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Operational optimization of Ultrasonic particle-liquid separation

Optimizing the separation efficiency of an ultrasonic separator for the recovery of starch particles

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Operational Optimization of Ultrasonic particle-liquid separation

Optimizing the efficiency of an ultrasonic separator for the recovery of starch particles

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Preface

This research report is a product of my internship with the water technology research group at the HZ University of Applied Sciences. I was especially drawn to the specific topic of using ultrasound as a method of filtration because it seemed so innovative to me, the idea fascinated me and ever since I read about it I became determined to make it the topic of my final thesis. I have always been interested in the water technology field since beginning my study of Aquatic Eco Technology in 2014. I thought this research would be the best way to finish my study and receive my bachelor degree by challenging myself with a new and interesting topic while remaining in the field that I wished to specialise in. A research proposal was written first, however, you do not necessarily have to read it before this report.
I started this research at the start of February 2017 with H.J. Cappon as my research supervisor which was lucky for me since he knew everything there was to know about the topic. Although it was challenging, and at times, even frustrating, I am really happy with the end result and I hope that others can become as interested in this topic as I have become.

Firstly, I would like to give a big thank you to my supervisor H.J. Cappon for sharing his valuable time and expertise with me. I would also like to extend my thanks to A. Verkruysse, for all his feedback and constructive criticism. I would also like to acknowledge my friends, my desk buddies and my partners in crime; Bo Schreur and Gabrielle Verbeeke. There has never been a day when I felt alone, especially away from home and without any family nearby because of their continued support, help and advice that has kept me motivated and secure in the fact that they have my back. Their presence in my life is truly a blessing. And last but definitely not least, I would like to thank my family, the people who keep messaging me asking for daily updates on the progress of my research and my well-being, for always encouraging me and trusting in me no matter what. Physically speaking, they are furthest away from me but they are the people closest to my heart.

Summary

A lot of industries in the world use water and produce waste, and with limited water resources and an increasing population, industrial development has grown, putting even more stress on resources. This issue of concern has been recognized and efforts into conserving these precious resources has been made in the form of limiting usage of water and maximizing reuse. Recovering material from waste water is also a priority. The separation of solid-liquid suspensions is a process that is commonly used for this and more recently acoustic separation has been investigated as new technique.

Ultrasonic standing waves (USW) are able to move suspended particles in a direction or fix them in specific locations, in order to generate USW, two opposite sound waves with the same frequency and magnitude are needed to create a fixed wave pattern. An USW filtration device design was made by H. J. Cappon and the operational settings were then further optimized by David Verschoor where he found two optimal settings, one for obtaining clean water and another for one for collecting the suspended particles. It was concluded that testing the system with an increase in power was needed in order to truly determine the effect of power on the filtration efficiency.

The aim of this research was to test this separator device and try to optimize the operational efficiency further by a combination of experiments and models involving influent flow rate, electric power input and frequency. The main research question being; How can the operational settings be optimized to achieve the highest filtration efficiency for starch recovery varying power, flow rate and frequency?

A model based optimization method known as the response surface methodology was used to form a response plot using MATLAB and the optimal settings for efficiency were derived from there.

The frequency was defined in two ways; filtration efficiency for producing clean water and retention efficiency for recovering starch particles. It was concluded that operational settings of 0.5 ml/s, power of 12.5 and a processed starch mass of 560mg (switching interval of 19 minutes and 40 seconds) are the optimal settings for filtration. The optimal settings for the retention efficiency was concluded to be 0.5ml/s, 17.5W and a processed starch mass of 560mg. These settings resulted in a filtration efficiency of 83% and a retention efficiency of 228%, the highest reached yet with this device. Varying the frequency in order to create quasi wave was not feasible using this separation chamber as it always creates a standing wave and no travelling waves. Overall, these settings have still resulted in the highest efficiencies so reached so far with this separation system.

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1. Introduction

With an increasing population, the demand for water rises, whether it be to supply drinking water for human consumption, for industries of which most require water for processes or to meet the increasing global food demand in terms of agriculture. All of these demands are putting pressure on the limited water resources in the world, and due to global climate change, more stress on these resources is expected in the near future (UN-Water, 2013).

This is one reason why disposal of industrial wastewater is becoming recognised as an issue of concern and why farmers and industries are encouraged to limit their water usages and try to find ways to recycle their wastewater for reuse as well as recovering material from it. In this way, costs of water supply and resources can be greatly reduced.

Technology for water treatment and content recovery is already available, depending on the type of particle that needs to be removed, different processes are used. For example, for the removal of insoluble particles from water, physical processes such as settling or screening are used. More recently, the use of ultrasound for water purification and particle recovery is being investigated as a possible alternative to the conventional methods of separation.

1.1 Problem formulation

Current technology for removing suspended solids includes membrane filters which require periodical cleaning whether it was with backwash or chemicals. This technology filters out suspended particles by pumping water to pass through a membrane where particles bigger than the pore size of the membrane will be captured. However, with time, the particles will eventually clog the pores and decrease the filtration efficiency. This will then require cleaning and maintenance which is a temporary solution as the structure itself will degrade over time and will need to be replaced.

In the case of this research, the particle used is insoluble starch, which is quite a sticky substance that can easily clog filters. Hydrocyclones are used in the food industry to concentrate starch and compared to ultrasound, they face higher shear stress level and have a larger chance of fouling.

In this research, an acoustic separation device designed by H.J. Cappon, will be optimized to improve the performance of the separator. The approach being to experimentally optimize three of the most important operational parameters, found by previous design studies (Cappon & Keesman, 2013), in order to obtain the highest operational performance possible.

The most recent experiments on the device (Verschoor, 2015) resulted in optimal settings of 0.5mL/s flow rate, 12W of power and processed starch mass of 0.350 g (with a switching interval of 11 minutes and 40 seconds) resulted in a filtration efficiency of 82%. In fact, only 10W was effectively being used, and the power usage was recognized as a limitation and testing further increase in power was recommended to see if a higher range would have a big influence on the filtration efficiency.

Operationally optimizing this device is needed before this device can operably be used in a larger scale, the optimal operational settings that result in the best efficiency possible, need to be identified. The plan to upscale this device is to use an array of the same sized device, therefore optimizing this device will in fact also optimize it on a larger scale.

1.2 Aim and research question

This leads to the main question to be answered by this research:

How can the operational settings of ultrasonic separation be optimized to achieve the highest filtration efficiency for starch recovery varying power, flow rate and frequency?

The sub questions derived from this question:

1. How is the efficiency going to be defined?

2. What range of flow rate and electrical power will be tested?
3. What settings of flow rate and electrical power result in the highest filtration efficiency?
4. What variation of frequency is feasible and will result in the highest filtration efficiency?

The overall aim of this research is to find the optimal operational settings that result in the highest efficiency possible. How well the current design runs will be tested and the filtering capacity of the US filter will be improved without changing the integral design structure of the acoustic separator. This is because the efficiency needs to increase further before the device can be scaled up. The approach being to experimentally optimize the three operational parameters above.

1.3 Function and content of this report

In the next chapter, a theoretical background explaining the theories and previous research done on this topic will serve as a reference. That will then be followed by the method which will give an overview of what method is going to be used and what data will be collected and how they will be analyzed. After which the results and discussion of the results follows in one combined chapter for the reader's convenience. Lastly, the conclusion answering the main research question is presented, followed by recommendation for any follow up researches.
This report is a research report displaying the research results and conclusion so that this knowledge can be shared with others in a clear and informative way. And it can be used for references, background information, fill a knowledge gap or have it be continued by any interested parties.

1. Theoretical background

The discovery and development of new and innovative technology is a valuable skill to our society. Creating new services and products leads to improvements in quality and can bring about significant changes. The separation of solid-liquid suspensions is a process that is commonly used in many industries and a lot of operations depend on it. Technological advancements in this field are arising, but optimization of these technologies is needed first before they can be widely used.

2.1 Particle filtration

Every industry or laboratory that processes particle suspensions uses particle filtration technology whether they are centrifuges, filtrations screens or settling devices. Currently centrifuges and filters are used in the processing and filtration stages of many industries such as the food and beverage industry and in biopharmaceutical manufacturing. These devices have been used for decades now, and have some drawbacks, centrifuges do not run continuously and require regular cleaning and sterilization. Filters often experience clogging, fouling due to small sized particles and need periodical replacement. Now however, different technologies are being developed that have the potential to be an even better alternative for particle filtration.

The use of USW[1] to suspend particles within a fluid was first described by Kundt and Lehmann, their original goal to make ultrasonic fields visible was quickly overshadowed by how the observed interaction could be utilized for the separation of particles from a fluid (Kundt & Lehmann, 1874).

Researches performed afterwards mostly focused on the miniaturizing of the USW system for particle manipulation and fluid filtration. The two main approaches for US filtration is ultrasound enhanced sedimentation and hydrodynamic acoustic filtration (Prest, Treves, Fielden, Wilkinson, & Hawkes, 2015).

2.2 Ultrasound Standing wave filtration

Acoustic waves are known to exert forces on particles in a liquid or gas. At ultrasonic frequencies, these forces become great enough to concentrate particles at pressure nodes, if particles are denser than the surrounding medium, and these forces are even greater with standing waves.

In the case of this research, the separation process occurs inside a resonance chamber, when the ultrasound is on and a suspension is passed through the chamber, the acoustic pressure will cause particles to line up and be suspended at the nodes (Figure 1b), even while the water is flowing, effectively forming an acoustic filter. In the case of this research, the separator used will create an acoustic wave that is parallel to the flow direction, meaning that the transducer is placed on the top and bottom of the chamber (Figure 3).

There is no disturbance of the wave and no acoustic pressure exerted at the stationary nodes. This is the reason why particles in a suspension will band at these nodal planes and form relatively large clumps (figure 1c) due to inhomogeneity in the sound field (Schram, 1991). As more particles become trapped in the wave pattern, the particles will rapidly begin to aggregate, forming clumps. Once the aggregations reach critical size, they overcome fluid drag forces and settle, as can be seen in figure 1d (Lipkens & McCarthy, 2014). This process can be described as ultrasound enhanced sedimentation.

[1] A standing wave (stationary wave) is a wave in a medium where along the length of the wave there are points (nodes) that are stationary because no acoustic pressure is exerted at those nodes in the axis. In case of this research, a standing wave is generated by two waves travelling in opposite directions combining to form a greater magnitude harmonic wave (Townsend, Hill, Harris, & Mcdonnel, Performance of quarter-wavelength particle concentrator, 2008)

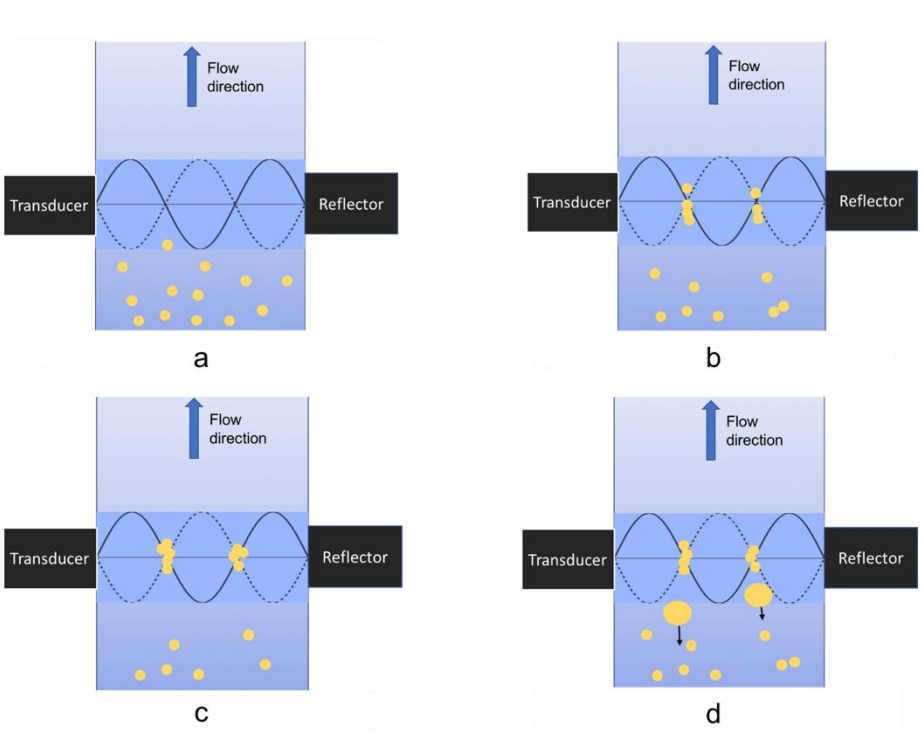

Figure 1 Diagram illustrating the particle separation process using Ultrasound waves. Yellow circles represent the suspended particles in the fluid. (a) standing wave created by transducer and reflector (b) particles become trapped by acoustic forces at the nodes (c) particles stick together forming agglomerations (d) Agglomerations sediment.

Using USW offers an alternative to the traditional filtration methods without some of their drawbacks. It is a continuous operation, which does not contain any mechanical parts so the risk of clogging and membrane fouling is removed. It has potential applications in many fields, for example, separating oil from contaminated water, removing yeast masses from fermented drinks and separating the different components of blood for transfusions.

2.3 Frequency alterations

Interest in the acoustic particle manipulation field has recently been more focused on micro fluidic (lab on chip) applications, which uses ultrasonic standing waves. These devices are usually limited to moving particles into specific planes (nodes). Research has been done on the use of progressive waves in which the trajectory of spherical particles subjected to ultrasonic progressive waves[2] was predicted by use of a numerical model. The model was validated with experiments using small glass spheres dropped in front of a 1 MHz transducer and their trajectory observed, which confirmed the trajectory predicted by the numerical model (Andrade, Buiochi, & Adamowski, 2009).

Switching between modes has been reported by Glynne-Jones *et al* as a technique for electronically changing the position of agglomerations in an acoustic particle manipulator. Switching between two modes of frequency with two different nodal positions causes the particles to migrate from one position to another effectively. It was shown that using this technique it is possible to control the agglomerated position of the particles by switching in a controlled manner between modes (Glynne-Jones, Boltryk, Harris, Cranny, & Hill, 2009).

