

Rheological and Functional Characterization of Oat Drink By-Product:

Effects of β -Glucan Molecular Weight, Solids Content, and Processing Conditions

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MASTER THESIS



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Examiner: Jeanette Purhagen

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From Side-Stream to Food: Unlocking the Potential of Oat Drink By-Product

As the world's population continues to grow, finding sustainable ways to produce food has become increasingly important. One promising approach is to make better use of side-streams from food manufacturing instead of letting them go to waste. This can help reduce both the environmental impact of food production and reduce food waste.

One such side-stream is oat drink by-product (ODBP), a nutrient-rich residue, created during the production of oat-based beverages. ODBP contains high levels of a type of dietary fiber called, β -glucan (BG) which is linked to various health benefits such as lower risk of cardiovascular disease and high blood pressure. Despite its nutritional value, ODBP is currently underutilized largely because of its thick consistency, which makes it difficult to transport and incorporate into new food products. Part of this thickness is believed to come from these β -glucans.

The key to overcoming this challenge lies in understanding the material's rheology. Rheology is the science of how materials flow and deform when pressure is applied. By understanding how ODBP behaves under different conditions, it becomes possible to design efficient production processes that can make better use of this valuable resource.

The results from the study show that the flow behavior of ODBP is strongly influenced by the temperature, water content and the size of the β -glucan molecules. β -glucans are actually long chains of sugar molecules, and the length of these chains determines how they interact with each other and other molecules in the ODBP. These interactions play an important role in determining the material's properties. Longer molecules were shown to make ODBP thicker and more resistant to flow, while shorter molecules resulted in less viscous material.

Temperature was also shown to have a significant effect. As ODBP becomes warmer, it flows more easily and requires less force to move through pipes and processing equipment. However, when reaching high enough temperatures, these benefits became smaller. In practice, this insight can be used to design food production processes that apply just the right amount of heat to make the product flow more easily (and keep them safe from harmful microbes) without wasting energy on excessive heating.

Ultimately, these findings bring ODBP one step closer to becoming a practical ingredient in new food products, which could reduce waste, create more sustainable food and unlock the potential of this nutritious material that is currently overlooked.

Abstract

With a growing global population, the demand for food increases, and the development of more sustainable food production becomes increasingly important. One way to improve sustainability within food production is to repurpose nutritious by-products to new food ingredients. Oat drink by-product (ODBP), generated during the production of oat-based beverages, is one such stream. ODBP contains significant amounts of β -glucan (BG), a type of dietary fiber associated with various health benefits such as preventing diabetes, reducing hypertension, and lowering glycemic index. While BG adds desirable nutritional value to ODBP, its remarkable influence on the rheological properties of food systems, could be a contributing factor to the complicated handling of ODBP, which is essential for its repurpose into new food products. Therefore, this study aims to investigate the rheological and functional properties of ODBP and the contribution of β -glucans to these properties.

Flow behavior and apparent yield stress were characterized across varying temperatures and total solids (TS) content using a rheometer and Rapid Visco Analyzer (RVA). To assess the contribution of BG, enzymatic hydrolysis was applied to generate samples with different BG Mw. The resulting flow curves were fitted to the Herschel-Bulkley and Bingham plastic fluid models, and the resulting parameters were used to evaluate the effects of temperature, TS, and BG Mw on ODBP yield stress and flow behavior.

The results demonstrated that BG Mw, temperature, and TS content all significantly affected the rheology of ODBP. Higher BG Mw increased the resistance to flow and promoted shear thinning behavior at low shear rates. Increasing the temperature significantly lowered the apparent viscosity, though the effect was smaller at higher temperatures. Lower TS content markedly decreased apparent yield stress.

Comparison of analytical methods indicated similar overall rheological trends, although some differences were observed. The rheometer provided greater precision and enabled measurements at lower shear rates, while the RVA was able to cover a broader shear rate range. Absolute values of apparent yield stress varied substantially depending on the measurement method and the suitability of the two rheological models, where shown to be dependent on both the shear rate interval measured as well as the BG Mw.

To assess potential storage effects on fresh and freeze-thawed ODBP, texture analyses were performed. The results indicate no effect on fresh samples over a three-day period, regardless of storage temperature, while there were significant effects in the hardness of freeze-thawed ODBP over the same testing period.

A strong influence of BG Mw and TS on the functional properties of dried ODBP could not be confirmed. Instead, the average particle size of the dried by-product exhibited the greatest impact on the measured properties. Thus, further studies are required to conclusively determine the role of BG in shaping the functional properties of dried ODBP.

Sammanfattning

Världens befolkning ökar, och med det även behovet av mängden livsmedel, vilket förstärker vikten av hållbar livsmedelstillverkning. En metod för att öka hållbarheten inom livsmedelsproduktion är att omvandla biprodukter till nya livsmedel. Restprodukten som bildas vid havredryckstillverkning (ODBP, *oat drink by-product*) utgör en sådan biprodukt. ODBP innehåller nämligen betydande mängder β -glukaner (BG), en typ av kostfiber som har visat sig bidra till hälsofördelar såsom förebyggande av diabetes och hjärt- och kärlsjukdomar. Samtidigt har BG visat sig påverka de reologiska egenskaperna hos livsmedel på ett betydande sätt, och skulle därför också kunna vara en bidragande faktor till det som idag innebär stora komplikationer i hanteringen av ODBP. Syftet med denna studie är därför att undersöka ODBPs reologiska och funktionella egenskaper samt att kartlägga BGs inflytande över dessa.

Flödesegenskaperna och den skenbara flytgränsen (*yield stress*) undersöktes vid olika temperaturer och torrsubstans (TS, *total solids*) med hjälp av en reometer och Rapid Visco Analyzer (RVA). Enzymatisk hydrolys användes för att framställa prover med olika BG molekylvikt för att på så sätt kunna undersöka BG:s bidrag till de reologiska egenskaperna. De erhållna flödeskurvorna passades sedan till två flödesmodeller, Herschel-Bulkley och Bingham plastisk. Modellparametrarna användes sedan för att utvärdera hur temperatur, torrhalt och BGs genomsnittliga molekylvikt påverkar ODBPs reologiska egenskaper.

Resultaten visade att både BGs molekylvikt, temperaturen och torrhalten påverkade ODBPs reologi på ett betydande sätt. Högre molekylmassa ökade flödesmotståndet och förstärkte provets skjuvtunnande egenskaper, även vid låg skjuvhastighet. Flytgränsen visade sig vara kraftigt påverkad av torrsubstansen och ökande temperatur minskade flödesmotståndet, även om effekten avtog något vid högre temperaturer.

Trots vissa mindre skillnader visade de olika analytiska metoderna liknande övergripande reologiska trender. Reometern gav högre precision och möjliggjorde mätningar vid lägre skjuvhastigheter medan RVAn täckte ett bredare skjuvhastighetsintervall. De absoluta värdena för de skenbara flytgränserna varierade dock avsevärt mellan de olika mätmetoderna. Resultaten visade även att de två reologiska modellernas lämplighet berodde både på BGs molekylvikt och skjuvhastighetsintervallet.

För att undersöka eventuella lagringstidseffekter hos färsk respektive upptinad ODBP genomfördes texturanalyser med en Texture Analyzer. Resultaten visade att lagring av färska prover inte påverkade hårdheten (*hardness I*) under en tredagarperiod oavsett vid vilken temperatur proverna lagrats. Däremot observerades en markant förändring i hårdhet hos de upptinade proverna med ODBP.

Det gick inte att utläsa någon tydlig påverkan av BG eller TS på de funktionella egenskaperna hos torkad ODBP. Den genomsnittliga partikelstorleken hos de torkade proverna var istället det som visade sig vara den mest avgörande faktorn. Ytterligare studier krävs därför för att säkerställa BGs effekt på de funktionella egenskaperna hos torkad ODBP.

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Contributions and the use of AI

Maya Eriksson did all the sample preparations and rheology experiments, including measurements on the RVA, rheometer, and texture analyser.

Maya Eriksson did the analysis on functional properties.

The Python code was generated aided by Claude AI-model Sonnet 4.6.

The analysis of beta-glucan molecular weight was performed by Nikolina Barchan at Oatly

The microscopy work was performed by Nick Sirijovski at Oatly

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Nomenclature

Abbreviation	Meaning
ANOVA	Analysis of Variance
BG	β -glucan
BP	Bingham Plastic
HB	Herschel-Bulkley
HPLC	High-Performance Liquid Chromatography
LVR	Linear Viscoelastic Region
Mw	Molecular Weight
ODBP	Oat Drink By-Product
OHC	Oil Holding Capacity
RVA	Rapid Visco Analyzer
RPM	Revolutions per Minute
SWC	Swelling Capacity
TPA	Texture Profile Analysis
TS	Total Solids
WHC	Water Holding Capacity
WSI	Water Solubility Index

Symbol	Description
A	Area
Δx	Deformation (distance)
δ	Angle
F	Force
G'	Storage modulus
G''	Loss modulus
G^*	Complex modulus
γ	Shear strain
$\dot{\gamma}$	Shear rate
$\dot{\gamma}_{max}$	Maximum shear rate
h	Length
η_a	Apparent viscosity
η_p	Plastic viscosity
k	Consistency index
n	Flow behavior index
t	Time
T	Temperature
τ	Shear stress
τ_0	Apparent yield stress
τ_{max}	Maximum shear stress

1 Introduction

1.1 Background

The global food system has a substantial impact on the environment and is a major contributor to greenhouse gas emissions, deforestation, and biodiversity loss (Benton, et al., 2021). With a growing global population, the demand for food increases, and the development of more sustainable food production systems becomes increasingly important. Customers today are looking for more sustainable food alternatives (Sharma, et al., 2024), and one way to improve sustainability within food production is to effectively repurpose by-products from food processes into new food ingredients or products.

Oatly is the world's first and largest oat drink company. The production of oat drink at Oatly is presented in Figure 1.1 below. It begins with mixing kilned oat kernels with hot water. This process breaks up starch granules and makes the starch more available for enzymes, which are added in the following step. After the enzyme process, the slurry is separated with a decanter into two separate fractions: the oat base, which later becomes the oat drink, and the oat drink by-product (ODBP).

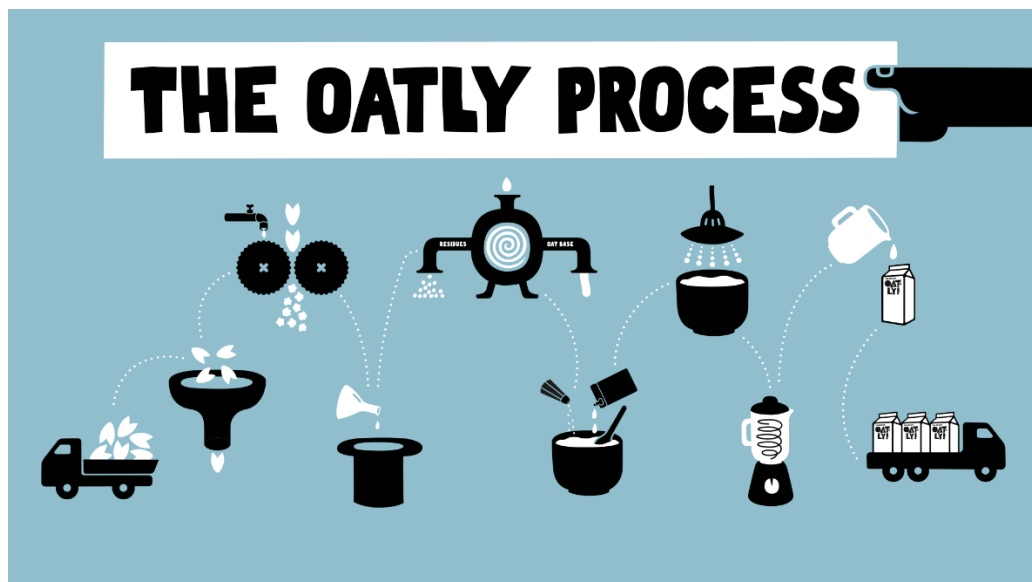


Figure 1.1. Overview of the oat drink production process (from oatly.com)

While the oat base undergoes additional formulation steps such as mixing with other ingredients, heat treatment, and homogenization to become the final product, the ODBP is not used for human consumption. Today, over 60 % of this by-product is converted to animal feed, while the rest is converted to energy or used for soil improvement, even though the nutritional composition of ODBP ranges from 30-40 g of fiber, 25-35 g of protein, 10-20 g of fat, 10-20 g of carbohydrates (Ehlde & Petersson, 2021), would have great potential in being repurposed as food products.

One of the main challenges limiting the use of ODBP in food applications is processing the by-product at large scale. For food safety purposes, immediate processing at elevated temperatures is required after the separation step, which necessitates transporting the ODBP. The difficulty in doing so is due to its complex rheological properties, which are largely unexplored. About 7

% of the ODBP consists of β -glucans (BG), a dietary fiber, which can be used as e.g. a thickening agent in different food applications (Kaur, et al., 2020). Therefore, investigating the rheological properties of ODBP and the contribution of BGs, is a key step towards turning ODBP into food products in the future.

1.2 Aim

The aim of this Master's Thesis was to investigate the rheological and functional properties of oat drink by-product (ODBP) and the contribution of β -glucans to these properties.

1.3 Research Questions

- How do the temperature, total solids, and β -glucan molecular weight affect the flow behavior of the ODBP?
- Which rheological model most accurately describes ODBP?
- How do different methods for determining apparent yield stress compare?
- How do rheological measurements of ODBP compare when using a Rapid Visco Analyzer (RVA) versus a rheometer?
- Is there a storage time effect on 1) fresh and 2) freeze-thawed ODBP?
- What impact does total solids and β -glucan molecular weight have on the functional properties of dried ODBP?

2 Theoretical Background

2.1 β -glucan

β -glucans (BG) are a type of dietary fiber that has been shown to provide a number of health benefits, such as a reduction in glycemic index and preventing diabetes, hypertension, and cardiovascular diseases (Kaur, et al., 2020). They consist of D-glucose monomers that are linked together by β -glycosidic bonds, depicted in Figure 2.1. Their exact molecular structure differs depending on their source, as they can be found in both plants, yeast, fungi and some bacteria (Maheshwari, et al., 2019). They exist in multiple different conformations, such as aggregates, single helix, and random coil, and depending on origin, structural features, and molecular weight, they can display different rheological properties (Kaur, et al., 2020). In oats, the fibers have β -(1,3;1,4) bonds, and together the monomers form a linear structure that is water-soluble. Because of these β linkages, the fibers are nondigestible to humans (El Khoury, et al., 2012) and that part of the health benefits observed might stem from the process of creating a highly viscous gel in the intestines (El Khoury, et al., 2012). They are also readily fermented in the human gut and thus serve as a prebiotic source (Connolly, et al., 2016).

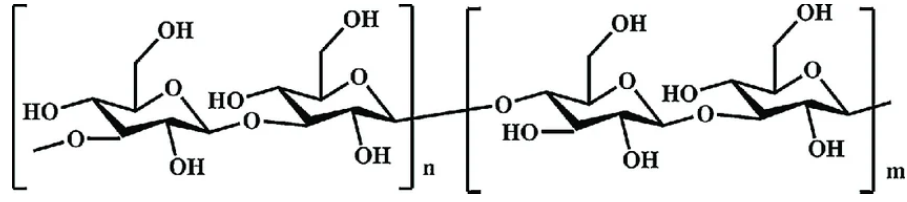


Figure 2.1. β -glucan with 1,3:1,4 glycosidic bonds (from researchgate.net)

In oats, BG are prevalent in multiple parts of the seed, and when the oats are developing, the amount of BG is enzymatically regulated as a response to changing environmental conditions (Hozlár, et al., 2022). Thus, oats inherently contain enzymes that break down β -(1,3;1,4) bonds, called lichenase (El Khoury, et al., 2012).

In previous studies, BG has had a significant impact on some of the functional properties of foods. For example, it has been shown that the molecular weight of BG can have an effect on the solubility, texture and rheology in food systems (Kaur, et al., 2020). Thus, the molecular weight of the BG could potentially have an impact on the functional properties of dried ODBP. Therefore, this aspect will be investigated in this thesis.

2.2 Rheology Theory

Rheology is the branch of science that studies the deformation and flow of matter caused by an external force (Rao, 2014). The applied force is commonly referred to as *shear stress* and is measured in force per unit area and can thus be expressed as seen in Equation 1,

$$\tau = \frac{F}{A} \quad (1)$$

where F [N] is the applied force and A [m²] is the area over which the force acts. The response of the material to stress can be measured either in amount of deformation, expressed as *shear strain*, or in rate of deformation, expressed as *shear rate* (Wilson, 2018) .

For fluids, shear deformation can be illustrated using a parallel-plate model, depicted in Figure 2.2, in which a fluid is confined between two plates where one plate moves relative to the other.

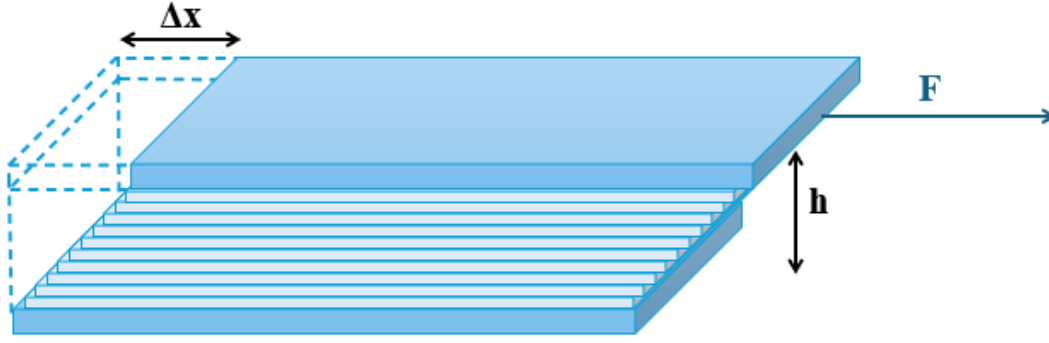


Figure 2.2. Shear deformation between parallel plates illustration.

As the force from the moving plate is applied, adjacent fluid layers start to slide past one another, generating internal friction within the fluid. This internal resistance to flow is described as the apparent viscosity, η_a [Pa s], which can be defined as seen in Equation 2,

$$\eta_a = \frac{\tau}{\dot{\gamma}} \quad (2)$$

where τ [Pa] is the shear stress and $\dot{\gamma}$ [s^{-1}] is the shear rate (Dogan & Kokini, 2007).

Shear strain, γ , is dimensionless and is given by the ratio between the deformation, Δx [m] and the original length, h [m] (Martínez-Padilla, 2023), see Equation 3.

$$\gamma = \frac{\Delta x}{h} \quad (3)$$

Shear rate, $\dot{\gamma}$ [s^{-1}] represents the change in shear strain as a function of time (Ramli, et al., 2022), see Equation 4.

$$\dot{\gamma} = \frac{d\gamma}{dt} \quad (4)$$

2.2.1 Non-Newtonian fluids

For some fluids, e.g. water, the relationship between shear stress and shear rate is linear, resulting in constant viscosity that is independent of the shear stress. These fluids are referred to as Newtonian. Most fluids, however, exhibit a viscosity that varies with the rate of deformation and thus have a non-linear relationship between shear rate and stress. Such fluids can exhibit many different behaviors, but are all classified as non-Newtonian. *Shear-thinning* fluids have a decrease in apparent viscosity with increasing shear rate, while *shear-thickening* fluids are characterized by an increase in apparent viscosity with increasing shear rate. The type of response a fluid has to increasing shear rate is determined by the interactions between the different components inside the fluid (Ngyen & Ngyen, 2012).

Viscoelastic liquid:

Some shear-thinning fluids exhibit both a plastic, solid-like, as well as viscous, or fluid-like behavior depending on the rate of deformation. When increasing the shear, such fluids first respond as an elastic solid until reaching a certain point where they start to flow like a liquid. This trait is called viscoelasticity and can be caused by the interactions between particles within the fluid, such as suspended particles like fibers, cells, or bubbles (Wilson, 2018).

Storage and Loss Modulus

A way to quantify elastic and viscous behavior is through the storage and loss modulus. By placing a sample between two plates and letting the upper plate rotate in an increasing oscillatory motion at a set frequency, the angle difference between the maximum shear strain and shear stress can be measured (Ramli, et al., 2022).

If the maximum shear stress and strain, during the oscillation, are perfectly aligned, the material is considered to be completely elastic. In contrast, if the angle difference is exactly $\frac{\pi}{2}$, the material is viscous. All angles between 0 and $\frac{\pi}{2}$ can be referred to as viscoelastic (Dogan & Kokini, 2007). This is illustrated in Figure 2.3 below.

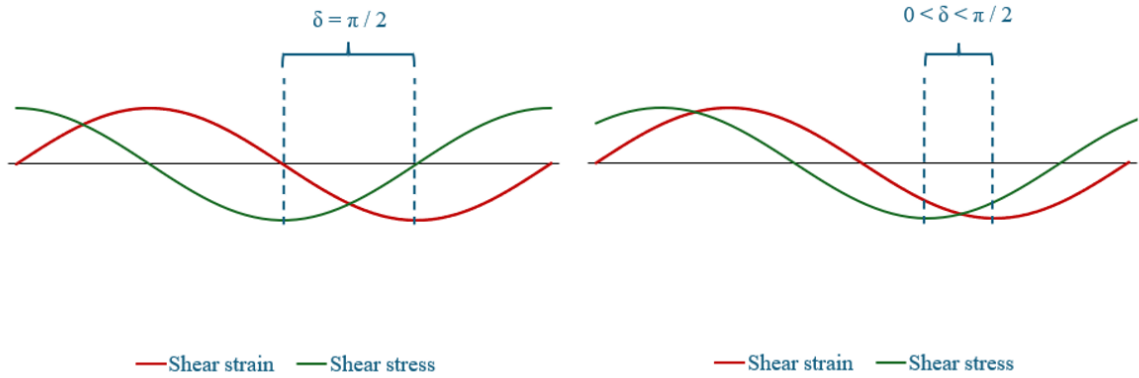


Figure 2.3. The sinus curve of shear stress and shear strain for a) a viscous material b) a viscoelastic material.

In this type of experiment, the ratio between the maximum shear stress and the maximum shear strain is known as the *complex modulus*, G^* [Pa], according to Equation 5,

$$G^* = \frac{\tau_{max}}{\gamma_{max}} \quad (5)$$

where τ_{max} [Pa] is the maximum shear stress and γ_{max} is the maximum shear strain. The complex modulus describes the total viscoelastic behavior and is used to calculate the separate elastic and viscous contributions, the storage and loss modulus (Dogan & Kokini, 2007).

