Varnishing of Fiber-based Closures

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MASTER THESIS

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Abstract

This research aims to investigate water-based varnishes and technologies to coat fiber-based closures to attain water barrier properties. To ensure recyclability the repulpability of coated and uncoated lids is tested. The research approach is exploratory. The investigation begins with the application of varnishes using a spray gun, brush, and dip coating followed by spraying equipment from Spraying Systems.

Nine varnishes are tested in the research. Based on the chosen minimum criteria to select the varnish, the primary objective is to analyze which of the varnishes show the following results 1) lower amount of dry coat weight(g/m^2) (2) long penetration time (min) (3) high contact angle (°). The appearance (color, texture, and homogenous coating) of the coated lid is evaluated through visual observation. Hydrophobicity is evaluated through water contact angle measurements using a goniometer, penetration time and visual observation. The repulpability trials of the uncoated lid and coated (selected varnish) material is tested using the lab equipment at Karlstad University.

The results show that closures coated with varnish 6 and 8 meet the minimum requirement; have a good visual appearance in terms of color and texture, high contact angle and relatively long penetration time. Repulpability results show that lids coated with varnish 6 are recyclable. Spray coating technology appears to be a potential process for implementation, although up-scaling is yet to be evaluated.

Keywords: Varnishing, fiber-based closures, water-based spray coating, packaging, repulpability

Executive Summary

Introduction:

Marine pollution from plastics has been a growing concern. Among the many wastes that end up in the ocean, beverage containers made of plastic which is non-biodegradable are among the most frequently dumped wastes entering the ocean. These plastics have a long span of life and can take centuries to degrade. The ingestion of plastics by marine animals is one of the most serious environmental impacts on the marine environment. Hence, policies are being implemented to reduce the use of single-use plastics.

Producers and consumers have also observed an increase in awareness of the environmental impact caused by packaging, the need for recycling, and the value of eco-design. Retailers and brands are under pressure to fulfill these requirements. Thus, packaging industries are making a shift to find packaging that is sustainable. BOC is one such Swedish sustainability start-up that is revolutionizing the packaging sector by creating fiber-based screw closures and lids to replace plastic.

Fiber-based packaging is gaining importance due to its recyclability and biodegradability. It has also been proven to have less environmental impact as compared to metal or plastic in terms of carbon emission and effect on climate change. One challenge is the natural porous and hydrophilic nature of fiber-based materials that prevents its application in certain products that require good barrier properties. To improve the technical performance, coating is required. Therefore, the focus of the study is the varnishing of fiber-based closures to attain water barrier properties.

Objective

- To analyze the type and amount of varnish to apply.
- To analyze the water barrier properties of the above-coated closures.
- To study the pros and cons of coating technology.
- To evaluate and compare the recyclability of the coated (selected) closure with the uncoated.

Hypothesis

Based on the correlation between the coat weight and barrier properties, and in alignment with company's requirements, the assumption is that:

1. The varnish that shows the below result is the most desirable, viable, and feasible:
    a. Lower coat weight.
    b. High contact angle measurements
    c. Relatively long penetration time
2. The spray coating technology appears to be a potential technology for coating due to proven studies of improving barrier properties.
3. The material with a yield of 70% and above is recyclable.

Materials and Methodology

The materials used in this project are cellulose fiber-based closures (produced at BOC) and nine varnishes from different suppliers. The investigation began with testing the varnishes by applying them on the closures using a Sames Kremlin SAS Spray gun. Due to technical complications in the equipment, brush coating and dip coating were adapted to evaluate the performance while a new spraying equipment was being arranged. After coating, the dry coat weight of the coated samples was calculated using the following formula and evaluated for performance.

Dry Coat weight= $\frac{(final\ weight\ - initial\ weight)}{surface\ area\ of\ sample} X\ dry\ content$ %

Once the new spraying equipment arrived, the varnishes that showed good performance were tested using the new spraying equipment from Spraying Systems to select the most desirable, viable, and feasible varnish.

The coated samples were analyzed for contact angle measurements using an Oscilla contact angle goniometer, to measure hydrophobicity. The penetration time of the droplets was noted to check the lasting during of water droplet. The repulpability trials were conducted at the lab and facility of Karlstad University. The equipment used for the repulpability are: (a) disintegrator to repulp the material, (b)Somerville screening equipment with 40µm diameter pore mesh to screen the accept and reject material, (c) MESSMER BUCHEL Handsheet former and Lorentzen & Wettre sheet press to make the handsheets and (d) Memmert heating/drying oven to dry the handsheets.

The visualization of the results and the variance in coat weight, contact angle, and penetration time were determined through graph builder tool and multiple variance analysis (ANOVA) using the JMP Pro 16 software respectively.

Results and Discussion

Visual Observation:

V1, V2, V5, V6, and V8 showed good barrier properties, transparent color, and a smooth texture. V3, V4, V7, and V9 showed undesirable results: brown color of the varnish, crystallization, rough texture after coating and drying, and poor water barrier property.

First screening step:

In the quantitative analysis, V1, V2, V5, V6, and V8 fulfil the chosen minimum criteria of coat weight below $20g/m^2$, a contact angle of 90° and above, and a penetration time of 20 min and above which will further be tested using the new spraying equipment.

Second screening step:

V6 showed relatively low coat weight (11.8, 20 and 14.3 g/m^2) with high contact angle measurement (107.9º) and relatively long penetration time (82.6 and 23.3 min) respectively. V8 while using a lower coat weight ($11g/m^2$) showed high contact angle measurement (91.46 º).

Repulpability:

The V6 coated lids with a coat weight of 12.9 g/m^2 defibrillated for 10 and 20min at 3000rpm had a yield of 80.5 and 85.6% respectively. Whereas the yield for uncoated lid had yield of 86% for 10 and 20min defibrillation.

The sheet adhesion for the handsheets was absent, which implies there was no damage or breaking when the sheet was simply separated from the support and gloss sheet for both coated and uncoated lids. However, the rejected material from coated lids after screening was slightly blue which requires further research to study the phenomena or its recyclability.

Coating technology:

As hypothesized, spray coating from Spraying Systems equipment helped in achieving the results. It was easy to handle, possible to attain low coat weight by regulating parameters like airflow, liquid flow, and size of the droplet due to the availability of various nozzles.

Conclusions

The conclusions obtained from this study are presented as follows:

Nine varnishes and three technologies (spraying, dip, and brush coating) were evaluated. The results of qualitative and quantitative analysis matched. The varnishes were selected through two screening steps.

V1, V2, V5, V6, and V8 were selected in the first screening step. The selected varnishes were tested using the new spraying equipment. In the second screening step, V6 showed both high CA (107.9°) and relatively long penetration time (82.6 and 23min) while using a lower coat weight 11.8g/m^2, 20g/$m^2$and 14.3g/m^2 respectively. V8 showed the good results for contact angle (91.46°) while using a relatively low coat weight (11g/m^2).

The spraying equipment from Spraying Systems appears to be a potential process for implementation, although scaling up is yet to be evaluated.

Repulpability of the uncoated and V6 coated lids obtained a yield of 86% and 80% at 10 min defibrillation respectively. For 20 min defibrillation, the uncoated and coated lids showed a yield of 86% and 85.6% respectively. Therefore, samples coated with varnish 6 are recyclable.

Future Recommendations

Since varnish 8 displayed good results, it is interesting to check the repulpability.

Cobb tests were performed but due to time constraints, the results could not be analyzed. The results would be beneficial for further analysis of barrier properties.

The second spraying equipment has five different nozzles. It is interesting to test the varnishes with different nozzles to check the spat flow and coating homogeneity.

Apparently, all the varnishes need to be tested using the new spraying equipment.

The results would be more reliable if the tests were conducted in a controlled environment. Environmental factors, like wind affected the direction of the flow of the spat from the equipment in this project, because of outdoors experiments.

Another interesting parameter to check would be the effect of temperature on the varnish performance.

Lastly, some food applications require high barrier properties like gas and oil. It is interesting to test the coated samples for oil and gas barrier properties.

Popular Scientific Summary

Varnishing of Fiber-based Closures

Every product we see in the market these days is packaged in some material, for instance, plastic, glass, metal, paper, etc. Once the product is used, the packaging material ends up as waste. Research says that out of the many wastes that end up in the ocean, beverage containers made of plastic are the most dumped waste. Due to this, marine pollution has been a growing concern since plastics take centuries to degrade and affect the natural habitat of the oceans. As a result, regulations are being implemented to reduce the usage of single-use plastics.

BOC is a Swedish start-up that is producing fiber-based closures to reduce plastic. Fiber-based products are gaining importance due to their biodegradability, high recycling rate, and they cause lesser environmental impact compared to plastics and metals in terms of carbon emission. However, one disadvantage of fiber-based material is their porous structure. We might have come across some paper bags for example, they readily absorb water and are used in products that do not need to be stored for very long.

To make the fiber-based materials suitable for longer shelf-life products, water-resistant property is required. This can be attained by coating them with a material that is hydrophobic (water-repellent). The materials used for coating are called varnishes and the process to apply on closures is called varnishing or coating.

This project aims to help BOC in investigating the type and amount of varnish, and coating processes for fiber-based closures. The chosen minimum criteria to select the varnish is the varnish that displays the following results.

(a) lower amount of coat weight so that it is economical,

(b) high contact angle measurements (angle between the liquid and coated lid), a higher contact angle implies water repellency, and

(c) long penetration time (the lasting duration of the liquid on the lid).

Since we want the closures to be recyclable, the coated closures need to be repulpable. If the repulpability yield is 70% and above, it indicates the product is recyclable.

We have tested nine different water-based varnishes from different suppliers and three coating technologies, spray, brush, and dip coating.

In conclusion, V6 and V8 showed the best performance by meeting the minimum criteria. The selected varnishes are food-grade and recommended for water-barrier-demanding food products. The repulpability yield of the coated lids ensured that varnish 6 coated lids are recyclable. Spray coating technology appeared to be the most suitable because it was easy to handle and attained lower coat weights.

Lastly, one interesting further recommendation would be to investigate the ocean biodegradability of the coated closures.

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1.Introduction

1.1 About the company

BOC is a Swedish start-up that is revolutionizing the packaging sector by creating fiber-based screw caps and lids. Intending to be first to market, they are addressing the global challenge of plastic pollution by guaranteeing that their premium goods adhere to five key principles: being biobased, ocean-biodegradable, recyclable, affordable, and scalable. Businesses and entrepreneurs around the world are working to attain sustainability, and BOC is contributing to the advancement of packaging. BOC is looking into novel, sustainable packaging options and helping well established companies to reduce their carbon footprint.

In April’23, the disruptive start-up BOC teamed a Swedish brand producing jams, marmalades, jellies, fruit drinks, smoothies, juices, fruit soups, and compotes. Some of the questions they are investigating are: would it be possible to replace metal lids for consumer products such as jams and spreads with a lid made of biobased fibers?

Metal screw lids are the only available option for several products on the market right now. The proposal is founded on the certain notion that a recyclable lid concept composed of cellulose fibers would satisfy market demands for more sustainable solutions and be supported by requests from customers around the world. A biobased material might potentially replace metal and save more than 500 000 tons of carbon dioxide equivalents annually in terms of reducing climate consequences (BOC, 2023).

They have developed a cost and energy-effective production concept using advanced, proprietary vacuum press forming to shape the closures to reduce the impact on climate change by reducing the carbon footprint by the production units, which is one of the ways to achieve an economic-environment win-win situation (Varghese, Lewis, and Fitzpat, 2012). Their first research and production facility are based in Säffle. They are developing and aiming at producing fiber-based closures in the coming months. Some of these closures, depending on the targeted products, may need to be varnished inside and/or outside to protect them from e.g., water.

To continue the development of their product, the main tasks of the master's thesis work are centered on this topic and include:

- Analyze the type and amount of varnish to apply, how many layers are needed, how to dry efficiently, and how to handle the overspray.
- Investigate the processes and products to varnish the closures at lab scale.
- Set up and carry out varnishing trials with different technologies.
- Compare performances and pros/cons of the technologies (water-based spray technology).
- Evaluate performances of the coated closures (visual quality, technical performances…).

1.2 Background/Context

Reducing the overall environmental impact of packaging and lowering the number of single-use plastics put on the market has become a common ambition for all of us. The awareness of the environmental impact caused by packaging, the need for recycling, and the value of eco-design are spread among the customers and consumers. Retailers and brands are required to fulfill these expectations of the market.

According to Myer Kutz (2007) one of the main industries using plastics is the packaging sector. In terms of commercial use, packaging (35.9%), building (16.0%), textiles (14.5%), and consumer goods (10.3%) accounted for the biggest amounts of plastic globally in 2015. With 146 million tons produced in 2015, of which 141 million tons were not recycled (96.6%), packaging is thought to be the leading cause of garbage globally. Among all industrial plastics sectors, packaging has one of the shortest average working lives. The lifespan of single-use plastics, from use to disposal, can be as brief as a few minutes (Rosenboom, Langer, and Traverso, 2022). Mismanaged garbage and debris that end up in water bodies seriously harm wildlife, the general public's health, and the economy.

Many plastics that end up in the oceans are harmful to the natural habitats of the ocean (Martin, 2019). For many years, marine pollution from plastic has been a growing issue. Around 4.8 – 12.7 Mt of plastic was found in the ocean on a global scale in 2010. In 2014, a study conducted by Gyres Institute showed that 5.25 trillion particles of plastics were floating in the sea. Among the many wastes that end up in the ocean, beverage containers made of plastic which is non-biodegradable are among the most frequently dumped wastes entering the ocean (Schuyler et al., 2018). And these plastics have a long span of life and can take centuries to degrade. Among the many negative impacts of marine plastic pollution, the ingestion of plastics by marine animals is one of the most serious environmental impacts on the marine environment. Hence, policies are being implemented to reduce the use of single-use plastics (Xanthos and Walker, 2017).

To address the above-stated issue and reduce the burden on the environment, fiber-based products are becoming more popular in the field of packaging as they have one of the highest recycling rates of all the packages worldwide, making it both a sustainable and "circular" solution that closes the loop on resources to keep them in use or reuse longer (4evergreen, 2022). Another advantage is that fiber-based products are biodegradable, even if they are not collected or sorted and accidentally end up in the oceans or the environment they do not pose as serious a threat as compared to other packaging materials that are non-biodegradable. Royer et al (2021) conducted a test on wood-based cellulosic fabrics and they discovered that wood fabrics break down quickly in marine environments.

However, fiber-based packaging has certain challenges for its usage in the food industry, which are addressed below:

1.3 Challenges

1.3.1 Hydrophilic nature

Fiber-based materials' natural porous and hydrophilic nature prevents their application in certain food products that require good barrier properties. The hydroxyl group in the cellulosic material enables the molecules to bind to water and thus reducing the functional performance like mechanical strength and load-bearing capacity. To strengthen the technical performance (like barrier property), fiber-based products are coated with different varnishes which are hydrophobic in nature and are used to provide the barrier properties (Hebbar, Isloor, and Ismail, 2017) (Bandyopadhyay, Ramarao and Ramaswamy, 2002,).

1.3.2 Physicochemical properties

Finding a substrate's Armstrong level physicochemical characteristics can be very difficult. One way to comprehend the surface characteristic of a substrate is through the understanding of Young's model equation of contact angle. However, theoretically, it is expected that a surface's contact angle value will be a defining characteristic of that surface in a particular situation. Therefore, through water contact angle measurement we will be analyzing the hydrophobicity of the substrate.

1.3.3 Recyclability of the coated varnish

Barrier-coated fiber-based products face challenges in recyclability due to the presence of a mixture of polymers, adhesives, additives, etc. that are difficult to separate.

Although there are several articles on the varnishing of different products like paperboards, wood, and metals, there is no pre-existing and proven research yet for fiber closures. For further understanding, below presented is the correlation between coating, penetration time, and contact angle.

1.4 Correlation between coating, penetration time, and contact angle

First, the final property of the coated product depends on the type and amount of coat weight applied, as well as the application process and drying conditions. For economic viability, it is important to use the least amount of coat weight to attain the desired results (Kathuria and Zhang, 2022).

Second, the higher the contact angle the higher the hydrophobicity. We can tell from a liquid's contact angle how well (or how poorly) it will spread over a surface (Oscilla contact angle measurement manual). We can identify a material's hydrophobicity by looking at its water contact angle (Chen et al., 2021) (Hebbar, Isloor, and Ismail, 2017).

Third, the penetration time is the duration the water droplet lasts on the lid. A raw material (cellulosic fibers) without any pre-treatment displays compatibility and good adhesive strength due to its hydrophilic nature. The water tends to absorb into the substrate immediately. Since the material we use in this project is in its raw form, it appears to be suitable for varnishing and can readily allow the varnish to adhere to the substrate, especially the water-based varnishes. However, due to its natural porous structure it could require more coat weight to form a good barrier coating layer which makes it unsuitable. Hence, the need to conduct trials to test which among the varnishes gives us a good hydrophobicity with longer penetration time using less coat weight.

In alignment with the stated findings and the requirements of the company below hypothesis was generated in discussion with the BOC.

1.5 Hypothesis

1.5.1 Varnish

The varnish that shows the below result is the most desirable, feasible, and viable option.

1. Low coat weight
2. High contact angle
3. Long penetration time

1.5.2 Recyclability

It depends on the varnish that displays the results stated in 1.5.1. Once selected, if the coated closure has a recyclability yield above 70%, it is recyclable (4evergreen, 2022).

1.5.3 Coating technology

Research has been done on the coating of fiber-based materials like paperboards and papers using the spray coating technique which has shown some improvement in permeability and barrier properties. The products coated were microfibrillated cellulose (MFC) and shellac (Hult, Iotti, & Lenes, 2010). Research shows that spray coating technique has proved efficient in attaining the barrier property. This was tested for fiber-based materials, and they were diluted with liquid solvents (Teisala, Tuominen and Kuusipalo, 2013). In addition, through spraying, minimum coat weights as low as 5- 10g/m^2 can be attained.

Although spray coating has shown improvement in permeability and barrier properties. The same needs to be confirmed for fiber-based closures which have a different physical structure compared to paperboard which has a flatter surface. The closures are 3D objects, which makes it hard for them to be coated using traditional 2D methods that are currently employed in the paper industry. This is why we are exploring spray coating and appears to be a potential technology that can help meet the minimum criteria.

1.6 Research Question

Which varnish best exhibits good performance, such as an attractive aesthetic appearance, good barrier property, while fulfilling the minimum criteria for a fiber-based closure (described in the hypothesis)? What are the pros and cons of the selected technology? In other words, which varnish, and technology is the most desirable, feasible, and viable to integrate into the existing BOC machinery to varnish the fiber-based closure?

1.7 Aim/Objective

- To analyze the type and amount of varnish to apply, and how many layers are needed.
- To analyze the water barrier properties of the above-coated closures.
- To study the pros and cons of coating technology.
- To evaluate and compare the recyclability of the coated (selected) closure with the uncoated closure.

1.8 Limitations

This project aims to select the most desirable viable and feasible varnish and coating technology to coat the fiber-based closures to enhance the technical performance like barrier properties. Nevertheless, the study has limitations which are addressed below:

- Although we select a low coat weight for economic viability, this adds to the overall cost of the product due to the coating material, the technology, and the production cost.
- In spray coating, controlling the overspray is difficult which leads to wasted material, and the requirement of closed booth to treat the closures leads to an increased cost.
- Lastly, the technology is a limitation because coating in lab scale is one aspect, and scaling up for industrial coating is another aspect requires different kinetics and is uncertain.