Another way in which suspended particles have been transported was done by establishing a fixed frequency difference between two transducers (Whitworth, Grundy, & Coakley, 1991). A selected increase in frequency within the range of +/- 2Hz to 150Hz was applied which caused the fixed particles of polystyrene divinyl benzene to be moved towards the transducer with the lower frequency. It was discovered that the movement speed of the particle clumps is linearly proportional to the frequency difference.

[2] Progressive waves, also known as travelling waves, are waves in which particles in the medium move progressively in the direction that the wave propagates.

2.4 Operational settings and efficiency

An ultrasonic particle filter was designed and built by H.J. Cappon. The resonance chamber, seen in figure 3, is part of the separation unit, where two transducers attached to it create the actual standing wave that will separate the particle from the fluid in the flow direction which is perpendicular to the transducer. This chamber contains three flow lanes and was designed in such a way as to have equal flow profiles in these lanes (Cappon H. J., 2014) (Cappon et al, 2013).

The results of an optimization experiment using this exact filter for attaining the cleanest water from a starch suspension resulted in the following recommendations; set the filtration setup to a flow rate to 0.5 mL/s, a power setting of 12 W and a switching interval of 11 minutes and 40 seconds (with a backwash time of 20 seconds). As for the collection of suspended particles from the contaminated water, the best efficiency was gained when the filtration setup was at a flow rate of 1.37 mL/s, a power setting of 12 W and a switching interval of 4 minutes and 15 seconds (with a backwash time of 20 seconds) (Verschoor, 2015). This conclusion was based on the range that was tested. Power of 12 W (effectively 10 W) was the highest used in the entire experiment, and from the results it can be seen that a higher efficiency favored the use of higher power.

This however is not necessarily always true, as previous experimental studies show that variation exists. Figure 2 shows the concentration of the filtrate solution at different amplitudes. While it is clear the lowest particle, concentrations were indeed obtained at the highest voltage of 12 V, it does not explain the uneven trend (Bekker, Meyer, Pretorius, & Van der Merwe, 1997) (Muralidhara, Senapati, & Beard, 1988). This would most likely be dependent on the particle characteristics such as its agglomeration due to shear stress at a specific voltage. In that same experiment, flow rate effect on separation efficiency was tested, resulting of the best efficiency (65%) at a flow rate of 2.5 ml/s.

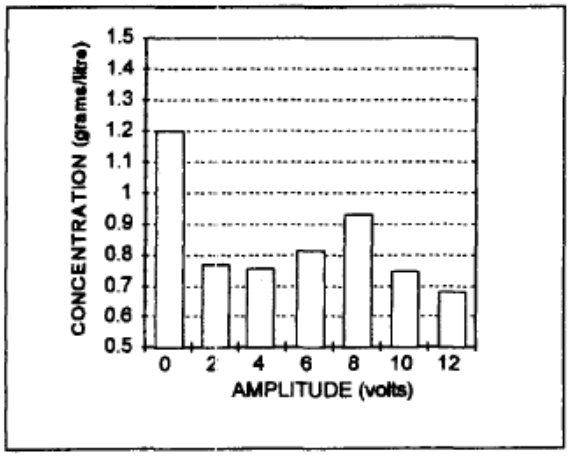

Figure 2 Separation concentration at applied voltages (Bekker, Meyer, Pretorius, & Van der Merwe, 1997)

In another research performed to harvest microalgae using the same standing wave separation technique, efficiencies of higher than 90% were reached at high biomass concentrations and 4-6 L/day flow rate. It was concluded that the ingoing flow rate had a large influence on the efficiency, with efficiency increasing at lower values (Bosma, van Spronsen, Tramper, & Wijffels, 2002).

When there is no ultrasound generated sedimentation is considered to be the form of filtration, (Hill, Townsend, & Harris, 2008) this will depend on the viscosity of the liquid, particle size in the solution and the flow rate.

2.5 Limitations

Despite the huge potential for this technology, it is not that widely used today. In fact, there is only one design that is currently available in the market which is the BioSep acoustic perfusion system by Applikon. Even though acoustic separation systems are second to centrifuges in efficiency, they still have the advantage of being easily adapted for continuous flow (Hawkes & Radel, 2012).

The separator in Figure 1 was used in a series of experiments to find optimal settings for high efficiency. Even though a separation efficiency of 76% was achieved, this is lower than was predicted. Reasons as to what may have affected the efficiency includes the limited power input, the pulsation caused by the pump and the presence of a wide range of particle size in the starch suspension (1-100 μm). The separator is able to trap a specific range of particles so any particles outside of that range may not be removed, the smallest starch particle size that was captured effectively was found to be 10 μm (Cappon, Stefanova, & Keesman, 2013).

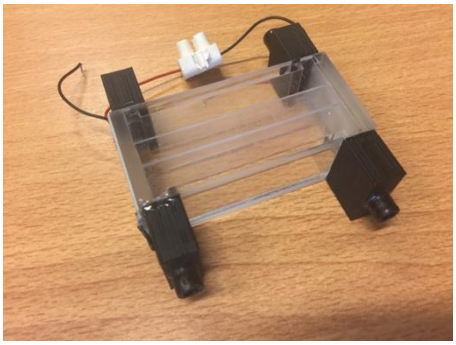

Figure 3 Separator designed by H.J. Cappon. wiring is for connection of transducers and black fixings are inlets and outlets for the fluid. (Image courtesy of author,2017)

While Ultrasonic standing waves can be used to trap biological particles of the micron scale, large variations in particle size may prove difficult to separate. Previous experiments have proved that particles extremely small in size show a lack of separation in these types of filters (Bekker, Meyer, Pretorius, & Van der Merwe, 1997).

2.6 Theory application in research

In order to capture smaller sized particles, the flow rate would need to be decreased or the power should be increased. In order to achieve higher power supply, the use of two synchronized amplifiers allowing an increase on the voltage limitations. As for the pulsation caused by the pumps, a pulsation dampener connected to the chamber may reduce its effects because it would absorb the energy from the pulse wave created by the pump, a lot like a shock absorber. Since high accelerations also causes high turbulence, a maximum flow rate of 3mL/s was set to allow laminar flow and efficient filtration.

For frequency alterations, quasi-standing waves will be used. If two sound forces are applied, one at each end of the chamber, with a minor frequency difference between them, a standing wave in which the nodes move will be created. This is known as an ultrasound quasi standing wave (UQSW) and it can be generated with the use of two amplifiers.

All in all, acoustic separators still need work when it comes to producing high yields in a consistent manner, however, this innovative separation method has great potential and possibilities in the filtration and material recovery field.

2. Method

3.1 Materials

The main apparatus and materials used in this research study:

- 1 L beaker (1)
- 500ml beakers (3)
- 250ml beakers (3)
- Insoluble Potato Starch powder
- Demineralized water
- Magnetic plate
- Stir bar
- Masterflex® L/S pump drives (2)
- Resonance chamber
- Bio Sep ADI 1015 Amplifier (2)
- Portable turbidity meter (Martini instruments MI415)

3.1.1 Suspended particle

In this experiment, potatoes starch suspension will be used, the same type of starch used previously in experiments with the separator used in here. This white powder is a polysaccharide of glucose and is primary food in global food production. Starch grains vary in size from approximately 5 μm to 100 μm, with potato starch having the largest sized grain. Filtration is a key step in the production process of starch since it is a wet process, and particle size and distribution has a big effect on the efficiency of this step (Sparks, 2012).

3.2 Experiment set up

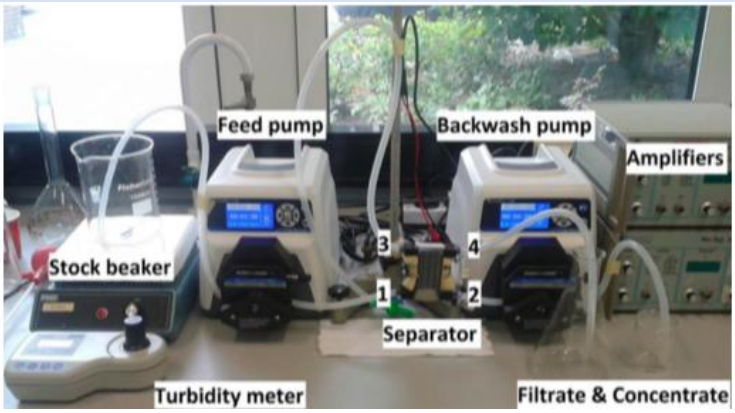

Figure 4 Particle separation unit set-up, equipment is labelled in the picture, numbered inlets/outlets is (1) inlet, (2) backwash, (3) air-fluid damper, (4) filtration outlet (Cappon, 2014)

The stock beaker contains the stock solution (starch suspension) which is kept in suspension using a magnetic stir bar. As is seen in the diagram, there are two pumps present, one controls the flow rate of the stock solution and the other the backwash flow rate. There are two glass beakers to store the filtrate (clean water) and the concentrate (suspended particle). The resonance chamber, is the main part of the separation unit and it will contain the ultrasonic waves that will separate the particle from the liquid. The amplifier provides the needed power, frequency and timing to generate the standing ultrasonic waves by sending a signal to the transducers on the separator. The amplifier will automatically find the frequency to produce the standing, the frequency can also be selected manually. The use of two linked amplifiers instead of one, should give a further increase in power output.

The separator has 4 outlet/inlet ports labelled in Figure 4 , during a normal filtration sequence, the starch suspension is fed by a pump to the separator through port 1. Inside the chamber the particles in the suspension are separated by the USW and the filtrate then exits through port 4 into the filtrate beaker. After the filtration run is over, the backwash run begins using the second pump, using the filtrate water from port 4 to wash out starch particles that were retained in the chamber out through port 2 and into the concentrate beaker.

A timer is used in each of the pumps and amplifiers so that they switch off and on according to the desired length of filtration sequence. Figure 5 shows a general timeline of what the on and off times look like for the pumps and amplifiers relative to each other. Pump 1 can only run when the amplifiers are on, and ultrasound in generated in the chamber for maximum filtration effect, and pump 2 backwashes after both pump1 and the amplifiers have switched off.

Figure 5 Time line of operation switching on and off times for the feed pump (pump 1) and the backwash pump (pump 2) and the amplifier in relation to each other (image courtesy of author).

3.3 Approach

The approach applied in this research was a numerical and experimental approach. A method based only on experiments would be both time consuming and expensive. Therefore, a design of experiments (DoE) is used to create a mathematical model that would predicted the effect of a parameter or several parameters on the response. For this only a limited number of experiments can be done, saving time and money, but still able to find the optimal response. Figure 6 shows the general process of finding the optimal settings from the design of experiments all the way to validating the optimal setting found.

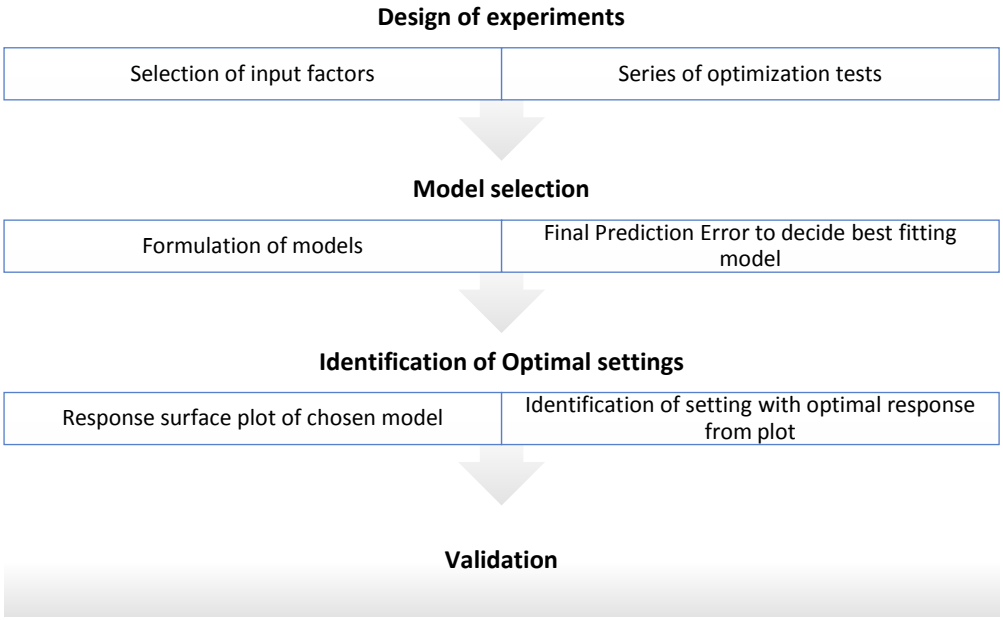

Figure 6 Schematic chart of the research approach

In the case of this research, the turbidity is used as the response variable. To measure the effect that the parameters have on particle filtration efficiency the turbidity (dependent variable) of the filtrate will be measured. The turbidity indicates the amount of light scattering in a liquid with particle suspension. In case of the filtrate for example, the turbidity should be less than that of the stock because the filtration will have reduced the suspended particles from the solution making it clearer, there by the turbidity is lowered. This is the most effective and reasonable method of measuring the effect of different variables on the filtration efficiency.

Efficiency in the case of this research can be defined in two ways. The first is filtration efficiency; efficiency in producing clean water in the filtrate volume. Or second, the retention efficiency; efficiency in recovering starch particles in the concentrate volume.

3.3.1 Response surface methodology

Response surface methodology (RSM) is the method that will be used in this research to answer the main research question. This is a mathematical technique for model building by use of experiments (Keesman, 2011). The objective being, the optimization of a response or output (turbidity), which is

influenced by independent variables (flow rate, power, processed starch mass). This experiment is basically a series of tests or *runs* in which the independent variables are changed so as to observe the changes in the output response (Response Surface Methodolgy, n.d.).

