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Optimized Rheological Properties of Silicon-based Paints

An Investigation of Thickeners

Master's Thesis in Materials Chemistry

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Cover: SiOO:X Panel Colour 02 Oyster Grey on fine sawn spruce boards.

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Abstract

Sioo Wood Protection AB produces silicon-based paints for exterior wood. Two of the products that the company produces are SiOO:X Panel Colour Wood Protection and SiOO:X Panel Colour Surface Protection which are used together to create a coating on wood. These products increase the lifespan of the wood it is applied on and gives the wood a silver-grey color. SiOO:X Panel Colour Wood Protection and SiOO:X Panel Colour Surface Protection suffer undesired sedimentation during storage and application as a result of low viscosity. The viscosity can be modified by using different thickeners. In this project, several thickeners were evaluated in SiOO:X Panel Colour Wood Protection and SiOO:X Panel Colour Surface Protection to see which thickeners increased the storage stability without impairing the other properties of the paints. The thickeners were evaluated by producing paint samples containing the thickeners and measuring the samples storage stability, color, viscosity, gloss, non-volatile matter, sag resistance, leveling and water repellency. The samples were also evaluated by performing natural weathering tests, leneta paper applicator tests, and tests of how easily the paints could be applied by brush. Most thickeners did not increase the viscosity more than the currently used thickener. However, thickeners based on diutan gums and xanthan gums could be used to improve the rheological properties of samples of SiOO:X Panel Colour Wood Protection without impairing the functionality of the samples. Combinations of thickeners based on one type of clay and xanthan gums or diutan gums also added good storage stability, improved application by brush, and made the SiOO:X Panel Colour Wood Protection samples protect wood well. One of the evaluated hydroxyethyl cellulose thickeners may be used in SiOO:X Panel Colour Surface Protection but at a lower amount than 0.9 wt% as that gave the sample good storage stability but too high viscosity for application.

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Petter Johansson, Gothenburg, July 2022

Contents

1
Introduction

Throughout history, wood has been of great importance to mankind and is currently one of the most consumed materials [1]. Its high mechanical strength and low processing costs combined with aesthetically desirable properties makes it useful as a building material [2]. One major issue with using untreated wood in construction is its relatively quick degradation which can reduce key properties and limit its applications [2]. The degradation is a result of several factors, for instance ultraviolet radiation, moisture, insects, and biological agents such as bacteria, algae, and fungi to name a few [3]. Wood can be protected from degradation in several ways, one being coating the wood surface with paint [3].

Sioo Wood Protection AB is a company that provides alternative wood protecting products to conventional paints. Two of their products are SiOO:X Panel Colour Wood Protection which will be called *wood protection* in this report and SiOO:X Panel Colour Surface Protection which will be called *surface protection. Sioo's panel paints* or simply *panel paints* will be used to refer to both of them. They are used together to create a protective coating on vertical exterior wood surfaces. Sioo's panel paints are based on potassium silicate and a mixture of acrylics, silanes and siloxanes. The coating increases the lifespan of the wood and gives it a silver-grey color. Pigments are added into Sioo's panel paints to modify the color of the coating. Sioo's panel paints have high water content, density differences, and low viscosity compared to traditional paints. The density differences and low viscosity makes some components sediment during storage [4] so that thorough stirring is required before application [5, 6]. The low viscosity complicates the application process as the paint may drip from brushes and even sag down on vertical surfaces if applied in excess [7, 8]. These issues have limited the types of wood surfaces Sioo's panel paints can be applied to.

The optimal rheology of a paint depends on the surface the paint is supposed to be applied on, the application method , and on personal preference [9]. In the case of Sioo's panel paints, the products must have suitable properties for application by brushing and for spraying. To prevent issues such as dripping from brushes, sag on vertical surfaces and sedimentation during storage the panel paints need high viscosity when the paints are exposed to low shear rates [10]. The panel paints also need to have low viscosity when the paints are exposed to high shear rates to counteract issues such as the paint not flowing out and covering surfaces evenly and being too thick to flow through pumps and spray nozzles [11]. A type of rheology suitable for paints is pseudoplastic rheology which has high viscosity during exposure to low shear rates and low viscosity when exposed to high shear rates [12]. This type of rheology can be obtained in paint formulations by adding certain pseudoplastic thickening agents [12].

Pseudoplastic thickeners can create rheological structures in paints which increase the viscosity of the fluid if it is exposed to low shear rates [9]. If high shear rates are applied to the fluid, particles in the rheological structure are moved and the structure is destroyed. For example, polymers may move parallel to each other and flocculated components can be deflocculated leading to a temporarily reduced viscosity [12]. How much the viscosity is affected by adding a thickener depends on the thickener, the liquid in which it is added, and the concentration of the thickener. A combination of thickeners can also be used to fine tune the rheology for a certain application. In the case of Sioo's panel paints, the rheology of the paints can be improved by changing the concentration of the thickening agent, using a new thickener or adding several thickening agents to the paints. This can result in the rheology being such that the storage stability and application process are improved.

1.1 Purpose

The purpose of this project is to investigate how the use of thickening agents can improve Sioo's panel paints by making them easy to apply and have adequate storage stability without impairing the protection of the wood. The effects different thickening agents have on the rheological properties of Sioo's panel paints are evaluated by producing paint samples with different thickeners or combinations of thickeners. These samples are tested using several methods. The data from these tests are used to evaluate the properties of the thickeners or thickener combinations. An improvement of the rheological properties of Sioo's panel paints can improve their storage stability and ease of application.

1.2 Limitations

Due to time restraints on the project, limitations are applied to how long samples are tested. Exterior wood coatings are required to last for several years so the performance of the panel paint samples are not evaluated over their full lifetime. During the project, a limited number of thickeners are tested as a result of limited time, resources, and since many thickeners provide similar rheological properties. Limitations are also made to the number of colors of the paints tested in this project. Only a single color is tested to reduce the number of parameters. The paints analyzed in this project are made for outside use since Sioo Wood Protection AB only makes external coatings. The application of the coatings is done with brushes in this project. Rollers and spraying machines are used frequently in the paint industry but were not used in this project due to time and resource restraints.

2

Theory

2.1 Silicon-based panel paints

Sioo's panel paints consist of two products, SiOO:X Panel Colour Wood Protection and SiOO:X Panel Colour Surface Protection. The first fluid which is applied is the wood protection which contains potassium silicate and silica. When the alkaline potassium silicate penetrates the surface of the wood, its pH is reduced as the wood is acidic and carbon dioxide dissolves in the mixture creating carbonic acid which further reduces the pH [13]. When the pH is reduced to a certain level and the temperature is above approximately 10 °C, the silica monomers begin to polymerize. As the monomers polymerize, an amorphous silica structure is created. The polymer particles grow through Ostwald ripening and the polymers can also bind directly to cellulose, hemicellulose or lignin. As the large amorphous silica structures bind to the wood, the wood becomes harder and biofouling is reduced. During this process the wood is mineralized and since the potassium silicate is alkaline, the pH of the previously acidic wood is increased. Wood protection without pigments does not create a film on the wood surface but as pigments are added a coating may be formed.

The second fluid, SiOO:X Panel Colour Surface Protection contains acrylics, silanes and siloxanes in an oil-in-water emulsion. As the surface protection is applied to the wood surface, water evaporates and the emulsion droplets eventually break. When the emulsion droplets break, the silanes and siloxanes flow out and a silane polymerization reaction is initialized. The monomers turn into a polymeric layer with a nonpolar side consisting of hydrocarbon chains and a polar side. Since the surface protection contains water which is polar, the polymer layer is positioned in the liquid-air interface with the polar side facing the surface protection and the wood. The side with the nonpolar hydrocarbon chains face away from the wood. As water evaporates, the polymeric layer creates a hydrophobic barrier on the wood surface. The barrier regulates the uptake and release of water by diffusion. By protecting the wood from water there is less shrinkage and swelling of the wood as less water is present. The bonding between the silanes and the wood is improved as the pH of the wood is increased due to the wood protection treatment. The surface protection forms a film on the surface of the wood and keeps the wood protection from being extracted by water. The surface protection together with the wood protection also protects the wood from rot and dirt.

2.2 Definitions in rheology

Rheology describes how materials flow and deform [14, 15]. There are several modes of deformation where the most common one is shear. Simple shear can be accomplished in a liquid by creating a velocity gradient across the material which is the same thing as letting it flow. This can be achieved by having the liquid between two walls where one of the walls is moving or by having the liquid flow through a tube. This can be used to do measurements on the liquid. In these cases the volume remains constant but there are types of deformation, such as dilatation and bulk compression where a volume change is used to deform the liquid [16]. If the deformation of a sample is the same in the entire material, it is considered a homogeneous deformation. However, this is often not the case in rheological measurements. In rheological measurements, the deformation and shear rates are often different in different parts of the material. The mathematical relationships concerning the deformation of the material and the applied stress can be simplified if the deformation is relatively small. If the deformation is large, considerations concerning geometries of the measuring equipment need to be considered [16].

2.2.1 Shear stress and shear strain

Shear stress can be visualised by having two parallel plates where both plates have an area of A and the distance between the plates is h. Between them is a liquid which is to be analyzed. If a force, F is applied on one of the plates while the other plate is held in place, the shear stress (γ) applied to the material is given by F/A [17]. When the plate under influence of the force moves a distance y, the shear strain ($\dot{\gamma}$) is given by y/h. If the moving plate is moving at a constant velocity v, the shear rate is given by v/h [17]. The shear viscosity (μ) is given by Equation 2.1 [16]. If the viscosity is examined in a specific shear rate interval it is called apparent viscosity.

$$\mu = \frac{\tau}{\dot{\gamma}} = \frac{Fh}{Av} \tag{2.1}$$

2.2.2 Power law fluids

Fluids can be characterized depending on their rheological properties. The viscosities of Newtonian liquids are independent of the applied shear rate. Most liquids are Newtonian over certain shear rates but usually not over all shear rates [16]. Pseudoplastic liquids have increasing viscosity as the shear rate decreases. Pseudoplastic liquids do not depend on the time it has been exposed to shear or previous shear that has been applied, only on the current shear rate. Dilatant fluids have increasing viscosity as the shear rate is increased and are in theory independent of the shear history of the sample. However, several dilatant liquids react to shear like a solid and break if the shear rate is high or increased too rapidly, making them depend on the history of shear on the sample. These rheological profiles can be called power law fluids because they can be described by the Ostwald–de Waele relationship given by Equation 2.2 [16].

$$\tau = K \left(\frac{\partial u}{\partial y} \right)^n \tag{2.2}$$

Where K is the consistency factor, also known as the viscosity coefficient which is greater than zero. n is the flow behavior index, also called power law exponent which defines the rheology of the fluid. $\frac{\partial u}{\partial y}$ is the shear rate, also called the velocity gradient. It can be written as Equation 2.3 for apparent viscosity (μ).

$$\mu = K \left(\frac{\partial u}{\partial y} \right)^{n-1} \tag{2.3}$$

The flow behaviour index can be used to analyze which rheology profile the paint has. For example, if the paint has a value of n greater than 1, the paint is dilatant. If the paint has a value of n equal to 1 it is Newtonian and if the n value is smaller than 1 it is pseudoplastic [18]. The relationship is however only valid at a certain interval as the relationship predicts that the viscosity approaches infinity as the shear rate approaches zero and that the viscosity approaches zero as the shear rate approaches infinity for pseudoplastic fluids. Real fluids have a limit on how high and low the viscosity can be which depends on the physical chemistry of the fluid on the molecular level. Therefore, the relationship is only valid in the interval where the constants were fitted [16].

2.2.3 Thixotropic and rheopectic liquids

Thixotropic and rheopectic fluids are dependent on the shear history of the sample as opposed to the pseudoplastic and dilatant fluids [16]. A thixotropic fluid becomes thinner as it is exposed to a shear force and when the shear force is removed it takes a certain amount of time for the fluid to return to its initial state. The viscosity of rheopectic fluids increases as the material is exposed to shear. As the shear is removed, the viscosity returns to its initial state over a certain time.

2.3 Rheology of paints

The optimal rheology of a paint depends on the surface the paint is supposed to be applied on, the application method [9], and on preference. In the case of Sioo's panel paints, the products must have suitable properties for application by brushing and for spraying. During application by brushing, the viscosity of the paint is required to be high when exposed to low shear rates to counteract dripping from the brush. When a loaded brush is drawn against a board, the paint is exposed to high shear rates [12] and the viscosity needs to be low so the paint flows out evenly and easily. After application, the paint needs to regain high viscosity to prevent sagging on vertical surfaces [9]. For application by spraying, the viscosity of the paint is required to be low when exposed to high shear rates to enable the paint to flow through pumps and nozzles [9]. When the paint lands on the wood surface it needs to get high viscosity to prevent sagging [9, 19]. It is also important that the paint has high viscosity at low shear rates to prevent sedimentation [12]. A type of rheology which is suitable for these applications is pseudoplastic rheology [12]. It has high viscosity during exposure to low shear rates and low viscosity when exposed to high shear rates. Pseudoplastic rheology can be obtained in paint formulations by adding certain pseudoplastic thickening agents.

