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Scaling of the modular synthesis of a cellulose derivative

Metropolia U of Applied Science

Bachelor of Engineering

Chemical engineering

Bachelor's thesis

15.5.2018

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Tutkinto	Insinööri (AMK)
Tutkinto-ohjelma	Bio- ja kemiantekniikka
Ammatillinen pääaine	Kemiantekniikka
Valvojat	Lehtori Timo Seuranen Professori Ali Harlin
<p>Työn tarkoitus oli tutkia hajautettua tuotantoa kontteihin asennettavilla prosessikokonaisuuksilla. Tutkimuksen kohteeksi valittiin selluloosan johdannaisen syntetisointi. Erityisesti prosessin skaalausta tuli tarkastella.</p> <p>Työ alkoi synteisiprosessiin ja prosessilaitteisiin perehtymisellä. Jotkin laitteet olivat ennalta määritettyjä tilaajan puolesta, esimerkiksi jauhin. Muut laitteet määritettiin työn aikana.</p> <p>Tuotantomäärä määritettiin synteisireaktorin kapasiteetin perusteella taselaskentaa varten. Loppu tieto taselaskentaa varten haettiin kirjallisuudesta tai oletettiin. Taselaskennan avulla mitoitettiin prosessilaitteet. Ainoastaan pääprosessilaitteet mitoitettiin tarkasti. Loput määritettiin kustannuslaskennassa.</p> <p>Myös prosessiin liittyvät lait ja määräykset käytiin läpi. Mielenkiinnon kohteena olivat rajoitukset, jotka liittyivät käytettyihin kemikaaleihin tai tuotantomäärän kasvattamiseen. Tarkoitus oli löytää mahdollinen raja-arvo, jonka ylittäminen tuottaisi huomattavasti enemmän töitä lakien ja määräyksien parissa.</p> <p>Lopuksi prosessi skaalattiin. Tarkat laskut suoritettiin yhdelle kapasiteetille havainnollistamisen vuoksi. Kapasiteettien ja kustannusten avulla piirrettiin käyrä kuvaamaan yhteisvaikutusta.</p>	
Avainsanat	selluloosa, johdannainen, kontti, modulaarinen, synteisi

Author Title	Mikko Näsi Scaling of the modular synthesis of a cellulose derivative
Number of Pages Date	40 pages + 5 appendices 15 April 2018
Degree	Bachelor of Engineering
Degree Programme	Bio- and chemical engineering
Professional Major	Chemical engineering
Instructors	Timo Seuranen, Senior Lecturer Ali Harlin, Research Professor
<p>The target of this Bachelor's thesis was to study distributed manufacturing by means of a flexible modular containerised process. A cellulose derivative synthesis well-known in literature was selected as a case for the project in order to examine the scalability of the containerised modular plant.</p> <p>This thesis started by gathering information about the synthesis process and by determining what unit processes it would include. Some process units, such as the mill, were pre-determined. Other equipment was determined and dimensioned during this thesis.</p> <p>Output for the balance calculations was selected based on the synthesis reactor's working capacity. The next task was to find the parameters relating to the balance calculations. Having chosen the machinery and using the information from the balance calculations, the unit processes could now be dimensioned. Only the primary unit processes were dimensioned accurately. The rest would be determined later during cost estimation.</p> <p>Regulations and legislation relating to the process were studied as well. Of interest were restrictions relating to the increase of output and the amount of chemicals used and produced. It was necessary to find out whether there existed a certain limit of produced/used chemicals that would increase the regulatory workload significantly.</p> <p>Finally, the scaling was done to the different capacities and the related costs. The specific calculations were done with a single capacity for demonstration. A graph with capacities and expenses was obtained to model the interaction.</p>	
Keywords	cellulose, derivative, containerised, modular, synthesis

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Abbreviations

CMP	Containerised modular plant
REACH	Registration, Evaluation, Authorisation and Restriction of Chemicals (European Union regulation)
Tukes	Bureau of safety and chemicals (Turvallisuus- ja kemikaalivirasto)

1 Introduction

The purpose of this Bachelor's thesis was to study distributed manufacturing by means of flexible modular containerised process in the context of regenerated cellulose textile fiber. Cellulose carbamate synthesis, known in literature and enabling separation of cellulose derivate manufacturing from dissolving and actual fiber spinning, was selected as a case for the project. The aim was to study was the scalability of the containerised modular plant without compromising the highest agility.

The units would be fitted into a standardised shipping container. The installation of the containerised plants must be flexible so that it can be performed in different factories with different layouts. Furthermore, the installation can take no more than 24 hours.

The process utilizes wet fibre mass to produce a cellulose derivate. Utilities, such as heating and wastewater management, are expected to be provided by the master factory itself. The raw material would be supplied by the master factory as well.

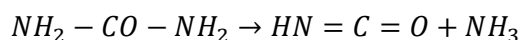
Particularly of interest are the factors and regulation relating to the scaling of the process. It is necessary to estimate cellulose derivate processes with different capacities and adjust the modular design so that a minimum amount of alterations to the design and layout of the process are needed.

2 Cellulose derivative

Regenerated cellulose fibers are produced by means of dissolving, spinning and regenerating cellulose. There are several solvents suitable for this, and the most commonly used process is the rayon process, where cellulose is dissolved by alkali. However, cellulose is poorly dissolving in alkali and that is why cellulose derivatives are applied, for instance cellulose xanthate when producing viscose fibers. Cellulose carbamate is considerably friendlier towards environment than carbon disulfide-based xanthate. In addition, it can be stored and transported unlike the alternatives. [1; 2; 3]

Carbamate is an organic compound derived from carbamate acid (NH_2COOH), formed from urea in elevated temperature. Carbamate group in cellulose molecule supports dissolution in brine (NaOH -solution). When cellulose is treated with urea at a temperature higher than 130°C , which is urea's decomposition temperature, it forms a derivative known as cellulose carbamate, CCA. The compound is thermally and chemically stable, non-toxic and biodegradable. [1; 2; 3]

Decomposition of urea can be expressed in the following reaction equation:



Formation of cellulose carbamate can be written as follows:

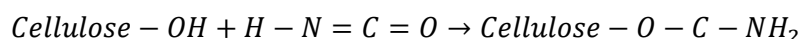
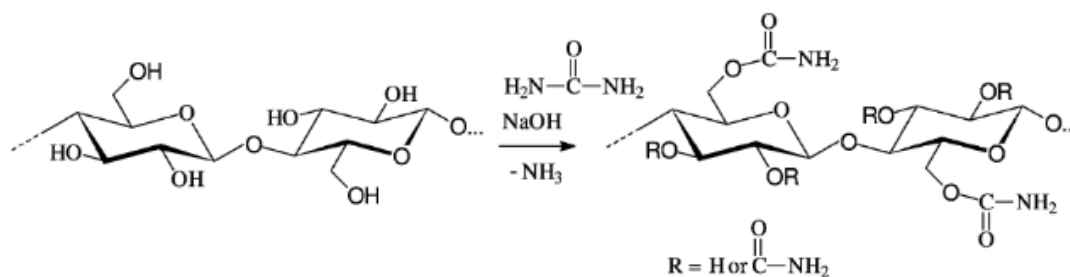


