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# Investigations into the rheological behavior of aqueous starch suspensions

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# Contents

Abstract						
Intro	Introduction 3					
1.	1. General					
	a.	Sustainability				
	b.	$CO_2$ and its impact				
	c.	Selection of Raw Materials				
2. Starch						
	a.	Overview7				
	b.	Chemical Structure				
	c.	Gelatinization and Retrogradation11				
3.	١	/iscosity 13				
	a.	Basics				
	b.	Newtonian- and Non-Newtonian Fluids15				
4.	(	Cleaning in Place (CIP) and Disinfection				
Mate	eria	ls and Methods 23				
1.	F	Powder analysis 23				
2.	F	Preparation of starch premix and its analysis				
3.	(	Cleaning Trial				
4.	1	4 Microbiology				
Resi	ults					
1.	F	Powder Analysis and Premix Production				
2.	١	/iscosity Measurements and Premix Quality				
	a.	July/August Trials				
	b.	September Trials				
	c.	January 2025 Trials				
3.	(	Cleaning Trials				
	a.	Cleaning Trial 1				
	b.	Cleaning Trial 2				
	c.	Cleaning trial 3 44				
	d.	Cleaning Trial 4				

e	e. Cleaning Trial 5	. 45				
f	Cleaning Trial 6	. 46				
Discu	Discussion					
1.	Starch premix – how powder specifications affect premix viscosity	. 47				
e	a. Quality by amount of lumps	. 47				
k	o. Premix viscosity	. 49				
2.	Cleaning trials	. 53				
Conc	Conclusion					
Appe	ndix	. 55				
1.	Trial Data for Premix Dispersion	. 55				
2.	Reference Pictures for the Cleaning Trials and Validation	. 60				
Indices						
1.	Figures	. 73				
2.	Equations	. 76				
3.	Tables	. 76				
Refer	References					

# Abstract

Biodegradable and renewable materials are heavily sought after in various industrial branches to help companies with a reduction in CO<sub>2</sub> emission. One cheap renewable resource is starch, as it is found in abundance in various grains and tubers.

In this work the preparation of a pre-cooked corn starch suspension in water and formic acid was monitored, looking at various process parameters with the goal of achieving the highest degrees of dispersion and a fast thixotropic recovery. The degree of thixotropy was found to be an important indicator of finely dispersed starch. This behaviour was reduced the more agglomerates were formed during the wetting stage of the premix preparation. It was found that finer powder particles had a detrimental effect on the fine dispersion of starch in the premix and led to a weaker thixotropic behaviour.

Furthermore, a cleaning process was proposed and optimized to reduce colony forming units (CfUs) in all used equipment to less than 10. The cleaning trials were further validated for success by employing UV light to detect fluorescent residue like biofilm.

# Introduction

# 1. General

# a. Sustainability

Sustainability has risen to a highly desirable goal most companies are striving to achieve. It has become a central concern not only within industries but also among the general public, whose increasing awareness of environmental issues has led to greater scrutiny of industrial practices. This shift in public perception has placed pressure on industries to adopt environmentally responsible methods and align with societal values. This represents an opportunity to innovate processes that minimize resource consumption and waste while meeting consumer demands for sustainable products. Beyond meeting these demands, industries benefit from enhanced brand reputation, increased market competitiveness, and access to new customer segments, particularly as sustainability becomes a key differentiator in global markets. Furthermore, adapting to public expectations fosters resilience by preparing industries for stricter environmental regulations and creating pathways to long-term economic and environmental viability. [1]

Sustainable industrial practices improve processes towards being more environmentally friendly, by reducing environmental impact, used resources and energy consumption [2]. At the same time, sustainable practices require economic viability to become a lasting change. One key component is resource efficiency, which focuses on optimizing the use of raw materials and energy. Examples are optimizing the choice of raw material for the production of bioethanol [3], or using raw materials in a more concentrated form or with less pre-processing [4]. As a result, companies reduce emissions and effluents through the adoption of cleaner production techniques and advanced pollution control, which helps the environment. Social responsibility rounds out these components, with industries striving to enhance employee well-being, uphold fair labor practices and support the development of local communities. Also, more governments around the globe demand stricter adherence to sustainable practices from companies. [5, 6] Figure 1 represents the important components of sustainable manufacturing processes.



Figure 1: Sustainable Manufacturing Processes [7]

# b. CO<sub>2</sub> and its impact

The impact on the environment is often closely related to the topic of energy. Most forms of power generators cause CO<sub>2</sub> emissions, which is the main contributor for man-made greenhouse gases and promotes global warming. Industries in the US were accountable for about 30% of the country's greenhouse gas (GHG) emissions in 2022, of which 7% was used for generating energy for the sector. [8]



Figure 2: GHG emissions in 2022 attributed to the described sectors. Left is a depiction of electric power as a separate consumer, right each sector is listed with electric power added accordingly. [8]

The amount of greenhouse gases emitted depends on the used form of power. Figure 3 shows this by plotting tons of  $CO_2$  equivalents per GWh for each form of power generation.



#### Figure 3: Lifecycle GHG Emissions Intensity of Electricity Generation Methods [9]

This graph shows how fossil fuels as energy providers are less sustainable than other technologies in terms of GHG emissions. To promote the switch to renewable energy it becomes clear, why certain governments started using taxes on CO<sub>2</sub>-generation, known as a carbon tax. [10] Another motivation to switch towards more sustainable energy sources is public perception and the aforementioned marketing benefits that result from building a well-perceived brand. Lastly, moving away from fossil energy can sever dependencies from markets that undergo heavy fluctuations, which leads to more accuracy for planning budgets and ensuring fluent and consistent production. [11, 12]

#### c. Selection of Raw Materials

Just like with energy, other industrial sectors can apply the same principle of optimizing the choice of raw material for a process as well, which can greatly reduce the overall carbon footprint of the process. When selecting materials, lower embodied carbon, which is defined as the total amount of fuel-related carbon emissions and process related carbon emissions accumulated during a cradle-to-gate-cycle, will lead to a more sustainable process. One cradle-to-gate-cycle describes the life cycle of a resources' extraction up to the point of leaving the factory. [13] This is one of the reasons why aluminum recycling is a desirable process: Greenhouse gas emissions are 32 times higher for primary aluminum than for secondary aluminum, while energy requirements are 23 times higher. [14] Another benefit of selecting recyclable or low embodied carbon materials aside of carbon savings is the reduction of waste and an improvement of recycling systems. This is also in line with the growing desire for circular economies, where resources are reused or recycled instead of being discarded. [15] One example of this is Renault, where a circular economy factory reuses parts and materials to lower the embodied carbon of their vehicles. [16]

# 2. Starch

## a. Overview

Starch is the main energy storage of many plants on the planet. It is used to store energy in seeds and kernels to help the plant grow and can also be found in various roots and tubers. [17] As such, it is found in a variety of plants, most notably grains, maize, potatoes and cassava, with corn being the biggest contributor to the global market. The global industrial starch market size was valued at around 57 Bn. US\$ in 2023. [18]



#### Global Starch Production by Source 2016-2022

Figure 4: This graph shows the amount of starch produced from 2016-2022 and clearly shows corns predominance on the market. [19]

Starch is also an important carbohydrate in human nutrition, making up about 17-25% of a typical western diet and being responsible for providing 50-70% of our needed energy. [20, 21] Its role in human and livestock nutrition and its availability made starch an essential resource for mankind. Its low price, sustainability and its uses in several industrial sectors like adhesives, paper, detergents, biofuels and, as a replacement of fossil carbon sources, plastics, draw scientists globally to this versatile material. [21, 22]

#### b. Chemical Structure

Starch is a granular solid consisting mostly of a mix of amylose and amylopectin, where amylopectin is the more abundant form usually making up about 80% of starch, heavily depending on the plant. The chemical composition of the primary structure is shown in Figure 5.





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Amylose and amylopectin are polysaccharides built from  $\alpha$ -D-glucose, differing in the size of the molecules and the branching behaviour of the glucose chains. Amylose is only slightly branched and consists of about 100-10000 glucose units linked by  $\alpha$ -1,4-glycosidic bonds and branching randomly with an  $\alpha$ -1,6-glycosidic bond. Amylopectin shows the same types of bonds, yet has a much higher branching rate, leading to its branching structure with molecular weights of ~10<sup>7</sup> g/mol. [22, 24, 25] This results in 3 different branch-types: A, B and C. A-chains are branches without an  $\alpha$ -1,6-glycosidic bond branching off, and with a reducing end. B-chains are chains that still branch off into other B- or A-chains. C chains are the long connecting chains, that connect blocks of B-chains together and can be viewed as the "roots". The length of amylopectin's A-chains is usually around 12-16 glucose units long. [26]

Chains of amylopectin form double-helices, as can be seen in Figure 6 and Figure 7. These double helices can reach a high degree of order, leading to semi-crystalline areas alternated by amorphic areas. These areas are shown in Figure 7 and are described by the lengths  $t_c$  (crystalline lamellae length) and  $t_a$  (amorphous lamellae length). It was reported that in this type of tapioca starch the distribution between the amorphous and the crystalline lamellae, that cause the typical growth rings in a starch granule, was 7.5 nm of length for the crystalline lamellae  $t_c$  and 1.1 nm  $t_a$  of length for the amorphous lamellae.



Figure 6: composition of a starch granule with a cut granule imaged via electron microscopy, showing the growth rings formed by periodically alternating crystalline and amorphous lamellae. [24]



Figure 7: Detailed view of semi-crystalline amylopectin layers. The relevant distances described are L = 8.6 nm,  $t_c$  = 7.5 nm and  $t_a$  = 1.1 nm [27]

Figure 7 also shows the packing of the double helices, which can be monoclinic (A-type) or hexagonal (B-type). [28] The helices are held by both inter- and intramolecular hydrogen bridges.

#### c. Gelatinization and Retrogradation

When starch is heated to a certain temperature, usually 60-80 °C in water, an irreversible process called gelatinization takes place. There, water molecules cause swelling of starch particles by moving into available spaces. [29] Due to its polarity, water molecules can then replace the intermolecular hydrogen bonds between the amylopectin and amylose molecules, causing swelling and the restructuring of crystalline phases to amorphous phases. Due to easier accessibility, this process starts at the amorphous regions of starch and moves to the crystalline regions at a later stage. Figure 8 shows SEM images of the process of gelatinization after treatment with heated water followed by

#### freeze-drying.





Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]

Retrogradation is the process when starch is cooled after gelatinization, leading to an increase in structuring and crystallinity. Amylose with its long and mostly linear structure forms a gel more quickly than amylopectin. [31, 32] The effects and the time needed for retrogradation is therefore dependent on the ratio of amylopectin to amylose.

During retrogradation a reorganization into double-helices takes place. At the beginning of the process the retrograded starch displays high viscosity and adhesive properties, which can be attributed to the restructuring of the longer amylose chains, which mainly takes place in up to 10 hours. [33] When amylopectin restructures and forms intermolecular hydrogen bonds and helical structures again, it results in agglomeration, which can lead to syneresis, a separation of solvent and polymer. [34] It also leads to reduced stickiness, since a higher degree of crystallinity means less mobility of the molecules. This effect is also believed to cause bread to turn stale. The schematic depiction of the process is shown and described very well by Goesart et. al. in Figure 9.