The *storage modulus*, G' [Pa], is defined according to Equation 6,

$$G' = G^* \cos \delta \quad (6)$$

where δ is the angle difference between the maximum shear stress and maximum shear strain. The storage modulus represents the elastic contribution, or solid-like response, of the viscoelastic behavior (Ramli, et al., 2022).

The *loss modulus*, G'' [Pa], describes the opposing, viscous, or liquid-like component. It is defined according to Equation 7 below (Ramli, et al., 2022).

$$G'' = G^* \sin \delta \quad (7)$$

If $G' > G''$, the sample displays predominantly elastic (solid-like) behavior. In contrast, a $G' < G''$ implies a viscous (liquid-like) behavior. At low deformation rates, the storage and loss modulus are constant. This is referred to as the *linear viscoelastic region*, LVR which is illustrated in Figure 2.4. For some materials, the moduli decrease at different rates as the applied shear increases. This can sometimes lead to reaching a so-called crossover, or flow point, where $G' = G''$ (Malvern, 2012).

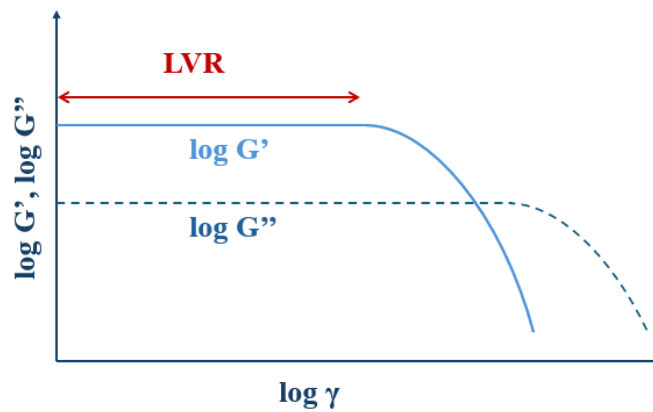


Figure 2.4. Linear viscoelastic region.

Yield Stress

The shear stress that needs to be applied in order for a sample to flow is what is defined as the *yield stress*. Viscoelastic material that exhibits yield stress behavior has a solid-like structure at low shear rate, though as a certain critical amount of stress is applied, this structure breaks down and the material starts to flow (Sun & Gunasekaran, 2009).

There is still no unanimous consensus on what true yield stress is exactly. However, since this parameter is useful in many practical applications, finding a critical point at which there is a clear decline in viscosity is of great interest. This shear stress is therefore often referred to as *apparent yield stress* (Malvern, 2012).

Because the meaning of true yield stress is still a topic of debate, there is not one singular way of measuring it. There are various ways of determining the apparent yield stress of a complex liquid and the results can vary greatly depending on the analytical method used. Therefore, this thesis explores multiple ways of determining this critical value for ODBP.

One way to measure the apparent yield stress is to find the crossover point from an oscillatory amplitude sweep. If there is no such point, the end of the viscoelastic linear region can be defined as the apparent yield stress. Another method is to measure shear stress versus shear rate data on a viscometer, RVA or rheometer and fit the data to a rheological model such as the Bingham plastic or Herschel-Bulkley model. The apparent yield stress is then found by extrapolating to zero shear rate (Malvern, 2012).

Thixotropy

Some non-Newtonian fluids also show time-dependent rheological properties denoted as *thixotropy*. Just like the concept of yield stress, the true definition of thixotropy has long been a topic of debate, despite being one of the oldest documented rheological phenomena in colloidal science (Mewis & Wagner, 2009). A widely cited article by Mewis and Wagner (2009) defines thixotropy as “The continuous decrease of viscosity with time when flow is applied to a sample that has been previously at rest and the subsequent recovery of viscosity in time when the flow is discontinued.”. In this review, Mewis and Wagner explain that even though there remains a lot to be uncovered about the details of thixotropy, it is a very reoccurring phenomenon in both industrial and natural systems.

2.2.2 Rheological models

To describe the flow behavior of these complex fluids, there are many different rheological models. One of the simplest models to describe shear-thinning and thickening behavior is the power-law model. It is defined according to Equation 8,

$$\tau = k\dot{\gamma}^n \quad (8)$$

where τ is the shear stress, k is the consistency index [Pa s^n] and n is the dimensionless flow behavior index. If n is smaller than 1, it represents a shear-thinning pattern and if it is greater than 1, it indicates a shear-thickening behavior (Ngyen & Ngyen, 2012).

A model that describes fluids that have a Newtonian-like behavior after a yield stress is exceeded, is the Bingham plastic model. It can be written as seen in Equation 9,

$$\tau = \tau_0 + \eta_p \dot{\gamma} \quad (9)$$

where τ_0 [Pa] represents the apparent yield stress and η_p [Pa s] is the plastic viscosity (Martínez-Padilla, 2023).

A third model that combines the power law and Bingham plastic model by including both a yield stress factor, consistency index and flow behavior index is the Herschel-Bulkley model. This model can be expressed as seen in Equation 10.

$$\tau = \tau_0 + k\dot{\gamma}^n \quad (10)$$

Fluids that fit the Herschel-Bulkley model have an apparent yield stress at low shear rates and a shear thinning (or thickening) pattern at higher shear rates (Martínez-Padilla, 2023).

Figure 2.5 below, illustrates the flow curves of a Newtonian fluid, as well as shear-thinning and thickening, Bingham plastic and Herschel-Bulkley fluids.

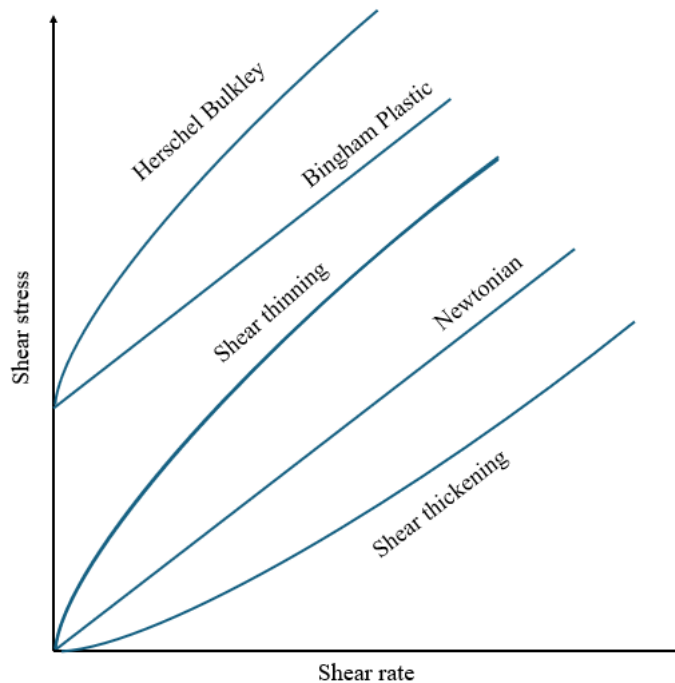


Figure 2.5. Common Newtonian and non-Newtonian models illustration

Yield stress behavior is commonly found in a variety of food products (Sun & Gunasekaran, 2009). Therefore, this study will investigate fitting rheological measurements of ODBP to the Herschel-Bulkley and Bingham plastic models, which both consider the apparent yield stress.

2.3 Rheological Instruments

2.3.1 Texture analyzer

A texture analyzer is a tool designed to measure different physical and mechanical properties in both solid and liquid samples. It is used in different applications such as formulation, processing and packaging and it works by moving a probe against a sample under controlled conditions while measuring the force and displacement. The measurements can be done in one or multiple compression cycles. Some of the parameters that can be analyzed with a Texture Profile Analysis (TPA) program are hardness 1 and 2, cohesiveness, springiness and adhesion (Brookfield Engineering Laboratories, Inc.).

Hardness 1 and 2 are the maximum force applied after the trigger and until the set displacement is reached for the first and second compression cycles, respectively. This force is measured in grams, which can be converted to Newtons by multiplying with the gravitational acceleration.

Cohesiveness is the ratio between the area under the compression stroke of the first cycle, A1, and the second cycle, A2, respectively. If the sample is perfectly elastic, the applied force will be equal in both the first and second cycle and the ratio will thus be 1. If the sample completely collapses in the first cycle, then there will be no resistance to overcome in the second round, meaning A2 will be zero and consequently also the ratio. The cohesiveness parameter can therefore be seen as a measure of elasticity.

Springiness is measured in mm and represents the distance that the sample recovers after being compressed.

Adhesiveness is calculated as the area of the negative force area during probe withdrawal after the first compression cycle.

2.3.2 Rapid Visco Analyzer

A rapid visco analyzer, or RVA, is an analytical instrument originally designed to rapidly analyze the quality of starch samples in a standardized way. A description of the operating principle is given in The RVA Handbook (Crosbie & Ross, 2021). The RVA measures the viscosity of a sample while controlling the temperature and works by rotating a paddle at a pre-set speed inside the sample. Because the instrument is meant to be able to simulate cooking and food processing, the paddle is designed to have good mixing capacity.

When the RVA is set to a certain RPM, the motor generates a torque that is equal and opposite to the torque caused by the sample's resistance to flow. The force applied by the motor is measured and converted internally to viscosity. To determine the viscosity from the torque, the shear rate can be calculated from the rotation of the paddle and the mechanical gap between the moving element and the fixed element, i.e. the paddle and the inside of the canister. To facilitate the calculations, an effective shear rate is applied. This average shear rate, between the RVA paddle and the canister, has been determined by Lai, et al., (2000) to 20.1 s^{-1} per RPS.

2.3.3 Rotational rheometer

A rotational rheometer is an instrument that is used to characterize a variety of rheological properties in both liquids and soft solids. The instrument operates either by applying a predefined shear stress (calculated from the applied torque) and measuring the resulting shear rate, or by rotating at a certain rate and measuring the corresponding stress (Wilson, 2018).

Rheological measurements can be performed using different geometries depending on the material properties and intended analysis. Some examples of such geometries are parallel plates, cone-and-plate and vane-and-cup. Choosing an appropriate geometry for a specific application is of great importance as it can significantly influence the results and introduce experimental artefacts (Feysa, et al., 2017).

The parallel plate geometry is widely used as it has an adjustable gap width and a well-defined flow field. However, this geometry presents certain limitations. If the sample has a low viscosity, it may flow out from the edges of the plates. In addition, large particles may become trapped

within the gap, introducing significant measurement errors. Selecting the gap width for parallel plate geometry is therefore often a compromise between a large enough gap for suspended particles, though small enough to maintain the sample in place. Parallel plate geometry is generally suitable for suspensions with solid particles up to about 200 microns (Satapathy & Bishnoi, 2026).

A vane-and-cup geometry may, on the other hand, be applicable for suspensions with larger particles, as well as low viscosity liquids. Nevertheless, this geometry is also associated with several challenges. One limitation is the less well-defined gap geometry compared with parallel plates and the more complex flow fields, making the measurement more geometry dependent. In addition, particle migration may occur, where heavier particles move toward the outer regions of the cup during measurement. Another issue is shear localization, in which the material closest to the vane separates from the surrounding sample, causing only part of the sample to deform and rotate with the vane (Satapathy & Bishnoi, 2026).

Below, in Figure 2.6, the parallel plate and vane-and-cup geometry are illustrated.

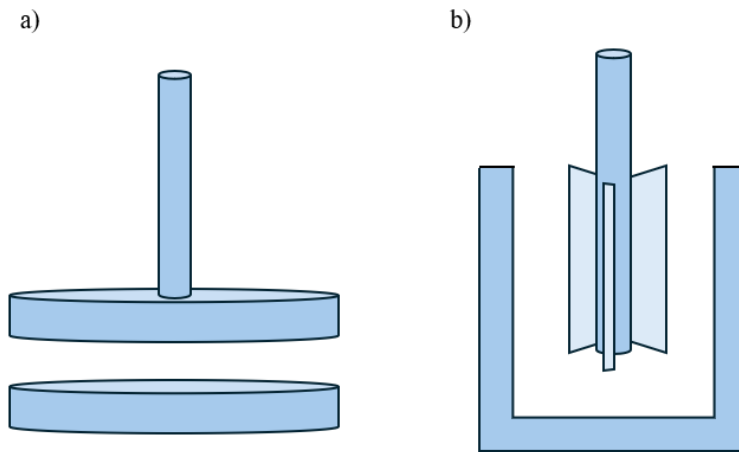


Figure 2.6. Rheometer geometries illustration. a) Parallel plates b) Vane-and-cup

A common issue that arises with all geometries is so called *wall slip*. Wall slip occurs when the sample loses contact with one of the plates or walls and moves together with the rotating plate or probe instead of deforming uniformly. To minimize wall slip, serrated or roughened plates and cups can be used (Feysa, et al., 2017).

3 Material and Methods

3.1 Material

To minimize variation in the start material, all ODBP was retrieved from a single production batch through the same decanter, during production at Oatly. The initial total solids (TS) were measured (see section 3.4). Part of the product was immediately analyzed with a texture analyzer (see section 3.2) while the rest was divided into smaller aliquots and stored frozen before further analysis.

A fungal β -glucanase (lichenase) from the Oatly lab was utilized during the trials to selectively hydrolyze the oat β -glucans.

3.2 Texture Analyzer Measurements

To analyze time-dependency and compare the hardness of freeze-thawed ODBP and fresh product, a TPA test with two compression cycles was carried out using a Brookfield Texture Analyzer (CT3 4500, USA).

Fresh ODBP was measured to 26.2% TS and was then diluted with water to six levels of TS, 14%, 18%, 20%, 22%, 24% and undiluted. 120 ml cups (height: 7 cm, diameter top: 5.5 cm, diameter bottom: 4.5 cm) were filled approximately halfway. One sample from each level of dilution was stored at room temperature, refrigerated and at 75 °C, respectively. After approximately one hour, the samples were analyzed in duplicates with the texture analyzer set to: Trigger: 10g, Deformation: 10,0 mm, Speed (compression and retraction): 2mm/s, using an acrylic TA3 probe with a 25.4mm diameter (height 35mm). The second cycle was initialized immediately after the first cycle. The same procedure was repeated twice on the same sample, after both one and two days.

The same measurement process was repeated for two freeze-thawed samples, one undiluted with no added enzyme, the other diluted to 18% TS with highly hydrolyzed BG. Both freeze-thawed samples were stored refrigerated in between measurements. The two freeze-thawed samples were analyzed twice on each sample, once a day for a total of four days.

3.3 Sample Preparation

To limit time-dependent rheological changes, samples were prepared the same day as measurements were carried out for both RVA and rheometer experiments.

RVA samples

Before RVA-measurements, samples were prepared by thawing ODBP in a water bath. The product was then diluted to 18% TS and immediately heat-treated in a boiling water bath to inactivate any inherent enzymes. During heating, the samples were stirred every other minute. When the sample temperature had surpassed 90 °C, after approximately 20 minutes, they were kept in the water bath for 4 more minutes before being cooled down in another water bath until the temperature was below 60 °C. The moisture content was then measured in duplicates before

adding the lichenase solution and mixing thoroughly. Once the enzyme was added, the samples were incubated in a 60 °C water bath for 60 minutes. To stop the enzymation process, the samples were then heat-treated at 90 °C a second time. Because small amounts of water could evaporate during the heat treatments, the TS content was measured once more in duplicates. Based on these measurements the ODBP was diluted to 18, 16 and 14 % TS.

Rheometer samples

Sample preparation for the rheometer-measurements was carried out in the same way as for the RVA-measurements, except the thawed ODBP was not diluted until after the enzymation-step, to allow for analysis of less diluted samples. The rheometer samples were diluted to 14, 16, 18, 20 and 22 % TS.

3.4 Total Solids Measurements

The dry content of ODBP was measured using a Moisture Analyzer (HS153 Mettler Toledo Switzerland and MB90 Ohaus Switzerland) using a standard protocol of heating approximately 1 g of sample to 140 °C with a built-in automatic detection for weight stabilization as a switch off criteria (typical drying time of 6-10 min).

3.5 β -glucan Molecular Weight Analysis

After the final sample preparation steps (see section 3.3), samples were stored frozen before the average BG molecular weight analysis was carried out. The measurements were performed in duplicates with size exclusion chromatography using a PL Aquagel-OH Guard Column (8 μ m, 50 $\times$ 7.5 mm; Agilent) coupled to a PL Aquagel-OH MIXED-M analytical column (8 μ m, 300 $\times$ 7.5 mm; Agilent). Detection was carried out with an excitation wavelength of 410 nm and an emission wavelength of 430 nm. The mobile phase consisted of 0.1 M Na₂HPO₄ (eluent A) and 0.1 M Na₂HPO₄ containing 5 mg/L Calcofluor (post-column reagent) (eluent B). The chromatographic system consisted of a Thermo Fisher Vanquish Duo HPLC equipped with a fluorescence detector (FLD).

3.6 RVA Measurements

A rapid visco analyzer from Perten (RVA 4800, Perkin Elmer, Australia) was used to generate flow curves. The viscosity of each sample was measured using a single program with stirring speeds ranging from 400 to 30 RPM at three different temperatures; To set the right temperature the samples were stirred at low speed (20 RPM) for 2,5 minutes while the RVA was heating up from the start-temperature of 50 °C to the set temperature of 55 °C followed by pre-shearing at 300 RPM for 1 minute. The viscosity was then measured at the highest stirring speed, 400 RPM, followed by 310, 220, 130, 40 and finally 30 RPM. Each stirring speed was kept constant for 2 minutes. Once the lowest stirring speed had been measured, the same sequence was repeated at a higher temperature. Measurements were performed at 55, 65 and 75 °C in one sequence, starting with the lowest temperature. The sequence for one temperature interval is illustrated in Figure 3.1.

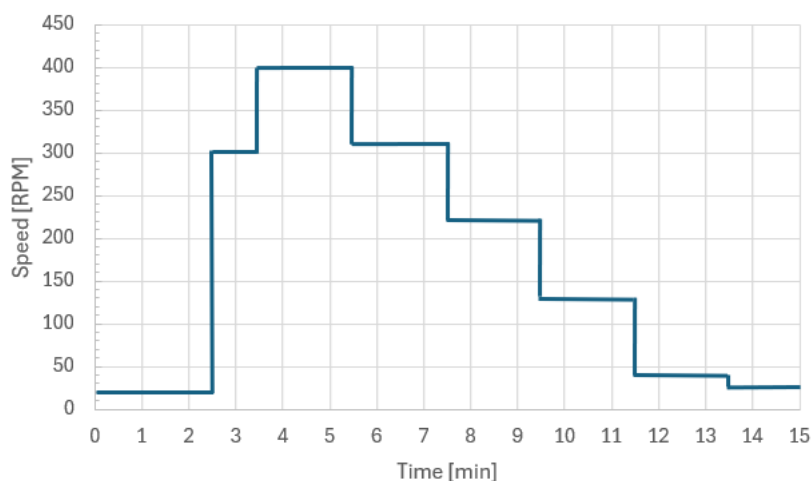


Figure 3.1. RVA sequence illustration.

In total, measurements were carried out on samples with three different levels of dilution, 14, 16, and 18% TS and four levels of enzyme dosing, no, low, medium, and high enzyme dose, meaning the BG are hydrolyzed to the point of being undetectable. Duplicates were performed on 5 of the 12 types of samples.

To compare RVA and rheometer data, shear rates were calculated from stirring speed using the effective shear rate (see section 2.3.2), and shear stress was calculated using the measured viscosity and calculated shear rate values.

3.7 Rheometer Flow Curve Measurements

To measure shear stress as a function of shear rate, a stress-controlled rotational rheometer (Kinexus Pro, Malvern Instruments Limited, UK) was used with a vane (4V14 SS0089SS) and serrated cup (PC25G A0008 AL) geometry. Once the sample had been loaded and the machine temperature had been reached, the samples were stirred at a shear rate of 1 s^{-1} for 1 minute to ensure an equal temperature throughout the sample. There was then a 30 second pause for temperature adjustment before an up-and-down shear rate ramp was realized. The program was set to measure the shear stress at shear rates with logarithmically spaced intervals between 0.1 and 50 s^{-1} . After each analysis, the sample was visually inspected to make sure there was no significant sedimentation, localized shearing, or particle migration. The sequence is illustrated in Figure 3.2.

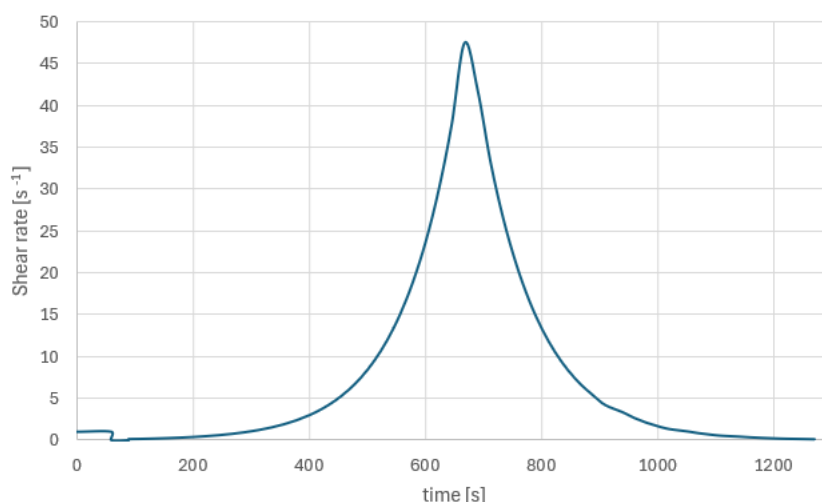


Figure 3.2. Rheometer sequence illustration

To make sure the results were reproducible, only data from the downward ramps were used to calculate the yield stress, as well as Herschel-Bulkley and Bingham plastic parameters. The samples that were measured with up and down-shear rate ramps are listed in Table 3.1.

Table 3.1. Samples measured with an up-and-down-shear rate ramp on a rheometer.

TS [%]	High Mw		Medium Mw		Low Mw	
14	25 °C	55 °C	25 °C	55 °C		
16	25 °C	55 °C	25 °C	55 °C	25 °C	55 °C
18	25 °C	55 °C	25 °C	55 °C	25 °C	55 °C
20			25 °C	55 °C		
22				55 °C	25 °C	55 °C

3.8 Rheometer Oscillatory Amplitude Strain Sweeps

Oscillatory amplitude sweeps were carried out using serrated plate geometry (PLCS65 C0002 SS with PU40 X L5776 SS) with a rheometer (Kinexus Pro, Malvern Instruments Limited, UK). The gap width was set to 3.5 mm and rapeseed oil was applied to the edges of the sample after trimming to avoid evaporation. The frequency was kept constant at 1 Hz, and the strain amplitude was set to sweep from 0.01 % to 100%. After each measurement, the samples were visually inspected to verify that they were intact. The amplitude sweeps were performed in duplicates, except on one sample, which was performed in triplicate, at 25 °C, both on samples with no enzyme treatment (high BG Mw) and on samples with completely hydrolysed BGs (Low BG Mw).