2.Literature Review

2.1 Packaging

From using a container for packaging to being an important element in the product design, the last 200 years have seen a major evolution in the packaging world. For instance, from the use of glass bottles for tomato ketchup to squeezable multi-layered plastic bottles, and canned foods that were stored in tin cans are now stored in paper-based packages providing the same shelf-life of the food product (Coles, McDowell, and Kirwan, 2003).

Traditionally, the purpose of packaging has been to store, contain, protect, and ensure safe delivery of the goods from producers to consumers (Löfgren and Witell, 2005). Packaging has majorly six functions – protection, containment, communication, convenience, unitization, apportionment. In the food industry, packaging not only helps in protecting but also acts as a medium to preserve and increase the shelf-life of the product.

In recent times, we have seen a massive drift in the role of packaging, from protection to marketing to a cause of environmental impact. Once the contents of the packaging have been used, the packaging often ends up as waste and is sorted at the waste collection centers. Out of which, some are recycled, and reused as raw material, some are incinerated to generate heat with the aim of reducing the amount of waste that ends up in landfills (Müller et al., 2012).

Demands for renewable, recyclable, compostable, and biodegradable alternatives to oil-based products have recently boosted interest in cellulose from both the scientific and commercial perspectives. Even though there will soon be a rapid increase in new eco-friendly materials and biopolymers, paper and paperboard are currently the only renewable materials that are frequently utilized in packaging applications. Their poor barrier qualities and high sensitivity to moisture, however, restrict their use (Hult, Iotti and Lenes, 2010). Some of the advantages and disadvantages of paper packaging can be seen in the Table 1 and in Figure 1 we can see a schematic diagram of how the cellulose fibers are extracted showing the natural composition and the microstructure which makes it biodegradable.

Table 1 Advantages and Disadvantages of paper and paper-based packaging adapted from Heldman, Lund and Sabliov, (2006)

Advantage	Disadvantages
Versatile	Negligible resistance to
• Rigid	• water vapor
• Semi-rigid	• aromas
• flexible	• gas
Mechanical protection	
Logistics functions	Not heat sealable
Barrier to light	
Renewable resource	
Recyclable	
Biodegradable	

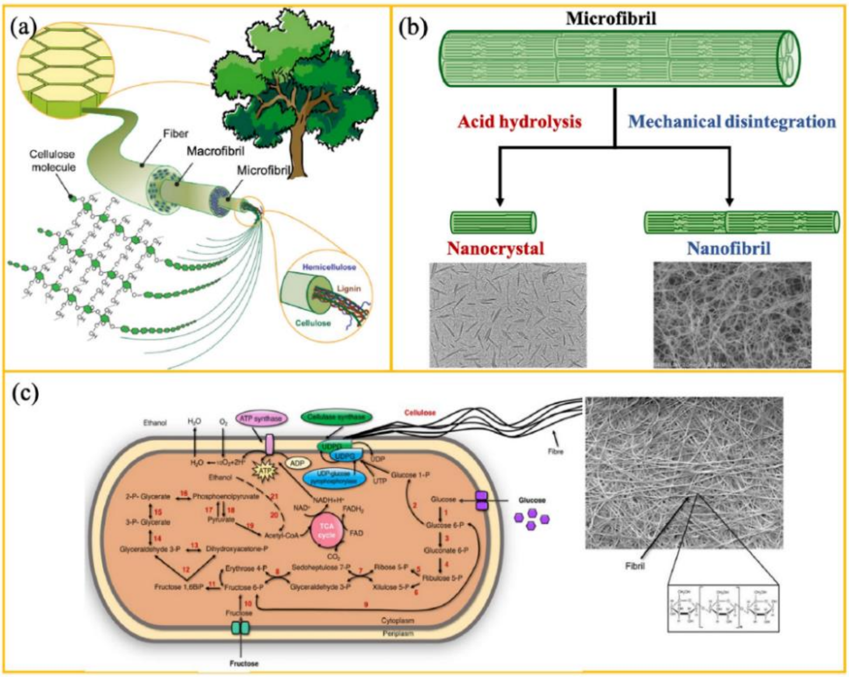

Figure 1 Microstructure of cellulose (Kathuria and Zhang, 2022)

To reduce marine pollution and address littering, biodegradable packaging material is beneficial. In addition, close loop recycling is efficient and environment friendly compared to down cycling e.g., waste glass bottles can be melted and reshaped into a new bottle, but with waste plastic, on recycling it loses its strength due to the weakening and reduction in the number of bonds, and is unfit for food applications (Verghese, Lewis and Fitzpatrick, 2012). On the other hand, fiber-based products can be recycled up to six to twenty-five times (Eckhart, 2021). After glass, paper-based materials have a long recycling time which adds to the circular economy by reducing the amount of waste.

Papers and boards are coated with varnishes (for e.g., with wax, fossil, or biobased materials) to attain the barrier property. The presence of some coatings like fossil based can hinder biodegradability. Yet, there are some compostable coatings in the market that in addition to improving the performance of the fiber-based products, are also biodegradable, compostable, and commercially viable.

Beeswax is one such example that is a functional lipid and protein-based coating that degrades fast (5-6 weeks) and comparably is good for food applications. Plant based Carnauba wax is also another example that provides good water barrier. Nanoclay coatings have a good scope of applications. Through forming a complex network, it hinders the penetration of molecules into the material. They appear to have improved the anti-bacterial property (Kathuria and Zhang, 2022).

Generally, where possible, the non-recyclable products are separated, and then they are taken to a landfill or for energy recovery. This disposal expense is a major problem, particularly for grocery chains who profit from the sale of old corrugated containers (OCC) and incur losses when wax items are transported to landfills or incinerators, which can amount to up to $20 million annually in some situations (A. Signoretti, 2022). However, it mostly depends on the nations under consideration. In most of the EU, if recycling is not an option, they will be burned to recover energy. For instance, it is illegal to dispose of organic or flammable garbage in landfills in Sweden.

Product characteristics demand certain packaging requirements (Pålsson, 2018). Based on the company goals, trade-offs are mutually made to attain a desirable outcome. For example, a producer might want low-price packaging whereas a retail store may require expensive packaging for marketing purposes, and some brand owners compromise on the shelf-life to switch to sustainable packaging to replace plastic. Packaging cannot be completely avoided, because it plays such an important role from protecting certain product to even impacting the environment both positively and negatively, a balance between the amount and type of the material is required to reduce the overall impact.

Reducing single-use plastic is becoming a growing concern worldwide, leading many governments and organizations to implement regulations to curb its use. Here are some examples of regulations that have been put in place:

1. Plastic Bag Bans: Many countries and cities have implemented bans on plastic bags, encouraging people to bring their reusable bags when shopping.
2. Straw Bans: Plastic straws have also been targeted with bans in many areas, and alternatives such as paper or metal straws are being promoted. (Directive (EU) 2019/904)
3. Bottle Deposit Schemes: Governments have introduced deposit schemes on plastic bottles, encouraging consumers to return them for recycling to reduce litter.
4. Plastic Tax: Some countries have introduced taxes on single-use plastics to discourage their use and encourage manufacturers to move towards more sustainable alternatives.
5. Packaging Regulations: Governments and companies are starting to introduce new regulations on packaging to reduce plastic waste, such as requiring a minimum percentage of recycled content in packaging. The EU is currently heavily revising Directive 94/62/EC on packaging and packaging waste and its amendments.

These regulations are just some of the many steps being taken to reduce the amount of single-use plastic that ends up in the environment. With increasing awareness of the damage that plastic waste can cause, we can expect to see more regulations and initiatives in the future.

2.2 Applications:

BOC is targeting several markets such as food, fresh drinks, liquor, home care, and cosmetics. For the closures we deal with in this project, there is no well-defined application yet. Nevertheless, the varnishes tested in this project are food safe and can be used for food applications. However, for products with high demand on the barrier properties, a thorough investigation and further shelf-study is required and recommended. And in some cases, the application of can come with trade-offs like lower shelf-life.

2.3 Characteristics of the cellulose fiber-based lid

Cellulose is a plentiful biopolymer that is frequently in paper goods. Numerous surface hydroxyl (OH) groups present in cellulose quickly form hydrogen bonds with water molecules, allowing water to diffuse across the surface. Additionally, cellulose can absorb water. In other words, cellulose is a naturally hygroscopic and hydrophilic substance. Water CAs observed on cellulose films with a flat surface range from 17° to 47°. Paper and cotton fabrics' rough and porous surface structures facilitate water dispersion and absorption via capillary action between the cellulose fibers. Paper-based microfluidic systems, for example, use capillary driven liquid transportation.

The fiber-based closure at BOC is made from cellulose fiber reels that are directly heat pressed under high pressure. The material used in this project is untreated and hence the need for post treatment of varnishing to attain the barrier property. Nevertheless, to attain the barrier property by varnishing, the characteristics of the formed closures affect the coating, like the surface energy.

A material's surface energy value can be used to gauge a substrate's susceptibility to wetting by liquids. Other factors that affect adhesion are density, cellulosic structure, basic compounds of wood, texture etc. In other words, the chemistry of the substrate plays a major role in the adhesion of the varnish on the substrate (Ghofrani, Mirkhandouzi and Ashori, 2016).

2.4 Varnish/Coatings

Varnish is any product that offers a protective coating while still allowing the substrate (such as the fiber closures) to be visible. Depending on the manufacturing processes utilized and the needed varnish quality, varnishes have quite complicated chemical compositions and structures (Butcher, 1992). In our experiments, all the varnishes used for the testing are water-based, food-grade and certified for food applications according to the supplier specifications.

Some of the parameters that affect the coating process are:

- Dry matter content
- Temperature
- Viscosity

- Substrate roughness/ smoothness
- Coat weight (dry or wet)
- Number of layers
- Homogeneity
- Drying temperature

Coating/Varnishing is a very well-known process in the paper industry. One of the issues faced by substrates is fouling which occurson adsorption or deposition of particles, colloids, or salts. Especially, when these products are going to encounter food products, it is important that they have a good barrier property and high contact angle to ensure hydrophobicity and anti-fouling property (Hebbar, Isloor and Ismail (2017).

Recyclability of the coated can be challenging in some cases. Nevertheless, according to Heldman, Lund and Sabliov (2018) advances in the polymer coatings have made it possible to coat the fiber-based products leading to a more recyclable packaging.

2.5 Water-based coatings

In the coming ten years, extrusion polymers will remain the dominant category of functional and barrier coatings. The next two most used raw material categories are wax and aluminium, both of which will continue to be in high demand. Alternatives for these product categories will nevertheless continue to gain market share for a variety of reasons, but mostly because of concerns about environmental sustainability and a desire for biodegradable packaging materials from both the public and the government.

Water-based coatings will experience the quickest growth, with coatings made with natural binders and cutting-edge emulsion polymers that are combined with structured pigments setting the standard for future developments. More durable packaging will also be demanded for certain demanding applications.

Water based coatings often are polymers emulsion and can contain additional materials such as pigments. In applications like corrugated boards and replaced plastic materials (like paper-based alternatives), water-based coatings are taking over and have selectively replaced some unattractive materials and appears to have the quickest growth in functional and barrier coating sector and is economic and sustainable alternative to other technologies like silicones, wax, fluorochemicals etc

Some of the common water basic coatings are styrene-butadiene, acrylic styrene and PVdC emulsion polymers. The wax, PE based coating are replaced by water-based emulsions of wax and PE and in North America some water-based coatings are aimed to replace silicones, wax, polyethylene, and fluorochemicals, and appear to be expanding at a rate of about 15% annually. Many packaging companies are slowly turning to water-based solutions to address packaging difficulties. When compared to other options, water-based solutions offer greater processing flexibility and can be applied using various techniques. These compounds can commonly be applied on and off machines using rod, air knife, and blade coating techniques, in addition to spray, size press, and other processes. After application, some coating types frequently need to dry and cure, which occasionally presents challenges (A. Signoretti, 2022).

2.6 Hydrophilicity/hydrophobicity analysis

When a drop profile contacts a surface, an imbalanced secondary force of interaction causes water molecules to bind to it. The contact angle (q), which analyzes this interaction, is measured and its value is connected to the materials' surface energies. It is widely acknowledged that a surface is hydrophilic if its contact angle with water is less than 90 degrees .

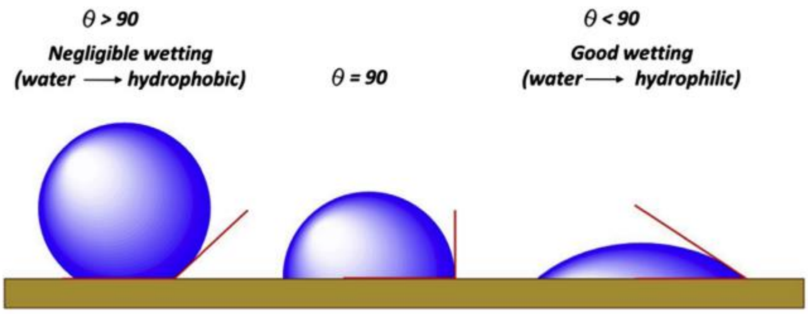

Figure 2 Contact angle measurement and their implications (Hebbar, Isloor, and Ismail, 2017).

Hydrophilic refers to a surface's propensity to become damp or to produce a thin layer of hydration on it. The nonwetting property of the surface or showing less affinity toward the liquid is represented by the value q > 90 degrees (see Figure 2). These surfaces are referred to as hydrophobic. Because there is no attraction between the water molecules and the hydrophobic solid surfaces, which are "water rejecting," water drops tend to form "beads." Superhydrophobic surfaces are those where the water contact angle is more than 140 degrees (Teisala, Tuominen and Kuusipalo, 2013).

2.6.1 Contact Angle

Contact angle is the angle created when the liquid-vapor and liquid-solid interfaces cross, as measured geometrically by drawing a tangent line from the contact point across the droplet's liquid-vapor interface. Surface roughness can lead to contact angle hysteresis. To get an average number that is indicative of the entire surface, contact angles should be measured several times on a relatively large substrate.

For accurate contact angle measurements Li and Neumann (1992) state a smooth surface of the substrate is required. Variations in the contact point of the three-phase contact line can also be caused by surface heterogeneity or roughness of a substrate. The dependency of the contact angle on the drop profile also leads to a systematic problem. Despite all these drawbacks, the sessile drop technique is believed to be the most effective method.

The surface energy for the solid material can be determined by measuring the contact angle between a solid surface and a droplet of liquid on the surface. A lower contact angle value indicates that a material is hydrophilic, or that water molecules have a strong affinity for the substrate. Due to the presence of active polar functional groups, substances that are referred to as hydrophilic easily adsorb water molecules. The surface's hydrophobic properties are shown by the increased contact angle, ones with this property, known as hydrophobicity, react to water in the exact opposite way to hydrophilic ones. Water tends to "bead" on the surfaces of hydrophobic materials, which are "water-hating" because they have little or no tendency to interact with water. (Hebbar, Isloor and Ismail, 2017).

2.7 Definitions of coating techniques

2.7.1 Spray coating

Spray coating is a process that is broadly used in many industries. With a substrate held vertically or slightly angled, the water-based varnishes are distributed over the surface by spraying.

At lab-scale, the spraying is done using a spray gun (see Figure 3(A)) and the time required for one layer to dry, and form is 1 minute approximately when dried at 105°C in an oven. The kinetics at industrial scale depend on coat weight, speeds, temperature, length of the oven. Spray drying is very feasible for flat surfaces; however, the challenge is spraying on the uneven surface like closures with threads on the inside which creates complications in coating every corner of the closure both inside and outside. Another disadvantage is that the consumption of the varnish in spraying is high and most of the varnish while spraying is lost in the air and majority of the content is lost through drainage and become useless afterward.

Some of the parameters that influence the spraying are as follows:

- The flow of the solutions
- Spray distance
- Polyelectrolyte ratio in solution
- Ionic strength and pH
- Temperature

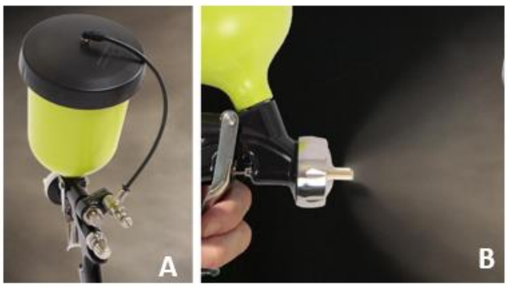

Figure 3 (A) Sames Kremlin Spray gun (B) Flow of spray splat from the nozzle of the spray gun.

As mentioned, the spray pattern is affected by the spray distance, speed, number of passes, the size of the spout/nozzle and the angle. The high velocity or the pressure of the spray and the size of the droplets plays a role in creating the interlock between the particles and forming a layer or coating on the substate. The emission from the spray gun is called splat (see Figure 3(B)), the splat with the help of air pressure is sprayed from the nozzle. When the spraying process proceeds and the particles strike the substrate, they form bonds with the substrate and provide a consistent coating with high bonding strength that is almost porosity free. Therefore, air pressure is crucial in coating and the nozzle aids in establishing a high-speed flow stream (Fauchais, 2016)

2.7.2 Dip Coating

Dip coating as the name suggests is a process of depositing any material (polyelectrolyte solution, water-based coatings etc.) on the substrate by dipping to form a coating (Scriven, 1988). The basic flow is constant, the rivalry between the forces of gravity, capillary (surface tension), and viscous force determines the thickness of the film (Scriven, 1988). After dipping, the lid is kept in the oven for drying and film formation where the water evaporates, and the particles coalesce and then cooled by keeping it at room temperature. Depending on the chemistry of the product, the minimum film formation temperature (MFFT) can vary from as low as ambient temperature to as relatively high as 105°C for a faster drying process.

In dip coating, one of the important aspects is the thickness of the film deposited on the substrate. The film is fundamental to numerous physical, chemical properties and applications. The number of factors that influence the deposited film are dip-coating time, withdrawal speed, the concentration of the coating solution, composition (Buhl et al., 2020).

Although dipping is an easy process for any product but is not economical since it can consume a lot of varnishing material and energy due to the longer periods of drying in the oven and the process can be time-consuming as well. It is significantly less frequently employed in precision coating manufacturing outside of product R&D labs than a few premetered coating techniques (Scriven, 1988).

2.7.3 Brush Coating:

Figure 4 Brush coating technique

As seen in Figure 4, a paint brush was used to apply the emulsion on the lid. This technique was used to simply test the performance of the varnishes for their barrier properties through visual observation, water contact angle measurements and penetration time. It is not a very commonly used technique in industries. Brush coating method was adopted when the spray gun showed some technical issues and to efficiently utilize the available time. Meanwhile, a new spraying equipment was being arranged.

2.8 Repulpability

In the current scenario, the circular economy is gaining importance.

Circular economy definition EU

"A production and consumption model which involves reusing, repairing, refurbishing, and recycling existing materials and products to keep materials within the economy wherever possible. A circular economy implies that waste will itself become a resource, consequently minimizing the actual amount of waste. It is generally opposed to a traditional, linear economic model, which is based on a 'take-make-consume-throw away' pattern." (European Parliament, 2015)

Recyclable materials are those that can be gathered, disassembled, shrunk in size, or processed before being used again as raw materials or in the creation of new products (Verghese, Lewis and Fitzpatrick, 2012). For paper and board recycling, repulpability is a process where the product is broken down into smaller parts using a blender to check if the product can return to its original state which is pulp. In this test, we can check the yield and this data helps us attain the percentage of recyclability.

