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The Effect of Different Oils on the Oil Resistance Properties of Molded Fiber

Metropolia University of Applied Sciences

Bachelor of Laboratory Services

Laboratory Sciences

Bachelor's Thesis

7 June 2025

Abstract

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Title:	The Effect of Different Oils on the Oil Resistance Properties of Molded Fiber
Number of Pages:	43 pages + 4 appendices
Date:	7 June 2025
Degree:	Bachelor of Laboratory Services
Degree Programme:	Laboratory Sciences
Specialisation option:	
Instructor(s):	Eija Koriseva, Senior Lecturer Annaleena Kokko, Senior Manager, Renewable Coatings, Kemira Oyj

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Molded fiber products (MFPs) made from renewable plant-based fibers have the potential to replace single-use plastic packaging. A large portion of single-use packaging is used for food packaging and often ends up in landfills or in nature after use. The pressure to reduce plastic use increases the demand for alternative ecological materials. For MFPs to effectively replace plastic, they must have packaging properties comparable to those of plastic. Currently, MFPs do not have the same resistance to grease and oil, which is essential for food packaging, as plastic does. Oil resistance is also influenced by the properties of the oil itself, such as viscosity. With vegetable oils, viscosity changes based on the composition and temperature of the oil.

In this study, the permeation times of olive oil, canola oil, palm oil, and soybean oil were examined from molded fiber samples at temperatures of 5°C, 23°C, and 60°C using an automatic computer-controlled camera system. Molded fiber samples were hand-made, and the effect of the mixing order of the chemicals on oil resistance was also investigated.

Statistical analysis concluded that the mixing order did not significantly impact results. It was also found that there were no significant differences in oil permeation times at 23°C and 60°C, or the test method employed did not provide sufficiently different test conditions.

Keywords: Molded fiber, Oil resistance

Tiivistelmä

Tekijä:	Ville Sjöblom
Otsikko:	Erilaisten öljyjen vaikutus kuituvalosten öljynkestoon
Sivumäärä:	43 sivua + 4 liitettä
Aika:	7.6.2025
Tutkinto:	Laboratorioanalyytikko (AMK)
Tutkinto-ohjelma:	Laboratorioanalytiikka
Ammatillinen pääaine:	
Ohjaajat:	Lehtori Eija Koriseva Senior Manager, Renewable Coatings, Kemira Oyj, Annaleena Kokko

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Uusiutuvista kasvipohjaisista kuiduista valmistetuilla kuituvaloksilla on potentiaalia korvata muovisia kertakäyttöpakkauksia. Kertakäyttöpakkauksista suuri osa käytetään ruuan pakkaamiseen, ja usein ne päätyvät käytön jälkeen kaatopaikoille tai luontoon. Paine muovien käytön vähentämiseen lisää kysyntää vaihtoehtoiselle, ekologiselle materiaalille. Jotta kuituvalokset voisivat syrjäyttää muovin materiaalina, sillä on oltava yhtä hyvät ominaisuudet kuin muovilla. Ruokapakkauksissa olennainen rasvan- ja öljynkestävyys ei vielä ole kuituvaloksella samalla tasolla muovin kanssa. Öljynkestävyyteen vaikuttavat myös itse öljyn ominaisuudet, kuten viskositeetti. Kasviöljyillä viskositeettiin vaikuttavat öljyn koostumus ja lämpötila.

Tässä työssä tutkittiin oliivi-, rypsi-, palmu- ja soijapapuöljyjen läpäisyaikoja kuituvalosnäytteistä 5 °C:n, 23 °C:n ja 60 °C:n lämpötiloissa käyttäen automaattista tietokoneohjattua kamerajärjestelmää. Työssä tutkittiin myös kuituvalosnäytteiden kemikaalien sekoitusjärjestyksen vaikutusta öljynkestoon.

Tilastollisten menetelmien avulla pääteltiin, että sekoitusjärjestyksellä ei näyttänyt olevan merkittävää vaikutusta. Lisäksi pääteltiin, että joko öljynläpäisyajoissa ei ollut merkittäviä eroja lämpötiloissa 23 °C ja 60 °C tai käytössä olleella testimenetelmällä ei saatu aikaan riittävän erilaisia testausolosuhteita.

Avainsanat: Kuituvalos, öljynkesto

Contents

List of Abbreviations

AKD	*Alkyl Ketene Dimer*
ANOVA	*Analysis of variance*
CNF	*Cellulose Nanofiber*
DLK	*Double Line Kraft*
MFC	*Microfibrillated Cellulose*
MFP	*Molded Fiber Product*
OCC	*Old Corrugated Cardboard*
OGR	*Oil and Grease Resistance*
ONP	*Old Newspaper*
PDMS	*Polydimethylsiloxane*

1 Introduction

There will be more plastic than fish in the ocean by 2050, stated a report published by the Ellen McArthur Foundation in collaboration with the World Economic Forum and McKinsey & Company in 2016 [1]. The shocking conclusion drew widespread attention and reminded people of the growing problems of ocean waste accumulation. The statement was calculated using the weight ratio of fish and plastics, which was 1:1. The calculations were based on the plastic production for that year and forecasts of its annual growth, as well as several other assumptions. In any case, according to the report, the main source of plastic in the oceans was plastic packages, which represented around 25% of all plastic produced in 2016. [1.]

