EXTENSIONAL VISCOSITY OF CURTAIN COATING COLORS EVALUATED AS EULER NUMBER

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Preface

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Abstract

The aim of this study was to evaluate different additives and their effect on the Euler number, and thus the extensional viscosity of barrier coating dispersions. The Euler number was measured over a wide range of flow rates, with an emphasis on a proposed standard of 15 m/s.

The Euler number was measured using a high shear capillary viscometer, using an orifice geometry. Additives used in this study were carboxymethyl cellulose, polyvinyl alcohol and polyethylene oxide. The results show significant differences in the effect on the Euler number.

The addition of high molar mass polyethylene oxide increased the Euler number significantly. Polyvinyl alcohol and lower molar mass polyethylene oxide showed a moderate increase in Euler number. Carboxymethyl cellulose and the barrier coating dispersion with no additives showed insignificant increases. The extensional viscosity is, however, also affected by the shear viscosity, which must be taken into account when comparing measurements.

Extensional viscosity plays an important role in several coating methods. In curtain coating, it plays a key role in the falling curtain and the impingement point. Extensional viscosity can slow down the expansion of holes in the curtain, and even prevent them from forming altogether. Insufficient extensional viscosity can also lead to uncoated areas on the surface due to inadequate stretching of the coating fluid onto it.

Nomenclature

Greek symbols

$\dot{\varepsilon}$	Constant extensional strain rate
η	Shear viscosity
η_E	Extensional viscosity
ρ	Density
σ	Surface tension
τ	Shear stress
$\dot{\gamma}$	Shear rate

Latin symbols and abbreviations

CMC	Carboxymethyl cellulose
ESR	Extensional strain rate
g	Gravitational acceleration constant
h_c	Curtain height
HPC	Hydroxypropyl cellulose
L	Length scale
p	Pressure
PAA	Polyacrylatic acid
PAM	Polyacrylamides
PEO	Polyethylene oxide
p_k	kinetic energy term
PMMA	Poly(methyl-methacrylate)
PS	Polystyrene
PVOH	Polyvinyl alcohol
PVP	Polyvinylpyrrolidones
Q	Volumetric flow rate
R	Radius
Re	Reynolds number
Tr	Trouton ratio
TS	Tensile stress
u	Flow velocity

v	Velocity
v_0	Initial velocity
v_c	Curtain velocity
We	Weber number
x	Distance from curtain edge

Table of Contents

1 Introduction

Plastics made from petrochemicals are widely used in several industries, such as in food packaging (Zhao et al., 2023). Though tremendously convenient, plastics need hundreds of years to degrade. This leads to pollution, which has severe negative impacts on the environment and food value chain. Concern over the appearance of microplastics in soil and water has increased over recent years (Ludwick and Aglan, 2020). The ecological toll of the single use plastics together with consumer demand is driving the development of biodegradable, recyclable or compostable packaging materials (Adibi, Trinh and Mekonnen, 2023). Paper packaging has been a front runner in this area. However, these products tend to have inadequate barrier properties for gas, water and grease, leading to problems with shelf stability. In order to achieve the desired barrier properties, coating of paper and paperboard with bio-based polymers has received much attention recently.

The coating vastly enhances the properties of the paper. Several coating methods can be employed to apply one or more layers, such as extrusion coating, blade coating, rod coating, film coating and curtain coating. Extrusion coating has been the most common process of applying barriers on paper due to its many benefits. However, an issue with extrusion coating is the difficulty in recycling the extrusion coated paper and paperboard (Khlewee, Al-Gharrawi and Bousfield, 2022).

Extensional viscosity is an important property in many coating methods, especially if the coating color is subjected to strong accelerating flows (Ojanen, Sinkko and Kunnas, 2007). While shear viscosity of coating colors is usually measured, extensional viscosity is typically not. Curtain coating is an example where the extensional viscosity plays an important role. Orifice rheometry is a method to characterize the extensional viscosity of coating colors. When a measurement is carried out, accelerating flow is created at the orifice entrance. The pressure drop is recorded and compared to the kinetic energy. The result is the Euler number, which indicates the level of extensional viscosity in the sample.

The objective of this study was to compare the effects of different polymers on the Euler number and thus the extensional viscosity of traditional barrier dispersions. Achieving a suitable extensional viscosity in the coating colors can enhance the final coating layers and improve efficiency and runnability in various processes. A method for measuring extensional viscosity that is fast and easy would be of great importance.

2 Theory

The theory portion of this study will focus on extensional viscosity, such as basic rheology, methods of measuring and controlling extensional viscosity, coating processes where extensional viscosity is relevant, as well as other miscellaneous areas where extensional viscosity can be important.

2.1 Rheology

Rheology is the study of the deformation and flow of matter (Strivens, 1999). When applying mechanical force or deformation on a material, the response can be irreversible (viscous), reversible (elastic), or a combination of the two (viscoelastic). In viscous flow, the energy is lost, while in elastic deformation it is stored and released once the force is removed.

In Newtonian fluids, the viscosity is independent of the shear rate or shear stress applied, at constant temperature and pressure. The viscosity will therefore stay the same, regardless of increased or decreased shear. Water is an example of a material that fits this description (Heinikainen, 2012). Most fluids do not fit into this description and are called non-Newtonian fluids. Shear dependent materials can either be pseudoplastic or dilatant, while time dependent materials are thixotropic or rheopectic (Chandran, Chandrasekharakurup and Thomas, 2020).

Pseudoplasticity is the most common non-Newtonian behavior. In these materials, the viscosity decreases when shear rate is increased. These fluids are therefore also called shear-thinning. This phenomenon occurs due to rearrangement of the microstructure in the direction of applied shear. In dilatant fluids, the viscosity increases when shear rate is increased and are, therefore, also called shear-thickening. This usually occurs due to the jamming of particles.

Thixotropic fluids show a decrease in viscosity over time, under constant shear conditions. This occurs when the intermolecular bonds of the fluid rupture. Rheopectic fluids show an increase in viscosity over time, under constant shear conditions. This occurs when particles aggregate, or cross links are formed.

2.1.1 Shear viscosity

Shear viscosity defines a fluid’s resistance to motion (Toivakka, 2015). The concept can be visualized as a fluid in between two parallel plates, as shown in figure 1.

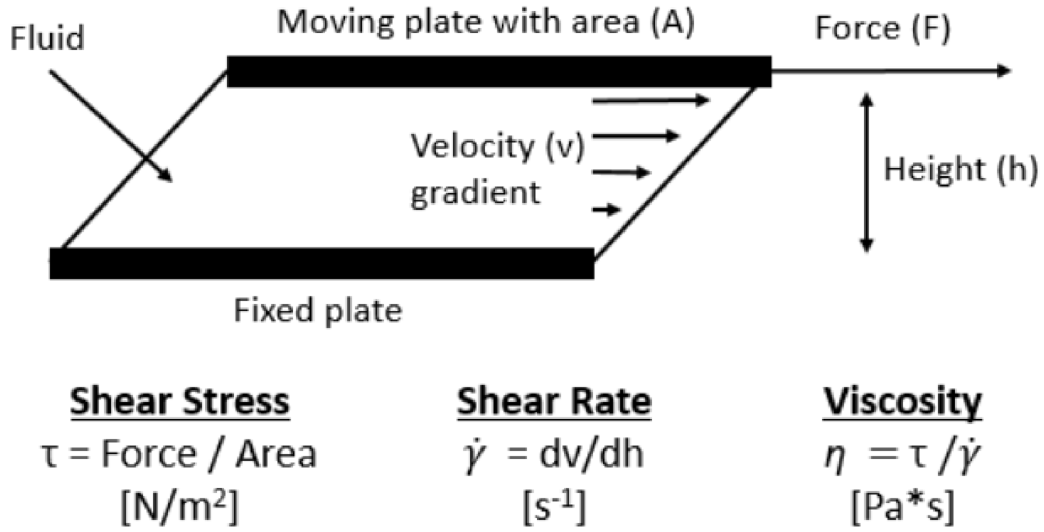

Figure 1. *Fluid sheared between a fixed and moving plate (Toivakka, 2015).*

The lower plate stays stationary, while the upper plate moves. Shear stress is described as the force needed to move the plate at a given speed divided by the area and is expressed in Pascals. Shear rate is described as the velocity of the plate, divided by the distance between the plates, and is expressed in reciprocal seconds. The shear viscosity can then be calculated as the shear rate divided by shear stress.

Viscosity is dependent on pressure and temperature (Lewis, 2023). These parameters should therefore be held constant and be clearly stated in the experiments. There are several methods to measure the shear viscosity. Gravity based capillary flow viscometers and falling sphere viscometers are relatively simple methods of measuring shear viscosity. However, a drawback is that they only measure the viscosity at one shear rate, making it difficult to determine if it is a Newtonian or non-Newtonian fluid. Rotational rheometers are more complex, and come with different geometries, such as parallel plates, cone and plate, and concentric cylinder. They have the ability to measure viscosity at different shear rates, which is important for determining viscoelasticity. For high shear rates, the Hercules viscometer (Smith and Applegate, 1948) or capillary rheometers (Pollak et al., 2017) can be used. ACA Systems Oy has recently developed a modern version of the capillary viscometer. The AX-100 model is capable of measuring viscosity over a wide shear rate range, from ca. 5000 to 1 000 000 1/s. In addition, it is equipped with orifice configuration enabling the measurement of extensional viscosity for various extensional rates.

For viscoelastic materials, it is common to use the loss and storage modulus to determine the properties of polymers or elastomers (Reshmin, Teplovodskii and Trifonov, 2011). The storage modulus (G’) represents stored energy, and the loss modulus (G’’) represents energy loss. The loss tangent is used to represent the ratio between energy loss and energy storage. For a completely elastic material, the stress and strain will be in phase at all frequencies. For a completely viscous material, the stress will lead the strain by π/2 rad. The phase angle for viscoelastic materials will therefore be between 0 and 90 degrees, 0 for completely elastic, and 90 for completely viscous materials.

The flow of a fluid can be either laminar, which can be described as smooth and predictable flow, or turbulent, which is a complicated flow structure of interacting vortices (Brody et al., 1996). In turbulent flow, the inertial forces are higher than the viscous forces. As turbulent flow is difficult to predict, the aim is usually to avoid it. The Reynolds number can be used to determine if flow is laminar or turbulent, by measuring the ratio of inertial forces to viscous forces. The formula can be seen in equation 1:

$$\boldsymbol{Re = \frac{\rho u L}{\eta},} \qquad [1]$$

where ρ is the density of the fluid, u is the flow velocity, L is the length scale (channel diameter over which changes in the fluid velocity occur), and η is the viscosity of the fluid. Low Reynolds numbers mean the flow is laminar, while a high number means turbulent flow. The flow is usually laminar at Reynolds numbers under 2000, and turbulent over 2000 (Reshmin, Teplovodskii and Trifonov, 2011). There are, however, situations where this is not the case, where flow can be laminar at higher Reynolds numbers, and turbulent at lower numbers.

2.1.2 Extensional viscosity

Extensional viscosity, also called elongational viscosity, is a fluid's resistance to extension (Vlachopoulos and Strutt, 2016). It is one of the most important rheological properties, second only to shear viscosity. It is also substantially more difficult to measure than shear viscosity. Extensional viscosity can be divided into three basic types of flow, uniaxial, biaxial and planar, as shown in figure 2 (Barnes, Hutton and Walters, 1989).

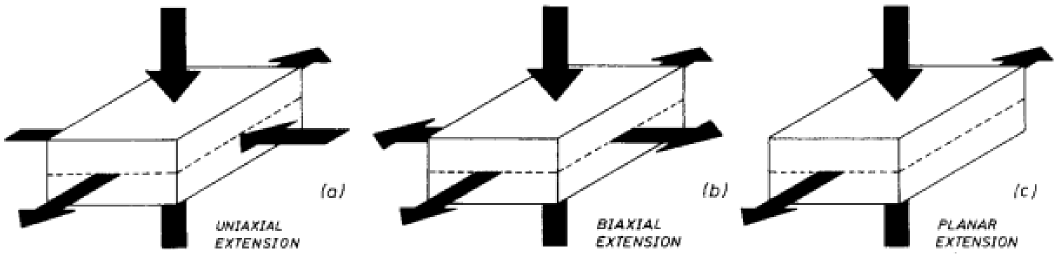

***Figure 2. a)** Uniaxial, **b)** biaxial, and **c)** planar extensional flow (Barnes, Hutton and Walters, 1989).*

Uniaxial extensional flow is the simplest one, where the material is stretched in one direction, while the sample dimensions of the two remaining directions are decreased. The velocity field is described in equation 2 as

$$\boldsymbol{v_x = \dot{\varepsilon}x, v_y = -\frac{\dot{\varepsilon}y}{2}, v_z = -\frac{\dot{\varepsilon}z}{2},} \qquad [2]$$

where $\dot{\varepsilon}$ is a constant extensional strain rate.

In biaxial extensional flow, the material is stretched in two directions, while the thickness of the material decreases. The biaxial velocity field is described in equation 3 as

$$\boldsymbol{v_x = \dot{\varepsilon}x, v_y = \dot{\varepsilon}y, v_z = -\dot{2}\varepsilon z.} \qquad [3]$$

Finally, in the planar extensional flow, the material is stretched in one direction, while the thickness decreases, and the width remains constant. The planar velocity field is described in equation 4 as

$$\boldsymbol{v_x = \dot{\varepsilon}x, v_y = -\dot{\varepsilon}y, v_z = 0.} \qquad [4]$$

Elastic polymer solutions which possess a viscosity that decreases with shear (pseudoplasticity) tend to have extensional viscosity that increases with strain rate (Hodder and Franck, 2005). Fluids that have extensional viscosity that increases with strain are called tension thickening, while fluids with extensional viscosity that decreases with strain are called tension thinning.

Using the Trouton ratio, extensional viscosity can be compared to shear viscosity. The shear viscosity is described in equation 5 as shear stress divided by shear rate

$$\boldsymbol{\eta = \frac{\tau}{\dot{\gamma}}.} \qquad [5]$$

The extensional viscosity is described in equation 6 as tensile stress divided by the extensional strain rate

$$\boldsymbol{\eta_E = \frac{TS}{ESR}.} \qquad [6]$$

The extensional viscosity can then be compared to the shear viscosity with the Trouton ratio, by dividing the former with the latter as seen in equation 7

$$\boldsymbol{Tr = \frac{\eta_E}{\eta}.} \qquad [7]$$

The Trouton ratio has been determined to be three for a perfectly Newtonian fluid, meaning the extensional viscosity is three times larger than the shear viscosity (Trouton, 1906).

The difference between shear flow and elongational flow is visualized in figure 3 (Zamani et al., 2015). If a fluid is exposed to shear flow, the distance between the points on the same streamline will not change. In elongational flow, the distance between the points will change. The easiest way to understand elongational viscosity is to imagine a fluid flowing through a tube, which narrows. For the flow to continue at the same rate, the fluid in the narrow part must move at a higher velocity and, therefore, accelerates and stretches. Converging streamlines in a flow field is a clear indication of extensional flow.

