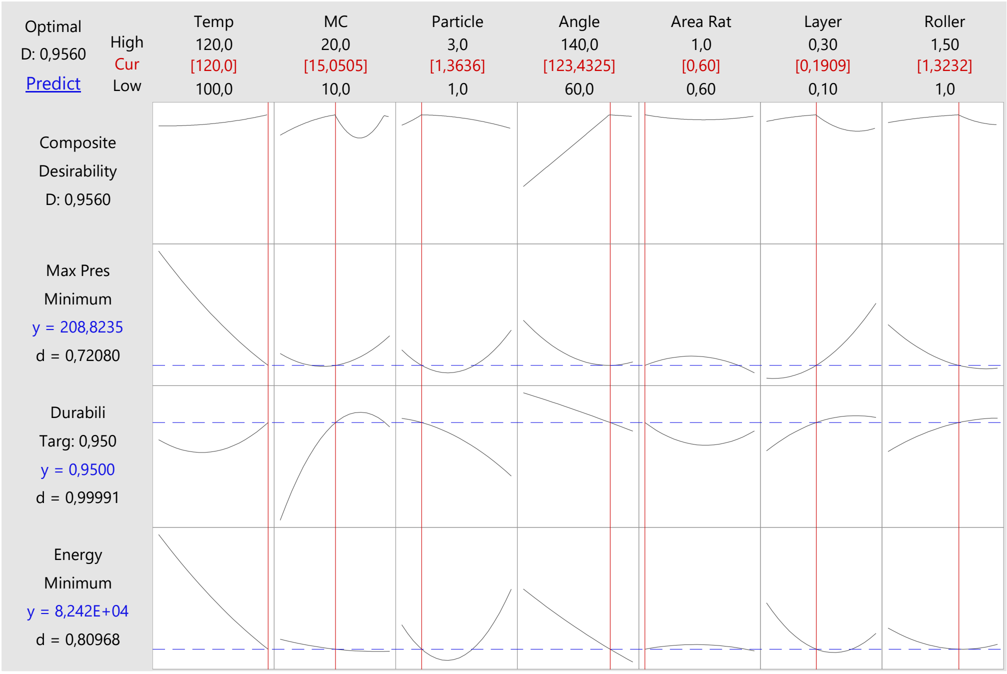

Figure A.4: Showing the optimized conditions for 95.0% durability

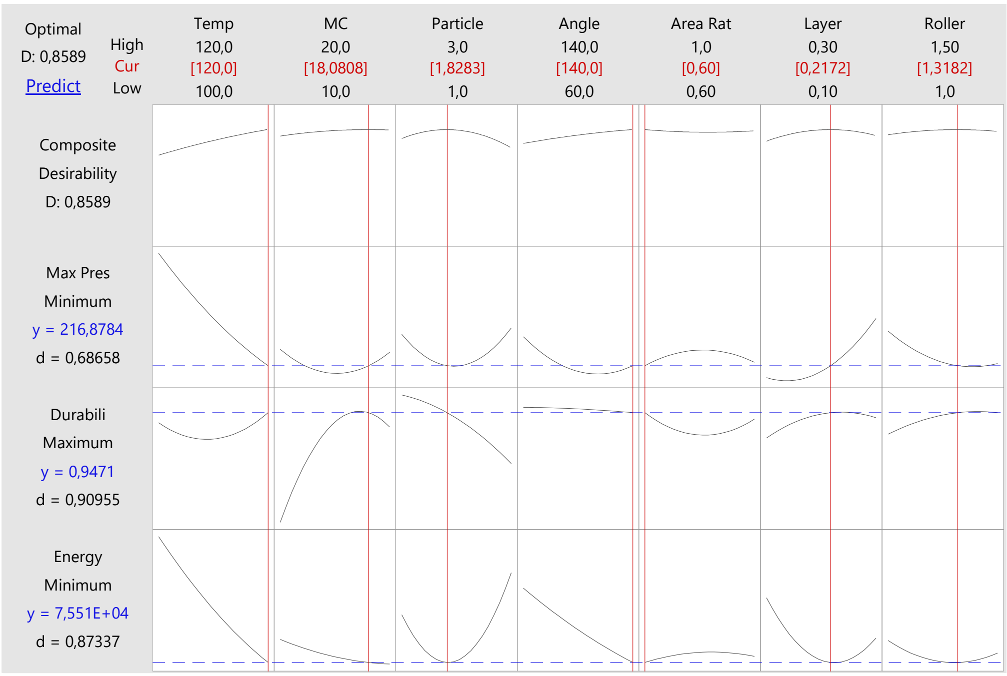

Figure A.5: Showing the optimized conditions for 91.3% durability

A.6 Backward MODEL

$Energy = 573344 - 2334Temp - 12192MC - 53486Particle + 282Angle - 82543AreaRatio - 833874Layer - 26021Roller + 14533Particle \cdot Particle + 1003732Layer \cdot Layer + 80.2Temp \cdot MC - 5.80Temp \cdot Angle + 2673Temp \cdot Layer + 3255MC \cdot AreaRatio + 55.2Particle * Angle - 12590Particle \cdot AreaRatio + 629Angle \cdot Layer + 35170AreaRatio \cdot Roller$

$MaxPressure = 1083.6 - 4.227Temp - 10.18MC - 98.5Particle - 0.375Angle - 274.7AreaRatio - 450Layer - 161.5Roller + 28.34Particle \cdot Particle + 2274Layer \cdot Layer + 0.1031Temp \cdot MC - 1.717MC \cdot Particle + 5.57MC \cdot AreaRatio - 12.43MC \cdot Layer + 0.1630Particle \cdot Angle - 24.60Particle \cdot AreaRatio - 47.0Particle \cdot Layer + 14.74Particle \cdot Roller + 147.8AreaRatio \cdot Roller$

$Durability = 0.750 - 0.001080Temp + 0.04617MC + 0.0162Particle - 0.001840Angle - 0.1233AreaRatio - 0.995Layer + 0.0883Roller - 0.001370MC \cdot MC + 0.00731Temp \cdot Layer - 0.001244MC \cdot Particle + 0.000065MC \cdot Angle - 0.02180MC \cdot Layer + 0.000165Particle \cdot Angle - 0.0311Particle \cdot Roller + 0.002291Angle \cdot Layer + 0.435AreaRatio \cdot Layer$