Repulpability and recyclability are affected by the presence of adhesives, minerals, pigments, inks, and coatings. Separation of fibrous and non-fibrous material is critical for achieving high circularity. Recycling wax-coated materials, as well as some laminated papers and paperboards, can be challenging. And some wax coatings limit the recyclability of corrugated boxes which end up in incineration. They become hard to recycle and recover.

In accordance with the terms of Directive (EU) 2018/852 of the European Parliament and of the Council of May 30, 2018, modifying Directive 94/62/EC on packaging and packaging waste, all packaging that is placed on the market must be accompanied by a Certificate of Conformity.

The latter has suggested several standards (EN 13427 - July 2004) to be applied to check the compliance of the packaging to meet the Directive's essential requirements:

- Prevention via source reduction requirements relevant to manufacturing and composition (EN 13428)

- Packaging recoverable through material recycling requirements (EN 13430)

- Requirements for packaging that can be recovered as energy, including a minimum inferior calorific value specification (EN 13431).

- Packaging recovery through composting and biodegradation requirements (EN 13432)

European Directive 2018/851/EC (amending Directive 2008/98/EC) states that the waste hierarchy shall be used as a priority order in waste prevention and management legislation and policy:

Eco-designing for prevention, getting things ready for reuse and recycling (which includes composting), other types of recovery like energy recovery, and disposal.

When determining if a product can be recycled, the following two aspects must be considered:

1 To ensure that in the final packaging design, a specific percentage of the packaging material can be designated as recyclable. To be considered a secondary material for the paper industry, packaging must include at least 50% paper and board.

2 The cautious choice of raw materials used in manufacturing operations to protect recycling procedures. To put it another way, packaging components must be compatible with recycling methods that are widely used, practical, and financially viable. To validate this component, laboratory tests are used. Recycling Study Report (2023).

3.Methodology

The project plan was subject to modifications from the beginning due to an explorative approach and novelty of the project. Some changes had to be made during the process due to unavoidable circumstances like the long lead time to arrange the materials. Additional coating techniques like brush and dip coating were used to test the performance of the coating to efficiently utilize the time available. The forecasted plan, actual plan and timeline for the project are presented in APPENDIX A. The project involved traveling to Saffle for varnishing trials, performing experiments at Karlstad University, and measuring water contact angle at Karlstad main office.

3.1 Materials Used

Cellulose Fiber-based Lids (see Figure 5)
Sames Kremlin SAS Spray gun
Varnish (details mentioned in the Table 2)
Paint Brush
Spraying equipment from Spraying Systems
Precision Weighing Balance (Fisherbrand – Moisture series)
Binder FD53 E2 Drying oven.
Contact angle Goniometer (Ossila)
Lab equipment and facility at Karlstad University

- MESSMER Disintegrator MK III C
- Mettler PM3000 weighing balance.
- Defibrator
- MESSMER BUCHEL Handsheet former
- Lorentzen & Wettre sheet press
- Saffle Verkstads Somerville screening equipment with 40μm mesh.
- Memmert heating/drying oven

The closures (with determined surface area of 0.00739 m^2 and the moisture content of 5.5%) were produced using the existing energy-efficient vacuum press forming BOC machinery at Säffle. The material is made from fiber reels, is hydrophilic, hence the need to coat the material to attain hydrophobicity. The coating process is carried out after the formation of the closures.

The varnishes were arranged by BOC from different suppliers. The varnishes were coated keeping their original form (solid content and viscosity) and diluted for some trials to reduce the viscosity. This was done to evaluate the performance of diluted varnishes, because the varnishes with high viscosity not only took up too much coat weight but also took longer time to dry.

Figure 5 Cellulose fiber-based closures

Table 2 Technical details of varnishes

Sl. No	Code	Description	pH	Viscosity	Dry content (%)
1	15-617040-9	Water-based overprint varnish for printing on top of water-based Flexo printing inks	7.5-9	25” – 35”	44
2	15-619763-4	Water-Based COBB lacquer	8 - 8.6	25”-35”	44
3	15-619798-0	Water-based overprint varnish for printing on top of water-based Flexo printing inks	7.0 - 8.0	20”-30”	30
4	10-609684-5	Water based sealable barrier coating to be applied on fibre-based substrates.		45 ± 5 sec/DIN 4mm	25
5	9455	coating based on vegetable waxes.	8.0 - 10.0	< 500 mPas	40 – 42
6	9466	coating based on a co-polymer dispersion and sustainable vegetable wax emulsion	7.5 - 9.5	500 - 2500 mPas	40 – 42
7	76525-1	NIL	NIL	NIL	20-25
8	76525-2	NIL	NIL	NIL	35-37
9	S-3136	Sucrose esters of fatty acids (UVCB, multi-constituent substance or complex mixture)	8.0 - 10.0	NIL	14-16

3.2 Varnishing Process

Each lid was given a code for identification. After which, the initial weight of the lid was noted using the precision weighing balance, followed by the application of coating using one of the technologies. After applying the varnish, the final weight of the lid was noted and then oven dried at 105 ± 5°C for a time of 1-2 minutes approximately. The initial and final weight is used to calculate the coat weight for the coated lid (see Equation 1). The varnishes were applied at room temperature and humidity which is approximately 20°C and 40-60% respectively.

Generally, the coat weight was calculated immediately after every coating. The value helped to assume the quantity for the next sample to be coated due to manual coating process.

Equation 1 Dry Coat weight

$$\text{Dry Coat weight} = \frac{(final\ weight - initial\ weight)}{surface\ area\ of\ sample} X\ dry\ content\ \%$$

For end applications, the entire surface of the lid needs to be coated. However, in our project, only the outer surface of the closure is coated as the aim is to evaluate the performance of the coating material. Additionally, since the inner and outer surface of the lid is plain, without any threads on the inside, it can be assumed that the coating would be similar on the inner surface as well. It is easier to calculate the coat weight of the outer surface only rather than the entire surface. Lastly, this process prevents material waste and helps in maintaining the same surface area for all the coating techniques. Therefore, it appears reasonable to only coat the top surface rather than the whole lid for efficiency and to reduce material waste.

For the first screening step, the samples were coated exploratively and randomly with brush and dip, except for varnish 1 which was coated using spray gun and brush. For the second screening step, the samples that showed the best results in the first screening step were coated using the new spraying equipment from Spraying Systems.

3.3. Coating Techniques

3.3.1 Brush Coating

The brush was dipped in the varnish in the ceramic bowl, careful consideration was taken to only take the required quantity of varnish on the brush for efficiency. The brush dipped in varnish was spread manually on the top surface of the lid as shown in Figure 6. The amount of varnish per lid varied from trial to trial because based on the prior testing, assumptions were made to apply less or more quantity.

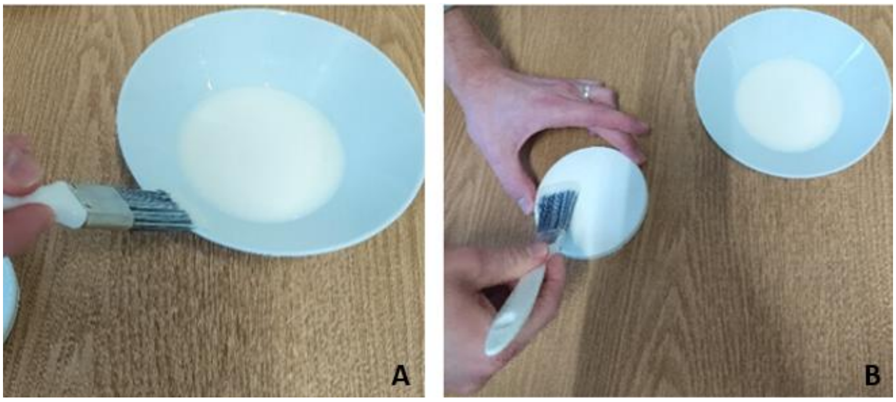

Figure 6 Brush coating: (A) Dipping into the varnish (B) painting on the lid.

3.3.2 Dip Coating

Unlike the general dip coating process where the substrate is completely dipped into the solution, only the outside surface of the lid is dipped (as seen in Figure 7). Since the purpose is to analyze the barrier property, it is reasonable to only coat one side as coating the entire surface of the lid would only lead to waste of the varnish.

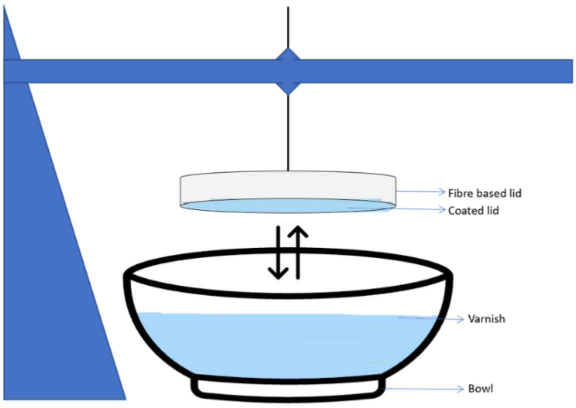

Figure 7 Schematic diagram of Dip coating

3.3.3 Spray coating

The lids were placed on the sample platform in the spray booth. The samples were sprayed using the Sames Kremlin SAS spray gun. Initially, the lid was placed horizontally as shown in Figure 8(A) and it was observed that the coating was not uniform. The lid was then assembled diagonally and sprayed with the help of a wooden cork behind to support the lid (see Figure 8).

The spray required an air compressor. Air pressure of 0 – 1 bar was used to create the splat of the varnish. The flow of liquid from the nozzle was regulated with a knob below the varnish cup to get a desired coating splat. It either created an eye shape spray format or a circular flow spray.

In some trials, the varnishes were diluted with water to reduce the viscosity, because highly viscous varnishes are difficult to spray due to small size of the nozzle. Additionally, to attain a low coat weight and to evaluate the performance with reduced viscosity, some varnishes were diluted.

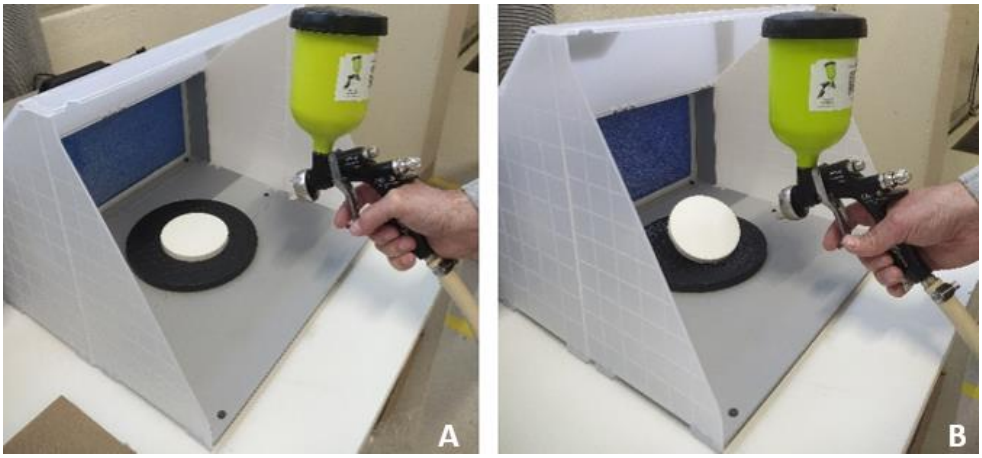

Figure 8 Spray coating (A) Horizontal placement of the lid (B) Diagonal placement of lid.

3.4 Setting up the new spraying equipment:

The schematic diagram of the new spraying system is presented in Figure 9. The different parts of the equipment arrived around the second week of April, and it took almost a week to set up the new equipment. The setting up took a long time because the manual for setting up the equipment was not sent by the supplier. The setting up was done via video call with the vendor, and with the help of Christer and Fredrik at BOC production facility. Some parts were missing which the vendor on notification arranged to dispatch to BOC a week or two later. The nozzles that were available are presented in Figure 10.

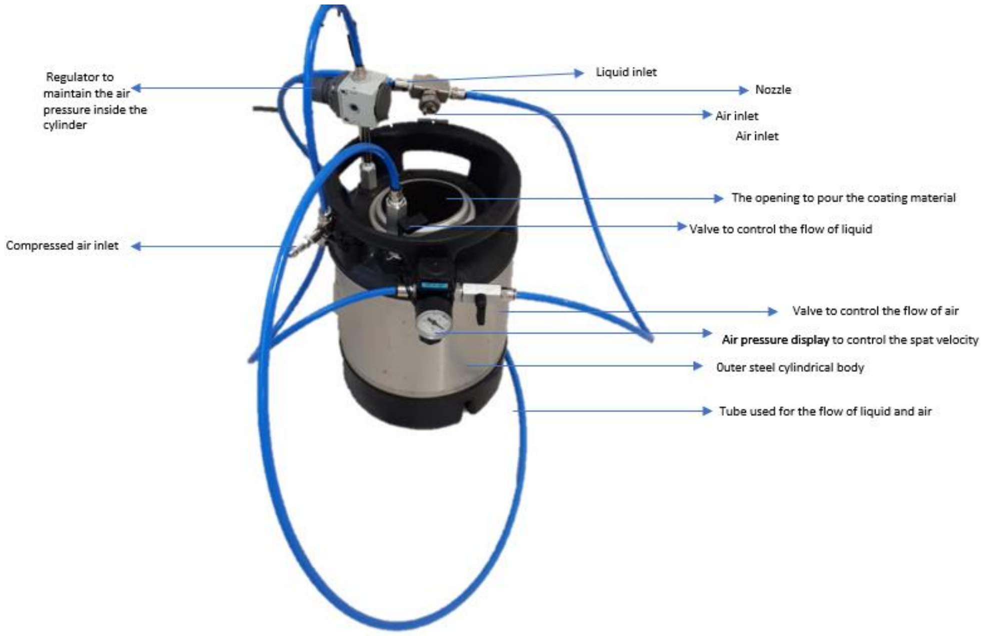

Figure 9 Schematic diagram of the new spraying system

Figure 10 Nozzles (2850, 2050, 1650)

After the equipment was set up, nozzle number 2850 was used to apply the coating with the selected varnishes. The air pressure was maintained between 0.2 – 1 bar. The flow of the liquid and air was regulated with the liquid and air valve respectively. The trials were conducted outdoors (see APPENDIX E) due to the absence of an evacuation or ventilation in the production unit.

3.5 Visual Appearance

The appearance of the lid during and after coating was observed. Some of the variables observed on encountering the varnish are the change in texture (like roughness/unevenness (see APPENDIX B), color like browning, crystal formation etc., and uniformity of the coating. The coated lids were observed for water barrier properties by dropping a few droplets of water with brushes spontaneously after drying the coated lid.

3.6 Contact angle measurement.

The contact angle was measured at Karlstad Office using a goniometer (Ossila, product code: L2004A). The equipment was calibrated using Ossila software version 4.

The frame rate (the number of pictures captured per second) was set to 20 frames per second. The recording time was 10 seconds. 1µl of tap water was deposited on the substrate using a variable volume micropipette (C2001) (ISO13485 CE) and the results were displayed on the monitor(See Figure 11). Random three spots on the surface were chosen to place the droplet on indicative of the entire surface.

In the panel display on the right side is the right angle, left angle, and average contact angle measurements. To attain the accurate contact angle, it is necessary to select the area where the droplet falls within the frame and the error is less than 1. The measurements of the same were noted on a separate Excel sheet (see APPENDIX C).

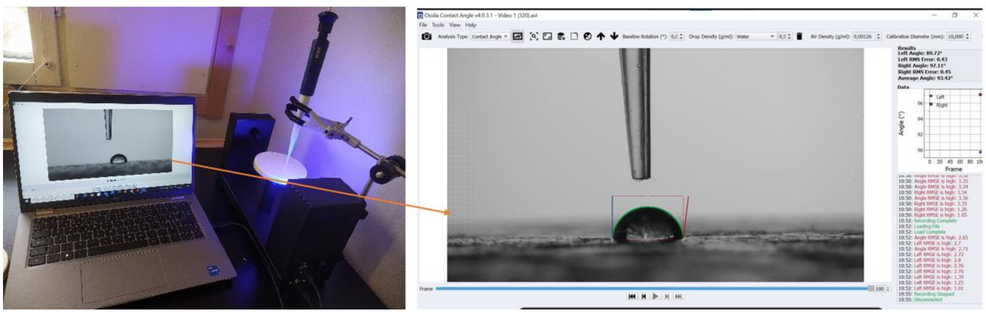

Figure 11 Contact angle equipment and the measurement set up.

3.7 Repulpability

The protocol for evaluating the repulpability for fiber-based packaging set by 4evergreen, (2022) and Leberle, (2022) was followed partially due to the availability of only certain equipment and screens. The repulpability of coated and uncoated lids was conducted in the lab and facility at Karlstad University. The procedure for repulpability trials is as follows:

3.7.1 Sample preparation:

The samples were cut into 3x2 ± 5 cm approximately in size. 50 ± 1g dry weight of sample was used. No prewetting or soaking was done. The coated samples used had a dry coat weight of 12.9 g/m^2 (see APPENDIX H).

3.7.2 Method:

3.7.2.1 Disintegration/ Defibrator

The samples were disintegrated using the disintegrator diluting with tap water at 40 ± 1% °C, (pH alkaline 7-8). The total volume of the sample and water was approximately 2000g to achieve a stock consistency of 2.5%. Disintegration time was done at 3000rpm for 10 and 20 min.

3.7.2.2 Screening of reject material/ strainer

Somerville fractionator was equipped with a perforated plate 40µm pore mesh and the deionized water flow set at 8.6 ± 0.2 l/min. Approximately 2.5cm of water was maintained to pour and mix the total repulped stock in the equipment. The screener was stopped until no more fibers fell on the screen wire. The rejected material was left above the screen and the accepted material passed through the screening equipment and collected separately from the outlet.

3.7.2.3 Sheet Formation

The handsheets (6.624g of fiber per lid) were formed from the accepted material. The reject material was directly kept in the oven for drying after draining the extra water . The handsheets were formed using the handsheet former and sheet press. The formed handsheet was oven dried for approximately 10 min. The sheet adhesion and visual appearance was visually observed.

The following day, the formed handsheets from the accept material and reject were oven dried for approximately 3 hours at 105 ± 5℃ and cooled in a desiccator. The weight of the accept material and reject were noted after drying. The repulpability yield was calculated using the below formula. The result is expressed in percentage with respect to the starting sample. (See APPENDIX H for the pictures of instruments used for repulpability trials)

$$Repulpability\ Yield = \frac{Accept}{Accept + Reject}\ 100\ \%$$

3.8 Statistical Analysis

In the analysis, the values of factor effects based on the varnish type, for contact angle and penetration were determined through multiple variance analysis (ANOVA) using the JMP Pro 16 software. The graph builder tool from the same software was used to plot the scatter plot to visualize the measurements of the sample representative of each varnish along with the correlation coefficient and standard deviation. All statistical calculations were based on 5% significance level ($P \leq 0.05$) level. The average values of each varnish were compared by Student -t test at a 5% significance level ($P \leq 0.05$).