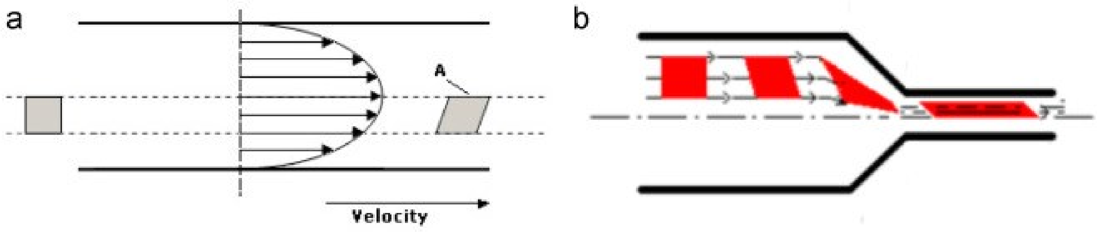

Figure 3. a) *Shear flow and* ***b)*** *elongational flow (Zamani et al., 2015).*

Euler number can be used to indicate the level of extensional viscosity in a sample (Heinikainen, 2012). It is a dimensionless number that is expressed as the ratio between the pressure loss (extrusion pressure) and kinetic energy term for flow through an orifice. The Euler number can be calculated as seen in equation 8:

$$\boldsymbol{Eu = \frac{p}{p_k} = \frac{p}{\frac{1}{2}\rho v^2} = \frac{p}{\frac{1}{2}\rho(\frac{Q}{\pi R^2})^2}}. \qquad [8]$$

As there will always be kinetic energy pressure drop, the Euler number can never be lower than 1. Calibrations can therefore be done with water, as it will have an Euler number of 1. The Euler number is typically around 1-15. The higher the Euler number a fluid has, the higher the extensional viscosity is.

2.2 Measurement of extensional viscosity

A challenge in measuring extensional viscosity is the difficulty to establish and maintain a uniform elongational flow field for an extended time, in order for a steady state to be reached (Arnolds et al., 2010). Methods used to measure extensional viscosity in the past are filament stretching devices and opposed jet rheometers, capillary breakup extensional rheometers (Zheng and Chen, 2023), orifice rheometers (Heinikainen, 2012), as well as fiber spinning, tubeless siphon and stagnation flow (Gauri and Koelling, 1997).

Several reliable methods for the measurement of extensional viscosity have been established (Yang et al., 2011). Suitable equipment is needed for different coating processes in order to obtain reliable results, as the equipment is suited for different extension rates. Figure 4 shows common measuring equipment and coating processes and their respective extension rates.

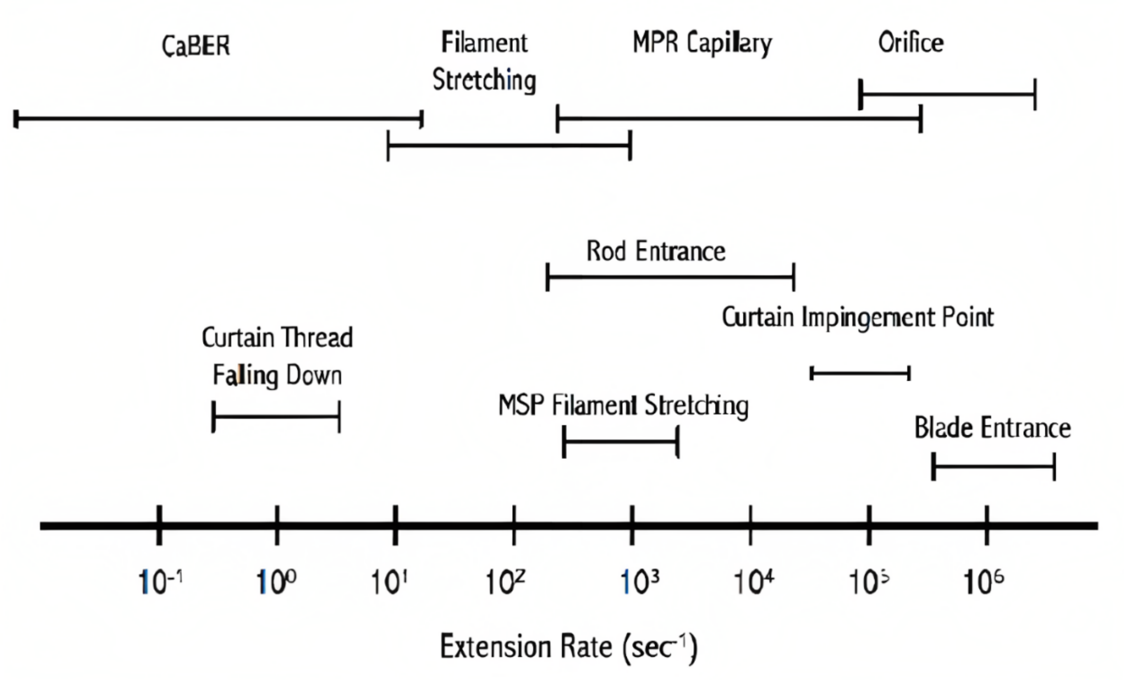

Figure 4. *Extension rate ranges for different coating processes and relevant test equipment (Yang et al., 2011).*

2.2.1 Orifice rheometer

The measurements of extensional viscosity in this study were performed with the ACA AX 100 high shear viscometer. The main parts for the measuring process are the main unit, sample cylinder, capillary holder with seals, orifice holder with seals, along with the capillaries and orifices themselves.

The orifice rheometer is a special kind of capillary viscometer (TAPPI, 2023). Instead of using the Bagley correction, where the capillary length is extrapolated to zero, an orifice with “zero” length is used instead. When running a test, accelerating flow is created at the entrance of the orifice. The pressure loss here is recorded and compared to kinetic energy. As a result, the Euler number is obtained, indicating the level of extensional viscosity in the sample. The Euler number is at lowest 1, as kinetic energy will always be present. High Euler numbers signify high extensional viscosity. The Euler number is measured as a function of velocity, with the standard velocity for coating colors being 15 m/s. It is, however, important to remember that the entrance pressure loss also depends on the shear viscosity of a fluid, which means the comparison of results can be problematic if the coating colors have very different shear viscosities. Schematics can be seen in figure 5 A.

2.2.2 Filament stretching rheometers

In filament stretching rheometers, a cylindrical liquid bridge is formed between two circular plates (Różańska, 2017). As the plates move apart, the sample is subjected to strong extensional deformation, creating a filament. If the sample has sufficiently high viscosity, the extensional viscosity can be derived from measuring the tensile stress as a function of strain. The capillary breakup extensional rheometer (CaBER) is a filament stretching rheometer that is used to measure the extensional viscosity of relatively low viscosity materials at low flow rates. The apparent extensional viscosity can be calculated by measuring the diameter of the formed filament with a laser as a function of time. Schematics of a typical filament stretching rheometer can be seen in figure 5 B.

2.2.3 Multi-pass rheometer

The multi-pass rheometer consists of two pistons and a test section that can hold either an orifice or capillary in between them (Mackley and Hassell, 2011). When using the orifice geometry, the extensional viscosity can be calculated by measuring the pressure difference across the test section and the movement of the pistons. Schematics can be seen in figure 5 C.

2.2.4 Capillary viscometer

Extensional viscosity parameters can be obtained with a capillary viscometer by analyzing the pressure loss when a fluid enters the capillary (entrance effect) (Lehtimäki, 2021). If the measured viscosities vary greatly when using capillaries with the same diameter but different lengths, the difference can be attributed to extensional viscosity. Schematics can be seen in figure 5 D.

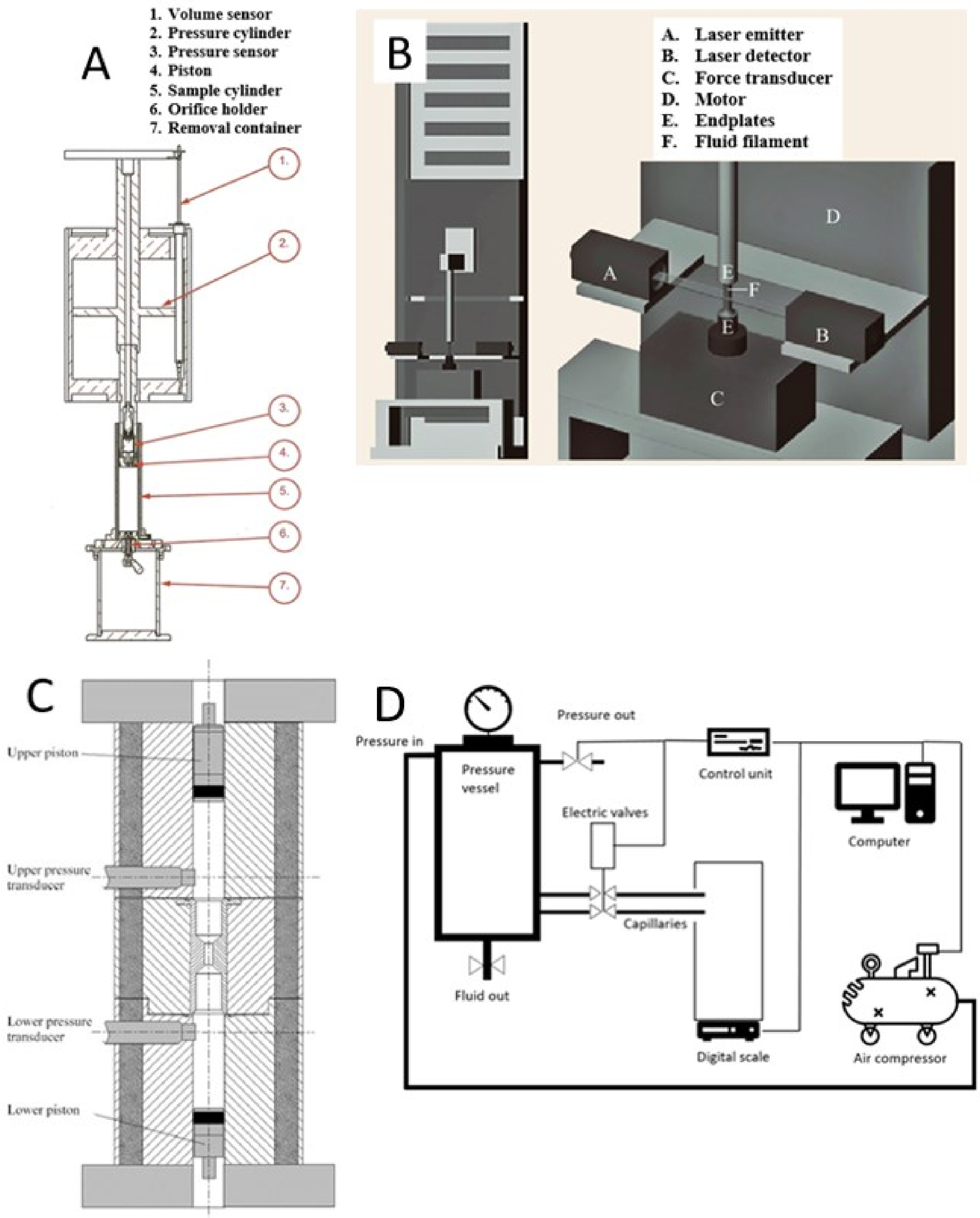

Figure 5. *Schematics of beforementioned rheometers.* ***A)*** *Orifice rheometer (TAPPI, 2023),* ***B)*** *Filament stretching rheometer (Nijenhuis et al., 2007),* ***C)*** *Multi-pass rheometer (Engmann and Mackley, 2006),* ***D)*** *Capillary viscometer (Lehtimäki, 2021).*

2.2.5 Miscellaneous rheometers

A patent by W. Gleissle (2002) proposed a rheometer that can determine both shear and extensional viscosity of a sample. Structurally viscous liquids such as polymer melts and solutions and other structural liquids are measured by passing the liquid through two capillaries connected in series. In the first capillary, the liquid is sheared only within a defined shear velocity range, where the shear viscosity is measured. The second capillary has a same or smaller shear velocity range. The liquid in the second capillary will face flow-through resistance comprising of both shear and strain resistance. Pressure measurements are used to determine the flow-through resistance. The strain viscosity is then separated by extracting the pressure fraction of the shear flow resistance. This is done by subtracting the portion of the pressure difference corresponding to this shear flow resistance by using the measurements in the first capillary. Three different configurations can be seen in figure 6 E.

Another patent by J. Parnaby, and C. Humphries (1986) describes a rheometer where extensional viscosity, elasticity and optionally shear viscosity can be determined for molten polymers or other fluids. The rheometer contains an open-ended die, which includes converging and diverging sections as well as optional sections of constant cross-sectional size and shape. Pressure measurements are made on the flow of the fluid at measured flow rates. The technique for determining the extensional viscosity and elasticity is based on the influence of reversible elastic energy storage in the converging flow, and the release of this stored energy in the diverging flow. The geometric characteristics of the die cavity, as well as the locations of pressure measurements are strategically selected in conjunction with the chosen fluid mechanical assumptions. This ensures that the elastic energy storage and release are compensated in relation to at least one function. This allows for a separation of pressure drop effects caused by fluid elasticity from that caused by the combined influence of shear and extensional viscosity. After this separation process, it becomes possible to calculate both elasticity and extensional viscosity. Schematics can be seen in figure 6 F.

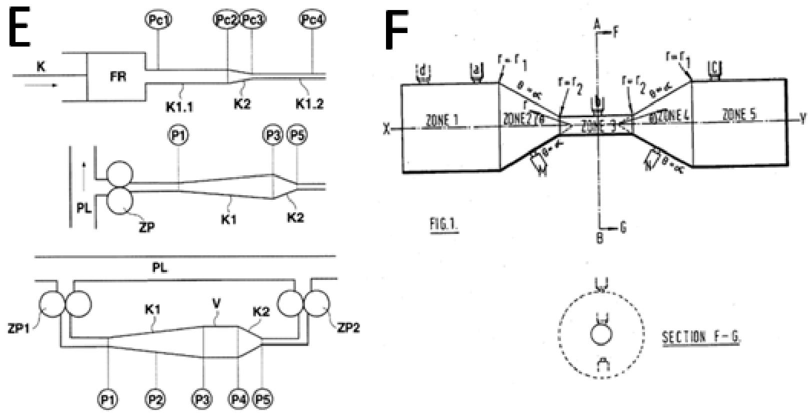

__Figure 6.__ Proposed rheometer schematics. __E)__ three different configurations of the rheometer proposed by Gleissle (2002), __F)__ rheometer proposed by Parnaby and Humphries (1986).

2.3 Control of extensional viscosity

Extensional viscosity can be modified through an addition of certain polymers into the coating color dispersion (Heinikainen, 2012). The properties of the polymer in question, its concentration and the solids content will all play a role in the extensional viscosity. The higher the molar mass and the longer the molecule chain is, the more this will increase the extensional viscosity. Long polymer chains that are curled up at rest and resist stretching out when faced with extensional stress, tend to have high extensional viscosity, as shown in figure 7. Long polymers with high molar mass alone are not enough to increase extensional viscosity, it is necessary for them to be coiled up at rest. Traditional thickeners tend to fail to increase the extensional viscosity because of this.