2.4 Thickeners in paints

A thickener can provide different properties to a paint and can be used to counteract several issues. Two common problems with paint is sag and settling. Resistance to sag and settling can be introduced to a paint by increasing its low shear rate viscosity. The low shear rate viscosity can be increased by incorporating small amounts of colloidal particles. These particles create a loose structure in the paint which can increase the viscosity substantially at low shear rates but also be destroyed quickly if higher shear rates are applied to the paint [20]. However, the structure is recreated as the shear rate is reduced again [12].

2.4.1 Thickening agents in water-based paints

There are many types of thickening agents which can be used in water-based paints. Two common thickeners used in the paint industry are polyurethanes and hydroxyethyl cellulose. Polyurethane thickeners are associative synthetic polymers. Associative thickeners create networks with themselves or other components of the formulation by interactions of hydrophobic end groups on the thickeners [21]. Hydroxyethyl cellulose are thickeners derived from cellulose and are also associative [21]. Different types of gums can also be used as thickeners. Xanthan gums are commonly used as thickeners in food due to their broad pH compatibility range [22]. Diutan gums are another type of thickener based on gum which are common in the concrete industry as they adsorb onto cement particle surfaces [23]. Xanthan gums and diutan gums are dispersed easily and increase the viscosity significantly even at low concentrations [23]. Microfibrillated cellulose thickeners are created from cellulose and consists of a three dimensional network of cellulose microfibrils that has a large surface area [24]. Fumed silica can also be used as a thickener and has a large surface area while being lightweight. Fumed silica is created by flame hydrolysis and consists of agglomerated silica aggregates [25]. Some types of clay can be used as thickeners. Clay thickeners have a small particle size and have a high affinity for water [26]. The product name of the thickeners used in this project are trade secrets. The thickeners will therefore be called by the type of thickener and a number, for example clay 1.

2.5 Common paint defects

Coatings and paints may suffer from defects which can appear and impair the performance of the paint. Several defects which are relevant in this project are described in this section.

2.5.1 Leveling and sag

In wood coating applications, an even coating thickness and total coverage of the surface is desired. In some cases the coating solidifies before it has become uniform on the wood surface. This is due to poor leveling properties. Leveling is the ability of a paint to flow out and remove surface defects right after application. Poor leveling can result in defects such as a structured surface, craters, crawling, brush marks, and pinholes [27]. The amount of leveling is dependant on the surface tension, drying conditions, application, and rheological properties. If the surface tension is high, proper leveling can be hindered. The surfactants influence the tension at the liquid-air, liquid-solid, and liquid-liquid interface. The surface tension of the paint and the surface tension of the substrate decides the wetting and leveling properties of the coating. If the paint has lower surface tension than the substrate, the paint will wet the surface of the substrate [28]. If the paint has higher surface tension than the substrate, the liquid will minimize the contact with the surface of the substrate and poor wetting will be achieved [28]. Leveling is also related to the ingredients such as solvents, binders, pigments, and extenders. Changing the ingredients may change the leveling properties of the paint. For example, a solvent with too fast evaporation will lead to poor leveling. The film thickness also impacts the leveling properties. A high viscosity of the paint can reduce the leveling properties. Leveling can be improved by changing the rheology. A lower viscosity will increase leveling but can cause problems with sagging. If the rheology of a paint is altered to make the viscosity increase slower after application, the time leveling can occur is extended but it may reduce the sag resistance of the paint [9].

2.5.2 Paint peeling

Paint peeling is when a paint film loosens from the substrate and cracks. Eventually the film will roll or fall off. It happens due to loss of adhesion between the substrate and the paint. One common reason of the loss of adhesion is that water separates the paint from the substrate.

2.5.3 Air entrapment

Air entrapment is a defect that can be present in coatings with high surface tensions and high viscosities. The defect is a result of small air bubbles in the coating. The air bubbles can be introduced during spraying, manufacturing, or during the preparation of the coating. As the bubbles eventually leave the coating a pinhole is created [27].

2.5.4 Craters and fisheyes

Craters are typically small circular defects found in coatings. These defects occur as a result of contaminant particles which reduce the surface tension of the coating locally [20]. The differences in surface tension create a flow of paint away from the particle with low surface tension, and a crater is formed [27]. A small circular defect can also be a result of a contaminant liquid which prevents wetting of an area. That will keep the paint from flowing over that area and circular defect called fisheye is created.

2.5.5 Mud cracks in paint

Mud cracks are cracks of different sizes in a coating which appear during drying. During drying, solvent leaves the paint and the paint coating shrinks. The coating optimally only shrinks in the normal direction to the surface of the coating. If the coating shrinks parallel to the surface of the coating, mud cracks can appear [27]. Mud cracks usually appear in paint coatings if a coating is too thick or if the drying process is inadequate. If mud cracks appear, the thickness of the coating should be reduced or the drying procedure should be evaluated [27].

3

Methods

The project was initiated with a literature study and consultation with the supervisor and coordinator of the project to decide which thickeners and methods to use. Samples of Sioo's panel paints with different thickeners were produced and analyzed with several methods which are described below. As the project continued, combinations of thickeners were evaluated using the same methods.

3.1 Literature study

Information about thickeners and paints was gathered in a literature study. Technical data sheets, safety data sheets, and product data sheets provided by companies were used as sources of information on specific thickening agents. General theory about thickeners, rheology and paints was obtained from books and research articles from established databases and journals.

3.2 Laboratory work

The initial laboratory work consisted of preparation of panel paints and proxy systems. A proxy system is a substitute system with similar properties to the original system. The proxy systems used in this project are simplified systems consisting of water, potassium silicate solutions or acrylic-silane-siloxane blends with different thickeners, in some cases combinations of two thickeners. The panel paints and proxy systems were tested using several methods to evaluate which rheological properties the thickeners add to panel paints.

3.2.1 Preparation of panel paint and proxy system samples

Samples of panel paint were made using thickeners of interest for the project. The samples were made from several ingredients put together in a series of steps. Since the exact procedures of making Sioo's panel paints are trade secrets, the procedures can not be fully described in this text. However, the main ingredients in the SiOO:X Panel Colour Wood Protection include pigments, water, potassium silicate, dispersants, and thickeners. The main ingredients of SiOO:X Panel Colour Surface Protection are pigments, water, thickeners, acrylics, silane, siloxane, and dispersants. Some thickeners were evaluated in simple proxy systems of water, potassium silicate solutions or acrylic-silicate-siloxane solutions before being introduced into panel paints to get an estimate of the required thickener concentration. The panel paints were mixed using a laboratory dissolver from Westerlins Maskinfabrik AB fitted with an eLA80b-2 engine from Loher GmbH producing 1.1 kW and a rotational speed between 1140 and 6840 rpm. The lab dissolver was used with a Lenart disc for low shear rate mixing and a Cowles mixing impeller for high shear rate mixing. The diameter of the mixing container was 3 times larger than the diameter of the impeller. The distance from the bottom of the container to the impeller and from the impeller to the liquid surface varied as ingredients were added to the containers. The mixing speed and mixing time depended on which ingredients were mixed as some ingredients required high shear rates to be dispersed in a mixture and some ingredients can't handle high shear rates. An immersion blender with variable rotational speed was used for mixing small volumes.

3.2.2 Storage stability

After a panel paint sample was produced, 4 dl of the finished paint sample was transfered into a transparent plastic container. 1 ml of the paint sample was also mixed with 4 dl water in another plastic container. The high water content in the second container acted to accelerate sedimentation due to system instability. Pictures were taken before and after a storage period of 1 month. These tests were performed to evaluate storage stability of the paint samples.

3.2.3 Color measurement

A Hunterlab UltraScan VIS color measurement spectrophotometer was used to test the color of the samples. After calibration of the spectrophotometer, a paint sample was put in a 1*5 cm cuvette. The cuvette was put in its designated position and a metal cover was put over it to create a dark background. The analysis was then executed and values of L, a, and, b were obtained based on Hunter Lab color scale. Duplicates were taken for each paint sample. L, a, and b shows where the color of the sample is on 3 different color scales. The L values indicate how light or dark the sample is where lower numbers (0-50) indicate a dark color and higher numbers (51-100) indicate a lighter sample [29]. The a values indicate how green or red a sample is where a values smaller than 0 indicate a green color and values greater than 0 indicate a red sample. The b value indicate how yellow or blue a sample is. b is negative for blue colors and positive for yellow colors. This test was used to see how the thickeners affect the color of the panel paints.

3.2.4 Spindle viscometer tests

The viscosities of the paint samples ware tested with a NDJ-5S spindle viscometer. The test was carried out by putting 600 ml of a paint sample in a 600 ml plastic beaker. A spindle connected to the viscometer was submerged to the right depth in the sample, indicated by an indentation on the spindle. When the analysis was initiated, the spindle started spinning with a predetermined frequency of rotation. The viscometer recorded the resistance the sample liquid exerted on the rotating spindle. From the resistance and shape of the spindle the viscometer calculated the viscosity. Several frequencies of rotation and spindles were used to get more data values. From the rotational speed and geometry of the spindles, the shear rates could be calculated and plotted against the viscosity. These data values were used to create a regression to the Ostwald–de Waele relationship which is shown in Equation 2.3. A graph was created, displaying the Ostwald–de Waele relationship for the paint sample and it was used to compare the rheological properties of the thickeners.

3.2.5 Natural weathering test

The test setup of the natural weathering test was made to replicate the standard SS-EN 927-3:2012, natural weathering test. Surface protection and wood protection samples were tested separately. When preparing wood boards for the natural weathering test of wood protection samples, four 50 cm boards were prepared. One of them was coated by applying two 90 g/m^2 layers of a wood protection sample and one 90 g/m^2 layer of unpigmented surface protection by brush on smooth planed wood boards of pine (Pinus sylvestris). The second sample was made by applying two 90 g/m^2 layers of wood protection sample and one 90 g/m^2 layer of SiOO:X Panel Surface Protection 06 Lava Grey, which will be called pigmented surface protection in this report, on smooth planed wood boards of pine. The smooth planed boards were used to accelerate the aging process of the coatings. The other two wood samples were made with the same paint combinations but on 50 cm fine sawn spruce (Picea abies). Since the surface area is larger on fine sawn wood compared to smooth planed wood, more paint per area was used. 143.5 g/m^2 was used for each layer compared to the 90 g/m^2 previously mentioned. Similarly to the samples for wood protection, when preparing the wood samples coated with surface protection samples, four 50 cm samples were made. One of them was prepared by applying two 90 g/m^2 layers of a SiOO:X Panel Wood Protection 06 Lava Grey, which will be called pigmented wood protection in this report, and one 90 g/m^2 layer of surface protection sample on smooth planed wood boards of pine. The second sample was made by applying two 90 g/m^2 layers of unpigmented wood protection and one 90 g/m^2 layer of surface protection sample on smooth planed wood boards of pine. The other two wood samples were again made with the same paint combinations but on 50 cm fine sawn spruce. 143.5 g/m^2 was used for each layer on the spruce boards. After drying, the planed samples were placed outside at a 90° angle to the ground, facing south, and the fine sawn samples at a 45° angle to the ground, facing south. The 45° angle speeds up the aging by a factor of 1.5 due to a higher impact of rain, dew, and ultra violet radiation from the sun as the angle increases the exposure to sunlight. The natural weathering test was carried out for one month to see how the samples ages outside.

3.2.6 Measurement of gloss

The specular reflection gloss of panel paint coatings on planed boards of pine was measured by a Sheen microgloss 60 glossmeter measuring at a 60° angle. When measuring the gloss of wood protection coatings, two wood samples with a length of 10 cm were made. One of them was prepared by applying two 90 g/m^2 layers of wood protection sample and one 90 g/m^2 layer of unpigmented surface protection by brush on smooth planed wood boards of pine. The other sample was made by applying two 90 g/m^2 layers of wood protection sample and one 90 g/m^2 layer of pigmented surface protection with a brush on smooth planed wood boards of pine. When measuring the gloss of surface protection samples, two wood samples with a length of 10 cm were also made. One of them was prepared by applying two 90 g/m^2 layers of unpigmented wood protection and one 90 g/m^2 layer of surface protection sample by brush on smooth planed wood boards of pine. The other sample was made by applying two 90 g/m^2 layers of pigmented wood protection and one 90 g/m^2 layer of surface protection sample. The measurements were made by placing the glossmeter on the area of the sample where the measurement was supposed to be made. The glossmeter was then turned on and after a short delay the measurement could be executed and a value of the gloss was obtained. For each wood sample, 10 measurements were made on areas with dense growth rings and 10 measurements were taken in areas with larger distances between growth rings. A mean value of the data was used to compare the difference in gloss between samples containing different thickeners.

3.2.7 Ease of application by brush

Samples of panel paints were applied on fine sawn spruce boards with a brush to evaluate how easily the paint is applied and if paint drips from the brush or sags on vertical surfaces.