Figure 1. Formation of cellulose carbamate [3]



3 Containerised modular plant

The containerised plant as a concept refers to processes that are situated in a shipping container for easier transportation. Modular refers to the option that the individual parts and machines can be swapped or left out based on what is wanted. Containerised modular plants, CMP:s, are used in a wide area of industries, for example, mining, pharmaceutical pilots and water treatment. CMP is an ideal choice when the production site is hard to access, available space is limited, or the production facilities have to be mobile for quick relocation. CMP brings versatility and flexibility to production in terms of faster reaction time to changes in production, available options to customize processes and unit distribution. [4; 5; 6; 7]

In this thesis the process was situated in containers specifically for the ease of transportation and relocation. The process would be situated next to the raw material and utility supplier and could be relocated if necessary. The process could be tailored depending on the needs of the customer. Two most common container models, the 20 ft. and the 40 ft. long one, were chosen. The most common models would be a ready and economically feasible solution. [8; 9]

4 Placement of the process

An ideal location for the synthesis process would be next to a cellulose- or refining master factory. In addition to a steady supply of raw material, certain crucial benefits are gained by this placement. Such benefits include utilities, wastewater disposal and an operating area that holds already the required laws and regulations relating to chemical production. [10]

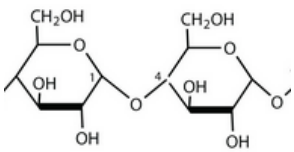
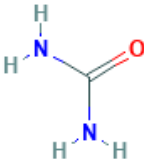
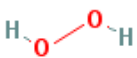

5 Design of the synthesis process

It was essential that the processes fit into a standardised container or containers. The goal was to first estimate the range of capacities with the spatial limitations in mind. The capacities would then be used to dimension the unit processes.

The cellulose-containing raw material would be supplied by the master factory via a belt conveyor. The raw material would be free of mechanical contaminants, mechanically opened for appropriate size and wetted. This means either re-pulperized cellulose or open carded cotton, which has been washed or bleached and thereafter pressed to known solid content as performed in the patent WO201404125A1 [11]. [10]

The process utilizes solutions of urea and hydrogen peroxide dissolved in water as chemical inputs. The hydrogen peroxide is to control the degree of polymerization which relates to the viscosity of the solution. [12; 13]

Table 1. Raw materials and their properties [14; 15; 16; 17; 18; 19]

Commercial name	Cellulose	Urea	Hydrogen peroxide
Formula	$(C_{12}H_{20}O_{10})_n$	CH_4N_2O	H_2O_2
Structure			
M (g/mol)	170	60	34
Boiling point (°C)			141
Melting point (°C)	260-270	132-135	-11
Density (g/cm ³)	1.50	1.32	1.40
Hazards			strongly oxidizing, corrosive, harmful
Hazard symbols			

The cellulose feed consistency was assumed to be 85% for the balance calculations. The value was obtained by comparing values from literature and existing trials and ad-


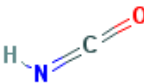
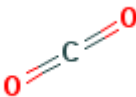


justing the value to ensure the necessary water content [10; 12]. Should the water content be any larger, there are two options. The chemicals could be added dry, which requires a longer mixing time so that the urea dissolves in the mixer. The other option would be to add the chemicals as solutions and extend the synthesis duration to evaporate the water. Of the two options the latter is preferable. It would be better that the chemicals are added as ready solutions to ensure that the required amount of urea will be dissolved and ready to penetrate the cellulose fibre. It would be harder to ensure proper dispersion of active urea when urea is input dry because the mass in the mixer is very viscous. Should the water content be less than in the assumption, it would help perform the synthesis faster but make the mixing and pumping of the mass harder. Additional water could be added if necessary.

Suitable conditions for the synthesis to occur are a temperature range of 140-170 °C and a reaction time of 2-3 hours [10; 12; 20; 21; 22]. This is the temperature range where carbamic acid forms autocatalytically from urea releasing ammonia. A temperature higher than 175 °C yields less reaction time but produces unwanted compounds from the decomposition of urea into isocyanate, biuret and ammonia gas. Moreover, cross-linking and carbonization of cellulose occurs [22]. The actual conditions in the reactor were determined to be 660 mbar vacuum for better evaporation of water, nitrogen atmosphere for stability, reaction temperature of 152 °C and reaction time of 3 hours based on existing trial runs of similar processes [10].

When the synthesis is complete, the product would be discharged into the mill. Since the product is in a powdery form, dust filters would be essential to install into the reactor to collect and return dust generated from the discharge. Milling is a crucial step. The function of the mill is to shred the formed derivative so that its active surface area is greatly enhanced for the dissolution process. [10]

The formed gasses from synthesis are basic and have to be processed accordingly. It was deemed appropriate to adjust the pH level of the outlet gasses with hydrochloric acid so that they could be discharged to the master plant's waste water treatment network. Carbon dioxide could be released through an outlet after condensing the water vapour and ammonia gas.

Table 2. Formed chemicals and their properties [23; 24; 25; 26]

Commercial name	Ammonia (anhydrous)	Isocyanic acid	Carbon dioxide
Formula	NH ₃	HNCO	CO ₂
Structure			
M (g/mol)	17	43	44
Boiling point (°C)	-33		
Melting point (°C)	-78		
Density (g/cm ³)	0.70	1.32	1.40
Hazards	corrosive, flammable, toxic, harmful	hazardous, corrosive,	
Hazard symbols			

The primary unit processes in this process were determined with information from literature and trial runs of similar processes [10; 12; 13; 20]. The primary unit processes would be the mixer, the compactors, the synthesis reactor and the mill. Cellulose and chemicals will be added into the mixer where the mechano-chemical impregnation begins.

Compacting follows mixing and the mass will be cycled through the unit several times. It is essential since cellulose crystal structure will not allow enough urea to penetrate. Compacting will finish the mechanical impregnation and allow the urea to penetrate the tighter regions of the cellulose fibre and textile structures. [10; 12]

Maximum capacity was determined by the maximum size of the process equipment that could still be fitted into containers. The smaller capacities would fit into the 20 ft. containers and the larger into the 40 ft. containers. The two containers do not differ in any other dimensions than length.

Lödige CGT Druvatherm Granulator was chosen as the optimal reactor for the synthesis. A typical use of the reactor is treating different powders, for example, starch modification. The CGT 6200 was chosen due to its availability and commonness on the market [10]. The other capacities provided by Lödige were used as the range of capacities studied in this thesis [27]. The CGT 6200 and the calculations related to it are presented in this thesis and the same method is applied to the other capacities.

The mixer tank could be designed at will. The compactor and the mill were predetermined as Kahl compactor and Atrex mill, respectively, and they would easily fit into a container [10; 28; 29]. Thus, from the dimensions provided by Lödige, it was clear that the synthesis reactor was the bottleneck. [27]

Information about the residence times was searched from the literature. The compactor would process the mass multiple times to ensure proper penetration of chemicals. The suitable amount of cycles would have to be determined empirically. Since the residence time differed greatly between the units, the operating style would be a continuous batch process. [10; 12; 20; 21; 22]

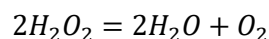
Table 3. Residence times of the units [10; 12; 20; 21; 22]

Mixer	10 minutes
Compactor	1-2 minutes per cycle
Reactor	2.5 hours
Mill	1 minute

5.1 Balance calculations

Following assumptions were made concerning the reactions occurring in the process [10; 13]:

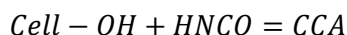
- All hydrogen peroxide reacts to oxygen and water.