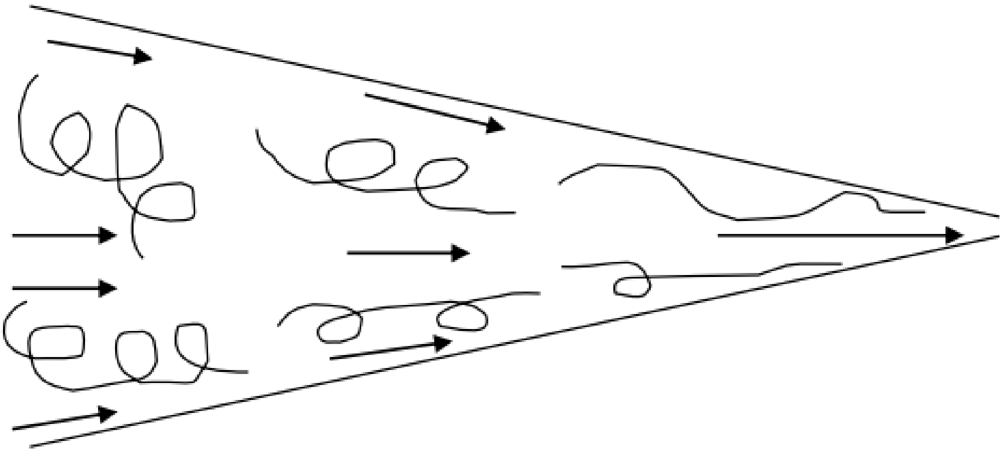

Figure 7. *Polymers in accelerating flow (Ojanen, Sinkko, and Kunnas, 2007).*

The use of starch does not give noteworthy increases in extensional viscosity, while the addition of carboxymethyl cellulose increase extensional viscosity only slightly (Ojanen, Sinkko, and Kunnas, 2007). The use of surfactants does not increase extensional viscosity either.

Xanthan gum has been used in food products and pharmaceuticals to increase extensional viscosity (Mackley et al., 2013). It is a better option than starch, since xanthan gum is able to increase the extensional viscosity better than starch at same shear viscosities.

Polyacrylamides (PAM) and polyvinylpyrrolidones (PVP) have also been found to be capable of increasing extensional viscosity and can for example be used to improve performance in inkjet printing (Branham, 1997).

Hydroxypropyl cellulose (HPC), polyacrylatic acid (PAA) and polyethylene oxide (PEO) have been used to increase extensional viscosity (Hidema et al., 2017). While the extensional viscosity of PEO increase with increasing extension rates, the extensional viscosity of HPC and PAA decrease with increasing extension rates. This was explained by the differing relaxation times of the polymer solutions. Polyacrylate has also been used as an anti-misting component in spray applications due to its extensional properties (Hodge et al., 2015).

Polystyrene (PS) and poly(methyl-methacrylate) (PMMA) may also give extensional viscosity under certain conditions (Narimissa, Poh and Wagner, 2021). The extensional viscosity is dependent on the number of entanglements, as well as the ratio of entanglement molar mass to critical molar mass in the linear viscoelastic region.

The use of polyvinylalcohol (PVOH) in water-based dispersions has been shown to increase the extensional viscosity to a degree (Lehtimäki, 2021). Both the degree of hydrolysis and the molar mass are important factors in how well it improves extensibility. A low degree of hydrolysis gives high extensibility, as it allows the use of a smaller coil. A higher molar mass and dry weight percentage also increase extensional viscosity.

Polyethylene oxide (PEO) increases the extensional viscosity well at a wide range of shear rates, making it a very promising additive for curtain coating colors. A higher molar mass and dry weight percentage increases the extensional viscosity. A considerable downside to the use of polyethylene oxide is its low solubility in water.

Papers on the behavior of polymeric fluids show that they can exhibit extensional thickening or extensional thinning behaviors (Yang et al., 2011). An explanation for this could be that in extensional thickening polymeric fluids, a three-dimensional network is created between latex, pigments and thickeners. This network retrogrades to its original shape during extensional stress. For extension thinning fluids, the lack of this three-dimensional structure based on the properties of the thickeners could lead to thinning behaviors. Under extensional deformation, the molecule chains have the required flexibility to align themselves, and therefore reduce the extensional viscosity as extensional rate increases.

2.4 Coating processes and relevance of extensional viscosity

There are several coating methods and processes that benefit greatly from suitable levels of extensional viscosity. The extensional viscosity of the coating color can have different effects in the process depending on the coating method in question. Such coating methods include curtain coating, film coating, blade coating, and roll coating.

2.4.1 Curtain coating

Curtain coating has gathered interest from the paper industry in recent years (Ojanen, Sinkko, and Kunnas, 2007). The method offers a process with economic advantages when compared to conventional coating methods. Due to curtain coating being a contactless method, it will reduce web breaks and improve the overall efficiency of the process. Curtain coating is a relatively straightforward coating technique. A curtain is formed, which falls with gravity on a moving web, evenly coating the material moving below. Applying thin films at high speeds is ideal, but there is still a minimum flow rate to take into consideration, as going below this will cause complications. Air bubbles should also be avoided, as it will compromise the coating layer. A schematic of a typical curtain coater can be seen in figure 8.

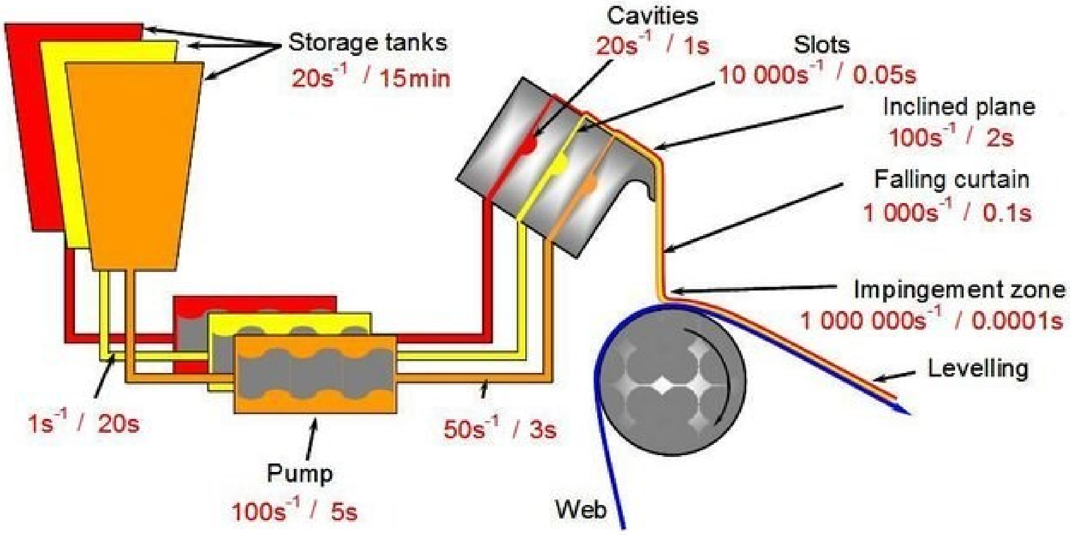

Figure 8. *Shear stress and residence time of traditional curtain coater (Martinez, 2011).*

Curtain coating can be performed either with a slot die, or a slide die. With a slide die, one or several layers can be coated simultaneously. With the slot die, only up to three layers can be applied at the same time. In curtain coating, the use of conventional coating colors can lead to problems in runnability and cause surface defects. A stable curtain can, however, be achieved by optimizing the parameters. This does not eliminate all problems, as some surface defects and problems with the stability of the curtain will remain. These problems originate from the coating color, namely the inadequate surface tension and extensional viscosity. Extension in the coating color during the coating process may result in uncoated areas in the coating. This can be prevented by using suitable additives in the coating color.

The material being coated moves at relatively high speeds, as the liquid coating falls from a considerable height before impinging on the material (Becerra and Carvalho, 2011). Due to the impingement velocity, air entrainment in the dynamic contact line is reduced, as well as helps in the wetting of the substrate. These factors have made curtain coating one of the preferred methods for precision coating. Curtain coating has some challenges, however, as a uniform layer of liquid can only be achieved at a limited region of operational parameters, which is usually called the “coating window”.

There are four potential major failures in curtain coating, namely bead pulling, heel formation, air entrainment and curtain breakup, as shown in figure 9. Bead pulling is when the falling curtain is pulled downstream, which happens at high web speed to curtain velocity ratio. Heel formation is when the contact line moves upstream, which happens at low web speed to curtain velocity ratio. Air entrainment is when thin air-pockets are entrained under the liquid bead, which happens at high web speeds. Finally, curtain breakup is when the curtain stops falling as a uniform sheet, and breaks into separate columns, which happens when the flow rate is below a critical limit.

Figure 9 shows the coating window and the above-mentioned coating failures (Mohammad Karim et al., 2018). U_{web}/U stands for the ratio of the web speed to the curtain velocity at impingement. The Reynolds number here is defined in equation 9 as

$$\boldsymbol{Re = \frac{\rho Q}{\eta},} \qquad [9]$$

where ρ is the liquid density, Q is the flow rate per unit width, and η is the liquid viscosity.

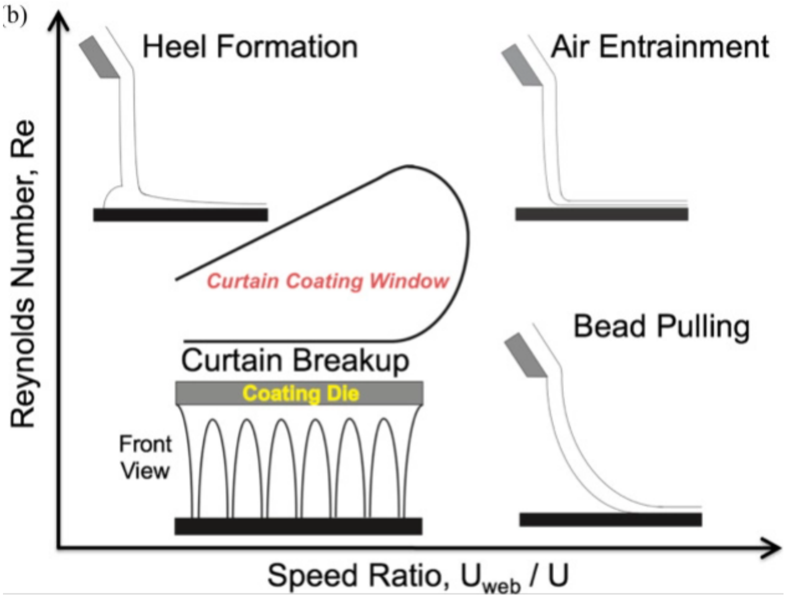

Figure 9. *The coating window in curtain coating (Mohammad Karim et al., 2018).*

According to Mohammad Karim et al (2018), the instability and breakup of liquid curtains was first studied by G. I. Taylor in 1959. Following the analysis by Taylor, D. R. Brown performed the first experimental study on the stability of Newtonian liquid curtains. Brown proposed a stability criterion for Newtonian liquid sheets, namely the Weber number. A Weber number of two or higher has been found to be needed for a stable curtain. This has, however, been found to not always be applicable, as the causes for the breakup of the curtain are far more complicated than anticipated. Some curtains have been proven to stay intact at Weber numbers under one. The formula for the Weber number is shown in equation 10 as

$$\boldsymbol{We = \frac{\rho Q v_c}{2\sigma}}, \qquad [10]$$

where ρ is the density of the liquid, Q is the curtain flow rate per unit width, v_c is the liquid curtain velocity, and σ is the surface tension of the liquid.

Surface tension

All materials have intermolecular forces that keep the molecules together (Rapp, 2023). Without these forces, material would decompose into individual molecules. These forces are balanced in all directions throughout the volume of the material. There is, however, an imbalance of forces at the surface of a material. The cohesive forces in a liquid are stronger than those in a gas. The outermost layer, for instance in water, has no neighboring molecule to one side to interact with, and the forces are therefore skewed towards the bulk of the mass. This can cause the surface to form meniscus, or curve at the top. The smaller the diameter of the container, the larger the curve.

The Weber number mentioned previously compares the inertial forces to the surface forces in a free-flowing curtain (Tripathi, 2005). In order for a stable curtain to form at low flow rates, the surface tension should be reduced in order for the Weber number to stay below 2. In order to reduce the surface tension, a wide variety of surfactants can be used. Choosing the correct surfactant is important, as they tend to differ in effectiveness and efficiency. The time it takes for a newly formed surface from the die exit to impinging is usually only fractions of a second (Birkert et al., 2005). It is therefore important to have fast moving surfactants, in order for them to have the desired effect. Slow moving surfactants may simply not have the time to move to the surface and should therefore be avoided.

As the curtain falls, the surface area, and therefore the surface tension, will increase (Tripathi, 2005). The curtain age is therefore an important factor to consider. The height the curtain falls determines the age of the curtain. The curtain age is the time it takes to leave the slot die, until it reaches the impingement point. The change in surface tension compared to the surface age is one of a surfactant's most important characteristics.

Curtain stability

The stability of the curtain in curtain coating is of great importance (Becerra and Carvalho, 2011). An unstable curtain will not leave an even smooth surface on the paper. The stability of the curtain sets the minimum flow rate that can be used. The breaking of the curtain can have different origins, it can start from the edges, or begin in the middle, caused by impurities such as dust particles. Viscous forces have been proven to hinder the growth of holes in the curtain, therefore enabling a lower minimum flow rate. Addition of high molar mass polymers such as PEO gives the curtain higher extensional viscosity, effectively stabilizing it, preventing the growth of holes. In the study by Becerra and Carvalho (2011), increasing extensional viscosity by the addition of small amounts of high molar mass polymers did not considerably change the shear viscosity, meaning the pressure inside the die, the pressure drop in the feeding system, and the characteristics of the leveling remained largely the same. The clear effects were limited to the parts where elongational deformation occurs, which is the falling curtain.

The coating color accelerates and stretches as the curtain falls towards the web, typically by a factor of 1.5-10, leading to strain rates of around 100 to 1000 1/s (Ojanen, Sinkko, and Kunnas, 2007). The curtain is stretched again within fractions of a millisecond, as it impinges on the moving paper web, usually resulting in an extension of 5-20. This stretching is stronger, leading to strain rates of 100 000 to 1 000 000 1/s. Typical extension rates of the coating color can be seen in figure 10. According to Ojanen, Sinkko, and Kunnas (2007) the Euler number should be at least 5 for shear rates of 20 000 1/s, or problems due to lack of extensional viscosity may occur in curtain coating.