Figure 9:. Schematic representation of changes that occur in a starch–water mixture during heating, cooling and storage. (I) Native starch granules; (II) gelatinization, associated with swelling [a] and amylose leaching and partial granule disruption [b], resulting in the formation of a starch paste; (III) retrogradation: formation of an amylose network (gelation/amylose retrogradation) during cooling of the starch paste [a] and formation of ordered or crystalline amylopectin molecules (amylopectin retrogradation) during storage [b]. [35]

# 3. Viscosity

#### a. Basics

Viscosity is a fundamental property of fluids that measures their resistance to flow under an applied force. It can be understood as the internal friction between two imaginary layers of the liquid. Viscosity is a property of great importance to plan and design processes. In case of transportation, viscosity is important to calculate the pressure necessary to overcome internal friction, friction with a pipe (or another transportation unit) and geodetic height. It is further important to control viscosity to ensure the flowability and thus the ability of material movement in reactions like polymerization. Viscosity also plays a role for certain products consisting of gels or suspensions, where a high viscosity can stabilize a suspension and prevent it from segregation.

The commonly used plate-plate model describes the force, which needs to be applied to a movable plate, that is placed on top of a layer of liquid, which also rests on another, stationary plate. In this model moving the plate very slowly causes the layers of liquid to move with it in a laminar way, causing no mixing between the layers. This leads to a velocity gradient from the top layer of the liquid, moving at the speed of the plate, to 0 velocity, for the bottom-most layer at the interface to the stationary plate. The described model is depicted in Figure 10. [36]



Figure 10: Plate-plate model for the description of viscosity. [36]

The gradient of velocity over perpendicular position to the direction of flow is called shear rate  $\gamma$  and uses the unit s<sup>-1</sup>. The force that is applied to the area of the moving plate is the shear stress  $\tau$ , using the unit  $\frac{N}{m^2}$ . These two parameters form the dynamic viscosity of a medium, as described in Equation 1, using the unit Pa\*s. Due to convenience the unit centi-Poise (cP) is also used to describe dynamic viscosity, with 1 cP = 1 mPa\*s.

 $dynamic \ Viscosity \ \eta = \frac{Shear \ stress \ \tau}{Shear \ rate \ \gamma}$ 

Aside from the dynamic viscosity  $\eta$ , the kinematic viscosity  $\nu$  can be used to describe the resistance to flow similarly. The difference between kinematic and dynamic viscosity is the division of the medium's density for kinematic viscosity  $\nu$ , as is described in Equation 2.

Equation 2: Kinematic viscosity

 $kinematic \ viscosity \ v = \frac{dynamic \ viscosity \ \eta}{density \ \rho}$ 

#### b. Newtonian- and Non-Newtonian Fluids

Newtonian liquids are fluids that display a constant viscosity independent of shear rate, while shear stress is directly proportional to the shear rate. The most prominent example for this would be water. Deviations of fluids from this rheological behaviour are called non-Newtonian. Looking at  $\eta$ ,  $\tau$  and  $\gamma$ , non-Newtonian fluids can be further categorized in time-dependent and time-independent rheological behaviour.

Figure 11 shows a diagram of 4 types of non-Newtonian rheology.



Figure 11: Shear stress over shear rate diagram for Newtonian and non-Newtonian fluids with time-independent rheological behaviour. [37]

Dilatant behaviour describes shear-thickening of the fluid, so a progressive increase in viscosity with rising shear rate. This behaviour can for example be observed with quicksand, where running over the quicksand and thus applying a lot of force, hardens the quicksand, while standing still causes sinking.

Pseudoplastic behaviour describes the opposite, where the viscosity is sinking with an increase in shear rate. A common model to describe the rheology of non-Newtonian fluids is the power law, described in Equation 3.

Equation 3: Power law  $\eta = K \cdot \gamma^{n-1}$  or  $\tau = K \cdot \gamma^n$ 

#### K, n ... Power law constants

Bingham plastic acts as a solid until a certain shear stress, called yield shear stress  $(\tau_0 > 0)$ , is exerted, at which point the behaviour is analogous to a Newtonian liquid. An example for this rheology type would be toothpaste or paint. The mathematical model is described in Equation 4.

Equation 4: Bingham model for non-Newtonian fluid behaviour

$$\eta = rac{ au_0}{\gamma} + \eta_\infty \ or \ au = au_0 + \eta_\infty \cdot \gamma$$

 $\tau_0$  ... Yield shear stress (Pa)

 $\eta_{\infty}$  ... Viscosity at infinite shear rate or a shear rate high enough that the viscosity is constant (Pa\*s)

Similarly, visco-plastic behaviour also requires a yield shear stress level to be reached before then acting shear-thinning like a pseudoplastic. One example of this behaviour would be grease. [38]

Measurements of viscosity require exactly defined process parameters to achieve reproducible results. Temperature is of utmost importance regardless of the rheology of the medium, since it affects the flowability of almost all materials to some extent. This is true for concentration and pressure as well. Furthermore, non-Newtonian fluids might require the same duration of measurement and of course can only be compared at the same shear rate. This often leads to vague or no literature values for liquids, suspensions

and solutions, since a given value would only be comparable to the exact same surrounding parameters.

Common measuring devices can be broadly categorized in 3 non-exhaustive categories:

- Time measurements of falling or rising objects: these objects are often spherical to simplify the geometry of the body that causes the resistance of the liquid to be measured. The object can range from steel balls (falling-sphere-viscometer) to gas bubbles. Since gravity is the driving force for these processes, the density of the materials involved plays an important role. Thus, for these methods the kinematic viscosity is measured.
- Vibrational viscometers: these viscometers operate on the principle of the dampening effect viscous liquids have on oscillating objects. This effect can for instance be measured by measuring the power input required for a constant oscillation, or the time it takes for a known initial oscillation to stop.
- Rotational viscometers:

These viscometers work by having a spindle rotate in the fluid of interest. There are two modes that can be used to calculate the viscosity: constant shear rate and constant shear stress. The constant shear rate mode uses a constant rotational speed and measures the torque needed for the rotation and the resulting shear stress. In the constant shear stress mode, the stress and torque applied are kept constant and the rotational speed is measured with the resulting shear rate.

There are several types of spindles that can be used for these measurements with varying suitability for different viscosities. If a cylinder is used in combination with a cylindrical cup that holds the fluid, the shear rate can be calculated by using the radii of the two parts according to Equation 5. This system is called a concentric cylinder system or a bob and cup system and is shown in Figure 12. ISO-3219 determines the geometries for easy comparisons of the method.



Figure 12: Concentric cylinder system or bob and cup system for rotational viscometers [39]

Equation 5: Equations for calculating the shear stress and the shear rate in a concentric cylinder system of a rotational viscometer. [39]

$$\tau = \frac{M}{2\pi L r^2}, \quad \gamma = \frac{1}{r^2} \cdot \frac{2R_i^2 R_a^2}{R_a^2 - R_i^2} \cdot \omega$$

#### M ... torque (Nm)

r ... distance between the rotational axis and any layer of the sample (m)

#### $R_a$ , $R_i$ ... see Figure 12

#### $\omega$ ... angular velocity (rad/s)

Another variant of rotational viscometer uses the concept of cone and plate. This setup is often preferred if the sample amount is limited, or cleaning of the sample is difficult. The setup has further advantages as the temperature control via the heating plate is quick and simple. There are, however, disadvantages as well, as liquids with a high vapor pressure tend to evaporate, causing the measurement to show too high viscosity values. This can be counteracted by solvent traps which slow the evaporation process down, allowing for extended measurements. Due to the narrow gap between cone and plate (typically around 15 $\mu$ m) and the low sample quantity, the sample should be homogenous and should not have any particles bigger than 15  $\mu$ m. [40] A typical cone and plate setup is shown in Figure 13.



Figure 13: Cone and Plate drawing with d as distance between cone and plate,  $\theta$  as angle of the cone to the plate and  $\omega$  as angular speed.

Time-dependent rheology of fluids is categorized depending on whether the viscosity rises or falls under constant shear rate. Decreasing viscosity is the more common occurrence in nature and is called thixotropy, while rising viscosity at constant shear rate is called rheopectic. The two behaviours are shown in Figure 14, where the graph does not show the behaviour under constant shear rate but rather as part of a hysteresis loop, that forms due to these effects.



Figure 14: Time-dependent rheology shows hysteresis loops as opposed to time independent rheological behaviour. [41]

Figure 15 shows the behaviour of a thixotropic fluid during a step test, which is a test, where a switch between two significantly different, yet constant shear rates takes place

for a certain amount of time. The fluid is suscepted to the lower shear rate first, then to the higher shear rate and after a certain time to the lower shear rate again. This will show if the liquid displays thixotropic behaviour and how stable the fluid is under the applied shear rate.



Figure 15: Flow curve of a thixotropic fluid during a step test. [42]

If the recovery is incomplete, as is shown in Figure 16, the fluid cannot recover its initial viscosity again, which could point to an irreversible change in the molecular structure of the fluid. An example of this would be the cleavage of covalent bonds of long, linear molecules, resulting in fewer intermolecular bonds forming as a result.



Figure 16: Near-thixotropic behaviour of a liquid. [43]

# 4. Cleaning in Place (CIP) and Disinfection

In many industrial processes cleaning is an essential part of the setup. It is necessary to remove colored residue from vessels like pipes and tanks to prevent different colors from mixing. Chemicals might react and form unwanted byproducts, or hygienic issues might arise if cleaning is not performed correctly. Due to the size of industrial plants, cleaning by hand is mostly cost inefficient, since workers need to work extra hours. Also, human errors are possible, especially at the end of shifts when concentration is low and focus shifts towards after-work activities. [44]

For this reason, cleaning in place (CIP) is used as an automated solution, capable of cleaning the setup autonomously without the need to take the setup apart. This not only saves time, but also resources like water. [45] Cleaning steps usually involve rinsing residue from the production runs with water as an initial step. Furthermore, chemicals can be used to assist in the removal of residue. Common materials used are acids, bases, enzymes and tensides (cationic, anionic or non-ionic). Each material class is used for the removal of specific residues. Caustic cleaners are often combined with tensides and are effective in removing organic residues like fats. Acids can be used to remove inorganic materials like minerals and salts, neutralize caustic residues. [46, 47]

CIP works on 4 main parameters, which all affect the overall result of a CIP cycle:

- 1. Concentration of chemicals
- 2. Duration of a cycle
- 3. Mechanical forces applied by looping liquids with enough speed to go from laminar to turbulent flow (above 1.5 m/s as a golden rule)
- 4. Temperature

Reducing one of the parameters usually necessitates raising another one.

In addition to cleaning a process plant, disinfection is another necessity in most cases. Cleaning chemicals might assist in disinfection but generally a separate step with more effective chemicals is foreseen to remove biofilm and microbiological remains. The chemicals used are usually strong oxidizing agents like hydrogen peroxide or peroxy acids, which kill microbes effectively. [44]

One essential prerequisite to utilize CIP is hygienic design of the plant. This starts with pipe routing being installed with a slope depending on the viscosity of the medium to enable draining. At a local minimum (deepest point of a pipe segment) a drain needs to be installed to prevent liquids from gathering. The same is true for dead ends in piping, which should be removed entirely or kept as short as possible.