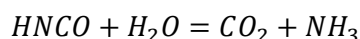
- Conversion of urea to ammonia and isocyanic acid is 60%.



- All cellulose reacts with isocyanic acid to form the derivative, cellulose carbamate.



- All left over isocyanic acid hydrolyses into carbon dioxide and ammonia.



The process starts with three input flows, wet cellulose from the factory, solution of urea and water and solution of hydrogen peroxide and water. Cellulose was estimated to be at 0.85 % consistency in input flow for the calculations. It was deemed necessary to dose urea 70% in relation to the dry mass of cellulose for successful derivatisation. This had been empirically tested to lead to a preferable outcome [10]. Furthermore, water content in mixing was necessary to be in the range of 20-25 % to promote successful compacting and also eliminating the need for drying of the mass before synthesis [12; 20].

It was calculated that in order to gain enough urea, the concentration of the urea solution had to be 70 %. At temperature levels of 50-70 °C it is possible to attain such a strong solution of urea [30]. The temperature should not exceed 65 °C during mixing so as to prevent the decomposition of urea occurring prematurely [13]. Moreover, the temperature should not exceed 50 °C to leave a margin, since compacting would raise the temperature of the solution. The concentration of the hydrogen peroxide solution would be 30 % and it would be dosed 3 % in relation to the mass of the dry cellulose [12; 31].

The working capacity of Lödige CGT 6200 was 3.1 m³. This value was used as the value for the volume of the mixer. Hydrogen peroxide is absent since it was assumed that all of it has reacted. Full results are in Appendix 1.

Table 4. Parameters in mixing phase

	Cellulose	Water	Urea	
m-%	0.44	0.25	0.31	kg
Mass	1793	1037	1255	kg
Mass_tot.	4085			kg
V_tot.	3.10			m ³
Water content	0.25			

The density of the solution in mixing was calculated with the mass percentages from table 4 and the densities from table 1.

$$\rho_{solution} = 1318 \frac{kg}{m^3},$$

where $\rho_{solution}$ is density of the solution

Compacting followed mixing. It was assumed that there were no waste flows and the balance would stay the same.

The following step was the actual synthesis. The flows were converted to molar flows to calculate the products of the synthesis reaction. Conversion of cellulose into cellulose carbamate was estimated to be 60 %, a somewhat realistic value, because 100 % conversion would not be achievable [10]. Molar mass of the derivative was assumed to be approximately equal to that of cellulose (170 g/mol compared to 162 g/mol of cellulose). Although the derivative would have the additional reacted isocyanic acid in its structure, the polymer structure of the cellulose was reduced in the reaction as well [10].

Table 5. Output (Full results can be seen in Appendix 2)

	Carbamate	
Moles	11.1	kmol
Mass	1882	kg

The accuracy of the estimations was checked by comparing the amount of cellulose carbamate in relation to the amount of cellulose feed. Comparable results were searched for in the literature. A pilot scale test to produce carbamate derivative performed by Chinese and Japanese Universities stated that if 2 kg of cellulose were used, 2 kg of derivative would be produced [21]. The relation in this thesis was approximately 2000 kg of both cellulose and derivative, which was sufficient.

It was assumed that all water would vaporise during the synthesis step to simplify the calculations. The left over isocyanic acid would hydrolyse into carbon dioxide and ammonia gas with the water present in the reactor. Ammonia gas would be produced as well by the decomposition of urea into isocyanic acid. The formed vapours were calculated from the molar flows in the balances.

Table 6. Formed vapours from the synthesis

	NH3	water(vapour)	CO2	
Moles	14	56	1.5	kmol
Mass	239	1010	65	kg

5.2 Dimensioning primary process equipment

This thesis aimed to dimension the equipment while keeping in mind that they had to be housed in the standardised containers. Safety margin of 10% was used where necessary.

The dimensions and weights of the chosen standard containers are presented in Table 7:

Table 7. Dimensions and capacities of the standard containers [32]

	20 ft. container	40 ft. container
Internal Length (m)	6.01	12.11
Internal Width (m)	2.34	2.34
Internal Height (m)	2.39	2.39
Inner Volume (m ³)	33.61	67.73
Container weight (kg)	2 170	3 750
Maximum payload weight (kg)	28 310	26 730

Due to the longer length, the 40 ft. is slightly weaker structurally.

The pre-mixer was assumed to be a tank and was calculated manually. The dimensions of the mill were estimated from pictures provided by the manufacturer. Lödige and Kahl both provided extensive information about the dimensions of their products.

It was estimated that the electrical installations and automation, for example, would take approximately 50% of the available space in the container [10]. This thesis concentrated on the primary unit processes and the auxiliary units such as pumps and conveyors. What was not covered in detail was the rest of the required related installations. The space requirement for the supplementary installations was taken in to account by leaving approximately 50% of the space empty.

5.2.1 Mixer

Cellulose would be immersed into urea and hydrogen peroxide in the mixer to begin the chemical impregnation. It was assumed that hydrogen peroxide reacts fully into its constituents, water and oxygen. The tank would have inlets and an outlet for formed gasses on top. Mixer was assumed to be a standard propeller-type mixer and would be fitted on top as well. [12]

The tank was assumed to be a round cylinder and its volume was calculated as such with formula 1. 10% margin was added as a safety measure. Values of diameter and height were estimated to reach the necessary working capacity and by keeping the spatial restrictions in mind.

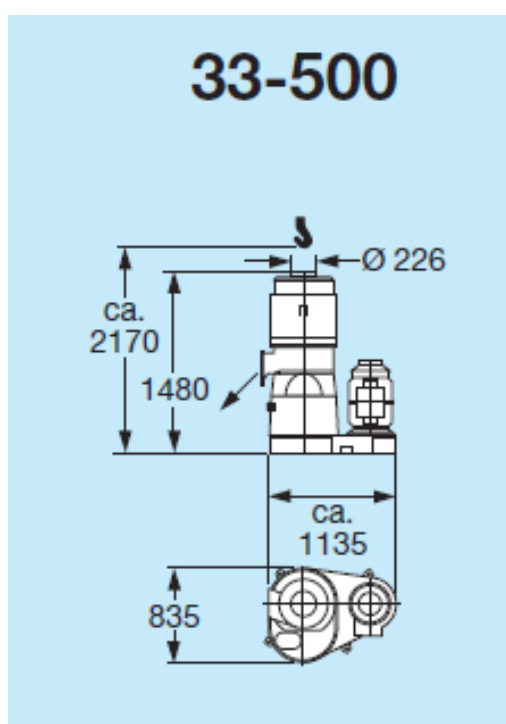
$$V_{tank} = 1.1 \frac{\pi d^2}{4} h = 1.1 * \frac{\pi * 1.46m^2}{4} * 1.7m = 3.1m^3, \quad (1)$$

where V_{tank} is the volume of the mixing tank

5.2.2 Compactor

The compactor manufactured by Kahl had been predetermined to be a suitable option [10]. The model 33-500 was chosen due to its small size and weight [28]. The larger models have their motors installed horizontally, taking more space. Information about the compactor was provided by a Kahl retailer (Hartek). The aim was to use two compactors to double the capacity. [10]. The model 33-500 fits into the container as a pair and leaves the desired empty space requirement as well.