3.2.8 Leneta paper applicator test

Leneta papers are tests charts produced from non-fluorescent paper which can be used to test wettability, adhesion, and surface smoothness of coatings [30]. The test was carried out by putting a coated, non-absorbing leneta paper on a horizontal surface. A 120 µm film of the sample of wood protection or surface protection was applied with a film applicator. The coating was allowed to dry. The paint's ability to bind to the surface was evaluated by how well the coating binded to the leneta paper and if defects in the coating appeared.

3.2.9 Non-volatile matter test

3 ml paint sample was put in a small, weighed, heat resistant container. The weight of the sample in the container was measured before being put in an oven for 24 hours which kept a temperature between 100 and 110 °C. The surface protection samples were weighed 3 times with a minimum of 3 hours between each measurement. The wood protection samples were only weighed once after the full drying period since the

dried wood protection started to crack when the samples were taken out of the oven. When the film cracked there was a risk that particles would fall out of the container. To prevent this, a plastic cover was put over the samples during the weighing. During the drying time in the oven, it was assumed that most volatile substances in the sample had evaporated. What was left was non-volatile components which was weighed to decide the percentage of non-volatile matter. Duplicates of each sample was made and analyzed.

3.2.10 Sag resistance test

Paint samples were stirred thoroughly before being put on a leneta paper with a syringe. A sag test blade containing notches with heights between 50 and 100 µm, shown in Figure 3.1, was pulled through the paint.

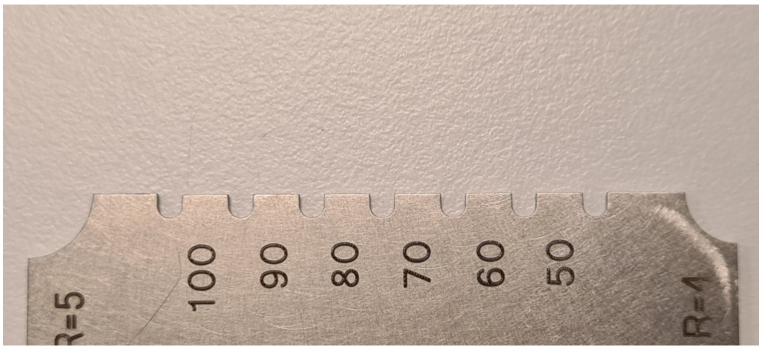

Figure 3.1: Picture of the edge of the sag test blade with small notches.

More paint sample was put on an unused part of the leneta paper and another test blade containing notches with heights between 110 and 160 µm, shown in Figure 3.2, was pulled through the new paint sample. This created parallel stripes with different thicknesses. The leneta paper was placed vertically with the thinnest stripes at the top. As gravity pushed the paint downwards the paint started to sag down the vertical surface of the leneta paper. The thickness of the thickest stripe which did not sag down on the stripe below was the anti sag index of the paint.

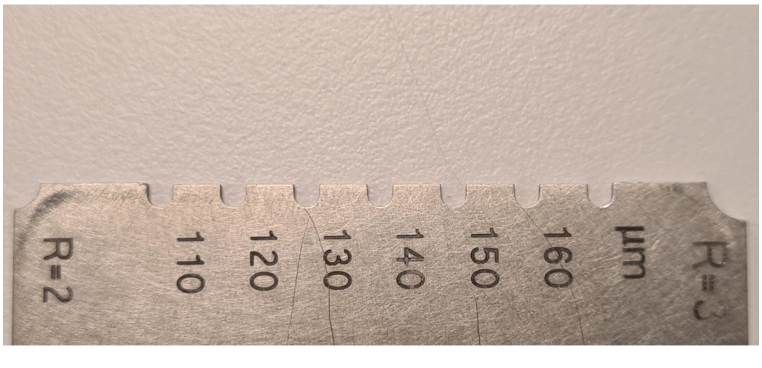

Figure 3.2: Picture of the edge of the sag test blade with large notches.

3.2.11 Leveling test

Paint samples were stirred thoroughly before being put on a leneta paper with a syringe. A leveling test blade shown in Figure 3.3 was drawn through the paint, creating 6 parallel tracks with 3 different sizes, labeled A, B, and C in Figure 3.3. The paint was allowed to dry. After drying, the leveling was evaluated depending on how the lines had connected due to leveling of the paint.

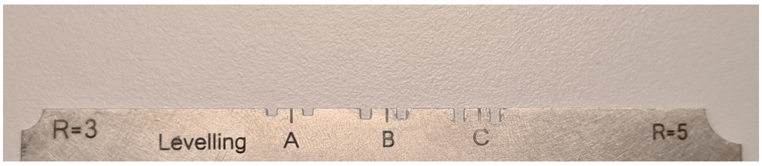

Figure 3.3: Picture of the edge of the leveling test blade used in the project.

3.2.12 Water repellency test

Sample boards from the natural weathering test were placed horizontally and water droplets were placed along the entire wood sample with a pipette. The droplets were examined visually for 5 minutes to see if the droplets were absorbed by the wood sample or if the water droplets were repelled. Since the surface protection is water repelling, the behaviour of the droplets give information about if the surface protection had fallen off or not. If the droplet was repelled, the surface protection was still present on the sample. If the water droplet was absorbed by the sample, the surface protection was no longer present on the sample.

4

Results and discussion

4.1 Storage stability tests of wood protection

The storage stability tests of wood protection showed that most thickeners did not add adequate storage stability. Thickeners based on fumed silica thickeners and polyurethanes had little or no effect on the viscosity of the wood protection. As a result, the storage stability of the samples was poor. These thickeners also had an effect on how the pigments were distributed in the paint. In wood protection samples containing fumed silica, white pigment fell to the bottom of the container while black pigment was present in the bulk of the sample which made the sample look black. A storage stability sample displaying these properties is shown in Figure 4.1.

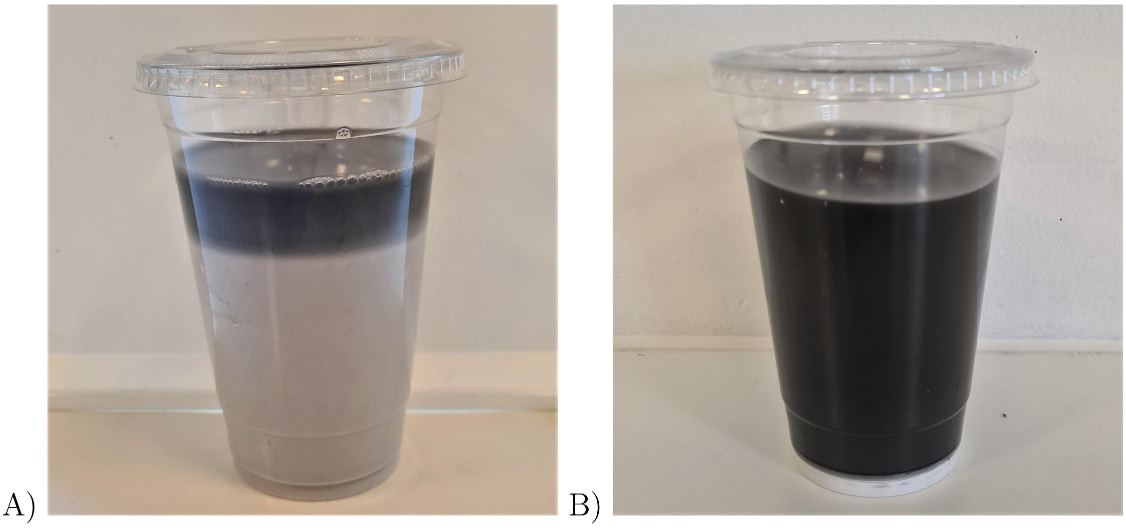

Figure 4.1: Storage stability sample of wood protection sample containing fumed silica 1. A) is after production and B) is after 1 month of storage.

The low viscosity of wood protection samples containing polyurethanes was probably because they are associative thickeners. Associative thickeners in paints create structures with other substances in the paint, especially binders. The wood protection has no binders and a high water content. As a result, the concentration of substances that the polyurethanes can create structures with is lower which decreases the thickening effect of the polyurethanes. After production of wood protection samples containing polyurethane thickeners, a layer of a black liquid with high viscosity was formed on the surface of the samples which can be seen in Figure 4.2. The layer may consist of polyurethanes which self associate at the air liquid interface together with black pigment. There was also sedimentation of white pigment on the bottom and small grains of white pigment in the bulk of the samples. Even after stirring the viscous black liquid remained in lumps.

Figure 4.2: Storage stability sample of wood protection containing polyurethane 1 after 1 month of storage.

All clay thickeners added some storage stability but some sedimentation was present in all samples. Clay 1 added the most storage stability and its storage stability sample is shown in Figure 4.3. Clay 1 was also most easily dispersed in the paint as clay 2 and 3 required high shear rates to be dispersed properly.

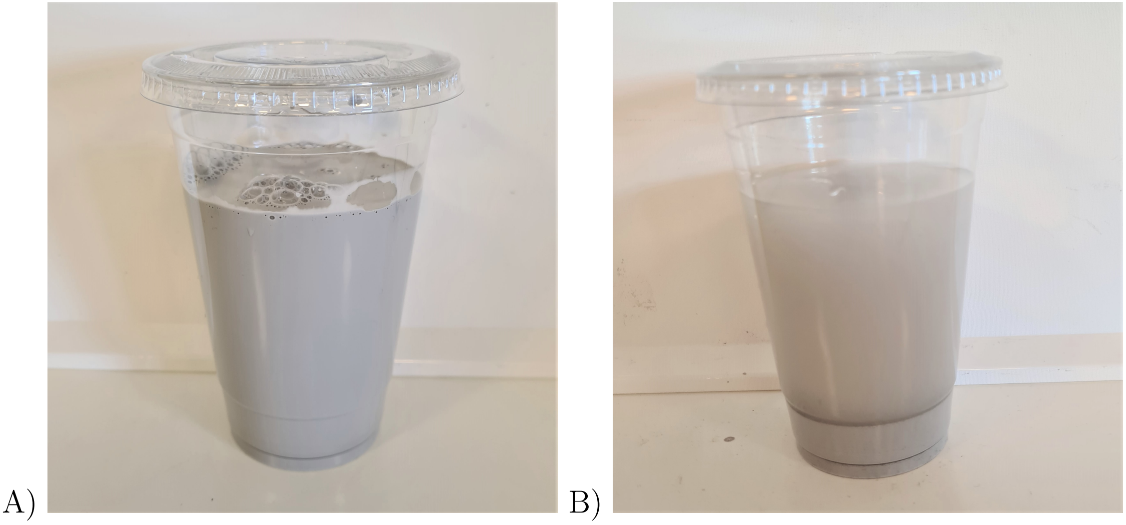

Figure 4.3: Storage stability test for a wood protection sample containing clay 1. A) is after production and B) is after 1 month of storage.

Storage stability tests conducted with wood protection samples containing xanthan gums and diutan gums showed that gums added exceptional storage stability to wood protection samples. All tested thickeners based on gums increased the viscosity of the wood protection samples so that no signs of sedimentation or separation could be seen after one month of storage. A storage stability test with 0.25 % diutan gum 1 is shown in Figure 4.4. The wood protection samples containing thickeners based on gums also had perfectly even colors after one month which indicate that very little stirring is needed before application.

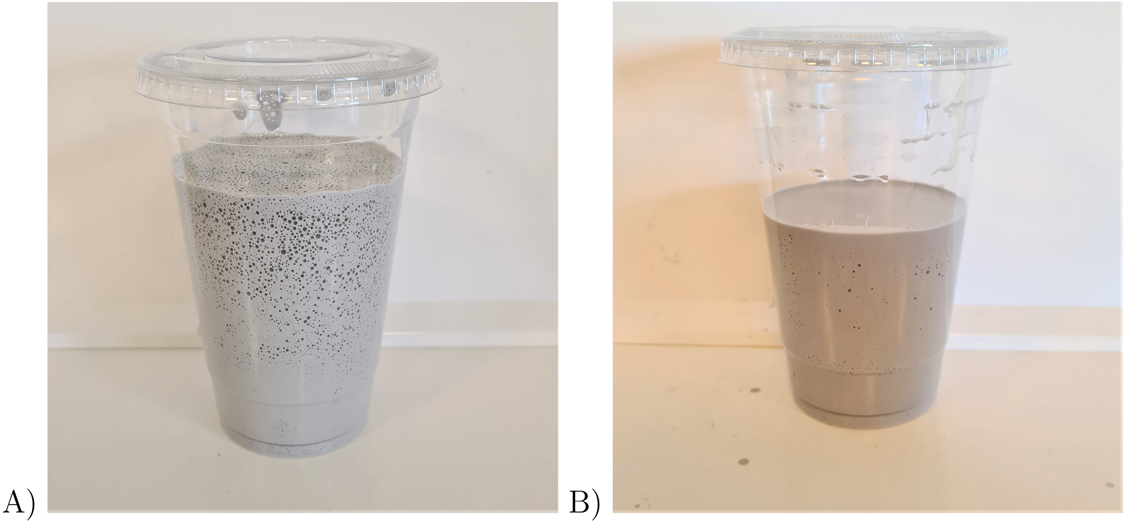

Figure 4.4: Storage stability test for a paint sample containing a thickener based on diutan gum. A) is after production and B) is after 1 month of storage.