Drainability is not only required of pipes but also of vats, tanks and other vessels. Optimally, these are equipped with spray nozzles through which the chemicals for cleaning can be dosed with high pressure, resulting in mechanical force applied to dirt or residues on the inside wall. To enable spray nozzles the connections to and from tanks need to be designed as short as possible and conically to improve accessibility of the jet produced by the nozzles. For that purpose, most providers have digital 3D computer simulations, which assist in the correct placement and the necessary number of spray nozzles for CIP. [47]

Spray nozzle placement can also be validated without the use of computer models by a Riboflavin test. This test requires a 0.2g/L Riboflavin (Vitamin B2, shown in Figure 17) dissolved in water. The yellow solution is highly fluorescent. It is filled into the tank as highly as possible and stirred until the solution has reached every part of the tank. The solution is then discarded. Pictures of the now empty tank can be taken, illuminated with a UV torch, showing yellow remains of the liquid on the surfaces. Afterwards, the spray nozzles are used to shortly clean the tank with water, which is discarded immediately.

Pictures of the UV-illuminated tank can again be taken to find any spray shadows, where water was unable to reach the surfaces sufficiently. [48]



Figure 17: Chemical structure of Riboflavin [49]

# Materials and Methods

# 1. Powder analysis

A total of 6 different precooked corn starch powder batches were tested. When procured, all powders were already cooked, dried, milled and sieved. The particle size distribution (PSD) in wt% was tested with sieve towers. The bulk density was measured by filling 700 ml of powder into a cleaned and dried glass beaker with 9.5 cm diameter with as little shaking as possible to measure the poured density. For some select samples a tapped bulk density was measured as well. The water content of the powder batches was given by the provider.

# 2. Preparation of starch premix and its analysis

To produce a premix 8 w% pre-cooked starch was suspended in 91.4 w% deionized (DI) water and 0.6 w% formic acid. Formic acid was first mixed with DI water. 1380 kg of the mix were then filled into a tank while 120 kg of starch powder were filled into a hopper. Once prepared, a high-shear dispersion tool (DA) working with a rotor-stator concept was

used to disperse the powder into the mix by creating a pressure difference between the hopper and the mixing chamber by using the Bernoulli-effect. The slits of the rotor were 4 or 9 mm apart and the slits of the stator 4 or 9 mm as well. The tool was always 9 mm in the trials done in August and from then on always 4mm.

The DA was recirculating the suspension back into the tank. The time it took to disperse the 120 kg of powder could be modulated by how far a butterfly valve at the DA powder induction was opened and varied between 50 and 160 seconds. The DA was used for a total of 4 or 8 minutes at 3000 rpm and delivered a suspension with several undispersed agglomerates. Samples of the premix were taken at 4- and 8-minutes dispersion time respectively and put through a sieve with 0.8 mm mesh size to check for large undispersed agglomerates. Pictures were taken for each trial with the results of the found agglomerates recorded in Table 18, Table 19, and

Table 20 in the Appendix-section.

During powder induction and while the premix was kept in the tank, a high-shear stirrer (DC) was used to prevent the premix from thickening in the tank. The used speed and power for all units are described in the trial reports in the Results- and Appendix- section.

Due to the incomplete dispersion of the starch powder after 8 minutes of dispersing with DA, another high-shear dispersion machine (DB) was used afterwards, containing a tool with a total of 3 rotor – stator pairs. The tool for this machine could be swapped from 3 rotor - stator pairs with 4 mm slits on both parts to 3 rotor stator pairs with 1 mm slits on the rotors and 1 mm slits on the stators. The gap between rotor and stator used a spacing of 0.5 mm. The maximum shear rate of the DB could be calculated using Equation 6.

Equation 6: Calculation of shear rate of a rotor-stator dispersion unit

$$\gamma = \frac{\pi \cdot n \cdot d}{60 \cdot s}$$

n ... rotations per minute (min<sup>-1</sup>)

d ... diameter of the outermost rotor/stator (m)

With a diameter of 0.2 m and 4500 rpm a maximum shear rate of over 96000 (1/s) could be reached. To mimic the planned industrial process and to ensure cycle time a pump was used to push the premix through DB at flow rates from 6m<sup>3</sup>/h to 22.5m<sup>3</sup>/h. The finished setup is displayed in Figure 18.



Figure 18: Premix dispersion setup with the following machines: DA: powder dispersion unit, DB: inline dispersion unit, DC: beam mixer, Hopper: starch powder funnel, IBC: intermediary storage, pumpe before DB: rotary lobe pump for consistent duration during DB

During the trials not only the used starch powder was varied but also the settings for the machinery and durations of dispersion time and time between dispersions. The tool of DB was permanently switched during the second trial in July to the smaller slit size, as indicated in Table 18.

Since most premixes were not dispersed finely enough even after one run through DB, the premix was put through the DB unit a second time. This was done by filling the premix into 2 IBCs and refilling those into the tank again. This approach was chosen due to convenience and to make sure the whole premix passed the DB unit the same number of times. Some trials had a strainer built into the premix stream after the DB unit with a mesh size of 0.5 mm.

Immediate effects of thixotropic recovery and swelling of the fresh premix was measured by using a Brookfield DV2T viscometer at a constant shear rate of 6.8 1/s and occasionally at a lower shear rate due to detection limits. This will be indicated in the graphs in the Results- and the Appendix-section. A flow curve and further maturation data was measured of more matured Premix with an Anton Paar Rheometer MCR 72.

# 3. Cleaning Trial

To validate the cleanability of a plant using Premix a pilot plant was designed and built to facilitate trials. Initially, rheological concerns of matured premix were tested with a prototype of the pilot plant, which was later further expanded into the setup shown in Figure 19.



Figure 19: P&ID of the cleaning trial with highlighted steps

The setup contained a production loop (PL), including a production tank made of 1.4404 stainless steel (SS) with a surface roughness Ra <0.8µm equipped with an agitator, a Packo ZL-120 pump with a max. capacity of 172 L/min and 5 exchangeable pipe segments connected with Tri-clamp connections. The pump was regulated with a frequency converter between 5 and 100Hz and a power indicator (JI). The piping was controlled with a flow indicator (FI) and a pressure indicator (PI). All material was made of SS 1.4404 (316L) and had a surface roughness Ra < 0.8µm. This setup was the initial prototype,

before adding the cleaning extension. For this, the tank was further equipped with two spray balls of the type Tanko S30 by AWH.

The spray balls were connected to the cleaning tank, which had a connection to a DI water ring, providing freshly produced DI water with a conductivity of 5 - 40  $\mu$ S and at ~10 °C. The piping connected to the spray balls was equipped with a Packo ZL-120 pump with a max. capacity of 61 L/min at 1.6 bar. Furthermore, a PI and an FI were installed to display monitor the efficiency of the spray balls. The pump was switched out during the trials since a wrong pump was installed first, reaching only 0.6 bar.

A cleaning trial started by filling the production loop (seen in the first row of Figure 19) and looping the premix for at least 24 hours while stirring. The premix was then discarded by opening the drain valve. The starch residue was rinsed with 2x 100L of DI water by filling the cleaning tank (blue tank on the left in the 2<sup>nd</sup> row of Figure 19) and looping the water in the production loop only (same as the premix route). Then, 2kg of acidic cleaning medium were added to ~98L of DI water that was pre-filled into the cleaning tank and was stirred. It was then looped either only in the PL or in both the PL and the cleaning loop (CL), as seen in the 4<sup>th</sup> row of Figure 19 in green. After a set duration of looping, the cleaning solution was drained via the two drain valves. Afterwards, a rinsing cycle was run with 100L of water and looped through PL and CL in that order. A pH check was performed to see if another rinsing step with about 25L was necessary.

As a next step, ~98L of DI water was prepared and 2 kg of caustic cleaning medium was added and stirred. The solution was then looped with the same settings as the acidic cleaning step was performed. The solution was then drained and rinsed with 100L of DI water again. In a last step, ~100L of DI water was prepared and 0.5 kg of disinfectant was added, stirred and looped in both PL and CL for a set amount of time. The solution was then drained.

After each draining step, pictures were taken under illumination of a UV torch to check the cleaning.

# 4. Microbiology

The cleaning trial was further checked for microbiological remains. This was done by taking swab tests from the agitator, the inner wall of the product tank and the pipe

segments. Additionally, a UV torch was used to detect otherwise hard to see biofilm and other fluorescent spots not properly cleaned.

The swab tests were carried out with in inactivator solution dipped cotton swab. The inactivator solution contained various salts and enzymes designed to eliminate the effects of disinfectant added to samples. In this case formic acid was used as a disinfectant. After the swabs were taken, the cotton swab was applied to agarose and dextrose plates, which were also prepared with inactivator solution. The plates were then stored at 25°C and 30°C respectively to provide optimal growth environment for bacteria and fungi like yeast.

Each visible spot on the plates was counted as one colony forming unit (CFU). According to an internal standard, values of <10 CFU were considered acceptable. An example is shown in Figure 20. Each plate was kept for 7 days, after which the amount of CFU was determined.



Figure 20: Agarose and Dextrose plates with inactivator solution and applied swab test. The top plate has 2 spots, which would count as 2 CFU and the bottom plate has 1 spot, so 1 CFU (seen in red). Both would be indicated as <10 CFU according to standard.

# Results

# 1. Powder Analysis and Premix Production

The analysis of each pre-cooked starch powder used in the trials is described in Table 1 and shown in Figure 21. The January 2024 powder batch 1601 was not taken further into consideration for this thesis.

Table 1: Pre-cooked starch powder specifications used in the trials

	_	Particle size distribution (wt%)					
		>1.6	1.6 - 0.6	0.6 - 0.1	< 0.1	Water content	Density
Trial	Batch #	mm	mm	mm	mm	(wt%)	(kg/l)
Jan 25	1716-2	2.1	20	70.8	7.1	5.8	0.37±0.04
Jan 25	1716-3	0.7	17.4	74.3	7.6	5.8	0.33±0.01
Sep 24	1624	0.5	10.2	77.5	11.8	5.98	0.35±0.01
Sep 24	1607	0.4	8.4	79.9	11.3	5.75	0.40±0.02
Aug 24	1602	0.3	1.4	59.7	38.6	7.48	0.51±0.02
Jan 24	1601	0.1	4.2	82.6	13.1	5.70	0.39±0.00



Figure 21: Powder Analysis Data including particle size distribution and water content of the used starch batches

# 2. Viscosity Measurements and Premix Quality

The setup for all trials can be found in the Appendix-section. There, the machine parameters are given as well as the process steps and the number of lumps counted after 4L premix were sieved with a 0.8 mm mesh.

The powder induction time had no noticeable effect on the premix production quality in regards to the observed lumps after dispersion.

## a. July/August Trials

This trial was planned to test whether a very fine powder also works for premix production in an industrial setting, cutting transportation costs by increasing the density of the powder. A criterium for success was powder dispersion to a degree high enough that the number of lumps in 4L premix on a 0.8 mm mesh was close to 0. This is necessary to protect further machines like pumps from abrasion and sieves from clogging, requiring more maintenance as a result and therefore lost time and efficiency.

Table 18 shows that low amounts of lumps were only achieved with hours of swelling time and a 2<sup>nd</sup> DB pass. This led to the realization that finer powders, although providing more surface for water to swell more quickly, were apparently not as accessible as assumed. This triggered the following further trials

The viscosity measurements of the measured samples of Trial 2 are shown in Figure 22, Figure 23, and Figure 24.