Figure 2. Dimensions of the chosen Kahl compactor [28]



5.2.3 Synthesis reactor

As mentioned before, the CGT 6200 was chosen as a starting point for the calculations with working capacity of 3.1 m³. CGT 6200 could be run as a continuous process and it was readily jacketed vessel. Moreover, the drum can operate in total vacuum and the jacket can hold pressure up to 5 bar. The unit can withstand temperatures up to 160 °C, which is above the critical temperatures for the synthesis. [27; 33]

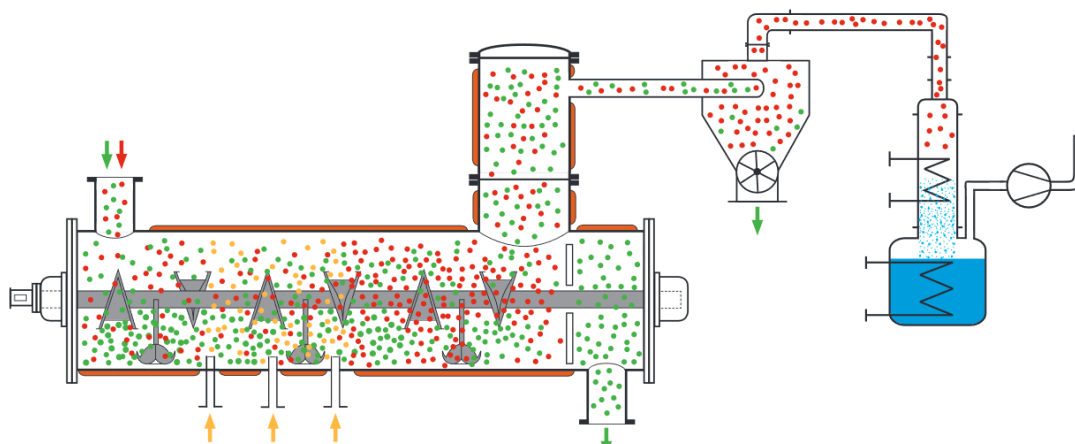
According to the literature, temperature of saturation of water vapour at a pressure of 5 bar would be 152 °C which is ideal to the synthesis [34]. The vapor could be overheated if necessary.

Lödige provided information, capacities and dimensions extensively for other commercial models as well. Even the largest model would fit into a container and still leave enough empty space. Furthermore, Lödige was the ideal choice since it had already been used in similar processes [10].

Table 8. Lödiges commercial models [27]

Type	Drum volume (l)	Working capacity (l)	Length (mm)	Breadth (mm)
CGT 300	300	150	3000	550
CGT 600	600	300	3800	590
CGT 800	800	400	4800	590
CGT 1200	1200	600	4900	700
CGT 2000	2000	1000	5050	880
CGT 2400	2400	1200	6050	880
CGT 2800	2800	1400	5300	1040
CGT 3500	3500	1750	6300	1040
CGT 4800	4800	2400	6300	1220
CGT 6200	6200	3100	6450	1380
CGT 8000	8000	4000	6450	1560

Figure 3. CGT 6200 DRUVATHERM [33]



The yellow arrows in Figure 3 represent the possibility of aeration drying. The red arrows indicate formed vapours and the green indicates raw material and product.

5.2.4 Mill

The Megatrex Atrex G-series mill whad been predetermined to be the mill that wouldlead to an acceptable outcome [10]. Atrex contains two counter-rotating drums that cause great shear, turbulence and impact forces to the processed mass. [29]

Figure 4. Atrex G-series mill [29]



The dimensions of the mill were purely assumed from the pictures in Megatrex's website by comparing the size of the surroundings to the mill (walkways, for example). It is assumed to be 2 metres long and 1 metre high. Accurate values were not provided by the manufacturer for this thesis.

Figure 5. Atrex mill in its natural habitat [29]



5.3 Basic gas condenser and pH adjuster

It is necessary to treat the formed vapours from the synthesis, due to the presence of ammonia. Hydrochloric acid would be used to neutralize the liquid due to its availability and cheapness. The neutralized liquid could be discharged into the master plants waste water system. The formed carbon dioxide could be released into the atmosphere through an outlet. The process was assumed to consist of a tank with the pH adjustment equipment.

This process would be placed into a container of its own. This unit was dimensioned to be able to handle the amount of basic solution from one batch from the process utilizing the CGT 6200. In the case of the smaller processes, this unit could accumulate the solution and handle it all at once or simply neutralize the smaller batches.

The amount to be treated was calculated from the balances. The mass was calculated from the amount of moles and the corresponding mass percentages were used with the densities to determine the density of solution. The density and the mass were used to calculate the volume of the basic solution.

$$V_{basic_solution} = \frac{m_{liquids}}{\rho_{liquids}} = \frac{1248.74 \text{ kg}}{808.93 \frac{\text{kg}}{\text{m}^3}} \approx 1.6 \text{ m}^3, \quad (2)$$

where $V_{basic_solution}$ is the volume of the basic solution

Hydrochloric acid would be added equal to the number of moles of ammonia; thus the volume of the pH adjuster had to be larger than just the volume of the basic solution. Moreover, hydrochloric acid would be added as 38 % solution which would be commercially available [35].

$$V_{HCl} = \frac{(n_{HCl} * M_{HCl})}{\rho_{HCl} * 0.38} = \frac{14.6 \text{ kmol} * 36.5 \frac{\text{kg}}{\text{kmol}}}{1.2 \frac{\text{t}}{\text{m}^3} * 0.38} \approx 1.17 \text{ m}^3, \quad (3)$$

where V_{HCl} is the volume of needed hydrochloric acid

Total volume of the pH adjuster containing the safety margin was calculated using results from formulas 2 and 3.

$$V_{pH_adjuster} = (V_{basic_solution} + V_{HCl}) * 1.1 \approx 3.05 \text{ m}^3, \quad (4)$$

where $V_{pH_adjuster}$ is the volume of the pH adjuster

5.4 Pumps, tanks, conveyors and piping

In addition to the primary process equipment, the chemical feed container holds two chemical feed pumps and two chemical feed tanks. The process containers would need four screw conveyors to facilitate the transportation of the mass from one unit to the next and of course, piping. The pipes would be connected by flanges or possibly by quick connectors. The quick connectors would enable faster start-up and faster installation on-site. The downside would be that the quick connectors are most likely vastly more expensive than connection by flanges. [36]

These units are assumed to fit into the containers and they are dimensioned in the cost estimation part.

5.5 Process description

The mass is assumed to stay unchanged during the transportation, compacting, synthesis and milling.

The chemical pumps PU101 and PU102 pump 1793 kg of urea solution and 179 kg of hydrogen peroxide solution to the mixer TK101. The raw material, cellulose, arrives by a belt conveyor from the factory. The amount of cellulose per batch would be 2110 kg. The cellulose is immersed into the chemicals in the mixer tank TK101 and is agitated with the agitator MI101. Hydrogen peroxide reacts into its constituents, oxygen and water. Total mass in the mixer tank would be 4085 kg.