The apparent yield stress was assumed to be the shear stress at the end of the linear viscoelastic region. The storage modulus in the LVR was calculated as the average of the first four

datapoints and the end of the linear region was considered to be where the storage modulus had decreased by 10 % compared to the LVR.

3.9 Functional Properties of Dried Oat Drink By-product

To analyze the functional properties of the by-product, the samples were first treated according to the *rheometer samples* method in section 3.3, after which they were dried in an oven at 85 °C for approximately 1.5 hours. After drying, the samples were milled using a food mixer (Puro S, Bianco di Puro, Germany) for 30 seconds. Finally, the samples were dried once more until reaching a dry matter level of at least 90 %.

Particle Size Measurements

To account for the potential impact of differences in particle size between the samples, they were analyzed in duplicates using a Mastersizer 3000+ (Malvern, Canada). The dried ODBP was added directly to milli-Q water in the master sizer.

Water and Oil Holding Capacity

The water and oil holding capacity was measured through the method described by Badia-Olmos, et al. (2023). 30 ml of water and 30 ml of rapeseed oil were added to 3 g of sample respectively. The samples were mixed with a vortex and let to rest for two hours. The oil-holding-capacity samples were put in room temperature and then centrifuged for 15 minutes at 1000 g. The water-holding-capacity samples were placed to rest in a 60 °C water bath and then centrifuged at 3000 g for 15 minutes. The centrifuge used was a Centrifuge 5804 R (Eppendorf, Germany). Measurements were performed in duplicates. The supernatant from the water samples was collected for water-solubility-index measurements, while the oil supernatant was discarded. The pellets were then weighed and the water and oil holding capacities were calculated according to Equation 11.

$$WHC = \frac{m_{pellet} - m_{sample}}{m_{sample}} \quad OHC = \frac{m_{pellet} - m_{sample}}{m_{sample}} \quad (11)$$

Swelling Capacity

The swelling capacity was analyzed with the method described by Badia-Olmos, et al (2023). 1 g of sample was mixed with 10 ml of water in graded test tubes. The samples were then vortexed and let to rest at room temperature for 24 hours. The measurements were made in duplicates in all but one sample. The total volume was then used to calculate the swelling capacity through dividing by the original sample weight, as described in Equation 12.

$$SWC = \frac{V_{sample}}{m_{sample}} \quad (12)$$

Water Solubility Index

The solubility index was analyzed using the method suggested by Choi, et al. (2012). The supernatants collected from the water holding capacity-measurements, see above, were poured onto pre-weighed aluminum dishes and dried overnight in a hot air oven at 105 °C. The measurements were performed in duplicates. The samples were then weighed, and the water solubility index was calculated according to Equation 13.

$$WSI = \frac{m_{dissolved\ solids}}{m_{sample}} \quad (13)$$

3.10 Data Analysis

Model fit

The flow curve data from both RVA and rheometer measurements were fitted to both Herschel-Bulkley and Bingham plastic models using the function `scipy.optimize.curve_fit` in Python. Bounds for the non-linear least square minimization were set to $[0, \infty]$, $[10^{-9}, \infty]$ and $[0, \infty]$ for τ_0 , k or η_p (depending on which model was used) and n , respectively. Because the shear rates were measured at logarithmically spaced intervals during the rheometer flow curve measurements, the data points were weighed by the inverse of the local data point density. The data points were also weighed so that the standard error would be proportional along the y-axis, assuming the absolute error is greater at lower shear rates. The resulting graphs were plotted using Python.

Statistical Analysis

All statistical analyses were performed using Minitab Statistical Software 22. Regression Analysis of Herschel-Bulkley and Bingham plastic model parameters as dependent variables were performed, with temperature, total solids and BG molecular weight as independent variables. Linear and first-order cross terms were included in the model and a stepwise, backward elimination algorithm with α to remove of 0.05 was used to identify statistically significant relationships. Interaction and contour plots were also generated using the same software.

Similarly, regression analysis was performed on the functional property results, but due to an unforeseen confounding impact from particle size, cross-terms had to be omitted. ANOVA with a following Tukey test was also performed on the functional property results.

3.11 Microscopy

To examine the microstructure of the ODBP samples and analyze the difference of the enzyme treatment, microscopy pictures, both brightfield and epifluorescence microscopy, were taken by an internal part at Oatly. A Nikon Eclipse Ci microscope was used.

Samples were first prepared as described in section 3.9 *Functional Properties of Dried Oat Drink By-product* and subsequently rehydrated in water overnight. The brightfield microscopy pictures were then taken of the samples directly mounted onto the microscope slide.

To visualize the effect of the enzyme treatment on the BG, the soluble phase of the samples was stained with Calcofluor White and imaged using a DAPI filter set.

4 Results

4.1 Texture Analysis

From the Texture Analyzer measurements of fresh ODBP, an interaction plot of the average hardness 1 values from two replicates for all types of samples was generated to illustrate the effects of dilution, storage time, and temperature. The results are presented in Figure 4.1.

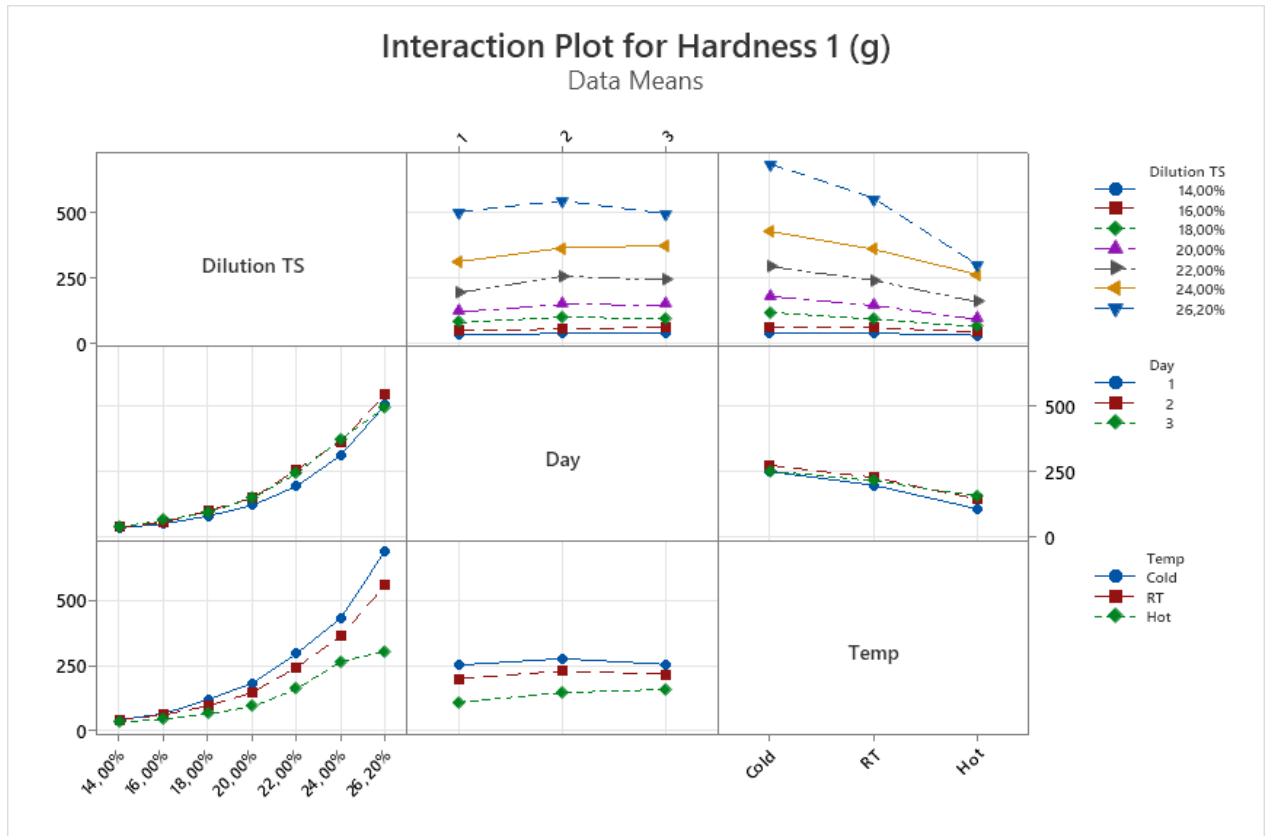


Figure 4.1. Interaction plot for hardness 1 from Texture Analyzer measurements carried out on fresh ODBP samples, days 1, 2 and 3. Each point is an average of two measurements.

Interaction plots of the other measured parameters from the texture analyzer were also plotted (see Appendix 9.1) although these are not thoroughly discussed in this thesis.

Hardness 1 was also measured on freeze-thawed samples without enzyme treatment (BG Mw average 235 kDa) at 26 % TS and with high enzyme treatment (BG not detectable) at 18%. In Figure 4.2, the hardness 1 of the freeze-thawed samples is plotted together with the hardness 1 values from comparable fresh samples.

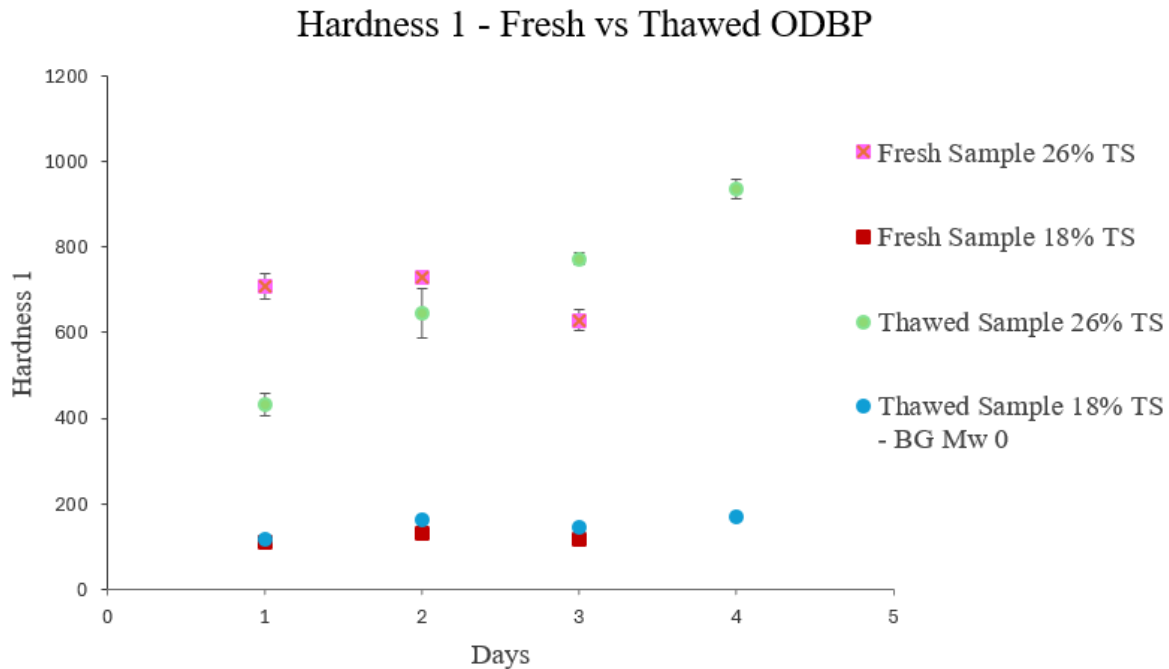


Figure 4.2. Hardness 1 for fresh and freeze-thawed ODBP samples with 26 and 18% TS. The thawed sample has no detectable amount of BG. The fresh samples were measured for 3 days, while the thawed samples were measured for four days. Each point is an average of two measurements.

4.2 Flow Curves Measurements

Supplemental flow curves from the RVA and rheometer can be found in Appendices 9.2 and 9.3.

Examples of the difference between rheometer samples with similar TS content yet different BG Mw and vice versa, are presented in Figures 4.3 and 4.4.

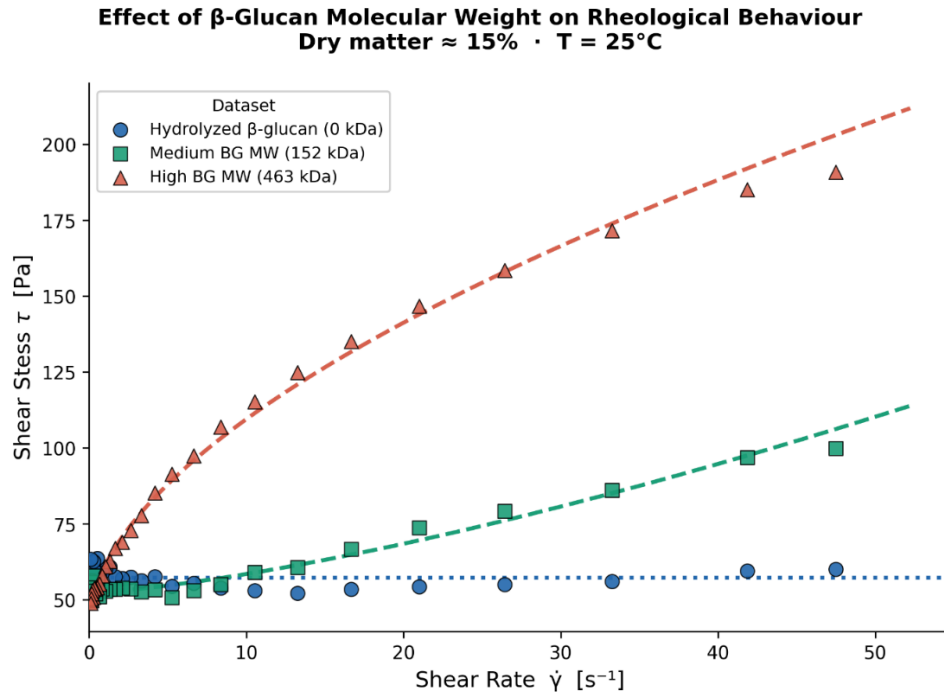


Figure 4.3. 3 flow curve measurements of ODBP samples with approximately 15 % TS and varying BG Mw at 25 °C on a rheometer. Each point is one data point. The raw data is fitted to the HB fluid model and plotted on a linear scale.

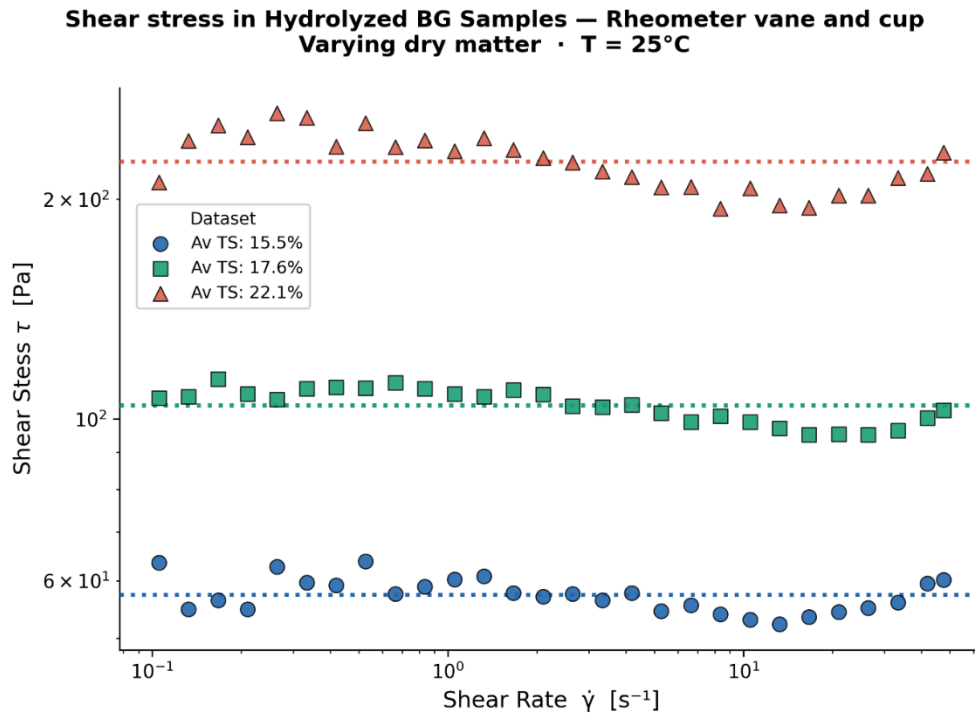


Figure 4.4. 3 flow curve measurements of ODBP samples with no detectable BG with varying TS content at 25 °C on a rheometer. Each point is one data point. The raw data is fitted to the BP fluid model and plotted on a logarithmic scale.

A comparison between one flow curve measured on the RVA and one analyzed on the rheometer is shown on a logarithmic scale in Figure 4.5. Both measurements have been carried out at 55 °C with similar TS content around 17%. None of the samples have been treated with lichenase enzyme, although the rheometer sample has an average BG Mw of 463 kDa while the average in the RVA sample is 235 kDa. In both cases, the raw data from the two measurements has been fitted to the HB fluid model.

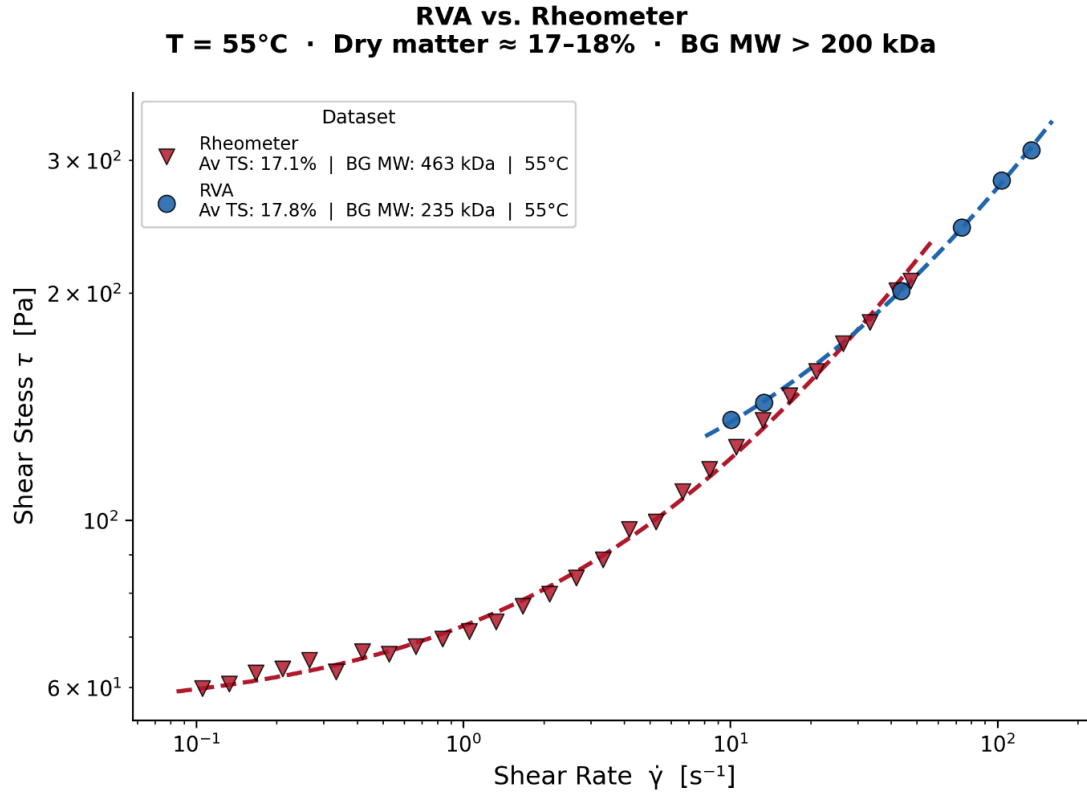


Figure 4.5. Flow curve of untreated ODBP diluted to approximately 17 % TS at 55 °C carried out once with a rheometer (red) and once on an RVA (blue). Each point is one data point. Raw data is fitted to the Herschel-Bulkley fluid model.

Using RVA measurement data, the effect of different temperatures on flow behavior and the difference in model fit between the HB and BP models on untreated ODBP are illustrated in Figures 4.6 and 4.7. Figure 4.6 is plotted on a logarithmic scale, while Figure 4.7 is plotted on a linear scale, hence the difference in the shape of the curves.

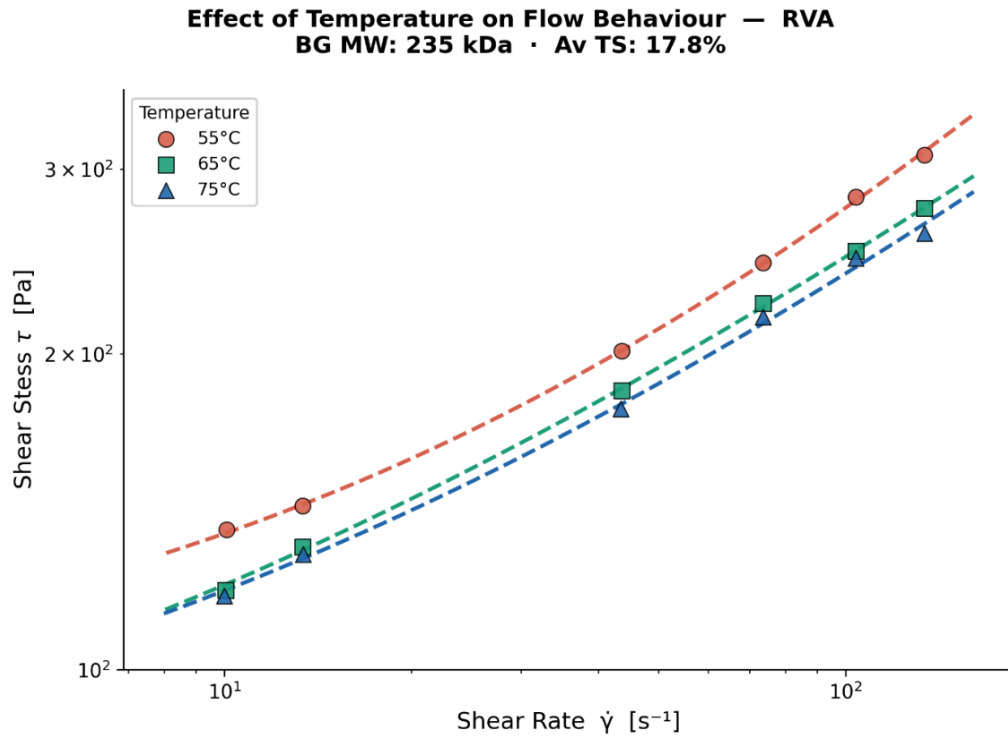


Figure 4.6. Flow curve raw data and HB model fit of untreated samples with 17.8% TS. Measured on an RVA at different temperatures. Each point is one data point.