Out of the hydroxyethyl celluloses in wood protection, hydroxyethyl cellulose 1 and 2 were tested the most as they had a better thickening effect than hydroxyethyl cellulose 3 and 4 in wood protection. The hydroxyethyl cellulose thickeners gave good thickening effect but they gave poor storage stability. Only microfibrillated cellulose 1 gave some storage stability in wood protection of the thickeners based on microfibrillated cellulose. However, after a short storage period, a transparent phase appeared at the top of the wood protection samples containing hydroxyethyl cellulose 1 which could be due to syneresis.

Microfibrillated cellulose thickeners are types of thickeners which often are combined with other thickeners. Because of this, combinations with microfibrillated cellulose and other thickeners were made. One combination was microfibrillated cellulose 1 with diutan gum 1. Two wood protection samples were made, one with 0.35 % microfibrillated cellulose and 0.05 % diutan gum 1, and one with 0.75 % microfibrillated cellulose 1 and 0.1 % diutan gum 1. The first sample had some storage stability issues as a transparent phase was created at the top of the paint similar to the wood protection samples with only microfibrillated cellulose 1. The second sample had good storage stability but it was very viscous and the wood protection was not completely homogeneous during storage. A sample was also made with 0.75 % microfibrillated cellulose 1 and 0.4 % hydroxyethyl cellulose 2 because it was a combination that the producer of microfibrillated cellulose 1 recommended. The sample showed excellent storage stability but it was very viscous. Another combination that was suggested by the producer of microfibrillated cellulose 1 was the combination of microfibrillated cellulose 1 and polyurethanes. A sample was therefore created with 0.75 % microfibrillated cellulose 1 and 0.5 % polyurethane 2. The combination did not help with the storage stability problems of microfibrillated cellulose 1 in wood protection. The problems of polyurethanes were also present as a dark layer of a viscous liquid was formed at the top of the sample.

Clay 1 was tested in combination with diutan gum 1, xanthan gum 1 and polyurethane 3. The combinations with gums gave excellent storage stability, similar to that of the wood protection samples containing only thickeners based on gums. The combination polyurethane 3 and clay 1 gave similar performance to just using clay 1 in stability tests and were therefore not evaluated further.

4.2 Natural weathering tests of wood protection

The natural weathering tests were a crucial part of testing the performance of the coatings. The tests varied greatly depending on which substrates and colors were used. Since the performance of some wood protection and surface protection combinations were poor, the performance of coatings are compared to a reference sample containing clay 1. Samples of wood protection and surface protection containing clay 1 was also used as a reference in other tests as the company has some laboratory experience working with that thickener in a test environment and because it does not interfere with the functionality of the wood protection or surface protection.

4.2.1 Wood protection in combination with pigmented surface protection on planed boards

When pigmented surface protection started to dry on planed wood boards coated with wood protection samples, yellow cracks appeared in most coatings. These cracks are believed to be mud cracks as similar cracks have been identified at the company when too much wood protection was applied to a substrate. Less wood protection should probably be used in future tests. The yellow color could be yellow pigment separating from the rest of the paint as yellow pigment is present in the paint to counteract the slightly blue tint of the black pigment. Coatings created with wood protection samples containing hydroxyethyl cellulose 1 and 2 as well as a combination of clay 1 and polyurethane 3 did not have mud cracks. A comparison between a coating with mud cracks and without mud cracks is shown in Figure 4.5.

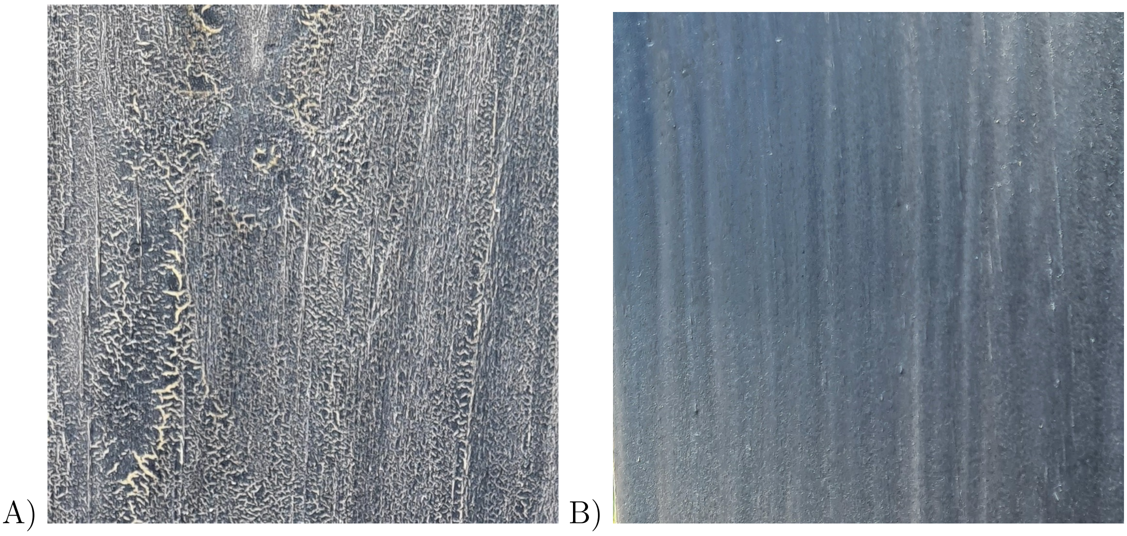

Figure 4.5: A) Planed wood board coated with wood protection containing clay 3 and pigmented surface protection with mud cracks. B) Planed wood board coated with wood protection containing hydroxyethyl cellulose 1 and pigmented surface protection without mud cracks.

The coating created using the reference sample containing clay 1 is shown in Figure 4.6. After the natural weathering test, a lot of surface protection and wood protection had fallen off as a result of paint peeling. Sioo's panel paints usually protect wood for years but in the weathering tests in this project the coating fails before one month of weather exposure for planed wood samples, especially when the surface protection and the wood protection have different colors. One reason for this can be differences in the recipes of surface protection with different colors. In the lighter surface protection products, more binder is added while more matting agents are added to darker colors. An increased amount of matting agent requires more binder but the amount of binder is decreased in the darker colors. More yellow pigment is also added in the darker colors to counteract the blue tint of the black pigment. The problem can also be a result of the dark surface protection shrinking quicker as it dries quicker than the lighter wood protection because the dark color absorbs light to a larger extent and becomes hotter.

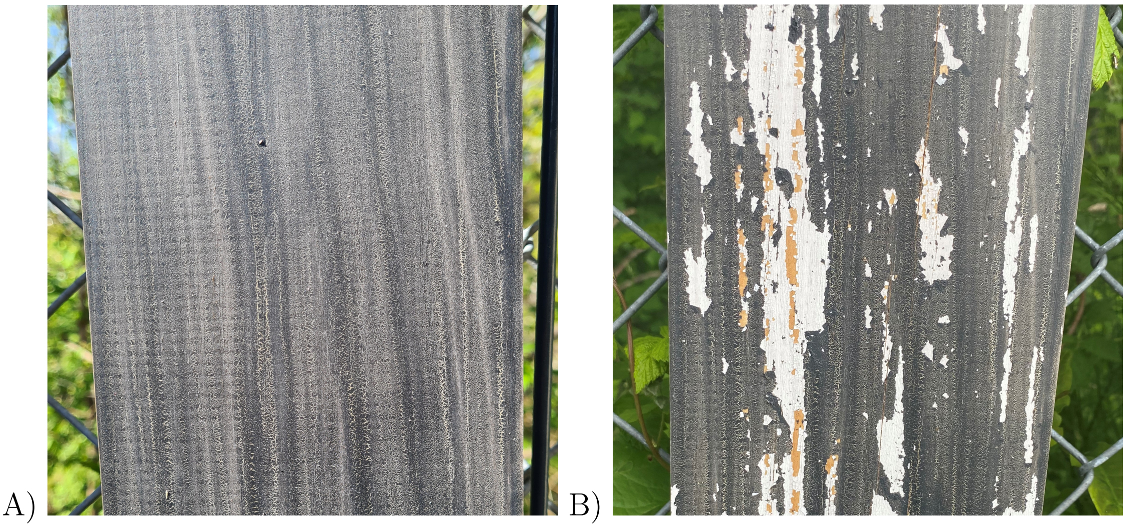

Figure 4.6: Planed wood board coated with a sample of wood protection containing clay 1 and pigmented surface protection used in the natural weathering test. A) is before the test and B) is after 1 month of weather exposure.

Since all of the evaluated thickeners based on gums gave the wood protection excellent storage stability, they were compared in a natural weathering test early on in the project. The thickeners based on gums that performed the best in the initial weathering test were diutan gum 1, xanthan gum 1 and xanthan gum 2. Further analysis of gums were carried out on these three thickeners. The coatings created with wood protection containing thickeners based on gums and pigmented surface protection on planed boards had similar or better performance than the reference as only some surface protection had fallen off which is shown in Figure 4.7. The weathering test with the combination of clay 1 and diutan gum 1 or xanthan gum 1 had good performance compared to the reference as the only defect was some peeling of surface protection but all of the wood protection was still present.

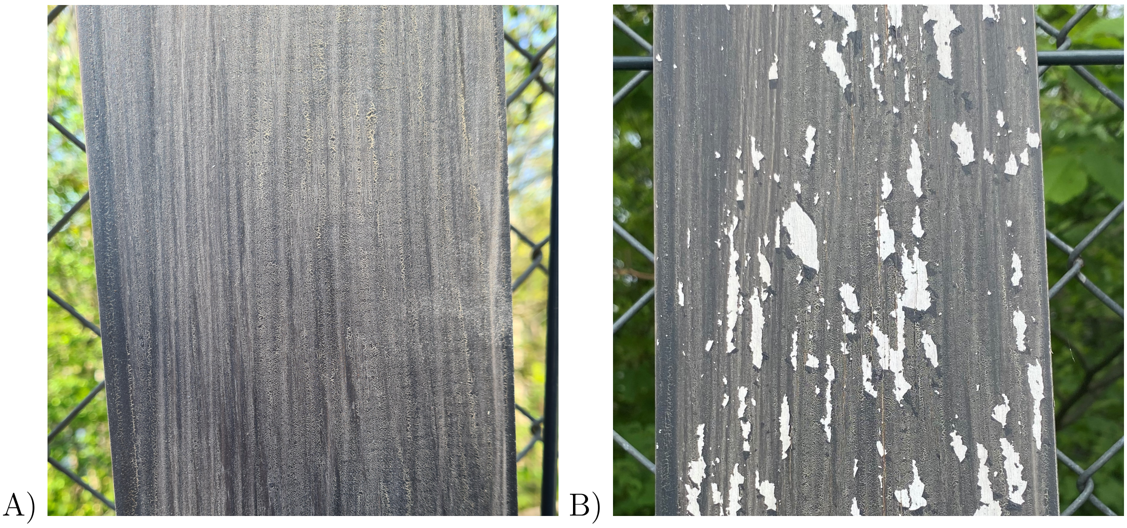

Figure 4.7: Planed wood board coated with a sample of wood protection containing diutan gum 1 and pigmented surface protection used in the natural weathering test. A) is before the test and B) is after 1 month of weather exposure.

The wood coated with wood protection containing hydroxyethyl cellulose 1 and pigmented surface protection had some loss of surface protection but very little loss of wood protection. The test with hydroxyethyl cellulose 2 had loss of surface protection but not wood protection. Coatings made with wood protection containing clay 3 and pigmented surface protection had loss of both surface and wood protection. Tests with microfibrillated cellulose 1 had some loss of both surface and wood protection but less than the reference. The planed wood coated with the wood protection sample containing microfibrillated cellulose 1 and pigmented surface protection had less loss of both surface protection and wood protection than the reference sample. The weathering test with the combination of clay 1 and diutan gum 1 performed well compared to the reference. The only defect was some peeling of surface protection but all of the wood protection was still present. The test with clay 1 and xanthan gum 1 only had very little loss of surface protection.

Microfibrillated cellulose 1 combined with hydroxyethyl cellulose 2 had better performance than the reference as less surface protection and wood protection had fallen off. The combination of clay 1 and polyurethane 3 had the best performance as barely any surface protection had fallen off and all wood protection was present.

The planed wood coated with the wood protection sample containing 0.75 % microfibrillated cellulose 1 and 0.4 % hydroxyethyl cellulose 2 and pigmented wood protection had lost some of its surface protection after the natural weathering test but all of the wood protection was still protecting the wood which was not the case for the reference. The natural weathering tests showed that the samples containing clay 1 and diutan gum 1 or xanthan gum 1 had similar performance to the reference.