A.7 Optimization [Backward MODEL] Results

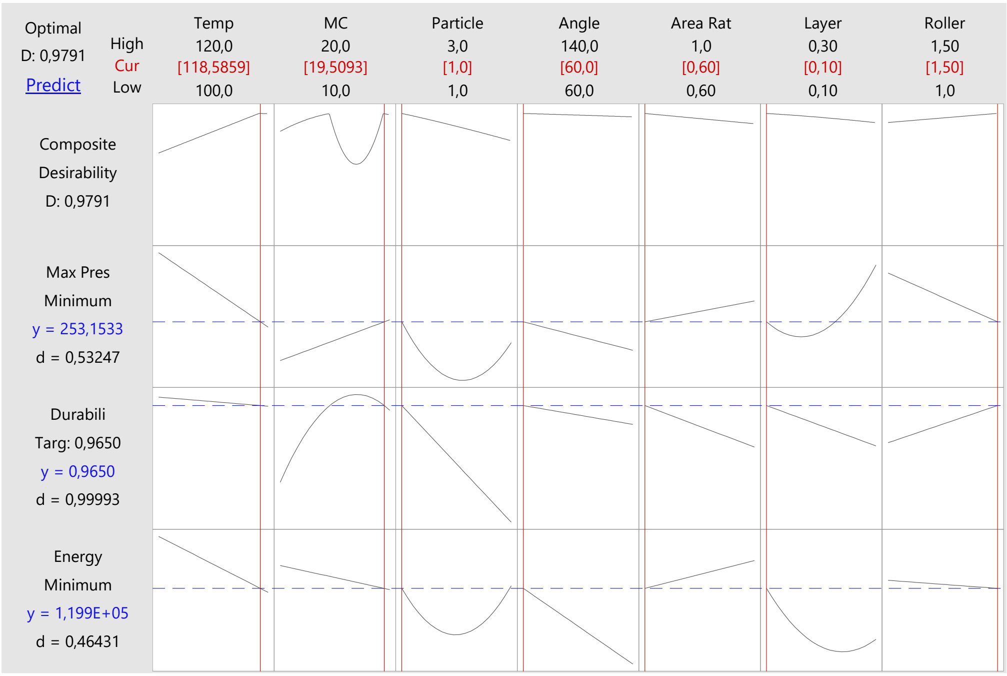

Figure A.6: Showing the optimized conditions for 96.5% durability

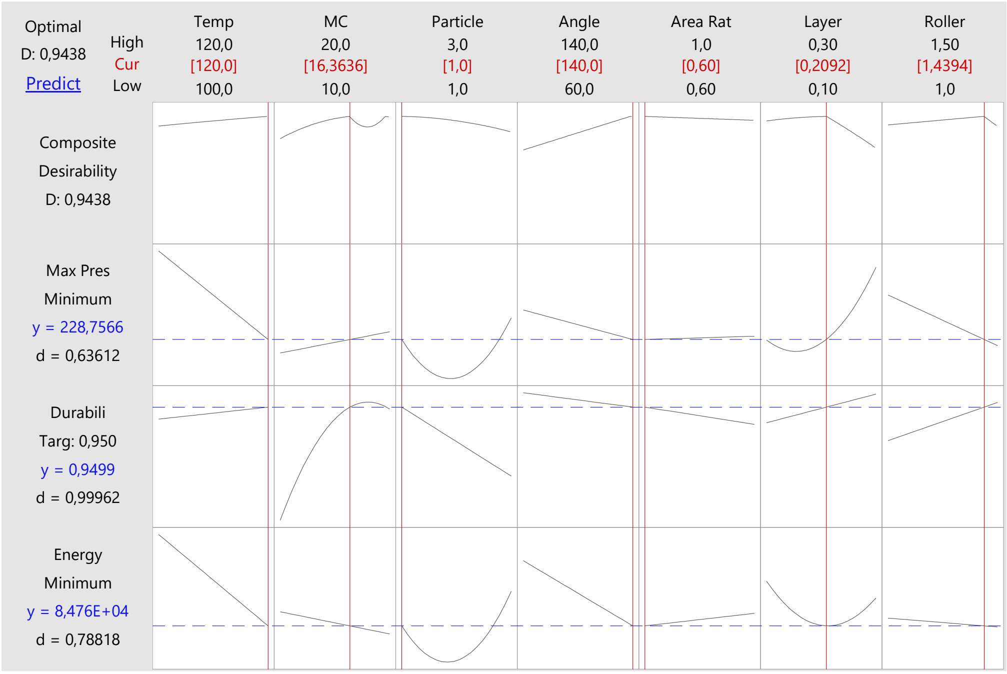