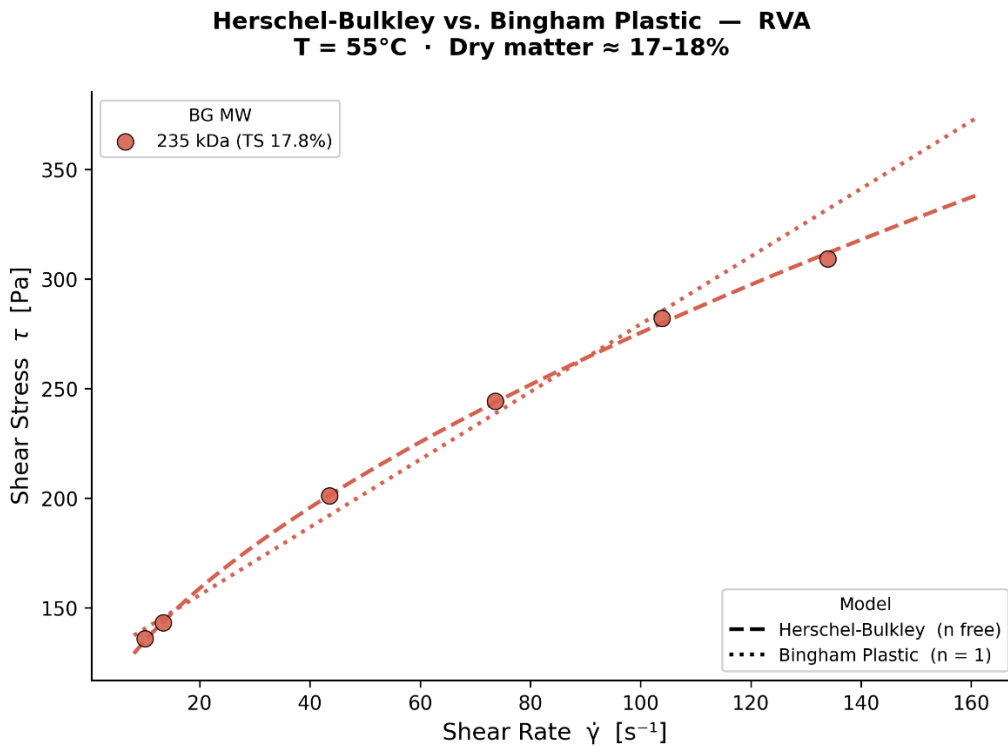


Figure 4.7. RVA flow curve of (enzymatically) untreated samples together with BP and HB model fit of untreated samples with 17.8% TS. Each point is one data point.

4.3 Flow curve Model fit

The τ_0 obtained from the HB and BP fluid models, for both rheometer and RVA measurements at 55 °C are plotted against TS in Figure 4.8.

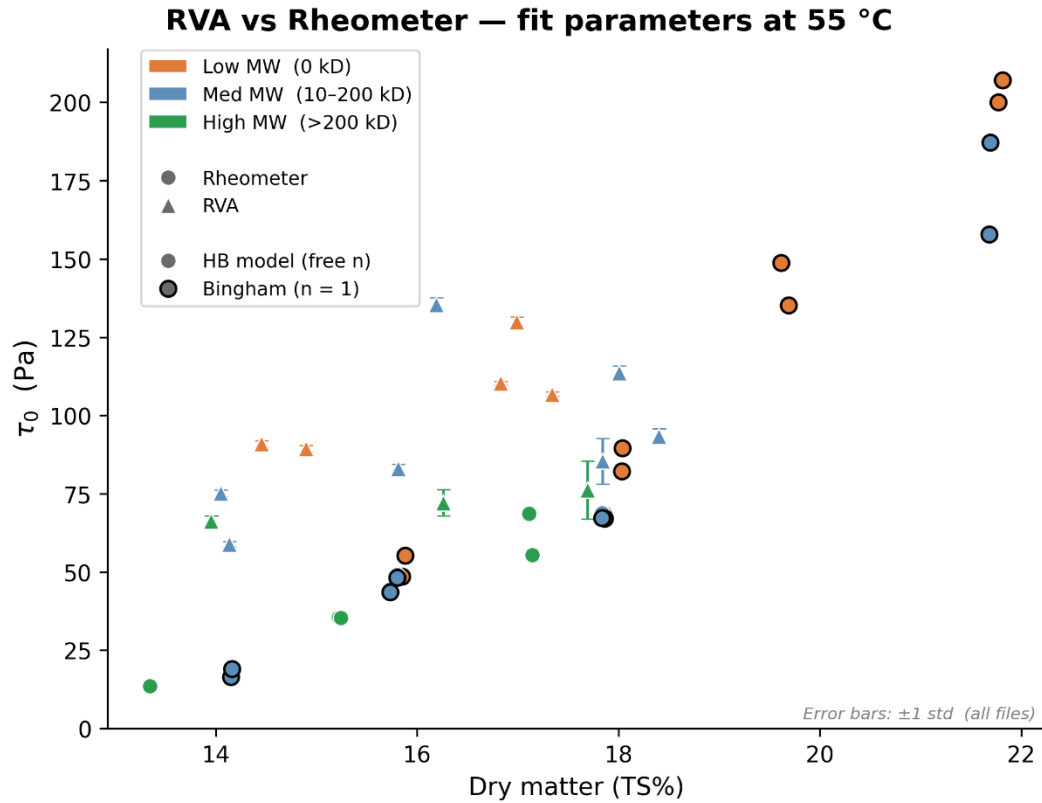


Figure 4.8. Apparent yield stress as a function of TS for rheometer and RVA measurements at 55 °C on samples with different BG Mw. The apparent yield stresses are calculated from fitting individual flow curve measurements to the Herchel-Bulkley and Bingham plastic fluid models. The error bars are based on the model fitting error.

In Figure 4.9, the apparent yield stress values for rheometer measurements at two different temperatures are shown. The results indicate a slight temperature effect on the apparent yield stress. Each point is calculated from a singular flow curve measurement.

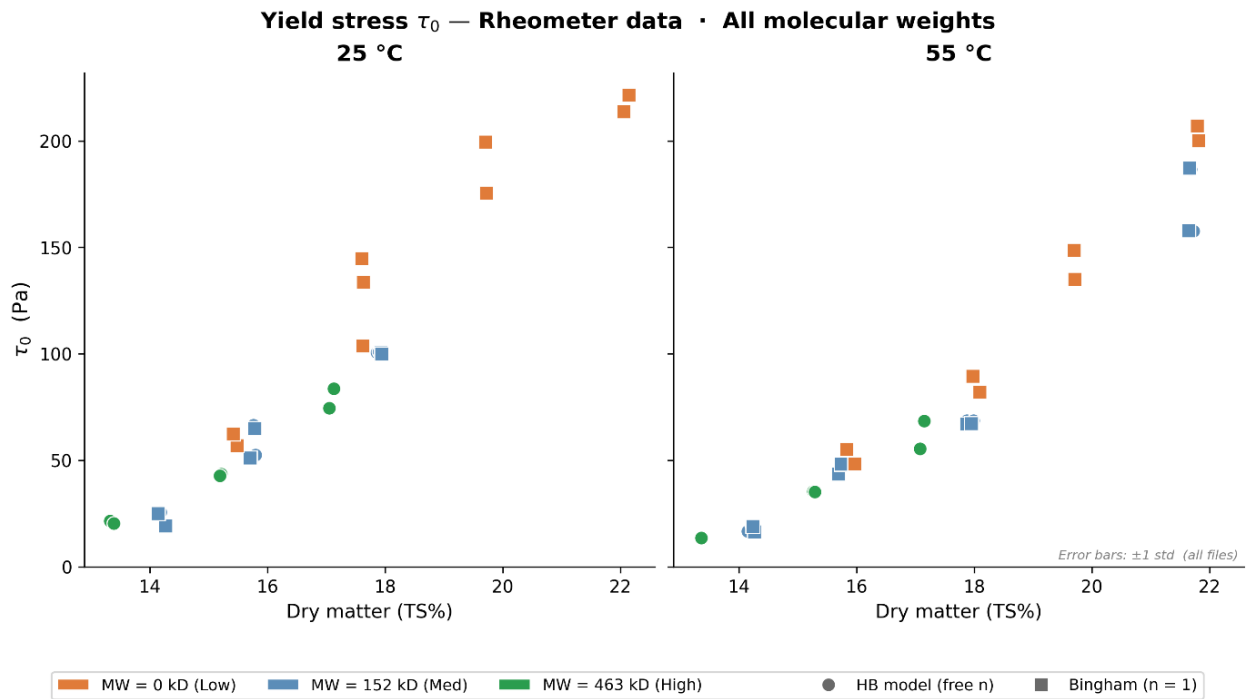


Figure 4.9. Apparent yield stress as a function of TS. for measurements carried out with a rheometer at 25 and 55 °C on samples with different BG Mw. The apparent yield stresses are calculated from fitting individual flow curve measurements to the Herchel-Bulkley and Bingham plastic fluid models. The error bars are based on the model fitting error.

4.4 Parameter statistics

4.4.1 Rheometer

The effect of TS, BG Mw and temperature and cross terms on HB and BP model parameters for the rheometer measurements were analyzed with a statistical regression model. The significant terms (α to remove = 0.05) and their respective P-values are presented in Tables 4.1, 4.2 and 4.3. Since both models generally fit poorly to raw data from samples with no detectable BG Mw (see Appendix 9.5), parameters estimated from those samples were excluded from the statistical analysis presented below.

Table 4.1. Coefficients and P-values from statistical regression analysis of apparent yield stress τ_0 from HB and BP models fitted to rheometer raw data.

τ_0 HB			τ_0 BP		
Factor	Coefficient	P-Value	Factor	Coefficient	P-Value
Temperature	-0.343	0.024	Temperature	-0.579	0.001
TS	18.286	0.000	TS	19.64	0.000
			BG Mw	0.0489	0.006

Table 4.2. Coefficients and P-values from statistical regression analysis of consistency index, k and plastic viscosity, η_p , from HB and BP models fitted to rheometer raw data.

k HB			η_p BP		
Factor	Coefficient	P-Value	Factor	Coefficient	P-Value
Temperature	1.543	0.005	Temperature	-0.0164	0.173
BG Mw	-0.1853	0.002	BG Mw	-0.01369	0.012
TS	1.41	0.335	TS	0.3186	0.000
Temperature*BG Mw	-0.001973	0.000	Temperature*BG Mw	-0.000075	0.050
Temperature*TS	-0.0784	0.007	BG Mw*TS	0.001621	0.000
BG Mw*TS	0.02054	0.000			

Table 4.3. Coefficients and P-values from statistical regression analysis of flow behavior index, n , from the HB model fitted to rheometer raw data.

n HB		
Factor	Coefficient	P-Value
Temperature	0.000646	0.015
BG Mw	-0.002174	0.000
TS	-0.0499	0.006

4.4.2 RVA

Tables 4.4, 4.5 and 4.6 present the significant terms (α to remove = 0.05) and their respective P-values from the statistical regression analysis of the HB model parameters based on RVA measurements. Because the BP model exhibited a poor fit to the RVA raw data (see Appendix 9.5), these parameters were excluded.

Table 4.4. Coefficients and P-values from statistical regression analysis of apparent yield stress, τ_0 , from the HB model fitted to RVA raw data.

τ_0 HB

Factor	Coefficient	P-Value
TS	9.45	0.000
Temperature	-1.073	0.000
BG Mw	0.458	0.036
TS*BG Mw	-0.0359	0.011

Table 4.5. Coefficients and P-values from statistical regression analysis of consistency index, k , from the HB model fitted to RVA raw data.

k HB

Factor	Coefficient	P-Value
TS	-0.301	0.112
BG Mw	-0.1136	0.000
TS*BG Mw	0.00851	0.000

Table 4.6. Coefficients and P-values from statistical regression analysis of flow behavior index, n , from the HB model fitted to RVA raw data.

n HB

Factor	Coefficient	P-Value
BG Mw	-0.00876	0.000

To illustrate the combined effect of BG Mw and TS on calculated apparent yield stress values, based on the HB model, a contour plot was generated. The result is shown in Figure 4.10.

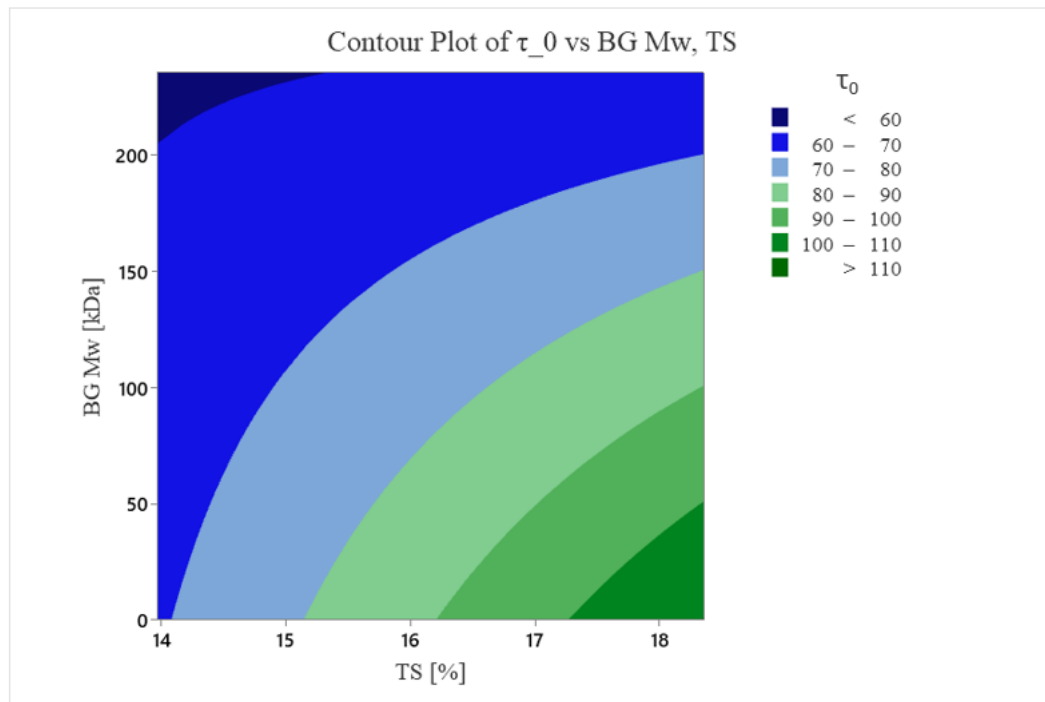


Figure 4.10. Contour plot of τ_0 with BG Mw vs TS from RVA measurements at different temperatures.

4.5 Amplitude Sweep

The average apparent yield stresses and their respective standard deviations from multiple amplitude sweep measurements are shown in Table 4.7. In the same table, τ_0 values obtained from the HB model fitted to rheometer data from comparable experimental conditions are also presented.

Table 4.7. Apparent yield stress with standard deviations from samples with varying TS and BG Mw. The values are calculated from amplitude sweeps and flow curve measurements on a rheometer fitted to the HB model.

Amplitude sweep				Rheometer Flow curve			
BG Mw [kDa]	TS [%]	τ_0 [Pa]	τ_0 STD	BG Mw [kDa]	TS [%]	τ_0 [Pa]	τ_0 STD
504	17.1	14.2	7.0	463	17.1	79.1	6.4
504	22.0	34.7	3.4				
504	26.0	90.5	21.7				

0	17.4	24.0	3.4	0	17.6	118.2	20.4
0	22.3	50.3	3.3	0	22.1	213.5	11.3
0	25.9	114.9	65.6				

4.6 Functional Properties

The results from the functional property analyses are shown in Figure 4.11. The different letters indicate a statistically significant difference according to the Tukey test. The colors of the bars indicate the average particle size of the sample. The results are corrected for dry TS content.



Figure 4.11. Oil holding capacity, swelling capacity, water holding capacity and water solubility index for ODBP samples that have been dried from different TS levels and with different BG Mw. All bars except one represent duplicate measurements. The swelling capacity for ODBP with high BG Mw and 22 % TS was measured once. The letters indicate statistical significance according to the Tukey test.

4.7 RVA and Rheometer - Method development

Before executing the flow curve measurements, the experimental conditions in which it was practically possible to generate accurate, and practically relevant results, had to be identified. This experimental window was shown to be different for the rheometer and RVA.

The TS content for the RVA measurements was set between 14 and 18%. More diluted samples were not deemed relevant for practical applications and less diluted samples generated too much resistance for the motor. At 18% TS, the maximum torque on the RVA was reached at higher rotational speeds than 400 RPM. Therefore, 400 was chosen as the maximum speed for all samples. At rotational speeds lower than 30 RPM, the rotation became uneven, and 30 was therefore chosen as the lowest RPM to be measured.

The temperatures in the rheometer measurements were set to 25 °C, to limit issues related to temperature changes, e.g. uneven temperature distribution or evaporation, and 55 °C, which is practically relevant for food production.

Serrated parallel plate geometry was tested with different gap widths for flow curve measurements. The results showed that even at 10 s⁻¹ the sample would flow out from the edges of the plates. It would thus be impossible to measure higher shear rates without introducing severe artifacts. However, solely analyzing shear rates lower than 10 s⁻¹ would limit the practical relevance of the results. Therefore, vane-and-cup geometry and 50 s⁻¹ as the maximum shear rate, were chosen for the rheometer-flow curves.

Although the vane-and-cup geometry eliminated the risk of samples flowing out to the sides, the risk of shear localization and particle migration was introduced. The minimum and maximum TS content and temperature for each level of enzyme dose were therefore determined with visual inspection from empirical trials. Measurements which were not deemed reliable were discarded, hence, the difference in TS and temperature range for samples with different BG Mw.

4.8 Microscopy

The brightfield microscopy pictures of ODBP samples with no enzyme treatment and samples treated with high enzyme doses show bran particles with identifiable aleurone cell layers, pericarp tissue fragments, and oil droplets. Two of the pictures, one of each sample is shown in Figure 4.12.

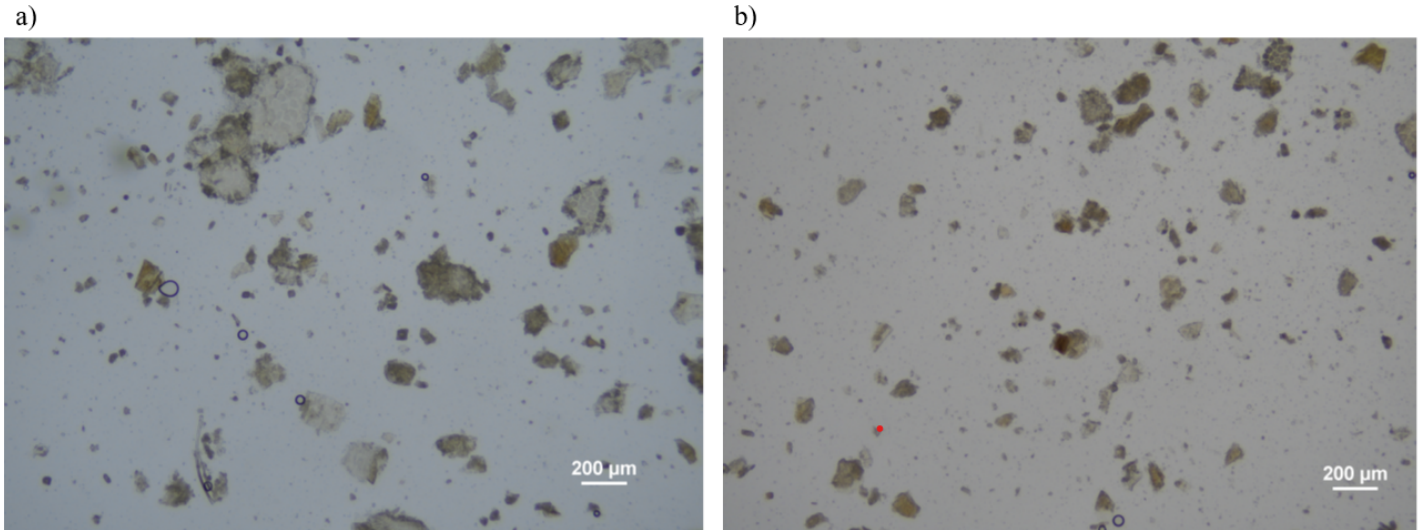


Figure 4.12. Brightfield Microscopy Pictures of ODBP samples with a) no enzyme treatment b) high enzyme treatment.

The calcofluor-stained samples, shown in Figure 4.13, displayed more significant differences between the different samples. Below is one ODBP sample treated with lichenase and one that has not been treated. The blue spots are likely BG aggregates.

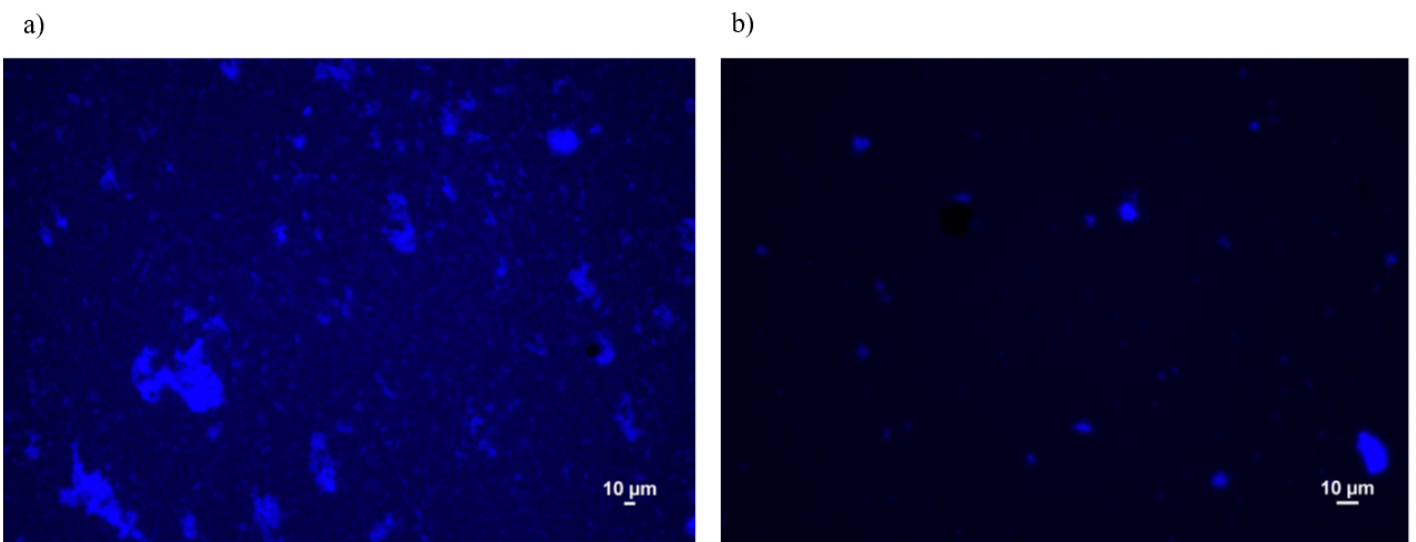


Figure 4.13. Calcofluor stained ODBP samples with a) no enzyme treatment b) high enzyme treatment.