Figure 22: Viscosity of first DB pass with 6m<sup>3</sup>/h flow rate through DB



Figure 23: Viscosity of first DB pass with 9m<sup>3</sup>/h flow rate through DB



Figure 24: Viscosity of first DB pass with 22.5m<sup>3</sup>/h flow rate through DB

The viscosity-plots will be further discussed in the Discussion-section. Whenever two graphs are displayed in one plot, the legend will explain whether the orange and/or grey graph describes the power law fitting with the actual time elapsed since passing DB (total time passed, see Figure 22) or if another corrective value was inserted to improve the fit of the model, as is shown in Figure 25.



Figure 25: Viscosity of first 4 minutes of dispersing with DA

Combined results for Trial one can be found in Figure 26.



Figure 26: Collected viscosity measurements for trial 1.

In Table 2 the parameters of the power law described in Equation 7, found on page 50, are shown for trial 1 of July.

Table 2: July/August trials fitted to power law models with the time correction factor fitting the model best (only 10second increments used)

	cf (s)	time passed (min)	А	В	R <sup>2</sup>
4 min DA	50	0	100,89	0,4000	0,9995
8 min DA	40	0	158,29	0,3528	0,9963
3000 rpm DB	20	5	254,43	0,2964	0,9989
2400 rpm DB	30	18,4	371,13	0,2460	0,9974
4500 rpm DB	0	50,25	723,99	0,1467	0,9834

In trial 3 only the first DB run was measured for viscosity and is shown in Figure 27.



Figure 27: Viscosity of first DB pass with 9m<sup>3</sup>/h flow rate through DB at 3600 rpm

# b. September Trials

In the September Trials the first two trials had technical difficulties, resulting in a more than 30 minute delay for DB1 passing in trial one and no viscosity data for trial two. In the other trials the measurements were not conducted as extensively, since new powders had to be validated after August's results and as many trials as possible should be conducted. One common denominator that was consistently measured was the viscosity of the 1<sup>st</sup> DB pass, called DB1 in the following graphs. A comparison of the runs can be found in Figure 28. The rate of increase in viscosity is noteworthy in this graph since it was the first time 2 powder qualities were measured on the same trial and had somewhat similar PSD. This lead to an early conclusion to get powder even coarser, reducing the fine particles as much as possible. Thus, the powder for the trials in January 2025 were ordered.



Figure 28: September trials viscosity data after passing DB for the first time

Table 3: Power law constants and R<sup>2</sup> of Sept trials of fitted curves after passing DB for the first time

	cf	А	В	R²
T1 DB1	20	170,72	0,4087	0,9973
T3 DB1	0	348,34	0,1756	0,9996
T4 DB1	20	165,37	0,2789	0,9977
T5 DB1	10	134,00	0,3997	0,9974
T6 DB1	20	154,16	0,3764	0,9998

In the September trial a rheometer was used in parallel to record a flow curve of trial 6 premix after one night of undisturbed swelling time. Additionally, the thixotropic behaviour at a shear rate of 6.8 1/s was recorded. The graphs can be found in Figure 29 and Figure 30.


Figure 29: September trial 6 rheometer measurements: double logarithmic flow curve with fitting



Figure 30: September trial 6 rheometer measurements: constant shear rate measurements at 6.8 1/s

## c. January 2025 Trials

The January trials had 2 powder qualities of even coarser starch granules delivered. In this trial the focus was again placed on processing the premix as quickly as possible to mimic

the industrial process closely. In parallel to the premix dispersion a rheometer was used again to record hysteresis loops to check thixotropy and to record flow curves of selected samples.

The viscosity was mostly checked after DB1 and after DB2. In this trial the temperature was varied as opposed to the previous ones since this trial happened in winter and there was no heating option for the DI water with formic acid (FA) mix. This resulted in the low temperatures of around 10 °C. For trials 4-6 the DI water/FA mix was heated through shear forces in the DA before dosing the powder, getting the temperature to levels of the previous trials. This confirmed the finding, that the thixotropic recovery was incomplete after passing through DB a second time. A clear example is provided in trial 2, displayed in Figure 31. This behaviour did not deviate once in the 8 trials.



Figure 31: Jan 25 Trial 2: Discrepancy of DB1 and DB2 with comparable time passing between passing through DB and the viscosity measurement (11 min and 11.5 min respectively)

It is noteworthy that the temperature of the DI water/FA mix increased in temperature due to being stored inside, thus slowly heating up to room temperature, which made trial 8 start at 15 °C already. Nevertheless, the effect of incomplete thixotropic recovery was still clearly observable, while heating the DI water/FA mix to 20 °C was sufficient to eliminate the effect again.

The following tables show the power law parameters of the fitting for the Jan 25 trials.

#### Table 4: Jan 25 trials: power law parameters for viscosity curves after the first passing of DB

	cf	А	В	R <sup>2</sup>
T1 DB1	-30	310,52	0,1302	0,9915
T2 DB1	10	385,01	0,1732	0,9971
T3 DB1	0	184,54	0,2632	0,9986
T4 DB1	0	253,5	0,2632	0,9978
T5 DB1	20	180,68	0,3155	0,9979
T6 DB1	20	209,29	0,2957	0,9999
T7 DB1	50	300,51	0,2404	0,9981
T8 DB1	60	251,74	0,2759	0,9995

Table 5: Jan 25 trials: power law parameters for viscosity curves after the second passing of DB

	cf	А	В	R <sup>2</sup>
T1 DB2	n.a.	n.a.	n.a.	n.a.
T2 DB2	-90	273,9	0,183	0,9997
T3 DB2	0	247,08	0,1817	0,9969
T4 DB2	20	242,79	0,2978	0,9977
T5 DB2	30	169,84	0,3339	0,9988
T6 DB2	0	372,45	0,217	0,9996
T7 DB2	30	262	0,2432	0,997
T8 DB2	10	358,95	0,2113	0,9958

The flow curves in the various trials from Jan 25 were measured after given time to swell over night. The rheometer was set to shear at 100 1/s for 60 seconds. After that period, the shear rate was reduced in logarithmic steps to 0.01 1/s. This was one of the ways to prove thixotropic behaviour of the premix, as can be seen in Figure 32, where the viscosity is reduced in the first minute of constant shear rate from 407 to 385 mPa\*s.

The flow curves in Figure 33 can be used as a reference to compare measurements of different shear rates.







Figure 33: Flow curves measured with a rheometer starting with high shear rate, reducing it over time



Figure 34: Hysteresis loop of the trials in Jan 25



Figure 35: Hysteresis loop of the trials in Jan 25



Figure 36: Hysteresis loop of the trials in Jan 25

## 3. Cleaning Trials

Due to the high amount of data collected in form of pictures a methodology to categorize the progress of tank, agitator and pipe segment cleaning is necessary to be implemented. For this, reference pictures will be assigned scores from 1 to 5 with 1 being a clean surface with hardly any spots under UV light, 3 being visibly clean (best score achievable without UV light check) and 5 being not clean. The reference images can be found in the Appendix section below. This allows for a more efficient report of each step of the cleaning trials performed.

## a. Cleaning Trial 1

The first cleaning trial (CT) 1 was conducted starting September 16<sup>th</sup>. Premix was filled into the tank and looped through the PL for 1 week. Each day, one pipe segment was swapped for a fresh one and set aside to dry and monitor. This showed that dry premix formed a plastic-like, thin and brittle layer on the pipe. This layer curled inwards and was thus easily removed. Wetting the layer did not, however, return this retrograded starch foil

gelly again. A certain crystallinity was noticeable by kneading the layer under running water between two fingers.

The pipe segments and the inside surface of the tank were swabbed daily to check for microbial issues, which always resulted in the acceptable score of <10 CFU. Since there were no findings, the swab tests in further trials were performed only sporadically on certain noticeable spots.



Figure 37: CT1 pipe segment with dried-up premix, showing retrogradation of the premix and a likely semicrystalline layer of starch

After 10 days, the cleaning was performed according to Table 6. For this trial the cleaning solution was prepared directly in the process tank, since the cleaning loop had not been built yet.

#### Table 6: CT1 with 2 cleaning steps performed

CT1 26.9.2024											
	Duration PL Speed PL Duration CL Speed CL CFU c										
Steps	(min)	(l/min)	(min)	(l/min)	Swab						
Rinse	20	71	n.a.	n.a.	<10						
Caustic	20	71	n.a.	n.a.	<10						

This initial trial served as a learning trial, where no UV torch had been procured yet. Thus, the scores for this trial could only reach 3 at best. The scores can be found in Table 7.

	Scores for cleaning steps						
Equipment	Rinse	Rinse	Acidic	Rinse	Caustic	Rinse	Disinfection
Tank	4	n.a.	n.a.	n.a.	3	n.a.	n.a.
Pipe segment	4	n.a.	n.a.	n.a.	3	n.a.	n.a.

## b. Cleaning Trial 2

This cleaning trial was a repeat of CT1 with additional swab tests being taken from the inside of the tank, O-rings, flanges and tri-clamp connections. These tests also showed no CFU >10. For this trial the UV-torch arrived and was used to analyze the cleaning progress more accurately. The cleaning steps can be found in Table 8 and the scores for this trial in Table 9. No pictures of the first rinse were taken. At this stage the equipment is usually still dirty and usually has a score of 5.

#### Table 8: CT2 cleaning steps performed

CT2 16.10.										
	Duration PL	Speed PL	Duration CL	Speed CL	CFU of					
Steps	(min)	(l/min)	(min)	(l/min)	Swab					
Rinse	30	71	n.a.	n.a.	<10					
Caustic	30	71	n.a.	n.a.	<10					
$1\% H_2O_2$	10	71	n.a.	n.a.	n.a.					

#### Table 9: CT2 score card

		Scores for cleaning step						
Equipment	Rinse	Rinse	Acidic	Rinse	Caustic	Rinse	Disinfection	
Tank	?	4	n.a.	n.a.	2	2	(1)	
Pipe segment	?	3	n.a.	n.a.	2	2	(1)	

Since a delay in delivery did not allow to use the proposed disinfectant, an initial trial with 1% H<sub>2</sub>O<sub>2</sub> was performed, which showed promising results, as the spots visible under UV light in the pipe segments were heavily reduced. This was done to disinfect the setup, since no trials could be prepared for the rest of the year due to the upgrade of the setup implementing the spray balls and the cleaning tank + cleaning loop.

## c. Cleaning trial 3

After seeing good success with using no acid but not reaching a score of 1, a trial with only 5 minutes of cleaning time was done, with the cleaning steps described in and the scores in Table 11.

Table 10 and the scores in Table 11.

СТЗ 10.1.											
Steps	Duration PL (min)	Speed PL (I/min)	Duration CL (min)	Speed CL (I/min)	CFU of Swab						
Rinse x2	5	131	n.a.	n.a.	n.a.						
Acidic	5	131	n.a.	n.a.	n.a.						
Rinse	5	131	n.a.	n.a.	n.a.						
Caustic	5	131	n.a.	n.a.	n.a.						
Rinse	5	131	n.a.	n.a.	n.a.						

Table 10: CT3 cleaning steps

#### Table 11: CT3 score card

			Scores for cleaning step					
Trial	Equipment	Rinse	Rinse	Acidic	Rinse	Caustic	Rinse	Disinfection
	Tank	?	4	4	4	2	2	n.a.
CT3								
	Pipe segment	?	4	4	3	2	2	n.a.