Plastic production has been growing ever since, and recent studies predict that the growth will continue, with total production doubling by 2050 [2]. Packaging is one of the largest sectors where plastic is used, and since most of the plastic packages serve a single-use purpose only, it creates a focus on finding sustainable alternatives [3]. The popularity of plastic packages can be explained by their desired properties, especially for packaging food. Plastic is cheap, lightweight, durable, and chemically stable [4]. Durability and chemical stability are the main reasons for the waste problem. Instead of degrading, plastic breaks down into smaller pieces over time and ends up becoming a permanent part of the ecosystem.

As pressure to reduce plastic waste grows, demand for new materials to be introduced to the market to replace plastic increases. One promising alternative for single-use plastic packages could be molded fiber products (MFPs). Using natural or recycled fibers as a material for disposable packaging would reduce problems caused by poor waste management, as natural fibers are fully biodegradable. [5.]

MFPs have many attributes suitable for packaging, some even better than comparable plastic products [4]. Single-use packaging is highly related to food: vegetables, fruits, and meals are often packaged to preserve them and prevent them from spoiling. Increasing the share of food packaging made of molded fiber material is especially relevant in fast food and takeaway portions. [6.] However, one important food-related limiting factor is still unsolved. Plastics are still superior to molded fiber in terms of oil and grease resistance (OGR) ability. Developing molded fiber OGR properties is critical for it to successfully replace plastic disposable packages. A first important step for creating OGR properties is to understand how oil and grease go through fiber-based products and what phenomena are behind them. Structure and source of fiber, density, and thickness are properties of plain molded fiber material that affect OGR [7]. Different chemical additives can be used to increase OGR, the most effective of them currently being fossil-based. [3.]

One thing to consider is the role of oil in the OGR of fiber material. The key factor is oil viscosity, which is dependent on both the temperature and the composition of the oil. [7.] Vegetable oils consist mostly of triglycerides, which contain certain fatty acids typical of the plant its extracted from. Fatty acids can be saturated or unsaturated depending on the plant species. The more saturated the oil, the more double bonds it contains in the fatty acid, which lowers the viscosity. [8.]

The aim of this study was to get more information on how different vegetable oils affect molded fiber products' oil resistance at different temperatures. Testing was done with an automated customized system that has similarities to the ASTM-F119 standard. The test used in this thesis was focused on oil permeation time measurements. Samples for the test were produced at Kemira’s R&D center. Vegetable oil viscosities were also measured at the same temperatures and compared to the results of the oil permeation times. The outcome of the study can be used to decide if it is beneficial to use a certain oil over others in the laboratory test when developing molded fiber products.

2 Molded Fiber

Molded fiber product (MFP) can be made of almost any natural fiber source, such as wood, bamboo, bagasse, straw, or any plant waste stream containing suitable fiber [6;9;10;11]. Fibers from different sources might have different mechanical properties, which may limit the options for production [9]. Recycled fiber can also be used, but it usually requires further chemical processing, for example deinking [12]. Recycled fiber sources are commonly old newspaper (ONP) and old corrugated cardboard (OCC) packages [10]. Recycled fiber was previously burned for heat and energy or simply dumped in landfills. The cycle of fiber is not endless since a single fiber can be used 5 to 7 times maximum before its quality reduces too much to be used for anything but energy [5]. Virgin fiber is also needed to meet the quality needs of high-end MFPs. To keep the transportation cost low, companies producing molded pulp products prefer to use a local source of fibers, whether it is wood, non-wood material like bagasse, or recycled newspaper [12].

2.1 Molded Fiber Packaging

A large part of all petroleum-based plastic produced is used for packaging [13]. Molded fiber products can replace plastic packages, which would have a significant impact on plastic waste pollution. The possibilities of what can be made out of molded fiber vary. [9.] Molded fiber products are used for egg trays, plant pots, cushioning material in electronics, support packages with heavy non-fragile products like auto parts, clinical healthcare products, and disposable dishes or trays [10]. Nowadays, there are three main product categories for molded fiber products, which are food packages, disposable clinical healthcare products, and support cushioning for electronics [10]. Packages made of molded fiber require different properties depending on use. Desirable attributes for molded pulp packages should also be at the same level as their similar plastic ones. For supporting purposes, MFPs meet the requirements and have

good shock absorption and a sturdy shape. Disposable dishes and trays need barrier properties to keep the content in and to prevent material migration between the content and the container. The desired properties also include cost-related properties such as production and transportation costs. The end product, therefore, needs to be both lightweight and stackable. [14.]

2.2 Molded Fiber Production: Category Types I-IV

The molded fiber manufacturing process can be divided into four groups according to the International Molded Fiber Association (IMFA). Different types are defined depending on molded fiber product wall thickness, how it is made, and the purpose of the final product. A schematic diagram of the categories is presented in Figure 1.