The mass proceeds, via the screw conveyor CV101, to the compactors. The compactors were classified as a special equipment ZZ101 and ZZ102. The mass transported and compacted stays unchanged as 4085 kg.

The mass proceeds to the synthesis reactor RC101, via the screw conveyor CV102. In the synthesis the urea decomposes into isocyanic acid and ammonia and the left over isocyanic acid hydrolyses into carbon dioxide and ammonia. The formed gasses would be led to the pH adjustment tank TK102. The mass of the formed gasses would be 1314 kg. The mass of the produced derivative would be 1882 kg.

The mass proceeds, via the screw conveyor CV103 to the mill GM101. The mass would be shredded in the mill GM101 and transported via the screw conveyor CV104 to storage. The mass transported to storage would be 1882 kg.

The flow diagram is in the Appendix 3.

5.6 Special aspects on container installation

The process units are heavy and during operation represent a dynamic payload. Certain aspects have to be taken into consideration when installing, transporting and operating the process containers.

5.6.1 Steel structure rigidity

The containers are built to withstand harsh conditions and stress caused by constant transportation. The containers are made of “corten” steel, which stands for corrosion resistant and tensile strength. In the event that the paint layer wears off, corten steel forms a protective layer of oxidized metal that protects the underlying layers from corrosion. [37; 38; 39]

The steel structure is durable and tough so that the containers can be stacked eight to nine units high while loaded to full capacity. This can be achieved during transportation, but not when the process is operational due to the dynamic mass. The point of placement has to be even to avoid warping of the container. In addition, all necessary regulation and marking has to be observed. [40]

When modifying the containers, one should always keep the structural integrity in mind. The containers will most likely be moved multiple times during the lifecycle of the modular units inside. This means that the container has to be able to withstand the stress caused by transportation. Piping through the containers is common, but if larger modifications, for example, hatches or larger connections are needed, a container professional should be consulted. [40]

5.6.2. Centre on gravity and balancing

It is possible that the process units are transported in the same position in the container in which they operate. The units could be installed by the machine shop that built them. Additional fastening would be added during transportation.

In the presented case, the CGT 6200 reactor fills approximately half of the container and could possibly be situated near the middle, stabilising the container. If the reactor has to be installed near the end, the centre of gravity has to be considered. The reactor could

also be much heavier on the other side. The electrical installations have to be taken into account as well and determine which components have to be installed at the shop and which can be installed at the destination. It is unlikely that this container would exceed the weight limit.

The mixing/compacting container presents an easier case. The compactor is the heaviest unit in this container, and they would be situated approximately in the middle in the operational layout. The compactor weights 1300 kg alone, but even together and with the mixer, there is considerable distance to reach the weight limit of the container [28]. It would most likely be easiest to transport this container and its contents readily installed and fastened. The milling container could most likely be transported readily installed, since it contains only the mill as a primary process unit. It is unlikely that this container would exceed the weight limit.

5.6.3 Internal support for transportation

The structure, both inside and outside, allows multiple points of contact for attachment and fastening [38, 39]. The payload should be distributed evenly, and the point of placement should be even to avoid warping of the container. Furthermore, if the payload contains units that distribute their mass to a very small area, they should be placed on weight-distributing platforms or load-transfer beams. [40] In the case of this thesis, no such case should exist.

The process equipment has to be fastened individually keeping the centre of balance in mind. The fastening has to be done so that in the event of tilting or any type of sudden change, the payload has to stay immobile. In addition, top-over lashings and friction increasing materials would be preferable. [40]

The other way would be to load to container fully so that no empty space remains for the payload to move into. In the case of this thesis, fastening would be much more practical. Extra care has to be administered if the transportation is done by roads, instead of by rails or by sea. Trucks are more sensitive to the centre of mass because it directly affects steering and braking. Professional should always be consulted when loading containers. [40]

6 Regulation and permits

One of the aims of this thesis was to inspect possible regulations and permits relating to connecting these individual unit processes to form the derivative synthesis process, scaling and consumed and produced chemicals. It was assumed that manufacturers of individual units have made sure that all machinery has passed and received CE labels and conforms with EU law regarding machinery. The process itself is labelled as joined machinery that form a process. The process would be constructed by a machine shop that makes sure that the machines would be joined and attached to the containers according to EU laws and regulations. [41; 42]

The main used chemicals include urea, cellulose, hydrochloric acid and hydrogen peroxide. The main formed chemicals include ammonia gas and cellulose carbamate. Cellulose, cellulose carbamate and urea can be considered harmless [3; 14; 15; 43].

The dangers relating to temperatures used in the process were critical to evaluate. The self-combustion temperature of ammonia gas is 651 °C and decomposition temperature > 540 °C, neither of which caused any concern in this case [44]. Hydrogen peroxide is not flammable or combustible [19]. Hydrochloric acid is not flammable nor combustible [45]. Nevertheless, hydrogen peroxide, hydrochloric acid and ammonia are considered as hazardous materials and need to be taken into account.

It was deemed necessary to check, depending on the amount of chemicals used and produced, whether any substantial regulatory workload would be caused. The search was conducted as per Finnish law due to the high possibility that this process would be situated in Finland. Furthermore, Finnish and EU regulation have been harmonized extensively and many degrees by the Finnish government are based on EU law. [46, 47, 48]

According to the Finnish government, the scale of the operation is determined by factors specific to each chemical and the amount of that chemical. If the cumulative value of these factors is less than or equal to one, the operation can be considered as a minor operation. Notification of an emergency official is necessary when minor operation is considered. If the value is greater than one, then the operation is considered as a large-scale operation and notification of Tukes is required. [46]

The scale and necessary notifications were determined with a calculator provided by Tukes [49]. Hydrochloric acid solution, ammonia solution and peroxide solution were inputted to the calculator. Maximum masses of the chemicals at any given moment were needed as well. The maximum mass per chemical would be no larger than the amount used per batch. The masses were calculated from the balances.

Table 9. Maximum amounts of hazardous chemical per batch

Chemical	Amount in tonnes
Ammonia	0.25
Hydrochloric acid	0.54
Hydrogen peroxide	0.06

It was clear from the result of the calculator that the operation could be classified as minor operation [49]. Moreover, fortuitously, the amounts of the chemicals in question were small enough that a formal notification to an emergency official would not be necessary. The results apply for all the smaller capacities and for the one larger, as well. The full results can be seen in Appendix 4.

According to REACH the production falls under REACH legislation and its regulatory policy. According to Annex XI of REACH, if certain tonnage limits are exceeded, certain regulatory policies apply. Depending on the tonnes produced per year, additional information may have to be submitted. In addition, all information included in the lower tonnages, if there are any, has to be submitted as well. The tonnes producer per year -limits are one, 10, 100 and 1000 tonnes. [50]

It was essential to estimate the tonnes produced per year to be able to consult REACH. To calculate the number of tonnes, it was estimated that the production efficiency would be 65% for one year and that the density of cellulose carbamate is 1300 kg/m³ [10].