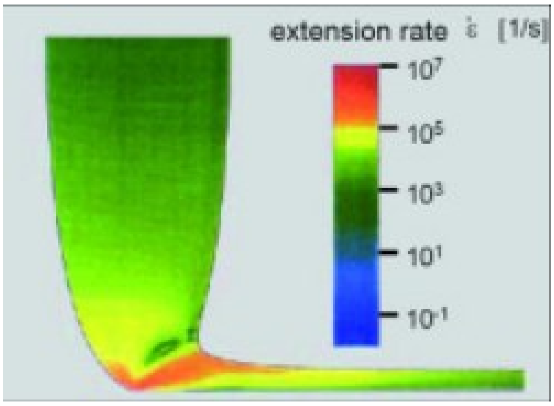

Figure 10. *Extension rates of coating color in falling curtain (Birkert et al., 2005).*

The velocity of a falling curtain can be calculated as seen in equation 11:

$$\boldsymbol{v_c = \sqrt{v_0^2 + 2gx},} \qquad [11]$$

where v_0 is the initial curtain velocity, g is the gravitational acceleration, and x the distance from the curtain edge (Birkert et eqal., 2005). The Reynolds number can then be calculated to determine flow type, seen in equation 12:

$$\boldsymbol{Re = \frac{\rho v_c h_c}{\eta},} \qquad [12]$$

where ρ is the density, v_c is the curtain speed, h_c is curtain height, and η is the viscosity.

The extensional viscosity plays an important role in the stability of the curtain, especially at previously mentioned moments of extension, at the impingement point and while falling. There are three important requirements for achieving better stretchability in coating colors. The molecules used must be very long, extending into the aqueous phase over long distances.

The molecules should also form hyperstructures in the aqueous phase when interacting with pigments. Finally, the polymer must contain functional groups which will interact with the continuous medium. The continuous medium is usually water, in which case the polymer backbone ought to contain hydrogen donor and acceptor moieties.

Pigments in the coating color play an important role during coating, in regard to the shape and size of particles. An obvious requirement is to keep the particle size under the minimum thickness of the curtain thickness, or the integrity of surface tension and the curtain dimensions can be compromised. Traditional blade coating formulations having high elasticity and water retention properties have been modified for curtain coating, as to prevent flocculation. This has mainly been done by the use of high molar mass polymers instead of thickeners, as they behave as extensional modifiers independent from the other coating color components and avoid the flocculation.

2.4.2 Film coating

In film coating, also called the (pre)metered size press, the coating color is metered onto a transfer roll as a film (Toivakka, 2015). The substrate, in this case paper, is then moved in between the transfer roll and backing roll, where the film is transferred to the paper at the nip. Both sides of the web are typically coated at the same time, allowing for simultaneous two-sided coating, which is a big advantage of film coating compared to methods that only allow coating of one side at a time. A schematic of a traditional film coater can be seen in figure 11.

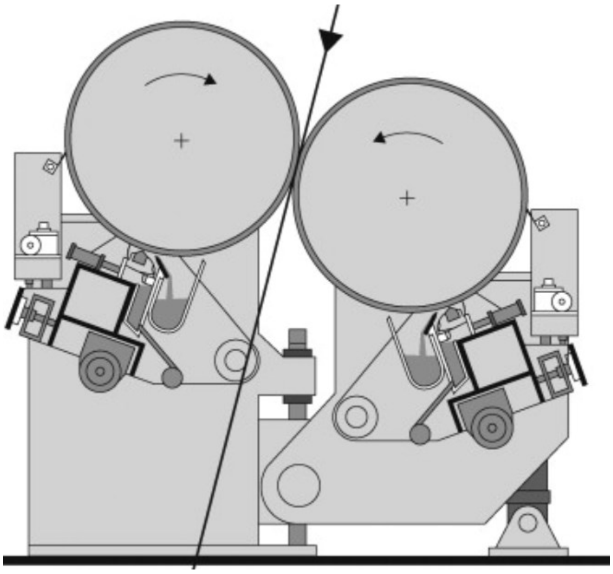

Figure 11. *Schematic illustration of a metered size press (Maurer, 2009).*

Both shear and extensional deformation are generated in film coating (Yang et al., 2011). These deformations happen as filament stretching at the nip exit in film coating. It was proven that the extensional viscosity has a significant effect on misting in film coating. As the extensional viscosity increases, so does the misting. This could happen due to the higher extensional viscosity slowing down the breaking up of growing filaments. This leads to the droplets being larger when the filament finally does break. Additionally, the breakup point of filaments will also be pushed further away from the nip with increasing extensional viscosity. One of the main problems with high-speed film coating is the occurrence of misting at the applicator roll nip outlet (Sugihara et al., 2003).

2.4.3 Blade coating

Blade coating is a method where a film with a well-defined thickness is formed (Cherrington and Liang, 2016). A sharp blade is pressed against the surface that is to be coated at a fixed distance. The coating color is placed in front of the blade, which creates an even wet film. The blade coating process can be seen in figure 12.

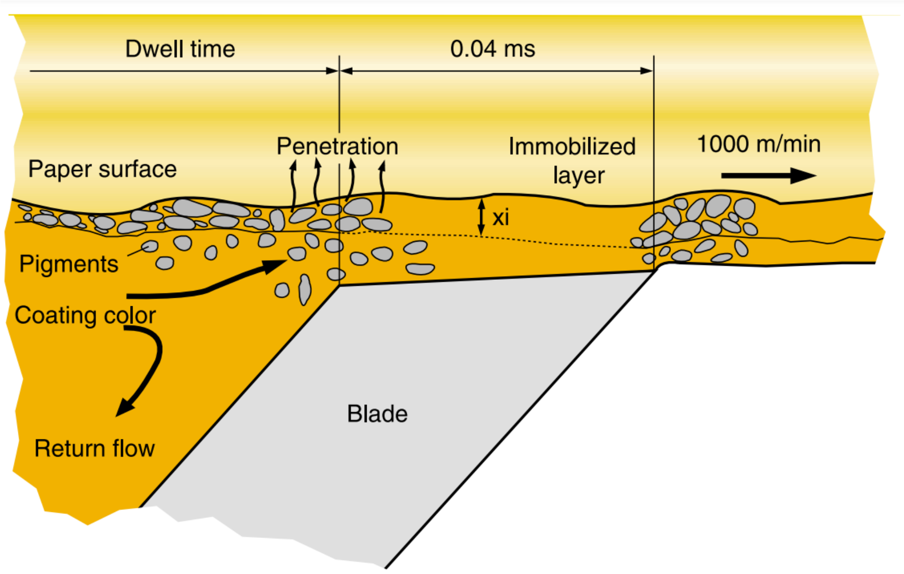

Figure 12. *Blade coating of paper surface (Lehtinen, Paulapuro, and Gullichsen, 2000).*

In blade coating, the blade and rod pressure increased with increasing extensional viscosity (Yang et al., 2011). The deformation can happen in the entrance flow of coating color into a metering blade or rod in blade coating. A perpendicular force is generated by the extensional viscosity compared to the shear direction, which affects the blade. This force increases the blade load, which in turn increases the blade lift.

2.4.4 Roll coating

In the nip region between a roll and the substrate, a high fluid pressure region can be found at the nip, and a low-pressure region at the nip-exit (Fernando, Xing and Glass, 2000). As the fluid travels through the variable pressure region, phenomena such as ribbing, web growth, filament formation and misting may occur. In certain process conditions, cavitation can occur when the pressure drops below the vapor pressure of the coating liquid. Mist droplets are then created as the cavitation leads to the formation of filaments that break up, as seen in figure 13. The deformation involved in the filament break up process is extensional.

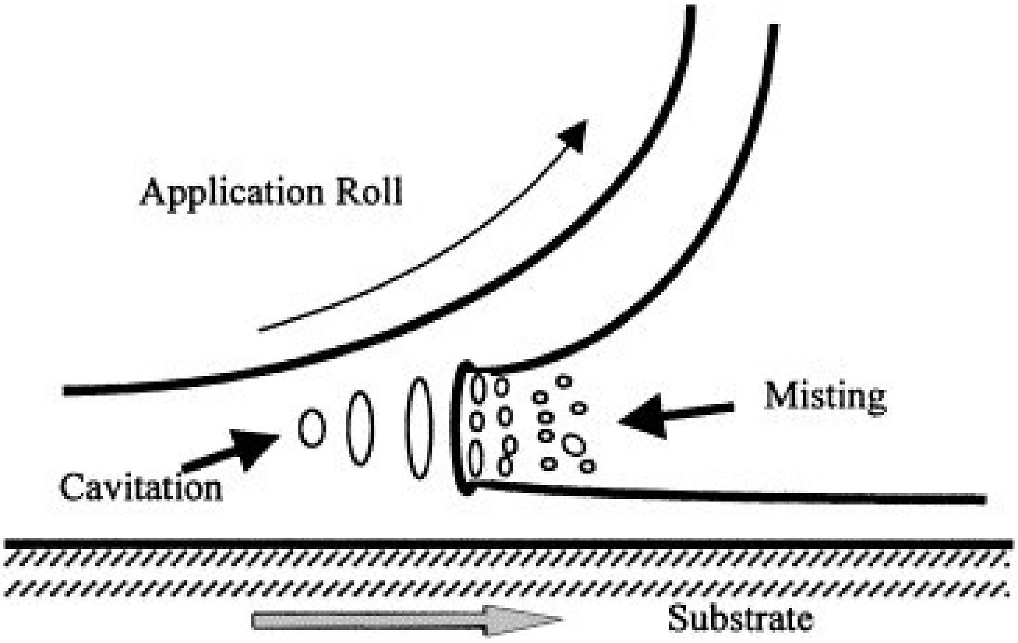

Figure 13. *Cavitation and misting in roll application (Fernando, Xing and Glass, 2000).*

At low speeds (5-59 m/min), coatings with low extensional viscosity does not support the filament growth that leads to misting. Coating formulations with higher extensional viscosity generates a higher degree of misting. At higher speeds (720 m/min), the growth of filaments that broke up and caused spatter is also observed.

2.5 Other processes and relevance of extensional viscosity

2.5.1 Gravure printing

Gravure printing is used to print large amounts of magazines and catalogues (Roth, Søndergaard and Krebs, 2015). The ink is transferred from engraved micro cavities embedded in the printing cylinder. An impression cylinder pushes the web towards the primary printing cylinder, and the ink is transferred to the web as seen in figure 14. Gravure printing is very dependent on ink viscosity, substrate speed and pressure.

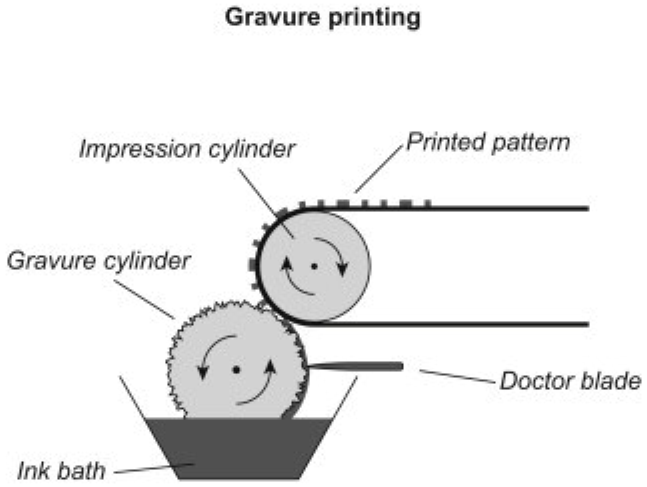

Figure 14. *Gravure printing (Roth, Søndergaard and Krebs, 2015).*

During gravure printing liquid bridges are formed, which influences the liquid transfer to the substrate (Sankaran and Rothstein, 2012). The liquid bridges are stretched, resulting in strong shear and extensional flow components. Due to extensional flow playing an important role, the extensional rheology of the coating fluid must be consistent in order to get optimal print quality and resolution.

2.5.2 Inkjet printing

Extensional viscosity can play a significant role in inkjet printing by influencing the behavior of the ink (Basaran, Gao and Bhat, 2013). When an inkjet droplet is formed, it involves the thinning of the liquid filament that connects the droplet with the rest of the liquid inside the nozzle. Extensional flow is predominant inside the thinning filament. The droplet size and velocity can also be affected by extensional viscosity (Yoo and Kim, 2015). Extensional viscosity must be taken into account for the ink formulation and printer design in order to ensure precise and high-quality printing results.

A patent by B. Branham (1997) presented an inkjet formulation with a method to reduce the generation of aerosols during inkjet printing using a viscoelastic polymer. The inkjet formulation which is comprised of one dye and an aqueous based vehicle reduced aerosol formation by addition of polyacrylamides with molar mass of 10 000 to 5 000 000 and polyvinylpyrrolidones with molar mass of 3000 to 1 000 000 g/mol, or a mixture of the two. The viscoelastic polymer serves several purposes. Firstly, it increases both extensional viscosity and surface tension of the expelled ink, increasing the likelihood of the tail portion to remain intact. Secondly, even if tail fragmentation occurs, the viscoelastic polymer shifts the point of fragmentation further away from the head, delaying the fragmentation so the detached ink remnants are closer to the print medium when formed. Any ink that manages to break away from the tail will likely result in satellite drops of sufficient size to not be dominated by aerodynamic forces. Implementation of the formulation in inkjet printing should enhance print quality and reduce aerosol formation.

2.5.3 Polymer systems

A wide variety of processing operations for polymeric melts are affected by extensional flow, such as fiber spinning, film blowing, blow molding and foaming (Arrigo, Malucelli and Mantia, 2021). Extrusion and injection molding, that are dominated by shear flow, are also affected by extensional flow. In extrusion molding, extensional flow plays a role in the entrance of the die and variation of the flow channels. In injection molding, it plays a role when the melt is injected into the mold gate.

Uniaxial extension occurs in melt spinning, while biaxial extension occurs in film blowing. Schematics of melt spinning and film blowing can be seen in figure 15.

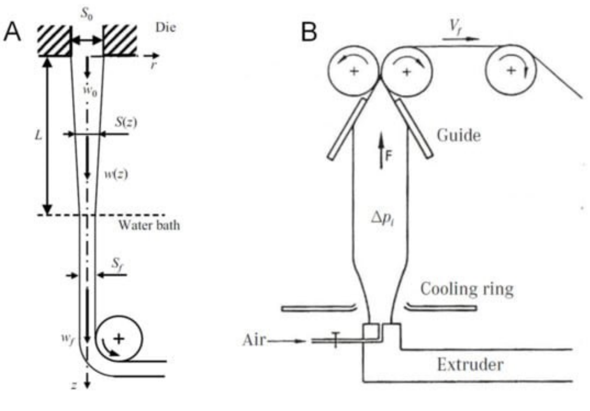

Figure 15. *Schematics of* ***A)*** *melt spinning and* ***B)*** *film blowing (Arrigo, Malucelli and Mantia, 2021).*

2.5.4 Aircraft safety fuel

Kerosene-based aircraft fuel was tested using a triple jet system, where the solution is stretched as it flows through a capillary tube (Oliver and Ashton, 1976). Cylindrical inserts were placed in the capillary tube, to alter the shear history. This showed to have a large effect on the extensional behavior of the fuel. The extensional viscosity of the fuel was raised by an order of magnitude when high shear was used for long periods of time, with the effects lasting up to a second. The aircraft safety fuel is intended to eliminate misting during crash situations.