4.Results and Discussion

BOC started in 2021 and is one of the pioneers in the packaging field to work on developing fiber-based closures. Since it is a start-up, not all the required material were established. An experimental and explorative approach was adopted which required flexibility and new ideas to perform the experiments with the available resources for the progress of the project. A qualitative and quantitative approach to analyze the samples was required as well.

The next steps of the projects were performed continuously after discussion and confirmation with the company supervisor Caroline Locre who is heading the project addressed in this thesis and academic supervisor Anders Warell was kept informed about the progress consecutively.

The project began with the testing of the varnishes by applying them on the lids. The number of lids coated for each varnish (Table 3) varied because of the explorative approach. The composition and specifications varied from varnish to varnish, decisions had to be made instantaneously to continue or discontinue coating with the material that showed undesirable results. For instance, five lids were coated with varnish 3, because the varnish was light brown and further trials with this varnish were pointless, since the company required transparent coated closures. On the other hand, the number of samples for varnish 1 is the highest because it was the first trial, new to experience of the author and required many trials to understand the analysis completely.

It is noteworthy that the samples were analyzed only for water barrier performance. Barrier properties like gas and oil barriers are interesting but are out of scope for this study. Initially, some samples were randomly tested for oil barrier property, and it was observed that the oil on the substrate (in case of poor oil barrier property) led to lower contact angle measurements which affected the measurements of contact angle. Therefore, to maintain accuracy in the contact angle measurements for water droplets, samples were tested for water barrier properties only.

Table 3 Number of samples coated for each varnish.

Sl. No	Code	No. of samples coated
1	15-617040-9	23
2	15-619763-4	15
3	15-619798-0	5
4	10-609684-5	7
5	9455	12
6	9466	8
7	76525-1	2
8	76525-2	6
9	S-3136	8

4.1 Reproducibility of coat weight

Due to manual process of coating, it was difficult to achieve a constant coat weight for the samples. The different coat weights obtained can also be attributed to the composition and technical details like viscosity and solid content, that varied from varnish to varnish (Ghofrani, Mirkhandouzi, and Ashori, 2016). In addition, during the trials, some varnishes were diluted to reduce the amount of coat weight, which led to changes in the moisture content and viscosity of the varnish, it was difficult to presume to coat a certain way to attain the desired coat weight as moisture content affects the adhesion of the varnishes on the substrate (Sonmez, Budakci and Bayram, 2009). The techniques were such that it required assumptions and presumptions to dip or brush coat or spray coat a certain way to attain an approximate a similar coat weight. For e.g., the coat weight obtained from dip coating technique is higher compared to brush coating, and through spray coating, it was possible to attain both the least and maximum amount of coat weight. Therefore, the difference in coat weight can be atributed to the varnish compostion, coatig techniques, and manual coating process.

4.2. The coating techniques

4.2.1 Spray coating

Most of the samples for Varnish 1 were coated using the spray gun and exploratively with brush coating for few samples. The spray gun technique showed some challenges as below:

- The clogging of the nozzle required cleaning after every second spray.
- The airflow – high or low airflow caused the varnish accumulation towards the corners and thus good barrier properties were observed on the corners (see Figure 12), and it was difficult to control the airflow accurately.
- The spraying also caused mist in the air, which could harm health.
- The right distance between the spray and the lid was difficult to attain, due to manual handling.

Figure 12 Spray coating showing good barrier property at the corners only.

A new supplier for spraying equipment that was more functional and industrially applicable was contacted where the parameters like the pressure of the airflow and liquid could be regulated more accurately. Meanwhile, to continue evaluating the performance of the other varnishes, brush, and dip coating were used. The reason being, it helped understand the performance when one of the samples for varnish 1 was coated using brush coating (see Figure 17). Therefore, it was decided to use brush and dip coating for varnishes 2-9.

4.2.2 Dip Coating:

Generally, it was observed that dip coating took up too much coat weight. It was difficult to control the amount of coat weight applied on the lids. This could lead to a large amount of product waste not only due to over-coating but also due to the waste from the unused coating material. To reduce the over-coating, varnishes were diluted. Since the substrate is hydrophilic it absorbed the water from the varnish immediately and left a rough texture on the substrate which is undesirable. The diluted varnish (V3) with the relatively low dry content (20-25% approximately after dilution in comparison to 30% before dilution), on application, led to roughening the texture of the lid as seen in Figure 13. The highly porous structure of the fiber enables the hydroxyl group to interact with the diluted varnishes and readily absorb into the substrate (Ghofrani, Mirkhandouzi, and Ashori, 2016).

The drying time after dip coating was longer, and for some varnishes like V9, it created a wafer-like layer, which could be peeled off, leaving the substrate uncoated (see Figure 14). According to Tang and Yan, 2016 dip coating is affected by the high viscosity of the varnish and results in a thicker coating layer. It appears that varnish with high viscosity and low solid content such as 12% is not an ideal solution for dip coating.

Figure 13 Rough texture of the sample coated by V3.

Figure 14 The wafer formation observed in V9 after drying for 10 min due to over coat weight.

Another observation of the dip coating was that it led to crystallization during and after the drying of the sample (see Figure 15). This observation was for varnish 4. Although some of the dip-coated samples showed excellent barrier properties for instance in Figure 16, the texture, the appearance after drying is not desirable. The bonding process between the fibrous material and coat solution is still not established (Tang and Yan (2016)).

Figure 15 Crystallization after drying in V4.

Figure 16 Bubbly texture with excellent water barrier property after drying in V6.

4.2.3 Brush Coating:

Brush coating helped in understanding the performance of the coating better compared to dip coating. It was possible to reduce or increase the amount of coating if required by taking less/more quantity on the brush and coating very lightly with the brush without pressing too hard. Pressing too much on the substrate can cause the surface of the substrate to wear off. In some samples, the brush used for this process left lines on the surface (see Figure 17).

Each coated sample was evaluated for visual performance (in the next section) followed by statistical analysis to narrow down the number of varnishes to test the same using the new spraying equipment.

Figure 17 Lines on the substrate due to brush coating observed in varnish 1.

4.3 Visual Observations / Qualitative analysis

The samples that showed the best performance visually representative of each varnsih are presented in this section.

4.3.1 Appearance

In general, the samples show a difference in appearance after coating in terms of color, texture, and barrier properties. The measurements of the samples used for visual observation are presented in Table 4. The samples presented on the first row (V1, V2, V5, V6 and V8) in Figure 18 are the ones that showed the best result. The samples showed good barrier properties, transparent color, and a smooth texture.

The representative samples from varnish V3, V4, V7, and V9 showed undesirable results like a brown color of the varnish, crystallization, rough texture after coating and drying, and poor barrier property.

Temperature can lead to the formation of cracking during drying after coating especially when the coat weight is very high. A small angle is formed when the liquid is spread out as seen for V9. The constant forces at the border between solid, liquid, and vapor are the main factor determining the magnitude of the contact angle, therefore in theory, contact angle is a characteristic property of a certain surface in each environment (Hebbar, Isloor, and Ismail, 2017). This explains the difference in contact angle among the different samples.

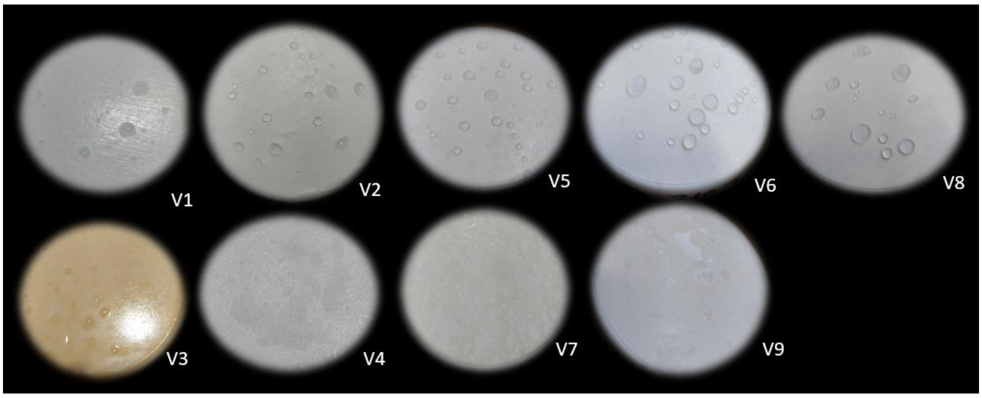

Figure 18 Acceptable results row 1, undesired results row 2

Table 4 Raw data of the samples selected from each varnish for the visual observation.

Varnish	Sample code	Coat weight (g/m2)	Penetration time (min)	Avg.Contact Angle (°)	Coating Technique
V 1	12419	26	53,67	87,06	Brush
V 2	20808	20	58,33	97,87	Brush
V 3	20818	48	65,33	81,57	Dip
V 4	20821	146	2	76,87	Dip
V 5	21008	21	58,33	95,177	Brush
V 6	21014	20	79,33	94,397	Brush
V 7	21021	36	2,33	55,89	Dip
V 8	21023	18,36	66	91,36	Brush
V 9	40308	49	2	42,5	Dip

V1, V2, V5, V6, and V8 will be tested with the new spraying equipment to select the final varnish. The next section presents the graph with the measurements obtained for the above samples for visualisation.

4.3.2 Visualization of samples for coat weight vs penetration time

According to the minimum criteria to select the varnish, our area of interest is in the top left corner of the graph, which represents lower coat weight and high penetration time. Figure 19 shows the measurements of penetration time and coat weight for the samples displayed in the previous section. Varnishes V1, V2, V5, V6, and V8 show the desired result of relatively low coat weight and high penetration time.

Although varnish 1 showed lines from the brush coating, it displayed very good barrier properties. Varnish 2, 5, 6, and 8 not only showed good barrier properties but also displayed a good overall appearance. It is noteworthy that V6 has the highest penetration time followed by V8. V8 has a lower coat weight compared to V6.

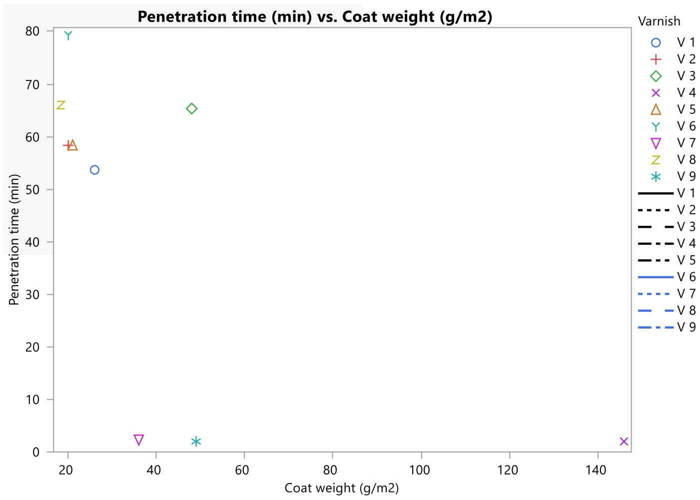

Figure 19 Visualization of samples with respective coat weight and penetration time

4.3.3. Visualization of samples for coat weight vs contact angle

The area of interest lies in the top left corner with lower coat weight and high contact angle (Figure 20). The varnishes that come in the interesting area are V1, V2, V5, V6, and V8. The reason for attaining the desired properties is attributed to the favorable composition of the varnish and the good adhesive strength of the varnish. Contact angle requires an even surface according to Youngs modulus equation. However, in our trials, we observe that due to uneven coating or crystal formation, the contact angle is either very high or it absorbs almost immediately due to very low surface energy (Kwok and Neumann, 1999). This is applicable for samples that were not selected due to uneven surfaces, rough texture, and crystallization.

Since both the sections 4.3.3 and 4.3.2 display the same varnishes V1, V2, V5, V6, and V8 and appear to display favorable results. It is interesting to test the selected varnishes with the new spraying equipment.

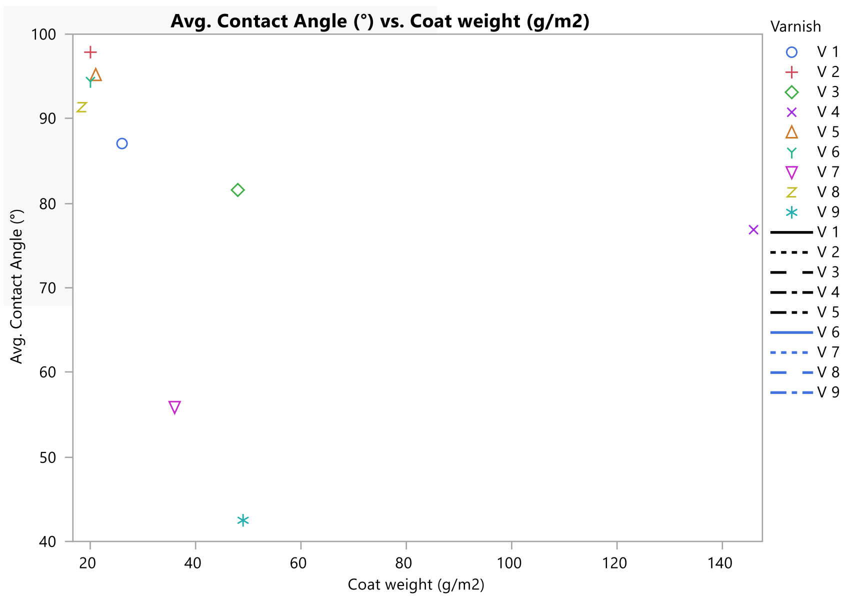

Figure 20 Visualization of samples with respective coat weight and contact angle

4.4 Quantitative analysis: the first screening

In this section, the effect of coat weight on the penetration time and contact angle are discussed. The varnish that falls in the range of (20 min and above penetration time and a coat weight of 20 g/m^2 and below) will be tested using the new spraying equipment. The samples are coated using the spray gun (for V1), brush coating (from V1-V9) and dip coating (from V2-V9).

The samples that showed penetration time of 2 min and below are not used for analysis. Since it is challenging to identify a tangent line when the liquid droplet profile is almost flat, this method's failure to measure more accurately at lower contact angle values (below 20 degrees) is another key shortcoming (Li and Neumann, 1992).

4.4.1 Mean Comparison of coat weight and penetration time

The scatter plot of mean coat weight and penetration time show variance. The penetration time is significant ($p<0.05$) for both samples within the varnish and among the different varnishes (see Table 5 and Table 6). The raw data of samples are presented in APPENDIX C.

The correlation coefficient between the coat weight and penetration is close to zero implying a weak correlation for almost all the varnishes except V4. Varnish 4 has a negative correlation coefficient.

It is observed that most of the values fall at a range of coat weight below 50 g/m^2, and the most value for penetration time is spread from 0 to 80 mins. And the preferred range or area of interest is in the top left corner of the graph. The varnishes V1, V2, V5, V6, and V8 of interest are boldened in Figure 21.

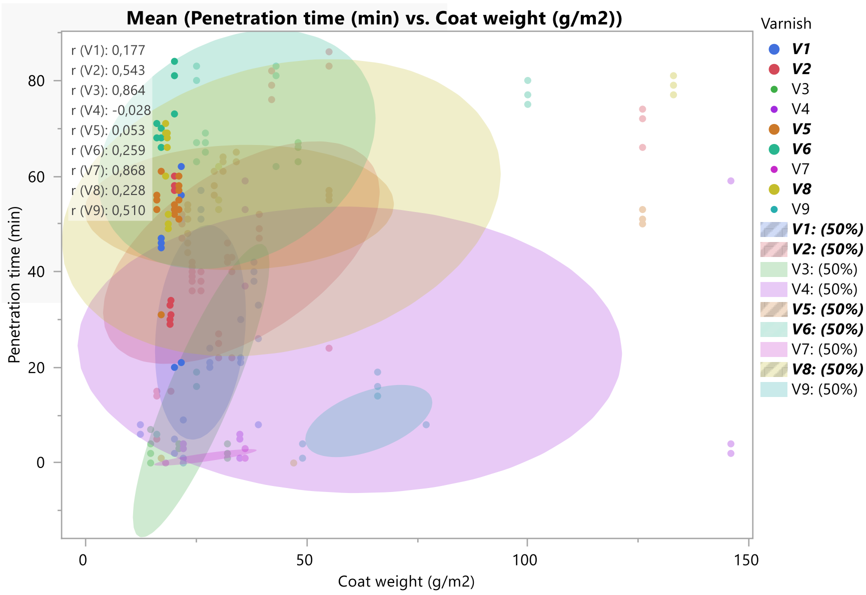

Figure 21 Scatter plot of mean penetration time and coat weight showing the weak correlation and the highlighted selected varnish (V1, V2, V5, V6 &V8)

Table 5 Analysis of Variance for penetration time by Sample.

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample code	63	112289,83	1782,38	14,9780	<,0001*
Error	128	15232,00	119,00		
C. Total	191	127521,83			

Table 6 Analysis of Variance for penetration time by Varnish

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Varnish	8	56256,21	7032,03	18,0572	<,0001*
Error	183	71265,62	389,43		
C. Total	191	127521,83			

4.4.2 Mean Comparison of coat weight and contact angle

The contact angle measurements are significant ($p<0.05$) for both samples within the varnish and among the different varnishes (see Table 7 and Table 8). It appears that a high contact angle with both a high coat weight and a low coat weight is achievable. V1, V2, V3, V5, V6 and V8 appear to meet the chosen minimum criteria. Although V3 has a high contact angle, the brown color of the varnish is not desirable.

Analyzing the contact angle of a liquid with a solid surface can measure its affinity. Contact angle values are sensitive to several factors, including heterogeneity, surface roughness, particle shape, and size. Accurate measurements, and reproducibility of contact angle values on actual surfaces are difficult to achieve. That explains the variance in Figure 22.

Generally, the substrate with higher roughness can also have a higher contact angle (high hydrophobicity) linked to the structure of the surface. In some samples, the coating led to the roughness of the texture and resulted in high contact angle which can be attributed to the high hydrophobic nature of the coating material.

The chemical makeup of the coating's composition supports achieving the desired characteristic and improves the substrate's barrier properties. According to Hebbar, Isloor, and Ismail (2017), the existence of polar functional groups in various varnishes is the cause of the difference in the contact angle.

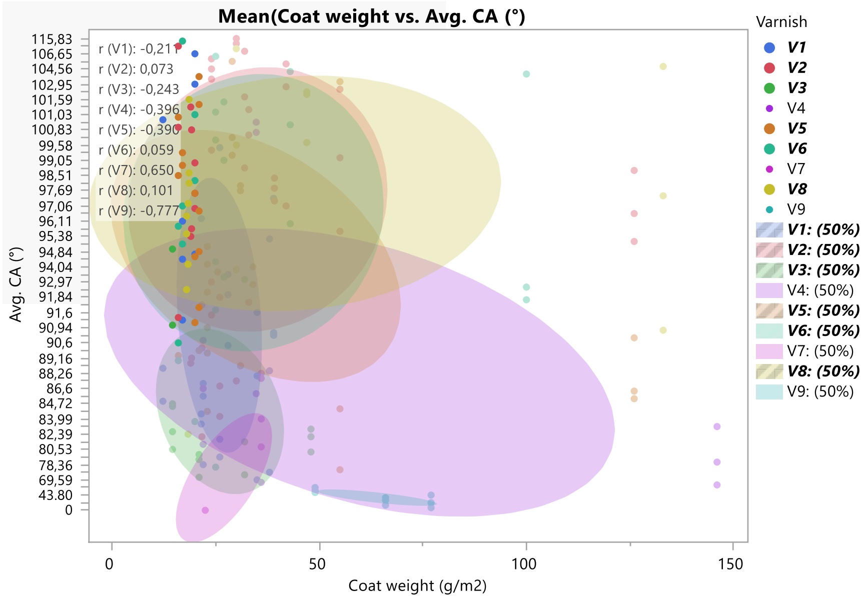

Figure 22 Scatter plot of mean avg. CA and coat weight showing the weak correlation and the highlighted varnishes that have good barrier properties.