This trial was the first where the cleaning solution was prepared in the cleaning tank and dosed via spray balls, adding an additional mechanical component to the cleaning process. Since the wrong pump was prepared for the setup, the pressure for the spray balls was lower than the supplier required for the spray balls, reaching only 0.6 bar instead of 2.

The results of using acid did not show any additional cleaning effect compared to a simple rinsing step with DI water. Since the duration was low to better see the effects of each step, the next trial focused on the effects of acid by increasing the rinsing duration.

## d. Cleaning Trial 4

In CT4 the duration of the initial rinsing steps was increased to see if acid has a similar effect on the tank and pipe segment surface as the caustic cleaning step. The process steps are shown in Table 12 and the scores in

Table 13.

CT4 14.1.											
	Duration PL Speed PL Duration CL Speed CL										
Steps	(min)	(l/min)	(min)	(l/min)	CFU of Swab						
Rinse	20	172	n.a.	n.a.	n.a.						
Rinse	20	172	n.a.	n.a.	n.a.						
Acidic	5	172	n.a.	n.a.	n.a.						
Rinse	5	172	n.a.	n.a.	n.a.						
Caustic	5	172	n.a.	n.a.	n.a.						
Rinse	5	172	n.a.	n.a.	n.a.						

Table 12: CT4 cleaning steps

#### Table 13: CT4 score card

			Scores for cleaning step						
Trial	Equipment	Rinse	Rinse	Acidic	Rinse	Caustic	Rinse	Disinfection	
	tank	5	3	3	3	2	2	n.a.	
CT4	pipe								
	segment	4	3	3	3	2	2	n.a.	

As the score card shows, the acidic cleaning step did not result in the same outcome as the caustic step and did not improve the cleaning progress any more than a rinsing step. To stay coherent, the step was not excluded from further cleaning trials yet, since a score of 1 had not been reached. As soon as that score is reached with a cleaning process, optimization towards eliminating acid from the cleaning process will be experimented with.

## e. Cleaning Trial 5

In this trial the cleaning time was doubled to understand if longer duration could lead to fewer spots visible under UV light. The process steps are shown in Table 14 and the score card in Table 15.

CT5 17.1.									
		Speed		Speed					
	Duration	PL	Duration	CL					
Steps	PL (min)	(l/min)	CL (min)	(l/min)	CFU of Swab				
Rinse	10	172	n.a.	n.a.	n.a.				
Rinse	10	172	n.a.	n.a.	n.a.				
Acidic	10	172	n.a.	n.a.	n.a.				
Rinse	10	172	n.a.	n.a.	n.a.				
Caustic	10	172	n.a.	n.a.	n.a.				
Rinse	10	172	n.a.	n.a.	n.a.				

#### Table 14: CT5 process steps

#### Table 15: CT5 score card

			-	Scor	es for c	cleaning s	step	
Trial	Equipment	Rinse	Rinse	Acidic	Rinse	Caustic	Rinse	Disinfection
	tank	5	4	4	4	2	2	n.a.
CT5	pipe							
	segment	3	3	3	3	2	2	n.a.

In this trial the score of 1 had not been reached either, since the surfaces showed still too many spots visible under UV light. For all the cleaning cycles the cleaning loop was not used so far. This was not done since there would be no effect on the pipe segments by employing the cleaning loop and because the wrong pump was installed to reach the specified pressure for the spray balls. This could be updated after CT5, since the preparation of another pump was finalized during the trial.

### f. Cleaning Trial 6

For this cleaning trial the same pump was installed for the CL as for the PL. Therefore, the cleaning process made use the CL for this trial. To maximize efficiency of the spray balls a tank should always be kept empty to allow the pressurized cleaning solution to hit the bottom of tank without a protective level of solution being there. For that reason, about 70l of cleaning solution was discarded after finishing the PL cycle. Only then the valves were prepared for the CL and the process was started. The process steps are shown in Table 16 and the score card in Table 17.

			CT6 2	24.1.		
			Speed		Speed	CFU
	Duration		PL	Duration	CL	of
Steps	PL (min)		(l/min)	CL (min)	(l/min)	Swab
Rinse		5	172	10	n.a.	n.a.
Rinse		5	172	10	n.a.	n.a.
Acidic		5	172	10	62	n.a.
Rinse		2	172	3	62	n.a.
Caustic		5	172	10	62	n.a.
Rinse		2	172	3	62	n.a.

#### Table 16: CT6 process steps

#### Table 17: CT6 score card

				Scor	es for c	cleaning s	step	
Trial	Equipment	Rinse	Rinse	Acidic	Rinse	Caustic	Rinse	Disinfection
	tank	5	4	3	3	2	2	1
CT6	pipe							
	segment	4	4	3	3	3	3	1

In this trial one of the unused and dirty pipe segments was used instead of the right-most segment that was used so far in all the other trials. The pipe segment was significantly dirtier than all the other pipe segments, which were just in contact with the starch premix. This was done to check how well the cleaning process could handle remains in the loop that would sit there over holidays or other long breaks of production. Since the proposed disinfectant arrived as well, the peracetic acid was used as a disinfection step in this trial showing great results. Both tank and pipe segment were sufficiently cleaned by this step.

# Discussion

## 1. Starch premix - how powder specifications affect premix viscosity

### a. Quality by amount of lumps

Comparing the results based only on the data measured with the viscometer is challenging. The variables between each trial differ greatly sometimes yet cannot be attributed to exact quantitative effects. This leaves only qualitative comparisons, if only looking at the resulting viscosities.

The premix quality was not only determined by the resulting viscosity behaviour, but also by the number of lumps after dispersion, which creates a much clearer picture. Considering the number of lumps, the premix created from batch 1624 was clearly superior to all the other batches. This holds true even after taking varying swelling times between the premix preparations into consideration, which had a vey positive effect on the dispersibility of lumps and agglomerates. The results were equally conclusive for the worst premix, which was prepared with batch 1602. There, the powder quality was a lot finer than with all the other batches. Since all the lumps found on the sieve were a lot bigger than most of the powder particles and swelling was unlikely to increase the size of the particles to that extent, agglomeration is suspected to have caused the bad results.

Small particles are known to have a way higher surface-to-volume-ratio, increasing the weight of electrostatic effects on the surface like the zeta potential, which describes the sum of charges in a thin electrochemical double-layer around the surface materials. While it was not measured for the powder, agglomeration can be caused by these electrostatic effects. If agglomeration were to take place before water could wet all the particles, insufficiently wetted powder would be enclosed in agglomerates and not swell to the full extent.

The lumps observed were mostly soft and gelly, passing through sieves easily by applying pressure, supporting at least a minimum of wetting happening, thus softening the agglomerates.

Dispersion of the smaller and likely harder particles resulted in worse results, which caused future powders to have only limited fines. Coarser powder was more expensive to ship due to a decrease in density, so it was attempted to find a reasonable compromise, increasing the coarse particles gradually.

The effect of coarser powder giving better results than finer powders held true for the September trial. The cause of this effect aside of the hypothesis of agglomeration of fines could also be explained by having higher forces applied to the edges of finer granules, breaking them apart and improving the accessibility to the granule for water. Fine particles might also be the reason for the breaking of bigger granules, as they are accelerated in the dispersion devices and could act as bullets, destroying bigger particles. Small particles are known to be harder than larger ones due to stochastically having a lower amount of material defects.

The positive effect of using coarser powder was no longer seen with batches 1716-2 and 1716-3. There, more lumps found on the sieve compared to batch 1624, which would support a hypothesis that fines might help with the dispersion of large particles. It would not explain why more gelly agglomerates were found on the sieve, though. To answer this question, further testing would be needed with very distinct particle sizes used in premix preparation to assign effects on dispersion quality to each particle size group and their combinations. With the data available, validation of the results with repeating trials should be done and a certain split between fine and coarse particles could be assumed as an optimum for this process.

Another effect could be observed with batches 1716-2 and 1716-3, which will be called "deshearing". The premix showed a distinct drop in absolute dynamic viscosity after passing through DB for a second time, which will be discussed in the next chapter.

## b. Premix viscosity

The viscosity of the sieved premix was measured as closely to the passing of DB as the process steps allowed. This caused a delay when one sample was still measured while other samples were already passing DB. Due to this, the time when the swelling process of the powder started was recorded as well as the time of passing through DB to then add the passed time when plotting the viscosity against it to account for thixotropic recovery. This did not help with finding a suitable model to describe the maturation and swelling of the premix, though. The measured data resembled a root function most closely, as can be seen in Figure 38.



Figure 38: July/August, Trial 2, 6m<sup>3</sup>/h pump speed, 4500 rpm DB, 3000 rpm DA

There was nothing to gain by adding the passed time as opposed to using the relative starting time of the measurement, which is what was done in Figure 38. The model would require the exact correction  $c_f$  according to Equation 7, which was shown in the result section.

Equation 7: Mathematical power law model describing the behaviour of freshly dispersed premix

$$\eta = A \cdot (x + c_f)^E$$

η ... dynamic viscosity (mPa\*s)

A, B ... constants describing the premix

cf ... correction value of the passed time (s)

Figure 39 shows the more accurate but less helpful plot.



Figure 39: Total time passed since premix passed DB added

Adding the total time elapsed is not a more accurate model regarding  $R^2$ .

As described in Figure 9 page 13 the used starch powder underwent a gelatinization process followed by complete retrogradation of amylose and amylopectin. The dispersion process aims to break the intermolecular bonds and the restored semi-crystallinity by adding water and shear stress. This causes the powder to swell again, which takes time. This total time is what was added as cr in Figure 39, resulting in a very bad fit. The effect appears to be so much smaller in comparison to the thixotropic recovery that including this effect in the model seems to make little sense. The thixotropic recovery triggered after passing DA or DB was anticipated for the samples. What was missing was the thixotropic effect of taking the sample, which apparently played a way bigger role in finding a model than previously thought. Otherwise, samples that were resting after passing through DB for 50 minutes should not show a recovery curve like the ones shown in Figure 38 but rather a decrease in viscosity, since recovery should have been quite advanced at that stage.

This means that while very fresh samples passing through DB would show a lower value for A and a higher value for B the effect would only be visible if the recovery was slower than the shear rate introduced by pouring the sample into the cup for measurement. This explains why the best models seen in chapter 2 of the results mostly used corrective value  $c_f$  of about 20 to 50, aligning with the time it took to fill the sample into the cup and starting the measurement. If the swelling effect would also be noticeable in the plotted data, it seems likely that the effect is small enough that another mathematical model cannot be detected looking at the residual plot or that the model is similar enough that it could be integrated well with the adaptions to A, B and  $c_r$ . It should be considered that A would be affected most notably by longer swelling time, as this value could be interpreted as the viscosity at the beginning of the process. A quantification of this effect could potentially be tested with a rheometer at high shear rate after a very short but thorough dispersion process. At high shear rate (~1000 s<sup>-1</sup>) the thixotropic recovery effect from the extreme shear rates present at DB and DA would not take long and the continuous rise due to swelling might be observable.

One observed behaviour of the premix was already briefly described in the previous chapter. Deshearing was observed in the January trial with both batches whenever the DI water/FA mix was not heated to at least 20°C. This effect could not be observed at the other trials since the mix was transported at ambient temperature to the trial facilities and the first trials were carried out in summer. Since at industrial scale the premix would have been prepared with ~10°C water, this learning proved to be essential.