2.5.5 Food processing

In the food industry, both shear and extensional viscosities are important to understand the behavior of products during processing (Arabo, 2011). One example is the production of dough. The understanding of dough viscosity is important for product quality control and equipment design for automated bakeries. The industrial processing of dough can involve extensional flows. The viscosity and behavior of the dough can depend on the properties of the wheat flour used, the combination of prepared dough, rest time and the mixing conditions. Understanding and having the correct extensional properties in the dough will help optimize the production process and ensure consistent product quality.

2.5.6 Cleaning compounds

A patent by I. Ajmani, L. Tin, and C. Choy (2005) describes the use of extensional viscosity in cleaning compounds. In particular, hypochlorite compositions were made to have optimal viscosity and elasticity. The thickened cleaners are designed to be used for the cleaning of vertical and inclined surfaces, as well as for opening clogged drains. The thickened cleaning compositions have the benefit of increasing the effectiveness of the cleaning agent by increasing the residence time and reducing unwanted effects such as spreading onto unwanted surfaces, misting and sputtering. This composition, when used as a drain opener, provides the advantage of ready penetration through standing water (blocked flow) and adherence to partial clogs (slow flow). This is due to viscous formulations being better suited for partial flows, and elastic formulations better suited for complete clogs. The viscoelastic properties were measured with a Bohlin VOR rheometer with oscillation. The viscoelastic thickening system is comprised of at least a hexadecyl dialkyl amine oxide, a tetradecyl dialkyl amine oxide and an organic counter ion. Optionally, an active cleaning compound can also be added. Additional components may also be added to improve stability, rheology, efficacy, and aesthetics for consumer acceptance of a commercial product.

2.5.7 Softening of tissue paper

A patent by Barnholtz et al. (2003) describes a composition that would soften absorbent tissue paper. One of the main uses for this tissue paper is to cleanse the skin without causing irritation. This composition would include a softening active ingredient, a vehicle in which the softening active ingredient is dispersed, an electrolyte dissolved in the vehicle, a bilayer disrupter and a high polymer. The high molar mass polymers are useful in order to achieve the desired extensional viscosity in the softening composition, which in turn can help to minimize spray fracture during spraying. The extensional viscosity was measured using a capillary thinning rheometer.

2.5.8 Spray applications

A patent by C. Hodge et al. (2015) describes concentrate sprayable compositions that include anti-misting components. Anti-misting components include polyethylene oxide, polyacrylamide and polyacrylate, or a mixture of them. In one variation, when the concentrated sprayable composition contains a sufficient amount of anti-misting component, the concentrate is diluted with water and dispersed through a transient trigger sprayer, leading to the resulting solution having increased median droplet size and decreased mist or aerosol formation. In one scenario, the sprayable solution produces minimal or no small particle aerosol. In another scenario, the median droplet size exceeds 50 microns. Enlarging the droplet size of the dispersed solution has been observed to reduce the risk of inhalation and minimize the formation of aerosol or mist. These sprayable compositions may be used in various environments where larger droplet sizes are desired when discharged from a transient trigger sprayer. Examples of areas where this can be used are food and beverage applications, vehicle care applications, medicinal applications, pest control operations, and laundering applications. Such applications include but are not limited to cleaning of laundry or textiles, antimicrobial cleaning, general purpose cleaning, surface cleaning, carpet and vehicle cleaning and destaining, and more. Polyethylene oxide with a molar mass of 3 million to 7 million and concentration of 0.01 and 0.3 w-% has been found to be suitable. Polyacrylamide has been found to be suitable with a molar mass of 8 million to 16 million and a concentration of 0.01 to 0.3 w-%.

2.5.9 Dysphagia

People with dysphagia, or difficulty swallowing, commonly use thickened fluids as a form of medical management (Hadde et al., 2019). Dysphagia is common in older people and can lead to malnutrition or even asphyxiation. The flow behavior of the food at the moment of swallowing (bolus) is affected by its rheological properties. Shear rheology plays an important role in the flow of the bolus, as thicker fluids have longer throat transit time. An increase in extensional viscosity reduces the elongation of the bolus during swallowing, which reduces risk of bolus breakage. Breaking of the bolus can cause problems, such as allowing the fluid to flow into the gullet or airways. Some of the bolus may also get stuck in the throat as residue, needing several swallows to clear, as well as being inhaled as breathing continues after swallowing.

The extensional viscosity of fluids with similar shear viscosity is dependent on the thickeners used. As an example, a study (Mackley et al., 2013) found that food thickened with xanthan gum show higher extensional viscosity than fluid thickened with starch, even if they have similar shear viscosity. A twin piston filament stretching device was used on the fluids, where the starch thickened fluids were stretched in a non-homogenous way, leading to early breaking of the bolus. The xanthan gum thickened fluid showed a uniform deformation. In the study by Hadde et al. (2019), it is concluded that higher extensional viscosity in the bolus is desirable, as it is stronger and will better resist elongation. The bolus can therefore better keep its shape as it is elongated during swallowing, and resists fracturing.

3 Experimental

3.1 Materials

A latex-based barrier dispersion was used as base in this study. The properties can be found in table 1.

Table 1. *Barrier dispersion properties.*

DISPERSION	SOLIDS CONTENT	DENSITY	BROOKFIELD VISCOSITY
CH-POLYMERS CHP EP 1 “Experimental product”	50%	1100 kg/m^3	97 mPas

Several additives were used in this study. The properties can be found in table 2.

Table 2. *Properties of used additives.*

ADDITIVE	PROPERTIES
NOURYON FINNFIX 4000G	CMC, 450 000 g/mol
KURARAY POVAL 95-88	PVOH, 88% hydrolyzed, 450 000 g/mol
SIGMA ALDRICH POLYETHYLENE OXIDE	PEO, 8 000 000 g/mol
SIGMA ALDRICH POLYETHYLENE OXIDE	PEO, 600 000 g/mol

3.2 Dispersion preparation

The dry weight percentages of the additives were measured with a Radwag MA 50.R (Radwag Balances and Scales, Poland) moisture analyzer, to add the correct amount into the solution.

The PEO and CMC solutions were mixed with the Silverson L5M-A (Silverson, United Kingdom) mixer. Once the correct amount additive was measured, it was added very slowly into the appropriate volume of distilled water under mixing with the Silverson mixer. To dissolve the additives faster, a water bath of 50 °C was used at times. The speed of the mixer was varied from below 1000 rpm in the beginning, to over 4000 rpm in the later stages. If mixing took several days, the solution was mixed overnight with the Heidolph mixer, while covered by aluminum foil to minimize evaporation. The addition of additives was then continued in the same way the next day.

The PVOH pellets were dissolved in a metal beaker with distilled water at 20 °C under constant stirring with a Heidolph mixer with propeller geometry. It was dispersed slowly over approximately 30 minutes. A solution of 12 weight-percent was the goal. The metal beaker with the solution was then placed in a water bath and heated to 95 °C for approximately an hour, still under stirring. The solution was covered with aluminum foil to minimize water loss due to evaporation. Afterwards, the solution was taken out from the water bath and left to cool under mixing until it was under 50 °C. The solution was left to cool further overnight to 20 °C.

After the solutions were mixed, the dry weight percentage was always measured with the Radwag MA 50.R and oven to ensure the correct amount would be added into the dispersion.

The solutions were added to the reference dispersion in correct amounts with a large syringe to obtain desired weight percentages of additives. The solids contents of the dispersions were kept at 45%. The Heidolph mixer was used to mix the dispersions. After the dispersion was well mixed, measurements could be performed.

3.3 Methods and apparatus

Measurements of the same sample were performed two or three times, in order to ensure correct measurements and repeatability. An example of this can be found in the results part of this study.

3.3.1 Rotational viscometers

A Brookfield viscometer was used to measure the viscosities of the dispersions. Spindle number 4 was used at a speed of 100 rpm. The measurement was taken after the reading had stabilized in approximately 30 seconds. All measurements were done at 20 °C.

A modular compact rheometer (MCR302, Anton Paar) was used to measure the low shear viscosities of the dispersions. The geometry used was cone and plate (CP-50).

3.3.2 ACA AX-100 high shear capillary viscometer

The ACA AX-100 was the main instrument for this study. An orifice plate with a 0.5 mm orifice diameter and 0.5 mm thickness was used to obtain the Euler numbers.

The shear viscosities were measured with two different capillaries. A capillary with a 0.5 mm diameter and 50 mm length was used at high pressure, and a capillary with a 0.9 mm diameter and 90 mm length was used at low pressure. The used geometries can be seen in figure 16 below, while the ACA AX-100 schematic can be found in figure 5 A in the theory part above.

The orifice and the smaller capillary were calibrated with water before measurements. The orifice had to have an Euler number of 1 ± 0.02. This was obtained by changing the diameter of the orifice in the program until the desired Euler number was achieved. The capillary was calibrated by measuring the flow of the water, which was then put into a third-party program that calculated the effective diameter of the capillary.

The sample cylinder was filled with a dispersion, approximately 0.2 L where after the measurement took place. After a measurement, any excess material left in the cylinder was removed and the cylinder was refilled. All measurements were done the same day the dispersions were mixed, at 20 °C.

Water calibrations of the capillaries were done afterwards in order to wash out any remaining dispersion.

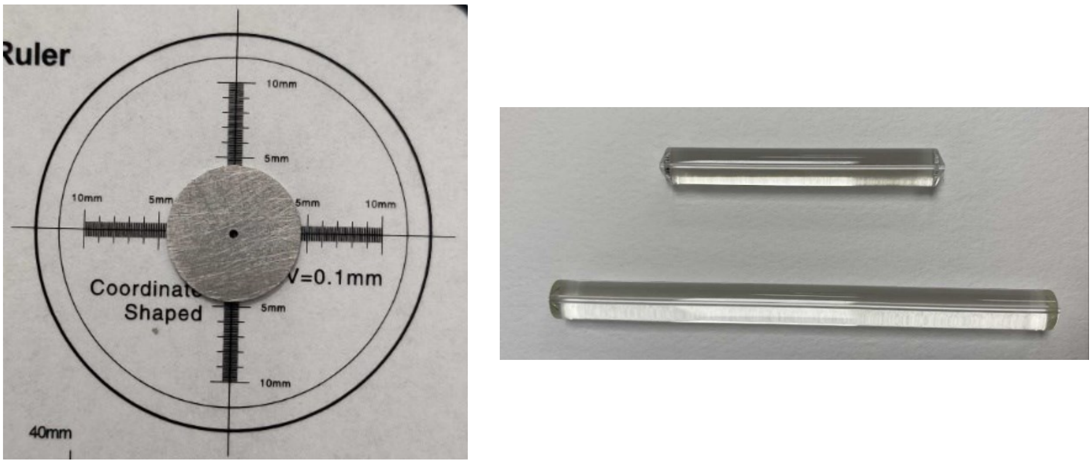

Figure 16. *Orifice plate 0.5x0.5 mm and capillaries 0.5x50 mm & 0.9x90 mm.*

3.3.3 Mixers

The mixer used for the PEO and CMC solutions was a Silverson mixer. The polyvinyl alcohol solution was mixed in a heated water bath with a Heidolph mixer. The final dispersions were mixed with a Heidolph mixer.

4 Results

This chapter shows and explains the results gathered from the ACA AX-100 high shear capillary viscometer, as well as MCR and Brookfield measurements. Six different dispersions were measured and compared. The CHP EP 1 “Experimental product” with no additives was used as a reference dispersion. Dispersions with PEO 8 000 000 g/mol were tested at 0.3 w-% and 0.15 w-%, PEO 600 000 g/mol at 0.3 w-%, PVOH at 1.5 w-%, and CMC at 0.3 w-%.

The repeatability of measurements was ensured by measuring each dispersion up to three times. An example of this can be seen in figure 17 below. The repeatability was good.

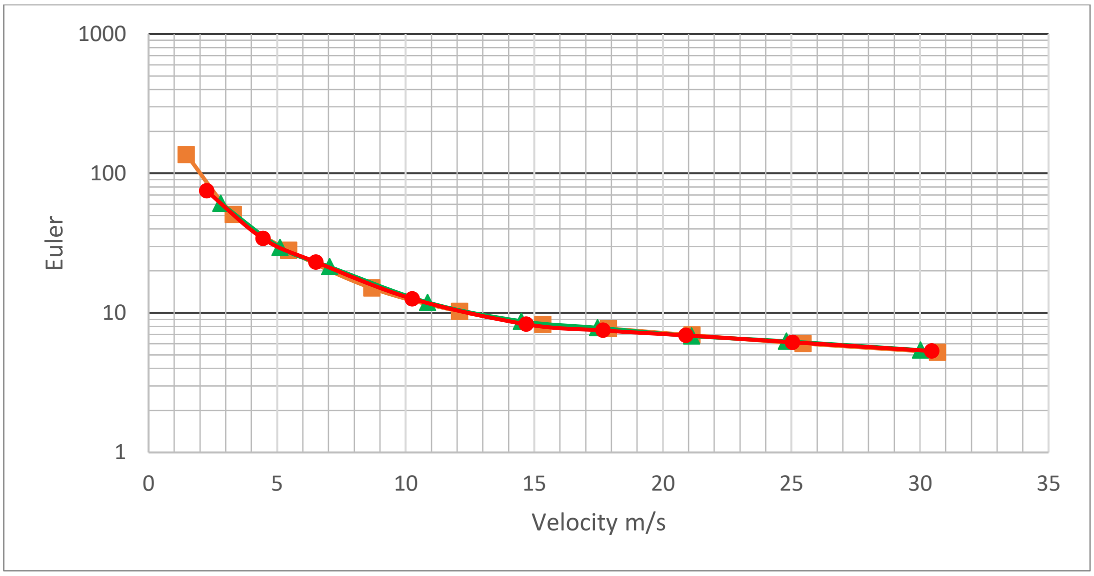

Figure 17. *Example of three consecutive measurements of PEO 8 000 000g/mol 0.3w-% with ACA AX-100 orifice 0.5x0.5mm geometry.*

4.1 Shear viscosities of dispersions

When measuring the viscosity of the dispersions, a wide variety of parameters can affect the results. These include, but are not limited to, temperature, concentration, molar mass, structure, and shear rate. The viscosity of high molar mass polymers tends to be shear thinning (George, 2009). The polymers used in this study have relatively high molar mass, ranging from around 450 000 g/mol up to 8 000 000 g/mol.

An MCR302 was used to measure the viscosities of the dispersions at low shear rates, as seen in figure 18. The geometry used was cone and plate (CP-50). Each measurement was done twice to ensure reliable results. In the figures below, 8 000 000 g/mol and 600 000 g/mol is written as “8M” and “600k” respectively.

All the dispersions showed pseudoplastic behavior. This behaviour can be attributed to the alignment of the polymers in the direction of flow, which reduces the flow resistance due to the more streamlined arrangement of the polymer chains. The PVOH had the highest viscosity, followed by CMC. The PEO dispersions came next, with the 8 000 000 g/mol 0.3 w-% dispersion having highest viscosity out of the three. The two other PEO dispersions, 8 000 000 g/mol 0.15 w-% and 600 000 g/mol 0.3 w-% had a relatively similar viscosity. Lowest shear viscosity by far was that of the reference dispersion, whose slope is also less steep compared to the other dispersions.