4.2.2 Wood protection in combination with unpigmented surface protection on planed boards

When the wood protection samples containing clays were applied on planed wood, most of the wood protection, or at least the pigment, absorbed at the light growth rings, also called earlywood. This was visible in the wood samples with wood protection and unpigmented surface protection which is shown in Figure 4.8. This is possibly due to differences in the structure of the growth rings and the low amount of pigment due to the use of unpigmented surface protection. The wood board also had areas where paint had been in a puddle which made a defect on the wood.

The wood protection samples based on the three clays had similar performance in the natural weathering test with unpigmneted surface protection on planed boards. Some paint peeling made some of the wood protection fall off the coated boards which can be seen in Figure 4.9.

Figure 4.8: Planed wood board coated with wood protection containing clay 3 and unpigmented surface protection with an uneven distribution of pigment or wood protection.

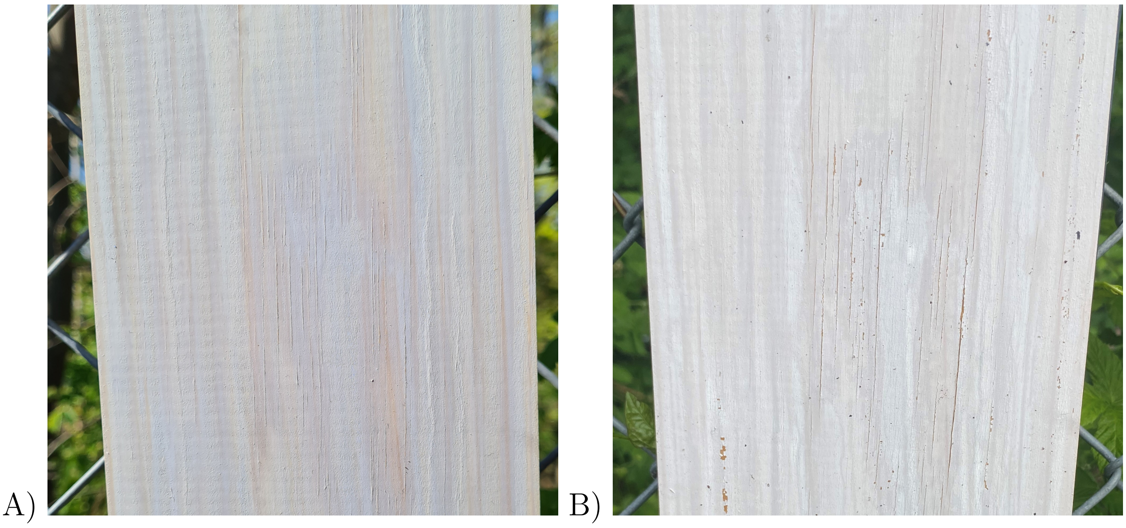

Figure 4.9: Planed wood board coated with a sample of wood protection containing clay 1 and unpigmented surface protection used in the natural weathering test. A) is before the test and B) is after 1 month of weather exposure.

The planed wood boards coated with wood protection samples containing gums and unpigmented surface protection performed almost identically to the reference sample. The coating was mostly intact for both samples but some very small areas had exposed wood which can be seen in Figure 4.10 and Figure 4.9.

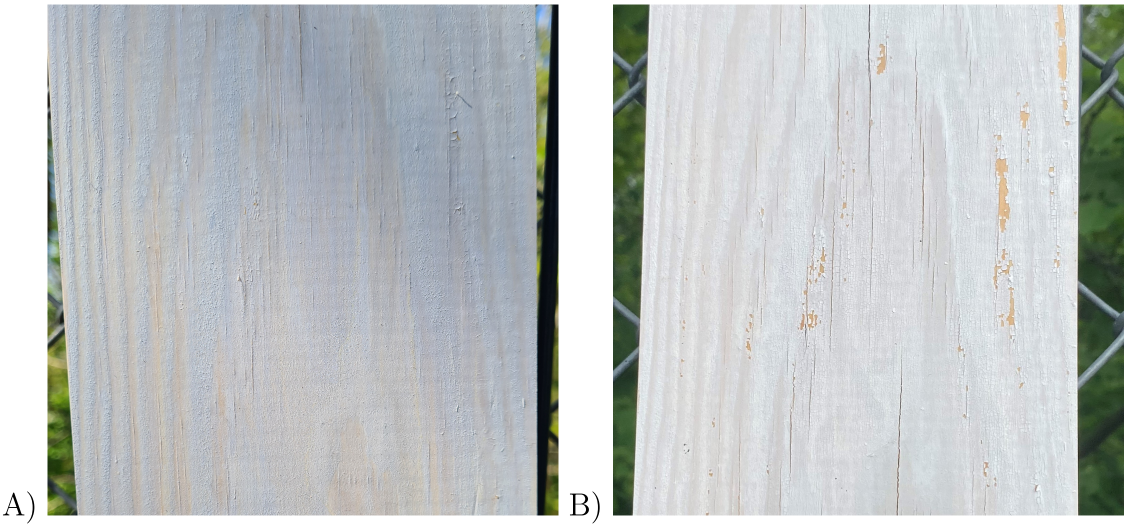

Figure 4.10: Planed wood board coated with a sample of wood protection containing diutan gum 1 and unpigmented surface protection used in the natural weathering test. A) is before the test and B) is after 1 month of weather exposure.

For wood protection containing hydroxyethyl cellulose, either paint or just pigment gathered in the lighter annual rings during application on planed wood boards. Some signs of paint puddles were present on planed wood coated with wood protection samples with hydroxyethyl cellulose thickeners and unpigmented surface protection.

The planed wood coated with wood protection containing microfibrillated cellulose 1 and unpigmented surface protection had signs of paint puddles. The planed wood sample with unpigmented surface protection showed almost identical results as the reference as very small amounts of wood protection had fallen off.

The wood protection containing 0.75 % microfibrillated cellulose 1 and 0.4 % hydroxyethyl cellulose 2 became unevenly distributed on planed wood. Most of the wood protection was at the light growth rings. The planed wood coated with the wood protection sample and unpigmented surface protection had some loss of wood protection similar to that of the reference after the natural weathering test. The natural weathering tests showed that the samples containing clay 1 and diutan gum 1 or xanthan gum 1 had similar performance to the reference.

4.2.3 Wood protection in combination with pigmented surface protection on fine sawn boards

The fine sawn wood coated with a wood protection sample containing clay 1 and pigmented surface protection had lost almost half of the surface protection after the natural weathering test which can be seen in Figure 4.11. However, the wood protection was still present except on some knots at which some wood protection had fallen off, probably due to the harder wood at knots.

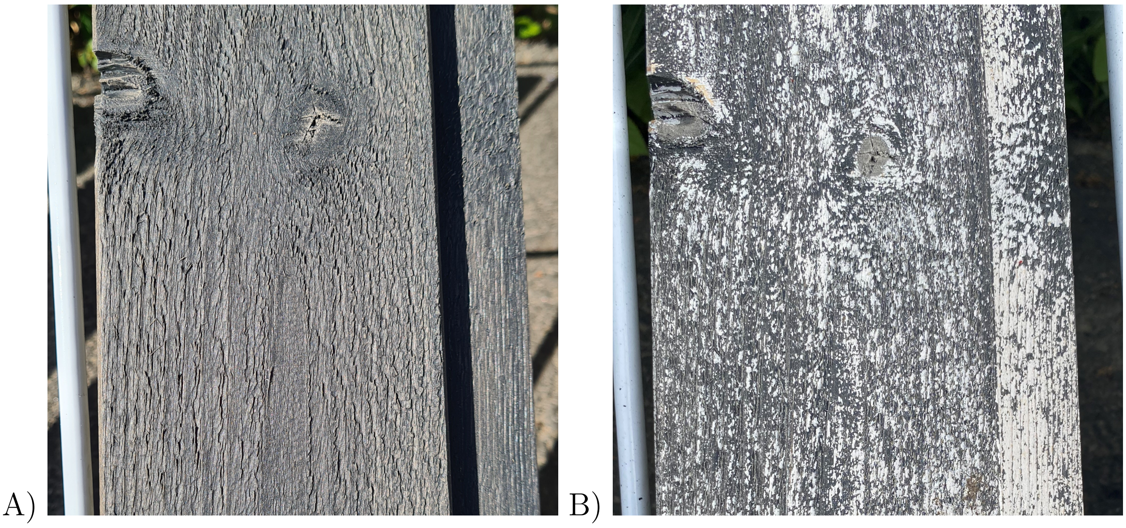

Figure 4.11: Fine sawn wood board coated with a with a sample of the reference: wood protection containing clay 1 and pigmented surface protection used in the natural weathering test. A) is before the test and B) is after 1 month of weather exposure.

When comparing wood protection samples containing thickeners based on gums and wood protection containing the reference thickener on fine sawn wood boards, the performances of the coatings were similar. Samples with the pigmented surface protection had problems with surface protection leaving the wood board and exposing the wood protection which can be seen when comparing Figure 4.12 and Figure 4.11. The wood protection was still present everywhere except on small parts of knots in the wood.

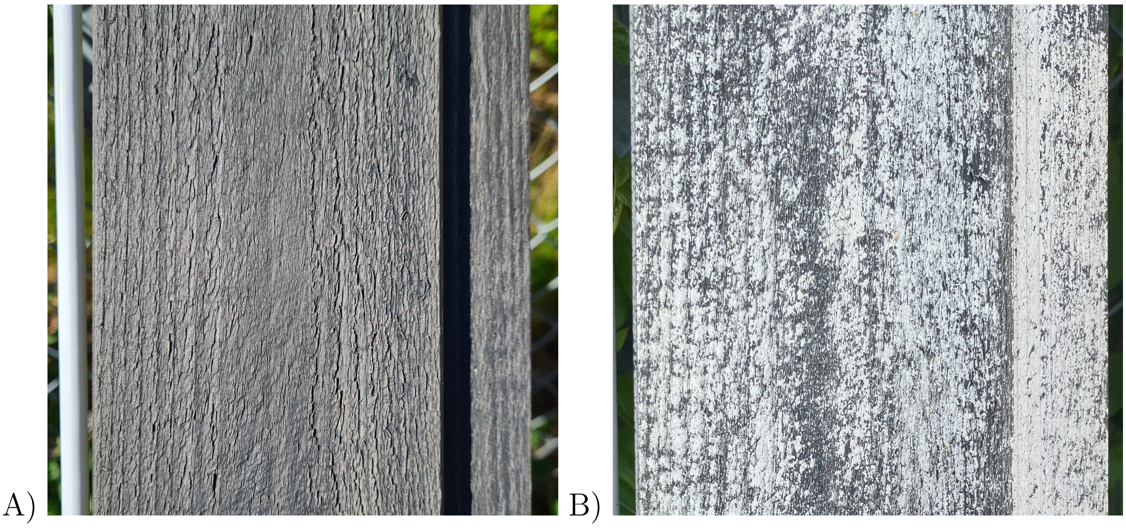

Figure 4.12: Fine sawn wood board coated with a sample of wood protection containing diutan gum 1 and pigmented surface protection used in the natural weathering test. A) is before the test and B) is after 1 month of weather exposure.

The fine sawn wood sample containing microfibrillated cellulose 1 with pigmented surface protection had less surface protection falling off but it had some wood protection falling off at knots. The fine sawn wood coated with the wood protection containing 0.75 % microfibrillated cellulose 1 and 0.4 % hydroxyethyl cellulose 2 and pigmented surface protection had loss of surface protection similar to the reference after the natural weathering test but it also had some loss of wood protection at knots which the reference did not have. The natural weathering tests showed that the samples containing clay 1 and diutan gum 1 or xanthan gum 1 had similar performance to the reference.

4.2.4 Wood protection in combination with unpigmented surface protection on fine sawn boards

The weathering tests with fine sawn wood coated with wood protection sample containing clay 1 and unpigmented surface protection had good performance which is shown in Figure 4.13 as the water droplet test showed that all of the surface protection was intact. There was however some loss of wood protection on some knots.

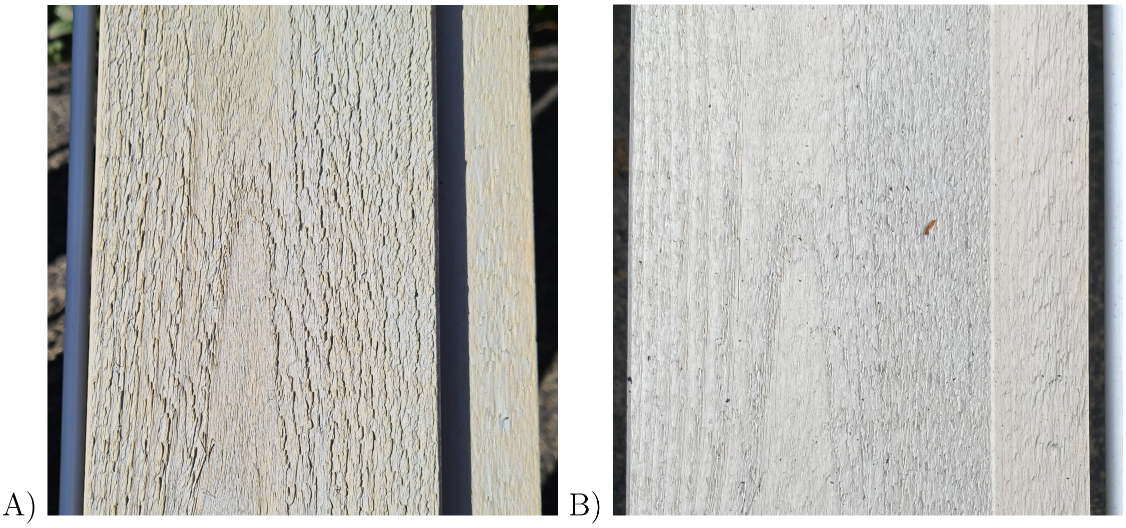

Figure 4.13: Fine sawn wood board coated with a sample of wood protection containing clay 1 and unpigmented surface protection used in the natural weathering test. A) is before the test and B) is after 1 month of weather exposure.