A possible explanation for deshearing might be a more rigid granule at colder temperature which could potentially be destroyed by the high shear forces applied by DB on a molecular level. Breaking off chains would limit the ability to closely entangle again, which is the cause for retrogradation and generally the thixotropic behaviour of the starch premix. This would need to be confirmed with methods like soft mass spectrometry (MALDI or ESI for example) for example comparing the molecular weight before and after passing DB several times.

Besides processability, which was discussed in section 1.a. of the discussion, the premix should also reach a high viscosity in a short amount of time. Looking at the premixes prepared throughout the trial, the most complete data exists after passing through DB for the first time. The parameter B of the power law model applied in the tables found in section 2 of the results shows how quickly the viscosity rises. Parameter A shows to be influenced by the swelling time that was granted to the premix and is more difficult to compare. Since the quick rise in viscosity is responsible for the stabilization of a suspension and keeping particles afloat, parameter B will suffice in rating the premixes prepared. This method shows analogous results as the quality determined by the number of lumps found after sieving. Batch number 1624 shows on average a much higher recovery rate than the other batches. For industrial validation a powder similar to batch 1624 should be used to achieve the best results.

## 2. Cleaning trials

The cleaning trials were used to accelerate the optimization process of the industrial process by outlining a clear path towards the first steps, minimizing the risk of failed validations. The proposed cleaning process involved the use of hot chemicals, which was not tried in the trials due to the lack of heating equipment available. The supplier of the CIP chemicals mentioned that the chemicals are designed for a certain temperature range which was not applied in either cleaning trial. This means that the results might differ substantially when hot cleaning medium is used for the process, which will be tried in the future.

Presently, the cleaning trials were still successful, as the CT6 showed. The use of disinfectant is strongly recommended as the effect was immediately noticeable when checked with UV light. The optimization process should also look to exclude the acidic step from further processes as no additional effect could be observed throughout the trials compared to simple rinses with water. This would save not only costs for chemicals but also reduce water consumption as both one rinsing step and one chemical cleaning step could be avoided. Another cost decreasing step would be to lower the proposed temperature of the chemicals, which should be discussed with the supplier of the chemicals first though, to see if other caustic cleaning chemicals might be available designed for use at lower temperatures.

# Conclusion

The results show how powder quality combined with dispersion temperature not only affect how finely dispersed starch will be after a set amount of time, but also how quickly the rise in viscosity takes place. This behaviour can help finished products in regard to stabilizing further particles by preventing segregation. Further tests with similar powders could help with finding an optimal powder for the proposed dispersion process. Also, powder from another supplier with similar particle size distribution but a different precooking and milling process could give insights on how the production of the powder affects the resulting premix quality.

The cleaning trials resulted in sufficiently cleaned equipment as long as a disinfection step was added after the caustic cleaning. This needs to be validated on industrial scale. Furthermore, tests, where peracetic acid is already dosed into the caustic cleaning solution might reduce the amount of steps, which can also be achieved by cutting the acidic cleaning step. Further validation trials are required for these hypotheses.

# Appendix

## 1. Trial Data for Premix Dispersion

Table 18: Machine settings, procedure and number of lumps for Trial 1 in July/August

TRIAL Batch 1602, 31.07.2024 Image: Figure Fi		JULY/AUGUST										
131.07.2024 $\Box At$ $\Box Parces Step$ $\Delta t$ $Speed$ $Power$ $\Box B$ $speed DB$ $DC$ $T$ 1Process Step $\Delta t$ $Speed$ Power $Speed$ $Power$ $Volumeflow$ $Speed$ $Power$ $Power$ $T$ 1 $h:mm$ $rpm$ $kW$ $rpm$ $kW$ $rpm$ $kW$ $m^3/h$ $rpm$ $kW$ $°C$ $Iumps$ 08:45Filling of tank Powder $rpm$ $kW$ $rpm$ $kW$ $m^3/h$ $rpm$ $kW$ $°C$ $Iumps$ 09:30Induction $50$ $3000$ $50$ $C$ $C$ $C$ $C$ $C$ $C$ 09:34Amin disperion $240$ $3000$ $50$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ 09:34Amin dispersion $240$ $3000$ $50$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ 09:35Amin dispersion $240$ $3000$ $50$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ 09:36Amin dispersion $240$ $3000$ $50$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ $C$ 09:37Ist pass DB $C$ 10:20Varying DB rpm $C$ <td>TRIAL</td> <td>Batch 1602,</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td>Pump</td> <td></td> <td></td> <td></td> <td></td>	TRIAL	Batch 1602,						Pump				
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	1	31.07.2024		D	A	D	В	speed DB	C	C		
hh:mm s rpm kW rpm kW m³/h rpm kW °C lumps   08:45 Filling of tank Powder Powder 26,6 <td>Time</td> <td>Process Step</td> <td>Δt</td> <td>Speed</td> <td>Power</td> <td>Speed</td> <td>Power</td> <td>Volumeflow</td> <td>Speed</td> <td>Power</td> <td>Т</td> <td></td>	Time	Process Step	Δt	Speed	Power	Speed	Power	Volumeflow	Speed	Power	Т	
08:45 Filling of tank Powder 26,6   09:30 Induction 50 3000 50 700 1,1 27,5   09:36 4 min disperion 240 3000 55 2000 14 30 >100   09:40 8 min dispersion 240 3000 50 2000 14,5 32,1 >100   10:20 1st pass DB 3000 20 22,5 500 0,5 33,2 >50   10:20 varying DB rpm 2400 12 22,5 33,2 >100   10:20 1800 7.5 22.5 33,2 >100	hh:mm		S	rpm	kW	rpm	kW	m³/h	rpm	kW	°C	lumps
Powder 700 1,1 27,5   09:30 Induction 50 3000 50 700 1,1 27,5   09:36 4 min disperion 240 3000 55 2000 14 30 >100   09:40 8 min dispersion 240 3000 50 2000 14,5 32,1 >100   10:20 1st pass DB 3000 50 2000 14,5 32,2 >50   10:20 varying DB rpm 2400 12 22,5 33,2 >100   10:20 1800 7.5 22.5 33,2 >100	08:45	Filling of tank									26,6	
09:30 Induction 50 3000 50 700 1,1 27,5   09:36 4 min disperion 240 3000 55 2000 14 30 >100   09:40 8 min dispersion 240 3000 50 2000 14,5 32,1 >100   10:20 1st pass DB 3000 20 22,5 500 0,5 33,2 >50   10:20 varying DB rpm 2400 12 22,5 33,2 >100   10:20 1800 7.5 22.5 33,2 >100		Powder										
09:36 4 min disperion 240 3000 55 2000 14 30 >100   09:40 8 min dispersion 240 3000 50 2000 14,5 32,1 >100   10:20 1st pass DB 3000 20 22,5 500 0,5 33,2 >50   10:20 varying DB rpm 2400 12 22,5 33,2 >100   10:20 1800 7.5 22.5 33,2 >100	09:30	Induction	50	3000	50				700	1,1	27,5	
09:40 8 min dispersion 240 3000 50 2000 14,5 32,1 >100   10:20 1st pass DB 3000 20 22,5 500 0,5 33,2 >50   10:20 varying DB rpm 2400 12 22,5 33,2 >100   10:20 1800 7.5 22.5 33,2 >100	09:36	4 min disperion	240	3000	55				2000	14	30	>100
10:20 1st pass DB30002022,55000,533,2>5010:20 varying DB rpm24001222,533,2>10010:2018007.522.533.2>100	09:40	8 min dispersion	240	3000	50				2000	14,5	32,1	>100
10:20 varying DB rpm 2400 12 22,5 33,2 >100   10:20 1800 7.5 22.5 33.2 >100	10:20	1st pass DB				3000	20	22,5	500	0,5	33,2	>50
10.20 1800 7.5 22.5 33.2 >100	10:20	varying DB rpm				2400	12	22,5			33,2	>100
10.20 1000 7,5 22,5 55,2 7100	10:20					1800	7,5	22,5			33,2	>100
10:21 3600 30 22,5 33,2 >100	10:21					3600	30	22,5			33,2	>100
4500 45 22,5 33,2 >50						4500	45	22,5			33,2	>50

TRIAL	Batch 1602,						Pump				
2	31.07.2024		D	A	D	B	speed DB	C	C		
Time	Process Step	Δt	Speed	Power	Speed	Power	Volumeflow	Speed	Power	Т	
hh:mm		S	rpm	kW	rpm	kW	m³/h	rpm	kW	°C	lumps
11:00	Filling of tank										
	Powder										
11:20	Induction	152	3000	50				900	2,2		
11:33	4 min disperion	240	3000	45				1800	15	30,7	>200
11:45	8 min dispersion	240	3000	45				1800	13	32,9	>200
13:17	1st pass DB				4500	20	6			33,3	20
	2nd pass over										
	DB				4500	27	9				30
					4500	45	22,5				>50
15:00	Stator to 1mm										
					4500	22	9				4
					4500	36	18				6
					4500	43	22,5				15

DA: powder dispersion unit

DB: inline Dispersion unit

TRIAL	Batch 1602,						Pump				
3	01.08.2024		D	A	D	В	speed DB	D	C		lumps
Time	Process Step	∆t	Speed	Power	Speed	Power	Volumeflow	Speed	Power	Т	
hh:mm		S	rpm	kW	rpm	kW	m³/h	rpm	kW	°C	
08:20	Filling of tank									26,7	
	Powder										
09:00	Induction	75	3000	45				800	1,7		
09:15	4 min dispersion	240	3000	45				1900	14,5	30	>200
09:22	1st pass DB				4500	26	9	700	1,1	32,5	>200
					3600	16	9	700	1,1	32,5	>200
					4500	28	12	700	1,1	32,5	>100
					3600	20	12	700	1,1	32,5	>100
					4500	37	15	700	1,1	32,5	>100
					3600	24	15	700	1,1	32,5	>100
					4500	39	18	700	1,1	32,5	>100
					3600	28	18	700	1,1	32,5	>100
					3600	22	9			32,5	>100
09:50	2nd pass DB	-			3600	37,5	9				30

Table 19: Machine settings, procedure and number of lumps for Trial 2 in September

	SEPTEMBER	_									
TRIAL	Batch 1624						Pump				
1	03.09.2024		D	A	D	B	speed DB	D	C		
Time	Process Step	Δt	Speed	Power	Speed	Power	Volumeflow	Speed	Power	Т	
hh:mm		S	rpm	kW	rpm	kW	m³/h	rpm	kW	°C	lumps
	Filling of tank									25,6	
	Powder										
09:39	Induction	82	3000	44				900	1,9	27,6	
09:44	4 min disperion	240	3000	46,5				1800	11,6	29,4	>100
10:05	8 min dispersion	240	3000	48				1800	12,1	31,4	>100
10:13	1st pass DB				4500	39	22,5			33,8	3
11:07	2nd pass DB				4500	37,6	22,5			33,8	0

TRIAL	Batch 1624						Pump				
2	03.09.2024		D	A	D	B	speed DB	D	C		
Time	Process Step	Δt	Speed	Power	Speed	Power	Volumeflow	Speed	Power	Т	
hh:mm		S	rpm	kW	rpm	kW	m³/h	rpm	kW	°C	lumps
11:30	Filling of tank									25,9	
	Powder										
11:38	Induction	88	3000	44				900	2,1	27,5	
11:41	8 min dispersion	480	3000	48				1800	13,3	31,2	>100
11:51	1st pass DB				4500	35,7	22,5			32,4	8
13:10	2nd pass DB	_			4500	35,4	22,5			33,3	0