The addition of polymers to the dispersion will increase the viscosity (Kalyanasundaram, Sundaresan and Hemalatha, 2000). This is due to the interactions between the solvent and the polymer. Different polymers will also affect the viscosity differently, depending on their properties, such as size of the polymers and structure. It is therefore easily understandable that the reference dispersion with no additives will have the lowest viscosity of all samples.

As stated before, the concentration of polymers in the dispersion will also have an effect on the viscosity. A higher concentration will lead to higher viscosity, due to there being more large molecules causing drag and interacting with each other (Anon, 2021). The concentrations of additives in this study ranged from 0.15 w-% up to 1.5 w-%. The PVOH dispersion, with the highest additive concentration at 1.5 w-%, also had the highest viscosity, as seen in figure 18. The relation between concentration and viscosity can best be seen in the two PEO 8 000 000 g/mol dispersions, where the only differing parameter was the concentration. The higher concentration of 0.3 w-% dispersion clearly had higher viscosity than that of the 0.15 w-% dispersion over all shear rates. It is, however, important to remember that the concentration is just one parameter that affects viscosity, meaning concentration alone will not determine which dispersion has the highest viscosity.

The molar mass is also an important parameter for viscosity measurements. The molar mass of the additives in this study ranged from approximately 450 000 g/mol to 8 000 000 g/mol. Higher molar mass will lead to higher viscosity, due to increased drag and intermolecular attraction (Anon, 2021). This can be observed in the case of the two PEO dispersions with different molar masses, each having the same concentration of 0.3 w-%. The dispersion with 8 000 000 g/mol had a higher viscosity over all shear rates than that of the 600 000 g/mol.

A. Gertan (2012) studied the rheological characteristics of PEO and concluded that low molar mass PEO had Newtonian behavior, while higher molar mass showed pseudoplastic behavior depending on the concentration. Due to the high molar masses used in this study, pseudoplastic behavior can clearly be seen in the measurements.

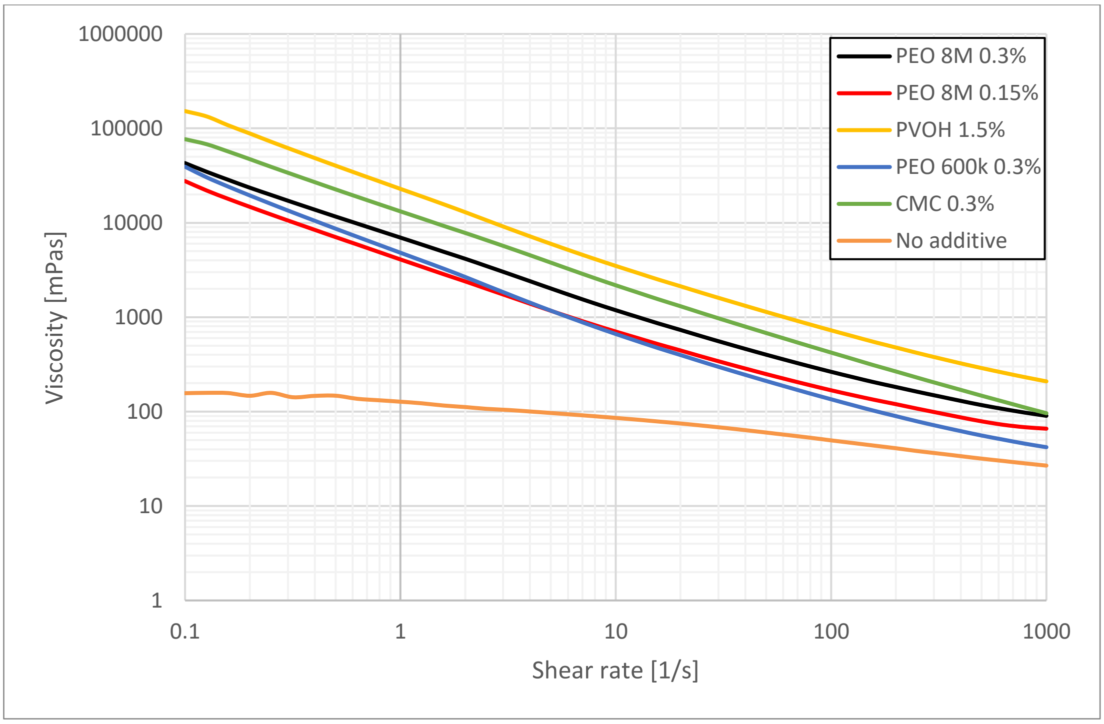

Figure 18. *Flow curve of dispersions with MCR302.*

The ACA AX-100 high shear capillary viscometer was used to measure the shear viscosities at higher shear rates, as seen in figure 19. Two capillaries were used, 0.5x50 mm at high pressure and 0.9x90 mm at low pressure. The 0.5x50 mm capillary at high pressure manages to reach higher shear rates, while the 0.9x90 mm capillary is able to measure lower shear rates. They do, however, overlap in shear rates for all dispersions. The majority of datapoints gathered were between 10 000 and 1 000 000 1/s. At higher shear rates, most dispersions have relatively stable viscosities.

The PVOH dispersion has a unique curve, where it displayed pseudoplastic behavior at lower shear rates starting at under 10 000 1/s, after which it suddenly showed strong dilatant behavior at around 100 000 1/s, as seen in figure 19. The CMC dispersion also exhibited pseudoplastic behavior at lower shear rates, only to stabilize at approximately 20 mPas around 100 000 1/s.

The PEO 600 000 g/mol 0.3 w-% dispersion had a viscosity around 30 mPas. PEO 8 000 000 g/mol 0.15 w-% had a little higher viscosity at most shear rates, until finally overlapping with the PEO 600 000 curve at little under 1 000 000 1/s. Their viscosities did not change significantly over the measured shear rates. The PEO 8 000 000 g/mol 0.3 w-% dispersion viscosities did vary more than the rest between the two capillaries. The viscosity stays somewhat stable, however, at around 50 to 60 mPas at most shear rates. The differences in shear viscosity between the PEO dispersions can be attributed to the differences in concentration and molar mass. Higher molar mass and concentration leads to higher viscosity. A lower concentration of the same polymer led to lower viscosity (PEO 8 000 000 g/mol, 0.15 w-%), as did lowering the molar mass while maintaining the same concentration (PEO 600 000 g/mol, 0.3 w-%).

Finally, the reference dispersion without additives maintained a stable viscosity throughout all shear rates, at little under 20 mPas.

The shear thinning effects of CMC can be attributed to the disentanglement and increased orientation in the direction of the flow of the polymers in the dispersion (Chalah et al., 2022). This leads to less resistance, and therefore lower viscosity. The same reason for the shear thinning effects can be applied to PEO (Ebagninin, Benchabane, and Karim, 2009). Slight shear thickening behaviors could be the result of a formation of a stiff inner structure caused by the entanglement and increased intermolecular interactions. The shear thickening behavior of PVOH may be related to macromolecular conformation changes induced by the flow (Lewandowska et al., 2019). In dilatant fluids, the liquid tends to lubricate the motion of the particles past each other in at lower shear rates, leading to less resistance and lower shear viscosity (Amoo and Layi Fagbenle, 2020). At higher shear rates, however, the material dilates or expands, leading to there being an insufficient amount of liquid to fill the void. This can lead to solid-on-solid contact, which will increase the friction and therefore also the viscosity. This can explain the sudden shift from pseudoplasticity to dilatancy in the PVOH dispersion.

Separate graphs for 0.5x50 mm and 0.9x90 mm geometries can be found in the Appendix.

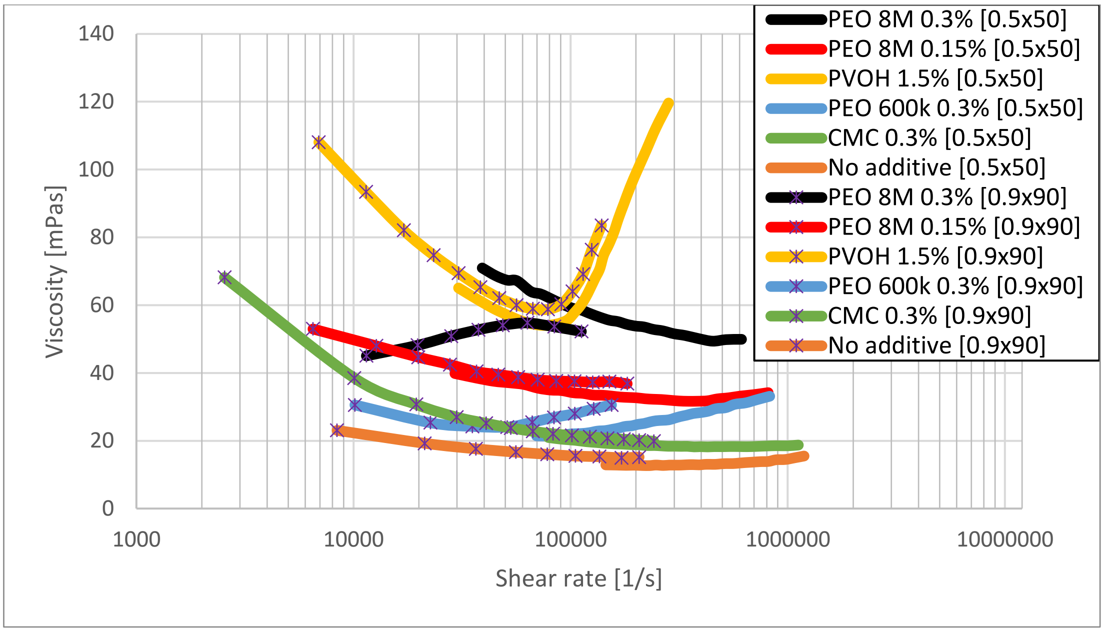

Figure 19. *Viscosities of dispersions using ACA AX-100 with geometries capillary 0.5x50 mm at high pressure and 0.9x90 mm at low pressure.*

For the Brookfield measurements seen in table 3, spindle 4 was used at a speed of 100 rpm for all dispersions except PVOH. Spindle 5 had to be used for PVOH, as spindle 4 could not give a reading. As with the MCR302 measurements, the order of highest viscosity to lowest is the following: PVOH, CMC, PEO 8 000 000 g/mol 0.3 w-%, PEO 8 000 000 g/mol 0.15 w-% and PEO 600 000 g/mol 0.3 w-% with very similar viscosity, and the reference dispersion last.

Table 3. *Brookfield results for all dispersions.*

Dispersions	**Brookfield [mPas]**
PEO 8 000 000g/mol 0.3%	914
PEO 8 000 000g/mol 0.15%	512
PEO 600 000g/mol 0.3%	492
PVOH 1.5%	2380
CMC 0.3%	1310
No additive	97

4.2 Euler number of dispersions

The Euler numbers of all six dispersions were measured and compared using the ACA AX-100 high shear capillary viscometer, with the standard 0.5x0.5 mm orifice geometry. According to ACA Systems, the Euler numbers can be interpreted as shown in table 4 at the standard of 15 m/s.

Table 4. *Interpretation of the Euler number.*

Euler Number	Extensional viscosity
1 – 2	Low/insignificant
2 – 5	Moderate
Over 5	High

As seen in figure 20, polyethylene oxide 8 000 000 g/mol 0.3 w-% had a significantly higher Euler number than all other dispersions. The PEO 8 000 000 g/mol 0.15 w-% and PVOH showed similar moderate increase in the Euler number. PEO 600 000 g/mol 0.3 w-% had a slightly lower impact on the Euler number, while CMC had a very low impact. The reference dispersion with no additives showed barely any increase in Euler number. The increase of the Euler number at low flow velocities, below 10 m/s, as shown in figure 20, is due to the low kinetic energy of the flow and should not be interpreted as an indication of high extensional viscosity. The area of interest is between velocities of 10 and 20 m/s, with an emphasis on the proposed standard of 15 m/s. A zoomed in graph can be found in the appendix.

Several factors play a role when increasing the extensional viscosity of a dispersion. The properties of the polymer in question, its concentration and the solids content are important factors to consider. Higher molar mass and longer molecule chains will increase the extensional viscosity more. It is necessary for the polymers to be curled up at rest, and resist stretching out when faced with extensional stress to have high extensional viscosity. The solids content for the polymer dispersions were kept at 45%. The different additives used and their effect on the Euler number will be discussed in further detail below.

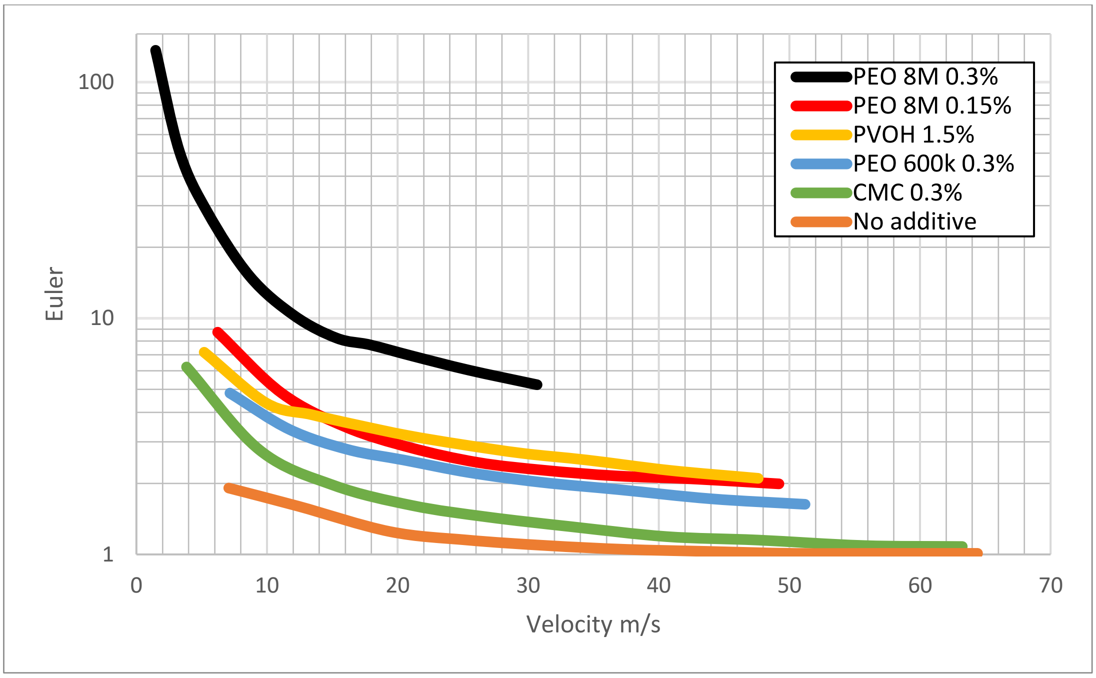

Figure 20. *Graph of the Euler numbers of all six dispersions.*

The Euler numbers rounded to one decimal at the standard velocity of 15 m/s can be seen in figure 21. Once again, PEO 8 000 000 g/mol clearly had the highest Euler number at 8.5. PVOH and PEO 8 000 000 g/mol 0.15 w-% share the same Euler number of 3.7. They are followed by PEO 600 000 g/mol 0.3 w-% with an Euler number of 2.8, CMC at 2.0, and the reference dispersion at 1.4.