Table 10. Estimated output per year

per batch	per day	per year	per year
Cap. (m3)	Cap. (m3)	Cap. (m3)	Cap.(t)
0.15	0.9	214	278
0.3	1.8	427	555
0.4	2.4	569	740
0.6	3.6	854	1110
1	6	1424	1851
1.2	7.2	1708	2221
1.4	8.4	1993	2591
1.75	10.5	2491	3238
2.4	14.4	3416	4441
3.1	18.6	4413	5737
4	24	5694	7402

It is clear that all except for the three smallest capacities (red) fall under the jurisdiction of Annex XI, Article 12(1)(e) that regulates manufacturing of 1000 tonnes or more per year. The Article 12(1)(e) sets additional demands for submitted information about topics such as reproductive and developmental toxicity, carcinogenicity and fate and behaviour in the environment. [50]

7 Cost estimation

Expenses for the primary unit processes were estimated, and the rest were calculated as percentages of the total expenses. It was far too soon to do anything more than a simple, preliminary analysis with available information. The manufacturers of the primary process units were consulted for a price range, except for the mixer. The mixer was a simple tank and could be made by any machine shop and was thus estimated with Matche.com

Lödige was consulted for a possible price range of the synthesis reactor, since it constituted the primary process unit. On the basis of on the cost of the reactor, the rest could be evaluated. Unfortunately, Lödige did not answer the query. It had been predetermined that the synthesis reactor would cost approximately 850 000 euros, and the value was used in this thesis as well [10].

Atrex, the manufacturer of the mill, answered. They provided a preliminary estimate for the price range of the mill. The range was 130 000 – 500 000 euros. It had been predetermined that 150 000 would be the approximate price of the mill.

The Kahl retailer (Hartek) provided a price range of 70 000 – 140 000 euros, depending on whether the customer required just the unit or all the supplementary equipment. An average value of 105 000 euros was deemed sufficient for the price estimate of this thesis.

Matche.com was deemed sufficient for the estimates for the costs of the non-primary units and the mixer. The website had an online calculator for individual process units. The calculator was based on relevant literature and actual costs of purchased equipment. The given prices represented values in dollars in the year 2014. [51]

The inflation was adjusted and finally, the total values were converted to euros according to the currency rates at the time of this thesis (February-May 2018) [52; 53].

Ammonia was the only material causing concern due to corrosion hazards in the main process. The added peroxide was in such small amounts, and it decomposes quickly that it did not cause any concern. The chosen material was chosen to be stainless steel 304 which is cheaper than the tougher 316 variant, and it is rated as corrosion proof against ammonia. [54]

Matche.com website required starting information specific to the equipment and calculated a generic value as shown below.

Mixer:

- Tank
- Vertical, cone top & bottom, small
- Stainless steel 304
- Capacity 3.1 m³ (820 gallons)
- Cost ≈ 13 000 €

Agitator

- Propeller, top entering
- 100 hp
- Stainless steel 304
- Atmospheric
- Cost ≈ 33 000 €

Chemical feed tanks

- The tanks were calculated as one volume, taking 40 % of the room of the 40ft. container.
- Tank
- Vertical, cone top & bottom, small
- Stainless steel 304
- Volume 26 m³ (6942 gallons)
- Cost ≈ 44 000 €

Centrifugal chemical pumps

- Horizontal, 1 stage, horizontally split case
- Stainless steel 304
- Packing seal
- Discharge pipe diameter 3 in. (7.6 cm)
- Cost ≈ 8100 €

pH adjustment unit

- Total cost $\approx 10\,100\text{ €}$

Tank

- Vertical, cone top & bottom, small
- Stainless steel 304
- Volume 2 m^3 (528 gallons)
- Cost $\approx 10\,000\text{ €}$

Hydrochloric acid consumption

- Hydrochloric acid consumption was deemed necessary to estimate as well and add it to the total value of the pH adjustment unit.
- The price per metric tonne of hydrochloric acid was estimated from Alibaba.com [35].
- Cost of 200 dollars per metric tonne which made its contribution irrelevant.

$$m_{HCl} = n_{HCl} * M_{HCl} = \frac{14.6\text{ kmol} * 36.5 \frac{\text{kg}}{\text{kmol}}}{1000} \approx 0.5\text{ tonnes} \quad (5)$$

- Cost per batch $\approx 100\text{ €}$

The equipment would represent the largest percentage of the total price since this would be a small, containerized process. The other costs were estimated as percentages. The process would be highly automated, which meant that installation of instrumentation and electrical components represented a larger portion of the total price. Containers are inexpensive, and due to the proximity of the equipment to each other, piping would be minimal. A certain amount of funds would have to be directed to efforts to level the location in which the container would be situated.

It was estimated that the expenses would be distributed as follows. [55]

Table 11. Distribution of costs

	Costs (%)
Equipment	40
Installation	14
Instrumentation and controls	12
Piping	8
Electrical systems	10
Containers	2
Yard improvements	5
Service facilities	8
Land	1
Total	100

Total cost approximation was calculated with the percentage representing the equipment and the cost of the equipment.

$$\text{Total costs} = \frac{2\,124\,475}{0.4} = 5\,311\,188 \text{ €} \approx 5\,400\,000 \text{ €} \quad (6)$$

8 Scaling

Of particular interest was the relation between the expenses of building the process and the capacity during scaling. The maximum and minimum capacities were based on the largest and smallest commercial models by Lödige [27]. Costs relating to different capacities for non-primary equipment were estimated from Matches.com website calculator in the same way as shown above [51].

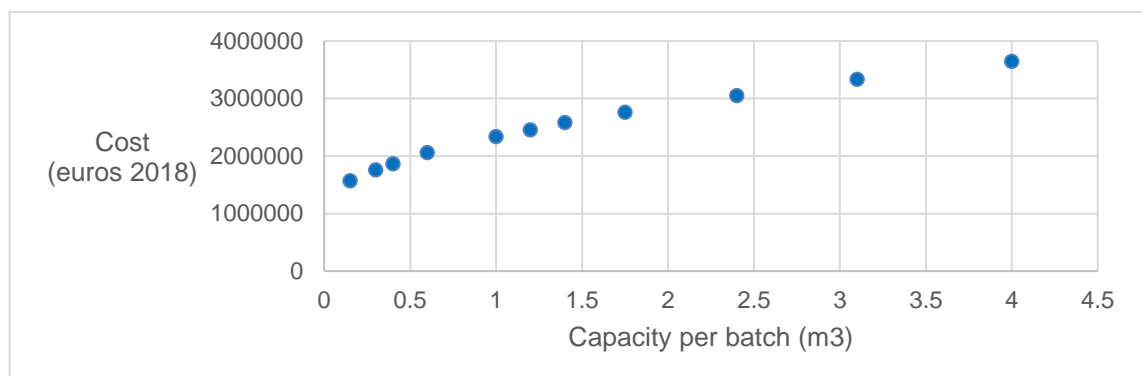
Of the primary process equipment, the mill and the compactors were assumed to stay constant during scaling. They could be thought of as a one-size equipment that could handle the lesser capacities as well as the large ones. The price ranges for the other primary process units were based on the values obtained from the manufacturers.