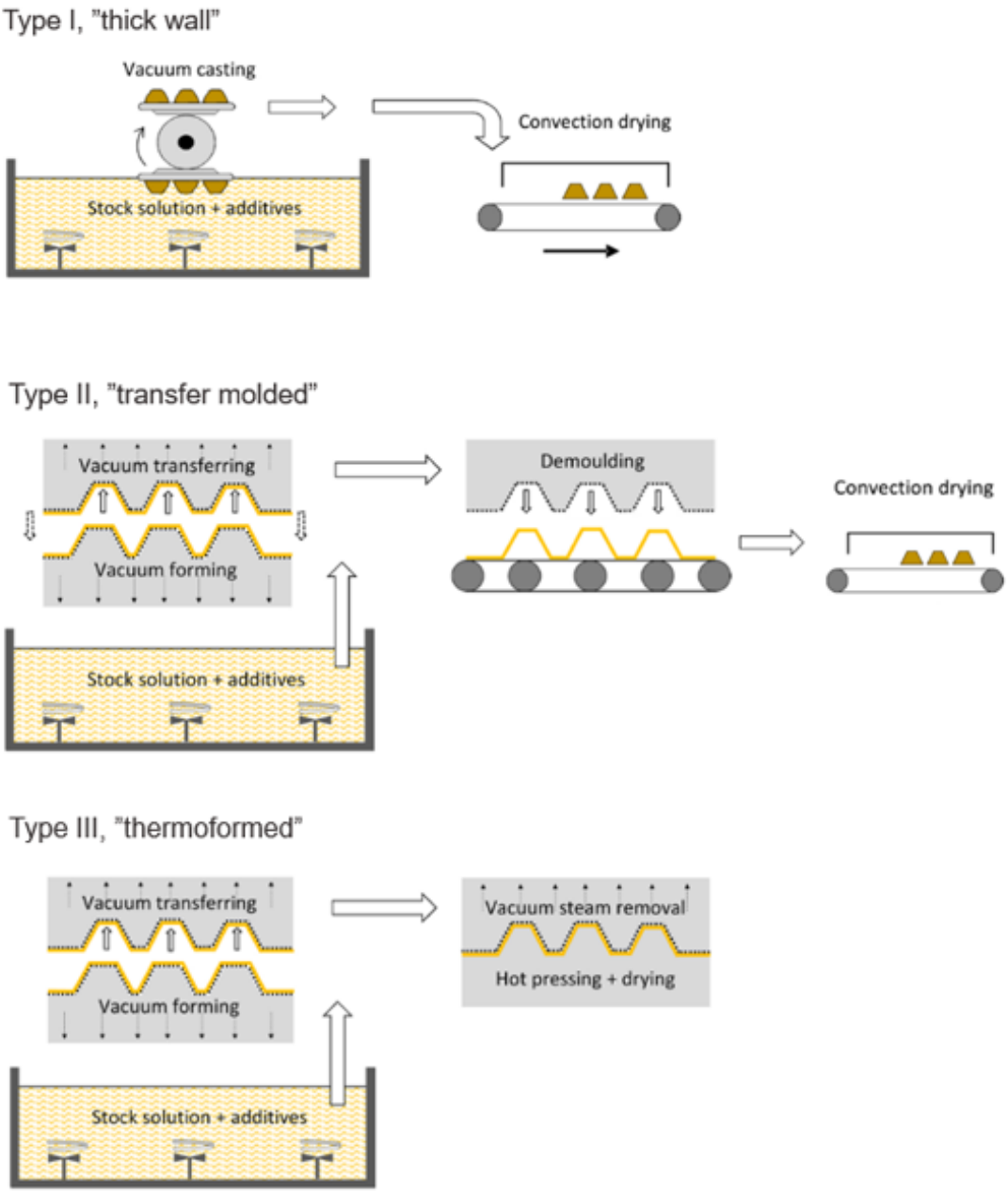

Figure 1. Schematic diagram of molded fiber manufacturing methods, types I-III. Adapted from [3, 33].

Type I

The first category is “thick wall” molded fiber. Wall thickness is usually 6 mm to 8 mm. It is strong and resilient and used as a support and protection for heavy-duty products and non-fragile heavy items such as vehicle parts and furniture. It can also be used as plant pots as the material is fully biodegradable. The

manufacturing process includes dipping a mold in the pulp tank, then a long vacuum is applied, and excess water is removed. Fibers create a layer on top of the surface of the mold. After that, the mold is lifted, and the product is released from the mold and dried in an oven. The final product has a fine, smooth inside and a rough outside. Pulp consistency is usually 1-2% with long vacuum cycles. Raw material is also typically recycled fiber like double-lined kraft (DLK), OCC, ONP, or a mix of different fiber sources. [3.]

Type II

The second type, "transfer molded", is the category that includes the largest volume of molded fiber products produced globally. There is a variety of specialized molding machinery solutions to make type II MFPs. They have different advantages depending on the final product, quality, and production rates. It typically uses one formation mold and one transfer mold. Final product wall thickness is typically between 3 mm and 5 mm. The most common drying method is oven drying. In Type II MFP, the smoother side is even smoother compared to Type I. Pulp fiber content is usually 1-2%, leaning towards 1-1.2% pulp consistency. Raw material can be virgin fiber, DLK, OCC, ONP, or mixed fiber. Typically, type II category products are used to produce egg trays, protection for heavy-duty products similar to type I, and finer support packages, office equipment, drink trays, electronic cover packages, and hospital disposal packages. [3.]

Type III

The third type, “thermoforming”, uses even more advanced techniques. Multiple heated molds define the third category. A fiber layer is formed in the first mold as in the previous categories. When the first mold is lifted from the tank, a second mold of the opposing shape is pressed against it. The heating system and vacuum are applied to both molds. Pressure, heat, and vacuum quickly dry the fiber cast, so no oven drying is needed. Final product surfaces are smooth, and wall thickness typically varies between 0.4 mm and 1.5 mm. Thermoforming creates well-shaped and detailed, dimensionally stable products. Using two molds pressed together has little to no shrinkage problems.