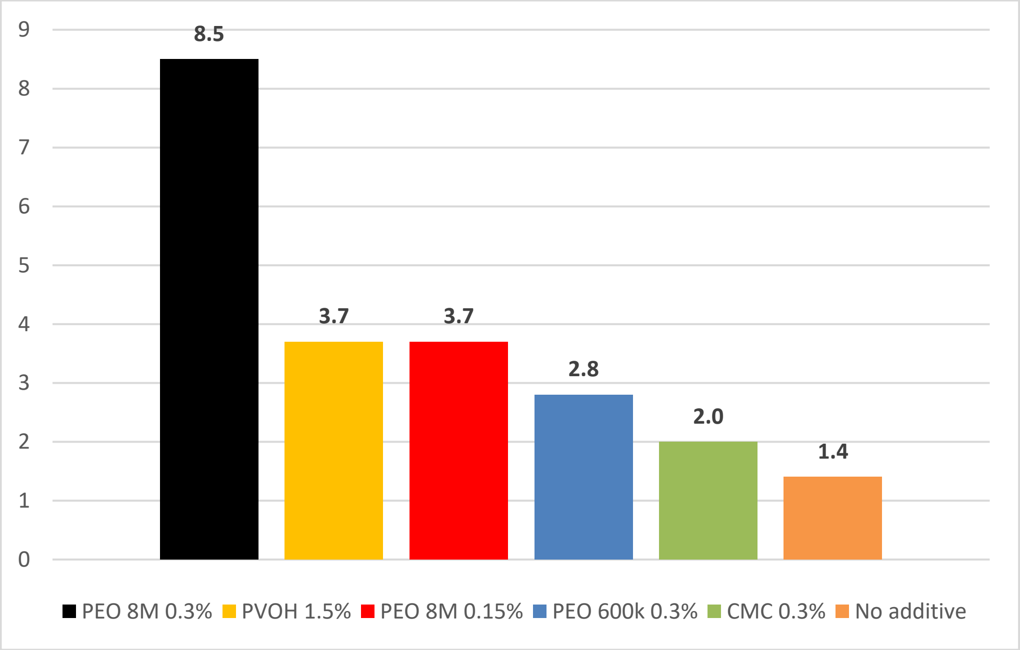

Figure 21. *Euler numbers of dispersions at 15m/s at one decimal.*

As per ACA Systems interpretation, the results can be summarized as the CMC and the reference dispersion (no additive) having low/insignificant extensional viscosity, the PVOH, PEO 600 000 g/mol 0.3 w-% and PEO 8 000 000 g/mol 0.15 w-% having moderate extensional viscosity, and the PEO 8 000 000 g/mol 0.3 w-% having high extensional viscosity.

4.2.1 Effects of CMC on Euler number

The effects of CMC on extensional viscosity was studied by Kokko (2001). In her experiments, CMC failed to show an increase in extensional viscosity in pigment-based coating colors. Her theory was that CMC was incapable of creating networks necessary for increasing the extensional viscosity.

Lehtimäki (2021) did further studies on CMC and its role in extensional viscosity in curtain coating colors. Like Kokko, he found no significant impact of CMC in extensional viscosity.

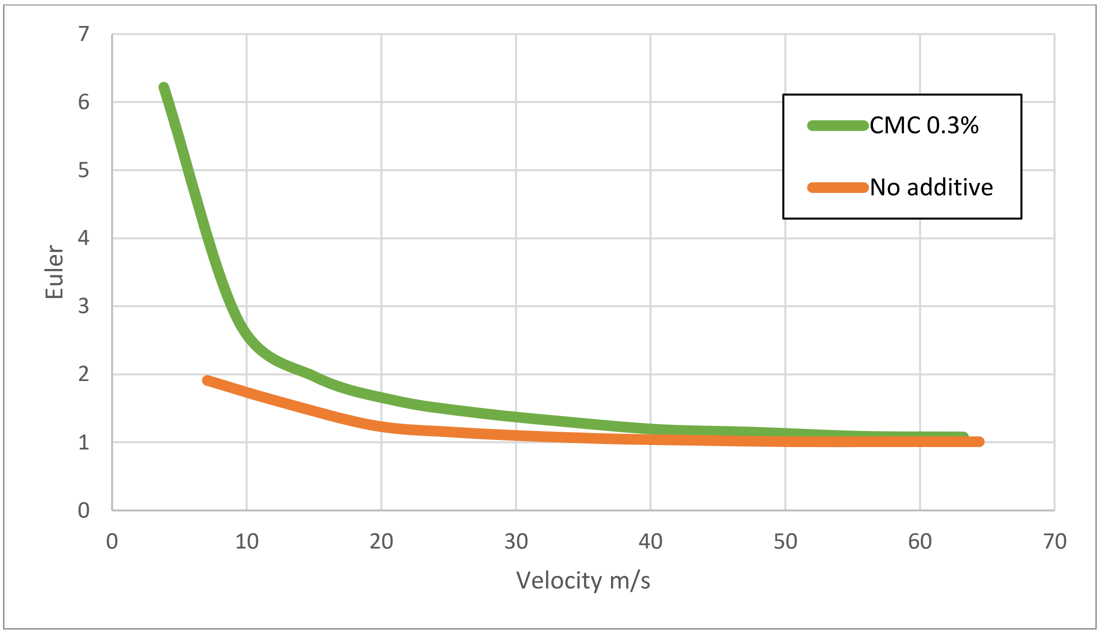

Figure 22. *Effect of 0.3 w-% carboxymethyl cellulose dispersion on Euler number.*

The measurements done in this study with CMC has also failed to show any significant increase in Euler number and thus extensional viscosity, as seen in figure 22. Once again, it is important to point out that the increase of the Euler number at low flow velocities below 10 m/s is due to the low kinetic energy of the flow and should not be interpreted as an indication of high extensional viscosity.

The lack of extensional viscosity can be explained due to CMC being a polyelectrolyte, where the charged segments will stiffen due to repulsion, creating a chain that is far less coiled than the ideal chain. CMC is therefore not a suitable polymer for increasing extensional viscosity.

The Brookfield viscosity for the reference dispersion with no additive was 97 mPas and the 0.3 w-% CMC dispersion was 1310 mPas.

4.2.2 Effects of PVOH on Euler number

Kokko (2001) studied the effects of PVOH on extensional viscosity. Kokko concluded that dispersions with high molar mass PVOH, which show dilatant behavior, show good extensional viscosity due to strong PVOH network that resists high shear. The low molar mass PVOH showed very low extensional viscosity, similar to that of CMC. No clear tendency for dilatancy was apparent either.

Lehtimäki (2021) studied various different polyvinyl alcohols and their effect on extensional viscosity. The PVOH was capable of increasing the extensional viscosity to an extent. A high molar mass is needed for PVOH to increase the extensibility, with the degree of hydrolysis also being important. A lower degree of hydrolysis allows for smaller coil, which will increase the extensibility.

The polyvinyl alcohol used in this thesis is Kuraray Poval 95-88. In Lehtimäki’s thesis, this was the polyvinyl alcohol that gave the highest apparent extensional viscosity.

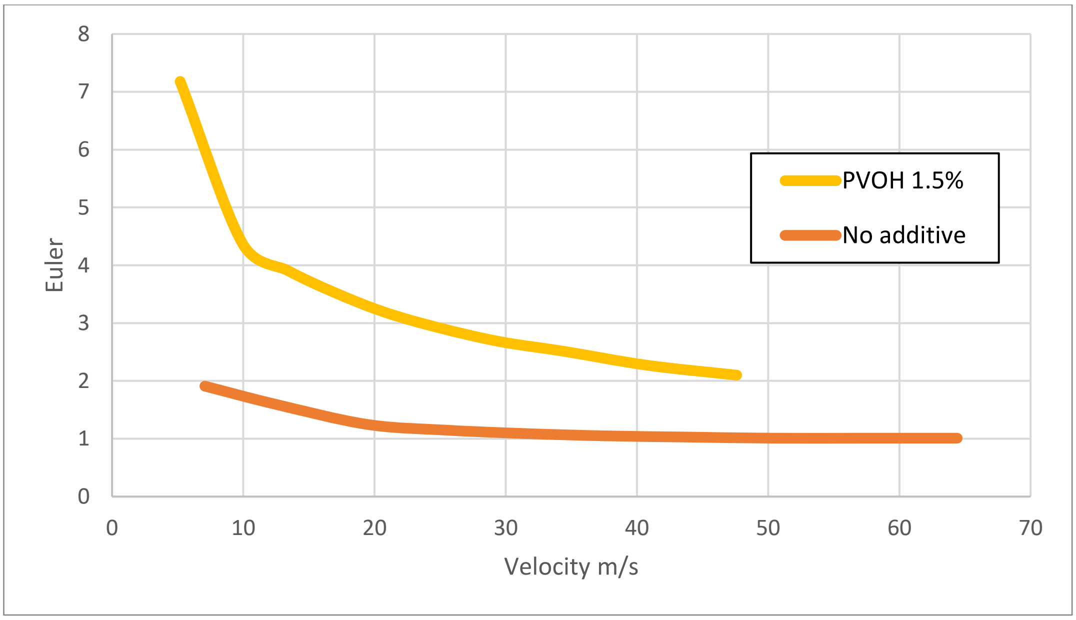

Figure 23. *Effect of 1.5 w-% polyvinyl alcohol dispersion on Euler number.*

As seen in figure 23, the PVOH dispersion used in this study shows a moderate increase in Euler number. The PVOH showed strong dilatant behavior at high shear rates, had relatively high molar mass and lower degree of hydrolysis, which are factors that were important for extensional viscosity according to Kokko and Lehtimäki respectively. PVOH seems to be a relatively good polymer for increasing extensional viscosity, as long as the before mentioned criteria are met.

The Brookfield viscosity for the 1.5 w-% PVOH dispersion was 2380 mPas.

4.2.3 Effects of PEO on Euler number

The addition of high-molar-mass polyethylene oxide can significantly increase the Euler number. Three different PEO-based dispersions were measured. In Lehtimäki's thesis (2021), the PEO dispersion measured gave far higher apparent extensional viscosity compared to all other dispersions.

Gauri and Koelling (1997) studied the extensional viscosity of PEO solutions with different concentrations. With higher concentrations, the strain rates needed to reach a Trouton ratio of 3 decreased. They attributed this to an increase in entanglements due to the higher concentration. Due to the flexible structure of the PEO chain, high molar mass PEO solutions were extension thickening in elongational flow. With increased shear, high Trouton ratios were reached. PEO showed good ability to increase the extensional viscosity.

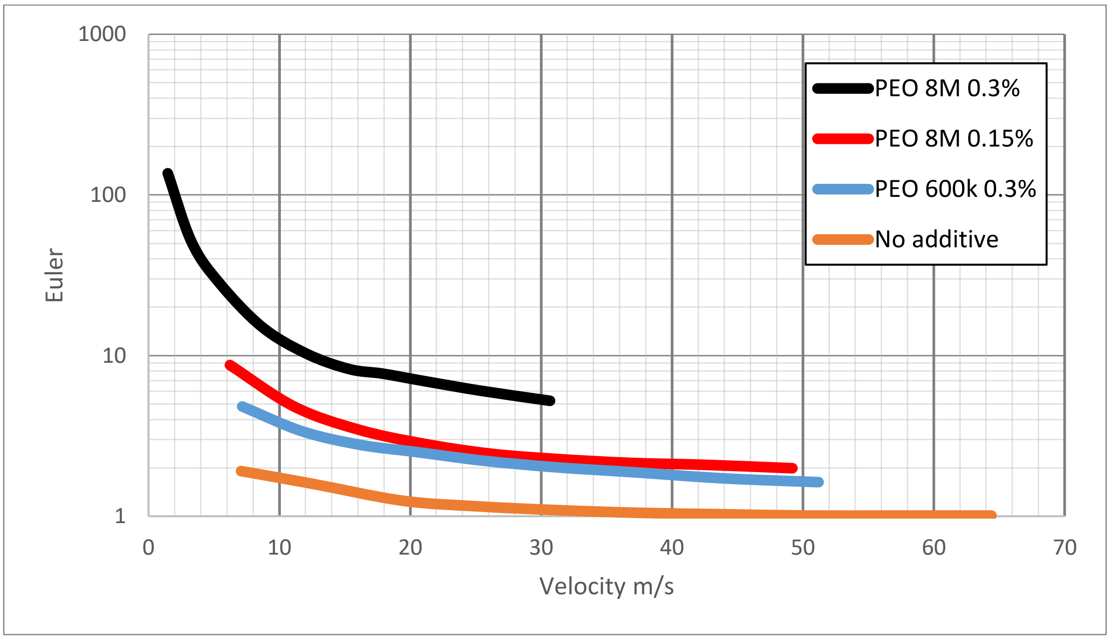

Figure 24. *Effect of PEO dispersions on Euler number.*

In this study, as in Lehtimäki's study, PEO gave the highest Euler number and thus the highest extensional viscosity of all used polymer dispersions. As expected, higher molar mass and higher concentration of PEO clearly correlates to higher Euler numbers. As seen in Figure 24, the Euler number of PEO 8 000 000 g/mol 0.3 w-% is significantly higher than the other PEO dispersions.

Even the lower molar mass PEO 0.3 w-% 600 000 g/mol, and the lower concentration 0.15 w-% 8 000 000 g/mol give moderate increases in Euler number, similar to that of the 1.5 w-% PVOH dispersion. The lower concentration dispersion had, however, a slightly higher Euler number over the lower molar mass dispersion at all velocities.

PEO seems be a very suitable polymer for achieving high extensional viscosity. This could be attributed to the high molar mass of the polymers, the entanglement of the polymers, the flexibility of the polymer chain, and the resistance to stretching out from their coiled state at rest.

The Brookfield viscosities for the 8 000 000 g/mol PEO dispersions were 914 mPas for 0.3 w-%, and 512 mPas for 0.15 w-%. The 600 000 g/mol PEO dispersion had a Brookfield viscosity of 492 mPas.

5 Conclusions

Extensional viscosity, also called elongational viscosity, is a fluid’s resistance to extension. It is one of the most important rheological properties, second only to shear viscosity. It is also substantially more difficult to measure than shear viscosity.

Extensional viscosity plays an important role in several different areas. In the paper coating industry, it has significant importance in methods such as curtain coating, as well as film, blade, and roll coating. It can affect misting, stability, layer formation and more. It has in addition many important implications in various other areas, such as in printing, polymer processing, food processing, and medicinal products.