The wood protection samples containing thickeners based on gums on fine sawn wood with unpigmented surface protection had similar performance when compared to the reference which can be seen in Figure 4.14 and Figure 4.13. Both the surface protection and wood protection remained which could be seen visually and was shown by the water droplet tests.

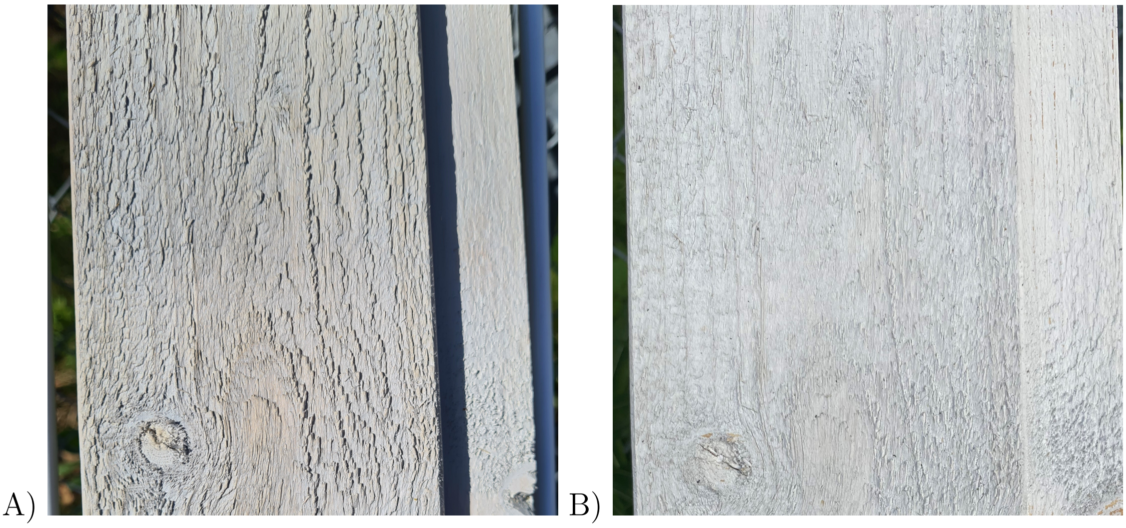

Figure 4.14: Fine sawn wood board coated with a sample of wood protection containing diutan gum 1 and unpigmented surface protection used in the natural weathering test. A) is before the test and B) is after 1 month of weather exposure.

The fine sawn wood coated with a wood protection sample containing microfibrillated cellulose 1 and unpigmented surface protection had good performance except some loss of wood protection on knots. Because of this it performs worse than the reference. The natural weathering tests showed that the samples containing clay 1 and diutan gum 1 or xanthan gum 1 had similar performance to the reference.

4.3 Viscosity tests on wood protection

Data values from viscosity tests with clay 1 is shown in Figure 4.15 together with a graph of a power law model fitted to the data. The power law model had a coefficient of determination, R^2 of 0.99 and a n of 0.52 which indicates that the liquid is pseudoplastic as $n<1$.

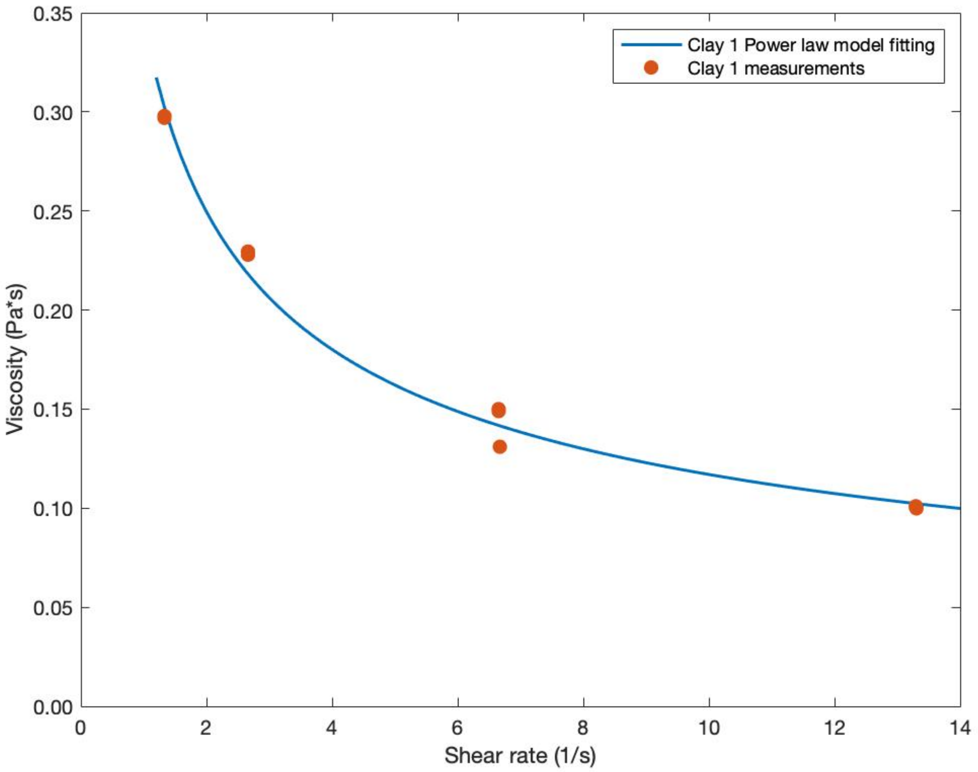

Figure 4.15: Data from viscosity measurements on wood protection containing clay 1 and a power law model curve fitted to the data.

The thickeners based on gums that performed the best in the initial weathering test are called diutan gum 1, xanthan gum 1 and xanthan gum 2. Further analysis of gums were carried out on these three thickeners. The values obtained from viscosity tests showed that the wood protection samples containing thickeners based on gum followed the power law model very well with an R^2 of 0.998, 0.988, and 0.988 for diutan gum 1, xanthan gum 1, and xantan gum 2 respectively. A graph of the viscosity measurements of wood protection with xanthan gum 1 and its power model regression is shown in Figure 4.16.

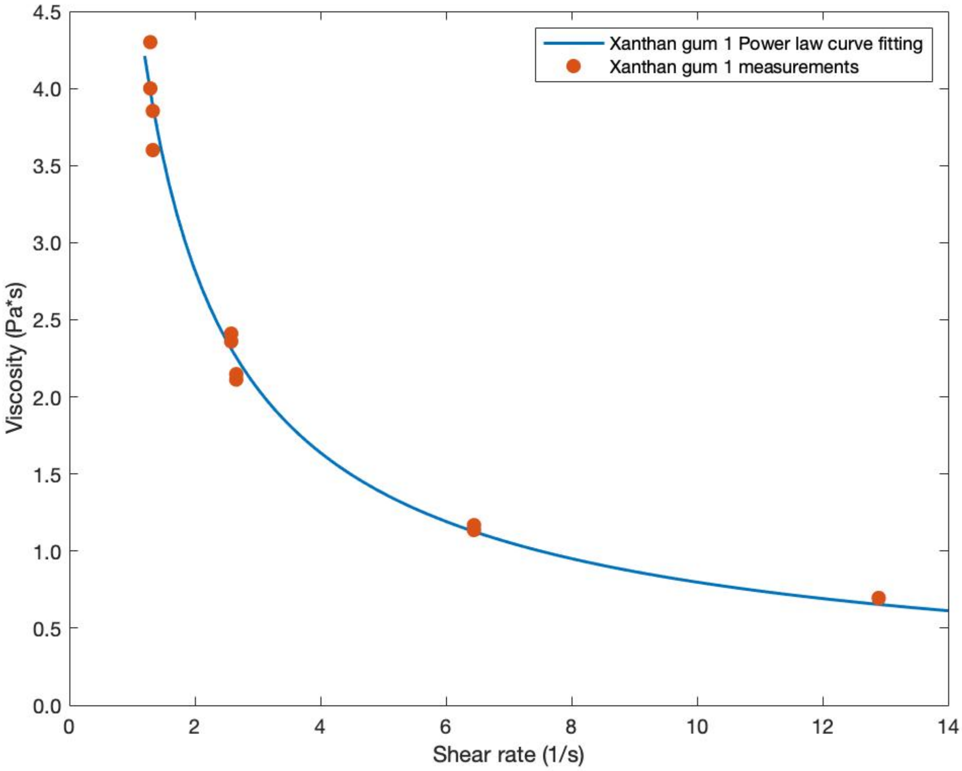

Figure 4.16: Data from viscosity measurements on wood protection containing xanthan gum 1 and a power law model curve fitted to the data.

Thickeners consisting of diutan gum and xanthan gum added a higher viscosity to the wood protection samples compared to the reference which is shown in Figure 4.17. The different types of xanthan gums give similar properties but not identical with regards to viscosity. Diutan gum 1 is a more effective thickener as only 0.25 % of the weight of the total wood protection batch was added, compared to 0.4 % for xanthan gum 1 and 0.7 % for xanthan gum 2, yet it had adequate viscosity. The viscosity of the samples containing xanthan gum 1 and 2 had similar viscosities even though more xanthan gum 2 was used which indicates that xanthan gum 1 is a more effective thickener. The wood protection samples get a pseudoplastic rheology from the gum thickeners which is displayed by the graphs in Figure 4.16 and Figure 4.17 but it can also be determined by the values of n is equal to 0.1748, 0.216, and 0.238 for diutan gum 1, xanthan gum 1, and xanthan gum 2 respectively. Since $n < 1$, all three gum thickeners are pseudoplastic.

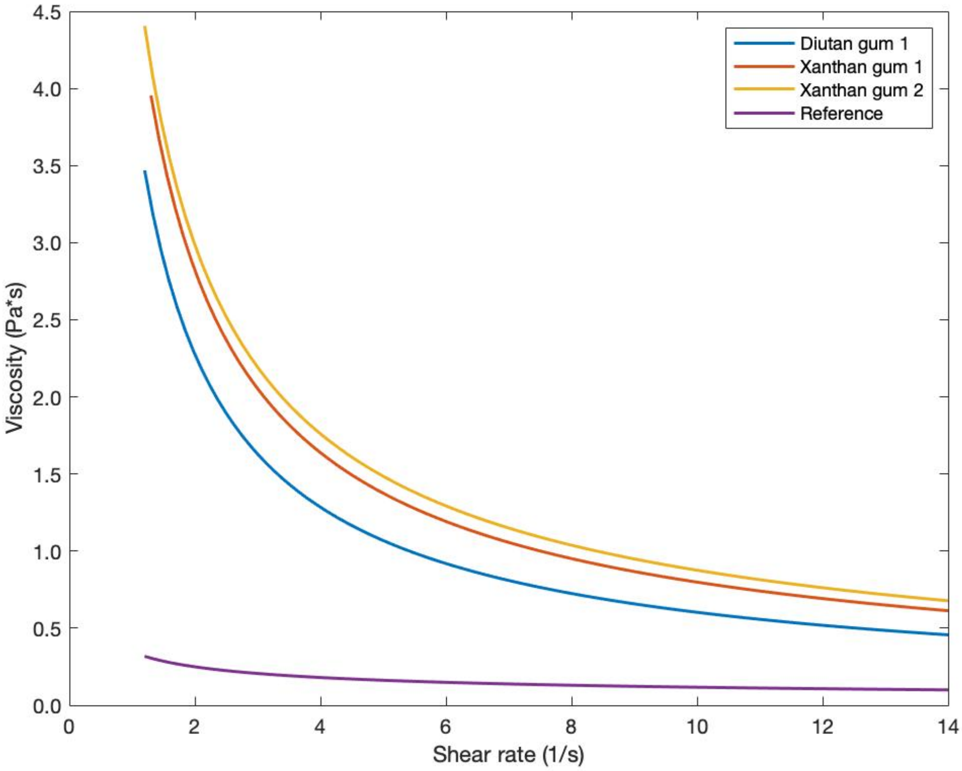

Figure 4.17: Power law model curve fitted to data from viscosity measurements on wood protection samples containing Diutan gum 1, Xanthan gum 1, Xanthan gum 2, and the reference thickener.