For example, in the case of this research, the level of Power (x_1) and flow rate(x_2) to optimize efficiency (*y*) of the separator can be represented in the following way;

$$y = f(x_1, x_2) + \varepsilon \qquad \text{Equation 1}$$

Where ε represents the error observed in the response *y*. The efficiency is a function of the levels of the power and flow rate as seen in formula 2, and the surface represented by $f(x_1, x_2)$ is the response surface. The response is usually represented graphically in 3-dimensional space so the shape of the response surface can be visualized.

This method was chosen because compared to others it is an inexpensive analysis method that allows you to gain as much information as possible from a limited number of experiments.

3.3.1.1 Design of experiments

This is a series of tests (runs) which will be used to formulate a matrix which uses a range of the input factors. Normally, the design variables to be considered are determined first, however, the three most influential variables have already been identified by the client due to previous research on the topic.

A filter run with three batches will have three samples of filtrate and three samples of 50 ml concentrate, the turbidity of each sample is measured thrice so there were nine measurements for each stream. Four levels were used for each factor; the range for flow rates tested was 0.5 ml/s to 2m/s, this is because sedimentation is the dominant process at flow rates below 0.5 ml/s and flow rates above 2 ml/s because at higher flow rates, the difference between the filtrate and stock turbidity is too small (<100FNU) (Cappon H. J., 2014).

As for the power range, four levels were also used starting at 10W up to 17.5%, this is because the most recent experiments on this device used 10W as the highest power tested, so to find out if a higher power range would have more influence on the efficiency, 10W was the first level used in the runs (Verschoor, 2015).

Since the mass of starch entering the chamber continuously before backwash might also influence the efficiency, this was also a factor, the levels depended on the flow rate since the run time was the same for all the runs, the starch mass had four levels; 0.14g, 0.28g, 0.42g, 0.56g which is a completely different range from what was tested previously. Not all the combinations of levels were run however, due to time constrictions which was not enough to run all 64 combinations. The combinations that were done however, can be seen in Table .

Table 1 Experimental runs combinations of flow rate, power and processed starch mass (switching interval)

Flow rate (mL/s)	Power (W)	Starch mass (mg)
0.5	0	140
0.5	10	140
0.5	12.5	140
0.5	15	140
0.5	17.5	140
1	0	280
1	10	280

1	12.5	280
1	15	280
1	17.5	280
1.5	0	420
1.5	10	420
1.5	12.5	420
1.5	15	420
1.5	17.5	420
2	0	560
2	10	560
2	12.5	560
2	15	560
2	17.5	560
0.5	12.5	420
1	12.5	420
2	12.5	420

3.3.1.2 Model formulation

The first step to establishing the structure between the dependent and independent variable is to find a suitable approximation to their true relationship. Usually, low order polynomials (1st or 2nd order) are most commonly used. Below is a general model (2nd order):

$$F(Q, m, P) = a_0 + a_1Q + a_2m + a_3P + a_4Q^2 + a_5m^2 + a_6P^2 + a_7Qm + a_8QP + a_9mP \quad \text{Equation 2}$$

When independent factors are assumed and cross terms are neglected this results in the following:

$$F(Q, m, P) = a_0 + a_1Q + a_2m + a_3P + a_4Q^2 + a_5m^2 + a_6P^2 \quad \text{Equation 3}$$

f(Q, P) = Relative turbidity [-]
Q = flow rate [mL/s]
P = power setting [W]

Relative turbidity can be calculated in the following way:

$$\textbf{Turbidity percentage} = \frac{Concentrate/filtrate\ turbidity}{Stock\ turbidity} * 100 \quad \text{Equation 4}$$

The data collected from the experiment will be tested with several models and their error calculated using MATLAB.

3.3.1.3 Final Prediction Error

The Error of the model fit is defined as the difference between, in this case, the measured turbidity and the calculated turbidity using the formulated model. Akaike's Final Prediction Error criterion (FPE) provides a way of measuring the model's quality, according to the theory of Akaike, the most accurate model has the smallest FPE. The below FPE formula will be used to determine which model fits the data most accurately using MATLAB:

$$J_{\mathrm{FPE}}(\mathcal{M}) := \frac{1 + d_{\mathcal{M}}/N}{1 - d_{\mathcal{M}}/N} \frac{1}{N} \sum_{t=1}^{N} \frac{1}{2} \varepsilon^2\left(t, \hat{\vartheta}\right) \quad \text{Equation 5}$$

N = number of values in the estimation data set

d_m = number of estimated parameters.

The optimal model for the data is decided using FPE by allowing the identification of the point where further increase in the number of fit coefficients (a_i) results in no significant difference to the estimation error, and this is done by searching for the 'knee' in the graph. Once this point is identified, the number of parameters needed and therefore the model complexity is decided. The next step would be to evaluate all the possible combinations for that specific number of parameters. The final model from that specific parameter number will be the one with the smallest FPE value.

3.3.1.4 Response surface

Once a model is selected, it is then used to plot a response surface plot in MATLAB. This plot will be used to identify the optimal settings for the factors tested in order to attain the desired filtration or retention efficiency.

3.4 Experiments

3.4.1 Preliminary experiments

Preliminary experiments are important to establish relationships between variables and to identify the ranges that can be used in this experiment. They are also used to identify any potential problems that may occur and allow their correction before the main experiment takes place.

3.4.1.1 Initial Saturation run

After a certain time of filtration, the separation chamber becomes saturated with the starch and efficiency decreases. The time at which this happens can be set manually as will be the case in this research. When saturation occurs is dependent on the processed starch mass going into the chamber within a specific time. Therefore, to establish the best run time for a flow rate will depend on this initial experiment. The time at which the filtrate turbidity becomes equal to that of the stock solution is the saturation point.

This will be achieved by approximately 2 runs, each with a different flow rate. During each run samples are taken from the filtrate at regular time intervals. The power level and starch solution volume and concentration are controlled so that they are constantly the same for both runs. Two samples are taken at a time, each measured twice, resulting in 4 data points, per measurement time. The results will then be plotted in a line graph, turbidity against time, and from there, the saturation time for each flow rate can be determined. The filtration duration will be flow dependent on these results so that the same level of saturation (processed starch mass) is maintained for the final optimization runs. See 8.1: procedures for the full method.

3.4.1.2 Concentration- Turbidity relationship

From previous research (Verschoor, 2015) it is known that starch turbidity and concentration have a linear relationship. Therefore, the turbidity will be used to measure the concentration. Another main reason for this experiment is to see how accurate the measures are. As mentioned, the turbidity will set the efficiency, so if it is seen that there is a large overlap in turbidity between the different concentrations then that will indicate that there is not enough difference between turbidity measurements and so more measurements will probably be needed during the optimization test runs.

In this experiment, a dilution series is created, with the concentrations known for each dilution, three samples are taken from each. The turbidity is measured thrice for each sample, resulting in 9 data points per concentration.

Then a turbidity against concentration graph is plotted, and its formula derived. The standard deviation lines in the graph will show clearly the extent of overlap between the points. From this,

the number of measurements needed for the optimization runs will be concluded. The full method can be found in 8.1: procedures.

3.4.2 Optimization experiment

After the initial experiments are through, the ranges of the variables and number of measurements needed is known. The range of the power and flow rate that will be tested depends upon the initial experimentation as it depends greatly on the capacity of the equipment used.
This experiment uses a series of experiments with different combinations of the independent variables that are run in the USW separator, and their turbidity measured. A blank run for each flow rate, with no ultrasound, will be done as a reference point for comparison since sedimentation is still occurring. A starch suspension of 1 g/l was run for a time depending on the required processed starch mass. The turbidity of each sample is then measured thrice. The data collected were then normalized and used to find which model was the best fit as explained previously.

3.4.3 Validation Experiment

After the optimal settings are decided according to RSM approach a validation experiment using those optimal settings was done in order to determine whether these settings are in fact optimal as predicted by the model. This involved two run using those settings.

3.4.4 Frequency

After the optimal power input and flow rate are decided, the frequency difference needed to produce a slowly travelling wave needed to be figured out. The aim was to introduce a frequency shift between the two transducers which would force the starch particles to moved downwards instead of being fixed in place as described in literature previously (Glynne-Jones, Boltryk, Harris, Cranny, & Hill, 2009) (Whitworth, Grundy, & Coakley, 1991). This can be done by trial and error method, since there are two amplifiers available, the frequency can be changed manually. An oscilloscope will also be used to view the waves on screen and observe how the signals from the amplifier affect the waves. This will help in finding the best frequency difference to create the quasi standing wave. After that is decided, it can be validated by testing this frequency with an experimental run to see if it in fact can be more efficient than the conventional USW. The results of this experiment are discussed in the Results and discussion chapter.

3. Results and discussion

The results of the preliminary, optimization and validation experiments are presented in this chapter. The results are described and discussed together for the reader's convenience. All raw data will be present in 8.2: Experimental data.

4.1 Preliminary experiments

4.1.1 Turbidity- Concentration relationship

This experiment measured the turbidity of six concentrations, three samples per concentration each measured thrice resulting in 54 measurement points, full method in 8.1: procedures.
The relationship between turbidity and concentration is linear as expected, see Figure 7, and the points line up well (R^2 = 0.99896). The error bars are quite narrow and do not overlap with each other, meaning that the measures are quite accurate and so distinguishing between the concentrations of the filtrate and concentrate is possible. Therefore, the same number of measurements will again be used for the optimization experiment. Now that the relationship is established, turbidity can be converted to concentration and vice versa using the line equation.

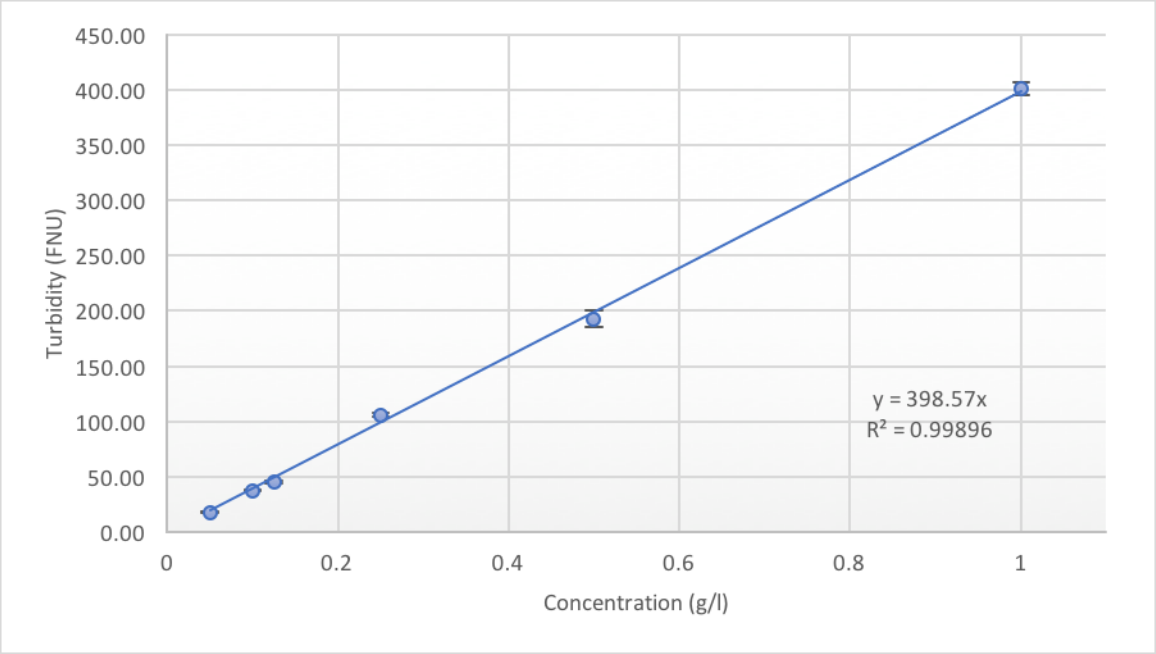

Figure 7 Graph displaying the relationship between turbidity and concentration. The error bars show the standard error per point, and the line equation (y = 398.57x) and fit of the line (R^2 = 0.99896) are also displayed.

4.1.2 Saturation progress

The breakthrough point is where the turbidity of the filtrate is equal to or higher than that of the stock turbidity. At this point the chamber is saturated with processed starch and is no longer able to capture starch particles efficiently. In figure 8, it can be seen that the run for 0.5 mL/s does not reach a saturation after 34 minutes of continuous filtration. As for the 2mL/s run, the saturation point was reached at 6 minutes, where the average turbidity at that point was equal to 289.75 FNU, and the turbidity of the stock solution has a band width of 271-482 FNU (stock bandwidth in figure 8). Therefore, the run time for the system has to be below this point, that is reached after 6 minutes of continuous filtration. The raw data is available in 8.2: Experimental data, and it shows that the range of turbidity measures for the filtrate and stock overlap. At a flowrate of 2 mL/s the processed starch mass in the chamber at the saturation peak would theoretically be 0.72 g. The third point for the 2 ml/s run, reached a max turbidity of 329 was excluded, the other 3 measurements taken at that point all had been below the stock bandwidth so the turbidity at that points was most likely due to some error when measuring the turbidity. The chosen run time was therefore 5 minutes, well before the chamber is saturated.

A flow rate of 2mL/s was the highest flow rate used in the experiment, and so its saturation peak is going to be reached sooner than any of the other slower flow rates tested. And since the saturation time for this flow rate will not be exceeded, any lower flow rates will not reach their saturation peak at this chosen run time. That is why it was unnecessary to make a saturation run for all of the flow rates that will be tested.