DA: powder dispersion unit

DB: inline Dispersion unit

DA: powder dispersion unit

DB: inline Dispersion unit

Table 20: Machine settings, procedure and number of lumps for Trial 3 in Jan 2025

	January 2025	_									
TRIAL	Batch 1716-2						Pump				
1	20.01.2025		D	A	C	В	speed DB	D	C		
Time	Process Step	Δt	Speed	Power	Speed	Power	Volumeflow	Speed	Power	Т	
hh:mm	·	S	rpm	kW	rpm	kW	m³/h	rpm	kW	°C	lumps
08:36	Filling of tank										. '
09:00	Powder Induction	84	3000	48				700	1.1	9.8	
09:13	4 min disperion	240	3000	56				1700	10	-,-	>100
09:21	8 min dispersion	240	3000	58				1700	13	16.6	>50
09:30	1st pass DB				4500	48	22.5	600	1.6	19	20
	2nd pass DB +						,=		_,.		
10:02	filter				4500	49	22,5			20,2	2
							,	-		- /	
TRIAL	Batch 1716-2						Pump				
2	20.01.2025		D	A	D	B	speed DB	D	C		
Time	Process Step	Δt	Speed	Power	Speed	Power	Volumeflow	Speed	Power	т	
hh:mm		5	rpm	kW	rpm	kW	m <sup>3</sup> /h	rpm	kW	°C	lumps
10.35	Filling of tank						,			10	lamps
10.33	Powder Induction	86	3000	43				700	1	10	
10.10	8 min dispersion	480	3000	56				1700	12	16	>100
10.50	1st nass DB + filter	400	5000	50	4500	48	22.5	600	1.6	18	>50
10.50	2nd nass DB +				4500	40	22,5	000	1,0	10	200
11:26	filter				4500	49	22.5			19.5	2
									-		
TRIAL	Batch 1716-2						Pump				
3	20.01.2025		D	A	C	B	speed DB	D	C		
Time	Process Step	Δt	Speed	Power	Speed	Power	Volumeflow	Speed	Power	т	
hh:mm		S	rpm	kW	rpm	kW	m <sup>3</sup> /h	rpm	kW	°C	lumps
11:43	Filling of tank		1-							9.4	
12:50	Powder Induction	84	3000	46				700	1.3	11.5	
12:52	8 min dispersion	480	3000	56				1700	12	15.6	n.a.
13:03	1st pass DB				4500	48	22.5	600	1.6	17.5	>50
	2nd pass DB +						/-		_,-		
13:20	filter				4500	49	22,5			18,8	4
	-										
TRIAL	Batch 1716-2						Pump				
TRIAL 4	Batch 1716-2 21.01.2025		D	A	C	B	Pump speed DB	C	C		
TRIAL 4 Time	Batch 1716-2 21.01.2025 Process Step	Δt	D Speed	A Power	C Speed	B Power	Pump speed DB Volumeflow	D Speed	C Power	т	
TRIAL 4 Time hh:mm	Batch 1716-2 21.01.2025 Process Step	∆t s	D Speed rpm	A Power kW	D Speed rpm	PB Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h	D Speed rpm	PC Power kW	T °C	lumps
TRIAL 4 Time hh:mm 08:00	Batch 1716-2 21.01.2025 Process Step	∆t s	D Speed rpm	0A Power kW	C Speed rpm	B Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h	D Speed rpm	OC Power kW	T ℃ 24	lumps
TRIAL 4 Time <u>hh:mm</u> 08:00 08:05	Batch 1716-2 21.01.2025 Process Step Filling of tank Powder Induction	Δt s	D Speed rpm 3000	PA Power kW 46	C Speed rpm	B Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h	D Speed rpm 700	Power kW	T °C 24 25.7	lumps
TRIAL 4 Time hh:mm 08:00 08:05 08:09	Batch 1716-2 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion	Δt s 126 480	D Speed rpm 3000 3000	Power kW 46	C Speed rpm	B Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h	D Speed rpm 700 1700	POWER kW 1,3 12	T °C 24 25,7 29.6	lumps
TRIAL 4 Time hh:mm 08:00 08:05 08:09 08:20	Batch 1716-2 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB	Δt s 126 480	D Speed rpm 3000 3000	PA Power kW 46 56	C Speed rpm 4500	Power kW 48	Pump speed DB Volumeflow m <sup>3</sup> /h	D Speed rpm 700 1700 600	C Power kW 1,3 12 1 6	T ℃ 24 25,7 29,6 30	lumps
TRIAL 4 Time hh:mm 08:00 08:05 08:09 08:20	Batch 1716-2 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB +	Δt s 126 480	D Speed rpm 3000 3000	Power kW 46 56	D Speed rpm 4500	PB Power <u>kW</u> 48	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5	D Speed rpm 700 1700 600	Power kW 1,3 12 1,6	T °C 24 25,7 29,6 30	lumps >100 30
TRIAL 4 Time hh:mm 08:00 08:05 08:09 08:20 08:36	Batch 1716-2 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter	Δt s 126 480	D Speed rpm 3000 3000	PA Power kW 46 56	C Speed rpm 4500 4500	PB Power kW 48 48	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5	D Speed rpm 700 1700 600	0C Power kW 1,3 12 1,6	T ℃ 24 25,7 29,6 30 31.8	lumps >100 30
TRIAL 4 Time hh:mm 08:00 08:05 08:09 08:20 08:36	Batch 1716-2 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter	Δt s 126 480	D Speed rpm 3000 3000	Power kW 46 56	C Speed rpm 4500 4500	Power kW 48 49	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5 22,5	D Speed rpm 700 1700 600	PC Power kW 1,3 12 1,6	T ℃ 24 25,7 29,6 30 31,8	lumps >100 30 12

DB: inline Dispersion unit

TRIAL	Batch 1716-3						Pump				
5	21.01.2025		D	A	C	ЭB	speed DB	C	C		
Time	Process Step	Δt	Speed	Power	Speed	Power	Volumeflow	Speed	Power	Т	
hh:mm		S	rpm	kW	rpm	kW	m³/h	rpm	kW	°C	lumps
08:57	Filling of tank									24	
09:15	Powder Induction	145	3000	46				700	1	26	
09:17	8 min dispersion	480	3000	56				1700	12	29,5	>100
09:26	1st pass DB				4500	48	22,5	600	1,6	30,5	>100
	2nd pass DB +										
09:48	filter				4500	49	22,5			31,7	30
TRIAL	Batch 1716-3						Pump				
6	21.01.2025		D	A	C	)B	speed DB	C	C		
Time	Process Step	Δt	Speed	Power	Speed	Power	Volumeflow	Speed	Power	Т	
hh:mm		S	rpm	kW	rpm	kW	m³/h	rpm	kW	°C	lumps
	Filling of tank									20	
10:25	Powder Induction	130	3000	46				800	1,7	21,6	
10:27	8 min dispersion	480	3000	56				1700	12	25,5	>200
10:49	1st pass DB				4500	48	22,5	600	1,6	27,3	>100
	2nd pass DB +										
11:09	filter	-			4500	49	22,5	-		28,2	10
TRIAL	Batch 1716-3						Pump				
TRIAL 7	Batch 1716-3 21.01.2025		D	A	C	ЭB	Pump speed DB	C	)C		
TRIAL 7 Time	Batch 1716-3 21.01.2025 Process Step	Δt	D Speed	A Power	D Speed	)B Power	Pump speed DB Volumeflow	D Speed	OC Power	Т	
TRIAL 7 Time hh:mm	Batch 1716-3 21.01.2025 Process Step	∆t s	D Speed rpm	0A Power kW	D Speed rpm	)B Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h	C Speed rpm	OC Power kW	T °C	lumps
TRIAL 7 Time hh:mm 12:00	Batch 1716-3 21.01.2025 Process Step Filling of tank	∆t s	D Speed rpm	A Power kW	C Speed rpm	B Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h	D Speed rpm	DC Power kW	T °C 14,8	lumps
TRIAL 7 Time hh:mm 12:00 12:42	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction	Δt s	D Speed rpm 3000	A Power kW 46	D Speed rpm	9B Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h	D Speed rpm 800	DC Power kW 1,7	T °C 14,8 17,3	lumps
TRIAL 7 Time hh:mm 12:00 12:42 12:44	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion	Δt s 127 480	D Speed rpm 3000 3000	Power kW 46 56	D Speed rpm	Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h	D Speed rpm 800 1700	DC Power kW 1,7 12	T °C 14,8 17,3 21	lumps
TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB	Δt s 127 480	D Speed rpm 3000 3000	PA Power kW 46 56	C Speed rpm 4500	Power kW 48	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5	C Speed rpm 800 1700 600	DC Power kW 1,7 12 1,6	T °C 14,8 17,3 21 22,7	lumps >100 >100
TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB +	Δt s 127 480	D Speed rpm 3000 3000	Power kW 46 56	C Speed rpm 4500	Power kW 48	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5	C Speed rpm 800 1700 600	DC Power kW 1,7 12 1,6	T °C 14,8 17,3 21 22,7	lumps >100 >100
TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56 13:10	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter	Δt s 127 480	D Speed rpm 3000 3000	PA Power kW 46 56	C Speed rpm 4500 4500	Power kW 48 49	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5 22,5	D Speed rpm 800 1700 600	DC Power kW 1,7 12 1,6	T ℃ 14,8 17,3 21 22,7 24,2	lumps >100 >100 20
TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56 13:10	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter	∆t s 127 480	D Speed rpm 3000 3000	PA Power kW 46 56	C Speed rpm 4500 4500	9B Power kW 48 49	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5 22,5	C Speed rpm 800 1700 600	DC Power kW 1,7 12 1,6	T °C 14,8 17,3 21 22,7 24,2	lumps >100 >100 20
TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56 13:10 TRIAL	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter Batch 1716-3	Δt s 127 480	D Speed rpm 3000 3000	Power kW 46 56	C Speed rpm 4500 4500	Power kW 48 49	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5 22,5 Pump	C Speed rpm 800 1700 600	0C Power kW 1,7 12 1,6	T °C 14,8 17,3 21 22,7 24,2	lumps >100 >100 20
TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56 13:10 TRIAL 8	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter Batch 1716-3 21.01.2025	Δt s 127 480	D Speed rpm 3000 3000	PA Power kW 46 56	C Speed rpm 4500 4500	Power kW 48 49 0B	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5 22,5 22,5 Pump speed DB	E Speed rpm 800 1700 600	DC Power kW 1,7 12 1,6	T °C 14,8 17,3 21 22,7 24,2	lumps >100 >100 20
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TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56 13:10 TRIAL 8 Time hh:mm 13:25	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter Batch 1716-3 21.01.2025 Process Step Filling of tank	Δt s 127 480 Δt s	D Speed rpm 3000 3000 3000 Speed rpm	PA Power kW 46 56 26 Power kW	C Speed rpm 4500 4500 C Speed rpm	Power kW 48 49 B Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5 22,5 22,5 Pump speed DB Volumeflow m <sup>3</sup> /h	C Speed rpm 800 1700 600 C Speed rpm	OC Power kW 1,7 12 1,6 OC Power kW	T °C 14,8 17,3 21 22,7 24,2 T °C 15,1 17,2	lumps >100 >100 20
TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56 13:10 TRIAL 8 Time hh:mm 13:25 13:54	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction	Δt s 127 480 Δt s 131	D Speed rpm 3000 3000 3000 Speed rpm 3000	PA Power kW 46 56 Power kW 46	C Speed rpm 4500 4500 5peed rpm	Power kW 48 49 B Power kW	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5 22,5 Pump speed DB Volumeflow m <sup>3</sup> /h	Speed rpm 800 1700 600 C Speed rpm 700	OC Power kW 1,7 12 1,6 OC Power kW 1,2	T ℃ 14,8 17,3 21 22,7 24,2 T ℃ 15,1 17,3	lumps >100 >100 20 lumps
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TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56 13:10 TRIAL 8 Time hh:mm 13:25 13:54 13:56 14:07	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB	Δt s 127 480 Δt s 131 480	D Speed rpm 3000 3000 3000 Speed rpm 3000 3000	PA Power kW 46 56 PA Power kW 46 56	C Speed rpm 4500 4500 C Speed rpm	Power kW 48 49 0B Power kW 30	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5 22,5 22,5 Pump speed DB Volumeflow m <sup>3</sup> /h	C Speed rpm 800 1700 600 C Speed rpm 700 1700 600	DC Power kW 1,7 12 1,6 DC Power kW 1,2 12 1,6	T °C 14,8 17,3 21 22,7 24,2 T °C 15,1 17,3 21,1 23,2	lumps >100 >100 20 lumps >100 ~50
TRIAL 7 Time hh:mm 12:00 12:42 12:44 12:56 13:10 TRIAL 8 Time hh:mm 13:25 13:54 13:56 14:07	Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter Batch 1716-3 21.01.2025 Process Step Filling of tank Powder Induction 8 min dispersion 1st pass DB 2nd pass DB + filter	Δt s 127 480 Δt s 131 480	D Speed rpm 3000 3000 3000 Speed rpm 3000 3000	PA Power kW 46 56 Power kW 46 56	Speed rpm 4500 4500 Speed rpm 4500	Power kW 48 49 0B Power kW 30	Pump speed DB Volumeflow m <sup>3</sup> /h 22,5 22,5 Pump speed DB Volumeflow m <sup>3</sup> /h	C Speed rpm 800 1700 600 C Speed rpm 700 1700 600	DC Power kW 1,7 12 1,6 DC Power kW 1,2 12 1,6	T °C 14,8 17,3 21 22,7 24,2 T °C 15,1 17,3 21,1 23,2 25,2	lumps >100 >100 20 lumps >100 ~50