The extensional viscosity of dispersions can be increased by an addition of coiled, high molar mass polymers. The higher the molar mass and the longer the chain is, the more the polymer will increase the extensional viscosity. Long polymer chains that are curled up at rest and resist stretching out when faced with extensional stress tend to have high extensional viscosity. Increasing the concentration of the additive or increasing the solids content will increase the extensional viscosity of the dispersion.

In this study, the extensional viscosity of dispersions was measured as the Euler number. It represents the relationship between loss of pressure and kinetic energy per volume. The Euler number is as its lowest equal to 1. The higher the Euler number is, the higher the apparent extensional viscosity is.

ACA AX-100 orifice rheometer was used to measure the Euler number. Six different dispersions were tested: CMC, PVOH, three different PEO and one with no additives. The CMC and reference dispersions showed no significant increase in Euler number. The PVOH and two PEO dispersions, one with lower molar mass (600 000 g/mol, 0.3 w-%) and one with lower concentration (8 000 000 g/mol, 0.15 w-%), showed a moderate increase. The last PEO dispersion of higher molar mass (8 000 000 g/mol) and concentration (0.3 w-%) showed a significant increase.

In conclusion, the orifice rheometer seems to be a suitable instrument to characterize extensional viscosity in coating colors. It is easy to use and has a good repeatability.

6 Suggestions for further research

While this study focused on the influence of molar mass and concentration of polymer additives on Euler number of barrier coating dispersions, other parameters such as solids content, pigment content, and temperature should also be investigated. Potential effects of natural polymers and other additives, as outlined in chapter “2.3 Control of extensional viscosity” could also be studied. Furthermore, examining the correlation of viscoelastic properties of materials and their extensional flow properties might provide interesting results.

Beyond material properties, a comparative analysis of results obtained from different orifice geometries would be essential in future work. The roles of the orifice diameter and the entrance angle should be clarified. Sources for the noise in measurements carried out at flow rates below 10 m/s should be paid attention to.

7 Swedish summary

Töjviskositet av ridåbestrykningssmeter mätt som Eulertal

Engångsplaster används i stor utsträckning i industrier, speciellt i förpackningsprodukter, såsom livsmedelsförpackningar. Dessa plaster tar århundranden att nedbrytas, vilket leder till föroreningar. De ekologiska effekterna och konsumenternas efterfrågan har drivit utvecklingen av förnybara, miljövänliga förpackningsmaterial. Pappersförpackningar har haft stor framgång på detta område, men deras barriäregenskaper är klart svagare än traditionella plastförpackningars. För att uppnå önskade barriäregenskaper har ytbeläggningar med biobaserade polymerer utvecklats. Ytbeläggningen förbättrar papperets egenskaper avsevärt. Flera olika bestrykningsmetoder kan användas för att applicera ett eller flera skikt på papper.

Skjuvviskositet definieras som en vätskas resistans mot ett flöde, medan töjviskositet definieras som en vätskas resistans mot ett accelererande flöde. Medan skjuvviskositeten för en beläggningssmet vanligtvis mäts, görs det sällan för töjviskositeten. Töjviskositet är en viktig egenskap i många bestrykningsmetoder, speciellt om beläggningssmeten utsätts för kraftigt accelererande flöden.

Ridåbestrykning är ett exempel på en process där töjviskositeten spelar en viktig roll. Töjviskositeten kan stabilisera ridån och förhindra eller fördröja tillväxten av hål i ridån. Andra bestrykningsmetoder som kan förbättras med lämplig töjviskositet och som beskrivs kort i denna studie är filmbeläggning, bladbestrykning och valslackering.

Töjviskositet är inte endast viktigt i bestrykningsmetoder. Diverse användningsområden diskuteras kort i denna studie, såsom dysfagi, bläckstråletryck (inkjet printing), rengöringsmedel och livsmedelsbearbetning.

Töjviskositeten kan ökas med en tillsats av långkedjade polymerer. Hur mycket polymerkedjan kan töjas från avslappnat till utsträckt tillstånd avgör hur mycket töjviskositet den ger smeten. Det är därmed viktigt att använda sig av långa polymerer som är ihopringlade i vila och motstår utsträckning i ett flöde.

Töjviskositeten kan mätas med diverse reometrar. I denna studie användes ACA AX-100, en reometer med en hålplatta (”orifice rheometer”), för att karakterisera töjviskositeten hos bestrykningssmeten. En öppning med nollängd används. När ett test körs, skapas ett accelererande flöde vid mynningens ingång. Tryckfallet registreras och jämförs med den kinetiska energin. Resultatet är ett Eulertal, som indikerar nivån av töjviskositet i provet.

Målet med denna studie var att jämföra effekterna av olika polymerer på töjviskositeten hos traditionella barriärdispersioner. Att uppnå en lämplig töjviskositet i beläggningssmeterna kan förbättra de slutliga beläggningsskikten och förbättra effektiviteten och körbarheten i olika processer. Det är även viktigt att påpeka att töjviskositeten påverkas av skjuvviskositeten, vilket kan göra det problematiskt att jämföra vätskor med mycket olika skjuvviskositet. Skjuvviskositeten hos bestrykningssmeterna mättes med Brookfield, MCR302 och ACA AX-100 med kapillär geometri.

Polymerer som användes i denna studie var PEO, PVOH och CMC. PEO- och CMC- lösningarna blandades i destillerat vatten med en Silverson L5M-A-mixer, och tillsattes i små doser över flera dagar. Ett varmt vattenbad användes för att accelerera processen. Lösningarna blandades med en Heidolph-mixer över natten. PVOH löstes i en metallbägare med destillerat vatten vid 20 °C under konstant omrörning med en Heidolph-mixer. Metallbägaren med lösningen placerades sedan i ett vattenbad och värmdes till 95 °C under ungefär en timme, fortfarande under omrörning. Lösningen täcktes med aluminiumfolie för att minimera vattenförlust på grund av avdunstning. Därefter togs lösningen ur vattenbadet och fick svalna under blandning tills den var under 50 °C. Lösningen fick svalna ytterligare över natten till 20 °C. Lösningarna blandades sedan med referens-dispersionen för att uppnå de önskade viktprocenterna.

Töjviskositetsmätningarna gjordes tre gånger per dispersion, för att säkerställa repeterbarheten. Referens-dispersionen och CMC-dispersionen visade ingen betydlig ökning av töjviskositeten. PVOH-dispersionen, en lägre molmassa-PEO dispersion (600 000 g/mol, 0.3 viktprocent), och en lägre koncentrations-PEO dispersion (8 000 000 g/mol, 0.15 viktprocent) ökade töjviskositeten måttligt. PEO med både hög molmassa och koncentration (8 000 000 g/mol, 0.3 viktprocent) visade sig öka töjviskositeten av kraftigt. En högre molmassa visade sig därmed öka töjviskositeten mera kraftigt.

Avsaknaden av töjviskositet i CMC-dispersionen kan förklaras genom att de laddade segmenten i polymerkedjan stelnar på grund av repulsion, vilket skapar en kedja som är mycket mindre intrasslad än den ideala kedjan. CMC är därför inte en lämplig polymer för att öka töjviskositeten.

PVOH-dispersionen var kapabel att öka töjviskositeten måttligt. En hög molmassa behövs för att PVOH ska öka töjviskositeten. Hydrolysgraden av PVOH spelar även en roll, en lägre grad av hydrolys ökar töjviskositeten av dispersionen.

PEO verkar vara en mycket lämplig polymer för att uppnå hög töjviskositet. Detta kan hänskrivas till polymerernas höga molmassa, polymerernas intrassling, flexibiliteten hos polymerkedjan och motståndet mot att utsträcka från deras lindade tillstånd i vila. Fastän PEO visade sig vara mycket bra på att öka töjviskositeten, är en nackdel dess svårlöslighet i vatten. En lösning på 3 viktprocent PEO med en molmassa på 8 000 000 g/mol tog över en vecka att lösas i vatten, fastän ett varmt vattenbad och konstant omblandning användes.

Sammanfattningsvis verkar reometern med en hålplatta (”orifice rheometer”) vara ett lämpligt instrument för att karakterisera töjviskositeten i bestrykningssmeter. Den är lätt att använda och har en bra repeterbarhet.

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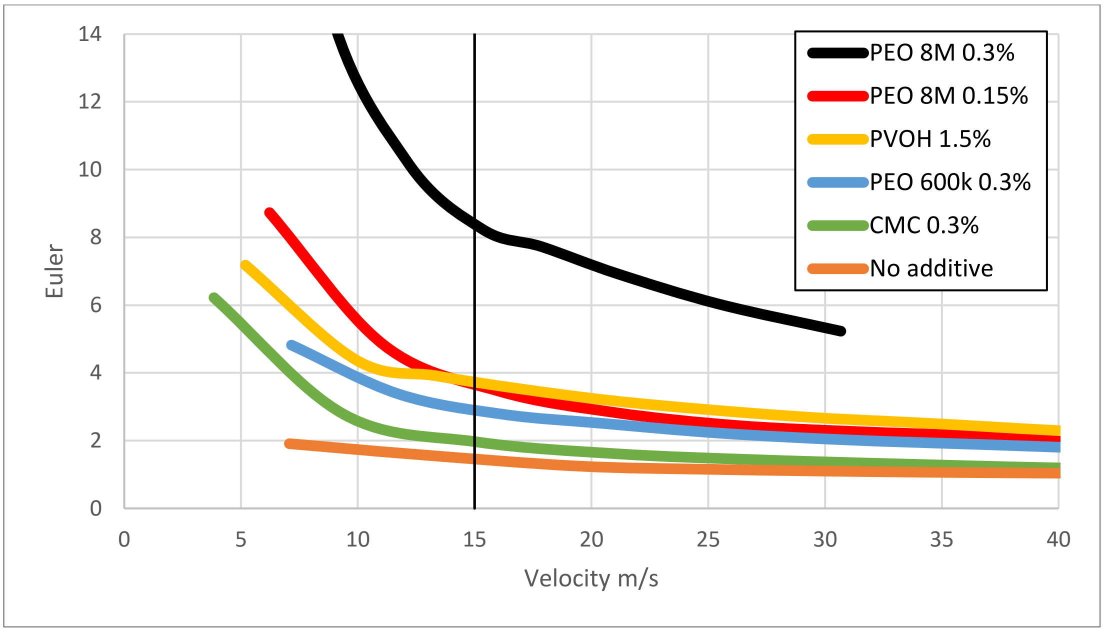
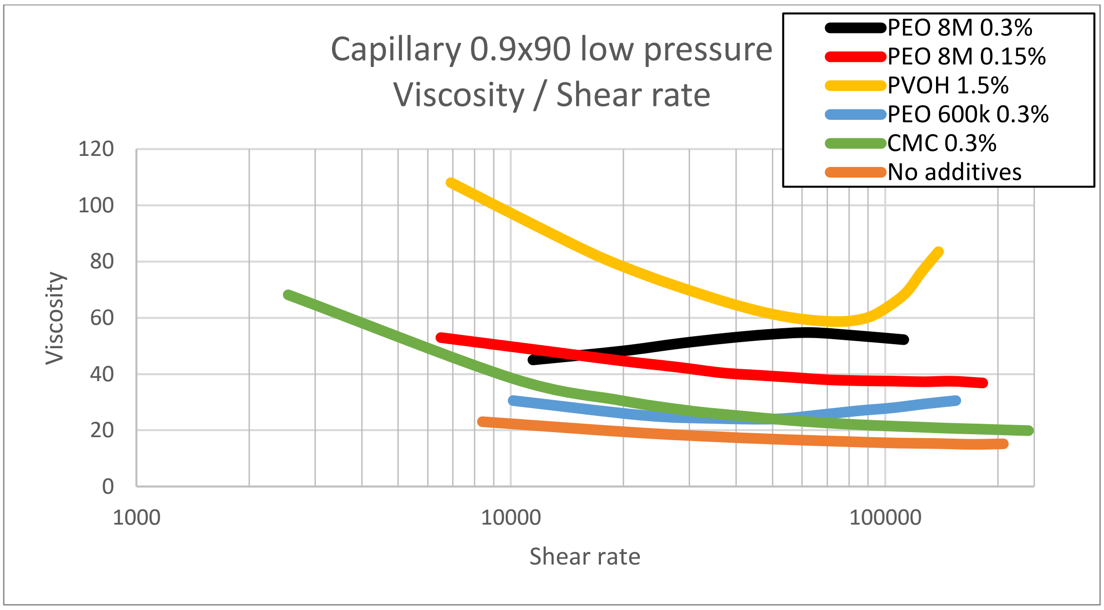
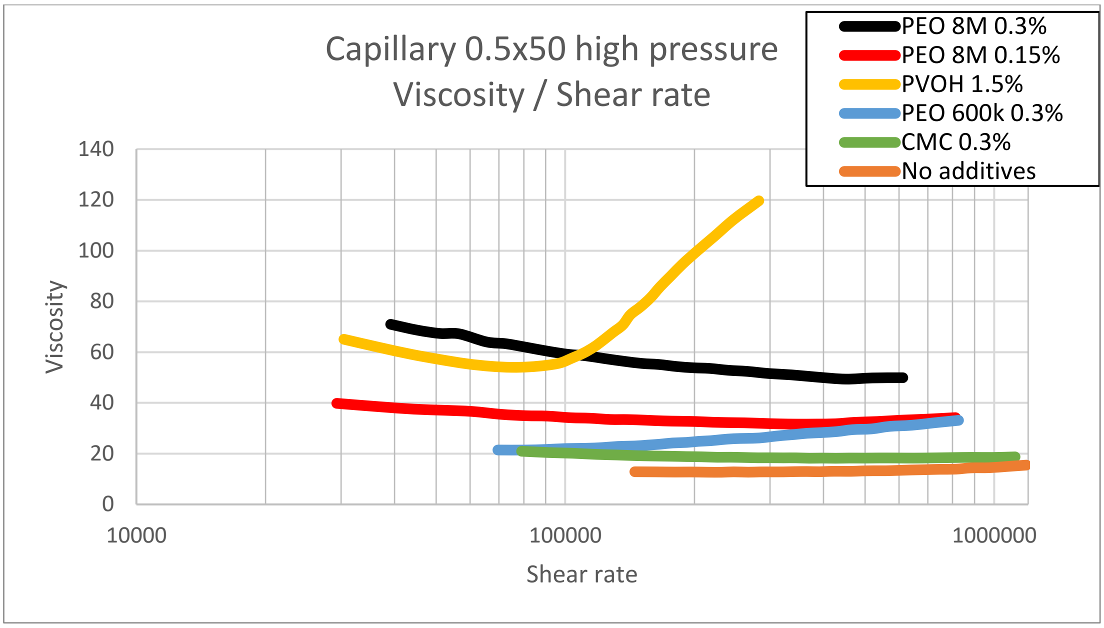
Appendix



Appendix 1. *Extensional viscosities of coating colors, zoomed in non-logarithmic.*



Appendix 2. *Viscosities of dispersions using ACA AX-100 with capillary 0.9x90mm geometry at low pressure.*



Appendix 3. *Viscosities of dispersions using ACA AX-100 with capillary 0.5x50mm geometry at high pressure.*