The agitator required additional estimations to its cost estimation. The agitator's estimation in Matche.com was based on horse powers, which were estimated to be in the range of 25-125 hp [51]. The horsepower value was increased by summing additional 25 hp after three consecutive values (see Appendix 5).

Pumps were assumed to be indifferent to the changes in the capacity. The different outputs would be gained by changing the rotational speed. The same assumption was applied to the conveyors. The pH adjustment device was assumed to be based on the capacity of the chosen CGT 6200 as explained above and be indifferent to changes in capacity as well. Moreover, the cumulative expenses of these secondary units did not cause much difference in the total expenses. These assumptions were applied because this is only a preliminary analysis and they do not constitute the primary unit processes.

The curve in Figure 6 was obtained from the data.

Figure 6. Cost as a function of capacity (see full results in Appendix 5)

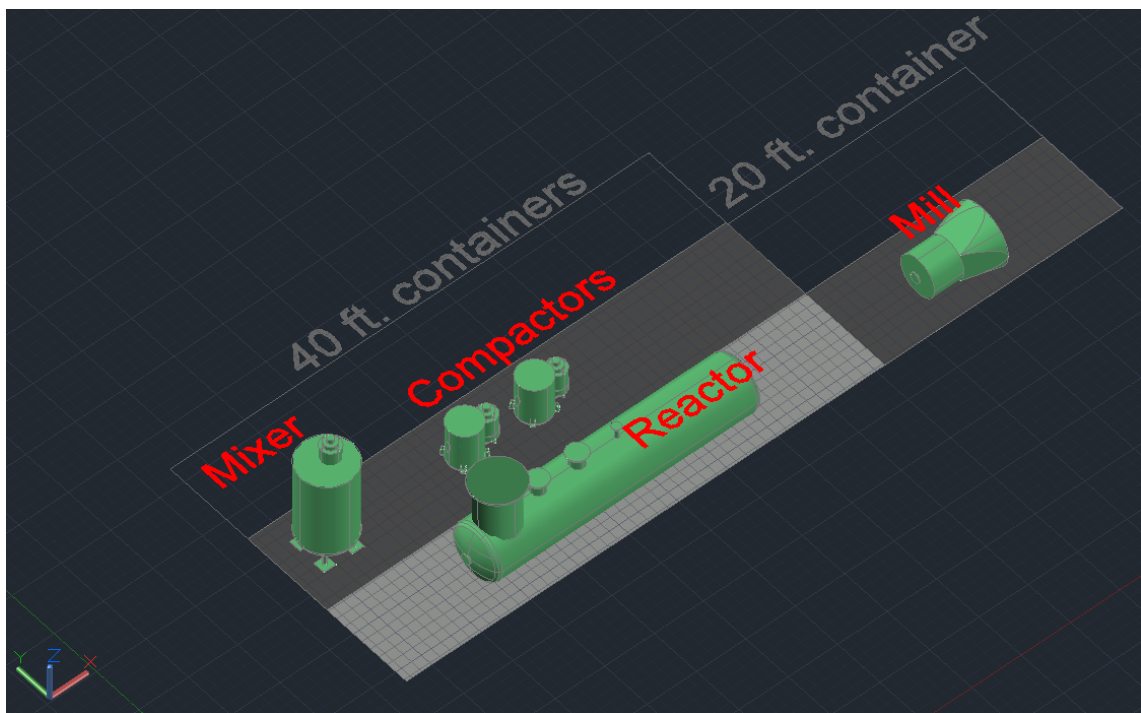


9 Discussion and conclusions

9.1 Scaling, installation and transporting

More attention was focused on the larger capacities since smaller ones did not present a practical or economical solution. Minimum amount of changes would be gained by situating mixing and compacting, the synthesis reactor and the mill into their own 40 ft. containers. It would be practical to house the mill in a separate 20 ft. container due to the emitted noise of approximately 130 dB. It is the last unit process and it is assumed to be one-size. This setup could be utilized in all capacities. The mixing/compacting and the reactor could be situated in 20 ft. containers in lesser capacities.

Figure 7. Lödige CGT 6200 -based process and its space requirement.



If possible, the process would be transported readily installed. This presents a ready process that could be shipped to any location where only starting of the process is required. The customer could choose which units they deem necessary and which they could provide themselves. The containers could be sold or leased to the customer.

All the process equipment would have to be situated so that the centre of gravity would be as close to the middle of the container as possible during transportation. In some cases, it could be a better option to ship the units fastened but not installed. The receiving end would take care of the installation, if possible.

9.2 Cost estimation

An attempt was made to obtain the values for the primary unit processes from the manufacturers because the process was centered around them and they constituted a major part of the costs. Their prices and capacities had to be as accurate as possible for this thesis to be of any use. The other equipment's prices and capacities contained more estimates and assumptions. Nevertheless, the form of the curve of figure 6 represents correctly a curve relating to cost/capacity and does not contain outliers.

It is worth noting that the values here represent order-of-magnitude -values. The values were crude estimates for the expenses of building the process and any conclusions based on this thesis should be handled with caution.

9.3 Regulations and permits

Carbamate is a safe and stabile compound. It is unlikely that the additional REACH regulatory requirements would cause any substantial workload. In addition, the operation was classified as a minor operation by Tukes, and a notification of the emergency official was not necessary. All in all, the approval of a containerized synthesis process can be hoped to be, in terms of regulatory workload, a smooth and quick process. Much is due to the stability and safety of the carbamate compound and urea and small amounts of hazardous chemicals used and formed in the process.

Since the aim is to produce on an industrial scale, utilizing the smaller capacities is hardly worthwhile. Larger capacities cause extra regulation according to REACH. Nevertheless, 1000 tonnes per year in relation to true factories, is a small amount and the regulation needed to go through is nothing out of ordinary. Thus, the recommendation of this thesis is to proceed with the setup that utilizes the working capacity of 3.1 m³ per batch which equals to using the CGT 6200.