Table 7 Analysis of Variance for contact angle by Sample

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample code	63	33029,361	524,276	6,8435	<,0001*
Error	128	9806,058	76,610		
C. Total	191	42835,419			

Table 8 Analysis of Variance for contact angle by Varnish

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Varnish	8	28404,659	3550,58	45,0258	<,0001*
Error	183	14430,760	78,86		
C. Total	191	42835,419			

4.5 Quantitative analysis: the second screening

The selected varnishes from the previous section were coated using the Spraying Systems equipment. Since the samples were already scrutinized for visual observation before the first screening step, the selected varnishes were directly analyzed quantitatively. Samples with a penetration time of 2 min and above were considered for further analysis. The raw data for the analysis are presented in APPENDIX F. The samples with below $20 g/m^2$ coat weight, above 90° contact angle and penetration time of 20min and above are screened. Among these, the most desirable, feasible and viable varnish will be selected. The graphs also present the standard deviation among the replicates per sample. The error bar is constructed using 1 standard deviation.

4.5.1 Mean Comparison of coat weight and penetration time

The penetration time presented in the graph is the mean of three replicates per sample representative of their respective varnishes. The observation shows a weak correlation between the coat weight and penetration time (see Figure 23).

V6 is observed to have the best combination of relatively low coat weight (14.3 and $20 g/m^2$) and low and high penetration time (23.3 and 82.7 min). Because of the good water barrier property, the penetration time is high implying good water resistance.

Changes in any characteristic, including heterogeneity, roughness, particle size, or surface chemistry, may affect how quickly all liquids penetrate a surface. It is crucial to note that the liquid's velocity of penetration has little bearing on the contact angle value but is closely associated with the kinetics. Surface roughness was shown to have a significant impact on liquid penetration kinetics but not on contact angle (Hebbar, Isloor, and Ismail, 2017).

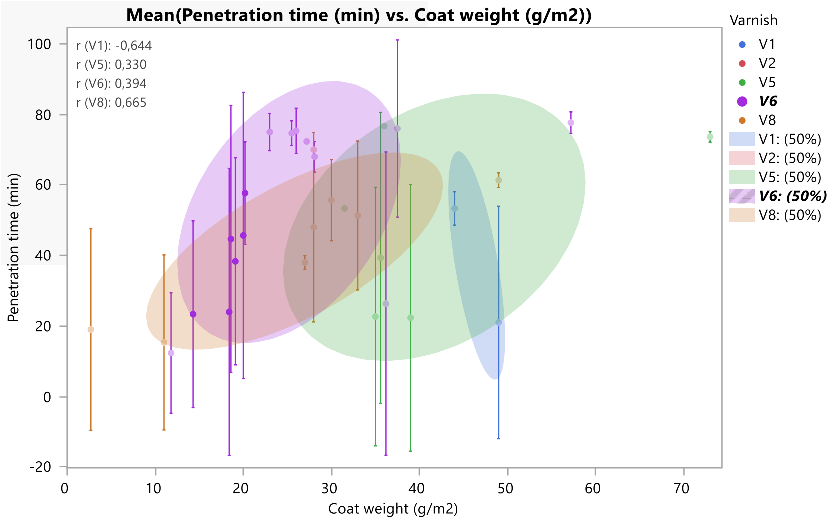

Figure 23 The scatter plot of mean coat weight and penetration time showing the selected varnish (V6)

The sample 50204 has comparatively low coat weight (14.3 g/m^2) but low penetration time (23.3 min). On the other hand, sample 50211 has a penetration time of 83 min with a coat weight of 20 g/m^2 (see Figure 24). Both the samples the showed good results belong to V6. The other samples that fall in the chosen minimum criteria (coat weight below 20 g/m^2 and CA above 90°) are presented in APPENDIX G.

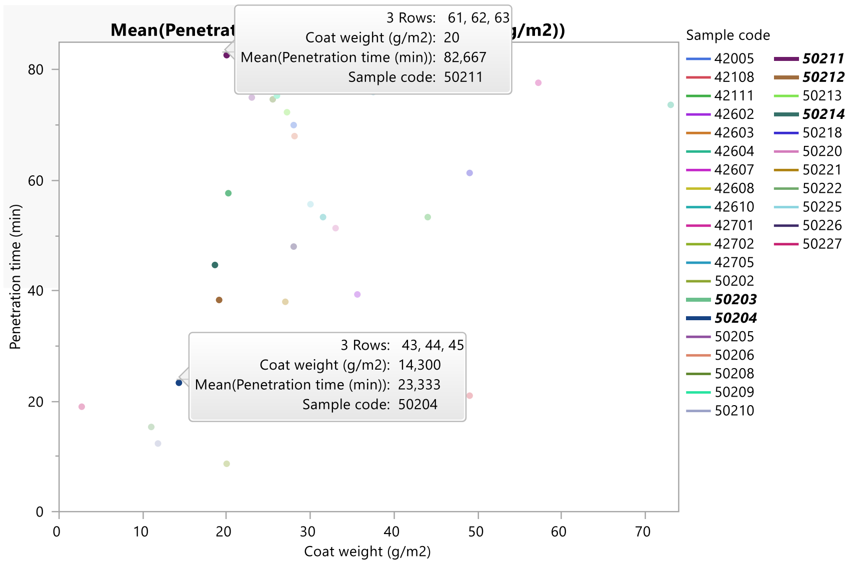

Figure 24 samples from the selected varnish and showed desirable results for penetration time and coat weight.

4.5.2 Mean comparison of coat weight and contact angle

The scatter plot presented in Figure 25 include the mean values of three replicates of contact angle and coat weight is constant per sample for different varnishes. The graph shows a weak correlation between the coat weight and contact angle measurements. The results show that varnish 6 and 8 have the desired measurements.

The result could be attributed to the composition of the varnish. The research conducted by Ghofrani et al 2016, stated that varnish type affected adhesion. Because of the good adhesion of the varnish on the substrate after drying, the substrate has a layer of coating on top of it, and the contact angle is impacted by the surface energy of this layer. The coating layer helped in reducing the surface energy of the substrate and hence to attain hydrophobicity.

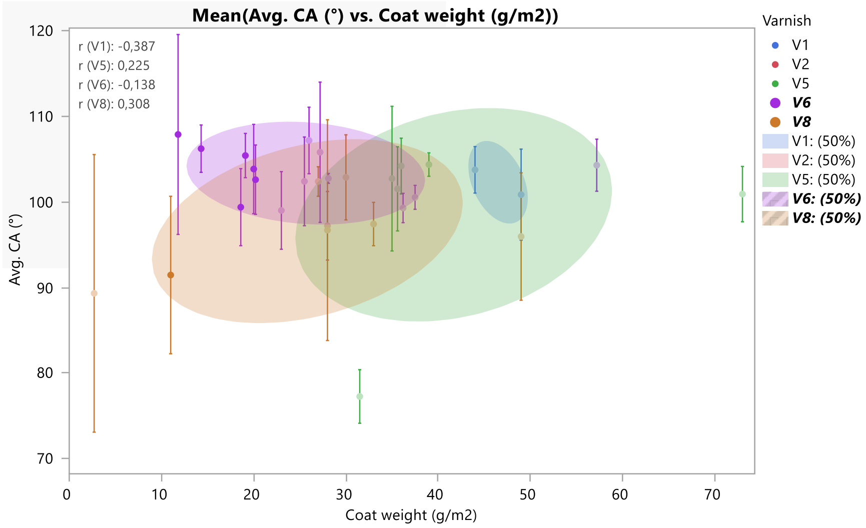

Figure 25 The scatter plot of coat weight and average contact angle shows the selected varnish (V6 &V8)

The two samples that are pinned in Figure 26 showed the best results. V6 has a coat weight of 11.8g/m^2 and higher contact angle of 107.8º. Whereas, V8 used 11g/m^2 coat weight and showed 91.5º contact angle. The other samples that fulfilled the chosen criteria (coat weight below 20 g/m^2 and CA above 90°) are boldened and presented in APPENDIX G.

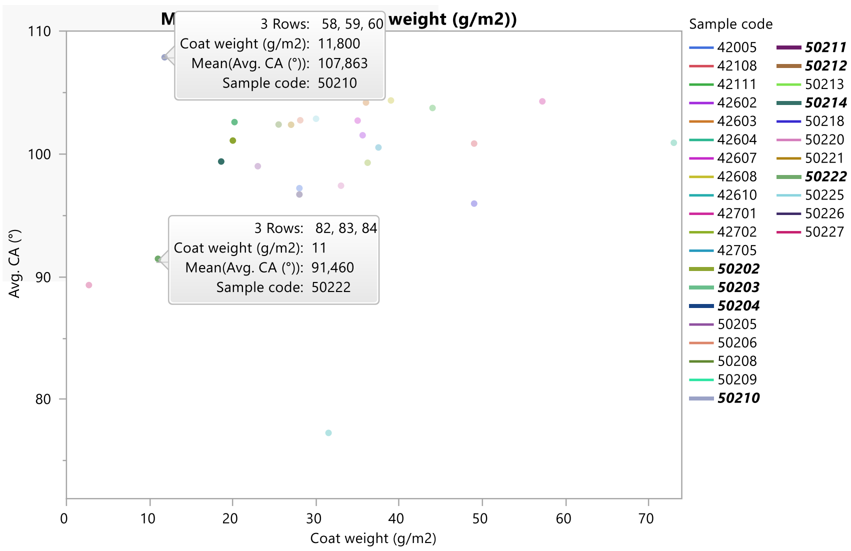

Figure 26 Samples from the selected varnish that showed desirable results for contact angle and coat weight.

4.6 Repulpability

The repulpability results showed a difference in the yield in coated and uncoated lids. It is noteworthy that the process flow was not completely followed according to 4evergreen alliance due to non-availability of the exact screens used in the screening equipment. The scorecard generated by the 4evergreen alliance is not completely comparable in this project. The implications of the repulpability yield are presented in Figure 27:

	YIELD	
	SCORE	DESCRIPTION
0-100 Suitable for Standard Mill recycling	100 - 90	The method indicates that the packaging is expected not to pose any repulpability issues in the standard mill and is therefore considered **Best in Class**.
	89 - 70	The method indicates that the packaging has **minor repulpability issues** that could have limited impact on the recyclability in the standard mill.

Figure 27 Implication of the yield score with 70% and above, adapted from 4evergreen (2017).

4.6.1 Repulpability of the uncoated lid

The yield for the uncoated lids is 86% for both 10 min and 20 min defibrillation. This can be attributed to the absence of coating or adhesives in the material. According to the scorecard generated by 4evergreen, 2022 description, the method indicates that the packaging has minor repulpability issues that could have a limited impact on the recyclability in the standard mill. It is noteworthy that this is applicable when the samples have been screened at 5mm and 150µm diameter holes. However, in this project, due to the non-availability of the later stated screens, the repulped samples were screened at 40µm which is three and a half times smaller than the original screen. Perhaps, it can be assumed that the uncoated lids can have a higher score, above 90% and can be declared recyclable in the best class.

The handsheets visual appearance can be seen in Figure 28. The adhesion was absent. Absence of adhesion means that the sheet was easily removed from the support and covered without any damage or breakage when removed from the oven after drying. The handsheets for 10 min repulped material are presented in APPENDIX H.

Figure 28 Handsheet of 20 min defibrillated accept material.

4.6.2 Repulpability of the coated lid

The coated lids had a mean coat weight of 12.9 g/m^2 (see APPENDIX H). The V6 coated lids repulped/defibrillated for 10 and 20 min had a yield percentage of 80.5 and 85.6% respectively. V6 coated lids are also recyclable. The specification shared by the supplier also says that varnish 6 is recyclable and is food safe. Additionally, since we used the same screen 40µm which is three and half times smaller than the second screen size of 150µm used in the actual process, it can be assumed that V6 coated lids can have a higher repulpability yield and is recyclable in the best class (see Figure 27).

The sheet adhesion for the handsheets was absent, which is good because there was no damage or breaking when the sheet when the sheet was simply separated from the support and gloss sheet (see Figure 29).

However, an interesting observation in the reject material is the color difference between the accept and reject material (see Figure 30). The reject material is slightly light blue compared to white handsheets. This could imply that most of the varnish during the screening stayed back in the tank with the rejected material. Further analysis needs to be done to investigate the recyclability of this material. Perhaps, how it would react once it ends up in the ocean. Will it form a film or degrade naturally?

Figure 29 Handsheet of 20 min defibrillated accept material for coated lids.

Figure 30 Light blue color of the reject material compared to accept material for coated lids.

4.7 Pros and Cons of the Technology

4.7.1 Spray gun coating

The equipment as shown in Figure 3 has a plastic body which makes it easy to handle on a lab scale due to its lightweight. However, accurate air pressure could not be regulated, and the size of the spraying particles could not be regulated because the equipment consisted of one nozzle only.

Table 9 Pros and cons of spray gun coating technology

Pros	Cons
1. Easy to handle. 2. Simple and easy to spray. 3. Suitable for lab-scale 4. Easy to clean – body design with smooth and polished walls.	1. Cannot regulate air pressure. 2. Only one nozzle 3. The nozzle kept clogging after every spray. 4. Cleaning after every spray 5. Difficulty in measuring the distance between the spray gun and the substrate. 6. Material waste 7. Mist in the air which can be harmful to human health

4.7.2 Brush coating

One interesting observation is that the brush used for coating has thick and hard bristles, made of plastic. Perhaps, a thinner and lighter material would help in attaining a better coating, with a smoother texture and uniform coating without affecting the fibers (like abrasion or wear off the fibers).

Brush coating is viable if one intends to quickly test the performance of a coating. However, for industrial viability, the process requires more research and development. Table 10 presents a summary of the pros and cons of brush coating.

Table 10 Pros and cons of brush coating

Pros	Cons
1. Easy to coat. 2. Possible to presume the amount of coat weight. 3. Less waste	1. Brush left a pattern in some samples. 2. Not industrially viable for BOC

4.7.3 Dip Coating

Table 11 presents the summary of pros and cons of dip coating.

Table 11 Pros and cons of dip coating.

Pros	Cons
1. Easy to coat. 2. Easy to attain barrier property due to thick layer of coating (for some varnishes).	1. Took up too much coat weight. 2. Crystallization after drying. 3. Wafer formation after drying in some varnishes. 4. Not industrially viable for BOC 5. Product waste

4.7.4 New Spraying Equipment

A study found out that measuring the temperature, velocity, or diameter of both the in-flight and spray jet particles was insufficient to predict the coating qualities. The flattening and solidification of each particle onto the substrate to form splats, the stacking of the splats to create the coating, and the actual contact of the splats with the substrate or earlier layers all affect the coating's properties (Hebbar, Isloor, and Ismail, 2017).

Compared to spray guns, the overspray could be regulated to a certain extent due to the air and liquid valves. It is noteworthy that the trials were coated with nozzle number (2580), the other nozzles (2050, 1650) have a smaller diameter. It is a possibility that the coating with the later nozzle would help meet the minimum criteria due to the smaller size of the spat. However, since the selected varnish is an acrylic polymer varnish, it is sticky and may result in clogging more often. One possible solution is diluting the varnish. This needs to be investigated by performing the experiments. Some of the pros and cons of the technology are presented in Table 12

Table 12 Pros and cons of using the new spray coating equipment.

Pros	Complications
1. Low coat weight is easy to attain. 2. Coat weight can be maintained to a certain range by layering. 3. Air pressure can be regulated. 4. The remaining varnish in the container can be emptied easily. 5. Various nozzles – Provide the option for different spraying diameters. 6. The flow of liquid and air pressure can be controlled. 7. Suitable for lab-scale coating	1. Since the experiment was carried out outdoors, the external environment affected the flow of the spray spat. 2. Due to wind, led to uneven coating. 3. To be tested for industrial viability 4. Cleaning requirements

5.Conclusion

Nine water-based varnishes were evaluated qualitatively and quantitatively for water barrier performance. The pros and cons of the four coating technologies spraying, dip, and brush coating were investigated.

The spraying equipment from Spraying Systems appears to be the most suitable because it is possible to attain very low coat weight, the air pressure and flow of the liquid can be regulated as per the requirement. The equipment consists of several nozzles of varying diameters allowing different spraying diameters.

The varnishes that were screened off through qualitative and quantitative analysis matched. The varnishes were selected through two screening steps. First, through visual appearance and quantitative analysis, five varnishes namely (V1, V2, V5, V6, and V8) were selected. The selected varnishes were tested using the new spraying equipment from Spraying Systems.

In the second screening step, V6 showed relatively low coat weight (11,8 and 20 and 14,3 g/m^2) with high contact angle measurements (107.9º) and relatively long penetration time (82.6 and 23.3 min) respectively. V8 while using a lower coat weight (11g/m^2) showed contact angle measurements (91,46 º).

Repulpability of the uncoated and V6 coated lids showed a yield of 86% and 80% at 10 min defibrillation respectively. For 20 min defibrillation, the uncoated and coated lids showed a yield of 86% and 85.6% respectively. Therefore, samples coated with varnish 6 are recyclable as stated in the specification supplied by the supplier.

6.Future Recommendations

Since varnish 8 also gave a reasonably good result, it is interesting to check the repulpability of the same for future applications.

Cobb tests were performed but due to time constraints, the results could not be presented. However, the cobb tester results would also be beneficial for further analysis of barrier properties.

The second spraying equipment has five different nozzles with different diameter holes. It is interesting to test the varnishes with different nozzles to check the spat flow and coating homogeneity.

Since the company's preference was to use water-based spraying coating technology, it is interesting to test all the varnishes using the new spraying equipment.

The coating process was done outdoors due to the absence of an evacuation/ventilation system in the new production unit. The results would be more reliable if the tests were conducted in a controlled environment. Because the external environmental factors, especially wind affected the direction of the flow of the spat from the equipment.

Another interesting parameter to experiment with would be to check the effect of temperature on the varnish performance.

> It is interesting to know how a varnish would perform with a temperature above room temperature. For example, thermal spray coating – try heating the coatings at different temperatures to measure the coating abilities or efficiency.

Some food applications require high barrier properties like gas and oil. It is interesting to test the coated samples for oil and gas barrier properties for high functional performance and demands.

Lastly, the ocean biodegradability of the reject material from the repulped stock of coated lids needs to be investigated.