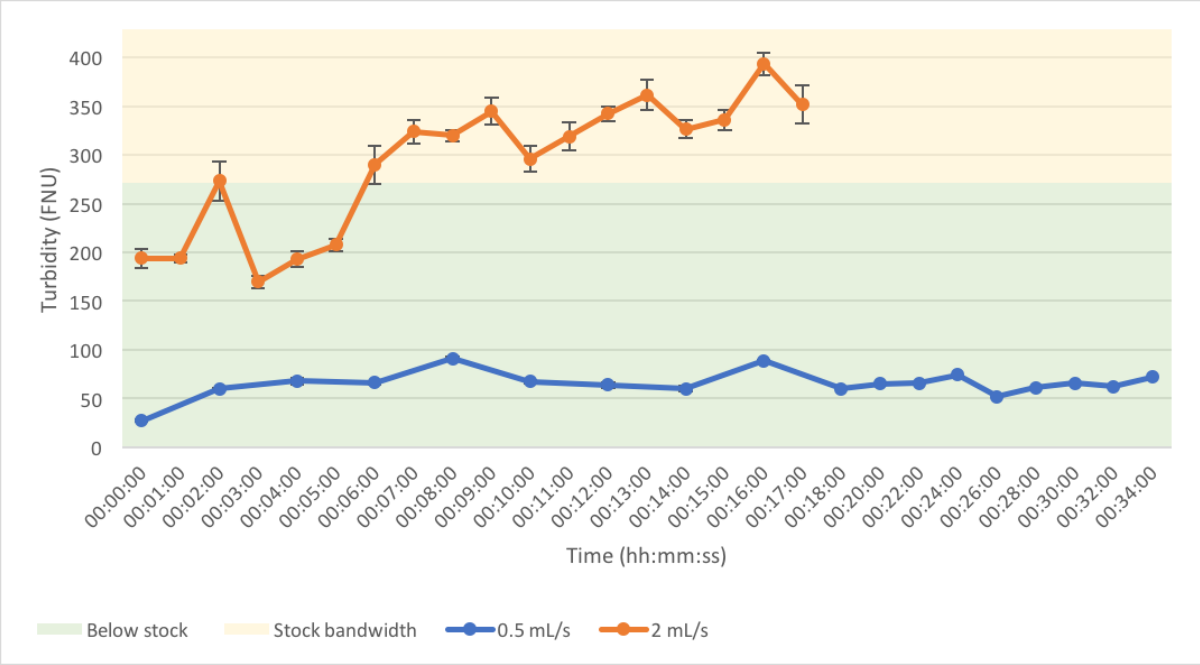

Figure 8 The turbidity of the filtrate through time when ultrasound is off. Turbidity is shown for two flow rates, 0.5 ml/s with 2 minute intervals between measurements and 2 ml/s 1 minute intervals between measurements. The bandwidth of the stock turbidity is shown in orange, and green represents the range of turbidities below that of the stock. Error bars show the Standard error ($\sigma/\sqrt{n}$).

The run time of the pumps was adjusted to 4 minutes and 40 seconds in the following experiments, due to timer restrictions of the amplifiers. Therefore, the exact pump run time actually depends on what the timer in the amplifier will allow. So, taking the chosen 5 minutes run time as an example, it was reduced to 4 minutes and 40 seconds, as the amplifier could only be set to 5 minutes exactly and according to the timing shown in Figure 8 the pump needs to switch on and off 10 seconds before the amplifiers.

4.2 Optimization experiments

The optimization experiments are shown in a number of bar charts, with each run having a bar for stock, filtrate and concentrate turbidity. The stock is included as a reference point as it is the starting turbidity and efficiency is calculated based on the difference from the stock. The runs are grouped into graphs depending on the flow rate, except for the last graph (figure 13) where the flow rates are compared with each other. The error bars in these graphs represent a 95% confidence interval so it is easy to see at a glance if there is a significant difference between the results.

In figure 9, the blank shows that there is quite a difference between the turbidity of the stock and filtrate, indication that some filtration did occur at a flow rate of 0.5 ml/s even when the ultrasound is switched off due to sedimentation. In fact, the turbidity is reduced by about 52% at this flow rate. The values of the concentrate were unexpected since they are below or higher than the turbidity of the stock, when it was expected they would be higher. The reason for this is due to the processed starch mass, which at a flow rate of 0.5 ml/s and a run time of 4 minutes and 40 seconds, only 0.14 g of starch is passed through. And since all the runs have the same run time but different flow rates, the processed starch mass varies depending on the flow rate. In this case, this flow rate processes the least amount of starch mass and therefore, there is in fact very little starch in the chamber when backwash occurs, resulting in the low readings for concentrate turbidity. This was confirmed with a mass balance for the starch. The highest filtrate efficiency reached at 0.5ml/s was 85% at a power of 12.5W, although the results of powers 12.5W-17.5W were all closely ranged and overlap indicating that an increase in power did not cause an increase in filtration efficiency. Full data of the optimization runs can be found in 8.2: Experimental data.

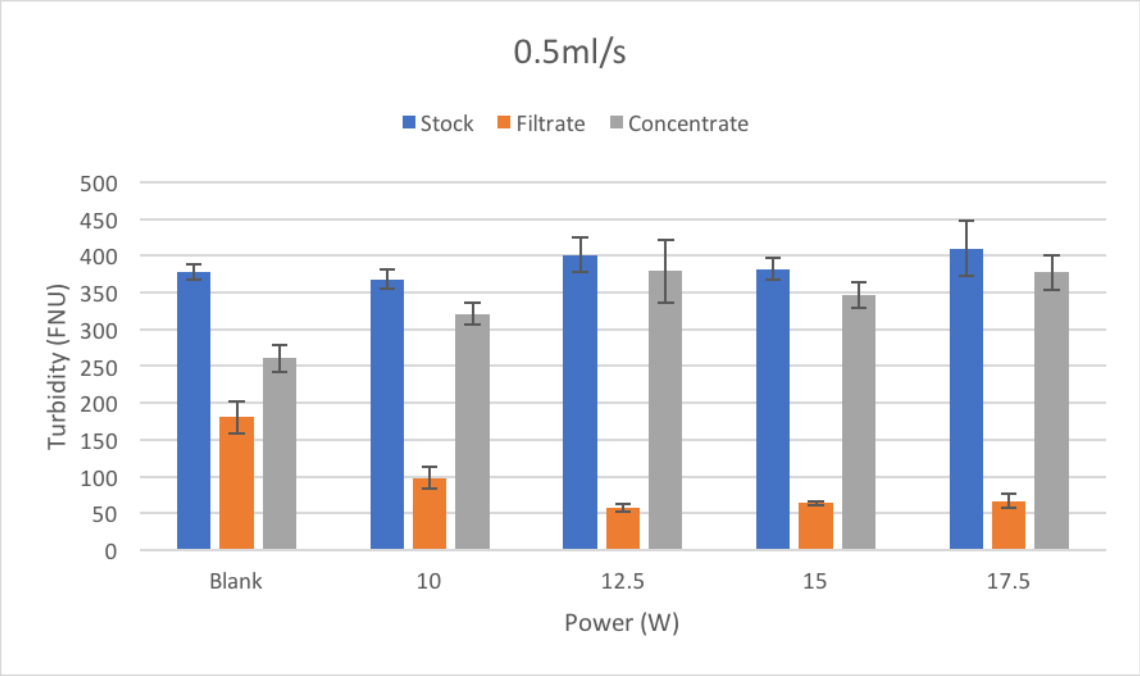

Figure 9 The effect of ultrasound on the turbidity of the filtrate and concentrate compared to that of the stock at varying power outputs at a flow of 0.5 ml/s. The error bars represent a 95% confidence interval.

At a flow rate of 1 ml/s and a processed starch mass of 0.28g (figure 10), the blank run shows that sedimentation does not have as big an effect on filtration as it did with 0.5 ml/s flow rate. This is due to the fact that sedimentation is more prominent at lower flow rates. The same pattern is observed with Figure 11 and Figure 12.

At this processed starch mass, the concentrate has increased to be up to twice that of the stock. The highest starch retention efficiency reached (123%) at a flow rate of 1 ml/s was at a power input of 10W. As for the filtration efficiency, it was 57% at a power input of 15W. Again, the filtration efficiency seems not greatly influenced by the varying power input, with the biggest filtration efficiency difference being 3%.

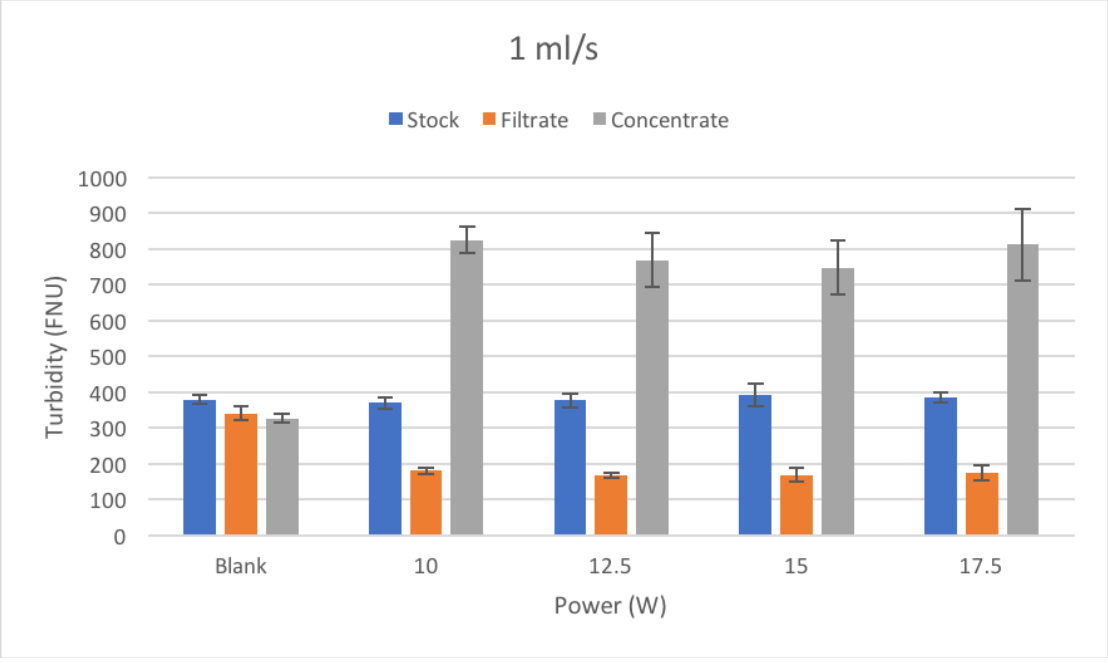

Figure 10 The effect of ultrasound on the turbidity of the filtrate and concentrate compared to that of the stock at varying power outputs at a flow of 1 ml/s.

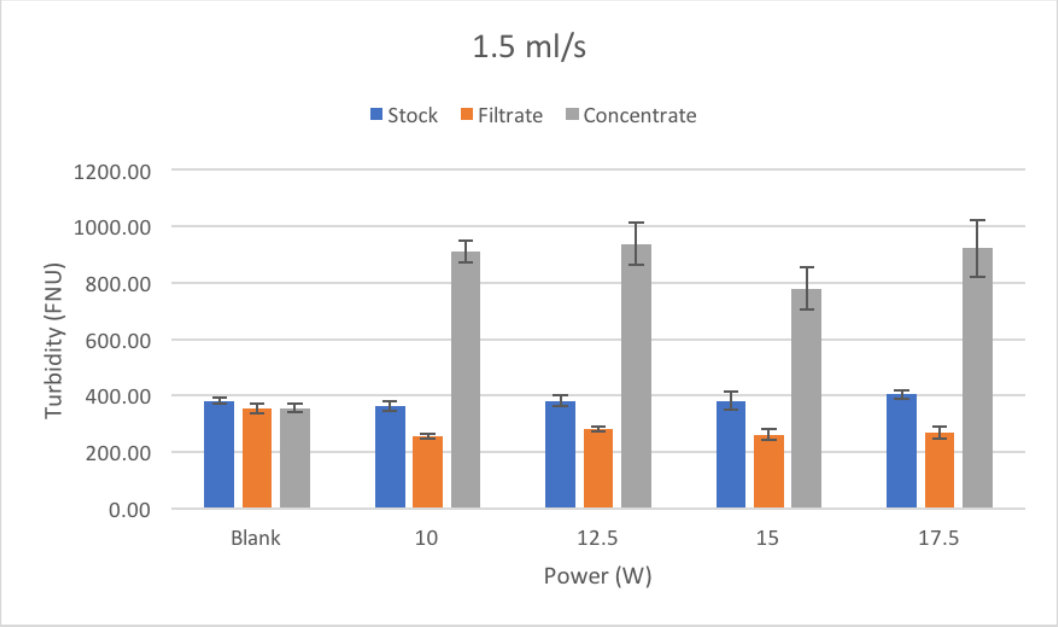

Figure 11 The effect of ultrasound on the turbidity of the filtrate and concentrate compared to that of the stock at varying power outputs at a flow of 1.5 ml/s.

At this point, figure 11, the turbidity of the stock, filtrate and concentrate in the blank run overlap. Indicating that at this flow rate sedimentation does not have an influence on the results. At a flow rate of 1.5 m/s, the processed starch mass is 0.42g for all of the above runs. For the concentrate the highest relative turbidity at 1.5 ml/s was achieved at 10W.

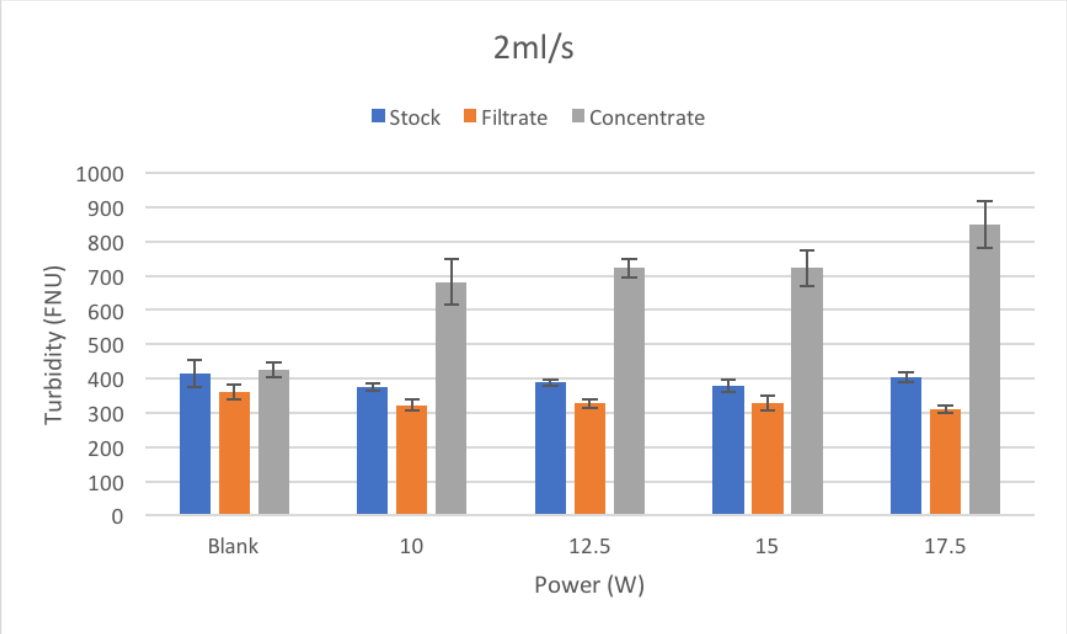

Figure 12 The effect of ultrasound on the turbidity of the filtrate and concentrate compared to that of the stock at varying power outputs at a flow of 2 ml/s.