Type III products have the highest potential to compete and replace plastic packages. Pulp consistency is typically 0.5% to 1%, and the production rate is 10-30 seconds. Raw material needs to be higher quality than for type I or type II, thus, virgin wood and non-wood fiber are used. Common mold materials are steel or aluminium. Aluminium heats faster and releases energy quicker, but steel is more durable. Type III products have an advantage in transportation since the products are more valuable, thinner, and weight less. The more tightly packed valuable products can be, the more profitable their transportation becomes from longer distances. [3.]

Type IV

Category IV is basically a type I-III product that is modified or enhanced in some way. For example type II product is pressed to improve its strength and then printed, coated, or labelled. The ways final products are modified could be for example die cutting, printed information, added enhancements like added barrier properties, or spray coating. [3.]

3 Edible Oils

Since one of the important requirements for molded pulp products is oil and grease resistance, it creates questions like what we need to know about oils and greases generally. Vegetable oil's main components, lipids, are a wide group including all kinds of fatty acid molecules. The simplest lipids are fatty acids, which are attached to glycerin to form triglyceride as shown in Figure 2 [15]. Fats and oils can be divided by their solidity at room temperature. Fats are usually solid, and oils remain liquid at room temperature. Greases, byproducts derived from fats and oils, usually stay semi-solid at room temperature.

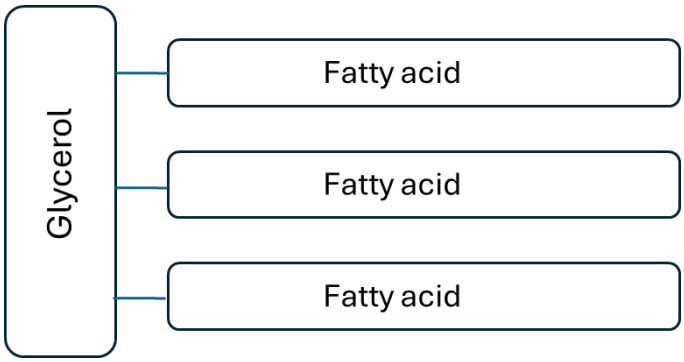

Figure 2. Diagram of a triglyceride.

As seen in Figure 3, fatty acids can be divided into saturated, monounsaturated, and polyunsaturated depending on the number of double bonds in their structure, and vegetable oils are usually a mix of these. The fatty acid profile in pure vegetable oils depends on the plant itself, several climate and environmental factors, and the way the oil is extracted [16]. An important environmental factor is temperature, which commonly leads to a case where plants growing in a colder environment produce more unsaturated fatty acids than plants growing in a more temperate climate. [17.]

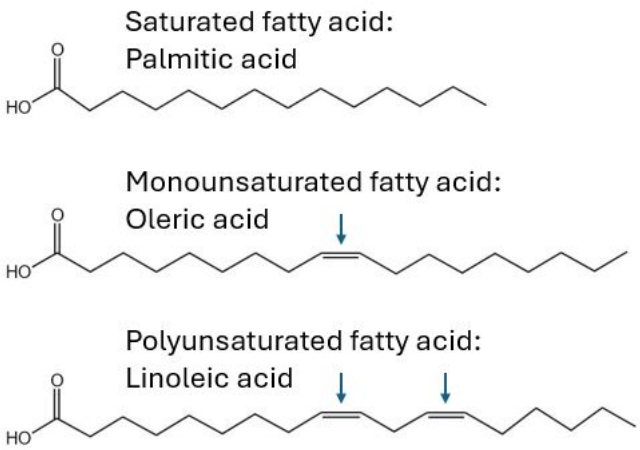

Figure 3. Diagram of fatty acids with different degrees of saturation.

The food we consume contains various sources of energy, the largest of which are lipids. There are lipids present in some form in almost any animal and plant-based food, making it an important part of the diet. Fatty acids are needed as building blocks for cell membranes, hormones, vitamins, and as an energy storage. Some of the fatty acids the body needs can be produced by breaking down carbohydrates or proteins. There are, however, some essential fatty acids that the human body cannot produce but are vital to body functions. These essential fatty acids are abundant in some vegetable oils, for example, omega-3 in canola oil. [15.]

3.1 Oil Properties Related to Oil Permeation

Carbon chain length is related to the viscosity of the oil. The longer the chain, the greater the viscosity. Carbon chain length also affects the melting point. Shorter carbon chains have a lower melting point. The melting point is also lowered by the degree of saturation of the fatty acids in the oil. The more double bonds a fatty acid has, the lower the viscosity and the melting point. The number of double bonds affects even more than the carbon chain length, as can be seen in Table 1. As previously stated, many vegetable oils are a mixture of different fatty acids, which is also influenced by the species' growth environment, making the properties of the oils variable. [8.]

Table 1. List of fatty acids of different saturation states, source, and melting point. Adapted from [15, 220].