5 Discussion

5.1 Texture Analysis

The interaction plots of the fresh ODBP samples seen in Figure 4.1, demonstrate that hardness decreased with decreasing TS content. However, the relationship does not appear to be linear, as the decrease in hardness per TS is lower at lower TS levels. Furthermore, samples stored at the lower temperature exhibit greater hardness values than those stored at room temperature, while heated samples show the lowest hardness values. The influence of storage temperature on hardness also appears to diminish with increasing dilution, indicating that the temperature has a smaller effect on the most diluted samples. Yet, no clear trend in hardness is observed over the storage period, implying the storage effect on fresh ODBP is limited with either storage temperature.

In contrast, in Figure 4.2 there is a clear trend for freeze-thawed samples containing BG in which the hardness increases each day. Such a tendency is not observed for the samples treated with lichenase, as these samples appeared to exhibit similarly small changes in hardness as the fresh samples. It should be noted that only four measurements per day were performed for the freeze-thawed samples, limiting the statistical strength of the observation. Furthermore, it remains unclear whether the observed time-dependent changes in hardness were primarily influenced by variations in TS content or by the BG Mw. However, previous studies have shown that BGs from barley, which have the same molecular structure as oat BG, develop network structures after freeze-thawing, altering sample viscosity (Vaikousi & Biliaderis, 2005). The same mechanism might be present in ODBP and could potentially be responsible for the changes observed in hardness 1.

In summary the hardness 1 results from the texture analysis indicate that TS content and freeze-thawing both effect the ODBP-texture. The other measured parameters from the texture analyses showed similar overall patterns and variations as hardness 1 (see Appendix 9.1). However, more detailed analysis of how storage conditions and enzyme treatment affect the different measured parameters, hardness 2, cohesiveness, springiness and adhesion are outside the scope of this thesis.

5.2 B-glucan, TS and Temperature Effects on Flow Curve Measurements

Overall, it was observed that samples containing BG, especially longer fiber chains, exhibit clear shear thinning behavior, whereby the shear stress does not increase linearly with the shear rate. This pattern was observed consistently in both RVA and rheometer experiments. It can for example be seen in Figure 4.3 and is further supported by the flow behavior index, n values (see Appendix 9.5) that remain below 1 for all untreated samples. This trend is also consistent with previous studies on BG, which persistently show that BGs form shear-thinning matrices (Du, et al., 2019). The behavior could perhaps be explained by both the entanglement of long fiber chains and the hydrogen bonds between the many hydroxyl groups on the BG, which form a structural network at rest and low shear. When shear is applied, these bonds would then be broken, and the BGs could start to slide more easily past each other, generating a shear-thinning behavior.

A pronounced effect of the enzyme treatment, reducing BG molecular weight, on the ODBP flow behavior can be seen both qualitatively in the flow curves (see Figure 4.3) and

quantitatively from Tables 4.2, 4.3, 4.5 and 4.6 (where $p < 0.05$ for all n and k -values). The untreated and least diluted samples display characteristic HB fluid behavior, whereas samples with low BG Mw exhibit considerably flatter flow curves. One possible explanation for this observation could be that BG may contribute to the formation of entangled structures which increase the apparent viscosity at lower shear rates. In some cases of samples with low BG Mw, the shear stress increases more than the shear rate, indicating shear thickening behavior. This effect is particularly prominent in the rheometer measurements, where the analysis was performed at lower shear rate intervals. This observation might indicate the presence of thixotropic effects, meaning there is time-dependent structural rebuilding of the sample at low shear rates. This restructuring of the sample might induce a downward deviation of the flow curve at low shear rates, generating a shear-thickening pattern in the flow curve.

When analyzing the rheometer measurements of samples containing highly hydrolyzed BG, see Figure 4.4, it appears as though the shear stress is elevated at the very lowest shear rates, before decreasing, only to increase again at higher shear rates, creating a ‘dip’ or ‘s-shaped’ flow curve. Since the analyses were performed by measuring shear stress as the shear rate decreases, these flow curves indicate a structural build-up of the sample at the lowest measured shear rates. Similar observations were also made on some of the RVA measurements, confirming the consistency of the pattern. Previous studies have shown that oat BGs with lower Mw have shorter gelation times compared to BG with higher Mw (Du, et al., 2019). This could perhaps be an indication that samples with low BG Mw have faster restructuring at low shear rates compared to samples with high BG Mw. In the rheometer measurements, the observed s-shaped curves appear to become more pronounced as the TS content increases. This could indicate more structural buildup in samples containing less water, which might also be explained by higher TS content, possibly causing more interaction or entanglement between the particles in the sample.

A clear temperature effect was also observed across measurements, where higher temperatures generated lower shear stresses. As can be seen in Figure 4.6, the effect of different temperatures on the shear stress was not linear. Instead, the effects of increasing the temperature were larger at lower temperatures. This was consistent with the effects seen on hardness 1 from the Texture Analyzer measurements. For practical applications, this would imply that increasing the temperature more than necessary for food safety purposes would only marginally increase any benefits in terms of decreased required shear.

Although the plastic viscosity, obtained from the BP model, and the consistency index derived from the HB model, describe related rheological properties, the parameters are not directly comparable. This could partly explain the smaller discrepancies in how these parameters are affected by BG Mw, TS content and temperature, seen in the variance analysis Table 4.2.

5.3 B-glucan, TS and Temperature Effects on Yield Stress Measurements

Although the apparent yield stress substantially varies depending on the analytical techniques used, as can be seen in Table 4.7, where comparable samples are listed, there are clear patterns in the obtained values across all experimental methods.

The TS content appears to exert the most important influence on the apparent yield stress. This trend is confirmed by the variance analyses presented in Tables 4.1 and 4.4, where the coefficients for TS are 18.286 and 19.64 for the rheometer measurements according to the HB and

BP models, respectively, and 9.45 for the RVA measurements according to the HB model (all with $p < 0.05$). This is also visually evident in the scatter plots 4.8 and 4.9 as well as the flow curves shown in Figure 4.4. As previously discussed (see section 5.2), the decrease in water content likely creates a more densely packed sample, creating a more mechanically intertwined structure, possibly allowing more physical interactions between the particles.

BG content also appears to influence the yield stress behavior of the samples. The contour plot, generated from the regression analysis (Figure.4.10), showing the apparent yield stress values by BG Mw and TS suggest a slight increase in yield stress as the BG molecular weight decreases. This can also be observed in the amplitude sweeps (Table 4.7). However, the subtle differences between the absolute numbers, the small number of measurements, as well as the large discrepancy between the measurements of the least diluted samples, significantly limit the certainty of these observations.

Temperature also shows a minimal yet statistically significant effect on apparent yield stress, as indicated by the analysis of both RVA and rheometer measurements shown in Tables 4.1 and 4.4 ($p < 0.05$). According to these analyses, increasing the temperature by 1 °C resulted in a decrease in apparent yield stress with either 1.073 or 0.343 Pa, depending on the rheological model applied. This reduction in apparent yield stress with increasing temperature is also consistent with the observed decrease in resistance to flow at higher temperatures.

5.4 Model fit - Herschel-Bulkley versus Bingham Plastic

The HB model generally provides a good fit for samples containing BG, particularly for samples with the highest molecular weight BG fractions. This is indicated both by the flow curve plots, e.g. Figure 4.7, and the corresponding r-squared values obtained from the RVA and rheometer measurements (see Appendix 9.3). For these samples, the flow behavior index, n , remained below 1, indicating shear thinning behavior. The model also describes the partially enzymatically treated samples reasonably well, with slight differences depending on the TS level.

For samples containing no detectable BG, the HB model performance differed substantially depending on the shear rate interval examined. In the rheometer measurements, where analysis was conducted on a lower and smaller shear rate interval, the model fit very poorly (see Appendix 9.3). Analyzing the flow curves of samples lacking BG (e.g. Figure 4.4), the shape of the curve does not resemble a classic HB curve, and thus fitting such data to the HB model might lead to overfitting. In contrast, the RVA measurements showed comparatively good agreement between the HB model and experimental data at higher shear rates, even for samples lacking BG.

These observations suggest that the applicability of the HB model depends more strongly on the shear rate interval investigated, rather than on the type of sample. The investigated shear rate region must extend far enough down to determine the apparent yield stress, τ_0 , and high enough to capture any possible power law feature of the sample. This might explain the discrepancy between some of the statistical results from the model parameters between the RVA and rheometer measurements (see Tables 4.2 and 4.5). At higher shear rates, the HB model

appears suitable for describing ODBP samples regardless of BG Mw, whereas at lower shear rates, the model only adequately describes samples containing high molecular weight BG.

As a result of a too narrow experimentally investigated shear rate region, there can be a rather high covariance between the fitted parameters. In turn, this may lead to an underestimation of the standard error in the fitted parameters. Consequently, although the r-squared and standard error values (see Appendix 9.5) might indicate a strong model fit, the n and k values should be interpreted with caution.

In comparison, the BP model generally provides a poorer fit to the RVA measurements (see Appendix 9.2). This can, for example, be seen in Figure 4.7 and may partly be attributed to the previously discussed broader shear rate range covered by the RVA measurements, which captures an upward curvature in shear stress at higher shear rates that cannot be adequately represented by the linear BP model. This stands in contrast to rheometer measurements, where less shear thinning is observed within the shear rate range measured. At these shear rates, the limited flexibility of the BP model becomes less of a liability, also limiting the risk of overfitting.

5.5 Rheological measurement methods - RVA vs Rheometer

In general, the measurements carried out on the RVA and rheometer showed similar results when analyzing the same shear rate intervals. An example of this can be found in Figure 4.5. The differences that could be observed were most prominent in samples containing no BG, where the RVA measurements were quite uneven. Several methodological factors may contribute to these discrepancies, including differences in temperature control, sample loading and history.

Furthermore, the applied shear rate is not even across the sample in these experimental setups. Under idealist conditions, the vane and cup geometry can give theoretically correct shear rates, but for complex, real-life samples the underlying assumptions are most likely not strictly valid, and the calculated shear rates must be seen as empirical. For the RVA setup an average is instead estimated using an empirically derived effective shear rate proposed by Lai, et al., (2000). This is because the paddle in the RVA is designed to mix the sample to simulate food processing, while rheometer geometries are generally intended to obtain as ‘ideal’ conditions as possible. While the mixing aspect of the RVA geometry generates an uneven flow field and therefore limits more precise rheological measurements, it also avoids certain problems observed with the rheometer measurement, such as localized shearing (see section 4.7).

Another important difference between the two measurement techniques is that the rheometer measurements were conducted over a more controlled and precisely defined shear rate range, whereas the RVA measurements covered a broader but less rigorously defined shear rate range. As a result, the rheometer generally captured more details, especially at low shear rates, while the RVA measurements primarily reflected broader rheological trends.

Additionally, the operational differences between the instruments likely influence the observed flow behavior. The rheometer is capable of rapidly adjusting rotational speed and maintaining more precise control over shearing conditions, allowing for capturing more precise stress responses. In contrast, the RVA requires a longer time to reach the target rotational speed, which might aggravate the impact of thixotropic effects on the measured response.

5.6 Functional Properties

Overall, the trends for the functional properties are not prominent. The differences in average particle size seem to have an impact on all properties measured, which could be one of the reasons why other effects, from TS and BG Mw, are more difficult to detect. In order to properly evaluate the effects of BG Mw and wet TS, the particle size would have to be more standardized. A larger particle size seems to increase all functional properties measured except for the WSI, where it appears to have the opposite effect. This is confirmed by regression analysis ($p < 0.05$, see Appendix 9.6).

Although observing the results shown in Figure 4.11, there are still some indications of other patterns. For example, there might be a slight effect of BG Mw on WHC and WSI, where the WHC decreases while the WSI increases with higher BG Mw. This is confirmed for WHC by the (multivariate) regression analysis ($p < 0.05$, see Appendix 9.6) and seems logical, since one of the properties is measured on the pellet and the other on the supernatant of the same sample. Larger BG might be able to form more structured networks that are able to ‘trap’ large amounts of water, while smaller BG with a higher degree of polymerization could be easily soluble in water. Increasing the Mw of oat BG has previously been shown to decrease water solubility (Du, et al., 2019). On the OHC and SWC, there could potentially be an effect of TS, where both properties decrease with higher wet TS content. These effects are also supported by regression analysis ($p < 0.05$).

5.7 Microscopy

The brightfield images of the ODBP samples (see Figure 4.12) revealed no remarkable differences between the enzyme-treated and untreated samples. The different structures observed confirm the heterogeneous nature of the ODBP. The most important difference between the samples was the particle size, which was also confirmed by the particle size analysis (see section 4.6). The stained ODBP, however, revealed the presence of large molecular weight BGs, which can be seen as blue aggregates in the untreated sample and almost no large molecular weight BGs in the enzyme-treated sample (see Figure 4.13).

6 Conclusion

The aim of this thesis was to investigate the rheological and functional properties of oat drink by-product (ODBP) and the contribution of β -glucans to these properties.

The findings demonstrate that BG Mw, temperature and TS all influence the flow behavior of ODBP. Higher BG Mw consistently increased the resistance to flow and promoted shear thinning at lower shear rates. Increasing the temperature significantly lowered the apparent viscosity, although the effect was smaller at higher temperatures. Lowering the TS content also showed a strong effect on rheology, particularly by lowering the apparent yield stress.

Across the shear-rate range studied, the Herschel-Bulkley model provided the most accurate description of untreated ODBP, whereas the Bingham plastic model was adequate at low shear rates for samples containing highly hydrolyzed BG. The HB model was not suitable for describing samples with low BG Mw at low shear rates.

The measured apparent yield stress differed substantially depending on the analytical method used. Amplitude sweep test generally generated significantly lower results compared to extrapolated values from flow curve measurements, although the overall patterns remained the same between the different measurement techniques.

Comparison of measurements obtained using an RVA and a rheometer revealed similar overall patterns in the flow behavior of ODBP. The main differences between the instruments, when analyzing ODBP, were in the precision and the operational range. While the rheometer provided more detailed information and could analyze the samples at lower shear rates, the geometries limited accurate measurements at higher shear rates. The RVA, on the other hand, could measure larger shear rate intervals, although with lower precision, and avoided issues such as localized shearing. Thus, the preferred method should be selected based on the intended application.

Storage time did not measurably affect the texture of fresh ODBP, regardless of storage temperature, whereas freeze-thawing introduced detectable changes during the storage period for untreated ODBP with high TS. Though the small number of measurements limits the statistical strength of the observation.

The functional properties of dried ODBP were not shown to be strongly affected by BG Mw or TS. Instead, the average particle size in the dried samples displayed the most significant influence. In order to conclusively determine the effect of BG Mw and TS on the functional properties, the experiment would have to be repeated with a standardized particle size.

7 Future Work

The results and conclusions from this study have also uncovered areas where future work could develop further valuable insights.

Some of this work includes further analyzing the rheological properties of fresh ODBP. Since freeze-thawing was shown to have an impact on ODBP texture, investigating the flow behavior and yield stress of fresh ODBP would be of great interest for gaining knowledge about the practical applications in using ODBP for future food products.

Another interesting aspect to investigate would be the fit of ODBP rheological data to more complex fluid models. Even though the HB, and partly BP, models fit relatively well for these specific measurements, other models that consider more aspects of rheology, such as thixotropy, might provide a greater understanding of ODBP rheological behavior.

Since the results from the functional properties of dried by-product were slightly indicative of BG Mw and TS impacts, further analysis of these effects, with standardized particle sizes might contribute more conclusive results.

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9 Appendix

9.1 Texture Analysis

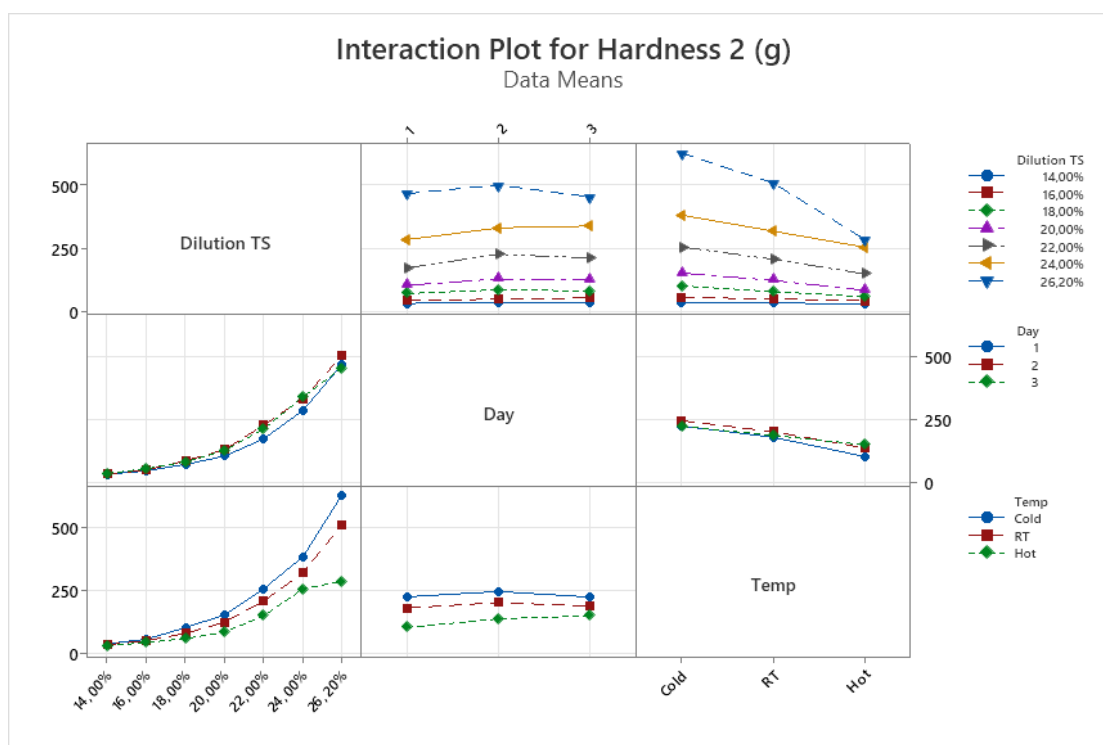


Figure 9.1. Interaction plot for hardness 2 from Texture Analyzer measurements carried out on fresh ODBP samples, days 1, 2, and 3.

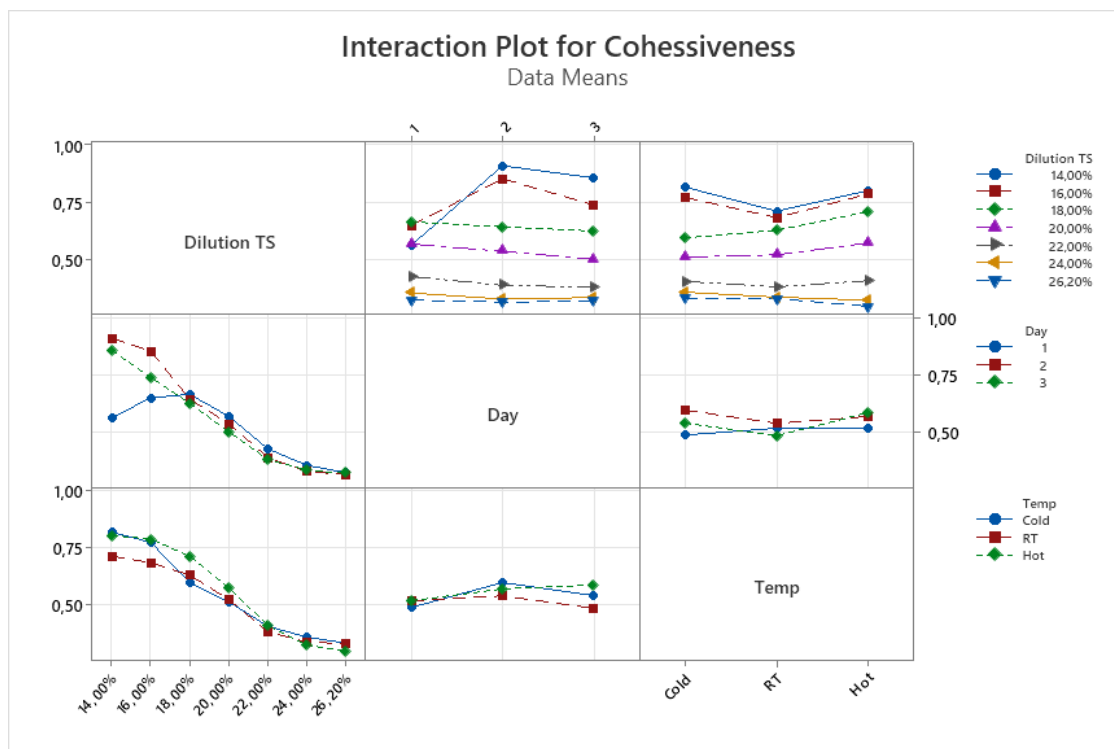


Figure 9.2. Interaction plot for cohesiveness from Texture Analyzer measurements carried out on fresh ODBP samples, day 1, 2 and 3. Measurements performed in duplicates.