The viscosity measurements indicated that certain viscosities at low shear rates were needed to achieve storage stability. Measurements on samples containing polyurethanes and hydroxyethyl cellulose showed that they had low viscosities at low shear rates which is why they have poor storage stability. The thickeners based on gums added high viscosity to wood protection which increased the storage stability. Some samples containing microfibrillated cellulose did have higher viscosities than the samples with thickeners based on gums but these samples did not have good storage stability due to phase separation. Most samples of wood protection had pseudoplastic rheology but samples with low viscosities had Newtonian rheology according to the measurements. This may be a result of the thickeners not having any effect on the viscosity of these samples and the rheology was therefore similar to the rheology of water which is Newtonian.

4.4 Tests of wood protection using leneta paper

The leveling test showed that wood protection containing clay 1 had excellent leveling and the leneta applicator test showed that the wood protection creates an even coating on a leneta paper. The test is shown in Figure 4.18

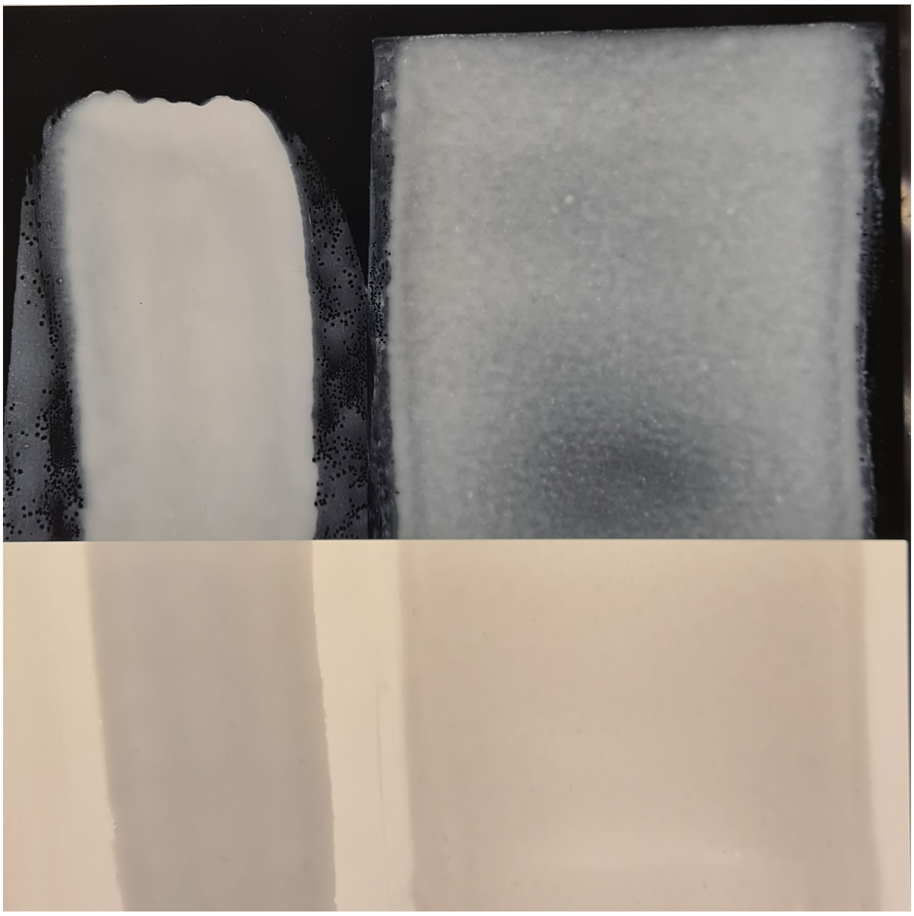

Figure 4.18: To the left: Leveling test on leneta paper with wood protection containing clay 1. To the right leneta applicator test with wood protection containing clay 1.

The leveling tests showed that the thickeners based on gums had adequate leveling properties. All samples covered the leneta paper evenly during the leneta applicator test. One sample had craters or fisheyes which most likely was due to a contamination on the paper which had other surface energies so that it was energetically favorable to form a crater instead of covering the area which is mentioned in Section 2.5.4. The coated leneta paper containing craters or fisheyes is shown in Figure 4.19.

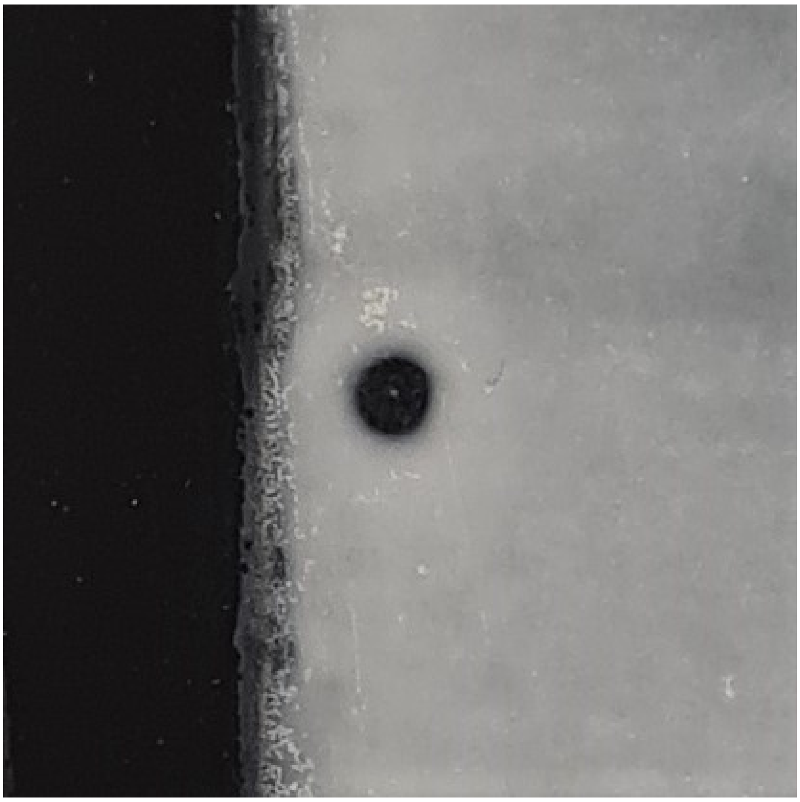

Figure 4.19: Craters in a coating from a leneta applicator test of wood protection with xanthan gum 1.

The low viscosity of the wood protection samples containing only polyurethanes gave the samples good leveling properties but paint peeling could be seen in both the leveling test and the leneta applicator test which is shown in Figure 4.20. The low viscosity at low shear rates resulted in poor sag resistance.

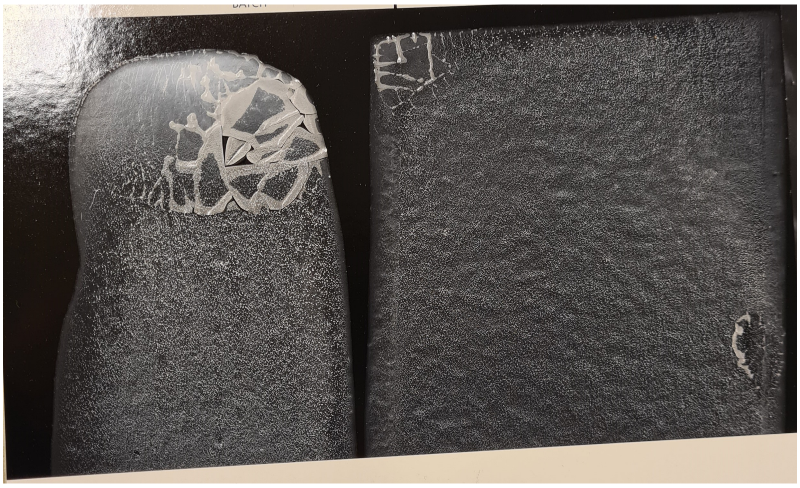

Figure 4.20: To the left: Leveling test with wood protection containing polyurethane 2. To the right: leneta applicator test on wood protection containing polyurethane 2.

The wood protection samples containing fumed silica had paint peeling after drying on leneta paper and the low viscosity of the samplesgace them good leveling but poor sag resistance. The samples containing hydroxyethyl cellulose had good leveling but little to no sag resistance. They also covered the leneta paper poorly in the leneta applicator test.

When a 0.5 % microfibrillated cellulose 1 wood protection sample was applied to leneta paper, a grainy coating was created with some pinholes, craters or fisheyes which could be a result of bubbles or contaminants. The 0.5 % sample had adequate leveling but poor sag resistance. A 1 % microfibrillated cellulose 1 sample was also evaluated and it had poor leveling due to a too high viscosity. Sag tests were performed on the 1 % microfibrillated cellulose 1 sample and no sag was identified.

The leveling of the sample containing 0.35 % microfibrillated cellulose 1 and 0.05 % diutan gum 1 was also poor and the coating had many craters or fisheyes in the leneta applicator test. A sample containing 0.7 % microfibrillated cellulose 1 and 0.1 % diutan gum 1 was also made which had poor leveling but the extra amount of thickener gave the wood protection significant sag resistance. The wood protection sample containing 0.75 % microfibrillated cellulose 1 and 0.5 % polyurethane 2 had poor leveling and good sag resistance but the coating on the leneta paper was grainy.

4.5 Gloss tests of wood protection

The coatings containing pigmented surface protection had higher gloss compared to coatings with unpigmented surface protection except for a coating containing wood protection with a combination of clay 1 and diutan gum 1. Most thickeners evaluated in wood protection had little impact on the gloss of the coating as most values were similar to the reference. However, microfibrillated cellulose 1 increased the gloss of the coatings when it was used in the wood protection. It also increased the gloss of the coatings it was used in when it was used in a combination with hydroxyethyl cellulose 2 in wood protection which is shown in Table 4.1. Clay 3 decreased the gloss by about 1 GU when it was used in wood protection compared to the reference which also is shown in Table 4.1.

Table 4.1: Mean values from some gloss measurements on planed wood boards coated with wood protection containing some evaluated thickeners and surface protection.

Microfibrillated cellulose 1	Gloss (GU)
Narrow growth rings, pigmented surface protection	7.37
Narrow growth rings, unpigmented surface protection	5.72
Broad growth rings, pigmented surface protection	8.26
Broad growth rings, unpigmented surface protection	7.40
Microfibrillated cellulose 1 and hydroxyethyl cellulose 2	Gloss (GU)
Narrow growth rings, pigmented surface protection	6.92
Narrow growth rings, unpigmented surface protection	6.20
Broad growth rings, pigmented surface protection	8.12
Broad growth rings, unpigmented surface protection	5.60
Clay 3	Gloss (GU)
Narrow growth rings, pigmented surface protection	4.19
Narrow growth rings, unpigmented surface protection	3.87
Broad growth rings, pigmented surface protection	4.26
Broad growth rings, unpigmented surface protection	3.59
Reference	Gloss (GU)
Narrow growth rings, pigmented surface protection	5.57
Narrow growth rings, unpigmented surface protection	4.41
Broad growth rings, pigmented surface protection	5.30
Broad growth rings, unpigmented surface protection	4.48

4.6 Non-volatile matter tests of wood protection

Most samples in the non-volatile matter tests had values around 17.4 % which was the value of the non-volatile matter of the reference clay 1 sample. Wood protection samples containing thickeners based on gums as well as hydroxyethyl cellulose had slightly lower values because a lower concentration of thickener was used. Samples of wood protection containing fumed silica had big differences in values as different amounts of thickeners were used. The samples with polyurethane thickeners had very low values. One reason is that polyurethanes are added as a liquid dispersion containing polyurethanes and water in which water will evaporate upon drying. However, since the amount of dry thickener added was around 1 % of the total formulation, the difference in the weight of the thickeners can only influence the value of the non-volatile matter by less than 1 %. In the samples with polyurethanes, there was a dark viscous layer at the liquid air interface. When the samples were mixed, the layer turned into dark lumps. The lumps may have more pigment and thickener. As a result, the values of the non-volatile test depends on the amount of lumps present in the sample used in the non-volatile matter test. There may however be some issues with the method since the non-volatile matter test with 0.5 % microfibrillated cellulose had higher non-volatile content values than the sample with 1 % microfibrillated cellulose.

4.7 Color measurements on wood protection

Values of the dark-bright scale of the wood protection samples were fairly similar. Wood protection samples containing hydroxyethyl cellulose, fumed silica and thickeners based on gum were significantly darker than the reference and other thickeners. The combination of microfibrillated cellulose 1 and hydroxyethyl cellulose 2 was also fairly dark, probably due to hydroxyethyl cellulose making the sample darker. These darker samples had values of L between 67.6 and 71.99 while the other samples had values between 75.16 and 77.63. These difference are so small that they will be difficult to see visually. The values on the green-red scale a was almost identical as all values were between -0.49 and 0.07. The values on the yellow-blue scale b were also similar, ranging between -1.29 and 1.61. The differences in the green-red scale and the yellow-blue scale is so small that it is impossible to see a difference visually. These tests show that the use of different thickeners has a very low impact on the color of the wood protection.