At a flow rate of 2 ml/s, the processed starch mass is 0.56g, the highest filtration efficiency was 23% at a power input of 17.5W. The filtration efficiency seems to decrease with increasing flow rate. On the other hand, the retention efficiency reached a 110% at a power input of 17.5W, see figure 12.

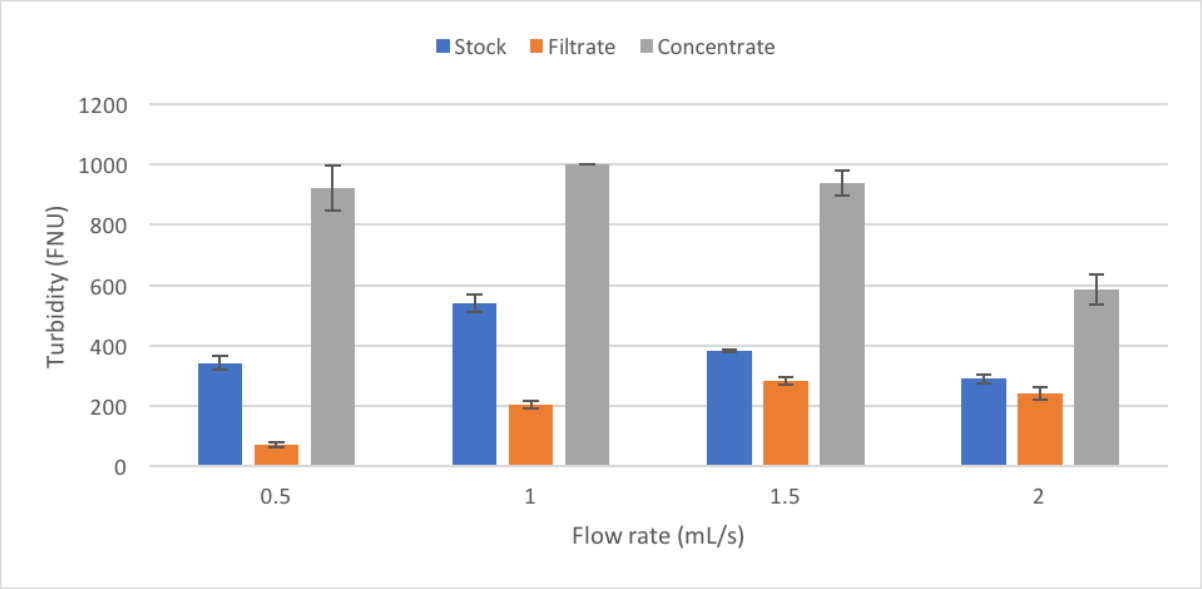

Figure 13 The effect of flow rate on the turbidity of the filtrate and concentrate compared to that of the stock at constant power output of 12.5W with a processed starch mass of 0.42g for all flow rates.

After studying the previous results, it was decided that a few more runs where the processed starch mass was the same for all four flow rates were needed. Making the power input 12.5W constant for all the flow rates, making it easier to compare the flow rates with each other by taking out the processed starch mass and power variations.
Figure 13 shows the results of the runs where the switching interval was changed for each flow rate to achieve the same processed starch mass of 0.42g for all four flow rates. These can be compared easily because chamber has the same quantity of starch coming in even at different flow rates. The lowest relative turbidity for filtrate was 20% at a flow rate of 0.5 ml/s, and the highest for the concentrate was also at a flow rate of 0.5 ml/s with a relative turbidity of 269%.

4.3 Frequency Experiment

Unfortunately, the plan to make a travelling (quasi) wave in this separator was not feasible. The reason for this was that the device was built to create a standing wave, therefore, the structural design of it always produced a standing wave. The chamber has two transducers opposite each other and the glass that the device is built from acts as a reflector. So, whatever wave is created by the transducer, no matter the frequency, is always reflected by the glass resulting an opposite wave of the same frequency also known as a standing wave. Using only one transducer did not work, because of the built-in reflector and changing the range of the frequency also did not solve this issue. Blocking the reflector with a piece of rubber would block the transducer at the same time. Therefore, the only way to do this would be to build a new device that is specifically designed to use a quasi-wave for particle separation.

4.4 Models and validation

The Selection of the model that has the best fit with my results and its response surface plot are displayed here. The optimal settings are also found from this surfaceplot

4.4.1 Final Prediction Error

The best fitting mathematical model was selected using the Akaike's Final Prediction Error. There was a total of 23 models derived from the general model previously mentioned in the method. The sum of error between the experimental and estimated data (FPE) was calculated for each model. The results are shown below for each of the filtrate and concentrate error.

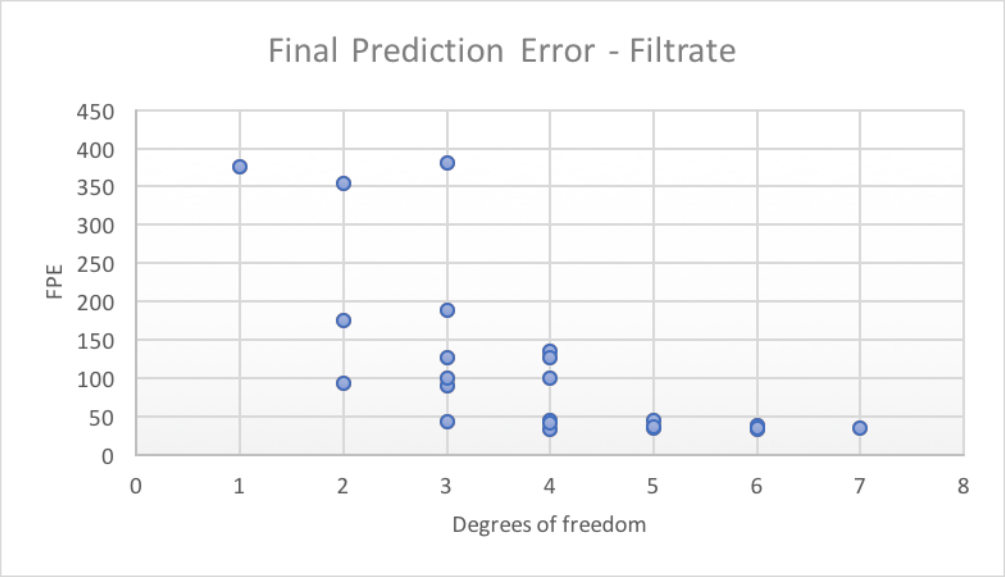

Figure 14 Final prediction error graph for the filtrate, the FPE value for each model is plotted against the degrees of freedom of that model.

The 'knee' in the graph is very clear for the filtrate, where there is a drop in error at 4 degrees of freedom(DOF) and error stabilizes afterwards with increasing DOF. Therefore, out of the six models with 4 DOF, the one with the smallest FPE value was chosen as the best fit: f (Q, m) = a_0+ a_1Q+ a_2m+a_3Q^2. The model does not include power as a variable because it does not have an influence in this specific model, however, the flow rate and the processed starch mass are quadratic and linear respectively.

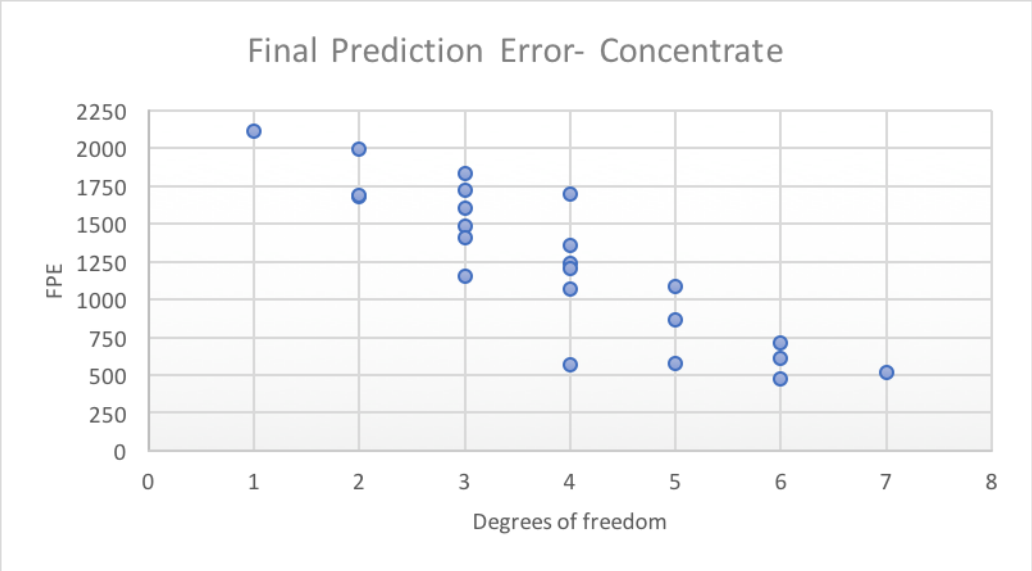

Figure 15 Final prediction error graph for the concentrate, the FPE value for each model is plotted against the degrees of freedom of that model.

As for the concentrate, the initial error drop is at 5 DOF, and where an increase in DOF does not cause an increase in error. Of the three models with 5 DOF, the model with the least error was chosen: f (Q, m, P) = a_0+ a_1Q+ a_2m+a_3P+a_4P^2. In this model, the flow rate and processed starch mass are linear and the power is quadratic.

4.4.2 Response surface

The selection of setting was done using the response surface plots of the models, the chosen filtrate model equation was:

$$f(Q, m) = 4 + 106Q - 23m - 28Q^2 \quad \text{Equation 6}$$

This was the model that fitted the data most accurately from all the other models according to the FPE. The response surface plot of this formula is shown in Figure 16, where mass refers to the processed mass starch and can be converted to time factor for the switching interval using this formula $t = \frac{\mathrm{m}}{\mathrm{Q} * \mathrm{C}}$.

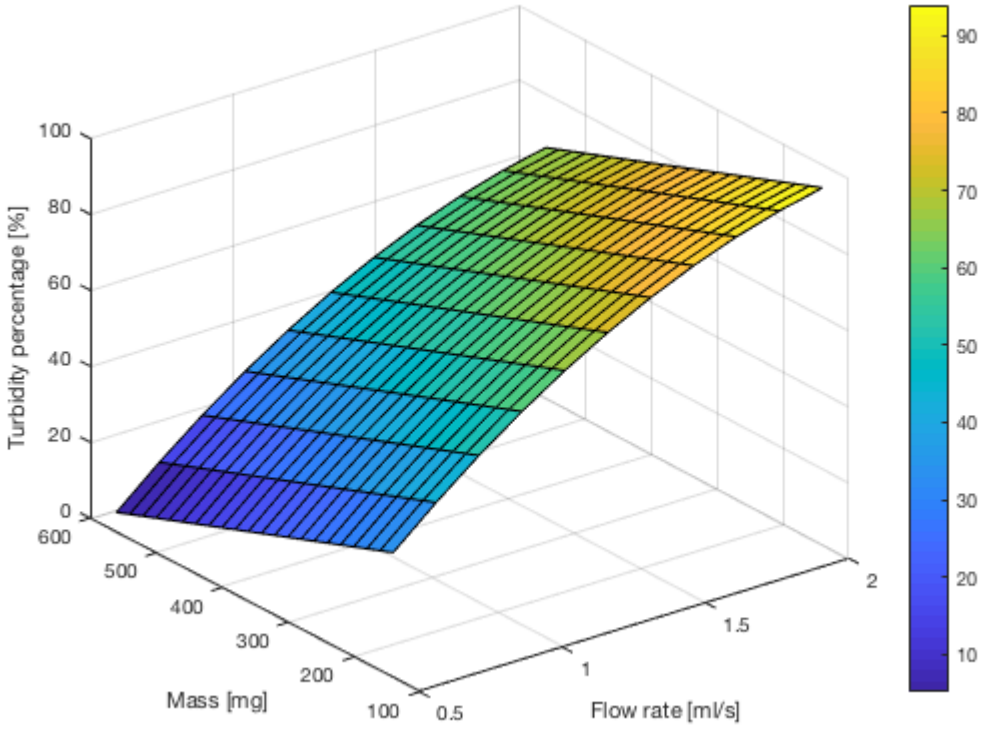

Figure 16 A 3-dimensional response surface plot for the filtrate model: $f(Q, m) = 4 + 106Q - 23m - 28Q^2$.

In this model power does not play a part, therefore, it is not seen in the graph as any level of power from the tested levels would do. The only factor which has a second order influence is flow rate. So, from this plot and using MATLAB, the lowest value for predicted relative turbidity is 5% at a flow rate of 0.5 ml/s and a processed mass of 560 mg with whichever power level as according to this model it should not have much of an influence.