Common name	Symbolic representation	Common source	Melting point (C°)
Saturated fatty acids			
Caprylic acid	C8:0	Coconut oil	16.7
Capric acid	C10:0	Coconut oil	31.6
Lauric acid	C12:0	Coconut oil	44
Myristic acid	C14:0	Butter, coconut oil	52
Palmitic acid	C16:0	Palm oil, butter, lard	63
Stearic acid	C18:0	Beef tallow, cocoa butter	70
Monounsaturated fatty acids			
Palmitoleic acid	C16:1, Δ^{9}	Palm oil	-0.5
Oleic acid	C18:1, Δ^{9}	Canola oil, olive oil	13
Erucic acid	C22:1, Δ^{9}	Rapeseed oil	33.4
Omega-6 polyunsaturated fatty acids			
Linoleic acid	C18:2, $\Delta^{9,12}$	Soy, safflower, corn oil	-9
Arachidonic acid	C20:4, $\Delta^{5,8,11,14}$	Minor constituent of foods	-49
Omega-3 Polyunsaturated fatty acids			
Linolenic acid	C18:3, $\Delta^{9,12,15}$	Linseed, parilla oil	-17
Eicosapentaenoic acid	C20:5, $\Delta^{5,8,11,14,17}$	Fish oils	-53

3.2 Selection of Vegetable Oils for the Tests

The practical part of the study was to measure the permeation times of different oils. The oils were selected based on their differences in global consumption, composition, availability, health aspects, and sustainability. Statistics on the global production of vegetable oil revealed that some oils are more common than others. [18.] Climate and growing environment greatly affect which species thrive best and whether their cultivation is profitable. Rapeseed oil is widely produced in colder climates, and palm oil in warmer environments. Table 2 shows the oils most consumed globally and the regions where they are most common. [18.]

Table 2. The most produced vegetable oils (2022) globally and in different regions [18].

	Production (Mt)				
Vegetable oil	World	Asia	Africa	America	Europe
Palm	78.87	68.86	3.48	5.68	
Soybean	58.05	21.75	1.63	30.55	4.11
Rapeseed	26.69	9.17		5.06	11.85
Sunflower	20.26		0.80	2.03	15.27
Palm kernel	8.29	7.27			
Groundnut	5.20	3.15	1.56		
Cottonseed	4.03				
Coconut	3.16				
Maize	2.93			1.51	0.39
Olive	2.74		0.57		1.48

Regional differences played a role in the decision-making process. Most of the oils produced in a region are also the most consumed there, and it would be wise to conduct tests with the oil that is statistically most likely to be used in that

geographical area. [18.] The difference in fatty acid composition was also considered an important factor in the possibility of obtaining more comprehensive results. The selected oils to conduct permeation tests were palm oil, soybean oil, canola oil, and olive oil.

Palm oil is the most used vegetable oil worldwide and is particularly dominant in the Asia region. It is also one of the most saturated oils there is. Soybean oil is also widely used and common in every region, especially in the Americas. Its composition is somewhere in the middle of having an average amount of unsaturated fatty acids. Rapeseed oil was listed in the statistics as the third most used. It is noteworthy that canola oil was not on the list, which could be because canola had not been distinguished from rapeseed oil. Canola and rapeseed, *Brassica rapa subsp. oleifera* and *Brassica napus subsp. oleífera* are two different species that resemble each other in appearance [19]. They are commonly thought of as one species instead of two. They also have very similar nutritional content, so the choice between the two should not significantly affect the test results [19]. Olive oil is not very common outside the Mediterranean area. Even in Europe, it is fourth abundant. Olive oil is interesting because it is considered to be an important part of a healthy Mediterranean diet [16]. It is commonly used in salads as a dressing, and it has a unique taste. The selected vegetable oils and their composition are shown in Table 3, including commercial brand information for comparison.

Table 3. Composition of selected vegetable oils and comparison with brand product information. Adapted from [15, 221].

Fatty acid composition (%) of common dietary fats and oils.

	Saturated				Monounsaturated		Polyunsaturated	
	4:0 -12:0	14:0	16:0	18:0	16:1	18:1	18:2	18:3
Canola oil			4	2		62	22	10
Olive oil			13	3	1	71	10	
Palm oil		1	45	4		40	10	
Soybean oil			11	4		24	54	7

Composition of the oils according to the brand labels (g)/100g of content.

	Saturated	All unsaturated combined
Canola oil	7	93
Olive oil	16	84
Soybean oil	15	85
Palm oil	49	51

4 Oil and Grease Resistance of Molded Fiber Product

The pulp from which molded fiber products are made consists mainly of cellulose, which is hydrophilic by nature. Cellulose molecules bond together under pressure and form a network of fibers [7]. SEM image of molded fiber products' surface is shown in Figure 4. As can be seen from the SEM image of the surface, the fibers stack on top of each other in a random orientation, forming a porous material.