Figure A.7: Showing the optimized conditions for 95.0% durability

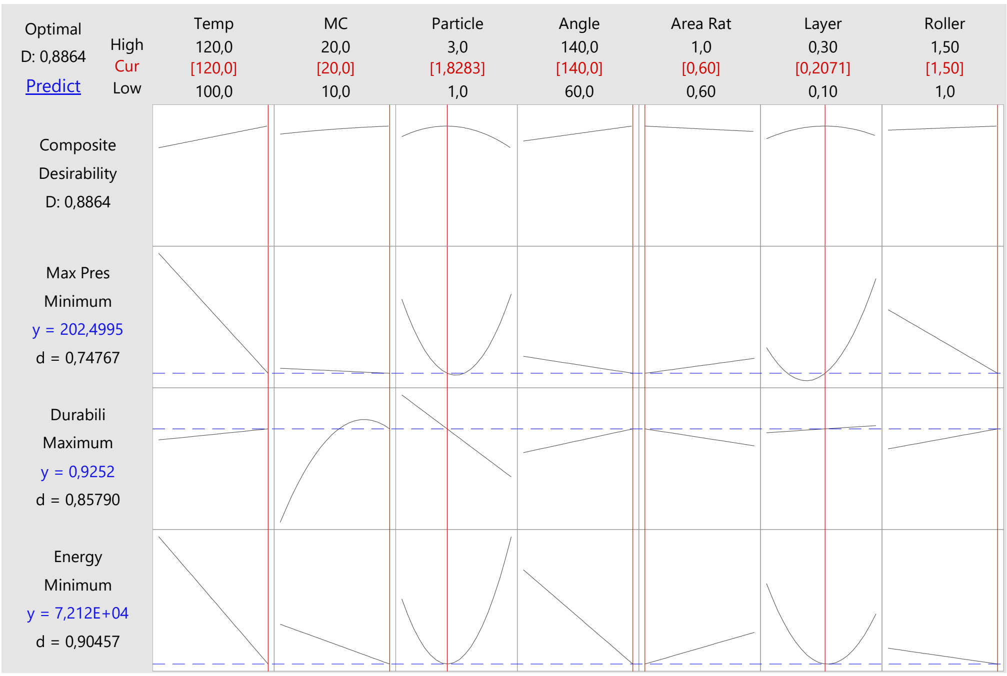

Figure A.8: Showing the optimized conditions for 92.5% durability