As for the concentrate, the best fitting model was:

$$f(Q, m, P) = -123 - 39Q + 75m + 494P - 192P^2 \quad \text{Equation 7}$$

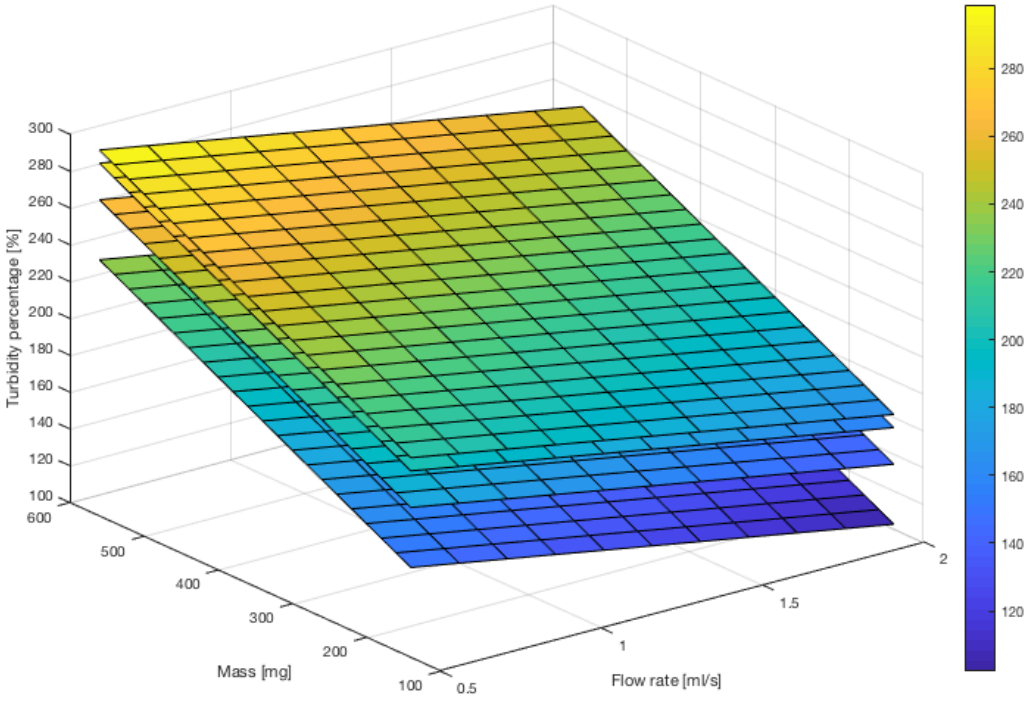

Figure 17 A 3-dimensional response surface plot for the concentrate model: f (Q, m, P) = -123+ -39Q+75m+494P-192P^2. the layers represent the power outputs tested, the lowest layer being the lowest power (10W) and the upper layer being the highest (17.5W).

The response surface plot for the concentrate model is shown in Figure 17, where the highest point in the plot can clearly be seen and the settings at which the highest turbidity is achieved is deduced. The highest predicted relative turbidity for the concentrate is 298.8% at a flow rate of 0.5 ml/s, a power output of 17.5W and a processed starch mass of 560mg (switching interval of 19 minutes 40 seconds).

4.4.3 Validation experiment

The validation experiments, produced the results shown in Figure 18, producing the highest retention efficiency yet of 227% (with a relative concentrate turbidity of 327%). This has even exceeded the model estimation for the relative turbidity at these settings which was 298.7%.
As for the filtration optimal settings, an efficiency of 83% (with a relative turbidity of 16%) was reached. This is different from the 5% relative turbidity predicted by the model. The reason why is likely due this value being extrapolated by the model since there was no run with these settings tested which makes the uncertainty much higher. When looking at the experimental results of all the runs, values of about 16% filtrate relative turbidity are the lowest values recorded and nothing is significantly below that was ever reached. Therefore, a range of 14-16% is concluded to be the absolute minimum value that can be achieved using this system. The 5% predicted by the model however, is not inaccurate with respect to the model results. The model does not register practical limitations so 5% relative turbidity for the filtrate should be doable in theory. Given this model, there should also be a setting where the turbidity would equal 0 (calculated to be at a processed starch mass of 1.87mg and flow rate of 0.5ml/s). But given the data, it is clearly beyond reason to get this value in reality.

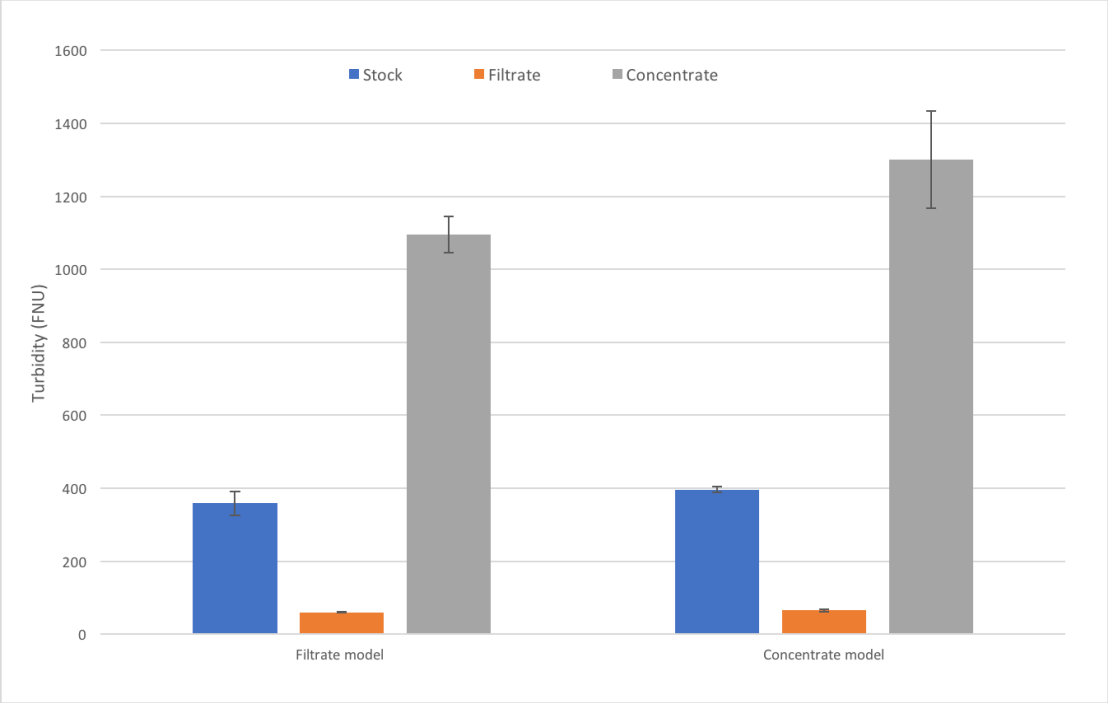

Figure 18 Validation runs for optimal settings for each of the filtrate model, with setting of 0.5 ml/s, 12.5W of power and 560mg of processed starch mass, and the concentrate model with settings of 0.5ml/s, 17.5W and 560mg of processed starch.

Comparing the results from this research are with the results from D. Verschoor's research on this same system, optimal settings found there produced a relative turbidity for the concentrate of 221%, and a relative turbidity for the filtrate of 17% (Verschoor, 2015). So, the retention efficiency reached in this research has exceeded that of the previous, and as for the filtrate, these results further prove that the system does have a lower limit for turbidity.

The difference in relative turbidity between this research and Verschoors' may likely be due to the different range of power and processed starch mass tested. A higher power range could be tested in this research because two amplifiers were linked to increase the power supply as opposed to the one amplifiers used in the previous research and maximum starch mass of 350mg. The reason for this was to provide a new set of data that could be used to find the true optimal settings for this system, within the limitations faced of course, in comparison with settings previously tested.

The best fitting model suggested that the power had no influence, from the tested range, on the filtration efficiency. This was also the case in Verschoor's research, however, from experimentation, it was noted that there was a difference between 10W and any power above it, with 10W producing lower efficiencies compared to 12.5 W, 15W and 17.5W. A significance t-test (95% confidence) proved that there was indeed no significant difference between the three higher power ranged tested, but there was a difference when compared to 10W.

Compared to the model, a less efficient process was expected in reality, as the experiments were not without faults. At low velocities, settling of the starch was dominant to the point where it would settle in some areas of the feed tubes. This is minimal when the filtration run is short, however, with longer runs they affects the results. This can be avoided by using an elevated reservoir as is the plan for scaling up the system.

The starch particles themselves have an effect on the efficiency as they vary in size and therefore, larger sized particles may be captured more by the ultrasound, causing agglomeration and saturation of the nodes to occur faster, so smaller sized particles of <10 *u*m may bypass them and enter the filtrate (Cappon, Stefanova, & Keesman, 2013). This however, has not been checked, and this remark is solely based on previous literature on the topic, therefore, considering the influence of particle size and agglomeration on the separation efficiency is recommended for further research.

In terms of power, the water filtration has proved that an increase in power does not lead to an increase in efficiency, as for the particle recovery process, the model assumed that it did. In reality however, the experimental results show that there is in fact is actually no significant difference between the concentrate results of different power levels used in relation to the retention efficiency according to a significance T test.
At the optimal flow rate of 0.5 ml/s the system can treat 1.8 L/h, so a multiple number of this separator would be needed for scaling up the process. Making an arrangement of parallel separators where two sides are used for each set of transducers, this way energy demand can be decreased.
Still, this system has reached the highest efficiencies so far for both water filtration and starch recovery. There are many potential implementations for this separator such as using it in the potato processing industry to recover starch which is a valuable commodity and to reduce their water consumption. Hydrocyclones are currently used for this process, with a separation efficiency of 90%, however, the drawbacks of this device are not present in acoustic separator and therefore, it has the potential to compete with it. Overall, this innovative device still needs some further investigation to reach its full potential.

4. Conclusion

The main research question to be answered by this research was: How can the operational settings of ultrasonic separation be optimized to achieve the highest filtration efficiency for starch recovery varying power, flow rate and frequency?
The frequency is defined in two ways; filtration efficiency for producing clean water and retention efficiency for recovering starch particles. From the results, it can be concluded that operational settings of 0.5 ml/s, power of 12.5 and a processed starch mass of 560mg are the optimal settings for filtration. The optimal settings for the retention efficiency is concluded to be 0.5ml/s, 17.5W and a processed starch mass of 560mg. These settings resulted in a filtration efficiency of 83% and a retention efficiency of 228%. These are the highest efficiencies reached with this device yet.
As for changing the frequency to form a quasi-standing wave, the experiment was not feasible using this separation chamber as it always creates a standing wave and no travelling waves. Overall, these settings have still resulted in the highest efficiencies so reached so far with this separation system.

5. Recommendations

Going forward with this research, there are a few suggestions for further investigation and small adjustments to the method that are recommended to gain optimal results and understand the potential of the system better. For one, checking the size of particles present in each of the filtrate and concentrate would indicate what particle size is captured more frequently. Explore the relationship between particle size of starch and turbidity further for a better understanding.
As for the backwash, to make it more efficient, switching the amplifier off for a period of time, allowing the particles to settle before backwashing would be a good idea. In these experiments a 10 second interval between the amplifier switching off and the backwash pump switching on is given, so doubling that time as a minimum might allow for an even higher retention efficiency. This combined with a higher backwash flow rate may be possible. Also, using a more precise amplifier and a turbidity meter with higher reading range would give more reliable results.
Testing the system with different particles for example homogenously sized particles like polystyrene spheres would give us a better idea of the potential of this system. So far, the system has only been tested with starch particles, however, it may have the potential to capture other insoluble particles so further experiments with other particles is recommended. This would also depend on the particle size that the device can capture, therefore, that is important to experiment with.

Lastly, using a flow rate of 2 ml/s was not practical because the difference between the filtrate and turbidity for those runs was too small (<100FNU) to be considered efficient compared to the other flow rates, therefore, it is not recommended to use this flow rate again.

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7. Appendices

8.1: procedures

8.1.1 Turbidity-concentration experiment

Materials:

- Insoluble potatoes Starch
- Measuring scale (up to 0.0001g)
- Turbidity meter
- 6 100ml volumetric flasks
- 50 ml pipet
- 10 ml pipet
- 5 ml pipet
- Demineralized water

Method:

A 100 ml dilution series is made, firstly by making a stock solution of 1000ppm, which is then diluted to make a series, as shown in the table.

Table 1 Dilution series concentrations and volumes summary

Total Volume (ml)	Concentration (g/l)	Addition of	Demineralized water added (ml)
100	1	0.1 g of starch	100
100	0.5	50 ml of 1 g/l suspension	50
100	0.25	50 ml of 0.5 g/l suspension	50
100	0.125	50 ml of 0.25 g/l suspension	50
100	0.1	10 ml of 1g/l suspension	90
100	0.05	5 ml of 1g/l suspension	95

1. Weigh out 0.1g of starch in the measuring scale and deposit it into the flask. Then add demineralized water to the flask, flushing the starch to the bottom, up to the 100ml mark. Cover with a lid and mix well until a suspension is formed.
2. Withdraw 50 ml of the suspension from step 1, using the pipet, and deposit it into a second 100 ml flask. Add water up to the 100ml mark. Cover and mix until a homogeneous suspension is formed.
3. Using a clean pipet, withdraw 50 ml of the suspension formed in step 2 into a new flask. And repeat.
4. Again, withdraw 50 ml of the suspension from step 3 and place into a new flask. Add demineralized water up to the 100ml mark. Place a lid and mix.
5. Withdraw 10 ml of the 1g/l suspension and place it into a clean flask. Make up the flask to the mark with demineralized water. Cover and mix well.
6. Withdraw 5 ml of the 1g/l suspension and place it into a clean flask. Make up the flask to the mark with demineralized water. Cover and mix well.
7. Take three 10 ml samples from each of the six flasks, measuring the turbidity for each sample thrice. Record results.
8. Clean all lab equipment and return to place.
9. Determine the relationship between the concentration and turbidity making a curve in MS Excel.

8.1.2 Saturation progress experiment

Materials:

- Ultra Sound filtration unit
- Starch
- Demineralized water
- Measuring scale
- Magnetic plate and stir bar
- Turbidity meter
- 3ml Pipets
- 2 1L beakers

Method:

1. To make the stock solution weigh out 1 g of starch into a 1 L beaker and add demineralized water to make up 1g/l starch solution. Place on the magnetic bar to stir the suspension.
2. Place the feed tube into the stock solution into the beaker.
3. Place the filtrate tube into an empty beaker.
4. Make sure the pump is calibrated before use, then set the pump to 'continuous' mode and select the flow rate, flow direction and tube size.
5. Have the backwash pump turned off.
6. Take two samples from the stock solution and measure their turbidity, twice each.
7. Start the filtration run
8. Take 30 ml samples every 30 seconds during the filtration process
9. Turn off the pump once the stock solution beaker is empty
10. Take two more samples from the filtrate and measure their turbidity, twice. Make sure to mix the samples before measuring.
11. Reset the filtration unit and repeat steps 1-10 for a different flow rate.