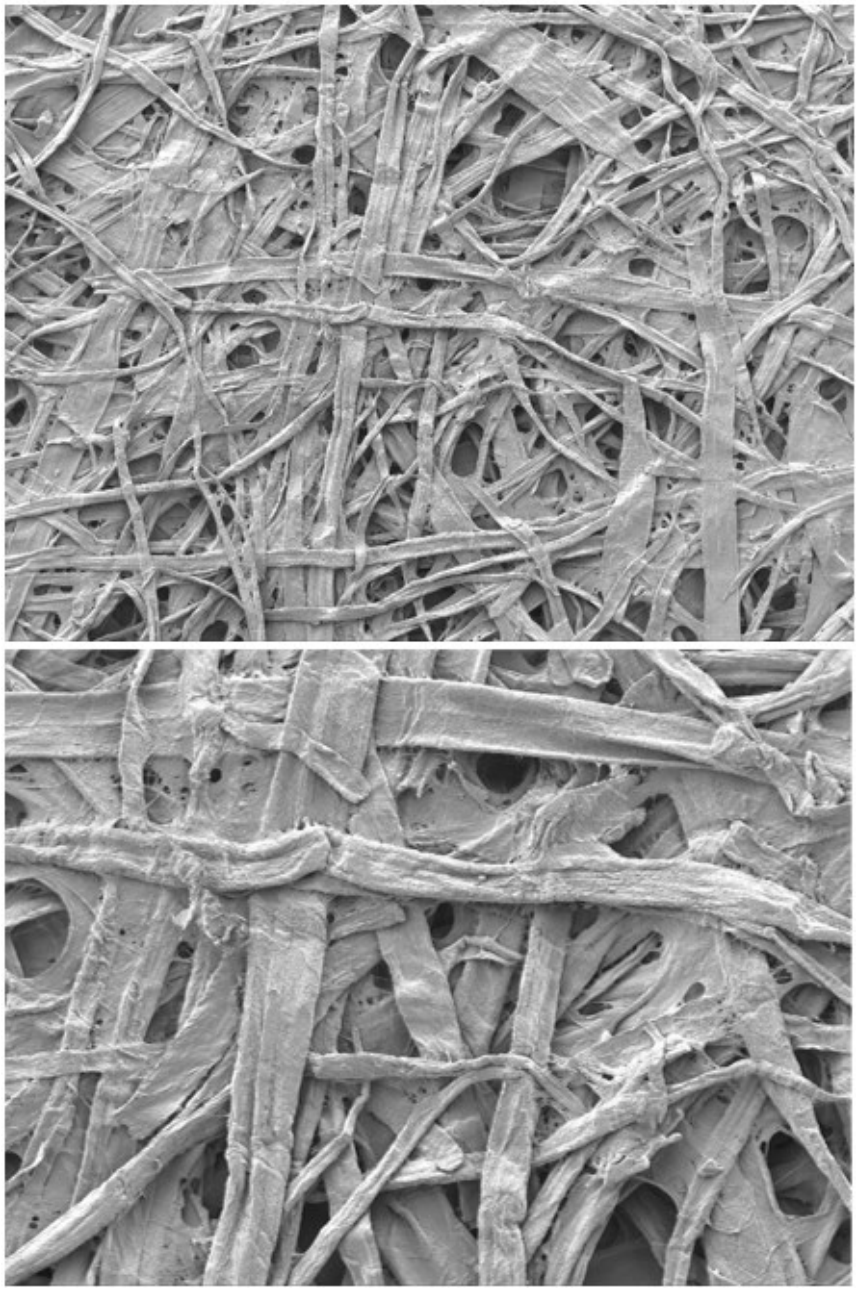

Figure 4. Generic SEM image of the surface of a molded fiber product. Image magnification x100 at the top and x300 at the bottom.

There are two ways to increase the oil resistance of fiber materials. Either creating a physical barrier or a low-energy surface that can effectively repel oil. Reducing the size and number of pores can effectively reduce the oil diffusion rate through fiber-based products. A physical barrier can be created inside the fiber-based product or on the surface. Single or multiple coating layers can be

applied to the surface, making it impermeable. Internally, the increase of oil resistance is done by filling the voids between the fibers with additives. [7;8.]

Reducing the surface energy results in an oil repellent surface. Low surface energy prevents wetting from happening, which is needed for diffusion. The properties of the liquid and surface roughness also affect the wetting phenomena. In practice, only fluorochemicals can achieve a sufficiently low surface energy to form an oil-repellent surface. [7;8.]

The source of fibers may also have a minor impact on oil resistance, although mainly affecting other properties such as mechanical strength and water absorption. Mixing hardwood and softwood may result in better water and oil barriers. [7.] Molded pulp products' 3-dimensional shapes limit coating techniques compared to paper products. Therefore, manufacturers would prefer low investment solutions like furnish additives rather than spending money on a spray coating system.

Some biodegradable additives have been successfully tested for enhancing oil resistance. One such additive, chitosan, made from shellfish, is proven to increase the oil resistance capabilities of MFPs [6]. Cellulose nanofibrils and microfibrillated cellulose created excellent oil barriers as well [20;21]. A few non-toxic and degradable additives with oil and grease resistance are listed in Table 4.

Additive	Safety and function
Cellulose nanofibers (CNFs)	Non-toxic. CNFs significantly improve strength and barrier properties and reduce linting
Microfibrillated cellulose (MFC)	Origin:natural plant fiber/non-toxic. Increase the barrier and mechanical properties
Chitosan	Sodium alginate. safe for food packaging. Improve oil-repellent properties
Polydimethylsiloxane (PDMS)	Non-toxic for humans. PDMS offers good water- and oil-repellent properties

5 Materials and Methods

5.1 Sheet Forming Process

Hand sheets for the practical part of the work were handmade locally at the Kemira R&D center. The first step was making a batch of the raw material, furnish. Usually, the furnish-making process involves several steps where the concentrated cellulose pulp stock is made by mixing dry pulp sheets and water. There was already suitable pulp stock to be used, so the first step was skipped, and some work time was saved. The next step was diluting the pulp stock to the right consistency, which was approximately 0.4% of solids according to plan. Target consistency, 4 g/L, was important for optimal sheet formation. Too high a consistency would not form high-quality hand sheets, and too low a consistency would be inconvenient to handle. Furnish composition was Schopper-Riegler (SR) 24-26 70% BHWK (Bleached Hardwood Kraft) +30% BSWK (Bleached Softwood Kraft) furnish mix, and batch size was 120L. Pulp stock was diluted using tap water. Furnish consistency was measured according to ISO standard 4119:1995 [22].