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APPENDICES

APPENDIX A

A.1 The project plan and timeline (Gantt chart) of the planned and actual project plan

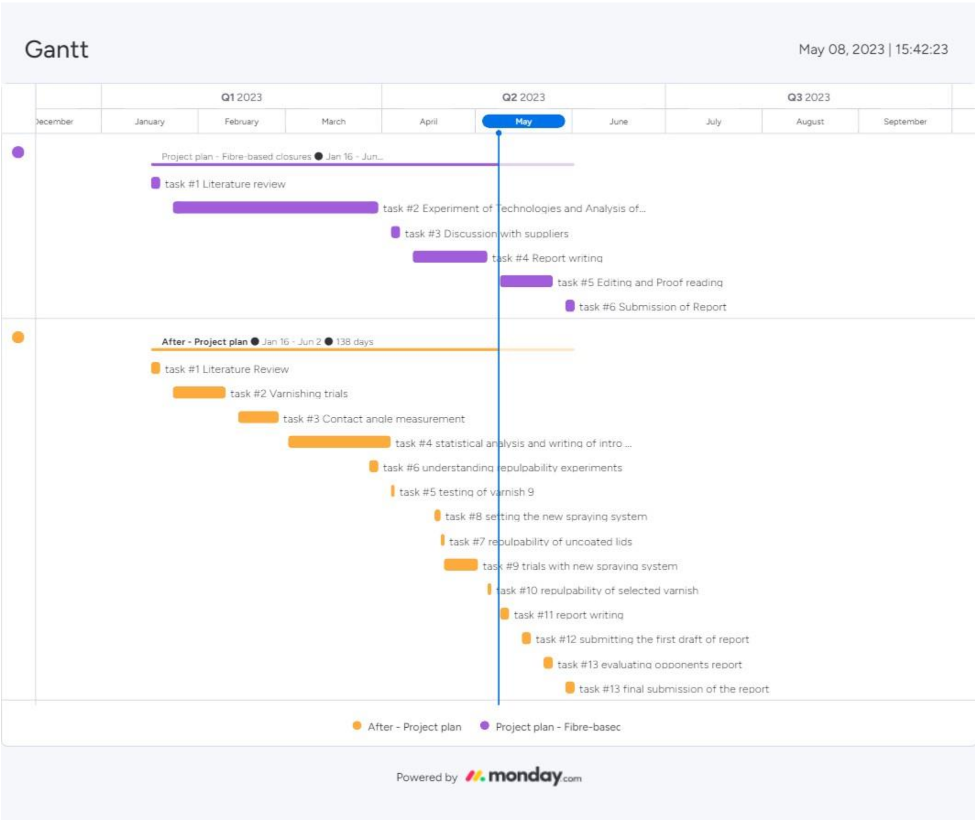


Figure 31 Ghantt chart for before and after project plan.

A meeting with the supervisor was scheduled either every week or month, to update on theprogress. Worked closely with the company supervisor and constantly in touch to maintain a good flow of the work.

After Project plan:

<u>Task 1:</u> 16th Jan – 20th Jan:

Literature review

- Literature study on coatings and technologies used for coating.

<u>Task 2:</u> 23rd Jan – 5th May

Experimentation

- Experiments/ trials using the different technologies – spraying, brush coating, dip coating.
- Analysis of the different technologies
- Qualitative analysis of the closures –visual, barrier properties, grease resistance
- Quantitative analysis of the closures - contact angle, penetration time
- Experiment at Karlstad University – repulpability for coated and uncoated lids
- Simultaneously report writing on intro, material, and method

<u>Task 3:</u> 5th May – 19th May

Report writing

- Results and Discussion
- Completion of the first draft of the thesis report
- Submit the first draft by the 15th to Advisors (company and academic)

<u>Task 5:</u> 22nd May – 27th May

- Editing and proofreading the report
- Opponent report reading

<u>Task 6:</u> 29th May – 2nd June

- Final submission of the report

APPENDIX B

B.1 The highly viscous texture of the varnish 9

Figure 32 Highly viscous varnish with small air bubbles.

Figure 33 The unevenness in dip coating varnish 9.

APPENDIX C

C.1 Raw Material of samples that were selected (samples with above 2 min penetration time) for statistical analysis along with the coating techniques used coat the sample.

Sample code	**Varnish**	**Coat weight (g/m2)**	**Penetration time (mins)**	**Left angle (°)**	**Right angle (°)**	**Avg. CA (°)**	**Coating Technique**
1248	V1	28	20	85,01	94,91	89,96	Spray gun
1248	V1	28	21	88,76	95,14	91,95	Spray gun
1248	V1	28	24	89,72	97,11	93,42	Spray gun
12411	V1	21,54	56	90,7	76,04	83,37	Spray gun
12411	V1	21,54	21	94,98	97,46	96,22	Spray gun
12411	V1	21,54	62	85,23	83,17	84,2	Spray gun
12412	V1	12,28	8	85,58	84,33	84,95	Spray gun
12412	V1	12,28	6	95,57	106,41	100,99	Spray gun
12412	V1	12,28	6	89,57	87,4	88,49	Spray gun
12413	V1	39	8	90,72	90,78	90,75	Spray gun
12413	V1	39	26	92,8	88,15	90,48	Spray gun
12413	V1	39	33	99,16	95,48	97,32	Spray gun
12414	V1	20	2	107,25	106,05	106,65	Spray gun
12414	V1	20	5	91,58	95,72	94,65	Spray gun
12414	V1	20	20	106,81	99,09	102,95	Spray gun
12416	V1	35	22	90,69	92,51	91,6	Spray gun
12416	V1	35	21	89,73	86,87	88,3	Spray gun
12416	V1	35	30	75,85	63,32	69,59	Spray gun
12418	V1	38	42	88,54	88,38	88,46	Spray gun
12418	V1	38	40	78,75	73,04	75,9	Spray gun
12418	V1	38	38	96,47	93,8	95,14	Spray gun
12419	V1	26	57	79,72	79,64	79,68	Brush
12419	V1	26	53	83,66	80,26	81,96	Brush
12419	V1	26	51	104,02	95,08	99,55	Brush
12420	V1	17	47	99,55	88,83	94,19	Brush
12420	V1	17	46	92,11	100,1	96,11	Brush
12420	V1	17	45	89,25	92,9	91,07	Brush

12421	V1	22	0	87,02	85,7	86,36	Spray gun
12421	V1	22	1	76,91	79,8	78,36	Spray gun
12421	V1	22	9	86,06	84,88	85,47	Spray gun
20801	V2	24	40	105,71	101,52	103,61	Brush
20801	V2	24	38	106,93	102,19	104,56	Brush
20801	V2	24	36	100,55	98,49	99,52	Brush
20802	V2	21,7	52	93,05	85,97	89,51	Brush
20802	V2	21,7	50	93,02	89,65	91,33	Brush
20802	V2	21,7	47	80,87	83,07	81,97	Brush
20803	V2	32	46	101,85	101,72	101,79	Brush
20803	V2	32	43	102,37	95,15	98,76	Brush
20803	V2	32	42	110,89	102,82	106,86	Brush
20804	V2	26	40	85,41	82,78	84,1	Brush
20804	V2	26	38	86,84	85,59	86,22	Brush
20804	V2	26	36	90,89	92,79	91,84	Brush
20805	V2	24	42	112,54	98,59	105,56	Brush
20805	V2	24	41	92,39	90,21	91,3	Brush
20805	V2	24	39	92,56	93,38	92,97	Brush
20806	V2	19,2	15	88,88	89,43	89,16	Brush
20806	V2	19,2	34	96,32	105,34	100,83	Brush
20806	V2	19,2	31	96,62	94,87	95,74	Brush
20807	V2	16	5	112,46	102,52	107,49	Brush
20807	V2	16	15	90,51	91,68	91,1	Brush
20807	V2	16	14	105,72	96	100,86	Brush
20808	V2	20	60	98,85	99,14	99	Brush
20808	V2	20	58	92,95	102,34	97,65	Brush
20808	V2	20	57	97,26	96,66	96,96	Brush
20809	V2	39,2	52	96,62	97,63	97,12	Dip
20809	V2	39,2	49	100,1	96,44	98,27	Dip
20809	V2	39,2	47	97	98,73	97,87	Dip
20810	V2	33	40	92,18	91,4	91,79	Dip
20810	V2	33	43	108,94	91,87	100,41	Dip
20810	V2	33	22	104,72	97,73	101,23	Dip
20811	V2	30	22	112,46	109,33	110,89	Dip
20811	V2	30	27	129,03	102,63	115,83	Dip
20811	V2	30	25	85,07	88,26	86,66	Dip
20812	V2	19	33	86,3	91,09	88,69	Brush

20812	V2	19	30	97,67	93,09	95,38	Brush
20812	V2	19	29	97,23	105,25	101,24	Brush
20813	V2	126	66	97,64	93,96	95,8	Dip
20813	V2	126	72	97,2	95,73	96,47	Dip
20813	V2	126	74	98,68	98,63	98,66	Dip
20814	V2	55	24	98,13	100,26	99,2	Dip
20814	V2	55	83	96,86	94,26	95,56	Dip
20814	V2	55	86	107,04	97,61	102,32	Dip
20815	V2	42	79	101,51	103,27	102,39	Dip
20815	V2	42	82	94,83	99,28	97,06	Dip
20815	V2	42	76	103,48	106,07	104,77	Dip
20816	V3	14,6	7	80,89	84,08	82,49	Brush
20816	V3	14,6	4	77,58	83,48	80,53	Brush
20816	V3	14,6	2	84,83	84,48	84,66	Brush
20818	V3	48	66	85,58	78,36	81,97	Dip
20818	V3	48	63	83,43	77,02	80,23	Dip
20818	V3	48	67	73,53	91,5	82,52	Dip
20819	V3	21	3	78,41	80,9	79,66	Brush
20819	V3	21	4	79,08	66,16	72,62	Brush
20819	V3	21	4	77,5	82,88	80,19	Brush
20820	V3	32	4	76,16	69,82	72,99	Dip
20820	V3	32	1	94,28	92,09	93,19	Dip
20820	V3	32	2	75,16	86,68	80,92	Dip
20821	V4	146	2	97,42	68,87	83,14	Dip
20821	V4	146	4	84,43	74,81	79,62	Dip
20821	V4	146	59	62,43	73,26	67,85	Dip
20823	V4	22	4	91,79	69,92	80,86	Brush
20823	V4	22	3	87,49	85,71	86,6	Brush
20823	V4	22	51	94,7	85,83	90,26	Brush
20824	V4	36	37	82,13	85,84	83,99	Dip
20824	V4	36	53	96,48	80,03	88,26	Dip
20824	V4	36	59	72,61	65,75	69,18	Dip
20825	V4	34,8	6	97,23	104,73	100,98	Dip
20825	V4	34,8	1	83,11	87,86	85,49	Dip
20825	V4	34,8	5	102,48	98,3	100,44	Dip
21001	V5	17	31	95,57	91,39	93,48	Brush
21001	V5	17	1	99,53	99,23	99,38	Brush

21001	V5	17	61	99,08	98,54	98,81	Brush
21002	V5	31	64	93,51	102,49	98	Brush
21002	V5	31	63	87,72	89,5	88,61	Brush
21002	V5	31	61	96,23	99,16	97,69	Brush
21003	V5	29	61	96,3	113,58	104,94	Brush
21003	V5	29	58	103,38	96,22	99,8	Brush
21003	V5	29	56	99,83	100,15	99,99	Brush
21004	V5	23	54	77,43	91,56	84,49	Brush
21004	V5	23	53	94,41	91,39	92,9	Brush
21004	V5	23	51	90,4	99,53	94,96	Brush
21005	V5	23	48	88,64	89,9	89,27	Brush
21005	V5	23	46	90,99	90,9	90,94	Brush
21005	V5	23	44	93,09	95,51	94,3	Brush
21006	V5	21	55	104,53	103,72	104,13	Brush
21006	V5	21	53	87,88	94,7	91,29	Brush
21006	V5	21	51	90,11	103,45	96,78	Brush
21007	V5	20	54	85,73	96,29	91,01	Brush
21007	V5	20	53	85,6	103,69	94,64	Brush
21007	V5	20	52	97,15	98,15	97,65	Brush
21008	V5	21	60	88,2	90,49	89,35	Brush
21008	V5	21	58	93,51	96,17	94,84	Brush
21008	V5	21	57	97,87	104,82	101,34	Brush
21009	V5	16	56	99,5	97,53	98,51	Brush
21009	V5	16	55	102,08	99,95	101,01	Brush
21009	V5	16	53	90,94	87,56	89,25	Brush
21010	V5	34	63	89,69	97,27	93,48	Dip
21010	V5	34	65	88,07	97,45	92,76	Dip
21010	V5	34	65	97,1	90,97	94,04	Dip
21011	V5	126	53	91,93	89,01	90,47	Dip
21011	V5	126	51	85,77	85,91	85,84	Dip
21011	V5	126	50	85,14	85,06	85,1	Dip
21012	V5	55	57	89,47	79,79	84,63	Dip
21012	V5	55	56	114,05	92,85	103,45	Dip
21012	V5	55	55	78,28	77,55	77,91	Dip
21013	V6	100	80	86,7	98,63	92,67	Dip
21013	V6	100	77	92,57	91,04	91,81	Dip
21013	V6	100	75	102,24	106,4	104,32	Dip

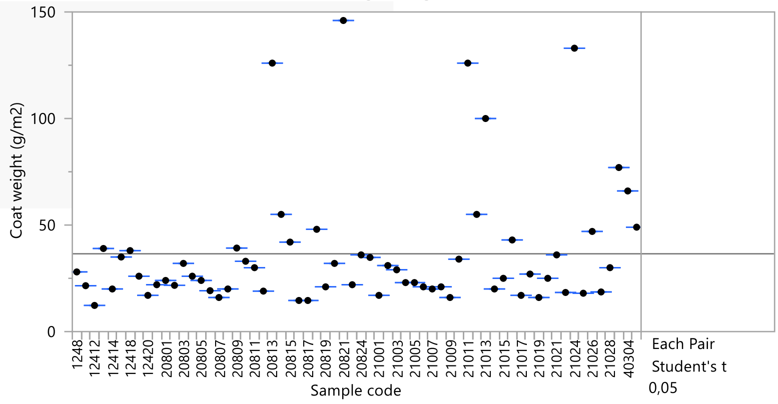
21014	V6	20	73	97,5	98,86	98,18	Brush
21014	V6	20	81	108,07	93,98	101,03	Brush
21014	V6	20	84	89,45	78,5	83,98	Brush
21015	V6	25	67	100,43	99,51	99,97	Brush
21015	V6	25	80	102,85	109,72	106,28	Brush
21015	V6	25	83	103,73	98,37	101,05	Brush
21016	V6	43	62	103,24	98,58	100,91	Dip
21016	V6	43	81	96,83	96,57	96,7	Dip
21016	V6	43	83	109,82	99,28	104,55	Dip
21017	V6	17	68	92,3	101,82	97,06	Brush
21017	V6	17	66	111,39	116,14	113,76	Brush
21017	V6	17	70	93,7	96,34	95,02	Brush
21018	V6	27	69	98,25	99,85	99,05	Dip
21018	V6	27	67	98,23	104,66	101,45	Dip
21018	V6	27	65	99,92	86,63	93,28	Dip
21019	V6	16	68	91,09	90,11	90,6	Brush
21019	V6	16	6	91,14	86,53	88,83	Brush
21019	V6	16	71	98,37	93,51	95,94	Brush
21020	V6	25	16	93,54	88,88	91,21	Dip
21020	V6	25	19	77,92	78,22	78,07	Dip
21020	V6	25	63	97,17	91,17	94,17	Dip
21021	V7	36	3	87,41	86,64	87,02	Dip
21021	V7	36	3	79,9	81,4	80,65	Dip
21021	V7	36	1	0	0	0	Dip
21023	V8	18,36	66	81,68	83,09	82,39	Brush
21023	V8	18,36	69	98,76	96,05	97,4	Brush
21023	V8	18,36	68	95,09	93,51	94,3	Brush
21024	V8	133	81	96,2	113,26	104,73	Dip
21024	V8	133	79	98,58	96,32	97,45	Dip
21024	V8	133	77	93,99	87,65	90,82	Dip
21025	V8	18	60	95,69	88,7	92,19	Brush
21025	V8	18	71	100,96	91,7	96,33	Brush
21025	V8	18	69	98,82	92,13	95,47	Brush
21026	V8	47	0	117,29	87,14	102,21	Dip
21026	V8	47	0	102,66	101,6	102,13	Dip
21026	V8	47	0	100,98	98,68	99,83	Dip
21027	V8	18,6	52	95,03	102,2	98,62	Brush

21027	V8	18,6	50	97,01	99,19	98,1	Brush
21027	V8	18,6	49	102,14	101,05	101,59	Brush
21028	V8	30	62	96,8	90,53	93,66	Dip
21028	V8	30	55	100,47	98,7	99,58	Dip
21028	V8	30	53	113,99	100,95	107,47	Dip
40302	V9	77	8	39.79	35.05	37.42	Dip
40302	V9	77	8	31.19	33.59	32.39	Dip
40302	V9	77	8	31.87	55.72	43.80	Dip
40304	V9	66	19	45.06	41.66	43.36	Dip
40304	V9	66	16	53.92	29.04	41.48	Dip
40304	V9	66	14	36.71	28.50	32.60	Dip
40308	V9	49	2	53.49	59.59	56.54	Dip
40308	V9	49	4	55.90	60.69	58.29	Dip
40308	V9	49	1	52.27	55.74	54.01	Dip

APPENDIX D

D.1 Statistical analysis - ANOVA of all samples

Fit Group
Oneway Analysis of Coat weight (g/m2) By Sample code



Oneway Anova
Summary of Fit

Rsquare	1
Adj Rsquare	1
Root Mean Square Error	8,259e-7
Mean of Response	36,51375
Observations (or Sum Wgts)	192

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample code	63	164445,29	2610,24	3,83e+15	<,0001*
Error	128	8,7311e-11	6,82e-13		
C. Total	191	164445,29			

Means Comparisons
Comparisons for each pair using Student's t
Confidence Quantile

t	Alpha
1,97867	0,05

Connecting Letters Report

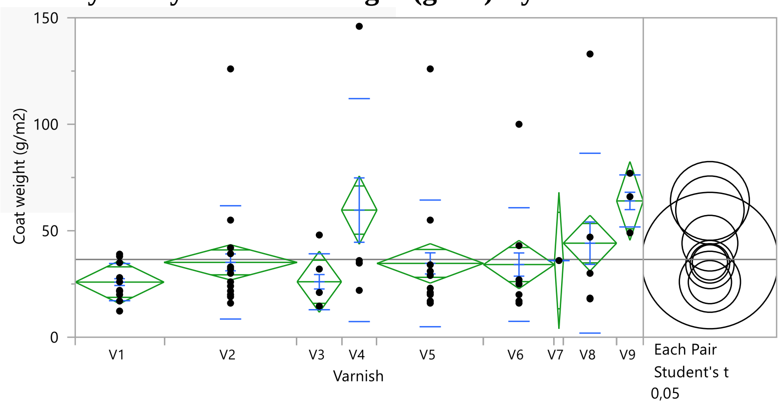
Level		Mean
20821	A	146,00000
21024	B	133,00000
21011	C	126,00000
20813	C	126,00000
21013	D	100,00000
40302	E	77,00000
40304	F	66,00000
21012	G	55,00000
20814	G	55,00000
40308	H	49,00000
20818	I	48,00000
21026	J	47,00000
21016	K	43,00000
20815	L	42,00000
20809	M	39,20000
12413	N	39,00000
12418	O	38,00000
20824	P	36,00000
21021	P	36,00000

Level		Mean
12416	Q	35,00000
20825	R	34,80000
21010	S	34,00000
20810	T	33,00000
20820	U	32,00000
20803	U	32,00000
21002	V	31,00000
20811	W	30,00000
21028	W	30,00000
21003	X	29,00000
1248	Y	28,00000
21018	Z	27,00000
12419	A1	26,00000
20804	A1	26,00000
21015	B1	25,00000
21020	B1	25,00000
20801	C1	24,00000
20805	C1	24,00000
21004	D1	23,00000
21005	D1	23,00000
12421	E1	22,00000
20823	E1	22,00000
20802	F1	21,70000

Level		Mean
12411	G 1	21,54000
21006	H 1	21,00000
20819	H 1	21,00000
21008	H 1	21,00000
12414	I 1	20,00000
20808	I 1	20,00000
21014	I 1	20,00000
21007	I 1	20,00000
20806	J 1	19,20000
20812	K 1	19,00000
21027	L 1	18,60000
21023	M 1	18,36000
21025	N 1	18,00000
21017	O 1	17,00000
12420	O 1	17,00000
21001	O 1	17,00000
21009	P 1	16,00000
21019	P 1	16,00000
20807	P 1	16,00000
20816	Q 1	14,60000
20817	Q 1	14,60000
12412	R 1	12,28000

Levels not connected by same letter are significantly different.