8.1.3 Optimization and validation experiment

Materials

- Ultrasound filtration unit
- volumetric flask (1000 ml)
- Starch powder
- Demineralized water
- Measuring scale
- Magnetic plate and stir bar
- Turbidity meter
- 3ml Pipets
- 2 beakers (1L)
- 3 beakers (250 ml)
- 3 beakers (500 ml)

Method:

1. Make a starch stock solution of 1g/l concentration using the conical flask and place it in a 1 L beaker with a magnetic stir bar to keep it in suspension.
2. Measure the turbidity of the stock solution using the turbidity meter. Take three 10 ml samples and measure each thrice. Make sure that the samples are mixed well before measuring the turbidity.
3. Prepare three 250ml beakers for the concentrate and three 500 ml beakers for the filtrate making sure they are clean and labelled.
4. Before using the pumps, make sure that they are calibrated and set both pumps to timed mode, with three batches, setting the run and stop time for each pump according to the processed starch mass required.

5. Attach the hose from the stock pump to the stock solution, making sure it stays submerged throughout the experiment, and attach the filtrate hose to the filtrate beaker. The hose from the second backwash pump into the concentrate beaker.
6. Prime the tubes making sure to get rid of any air bubbles, and fill the chamber and damper hose with the stock solution using the 'continuous' mode in the feed pump, once they are filled make sure to close the top of the damper hose. The level of water in it will decrease during the experiment.
7. Select the timer option on the amplifiers and immediately switch on timer, at the time mark switch on the stock pump, on and off times are shown in the table below.

Table 2 On and Off times for the stock pump, backwash pump, and the amplifiers.

Device	**Time duration (mm:ss)**	
	ON	OFF
Pump 1 (stock)	4:40	01:00
Amplifier (both of them)	05:00	00:40
Pump 2 (backwash)	00:20	5:20

8. Let the unit run until the backwash pump stops, then switch the filtrate beaker to new clean beaker and afterwards change the concentrate beaker.
9. Repeat for the next two cycles till you end up with 3 filled filtrate beakers and 3 concentrate beakers.
10. Turn the amplifiers off and clean the entire system at high speed with demi water, making sure that any starch left in the chamber is removed.
11. Measure the turbidity of the six sample, measuring each sample thrice using the turbidity meter, resulting in nine measurements total with the stock readings.
12. Repeat these steps for all planned runs (changing the power, flow rate, and if required time)

8.2: Experimental data

8.2.1 Saturation progress

Table 3 Raw data of the saturation progress experimental run at a flow rate of 0.5 ml/s. Turbidity of the filtrate is recorded at 2 minute intervals for 34 minutes, first row is the turbidity of the stock solution used in the run.

0.5 mL/s					Standard deviation	standard error
Time (hh:mm:ss)	Sample	Turbidity				
		measurement 1	measurement 2	Average		
stock	1	373	368	386	18,018509	9,009254501
	2	402	401			
00:00:00	1	25,54	25,5	27,2175	2,28365752	1,14182876
	2	30,35	27,48			
00:02:00	1	62	59	60,25	1,258305739	0,62915287
	2	60	60			
00:04:00	1	69	67	68,25	0,957427108	0,478713554
	2	69	68			
00:06:00	1	64	62	66,25	4,031128874	2,015564437
	2	71	68			
00:08:00	1	97	87	91,25	6,751543034	3,375771517
	2	84	97			

	1	63	67			
00:10:00	2	67	73	67,5	4,123105626	2,061552813
	1	59	66			
00:12:00	2	64	66	63,75	3,304037934	1,652018967
	1	61	62			
00:14:00	2	59	58	60	1,825741858	0,912870929
	1	92	91			
00:16:00	2	87	84	88,5	3,696845502	1,848422751
	1	58	58			
00:18:00	2	63	60	59,75	2,362907813	1,181453907
	1	69	63			
00:20:00	2	62	66	65	3,16227766	1,58113883
	1	68	64			
00:22:00	2	65	66	65,75	1,707825128	0,853912564
	1	72	71			
00:24:00	2	83	71	74,25	5,852349955	2,926174978
	1	64	58			
00:26:00	2	62	52	52	5,291502622	2,645751311
	1	67	60			
00:28:00	2	64	53	61	6,055300708	3,027650354
	1	69	60			
00:30:00	2	65	68	65,5	4,041451884	2,020725942
	1	62	60			
00:32:00	2	62	66	62,5	2,516611478	1,258305739
	1	76	78			
00:34:00	2	68	66	72	5,887840578	2,943920289

Table 4 Raw data of the saturation progress experimental run at a flow rate of 2 ml/s. Turbidity of the filtrate is recorded at 1 minute intervals for 17 minutes, first row is the turbidity of the stock solution used in the run.

2 mL/s- (120 mL/min - 2L)						
Time (hh:mm:ss)	Sample	Turbidity			Standard Deviation	Standard Error
		measurement 1	measurement 2	Average		
	1	271	369			
stock	2	428	398	366,5	68,0710413	34,0355207
	1	203	210			
00:00:00	2	164	198	193,75	20,4348558	10,2174279
	1	194	201			
00:01:00	2	183	199	194,25	8,05708798	4,02854399
	1	329	266			
00:02:00	2	232	267	273,5	40,4186426	20,2093213
	1	167	154			
00:03:00	2	180	178	169,75	11,9547759	5,97738795

	1	200	183			
00:04:00	2	211	178	193	15,2534149	7,62670746
	1	202	192			
00:05:00	2	219	218	207,75	13,0735101	6,53675506
	1	242	274			
00:06:00	2	331	312	289,75	39,6852198	19,8426099
	1	350	332			
00:07:00	2	293	320	323,75	23,9217474	11,9608737
	1	325	309			
00:08:00	2	312	334	320	11,6332856	5,81664279
	1	352	324			
00:09:00	2	368	306	344,5	27,7788889	13,8894444
	1	285	315			
00:10:00	2	278	337	296	27,3663419	13,6831709
	1	326	292			
00:11:00	2	321	264	319	28,7213625	14,3606813
	1	356	384			
00:12:00	2	365	385	342,25	14,3410832	7,17054159
	1	334	397			
00:13:00	2	329	355	361,25	30,9556134	15,4778067
	1	336	323			
00:14:00	2	291	313	326,25	18,9978069	9,49890345
	1	351	337			
00:15:00	2	342	383	335,75	20,6619618	10,3309809
	1	418	370			
00:16:00	2	403	376	393,5	22,6329406	11,3164703
	1	374	301			
00:17:00	2	356	388	351,75	38,1521078	19,0760539

8.2.2 Optimization

Table 5 Data collected from the optimization run for a flow rate of 2ml/s (560 mg processed starch) with no ultrasound. The turbidity readings of the stock, filtrate and concentrate are displayed.

2 ml/s - Blank (No US)						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	344	401	362			
2	409	545	431			
3	387	455	395	414,33	59,18	19,73
Filtrate						

Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	368	366	354			
2	293	372	411			
3	348	392	355	362,11	32,64	10,88
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	508	408	390			
2	413	407	451			
3	421	408	424	425,56	35,09	11,70

Table 6. Data collected from the optimization run for a flow rate of 2ml/s (560 mg processed starch) with power input of 17.5 W. The turbidity readings of the stock, filtrate and concentrate are displayed

2 mL/s - 17.5 W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	392	444	394			
2	380	438	402			
3	399	401	388	404,22	21,99	7,33
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	299	292	294			
2	299	347	316			
3	312	324	307	310	17,45	5,82
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	1000	782	749			
2	751	762	766			
3	965	959	913	849,67	106,65	35,55

Table 7 Data collected from the optimization run for a flow rate of 2ml/s (560 mg processed starch) with power input of 15W. The turbidity readings of the stock, filtrate and concentrate are displayed.

2mL/s - 15W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	367	381	359			
2	374	363	353			
3	432	371	417	379,67	26,97	8,99

Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	360	333	360			
2	342	353	340			
3	290	290	289	328,56	30,50	10,17
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	831	789	723			
2	646	604	622			
3	767	777	744	722,56	80,31	26,77

Table 8 Data collected from the optimization run for a flow rate of 2ml/s (560 mg processed starch) with power input of 12.5WW. The turbidity readings of the stock, filtrate and concentrate are displayed.

2mL/s - 12.5W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	372	386	375			
2	400	394	378			
3	413	386	387	387,89	12,94	4,31
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	340	323	314			
2	343	332	352			
3	280	334	324	326,89	21,00	7,00
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	715	651	690			
2	690	752	713			
3	787	754	752	722,67	42,23	14,08

Table 9 Data collected from the optimization run for a flow rate of 2ml/s (560 mg processed starch) with power input of 10W. The turbidity readings of the stock, filtrate and concentrate are displayed.

2mL/s - 10W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	382	372	378			
2	383	388	381	375,44	17,39	5,80

3	377	331	387			
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	302	286	303			
2	310	333	319			
3	350	350	351	322,667	24,331	8,110
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	672	697	620			
2	602	568	565			
3	827	797	794	682,44	102,50	34,17

Table 10 Data collected from the optimization run for a flow rate of 1.5ml/s (420 mg processed starch) with no ultrasound. The turbidity readings of the stock, filtrate and concentrate are displayed.

1.5 ml/s - Blank (No US)						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	391	353	390	380,78	12,76	4,25

2	372	387	392			
3	373	387	382			
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	334	358	340			
2	364	347	369			
3	367	362	346	354,11	12,66	4,22
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	366	375	379			
2	355	337	349			
3	365	337	338	355,67	16,45	5,48

Table 11 Data collected from the optimization run for a flow rate of 1.5ml/s (420 mg processed starch) with power input of 17.5 W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1.5 mL/s - 17.5 W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	445	359	409			
2	573	440	363			
3	254	398	394	403,89	85,08	28,36
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	250	275	234			
2	325	280	280			
3	239	251	273	267,44	27,88	9,29
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	826	883	799			
2	899	893	1000			
3	1000	1000	1000	922,22	80,29	26,76

Table 12 Data collected from the optimization run for a flow rate of 1.5ml/s (420 mg processed starch) with power input of 15 W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1.5mL/s - 15W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error

1	375	379	380			
2	388	375	394			
3	379	392	369	381,22	8,39	2,80
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	205	242	240			
2	309	291	281			
3	263	265	249	260,56	31,03	10,34
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	764	746	826			
2	761	774	775			
3	780	795	792	779,22	23,22	7,74

Table 13 Data collected from the optimization run for a flow rate of 1.5ml/s (420 mg processed starch) with power input of 12.5 W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1.5mL/s - 12.5W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	382	398	385			
2	368	382	383			
3	381	374	380	381,44	8,13	2,71
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	236	271	287			
2	304	281	283			
3	276	302	294	281,56	20,39	6,80
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	989	1000	1000			
2	850	853	1000			
3	918	924	912	938,44	61,62	20,54

Table 14 Data collected from the optimization run for a flow rate of 1.5ml/s (420 mg processed starch) with power input of 10 W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1.5 mL/s - 10W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard	Standard

					Deviation	error
1	390	224	389			
2	387	387	374			
3	373	364	382	363,33	52,99	17,66
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	213	217	233			
2	278	270	287			
3	275	255	281	256,56	28,56	9,52
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	1000	1000	1000			
2	837	827	836			
3	904	898	893	910,56	72,85	24,28

Table 15 Data collected from the optimization run for a flow rate of 1ml/s (280 mg processed starch) with no ultrasound . The turbidity readings of the stock, filtrate and concentrate are displayed.

1mL/s - Blank (No US)						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	360	384	377			
2	364	408	372			
3	360	388	404	379,67	17,92	5,97
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	347	331	327			
2	321	327	323			
3	412	343	333	340,44	28,19	9,40
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	346	344	327			
2	307	316	364			
3	311	322	305	326,89	20,33	6,78

Table 16 Data collected from the optimization run for a flow rate of 1ml/s (280 mg processed starch) with power input of 17.5 W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1mL/s - 17.5 W

Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	374	368	395			
2	402	415	413			
3	376	351	376	385,56	21,79	7,26
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	175	200	88			
2	199	179	192			
3	176	167	193	174,33	34,39	11,46
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	821	757	759			
2	683	597	690			
3	1000	1000	1000	811,89	153,99	51,33

Table 17 Data collected from the optimization run for a flow rate of 1ml/s (280 mg processed starch) with power input of 15 W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1mL/s - 15W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	373	305	406			
2	377	493	384			
3	385	392	418	392,56	49,17	16,39
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	182	183	200			
2	167	182	94			
3	176	166	162	168	30,046	10,015
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	712	734	655			
2	786	649	628			
3	839	1000	730	748,11	116,13	38,71

Table 18 Data collected from the optimization run for a flow rate of 1ml/s (280 mg processed starch) with power input of 12.5W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1mL/s - 12.5W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	402	391	350			
2	378	428	366			
3	376	381	325	377,44	29,56	9,85
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	156	171	161			
2	183	190	171			
3	163	160	165	168,89	11,24	3,75
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	779	932	901			
2	663	652	631			
3	880	785	690	768,11	115,58	38,53

Table 19 Data collected from the optimization run for a flow rate of 1ml/s (280 mg processed starch) with power input of 10 W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1mL/s - 10 W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	357	354	392			
2	415	362	334			
3	384	380	348	369,56	25,18	8,39
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	188	195	185			
2	159	169	168			
3	172	197	193	180,67	13,87	4,62
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	797	810	818			
2	738	794	790			
3	928	888	859	824,67	57,60	19,20

Table 20 Data collected from the optimization run for a flow rate of 0.5 ml/s (140 mg processed starch) with no ultrasound. The turbidity readings of the stock, filtrate and concentrate are displayed.