After the proper consistency was confirmed, work continued by adjusting the furnish chemical properties similar to factory conditions. Conductivity was set to

300mS/cm by adding a triple salt mix (70% calcium acetate, 20% sodium sulfate, and 10% sodium bisulfate). Sodium hydroxide was also added to settle the pH to 7.5. Zeta-potential measurements were also done of the furnish. Target zeta potential was between -10 mV and -15 mV. Zeta potential was adjusted by adding a cationic retention chemical. Several tests were made with different dosages of the retention chemical to find the right amount to be used. Conductivity and pH were required to be checked daily and adjusted if needed.

Hand sheets were produced with the Rapid Köthen-sheet former (RK). A few test sheets were made first to find the right volume of furnish to have a 400 grams/m^2 hand sheet. Furnish was pumped into a smaller container, which was placed under a mixer. Chemical additives were added one by one, mixing for 90 seconds after each chemical. The chemicals used to enhance the OGR properties of the sheets were mixed in the order shown in Table 5.

Table 5. Chemical additives and doses (dry chemical additive/dry pulp).

Alkyl Ketene Dimer (AKD)	Sizing agent. Adds hydrophobic properties and enhances water resistance.			
OGR-chemical	Adds oil and grease resistance.			
Retention chemical	Enhances chemical retention and speeds dewatering, which reduces the energy needed for drying the MFP.			
Sample	AKD	OGR chemical	AKD	Retention chemical
KP 1, reference sample	-	-	-	0.9 kg/t
KP 2	1 kg/t	20 kg/t	-	0.9 kg/t
KP 3	-	20 kg/t	1 kg/t	0.9 kg/t

Then the furnish with additives was poured into the RK-sheet former, and the automated sheet making program was started. After the program was finished,

a wet fiber cake was manually dried for 45 seconds with increased vacuum. The wet sheet was weighed and then carried to be cold pressed using a modified L&W type 5-1 sheet press. The wet hand sheet was placed between insulated ceramic tiles. Under the sheet were also added 3 blotting papers for moisture absorption and a rubber mat at the top for protection. Cold press times were set to 8 seconds to reach similar dryness as factory conditions. After cold pressing, the sheet was weighed again to ensure that enough water had been removed. The hand sheet was then hot pressed for 45 seconds using the same press but without the insulating tiles. A hot pressing temperature of 185°C was used under vacuum, and two metal forming wires were placed under the sheet prior to pressing. The modified press closely follows the conditions and materials of the thermoforming process. The hand sheet was weighed again after being pressed. All of the average sheet weights and dry solid contents are shown in Figures 5 and 6.

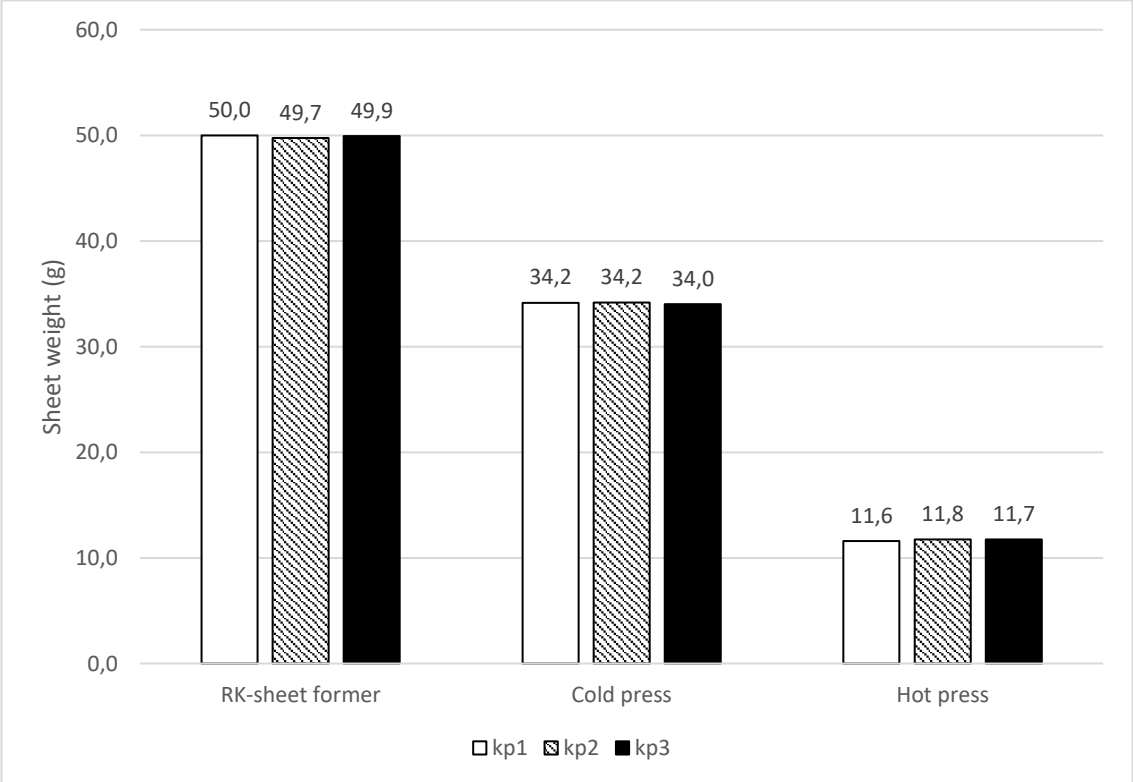

Figure 5. Sheet weight at all phases. Averages of KP1, KP2 and KP3.