Oneway Analysis of Coat weight (g/m2) By Varnish



Oneway Anova
Summary of Fit

Rsquare	0,119706
Adj Rsquare	0,081223
Root Mean Square Error	28,12543
Mean of Response	36,51375
Observations (or Sum Wgts)	192

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Varnish	8	19685,04	2460,63	3,1106	0,0026*
Error	183	144760,25	791,04		
C. Total	191	164445,29			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
V1	30	25,8820	5,135	15,751	36,013
V2	45	35,1400	4,193	26,868	43,412
V3	15	26,0400	7,262	11,712	40,368
V4	12	59,7000	8,119	43,681	75,719
V5	36	34,6667	4,688	25,418	43,915
V6	24	34,1250	5,741	22,798	45,452
V7	3	36,0000	16,238	3,962	68,038
V8	18	44,1600	6,629	31,080	57,240
V9	9	64,0000	9,375	45,503	82,497

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
V1	30	25,882	8,7434158	1,596322	22,617155	29,146845
V2	45	35,14	26,59914	3,9651657	27,148734	43,131266
V3	15	26,04	13,133208	3,3909796	18,767072	33,312928
V4	12	59,7	52,355463	15,11372	26,434926	92,965074
V5	36	34,666667	29,745588	4,957598	24,602208	44,731126
V6	24	34,125	26,678949	5,4458176	22,859468	45,390532
V7	3	36	0	0	36	36
V8	18	44,16	42,22715	9,9530348	23,160932	65,159068
V9	9	64	12,216792	4,0722639	54,609343	73,390657

Means Comparisons
Comparisons for each pair using Student's t
Confidence Quantile

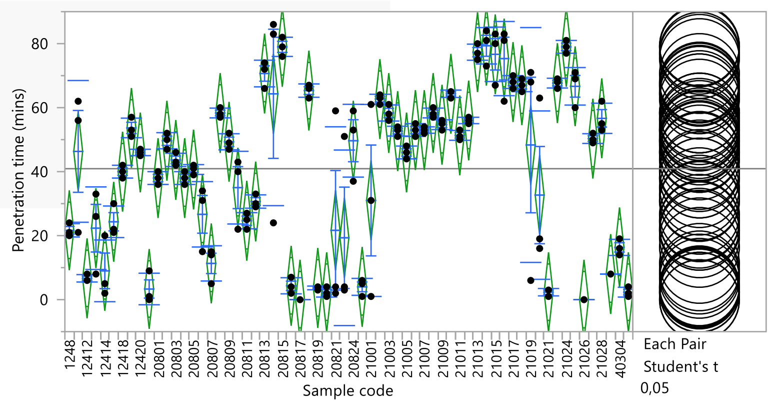
t	Alpha
1,97301	0,05

Connecting Letters Report

Level				Mean
V9	A			64,000000
V4	A			59,700000
V8	A	B		44,160000
V7	A	B	C	36,000000
V2		B	C	35,140000
V5		B	C	34,666667
V6		B	C	34,125000
V3		B	C	26,040000
V1			C	25,882000

Levels not connected by same letter are significantly different.

Oneway Analysis of Penetration time (mins) By Sample code



Oneway Anova
Summary of Fit

Rsquare	0,880554
Adj Rsquare	0,821764
Root Mean Square Error	10,90871
Mean of Response	40,92188
Observations (or Sum Wgts)	192

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample code	63	112289,83	1782,38	14,9780	<,0001*
Error	128	15232,00	119,00		
C. Total	191	127521,83			

Means Comparisons
Comparisons for each pair using Student's t
Confidence Quantile

t	Alpha
1,97867	0,05

Connecting Letters Report

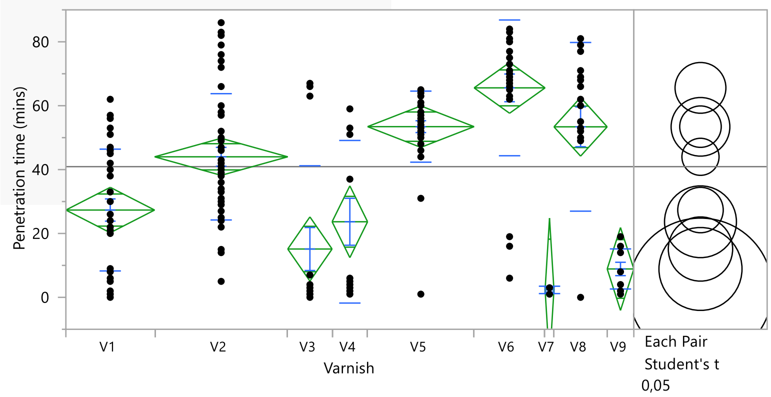
Level		Mean
21014	A	79,333333
20815	A	79,000000
21024	A	79,000000

Level																												Mean
21013	A																											77,333333
21015	A																											76,666667
21016	A	B																										75,333333
20813	A	B	C																									70,666667
21017	A	B	C	D																								68,000000
21023	A	B	C	D	E																							67,666667
21018	A	B	C	D	E	F																						67,000000
21025	A	B	C	D	E	F	G																					66,666667
20818	A	B	C	D	E	F	G	H																				65,333333
20814	A	B	C	D	E	F	G	H																				64,333333
21010	A	B	C	D	E	F	G	H																				64,333333
21002	A	B	C	D	E	F	G	H	I																			62,666667
21008		B	C	D	E	F	G	H	I	J																		58,333333
21003		B	C	D	E	F	G	H	I	J																		58,333333
20808		B	C	D	E	F	G	H	I	J																		58,333333
21028			C	D	E	F	G	H	I	J	K																	56,666667
21012			C	D	E	F	G	H	I	J	K																	56,000000
21009			C	D	E	F	G	H	I	J	K	L																54,666667
12419			C	D	E	F	G	H	I	J	K	L																53,666667
21007				D	E	F	G	H	I	J	K	L																53,000000
21006				D	E	F	G	H	I	J	K	L																53,000000
21004				D	E	F	G	H	I	J	K	L																52,666667
21011				D	E	F	G	H	I	J	K	L	M															51,333333
21027					E	F	G	H	I	J	K	L	M															50,333333
20802						F	G	H	I	J	K	L	M	N														49,666667
20824						F	G	H	I	J	K	L	M	N														49,666667
20809							G	H	I	J	K	L	M	N														49,333333
21019								H	I	J	K	L	M	N	O													48,333333
12411									I	J	K	L	M	N	O	P												46,333333
21005									I	J	K	L	M	N	O	P												46,000000
12420									I	J	K	L	M	N	O	P												46,000000
20803										J	K	L	M	N	O	P	Q											43,666667
20805											K	L	M	N	O	P	Q	R										40,666667
12418											K	L	M	N	O	P	Q	R										40,000000
20801												L	M	N	O	P	Q	R	S									38,000000
20804												L	M	N	O	P	Q	R	S									38,000000
20810													M	N	O	P	Q	R	S	T								35,000000
21020														N	O	P	Q	R	S	T	U							32,666667
21001															O	P	Q	R	S	T	U							31,000000
20812																P	Q	R	S	T	U							30,666667
20806																	Q	R	S	T	U	V						26,666667
20811																		R	S	T	U	V	W					24,666667
12416																		R	S	T	U	V	W					24,333333
12413																			S	T	U	V	W	X				22,333333
20821																			S	T	U	V	W	X	Y			21,666667
1248																			S	T	U	V	W	X	Y			21,666667
20823																				T	U	V	W	X	Y	Z		19,333333
40304																					U	V	W	X	Y	Z	A1	16,333333

Level							Mean
20807	V	W	X	Y	Z	A1	11,333333
12414		W	X	Y	Z	A1	9,000000
40302		W	X	Y	Z	A1	8,000000
12412			X	Y	Z	A1	6,666667
20816				Y	Z	A1	4,333333
20825					Z	A1	4,000000
20819					Z	A1	3,666667
12421					Z	A1	3,333333
21021					Z	A1	2,333333
20820					Z	A1	2,333333
40308					Z	A1	2,333333
20817						A1	0,000000
21026						A1	0,000000

Levels not connected by same letter are significantly different.

Oneway Analysis of Penetration time (mins) By Varnish



Oneway Anova
Summary of Fit

Rsquare	0,44115
Adj Rsquare	0,416719
Root Mean Square Error	19,73397
Mean of Response	40,92188
Observations (or Sum Wgts)	192

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Varnish	8	56256,21	7032,03	18,0572	<,0001*
Error	183	71265,62	389,43		
C. Total	191	127521,83			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
V1	30	27,3333	3,603	20,22	34,442
V2	45	44,0000	2,942	38,20	49,804
V3	15	15,1333	5,095	5,08	25,186
V4	12	23,6667	5,697	12,43	34,906
V5	36	53,4444	3,289	46,96	59,934
V6	24	65,5833	4,028	57,64	73,531
V7	3	2,3333	11,393	-20,15	24,813
V8	18	53,3889	4,651	44,21	62,566
V9	9	8,8889	6,578	-4,09	21,867

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
V1	30	27,333333	19,066731	3,4810929	20,213699	34,452968
V2	45	44	19,772571	2,9475208	38,059662	49,940338
V3	15	15,133333	26,062745	6,7293718	0,7002663	29,5664
V4	12	23,666667	25,453463	7,3477819	7,4943077	39,839026
V5	36	53,444444	11,123834	1,8539723	49,680681	57,208208
V6	24	65,583333	21,249893	4,3376163	56,61029	74,556376
V7	3	2,3333333	1,1547005	0,6666667	-0,535102	5,2017685
V8	18	53,388889	26,402181	6,2230537	40,259393	66,518385
V9	9	8,8888889	6,2738434	2,0912811	4,0663859	13,711392

Means Comparisons
Comparisons for each pair using Student's t
Confidence Quantile

t	Alpha
1,97301	0,05

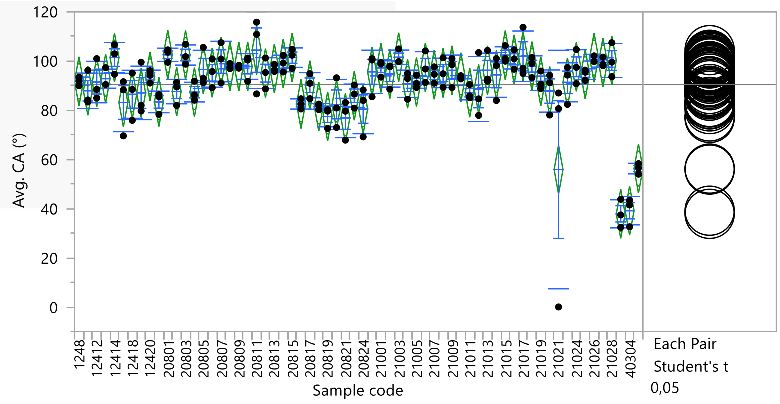
Connecting Letters Report

Level						Mean
V6	A					65,583333
V5		B				53,444444
V8		B	C			53,388889
V2			C			44,000000
V1				D		27,333333
V4				D	E	23,666667
V3				D	E	15,133333
V9					E	8,888889

Level		Mean
V7	E	2,333333

Levels not connected by same letter are significantly different.

Oneway Analysis of Avg. CA (°) By Sample code



Oneway Anova
Summary of Fit

Rsquare	0,771076
Adj Rsquare	0,658402
Root Mean Square Error	8,752704
Mean of Response	90,47052
Observations (or Sum Wgts)	192

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Sample code	63	33029,361	524,276	6,8435	<,0001*
Error	128	9806,058	76,610		
C. Total	191	42835,419			

Means Comparisons
Comparisons for each pair using Student's t
Confidence Quantile

t	Alpha
1,97867	0,05

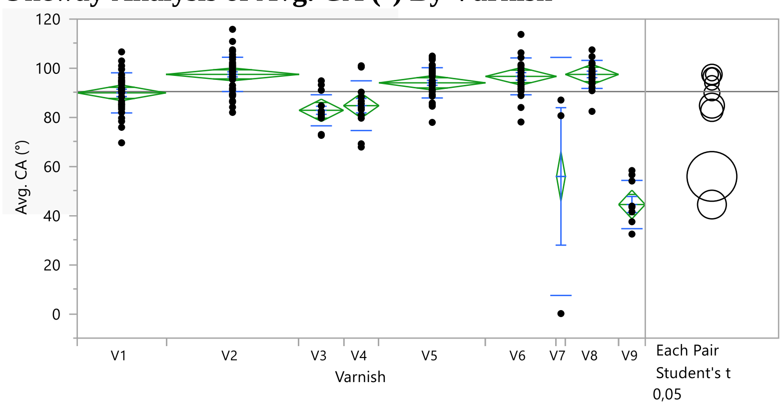
Connecting Letters Report

Level																	Mean
20811	A																104,46000
20801	A	B															102,56333
20803	A	B															102,47000
21015	A	B															102,43333
21017	A	B	C														101,94667
21003	A	B	C	D													101,57667
12414	A	B	C	D	E												101,41667
20815	A	B	C	D	E												101,40667
21026	A	B	C	D	E												101,39000
21016	A	B	C	D	E	F											100,72000
21028	A	B	C	D	E	F	G										100,23667
20807	A	B	C	D	E	F	G	H									99,81667
21027	A	B	C	D	E	F	G	H									99,43667
20814	A	B	C	D	E	F	G	H									99,02667
21018	A	B	C	D	E	F	G	H									97,92667
20808	A	B	C	D	E	F	G	H									97,87000
20810	A	B	C	D	E	F	G	H									97,81000
20809	A	B	C	D	E	F	G	H									97,75333
21024	A	B	C	D	E	F	G	H									97,66667
21006	A	B	C	D	E	F	G	H	I								97,40000
21001	A	B	C	D	E	F	G	H	I	J							97,22333
20813	A	B	C	D	E	F	G	H	I	J							96,97667
20805	A	B	C	D	E	F	G	H	I	J	K						96,61000
21013	A	B	C	D	E	F	G	H	I	J	K	L					96,26667
21009	A	B	C	D	E	F	G	H	I	J	K	L					96,25667
20825	A	B	C	D	E	F	G	H	I	J	K	L	M				95,63667
20806	A	B	C	D	E	F	G	H	I	J	K	L	M				95,24333
21008	A	B	C	D	E	F	G	H	I	J	K	L	M				95,17667
20812	A	B	C	D	E	F	G	H	I	J	K	L	M				95,10333
21002	A	B	C	D	E	F	G	H	I	J	K	L	M				94,76667
21025	A	B	C	D	E	F	G	H	I	J	K	L	M				94,66333
21007	A	B	C	D	E	F	G	H	I	J	K	L	M	N			94,43333
21014	A	B	C	D	E	F	G	H	I	J	K	L	M	N			94,39667
12420	A	B	C	D	E	F	G	H	I	J	K	L	M	N			93,79000
21010	A	B	C	D	E	F	G	H	I	J	K	L	M	N			93,42667
12413	A	B	C	D	E	F	G	H	I	J	K	L	M	N			92,85000
21019	A	B	C	D	E	F	G	H	I	J	K	L	M	N			91,79000
1248	A	B	C	D	E	F	G	H	I	J	K	L	M	N			91,77667
21005	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O		91,50333
12412	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O		91,47667
21023	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O		91,36333
21004	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	90,78333
20817		B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	90,18333
21012		B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	88,66333

Level																	Mean
12411	C	D	E	F	G	H	I	J	K	L	M	N	O	P			87,93000
21020	C	D	E	F	G	H	I	J	K	L	M	N	O	P			87,81667
20802		D	E	F	G	H	I	J	K	L	M	N	O	P			87,60333
20804			E	F	G	H	I	J	K	L	M	N	O	P			87,38667
21011				F	G	H	I	J	K	L	M	N	O	P			87,13667
12419				F	G	H	I	J	K	L	M	N	O	P			87,06333
12418					G	H	I	J	K	L	M	N	O	P			86,50000
20823						H	I	J	K	L	M	N	O	P			85,90667
12421							I	J	K	L	M	N	O	P			83,39667
12416								J	K	L	M	N	O	P			83,16333
20816									K	L	M	N	O	P			82,56000
20820										L	M	N	O	P			82,36667
20818											M	N	O	P			81,57333
20824												N	O	P			80,47667
20819													O	P			77,49000
20821														P			76,87000
40308															Q		56,28000
21021															Q		55,89000
40304																R	39,14667
40302																R	37,87000

Levels not connected by same letter are significantly different.

Oneway Analysis of Avg. CA (°) By Varnish



Oneway Anova
Summary of Fit

Rsquare	0,663111
Adj Rsquare	0,648384
Root Mean Square Error	8,880125
Mean of Response	90,47052
Observations (or Sum Wgts)	192

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Varnish	8	28404,659	3550,58	45,0258	<,0001*
Error	183	14430,760	78,86		
C. Total	191	42835,419			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
V1	30	89,9363	1,6213	86,738	93,14
V2	45	97,4733	1,3238	94,862	100,09
V3	15	82,8347	2,2928	78,311	87,36
V4	12	84,7225	2,5635	79,665	89,78
V5	36	94,0289	1,4800	91,109	96,95
V6	24	96,6621	1,8126	93,086	100,24
V7	3	55,8900	5,1269	45,774	66,01
V8	18	97,4594	2,0931	93,330	101,59
V9	9	44,4322	2,9600	38,592	50,27

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
V1	30	89,936333	8,1756761	1,4926674	86,883486	92,989181
V2	45	97,473333	6,9738388	1,0395985	95,37816	99,568506
V3	15	82,834667	6,3239385	1,6328339	79,332586	86,336747
V4	12	84,7225	10,130439	2,924406	78,285926	91,159074
V5	36	94,028889	6,1626816	1,0271136	91,943737	96,11404
V6	24	96,662083	7,5145609	1,5339033	93,488963	99,835204
V7	3	55,89	48,506838	28,005436	-64,60766	176,38766
V8	18	97,459444	5,7136652	1,3467238	94,618106	100,30078
V9	9	44,432222	9,8413855	3,2804618	36,867464	51,996981

Means Comparisons
Comparisons for each pair using Student's t
Confidence Quantile

t	Alpha
1,97301	0,05

LSD Threshold Matrix

Abs (Dif)-LSD

	V2	V8	V6	V5	V1	V4	V3	V7	V9
V2	-3,694	-4,872	-3,617	-0,473	3,407	7,059	9,415	31,136	46,643
V8	-4,872	-5,840	-4,666	-1,627	2,299	6,207	8,500	30,643	45,874
V6	-3,617	-4,666	-5,058	-1,984	1,928	5,745	8,061	30,043	45,382
V5	-0,473	-1,627	-1,984	-4,130	-0,239	3,466	5,810	27,610	43,067
V1	3,407	2,299	1,928	-0,239	-4,524	-0,771	1,561	23,437	38,845
V4	7,059	6,207	5,745	3,466	-0,771	-7,153	-4,898	17,523	32,564
V3	9,415	8,500	8,061	5,810	1,561	-4,898	-6,398	15,864	31,015
V7	31,136	30,643	30,043	27,610	23,437	17,523	15,864	-14,306	-0,223
V9	46,643	45,874	45,382	43,067	38,845	32,564	31,015	-0,223	-8,259

Positive values show pairs of means that are significantly different.