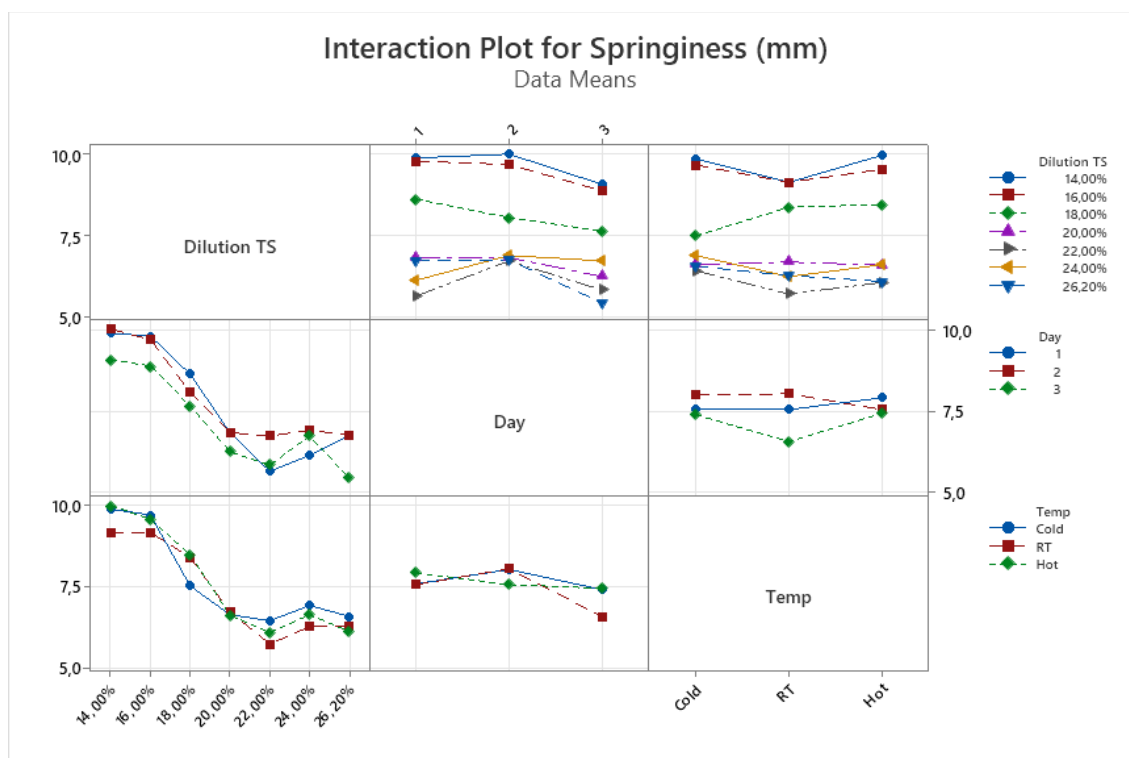


Figure 9.3. Interaction plot for springiness from Texture Analyzer measurements carried out on fresh ODBP samples, day 1, 2 and 3. Measurements performed in duplicates.

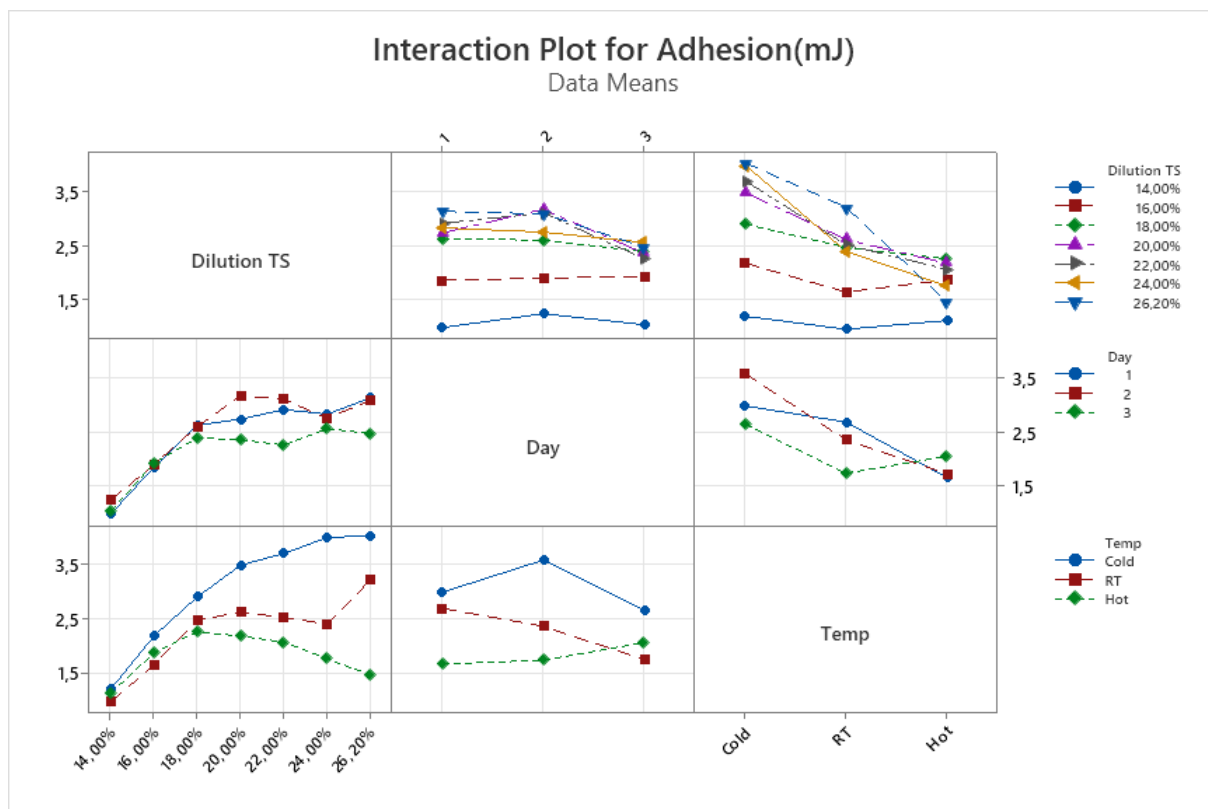


Figure 9.4. Interaction plot for adhesion from Texture Analyzer measurements carried out on fresh ODBP samples, day 1, 2 and 3. Measurements performed in duplicates.

9.2 RVA Flow curve measurements

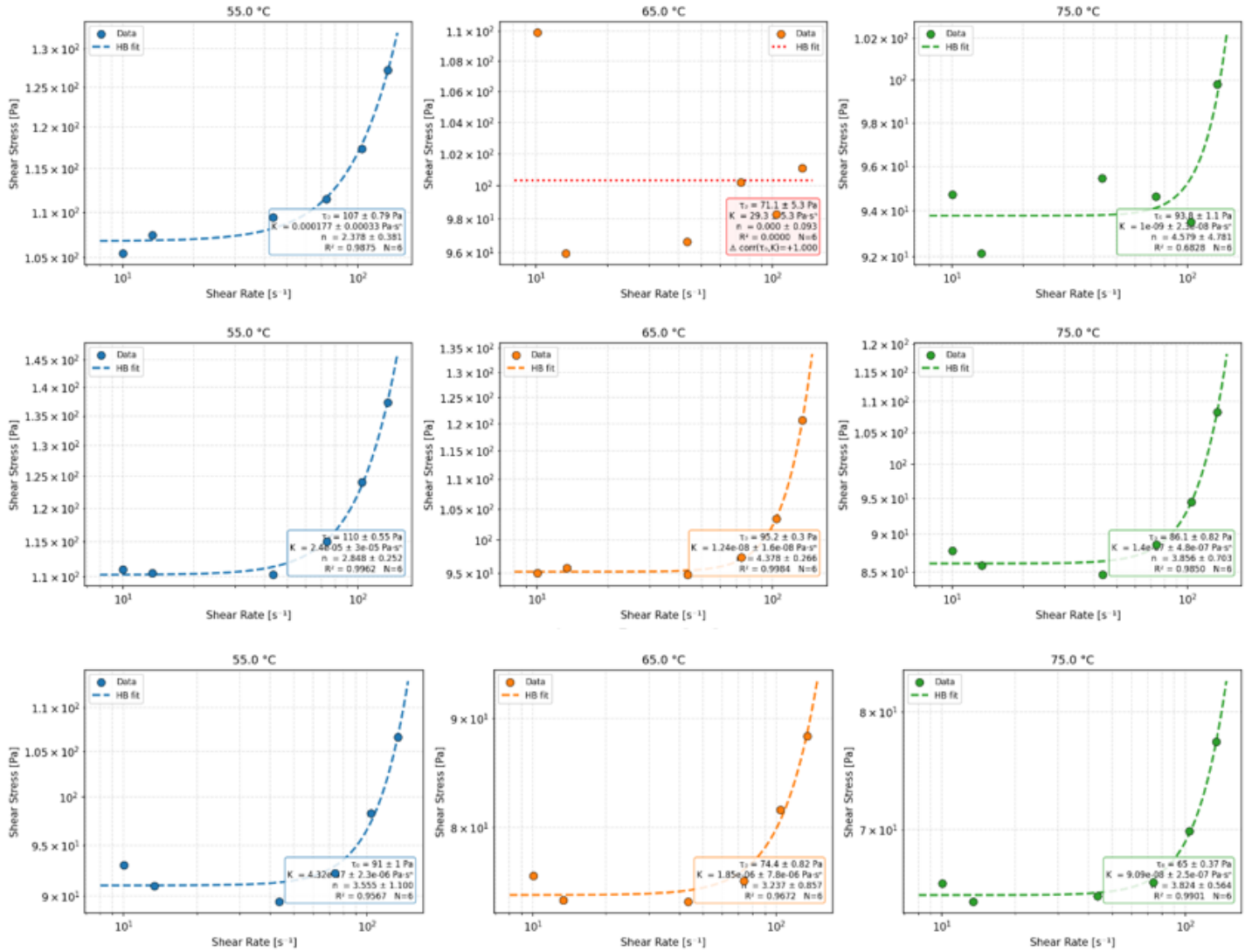


Figure 9.5. Flow curves from RVA measurements at different temperatures, 55, 65 and 75 °C from left to right. Measurements are performed on samples containing BG Mw 0 kDa with TS 17.4 %, 16.9 % and 14.5 % in the top, middle and bottom flow curve respectively.

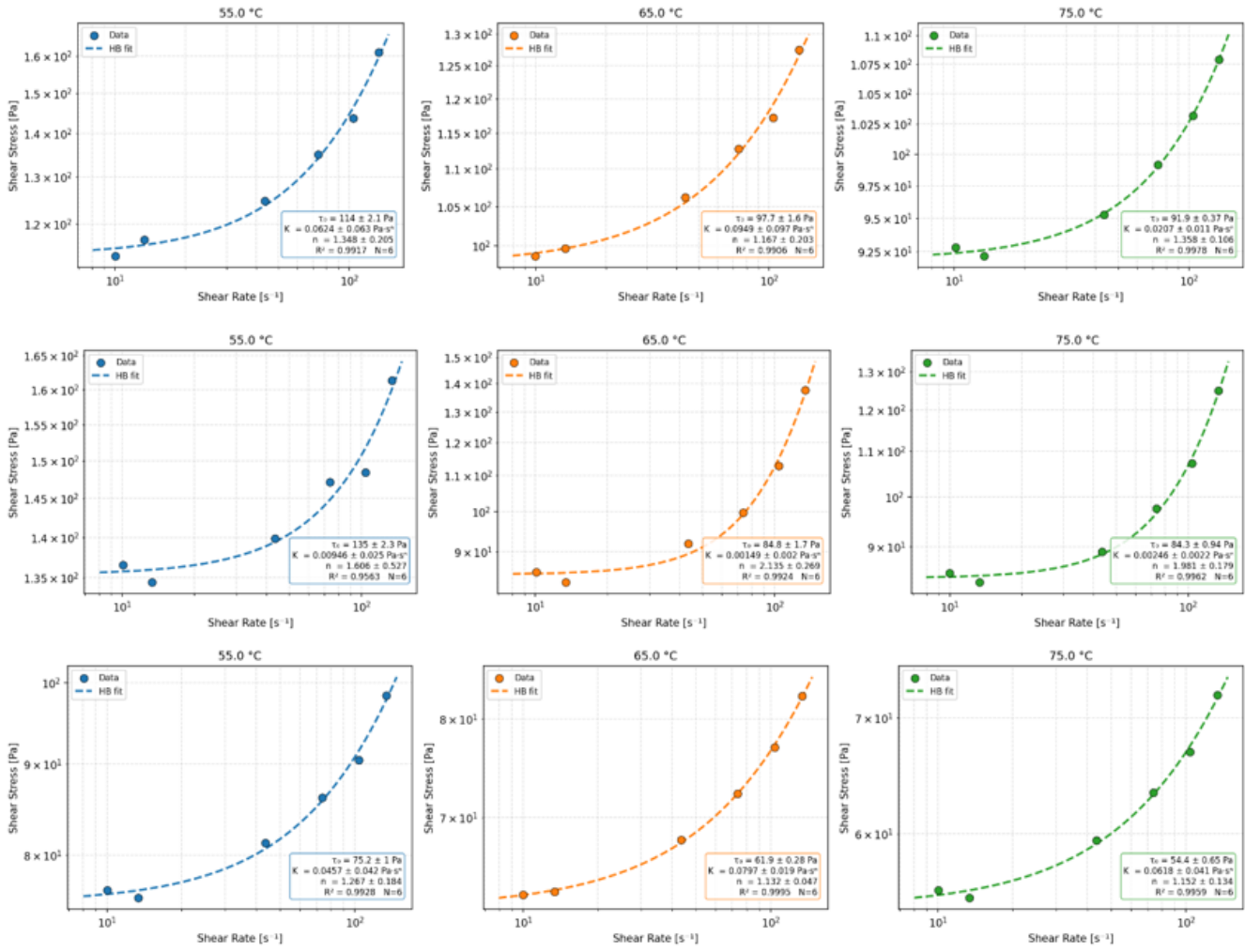


Figure 9.6. Flow curves from RVA measurements at different temperatures, 55, 65 and 75 °C from left to right. Measurements are performed on samples containing BG Mw 31 kDa with TS 18.0 %, 16.1 % and 14.1 % in the top, middle and bottom flow curve respectively.

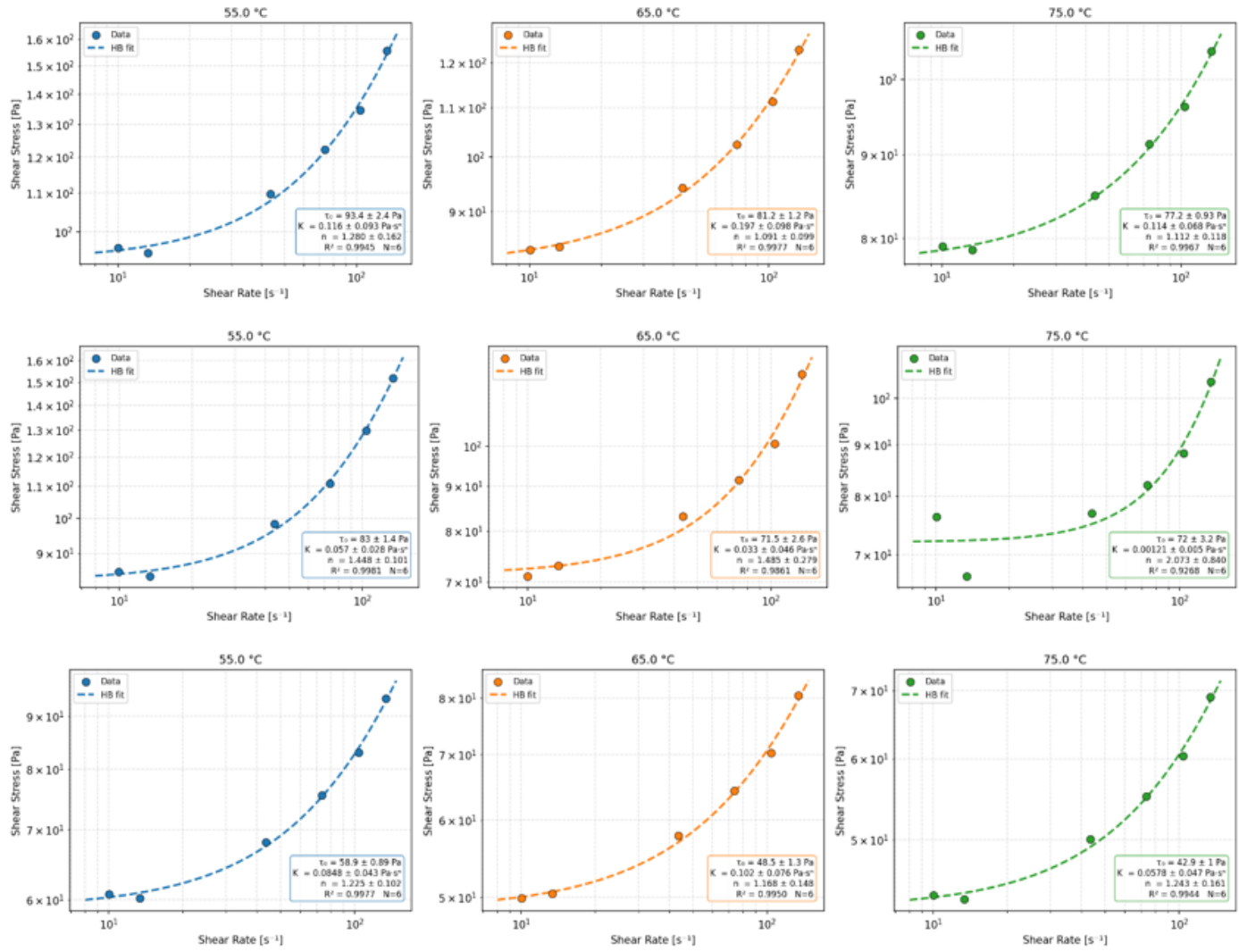


Figure 9.7. Flow curves from RVA measurements at different temperatures, 55, 65 and 75 °C from left to right. Measurements are performed on samples containing BG Mw 50 kDa with TS 18.4 %, 15.8 % and 14.1 % in the top, middle and bottom flow curve respectively.

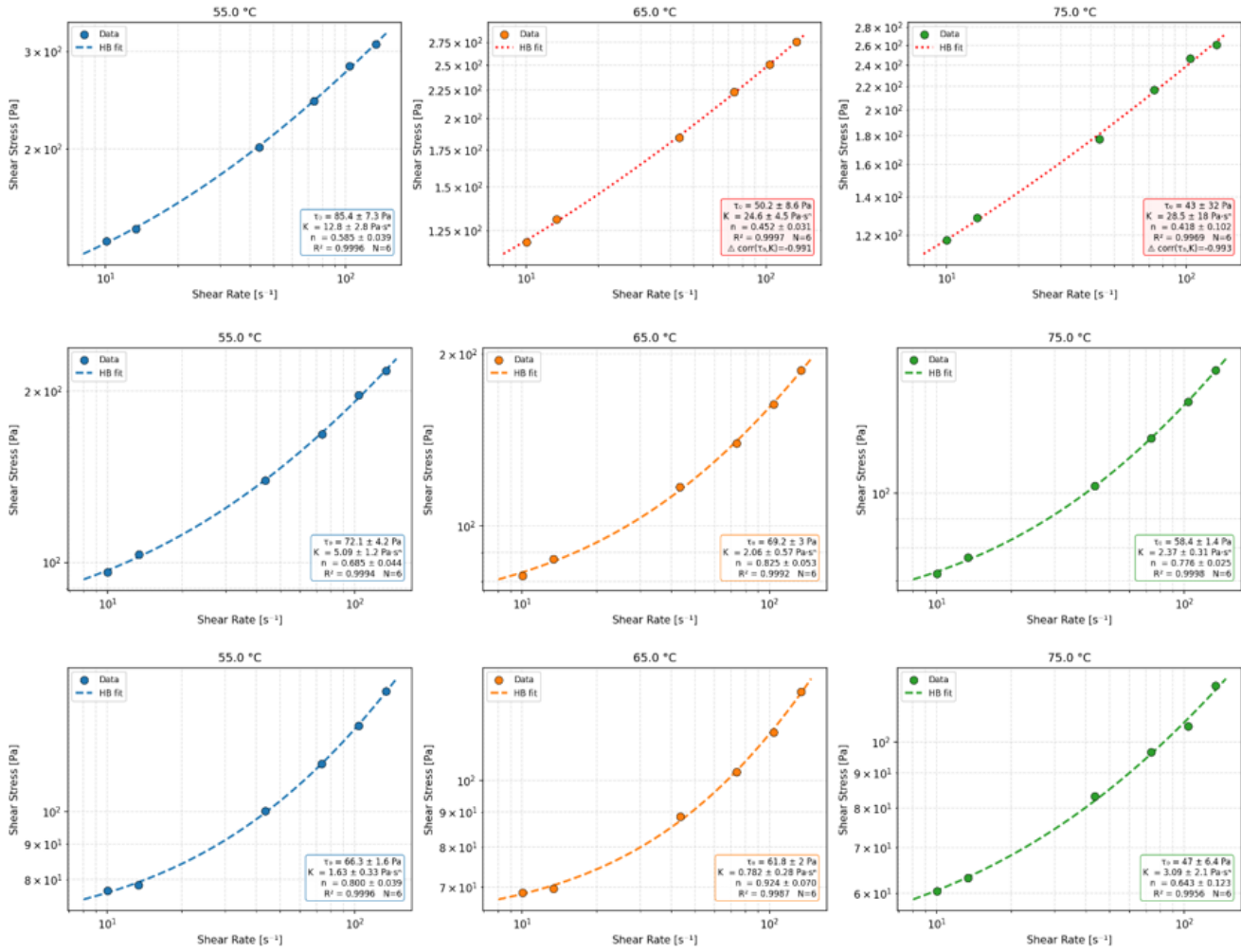
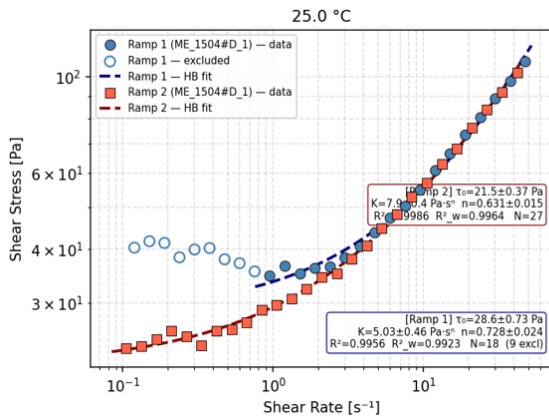


Figure 9.8. Flow curves from RVA measurements at different temperatures, 55, 65 and 75 °C from left to right. Measurements are performed on samples containing BG Mw 236 kDa with TS 17.8 %, 16.3 % and 14.0 % in the top, middle and bottom flow curve respectively.