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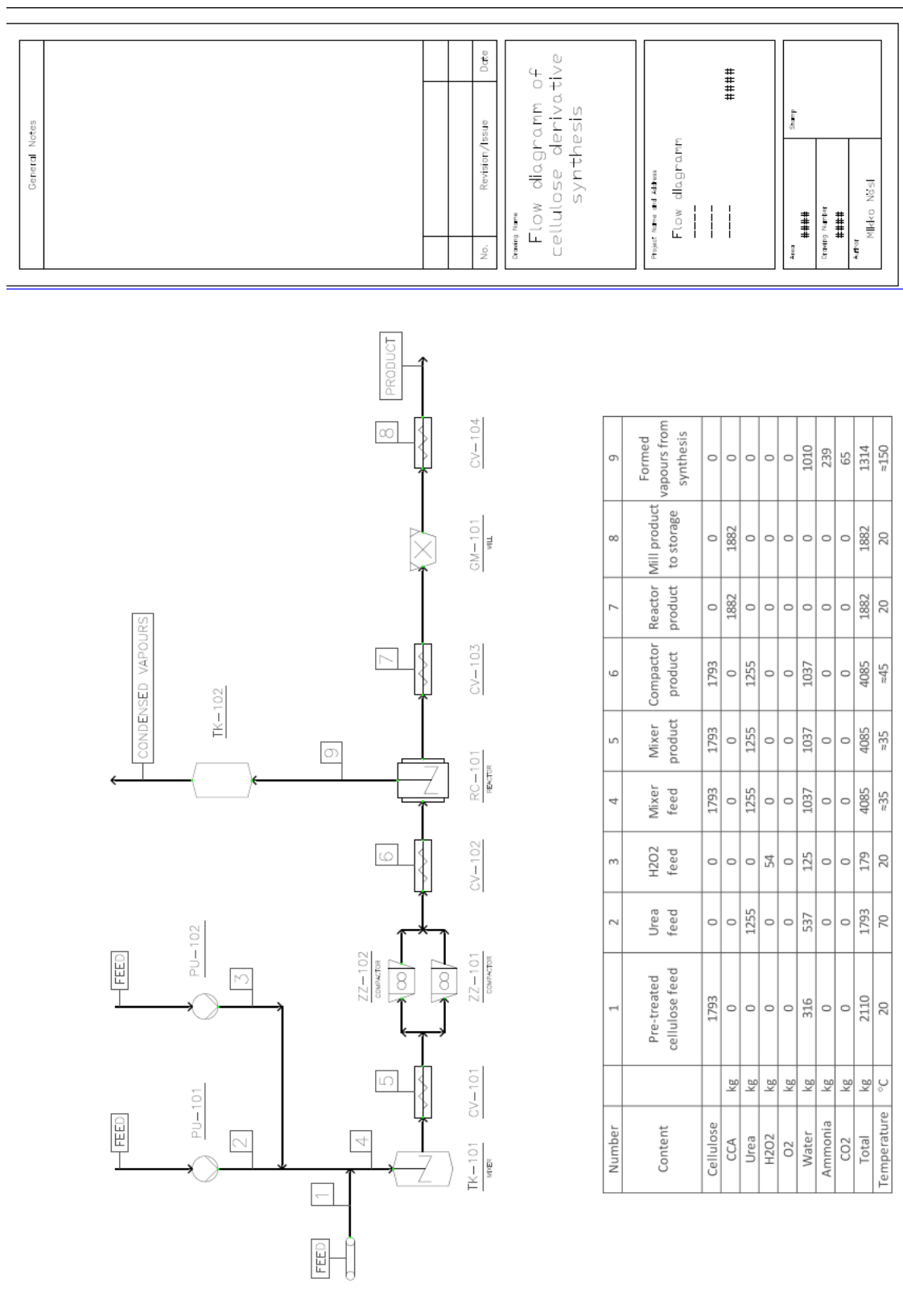
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Balance calculations, Mixing

Properties			SELKO						
	Urea	H2O2	Cellulose	CCA	Water	NH3	O2		
Rho_s	1320	1450	1500	500	1000	730		kg/m3	
Molar mass	60	34	162	170	18	17	32	kg/kmol	
O2									
m_flow	52,5346024	kg/h							
Molar flow	1,64170633	kmol/h							
Cellulose		Water							
m%	0,85	0,15							
m_flow	1860,600502	328,341265	kg/h						
Input flow									
2188,9418	kg/h								
			Mixing						
		Urea	H2O2						
m_flow		1302,420352	55,81801507	kg/h					
Molar flow			1,641706326	kmol/h					
		Urea_solution	H2O2 solution						
m_flow		1860,600502	186,0600502	kg/h					
tot.flow		2046,660553							

Flow diagram



Tukes calculator's full results

Vaaraluokka: Terveydelle vaaralliset kemikaalit									
Kemikaali	Määrä	Ilmoitusraja	Ilmoitussuhdeluku	Luparaja	Lupasuhdeluku	Toimintaperiaateasiakirjaraja	MAPP-suhdeluku	Turvallisuusselvitysraja	IS-suhdeluku
Ammoniakkivesi	0,25	10	0,025	1000	0,000	0	0,000	0	0,000
Vetyperoksidi	0,06	10	0,006	1000	0,000	0	0,000	0	0,000
Suolahappo (vesiliuos)	0,54	10	0,054	1000	0,001	0	0,000	0	0,000
Vaaraluokka: Ympäristölle vaaralliset kemikaalit									
Kemikaali	Määrä	Ilmoitusraja	Ilmoitussuhdeluku	Luparaja	Lupasuhdeluku	Toimintaperiaateasiakirjaraja	MAPP-suhdeluku	Turvallisuusselvitysraja	IS-suhdeluku
Ammoniakkivesi	0,25	1	0,250	10	0,025	100	0,003	200	0,001
Vaaraluokka: Fysikaalisesti vaaralliset kemikaalit									
Kemikaali	Määrä	Ilmoitusraja	Ilmoitussuhdeluku	Luparaja	Lupasuhdeluku	Toimintaperiaateasiakirjaraja	MAPP-suhdeluku	Turvallisuusselvitysraja	IS-suhdeluku
Vetyperoksidi	0,06	5	0,012	10	0,006	50	0,001	200	0,000
Vaaraluokka: Muut vaaralliset kemikaalit									
Kemikaali	Määrä	Ilmoitusraja	Ilmoitussuhdeluku	Luparaja	Lupasuhdeluku	Toimintaperiaateasiakirjaraja	MAPP-suhdeluku	Turvallisuusselvitysraja	IS-suhdeluku
Terveysvaarat			0,085		0,001				IS-suhdeluku
Ympäristövaarat			0,250		0,025		0,000		0,000
Fysikaaliset vaarat			0,012		0,006		0,003		0,001
Muut vaarat			0,000		0,000		0,001		0,000
Suhdelukujen summat vaaraluokittain									
Kemikaali	Ilmoitussuhdeluku	Lupasuhdeluku	MAPP-suhdeluku	IS-suhdeluku					
Terveysvaarat	0,085	0,001	0,000	0,000					
Ympäristövaarat	0,250	0,025	0,003	0,001					
Fysikaaliset vaarat	0,012	0,006	0,001	0,000					
Muut vaarat	0,000	0,000	0,000	0,000					

Cost versus capacity table

Cap. (m3)	Mixer	Agitator	Compactors	Reactor	Mill	pH adjustment	Feed tanks	Pumps	Conveyors	Units (euros)	Total (euros)
0,15	2343	16403	210000	171630	150000	10261	44262	16143	7985	629027	1572567
0,3	3472	16403	210000	245939	150000	10261	44262	16143	7985	704464	1761160
0,4	4079	16403	210000	287648	150000	10261	44262	16143	7985	746781	1866952
0,6	5121	23173	210000	356684	150000	10261	44262	16143	7985	823627	2059068
1	6856	23173	210000	466469	150000	10261	44262	16143	7985	935149	2337872
1,2	7637	23173	210000	513931	150000	10261	44262	16143	7985	983392	2458479
1,4	8332	28380	210000	558037	150000	10261	44262	16143	7985	1033399	2583498
1,75	9460	28380	210000	627552	150000	10261	44262	16143	7985	1104042	2760106
2,4	11283	28380	210000	742132	150000	10261	44262	16143	7985	1220445	3051112
3,1	13105	32719	210000	850000	150000	10261	44262	16143	7985	1334475	3336187
4	15101	32719	210000	973209	150000	10261	44262	16143	7985	1459680	3649201
		25 hp									
		50 hp									
		75 hp									
		100 hp									