Connecting Letters Report

Level						Mean
V2	A					97,473333
V8	A					97,459444
V6	A					96,662083
V5	A	B				94,028889
V1		B	C			89,936333
V4			C	D		84,722500
V3				D		82,834667
V7					E	55,890000
V9					E	44,432222

Levels not connected by same letter are significantly different.

APPENDIX E

E.1 The trials with the new spray system

Figure 34 Spraying of the lids using the new spraying equipment outdoors.

APPENDIX F

F.1 Raw Data for the statistical analysis of selected varnished samples with above 2 min penetration time

Sample code	Varnish	Coat weight (g/m2)	Penetration time (min)	Avg. CA (°)	Left angle (°)	Right angle (°)	Avg. CA (°)
42108	V1	49	4	106,83	98,1	115,56	106,83
42108	V1	49	59	96,67	90,42	102,91	96,67
42108	V1	49	0	99,02	98,65	99,4	99,02
42111	V1	44	48	105,12	114,03	96,21	105,12
42111	V1	44	57	105,48	105,01	105,96	105,48
42111	V1	44	55	100,61	108,47	92,75	100,61
42005	V2	28	70	100,44	100,26	100,62	100,44
42005	V2	28	70	98,45	97,03	99,87	98,45
42005	V2	28	70	92,73	95,49	89,96	92,73
42602	V5	35,6	34	96,77	93,58	99,96	96,77
42602	V5	35,6	83	106,58	107,2	105,95	106,58
42602	V5	35,6	1	101,2	100,01	102,39	101,2
42603	V5	36	77	102,52	100,6	104,43	102,52
42603	V5	36	77	107,93	107,94	107,91	107,93
42603	V5	36	76	102,09	106,11	98,07	102,09
42604	V5	73	75	104,62	106,66	102,58	104,62
42604	V5	73	74	99,33	102,34	96,32	99,33
42604	V5	73	72	98,76	103,32	94,19	98,76
42607	V5	35	65	94,01	94,71	93,3	94,01
42607	V5	35	2	110,87	115,58	106,15	110,87
42607	V5	35	1	103,25	98,9	107,59	103,25
42608	V5	39	66	102,93	105,12	100,74	102,93
42608	V5	39	1	104,46	102,6	106,31	104,46
42608	V5	39	0	105,66	103,95	107,38	105,66
42610	V5	31,5	53	76,36	81,32	71,41	76,36
42610	V5	31,5	53	74,72	81,79	67,64	74,72
42610	V5	31,5	54	80,77	82,5	79,04	80,77
42701	V6	57,2	77	107,65	107,59	107,7	107,65
42701	V6	57,2	75	103,43	101,14	105,72	103,43

42701	V6	57,2	81	101,75	101,53	101,96	101,75
42702	V6	36,2	2	101,2	113,33	89,06	101,2
42702	V6	36,2	76	98,72	99,26	98,17	98,72
42702	V6	36,2	1	97,92	95,09	100,75	97,92
42704	V6	18,4	71	91,21	94,28	88,14	91,21
42704	V6	18,4	1	81	80,19	81,81	81
42704	V6	18,4	0	0	0	0	0
42705	V6	37,5	47	102,04	105,52	98,55	102,04
42705	V6	37,5	90	100,24	104,98	95,49	100,24
42705	V6	37,5	91	99,29	99,63	98,94	99,29
50202	V6	20	7	97,69	102,34	93,04	97,69
50202	V6	20	6	101,22	99,82	102,61	101,22
50202	V6	20	13	104,34	105,02	103,65	104,34
50203	V6	20,2	41	105,77	101,45	110,09	105,77
50203	V6	20,2	68	98,03	100,85	95,2	98,03
50203	V6	20,2	64	103,96	103,68	104,24	103,96
50204	V6	14,3	2	109,36	103,94	114,77	109,36
50204	V6	14,3	15	104,2	104,76	103,64	104,2
50204	V6	14,3	53	105,06	105,94	104,18	105,06
50205	V6	23	69	99,17	104,37	93,96	99,17
50205	V6	23	77	94,38	98,88	89,88	94,38
50205	V6	23	79	103,44	102,99	103,88	103,44
50206	V6	28,1	66	102,98	96,7	109,26	102,98
50206	V6	28,1	65	102,08	96,13	108,03	102,08
50206	V6	28,1	73	103,15	103,42	102,88	103,15
50208	V6	25,5	71	97,77	89,55	105,99	97,77
50208	V6	25,5	75	107,99	104,31	111,68	107,99
50208	V6	25,5	78	101,41	97,82	105,01	101,41
50209	V6	26	68	102,83	117,65	88,01	102,83
50209	V6	26	80	110,33	114,66	106	110,33
50209	V6	26	78	108,33	112,52	104,14	108,33
50210	V6	11,8	1	102,92	101,36	104,49	102,92
50210	V6	11,8	32	99,48	99,48	99,48	99,48
50210	V6	11,8	4	121,19	124,91	117,47	121,19
50211	V6	20	82	111,26	114,92	107,61	111,26
50211	V6	20	83	100,03	99,59	100,47	100,03
50211	V6	20	83	108,45	106,52	110,39	108,45
50212	V6	19,1	18	107,67	101,93	113,41	107,67
50212	V6	19,1	25	102,59	101,2	103,98	102,59
50212	V6	19,1	72	105,95	112,81	99,1	105,95
50213	V6	27,2	72	114,74	129,17	100,32	114,74
50213	V6	27,2	72	103,96	104,29	103,63	103,96

50213	V6	27,2	73	98,67	101,24	96,1	98,67
50214	V6	18,6	1	102,29	103,11	101,47	102,29
50214	V6	18,6	65	94,2	99,15	89,24	94,2
50214	V6	18,6	68	101,65	101,63	101,68	101,65
50218	V8	49	63	99,48	102,82	96,14	99,48
50218	V8	49	59	100,97	101,98	99,97	100,97
50218	V8	49	62	87,41	92,91	81,91	87,41
50220	V8	33	27	95,95	94,65	97,25	95,95
50220	V8	33	62	100,33	101	99,67	100,33
50220	V8	33	65	95,95	96,67	95,24	95,95
50221	V8	27	36	103,83	107,15	100,52	103,83
50221	V8	27	40	100,47	100,82	100,11	100,47
50221	V8	27	38	102,82	101,75	103,9	102,82
50222	V8	11	44	84,51	82,04	86,97	84,51
50222	V8	11	1	87,99	88,46	87,51	87,99
50222	V8	11	1	101,88	102,65	101,12	101,88
50225	V8	30	44	98,59	104,82	92,37	98,59
50225	V8	30	56	101,68	101,09	102,27	101,68
50225	V8	30	67	108,31	107,68	108,94	108,31
50226	V8	28	17	92,48	92,78	92,19	92,48
50226	V8	28	64	111,16	114,85	107,48	111,16
50226	V8	28	63	86,45	80,19	92,71	86,45
50227	V8	2,7	3	70,68	75,2	66,15	70,68
50227	V8	2,7	2	97,26	102,88	91,63	97,26
50227	V8	2,7	52	100,02	99,95	100,09	100,02

APPENDIX G

G.1 The average values of the contact angle and penetration time of the samples in interest.

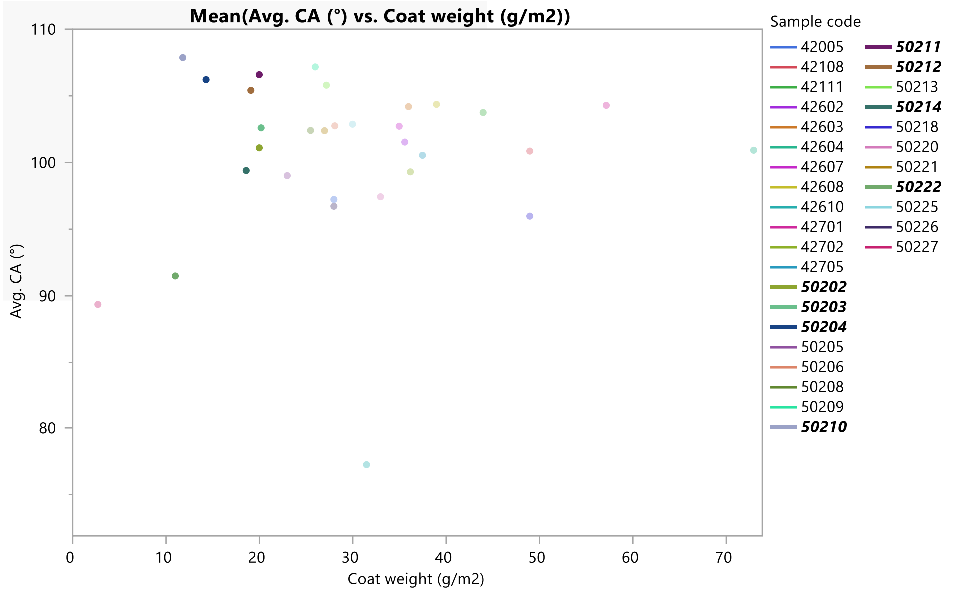


Figure 35 The samples of interest that meet the minimum criteria for coat weight and contact angle.

Table 13 Sample codes and their mean values of contact angle

Sample code	Varnish	Coat weight (g/m2)	Avg. CA (°)
50202	V6	20	101,08
50203	V6	20,2	102,59
50204	V6	14,3	106,21
50210	V6	11,8	107,86
50211	V6	20	106,58
50212	V6	19,1	105,40
50214	V6	18,6	99,38
50222	V8	11	91,46

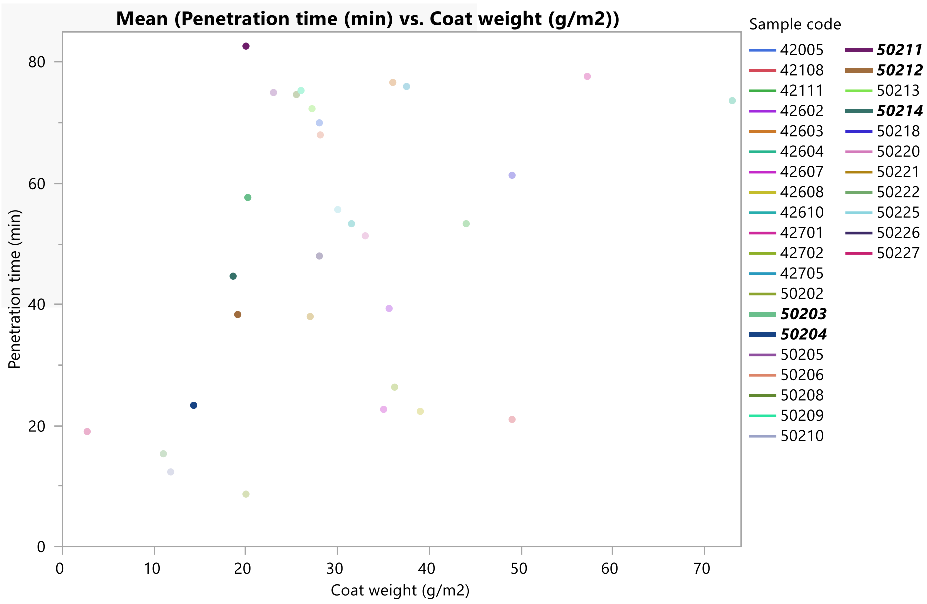


Figure 36 The samples of interest that meet the minimum criteria for coat weight and penetration time.

Table 14 Sample codes and their mean values of penetration time

Sample code	Varnish	Coat weight (g/m2)	Penetration time (min)
50203	V6	20,2	57,67
50204	V6	14,3	23,33
50211	V6	20	82,67
50212	V6	19,1	38,33
50214	V6	18,6	44,67

APPENDIX H

H.1 The coat weight of varnish 6 coated lids used for Repulpability experiment.

Varnish 6	
Sl. No	Coat weight
1	16
2	13
3	13
4	14
5	11
6	11
7	13
8	12
9	13
10	13
11	13
12	13
13	12
14	13
15	13
	12,87

H.2 The defibrillator and the cut samples used for analysis

Figure 37 Samples and water inside the disintegrator for repulping

Figure 38 The sampling size (3x2cm approx.) used for repulpability trials.

H.3 Sommerville screening equipment

Figure 39 (A)The inside of the defibrillator. (B) The sommerville screening equipment.

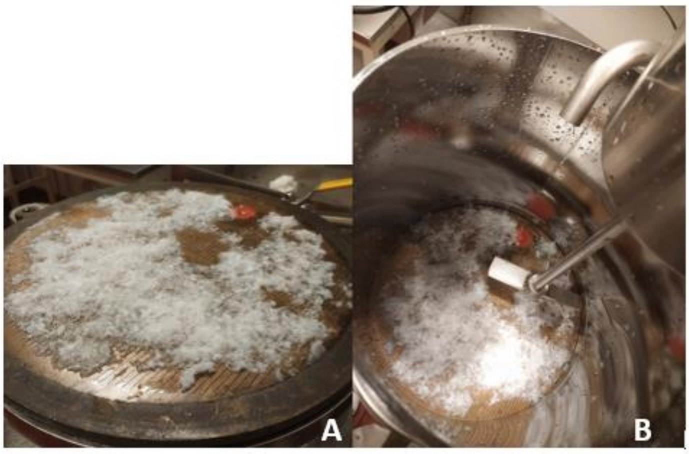


Figure 40 (A) Reject material from repulped uncoated material left after passing through 40µm screen (B) The sommerville defibrillator tank.

H.4 Hand Sheet Former and Press and other parts

Figure 41 The handpress (left) and the handsheet former (right)

Figure 42 The handsheet former (left) and the mesh used to form the handsheets (right)

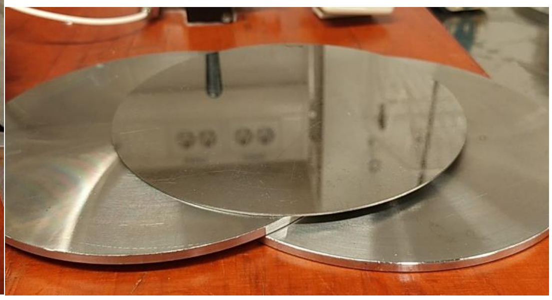


Figure 43 The gusk weight used to press the handsheet with the blotting paper to remove the excess water.

Figure 44 The lower and upper press sheet (left and right) and the gloss sheet (center).

H.5 Handsheets from the repulped material.

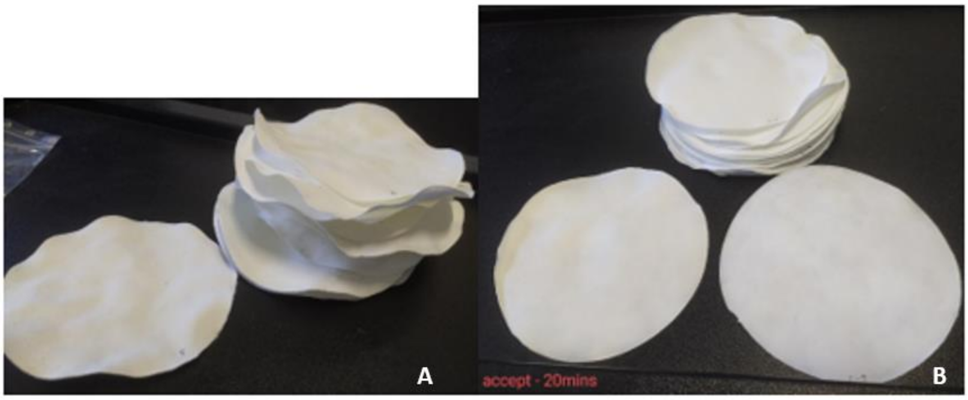


Figure 45 Handseets from the uncoated Lid’s repulped material (A) Handsheet of 10 min defibrillated accept material (B) Handsheet of 20 min defibrillated accept material.

Figure 46 Handsheets from the coated (varnish 6) lid's repulped material Handsheet of 10min (left) and 20min (right) defibrillated accept material.

H.6 Color of the reject material (coated with V6)

Figure 47 The difference in color (light blue) of the reject compared to 10 min disintegrated accept material from coated (V6) lid' repulped material.

Figure 48 Drying of handsheets and reject material for yield calculation.

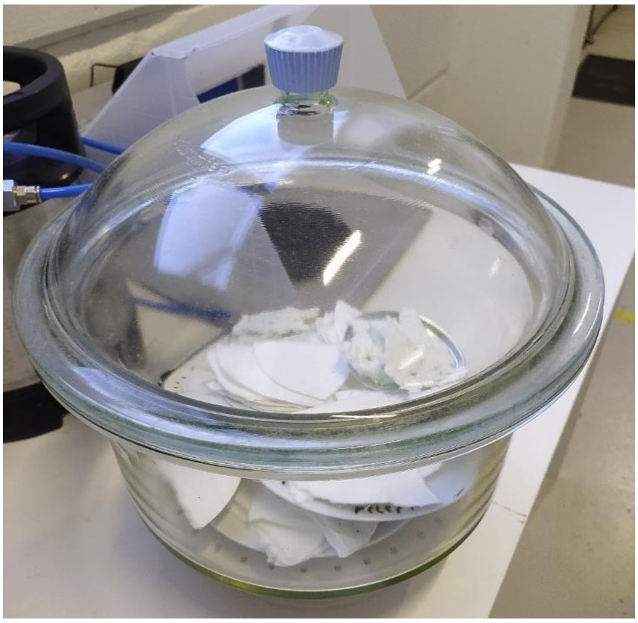


Figure 49 Cooling for the dried handsheets for yield calculation.

APPENDIX I

I.1 Specification of Varnish 6

[redacted]

[redacted] is a fully recyclable water-based coating based on a co-polymer dispersion and sustainable vegetable wax emulsion, which provides specific barrier properties, such as excellent OGR in combination with good MVTR, low Cobb and low CoF values.
In addition, is also offers excellent release properties while being heat sealable already at low temperatures (75-80°C).

It is recommended as a perfect alternative to film laminated and polymer extruded structures for food and other packaging systems.

Typical properties

Appearance	White Liquid
Solids Type	Co-polymer / Vegetable wax
Non-volatile content	39 - 41 %
pH	7,5 – 9,5
Viscosity (Brookfield, #3 @60 RPM)	500 - 2500 mPas