9.3 Rheometer Flow curve measurements

Dry matter: 13.4%; Enzyme Dose: No; Temp: 25 °C [Log-log]



Dry matter: 13.4%; Enzyme Dose: No; Temp: 25 °C [Log-log]

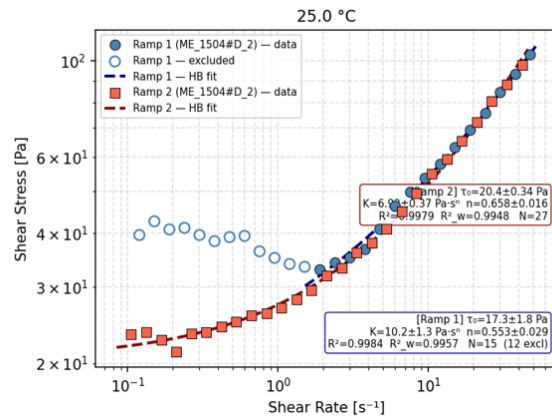
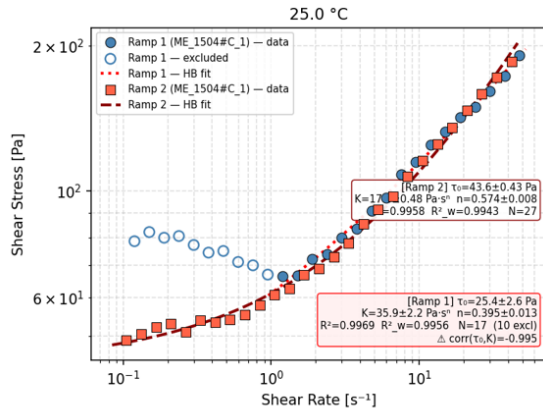


Figure 9.9. Duplicates of rheometer flow curves measured at 25 °C on untreated samples with 13.4 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 15.2%; Enzyme Dose: No; Temp: 25 °C [Log-log]



Dry matter: 15.2%; Enzyme Dose: No; Temp: 25 °C [Log-log]

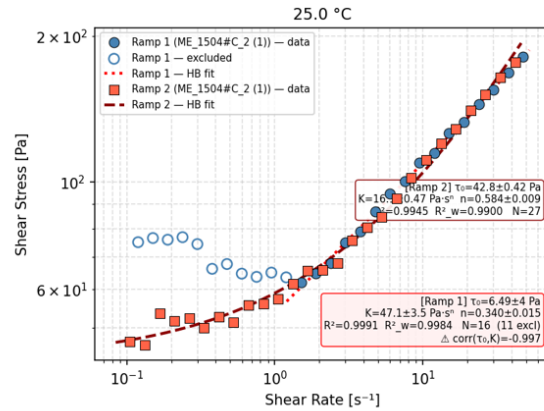
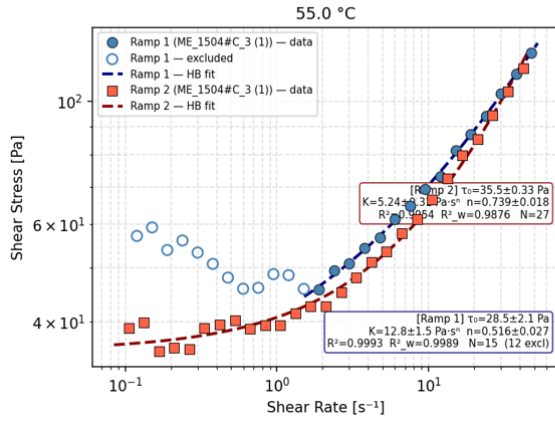


Figure 9.10. Duplicates of rheometer flow curves measured at 25 °C on untreated samples with 15.2 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 15.2%; Enzyme Dose: No; Temp: 55 °C [Log-log]



Dry matter: 15.2%; Enzyme Dose: No; Temp: 55 °C [Log-log]

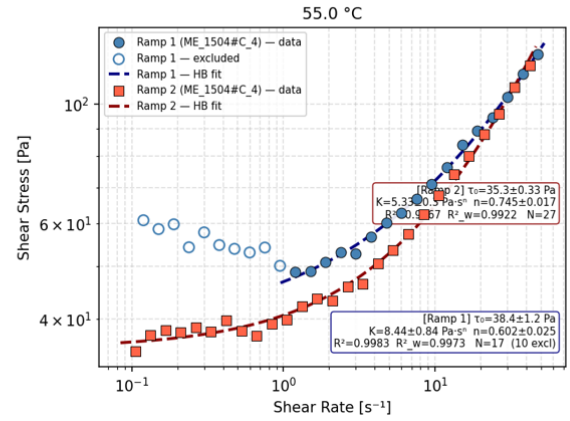
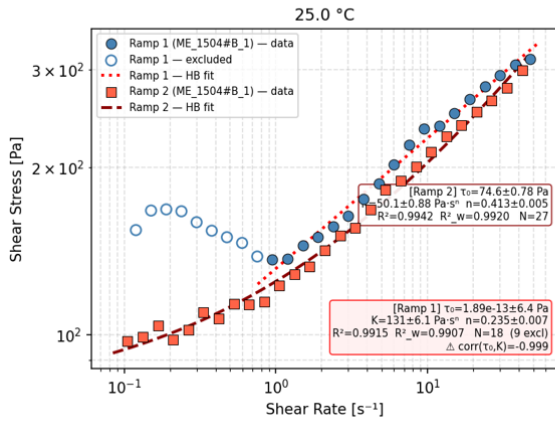


Figure 9.11. Duplicates of rheometer flow curves measured at 55 °C on untreated samples with 15.2 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 17.1%; Enzyme Dose: No; Temp: 25 °C [Log-log]



Dry matter: 17.1%; Enzyme Dose: No; Temp: 25 °C [Log-log]

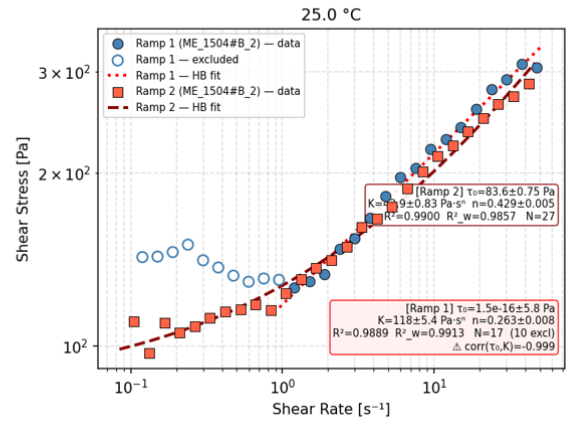
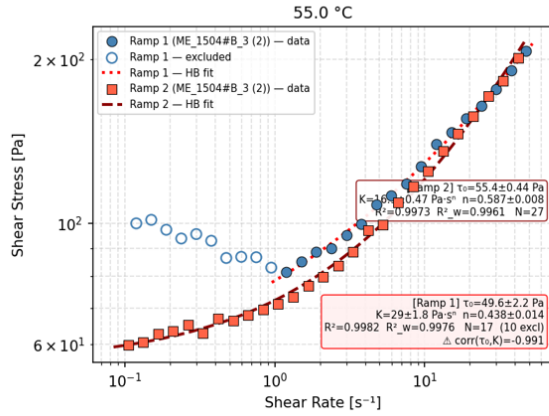


Figure 9.12. Duplicates of rheometer flow curves measured at 25 °C on untreated samples with 17.1 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 17.1%; Enzyme Dose: No; Temp: 55 °C [Log-log]



Dry matter: 17.1%; Enzyme Dose: No; Temp: 55 °C [Log-log]

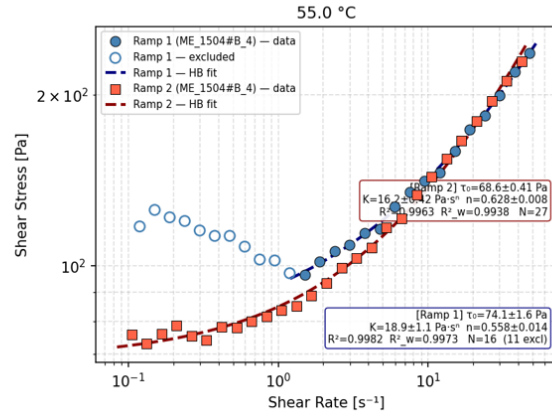
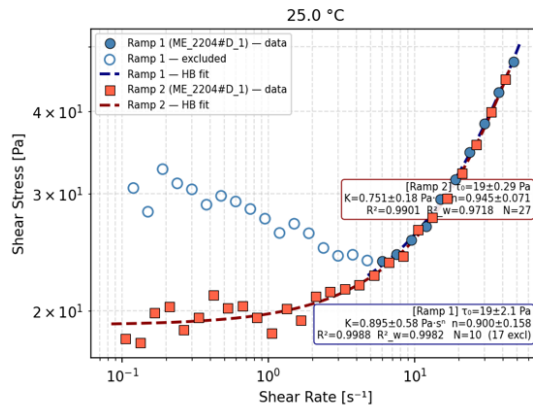


Figure 9.13. Duplicates of rheometer flow curves measured at 55 °C on untreated samples with 17.1 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 14.2%; Enzyme Dose: Medium; Temp: 25 °C [Log-log]



Dry matter: 14.2%; Enzyme Dose: Medium; Temp: 25 °C [Log-log]

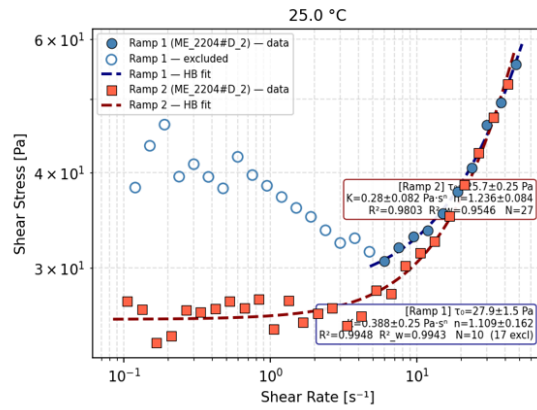


Figure 9.14. Duplicates of rheometer flow curves measured at 25 °C on samples with 152 kDa BG Mw, 14.2 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 14.2%; Enzyme Dose: Medium; Temp: 55 °C [Log-log]

Dry matter: 14.2%; Enzyme Dose: Medium; Temp: 55 °C [Log-log]

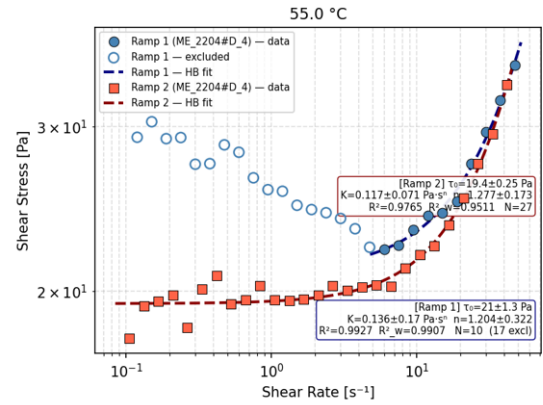
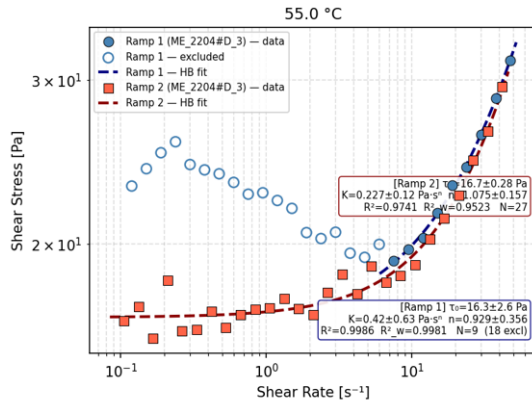


Figure 9.15. Duplicates of rheometer flow curves measured at 55 °C on samples with 152 kDa BG Mw, 14.2 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 15.7%; Enzyme Dose: Medium; Temp: 25 °C [Log-log]

Dry matter: 15.7%; Enzyme Dose: Medium; Temp: 25 °C [Log-log]

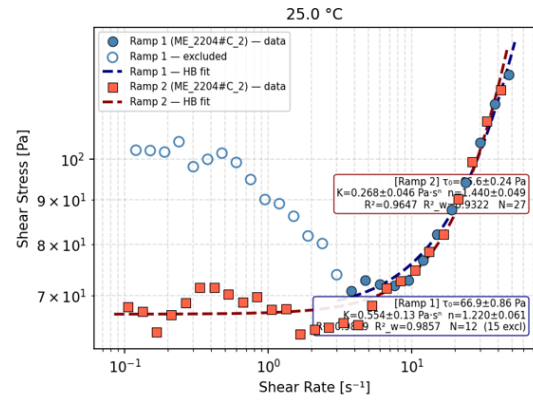
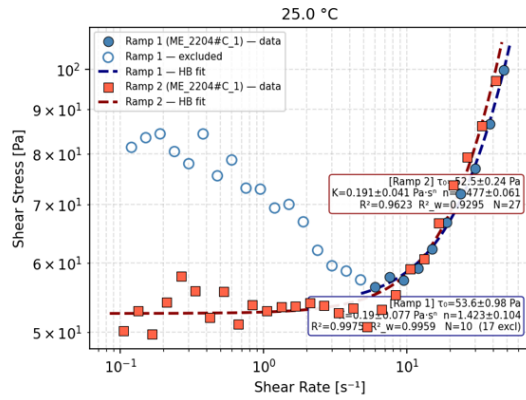
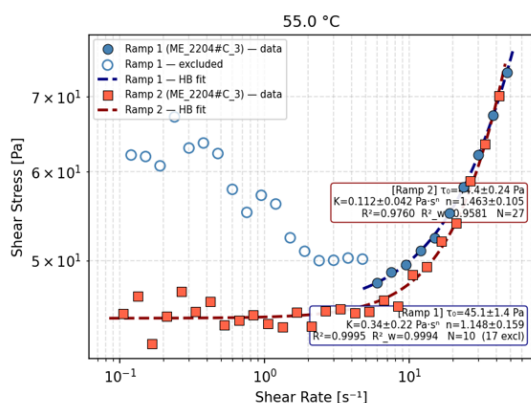


Figure 9.16. Duplicates of rheometer flow curves measured at 25 °C on samples with 152 kDa BG Mw, 15.7 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: N/A; Enzyme Dose: N/A; Temp: 55 °C [Log-log]



Dry matter: 15.7%; Enzyme Dose: Medium; Temp: 55 °C [Log-log]

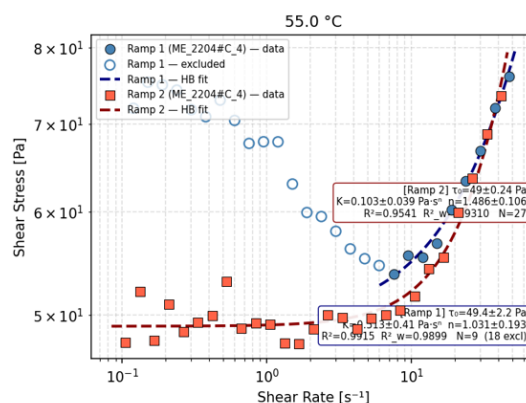
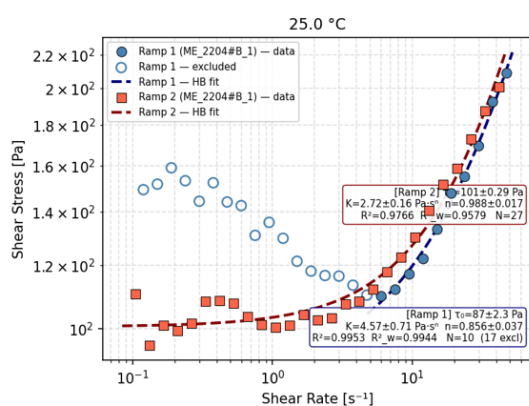


Figure 9.17. Duplicates of rheometer flow curves measured at 55 °C on samples with 152 kDa BG Mw, 15.7 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 17.9%; Enzyme Dose: Medium; Temp: 25 °C [Log-log]



Dry matter: 17.9%; Enzyme Dose: Medium; Temp: 25 °C [Log-log]

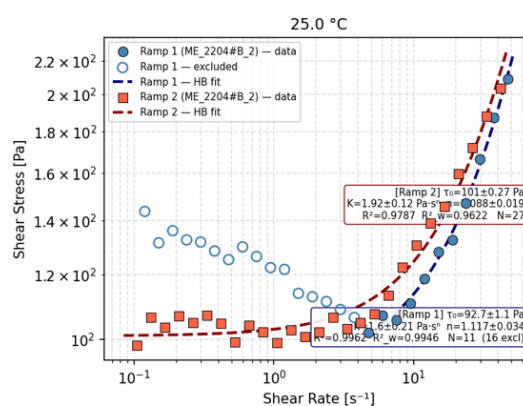
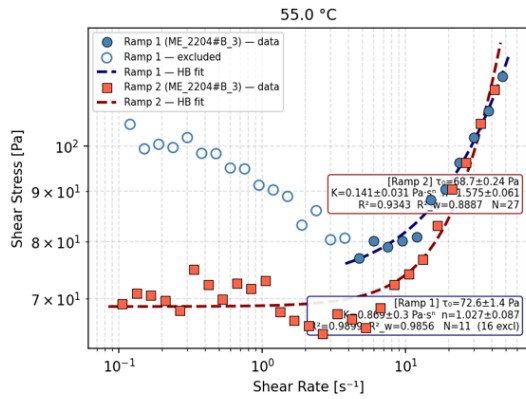


Figure 9.18. Duplicates of rheometer flow curves measured at 25 °C on samples with 152 kDa BG Mw, 17.9 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: N/A; Enzyme Dose: N/A; Temp: 55 °C [Log-log]



Dry matter: 17.9%; Enzyme Dose: Medium; Temp: 55 °C [Log-log]

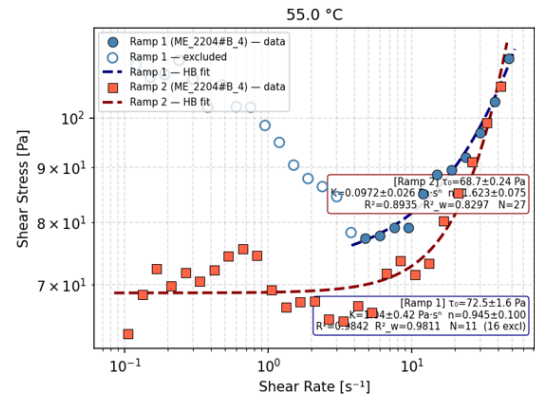
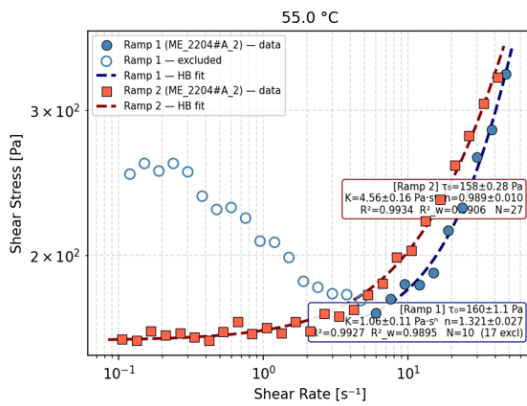


Figure 9.19. Duplicates of rheometer flow curves measured at 55 °C on samples with 152 kDa BG Mw, 17.9 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 21.7%; Enzyme Dose: Medium; Temp: 55 °C [Log-log]



Dry matter: 21.7%; Enzyme Dose: Medium; Temp: 55 °C [Log-log]

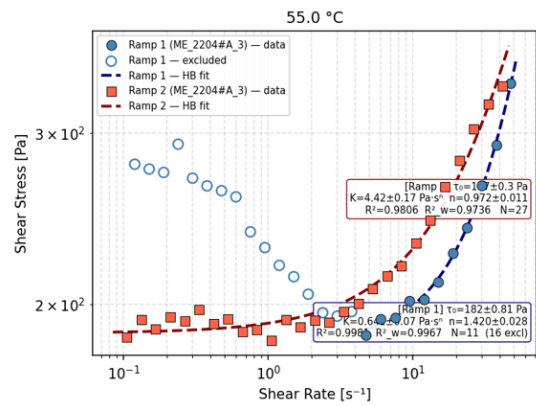


Figure 9.20. Duplicates of rheometer flow curves measured at 55 °C on samples with 152 kDa BG Mw, 21.7 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 15.5%; Enzyme Dose: N/A; Temp: 25 °C [Log-log] Dry matter: 15.5%; Enzyme Dose: N/A; Temp: 25 °C [Log-log]

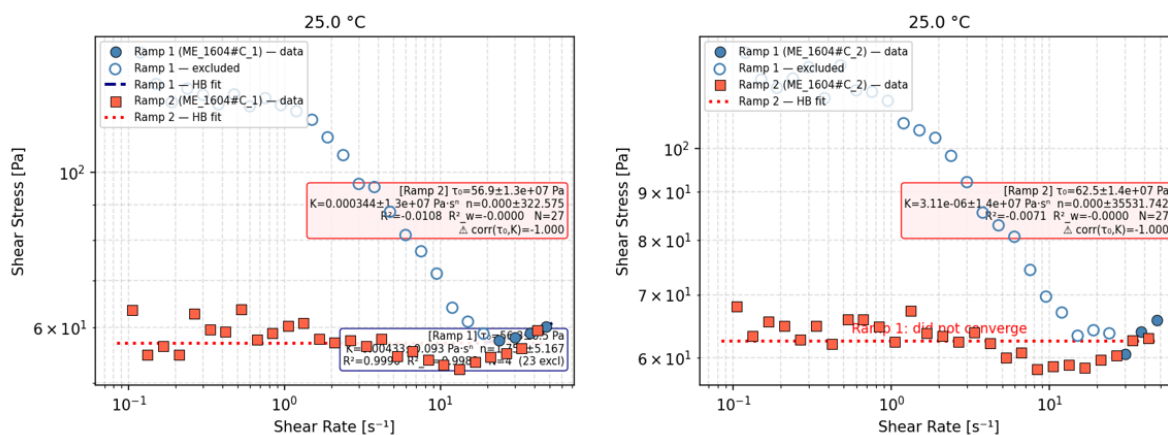


Figure 9.21. Duplicates of rheometer flow curves measured at 25 °C on samples with no detectable BG and 15.5 % TS. The blue points are the upward ramp and the orange points are the downward ramp.