0.5 ml/s - Blank (No US)						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	399	379	363			
2	374	361	387			
3	397	392	354	378,44	16,52	5,51
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	194	202	182			
2	183	180	97			
3	186	202	197	180,33	32,38	10,79
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	252	240	248			
2	245	228	250			
3	277	289	318	260,78	28,38	9,46

Table 21 Data collected from the optimization run for a flow rate of 0.5 ml/s (140 mg processed starch) with a power input of 17.5W. The turbidity readings of the stock, filtrate and concentrate are displayed.

0.5 mL/s - 17.5 W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	396	389	420			
2	433	372	523			
3	314	443	397	409,67	56,96	18,99
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	51	52	52			
2	88	86	85			
3	61	63	63	66,78	15,41	5,14
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	384	382	368			
2	387	319	391	377,78	36,11	12,04

3	427	416	326			

Table 22 Data collected from the optimization run for a flow rate of 0.5 ml/s (140 mg processed starch) with a power input of 15W. The turbidity readings of the stock, filtrate and concentrate are displayed.

0.5mL/s - 15W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	362	373	351			
2	372	392	371			
3	421	410	383	381,67	22,55	7,52
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	72	64	70			
2	64	61	63			
3	62	62	61	64,33	3,97	1,32
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	323	353	344			
2	320	322	342			
3	392	391	333	346,67	27,74	9,25

Table 23 Data collected from the optimization run for a flow rate of 0.5 ml/s (140 mg processed starch) with a power input of 12.5W. The turbidity readings of the stock, filtrate and concentrate are displayed.

0.5mL/s - 12.5W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	460	383	468			
2	377	396	398			
3	371	380	379	401,33	36,62	12,21
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	59	56	61			
2	66	64	70			
3	44,77	47	51	57,64	8,67	2,89
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	454	477	466	379,44	65,51	21,84

2	339	327	320			
3	337	348	347			

Table 24 Data collected from the optimization run for a flow rate of 0.5 ml/s (140 mg processed starch) with a power input of 10W. The turbidity readings of the stock, filtrate and concentrate are displayed.

0.5mL/s - 10W						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	378	404	371			
2	373	366	353			
3	334	359	377	368,33	19,29	6,43
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	98	102	99			
2	75	69	71			
3	113	122	131	97,78	22,38	7,46
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	357	335	342			
2	300	337	288			
3	317	304	309	321,00	22,83	7,61

Table 25 Data collected from the optimization run for a flow rate of 0.5 ml/s (420 mg processed starch) with a power input of 12.5W. The turbidity readings of the stock, filtrate and concentrate are displayed.

0.5mL/s - 12.5W - 0.42g						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	300	322	391			
2	355	347	347			
3	368	279	368	341,89	35,54	11,85
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	83	82	65			
2	77	77	74			
3	52	56	54	68,89	12,33	4,11
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error

1	770	758	776			
2	1000	1000	1000			
3	1000	1000	1000	922,67	116,09	38,70

Table 26 Data collected from the optimization run for a flow rate of 1 ml/s (420 mg processed starch) with a power input of 12.5W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1mL/s - 12.5W - 0.42g						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	552	530	546			
2	566	473	464			
3	553	616	552	539,11	46,51	15,50
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	186	190	189			
2	193	201	180			
3	229	223	232	202,56	20,01	6,67
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	1000	1000	1000			
2	1000	1000	1000			
3	1000	1000	1000	1000,00	0,00	0,00

Table 27 Data collected from the optimization run for a flow rate of 1.5 ml/s (420 mg processed starch) with a power input of 12.5W. The turbidity readings of the stock, filtrate and concentrate are displayed.

1.5mL/s - 12.5W - 0.42g						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	382	398	385			
2	368	382	383			
3	381	374	380	381,44	8,13	2,71
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	236	271	287			
2	304	281	283			
3	276	302	294	281,56	20,39	6,80

Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	989	1000	1000			
2	850	853	1000			
3	918	924	912	938,44	61,62	20,54

Table 28 Data collected from the optimization run for a flow rate of 2 ml/s (420 mg processed starch) with a power input of 12.5W. The turbidity readings of the stock, filtrate and concentrate are displayed.

2mL/s - 12.5W - 0.42g						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	325	236	287			
2	316	286	290			
3	288	291	278	288,56	24,93	8,31
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	208	191	206			
2	255	237	280			
3	277	261	235	238,89	32,09	10,70
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	657	699	679			
2	555	522	518			
3	494	574	574	585,78	74,91	24,97

8.2.3 Validation experiments

0.5mL/s - 12.5 W- 560mg (filtrate model)						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	376	411	388			
2	394	385	397			
3	98	392	390	359,00	98,33	32,78
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error

1	60	63	62			
2	58	57	55			
3	61	65	62	60,33	3,16	1,05
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	1000	1000	1000			
2	1000	1000	1000			
3	1000	1000	1000	1000,00	0,00	0,00

0.5mL/s - 17.5 W- 560mg (concentrate model)						
Stock						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	437	396	390			
2	396	361	424			
3	406	370	390	396,67	23,81	7,94
Filtrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	71	72	66			
2	49,42	55	51			
3	78	76	71	65,49	10,89	3,63
Concentrate						
Sample	measurement1	measurement2	measurement3	Average	Standard Deviation	Standard error
1	800	774	794			
2	1472	1406	1398			
3	1684	1648	1722	1299,78	399,97	133,32

8.3: Data analysis

8.3.1 Final prediction error

Table 29 The FPE value for each model of either the filtrate or concentrate. Shaded formulas are the chosen models for each of the concentrate and filtrate

Filtrate		
Model	**no. Parameters**	**FPE value**
$y=a_0+a_1Q+a_2m+a_3P+a_4Q2+a_5m^2+a_6P^2$	7	35,4813
$y=a_0+a_1Q+a_2m+a_3P+a_4Q^2+a_5m^2$	6	32,9147
$y=a_0+a_1Q+a_2m+a_3P+a_4Q^2+a_5P^2$	6	37,4152
$y=a0+a_1Q+a_2m+a_3P+a_4m2+a_5P^2$	6	34,1945

$y=a_0+a_1Q+a_2m+a_3P+a_4Q^2$	5	34,2203
$y=a_0+a_1Q+a_2m+a_3P+a_4m^2$	5	44,6250
$y=a_0+a_1Q+a_2m+a_3P+a_4P^2$	5	37,2671
$y=a_0+a_1Q+a_2m+a_3P$	4	45,1103
$y=a_0+a_1Q+a_2m+a_3Q^2$	4	32,7548
$y=a_0+a_1Q+a_2m+a_3m^2$	4	41,6259
$y=a_0+a_1Q+a_2P+a_3P^2$	4	99,9272
$y=a_0+a_1m+a_2P+a_3P^2$	4	134,8131
$y=a_0+a_1m+a_2P+a_3m^2$	4	127,3945
$y=a_0+a_1Q+a_2m$	3	42,6303
$y=a_0+a_1Q+a_2Q^2$	3	89,3652
$y=a_0+a_1Q+a_2P$	3	100,3874
$y=a_0+a_1P+a_2P^2$	3	188,9953
$y=a_0+a_1m+a_2m^2$	3	381,1475
$y=a_0+a_1m+a_2P$	3	126,5220
$y=a_0+a_1Q$	2	93,9498
$y=a_0+a_1m$	2	354,3169
$y=a_0+a_1P$	2	175,2666
$y=a_0$	1	376,0703
Concentrate		
Model	**no. Parameters**	**FPE value**
$y=a_0+a_1Q+a_2m+a_3P+a_4Q2+a_5m^2+a_6P^2$	7	516,6803
$y=a_0+a_1Q+a_2m+a_3P+a_4Q^2+a_5m^2$	6	710,6439
$y=a_0+a_1Q+a_2m+a_3P+a_4Q^2+a_5P^2$	6	610,2986
$y=a0+a_1Q+a_2m+a_3P+a_4m2+a_5P^2$	6	474,0592
$y=a_0+a_1Q+a_2m+a_3P+a_4Q^2$	5	863,7209
$y=a_0+a_1Q+a_2m+a_3P+a_4m^2$	5	1084,8000
$y=a_0+a_1Q+a_2m+a_3P+a_4P^2$	5	573,9822
$y=a_0+a_1Q+a_2m+a_3P$	4	1698,0000
$y=a_0+a_1Q+a_2m+a_3Q^2$	4	1236,9000
$y=a_0+a_1Q+a_2m+a_3m^2$	4	1358,3000
$y=a_0+a_1Q+a_2P+a_3P^2$	4	1201,6000
$y=a_0+a_1m+a_2P+a_3P^2$	4	566,8873
$y=a_0+a_1m+a_2P+a_3m^2$	4	1064,6000
$y=a_0+a_1Q+a_2m$	3	1480,2000
$y=a_0+a_1Q+a_2Q^2$	3	1835,3000
$y=a_0+a_1Q+a_2P$	3	1719,6000
$y=a_0+a_1P+a_2P^2$	3	1149,8000
$y=a_0+a_1m+a_2m^2$	3	1600,3000
$y=a_0+a_1m+a_2P$	3	1408,7000
$y=a_0+a_1Q$	2	1990,9000
$y=a_0+a_1m$	2	1675,5000

$y=a_0+a_1P$	2	1684,1000
$y=a_0$	1	2114,0000

Table 30 ai values for each of the selected optimal filtrate and concentrate model

a_i	Filtrate model	Concentrate model
a_0	4.3307	-123.0809
a_1	106.0379	-38.9846
a_2	-23.2360	75.1249
a_3	-27.6221	494.2155
a_4	-	-192.4662

8.3.2 MATLAB scripts

8.3.2.1 FPE

```
% Theorem for first order (linear) regression with two variables
% y = a0 + a1*v1 + a2*v2 = X*a
% with X = [1 v1 v2]; and a = [a0 ; a1 ; a2]
% (Note the difference in semicolons use
%
% Theorem for second order multiple regression with two variables
% y = a0 + a1*v1 + a2*v2 + a3*v1*v2 + a4*v1^2 + a5*v2^2 = X*a
% with coefficients ai and variables vi
%
% Thus there are six unknowns ai, which need to be estimated
% a = [a0; a1; ... ; a5];
% These ai describe the response y to the variables in X
% X = [1 v1 v2 v1*v2 v1^2 v2^2];
%
% Example variables
v1 = [0.5 1 1.5 2]; % flow rate in ml/s
v2 = [60 205 350]; % processed starch mass in mg
v3 = [10 12.5 12]; % power in W
%v1 = [33 66 100]; % flow rate in ml/s
%v2 = [20 60 100]; % processed starch mass in mg
%v3 = [25 75 100]; % power in W

% Build X (form in v [1 1 1; 1 1 2; 1 1 3; 1 2 1; 1 2 2; ... 3 3 2; 3 3 3];
vars = [];

X =[];
for n = 1:length(vars)
    % y = a0 + a1*v1 + a2*v2 + a3*v3
    xi = [1 vars(n,1) 0 vars(n,3)]; % vars(n,1)^2 v2(m)^2 v3(p)^2];
    X = [X; xi];
end

% Build Y

y = [];
% Experimental runs provide a range of y values

ai = X\y; % least squares estimate with multiple regression in Matlab
y_est = X*ai; % estimated y on the basis of the fit with ai

N = length(y);
d = length(xi);
loss = sum(0.5*(y_est-y).^2)/N
FPE = (1+d/N)/(1-d/N)*loss

var_y = sum((y-y_est).^2)/(length(y)-length(xi));
```

```
cov_ai = var_y*inv(X'*X);
sd_ai = sqrt(diag(cov_ai));
A = [ai-1.96.*sd_ai ai ai+1.96.*sd_ai]

plot(y,y_est,'r*'); hold on % plot the result y and y_est
xr = [0:100]; yr= xr; plot(xr,yr,'g'); hold off
axis('equal')
axis([0 105 0 105])

figure(2); plot(X(:,2),y,'r^',X(:,2),y_est,'*'); xlabel('Flow rate [%]'); ylabel('Turbidity [NTU]');
legend('Experiment','Estimated');
figure(3); plot(X(:,3),y,'r^',X(:,3),y_est,'*'); xlabel('Processed mass [%]'); ylabel('Turbidity [NTU]');
legend('Experiment','Estimated');
figure(4); plot(X(:,4),y,'r^',X(:,4),y_est,'*'); xlabel('Power [%]'); ylabel('Turbidity [NTU]');
legend('Experiment','Estimated');
% X_est = [];
% for n = 1:10:100;
%     for m = 1:10:100
%         for p = 1:10:100
%             x_est = [1 n m p n*m n*p m*p n^2 m^2 p^2];
%             X_est = [X_est; x_est];
%         end
%     end
% end
% Y_est = X_est*ai;
% surf(X_est(1:10,2),X_est(1:10:100,3),reshape(Y_est(1:100:end),10,10))
% Compare with results from statistics toolbox for instance fitlm function
% in linear regression toolbox
```

8.3.2.2 3-D response model

```
v1 = [0.5:0.15:2]./1.25; % flow rate in ml/s
v2 = [140:20:560]./350; % processed starch mass in mg
v3 = [10 12.5 15 17.5]./13.75; % power in W

ai = [-123.0809
-38.9846
75.1249
494.2155
-192.4662];

% Build X (form in v [1 1 1; 1 1 2; 1 1 3; 1 2 1; 1 2 2; ... 3 3 2; 3 3 3];

for p = 1:length(v3)
   X = [];
   for n = 1:length(v1)
      for m = 1:length(v2)
         % xi = [1 v1(n) v2(m) v3(p)/v1(n) v1(n)^2 v2(m)^2 (v3(p)/v1(n))^2];
         % your response model
         xi = [1 v1(n) v2(m) v3(p) v3(p)^2];
         X = [X; xi];
      end
   end
   y_est = X*ai; % estimated y on the basis of the fit with ai
   y_est = reshape(y_est,length(v2),length(v1));
   surf(v1.*1.25,v2.*350,y_est); hold on
end

xlabel('Flow rate [ml/s]')
ylabel('Mass [mg]')
zlabel('Turbidity percentage [%]')
% setfont(16)

 % Experimental runs provide a range of y values
```