DA: powder dispersion unit

DB: inline Dispersion unit

# 2. Reference Pictures for the Cleaning Trials and Validation



Figure 40: cleaning trial setup with equipment description



Figure 41: Filling of premix into the product tank



Figure 42: Reference for pipe segment score 1 - visibly clean and hardly any spots under UV



Figure 43: Reference for pipe segment score 2 - visibly clean and few spots under UV (teal in red circle)



Figure 44: Reference for pipe segment score 3 - visibly clean but many spots under UV



Figure 45: Reference for pipe segment score 4 - little residue visible without UV marked red



Figure 46: Reference for pipe segment score 5 - dirty



Figure 47: Reference for tank score 1: hardly any spots under UV



Figure 48: Reference for tanks score 2 - visibly clean with few spots under UV



Figure 49: Reference for tanks score 2 - same picture as Figure 48 but with flashlight, making UV spots mostly invisible



Figure 50: Reference for tanks score 3 – visibly clean



Figure 51: Reference for tanks Score 4 - rinsed, not clean



Figure 52: Reference for tanks score 5 - dirty


Figure 53: Cleaning validation with riboflavin using a UV torch to illuminate remaining stains. Left: filled tank with 20 ppm riboflavin solution. right: drained solution with clearly visible stains, serving as an indicator of sufficient cleanability



Figure 54: Cleaning validation with riboflavin using a UV torch - after rinsing for 30 seconds with DI water. left: small spray shadow on the agitator visible; middle: spray shadow on the spray ball pipe; right: upper tank wall with spray shadow (upside down).

# Indices

## 1. Figures

Figure 1: Sustainable Manufacturing Processes [7] 4
Figure 2: GHG emissions in 2022 attributed to the described sectors. Left is a depiction of
electric power as a separate consumer, right each sector is listed with electric power
added accordingly. [8]
Figure 3: Lifecycle GHG Emissions Intensity of Electricity Generation Methods [9] 6
Figure 4: This graph shows the amount of starch produced from 2016-2022 and clearly
shows corns predominance on the market. [19]7
Figure 5: Primary structure of the building blocks of starch, showing the two different
glycosidic bonds occurring in amylose and amylopectin [23]
Figure 6: composition of a starch granule with a cut granule imaged via electron
microscopy, showing the growth rings formed by periodically alternating crystalline and
amorphous lamellae. [24] 10
Figure 7: Detailed view of semi-crystalline amylopectin layers. The relevant distances
described are $l = 8.6 \text{ nm}$ t = 7.5 nm and t = 1.1 nm [27] 11
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x.
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]
Figure 8: corn starch treated with indicated temperature in °C at a magnification of 2500x. [30]

Figure 13: Cone and Plate drawing with d as distance between cone and plate, $\theta$ as angle
of the cone to the plate and $\omega$ as angular speed
Figure 14: Time-dependent rheology shows hysteresis loops as opposed to time
independent rheological behaviour. [41] 19
Figure 15: Flow curve of a thixotropic fluid during a step test. [42]
Figure 16: Near-thixotropic behaviour of a liquid. [43] 20
Figure 17: Chemical structure of Riboflavin [49] 23
Figure 18: Premix dispersion setup 25
Figure 19: P&ID of the cleaning trial with highlighted steps
Figure 20: Agarose and Dextrose plates with inactivator solution and applied swab test.
The top plate has 2 spots, which would count as 2 CFU and the bottom plate has 1 spot,
so 1 CFU (seen in red). Both would be indicated as <10 CFU according to standard 28
Figure 21: Powder Analysis Data including particle size distribution and water content of
the used starch batches
Figure 22: Viscosity of first DB pass with 6m <sup>3</sup> /h flow rate through DB
Figure 23: Viscosity of first DB pass with 9m <sup>3</sup> /h flow rate through DB
Figure 24: Viscosity of first DB pass with 22.5m <sup>3</sup> /h flow rate through DB
Figure 25: Viscosity of first 4 minutes of dispersing with DA
Figure 26: Collected viscosity measurements for trial 1
Figure 27: Viscosity of first DB pass with 9m <sup>3</sup> /h flow rate through DB at 3600 rpm 34
Figure 28: September trials viscosity data after passing DB for the first time
Figure 29: September trial 6 rheometer measurements: double logarithmic flow curve
with fitting
Figure 30: September trial 6 rheometer measurements: constant shear rate
measurements at 6.8 1/s
Figure 31: Jan 25 Trial 2: Discrepancy of DB1 and DB2 with comparable time passing
between passing through DB and the viscosity measurement (11 min and 11.5 min
respectively)
Figure 32: Flow-curve measurement for Trial 4 in Jan 25, displaying thixotropic behaviour.
Figure 33: Flow curves measured with a rheometer starting with high shear rate, reducing
it over time

Figure 34: Hysteresis loop of the trials in Jan 25
Figure 35: Hysteresis loop of the trials in Jan 25 40
Figure 36: Hysteresis loop of the trials in Jan 25 41
Figure 37: CT1 pipe segment with dried-up premix, showing retrogradation of the premix
and a likely semicrystalline layer of starch 42
Figure 38: July/August, Trial 2, 6m <sup>3</sup> /h pump speed, 4500 rpm DB, 3000 rpm DA 50
Figure 39: Total time passed since premix passed DB added
Figure 40: cleaning trial setup with equipment description
Figure 41: Filling of premix into the product tank
Figure 42: Reference for pipe segment score 1 - visibly clean and hardly any spots under
UV
Figure 43: Reference for pipe segment score 2 - visibly clean and few spots under UV (teal
in red circle)
Figure 44: Reference for pipe segment score 3 - visibly clean but many spots under UV64
Figure 45: Reference for pipe segment score 4 - little residue visible without UV marked
5
red

### 2. Equations

Equation 1: Dynamic viscosity	15
Equation 2: Kinematic viscosity	15
Equation 3: Power law	16
Equation 4: Bingham model for non-Newtonian fluid behaviour	16
Equation 6: Equations for calculating the shear stress and the shear rate in a c	oncentric
cylinder system of a rotational viscometer. [39]	18
Equation 7: Calculation of shear rate of a rotor-stator dispersion unit	24
Equation 8: Mathematical power law model describing the behaviour of freshly	dispersed
premix	50

#### 3. Tables

Table 1: Pre-cooked starch powder specifications used in the trials    29
Table 2: July/August trials fitted to power law models with the time correction factor fitting
the model best (only 10 second increments used) 33
Table 3: Power law constants and $R^2$ of Sept trials of fitted curves after passing DB for the
first time
Table 4: Jan 25 trials: power law parameters for viscosity curves after the first passing of
DB
Table 5: Jan 25 trials: power law parameters for viscosity curves after the second passing
of DB
Table 6: CT1 with 2 cleaning steps performed
Table 7: CT1 score card 43
Table 8: CT2 cleaning steps performed
Table 9: CT2 score card 43
Table 10: CT3 cleaning steps 44
Table 11: CT3 score card
Table 12: CT4 cleaning steps 45
Table 13: CT4 score card
Table 14: CT5 process steps 46
Table 15: CT5 score card 46

Table 16: CT6 process steps 4	.7
Table 17: CT6 score card 4	₽
Table 18: Machine settings, procedure and number of lumps for Trial 1 in July/August . 5	5
Table 19: Machine settings, procedure and number of lumps for Trial 2 in September 5	6
Table 20: Machine settings, procedure and number of lumps for Trial 3 in Jan 2025 5	8

## List of Abbreviations

- DA ... Powder dispersion unit
- DB ... Inline dispersion unit
- DC ... beam mixer
- IBC ... industrial batch container
- PL ... product loop in cleaning trial
- CL ... cleaning loop in cleaning trial
- CIP ... Cleaning in Place
- PSD ... particle size distribution
- DI ... deionized
- FA ... formic acid
- wt% ... percentage by weighth
- w ... angular momentum (rad/s)
- M ... torque (Nm)
- n ... rotations per minute (rpm)
- d ... diameter (m)
- $s \dots gap$  between rotor and stator in the tools of DA and DB
- r ... distance between two points (m)
- $\eta \ldots$  Dynamic viscosity (mPa\*s or cP)
- cP ... centi-Poise (=1 mPa\*s)
- $\tau$  ... shear stress (Pa)
- $\rho$  ... density (kg/m<sup>3</sup>)

- v ... kinematic viscosity (m<sup>2</sup>/s)
- γ... shear rate (s<sup>-1</sup>)

 $\eta_{\infty}$  ... Dynamic viscosity at an infinitely high shear rate (mPa\*s or cP)

 $\tau_{\scriptscriptstyle 0} \ldots$  Yield shear stress (Pa)

A ... interception on the Y-axis for the developed formula to validate the premix faster

B ... exponent for the developed formula to validate the premix faster, showing the speed with which the viscosity increases

CFU ... colony forming units

GHG ... greenhouse gas emissions

CT ... Cleaning trial

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