Dry matter: 15.9%; Enzyme Dose: N/A; Temp: 55 °C [Log-log] Dry matter: 15.9%; Enzyme Dose: N/A; Temp: 55 °C [Log-log]

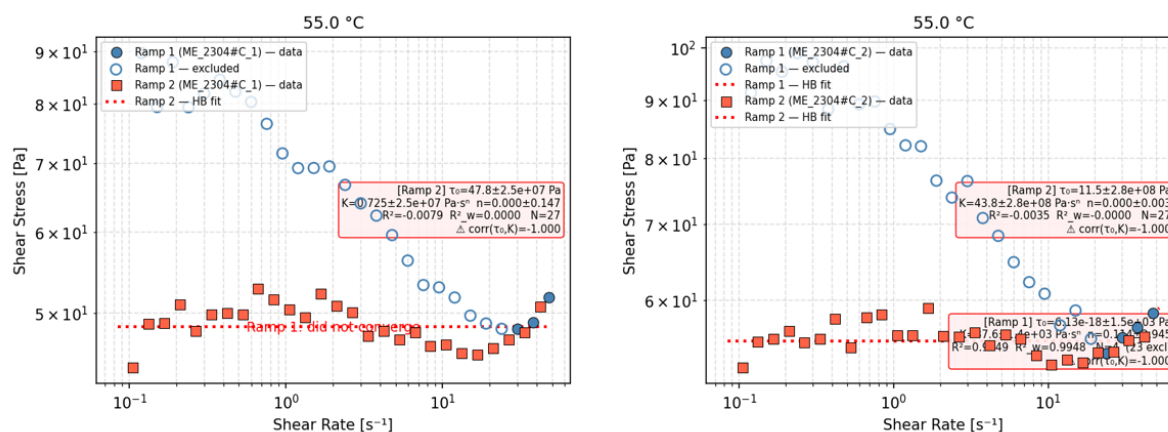


Figure 9.22. Duplicates of rheometer flow curves measured at 55 °C on samples with no detectable BG and 15.9 % TS. The blue points are the upward ramp and the down-ward ramp.

Dry matter: 17.6%; Enzyme Dose: N/A; Temp: 25 °C [Log-log]

Dry matter: 17.6%; Enzyme Dose: N/A; Temp: 25 °C [Log-log]

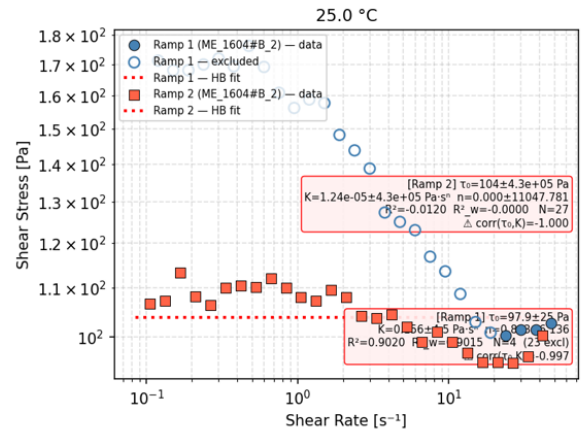
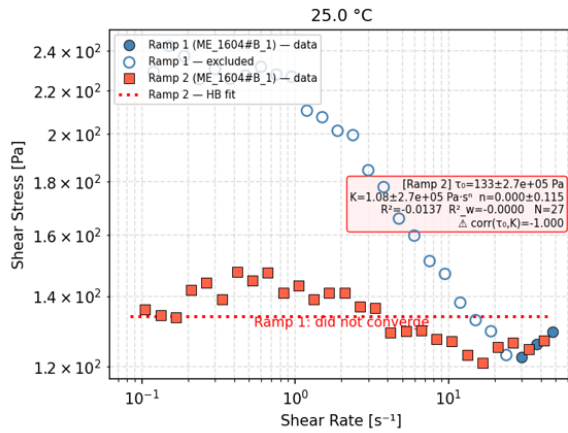


Figure 9.23. Duplicates of rheometer flow curves measured at 25 °C on samples with no detectable BG and 17.6 % TS. The blue points are the upward ramp and the orange points are the down-ward ramp.

Dry matter: 18.0%; Enzyme Dose: N/A; Temp: 55 °C [Log-log]

Dry matter: 18.0%; Enzyme Dose: N/A; Temp: 55 °C [Log-log]

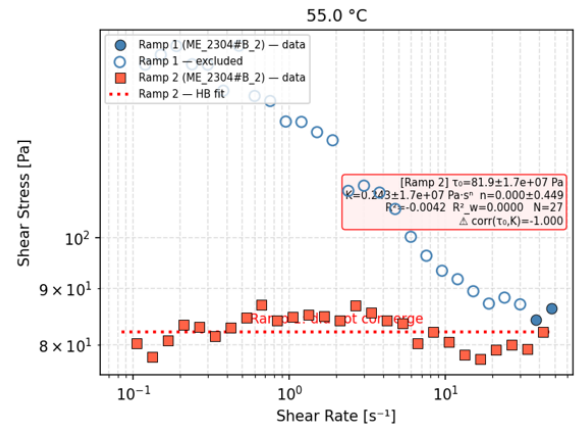
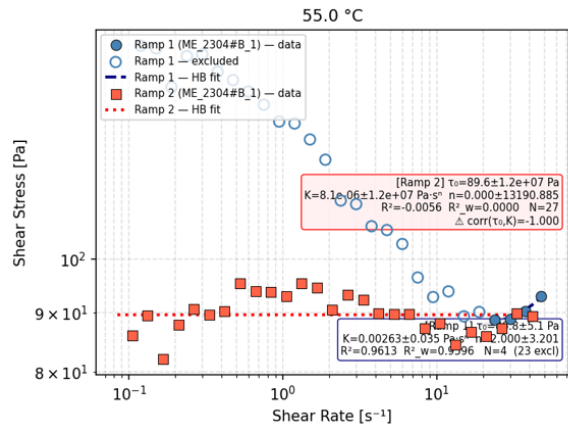
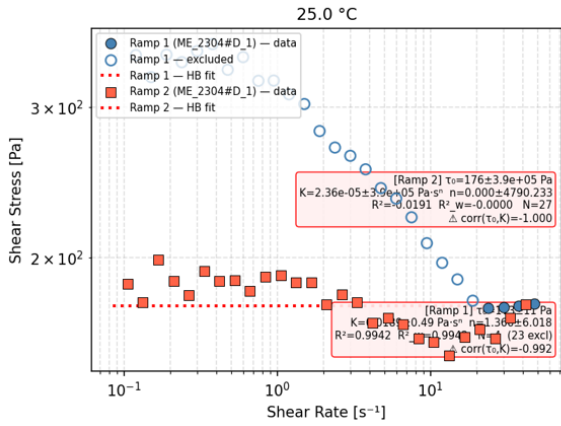


Figure 9.24. Duplicates of rheometer flow curves measured at 55 °C on samples with no detectable BG and 18.0 % TS. The blue points are the upward ramp and the orange points are the down-ward ramp.

Dry matter: 19.7%; Enzyme Dose: N/A; Temp: 25 °C [Log-log]



Dry matter: N/A; Enzyme Dose: N/A; Temp: 25 °C [Log-log]

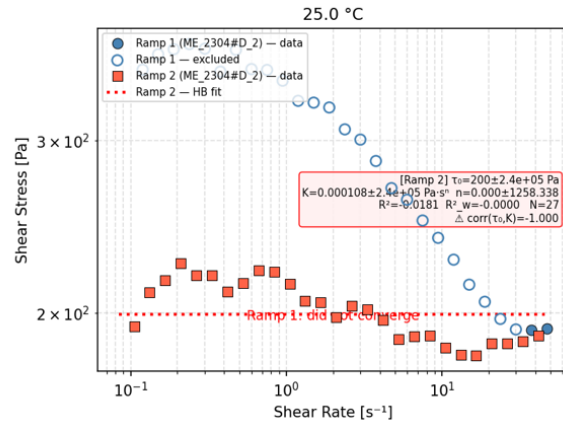
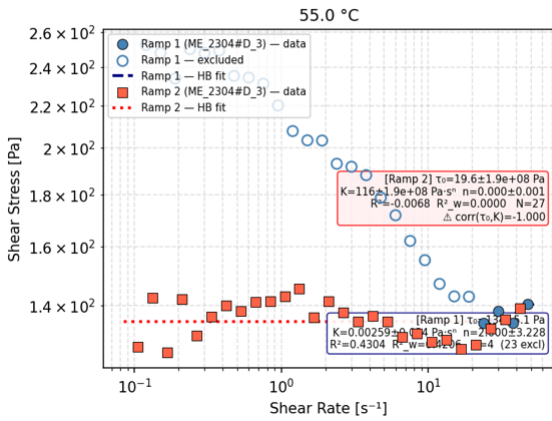


Figure 9.25. Duplicates of rheometer flow curves measured at 25 °C on samples with no detectable BG and 19.7 % TS. The blue points are the upward ramp and the orange points are the down-ward ramp.

Dry matter: 19.7%; Enzyme Dose: N/A; Temp: 55 °C [Log-log]



Dry matter: 19.7%; Enzyme Dose: N/A; Temp: 55 °C [Log-log]

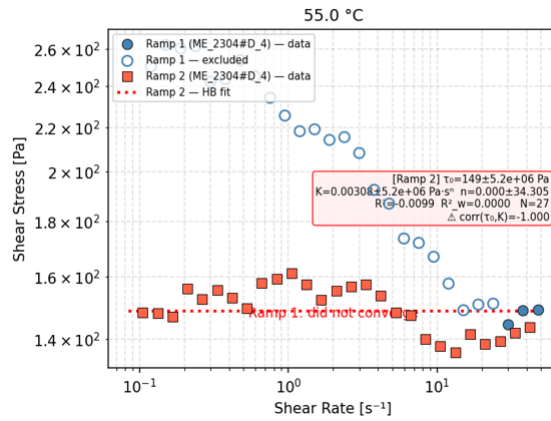


Figure 9.26. Duplicates of rheometer flow curves measured at 55 °C on samples with no detectable BG and 19.7 % TS. The blue points are the upward ramp and the orange points are the down-ward ramp.

Dry matter: 22.1%; Enzyme Dose: N/A; Temp: 25 °C [Log-log]

Dry matter: N/A; Enzyme Dose: N/A; Temp: 25 °C [Log-log]

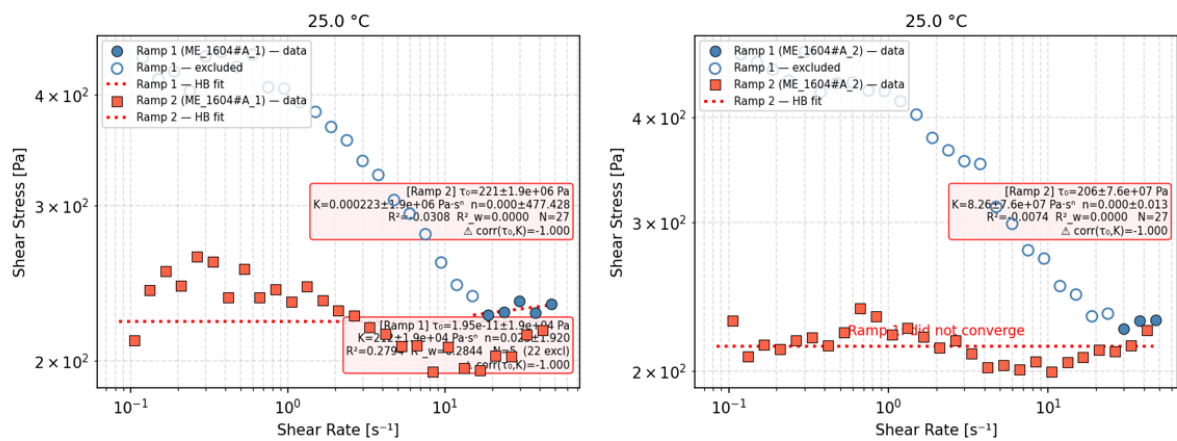


Figure 9.27. Duplicates of rheometer flow curves measured at 25 °C on samples with no detectable BG and 22.1 % TS. The blue points are the upward ramp and the orange points are the down-ward ramp.

9.4 Amplitude sweep

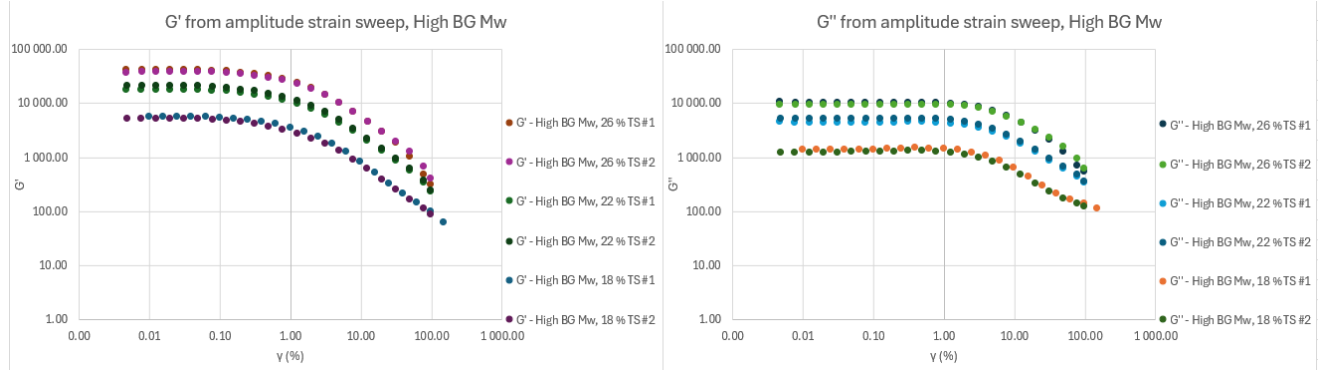


Figure 9.28. Storage and loss modulus from amplitude strain sweeps performed at 25 °C on untreated samples with varying TS content. Left: G' , Right: G'' .

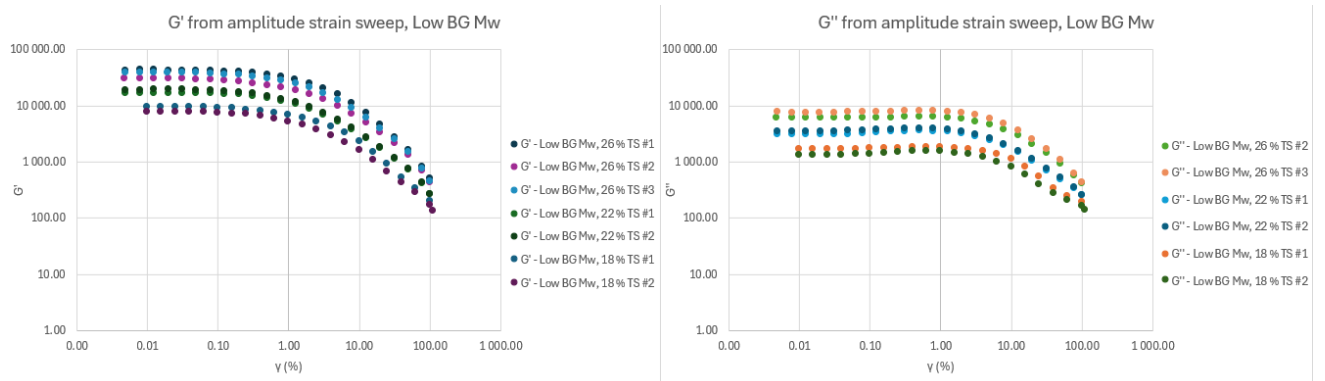


Figure 9.29. Storage and loss modulus from amplitude strain sweeps performed at 25 °C on samples with no detectable BG Mw and varying TS content. Left: G' , Right: G'' .

9.5 Parameter fit

9.5.1 Rheometer

Table 9.1. HB and BP model parameters and r squared values for the model fits. Raw data fit to rheometer-measurements of samples with different BG Mw and TS at 25 °C.

Run	Mw	TS	HB: τ_0	HB: k	HB: n	HB: R2	BP: τ_0	BP: k	BP: R2
1	463	17.09	74.578	50.088	0.413	0.994	112.255	6.227	0.828
2	463	17.09	83.594	43.912	0.429	0.990	117.063	5.743	0.831
1	463	15.25	43.635	17.581	0.574	0.996	54.963	4.181	0.901
2	463	15.25	42.810	16.105	0.584	0.995	53.140	3.960	0.906
1	463	13.39	21.495	7.899	0.631	0.999	26.115	2.337	0.932
2	463	13.39	20.408	6.918	0.658	0.998	24.304	2.250	0.937
1	151	21.71	149.492	16.772	0.665	0.979	161.187	5.218	0.924
1	151	17.91	100.629	2.719	0.988	0.977	100.760	2.614	0.976
2	151	17.91	100.970	1.922	1.088	0.979	100.076	2.594	0.980
1	151	15.74	52.529	0.191	1.477	0.962	51.205	0.964	0.934
2	151	15.74	66.627	0.268	1.440	0.965	65.070	1.192	0.939
1	151	14.21	19.045	0.751	0.945	0.990	19.191	0.624	0.990
2	151	14.21	25.654	0.280	1.236	0.980	25.161	0.622	0.974
1	0	22.1	221.458	0.000	0.000	-0.031	221.459	0.000	-0.031
2	0	22.1	205.525	8.264	0.000	-0.00739	213.788	0.000	-0.007
1	0	17.58	132.715	1.077	0.000	-0.014	133.792	0.000	-0.014
2	0	17.58	103.811	0.000	0.000	-0.012	103.811	0.000	-0.012
1	0	15.49	56.940	0.000	0.000	-0.011	56.940	0.000	-0.011
2	0	15.49	62.510	0.000	0.000	-0.007	62.510	0.000	-0.007
1	0	19.69	175.549	0.000	0.000	-0.019	207.117	0.000	-0.007
2	0	19.69	199.545	0.000	0.000	-0.018	200.042	0.000	-0.020

Table 9.2. HB and BP model parameters and r squared values for the model fits. Raw data fit to rheometer-measurements of samples with different BG Mw and TS at 55 °C.

Run	Mw	TS	HB: τ_0	HB: k	HB: n	HB: R2	BP: τ_0	BP: k	BP: R2
1	463	17.09	55.426	16.874	0.587	0.997	66.566	4.159	0.920
2	463	17.09	68.586	16.162	0.628	0.996	79.042	4.574	0.932
1	463	15.25	35.491	5.242	0.739	0.995	38.387	2.190	0.964
2	463	15.25	35.287	5.332	0.745	0.997	38.163	2.279	0.966
1	463	13.39	13.662	2.389	0.752	0.998	19.359	0.846	0.988
1	151	21.71	157.578	4.562	0.989	0.993	157.786	4.392	0.993
2	151	21.71	186.675	4.417	0.972	0.981	187.191	4.010	0.979
1	151	17.91	68.748	0.141	1.575	0.934	67.165	0.995	0.892
2	151	17.91	68.748	0.097	1.623	0.894	67.361	0.820	0.842
1	151	15.74	44.403	0.112	1.463	0.976	43.633	0.549	0.944
2	151	15.74	49.038	0.103	1.486	0.954	48.239	0.545	0.921
1	151	14.21	16.673	0.227	1.075	0.974	16.584	0.294	0.972
2	151	14.21	19.410	0.117	1.277	0.976	19.124	0.302	0.962
1	0	21.79	207.114	0.002	0.000	-0.007	207.117	0.000	-0.007
2	0	21.79	200.041	0.000	0.000	-0.020	200.042	0.000	-0.020
1	0	18.02	89.578	0.000	0.000	-0.006	89.578	0.000	-0.006
2	0	18.02	81.920	0.243	0.000	-0.004	82.162	0.000	-0.004
1	0	15.9	47.795	0.725	0.000	-0.008	48.520	0.000	-0.008
2	0	15.9	11.494	43.804	0.000	-0.003	55.298	0.000	-0.003
1	0	19.69	175.549	0.000	0.000	-0.019	175.549	0.000	-0.019
2	0	19.69	199.545	0.000	0.000	-0.018	199.545	0.000	-0.018
1	0	19.69	19.592	115.574	0.000	-0.007	135.166	0.000	-0.007
2	0	19.69	148.686	0.003	0.000	-0.010	148.689	0.000	-0.010

9.5.2 RVA

Table 9.3. HB and BP model parameters and r squared values for the model fits. Raw data fit to RVA-measurements of samples with different BG Mw and TS at 55 °C.

Run	Mw	TS	HB: τ_0	HB: k	HB: n	HB: R2	BP: τ_0	BP: k	BP: R2
1	235	17.765	85.390	12.841	0.585	1.000	129.413	1.425	0.983
2	235	17.765	76.181	4.420	0.750	0.997	93.235	1.208	0.992
1	235	16.33	72.140	5.093	0.685	0.999	91.486	0.983	0.990
1	235	13.97	66.305	1.632	0.800	1.000	72.433	0.581	0.996
2	235	13.97	66.305	1.632	0.800	1.000	72.433	0.581	0.996
1	50	18.355	93.359	0.116	1.280	0.994	89.254	0.470	0.989
2	50	18.355	93.359	0.116	1.280	0.994	89.254	0.470	0.989
1	50	15.79	83.013	0.057	1.448	0.998	76.524	0.532	0.984
1	50	14.13	58.878	0.085	1.225	0.998	56.936	0.263	0.994
1	31	18.02	113.670	0.062	1.348	0.992	110.004	0.356	0.983
1	31	16.12	135.364	0.009	1.606	0.956	132.521	0.192	0.935
1	31	14.09	75.232	0.046	1.267	0.993	73.761	0.174	0.988
1	0	17.405	106.781	0.000	2.378	0.987	103.367	0.154	0.908
1	0	17.02	129.816	0.000	3.494	0.987	121.574	0.318	0.765
1	0	16.895	110.264	0.000	2.848	0.996	105.488	0.199	0.844
1	0	14.48	91.015	0.000	3.555	0.957	88.304	0.107	0.705
1	0	14.92	89.327	0.000	3.135	0.945	86.798	0.104	0.749

9.6 Functional Properties Regression Analysis

Table 9.4. Coefficients and P-values from statistical regression analysis of OHC raw data.

OHC

Factor	Coefficient	P-Value
TS	-5.92	0.002
PS Dx 50	0.1685	0.000

Table 9.5. Coefficients and P-values from statistical regression analysis of SWC raw data.

SWC

Factor	Coefficient	P-Value
TS	-0.0979	0.000
PS Dx 50	0.002317	0.000

Table 9.6. Coefficients and P-values from statistical regression analysis of WHC raw data.

WHC

Factor	Coefficient	P-Value
BG Mw [kDa]	-0.115	0.018
PS Dx 50	0.1496	0.007

Table 9.7 Coefficients and P-values from statistical regression analysis of WSI raw data.

WSI

Factor	Coefficient	P-Value
PS Dx 50	-0.00772	0.000