4.8 Storage stability tests of surface protection

Clay 1, 2, and 3 were evaluated in surface protection. Samples with clay 1 had the best storage stability of the three clay thickeners but they all had some sedimentation after storage. Surface protection with thickeners based on gums did not get the same storage stability as the wood protection. Storage stability tests showed that some sedimentation took place and the thickening effect was not even, some areas of the paint were thicker than others. Storage stability tests showed that polyurethanes in surface protection gave little to no thickening effect. Sedimentation was present quickly after production. Since polyurethanes did not increase the viscosity noticeably, no further evaluations were made with polyurethane thickeners as the only thickeners in surface protection.

The fumed silica thickeners did not add storage stability to surface protection. White pigment sedimented on the bottom and the bulk got dark. Hydroxyethyl cellulose thickeners were tested in surface protection. Hydroxyethyl cellulose 3 performed the best in storage stability tests and showed good storage stability but the viscosity got very high.

Microfibrillated cellulose 1 did not add good storage stability to the surface protection. A layer of foam containing black pigments was formed on top of the bulk of the sample. White pigments had sedimented to the bottom of the sample. In between the foam and the bottom, there was a white layer with some white pigment.

4.9 Natural weathering tests of surface protection

The surface protection samples performed better than the wood protection samples in the natural weathering tests The results from the natural weathering tests were varied and the performance of the reference sample containing clay 1 was poor compared to most other samples.

4.9.1 Surface protection on planed boards with pigmented wood protection

The wood board coated with pigmented wood protection and surface protection containing clay 1 had some loss of surface protection or pigment but mo loss of wood protection. It could also be seen that cracks were present, possibly mud cracks. These cracks were not yellow which some of the cracks were in the natural weathering tests of wood protection. No mud cracks were however present on other wood boards coated with surface protection containing other thickeners. Most samples containing other thickeners had some loss of both wood and surface protection. The combination of clay 1 and xanthan gum 2 in surface protection had the worst performance as a lot of the wood and surface protection had fallen off due to paint peeling. The coating made from surface protection containing hydroxyethyl cellulose 3 had an uneven distribution of surface protection as the viscosity was too high during application. It had less loss of surface protection but more loss of wood protection compared to the reference.

4.9.2 Surface protection on planed boards with unpigmented wood protection

The coating made with unpigmented wood protection and surface protection containing clay 1 had mud cracks and more surface protection or at least pigment absorption at the light growth rings. A lot of surface protection had fallen off and it showed the worst result of all tested surface protection samples. Hydroxyethyl cellulose 3 had an uneven coating and loss of surface protection but less than the reference. Microfibrillated cellulose 1 had great performance as no wood or surface protection had fallen off. Most samples had some loss of surface protection but no loss of wood protection.

4.9.3 Surface protection on fine sawn boards with pigmented wood protection

The coating made with pigmented wood protection and surface protection containing clay 1 had good performance as only a little amount of surface protection had fallen off while all of the wood protection remained on the board. All samples showed similar performance to the reference, except coatings made with pigmented wood protection and surface protection containing clay 2 or a combination of clay 1 and xanthan gum 2. These samples had no or a very low loss of surface protection. Those combinations had poor performance on planed boards which indicate that the aging might not be the same when comparing a sample on different surfaces. The tests on planed boards gives an indication on how the paint samples performs on smooth surfaces and can not be used to predict the performance of a paint sample on a rough surface. The tests on planed boards can not be used to show accelerated aging but it does show the performance of the paint sample on smooth surfaces which also is of importance.

4.9.4 Surface protection on fine sawn boards with unpigmented wood protection

All samples showed good performance with unpigmented wood protection on fine sawn boards. The samples had no or very little loss of surface protection and no loss of wood protection. Since the time of the natural weathering test only was one month, not much aging can be seen. The time would probably need to be extended to see differences in the performances.

4.10 Viscosity tests of surface protection

The viscosity of the samples with different thickeners varied greatly. Measured viscosities of clay 1, which is the reference, and xanthan gum 2 as well as a power low model fitted to the values are shown in Figure 4.21. The graph shows that the viscosity of the surface protection containing xanthan gum 2 had higher viscosity than the surface protection sample containing clay 1.

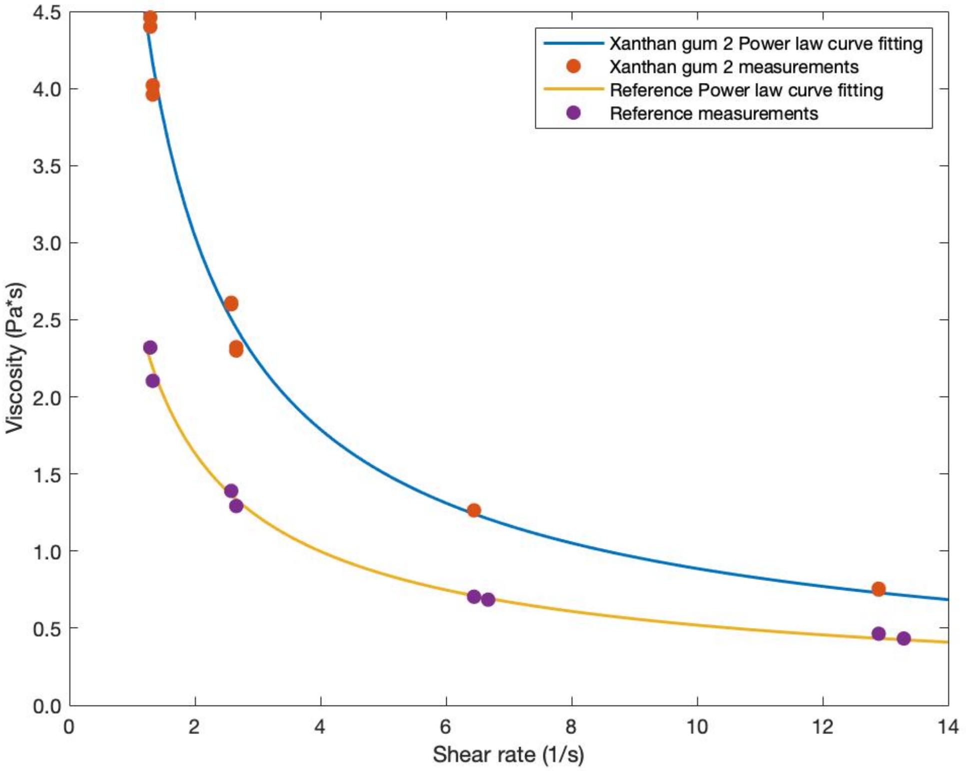

Figure 4.21: Data from viscosity measurements on surface protection containing xanthan gum 2 and a power law model curve fitted to the data as well as data and the curve for the reference sample.

The measurements on the surface protection sample containing xanthan gum 2 shows that the viscosity is high which is an indication of storage stability but the sample wasn't homogeneous like the wood protection samples containing thickeners based on gums. Xanthan gums and diutan therefore worked best in wood protection.

All samples had pseudoplastic rheology in the measurements on surface protection except samples with very low viscosities. The samples with very low viscosities had Newtonian rheology. This could be due to the of thickeners having a low effect on the viscosity of these samples and as a result the rheology was similar to the rheology of water. Most samples with low storage stability had low viscosity at low shear rates except samples containing microfibrillated cellulose 1 which had had high viscosity but relatively low storage stability due to phase separation. Hydroxyethyl cellulose 3, which had good storage stability, did not have a very high measured viscosity at low shear rates. The highest measured viscosity of the surface protection sample containing hydroxyethyl cellulose 3 was 1.775 Pas but the viscosity of the sample may increase significantly when the shear rate is lower than the shear rates that the viscometer can measure at.

4.11 Tests of surface protection using leneta paper

All samples with clays had good leveling and created an even coating in the leneta application test. All samples containing clay had too low viscosity to be tested in the sag resistance test. Sag resistance tests made with surface protection samples containing gums had sag between all stripes. The samples containing gums covered the leneta paper evenly in the applicator tests but small white pigment grains were present in the coating. The samples containing gums showed adequate leveling in the leveling test but had sag between all lines in the sag resistance tests. The low viscosity of the fumed silica samples gave the surface protection samples good leveling but the samples created an uneven coating with poor adhesion in the leneta paper applicatior test. The sample containing hydroxyethyl cellulose 3 did not pass the leneta leveling test due to the high viscosity. It covered the leneta paper well in the applicator test but there were some craters or fisheyes in the coating. The craters or fisheyes were probably due to contaminants. A sag test was also performed but there were sag between all stripes. When the surface protection with microfibrillated cellulose 1 was applied to planed wood, the coating became uneven with paint lines from the brush which indicate poor leveling characteristics. The leveling test confirmed this as the surface protection containing microfibrillated cellulose 1 did not pass the leveling test.

4.12 Gloss measurements of surface protection

The gloss measurements of surface protection samples varied depending on the thickeners, the color of the wood protection and the growth rings. Coatings with unpigmented wood protection had higher gloss in most samples. How narrow and broad growth ring impact the gloss depends on the thickener. The coatings made from surface protection containing clay 2 had lower gloss than clay 1. This could be a result of the high amount of thickener in the sample containing clay 2 which may lower the gloss. Hydroxyethyl cellulose had similar gloss as the reference. The values of the gloss measurements of coatings made from surface protection containing hydroxyethyl cellulose had a high standard deviation which could be due to the uneven distribution of surface protection due to the high viscosity of the surface protection sample. Some of the values from the gloss measurements are shown in Table 4.2.

Table 4.2: Mean values from gloss measurements on planed wood boards coated with wood protection and surface protection samples containing a variety of thickeners.

Clay 2	Gloss (GU)
Narrow growth rings, pigmented wood protection	5.51
Narrow growth rings, unpigmented wood protection	8.74
Broad growth rings, pigmented wood protection	4.91
Broad growth rings, unpigmented wood protection	9.36
Clay 1	Gloss (GU)
Narrow growth rings, pigmented wood protection	9.15
Narrow growth rings, unpigmented wood protection	13.8
Broad growth rings, pigmented wood protection	11.52
Broad growth rings, unpigmented wood protection	11.65
Microfibrillated cellulose 1	Gloss (GU)
Narrow growth rings, pigmented wood protection	6.62
Narrow growth rings, unpigmented wood protection	10.7
Broad growth rings, pigmented wood protection	7.13
Broad growth rings, unpigmented wood protection	15.27
Hydroxyethyl cellulose	Gloss (GU)
Narrow growth rings, pigmented wood protection	11.26
Narrow growth rings, unpigmented wood protection	9.53
Broad growth rings, pigmented wood protection	9.82
Broad growth rings, unpigmented wood protection	15.03

4.13 Non-volatile matter of surface protection

The reference sample which contained clay 1 had 17.6 % non-volatile matter according to the test. Most samples containing a similar amount of thickener had similar values. Several samples which had higher values contained more thickener. For example, a sample containing 3.17 % clay 2 had 19.7 % non-volatile matter. Since more thickener was used the non-volatile matter is increased as the thickeners does not evaporate at 100-110 °C. However, the samples containing polyurethanes had too low values to be explained by the fact that a low amount of thickener were used which can be seen in Table 4.3. These values indicate that there is a source of error in the method.

Table 4.3: Values of the percentage of non-volatile matter of surface protection samples containing polyurethane thickeners.

	Non-volatile matter (%)
0.5 % polyurethane 1	14.8
0.5 % polyurethane 2	15.5

4.14 Color measurement of surface protection

The L values of the samples varied the most in the color measurements. The L value was between 74.1 and 80 and these differences can be seen visually. All values were very similar on the green-red scale as all values were between -0.27 and -0.49. All values were also similar on the blue-yellow scale as the lowest value was 0.24 and the highest 2.75. This indicates that the thickeners have a low impact on the color of the surface protection, and the impact that the thickener has on the brightness can be adjusted for.

5

Conclusion

Several thickeners were evaluated in both the wood protection and surface protection. Most thickeners and thickener combinations did not add good storage stability to the panel paints. There were also issues regarding natural weathering tests as using different colors on the wood protection and surface protection impaired the performance of the coating. Wood protection and surface protection of the same color should be used in future natural weathering tests. The natural weathering tests showed than the performance of a coating on planed boards can not be used to predict the performance of a coating on fine sawn boards. The coatings should therefore be evaluated over a longer time period on fine sawn wood. Planed wood can however be used in natural weathering tests to evaluate how coatings performs on smooth surfaces. Xanthan gums and diutan gums gave the wood protection good properties in regards to stability, application and protection of wood. Combinations of thickeners based on some clays and xanthan gums or clays and diutan gums also added good storage stability, improved application by brush and made the coating protect wood well. Hydroxyethyl cellulose may be used in surface protection but at a lower amount than 0.9 wt% as it provided good storage stability but gave the surface protection too high viscosity. Surface protection samples should be made which contains less than 0.9 wt% hydroxyethyl cellulose in the future to see if still provides good storage stability.

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