The hand sheets were hot pressed completely dry, as seen in Figure 6. The reason behind it is the requirement for maximum flatness of the hand sheet needed for oil testing. Even a small amount of moisture is enough to twist the hand sheet slightly after pressing.

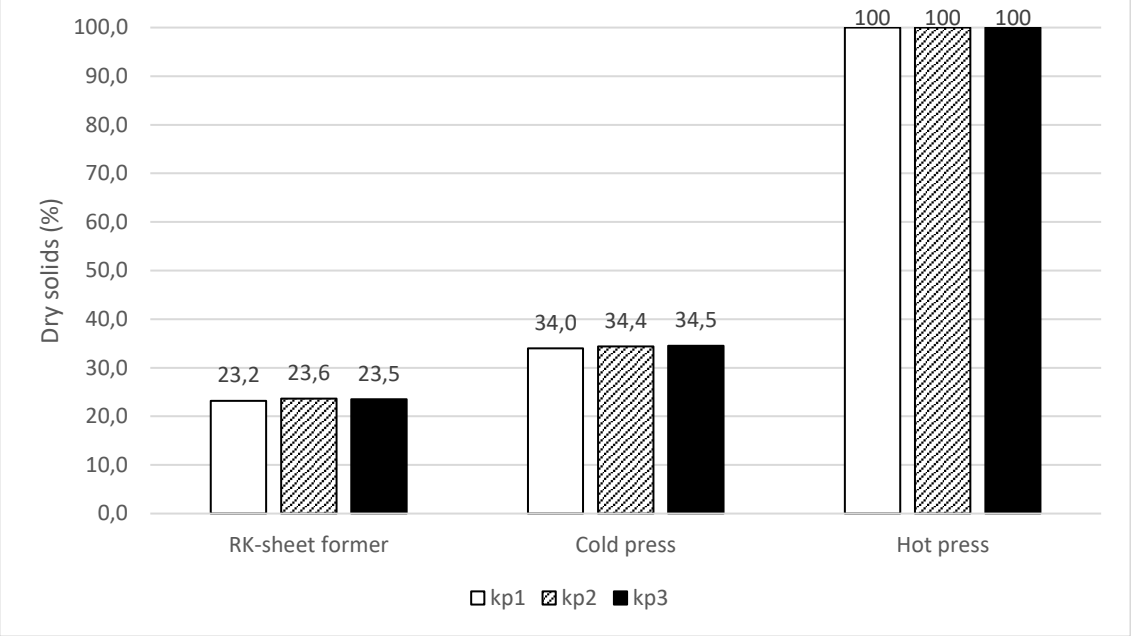

Figure 6. Dry solids content at all phases. Average values at KP1, KP2, and KP3.

5.2 Physical Measurements of the Sheets

To ensure the similarity of the hand sheets, a few basic measurements had to be conducted. Necessary physical measurements are listed in Table 6. Hand sheets were placed in the temperature and humidity-controlled room (23°C & 50% relative humidity) so the relative humidity change would have a minimum effect on any measurements taken.

Table 6. Basic physical measurements, equipment used, and the followed ISO standards.

Grammage	The hand sheet was weighed using an analytical balance	ISO 536
Thickness	5 measurements per sheet, L&W micrometer	ISO 534
Gurley and Bendtsen air permeability	L&W Air Permeance Tester	ISO 5636-3
Bendtsen roughness topside	L&W Bendtsen Roughness Tester	ISO 8791-2

Oil permeation speed is affected by the sample material's thickness and density. All sample materials should be similar to each other to minimize the deviation of the oil permeation tests. Grammage was measured by weighing the sheet and then dividing it by the area of the sheet. Thickness was needed for the density calculation. Thickness was measured at the centre of the sheet and at the corners, similar to a dice number of 5.

Gurley and Bendtsen air permeability were measured using the L&W Air Permeance tester. The high amount of air going through the sample could give information about how porous the material is. Results of the air permeability could also hint of structural defects of the sheets.

Bendtsen roughness measures the smoothness of the sheet surface. It is usually measured from the test side, in this case, the smoother side without wire mesh marks. A smoother surface is related to the wetting phenomena, but it mostly tells how well the sheet is formed.

5.3 Automated Oil Permeation Detection Test

The focus of this thesis was on measuring oil permeation times of selected oils on molded fiber samples. The commonly used oil permeation test is ASTM F119. It is a relatively simple test, but it has some weaknesses. It is best suited for thin paper samples rather than thicker molded fiber sheets. If the tested samples have good OGR properties, the test could take several hours up to a day to finish. Detection of the permeation point is evaluated by the tester, and it can vary depending on the skills and experience of the tester. The method used in this thesis work aims to minimize the user error factors, leading to fewer work resources needed and more accurate results.

Kemira uses a method for oil penetration measurements, in which oil penetration is monitored by a camera connected to a computer in a closed environment. This automated setup was developed and built by Labra AI. The automated test is done at 40°C in a large cabinet-sized oven with cameras installed on the bottom to observe samples on shelves above them, as shown in Figure 7. A heated oven was chosen so the atmosphere temperature can be controlled, and the cabinet also provides stable lighting conditions for better detection. Cameras are controlled by a specific program installed on the computer unit next to it. Samples are placed on a metal frame shelf, which has spots for twelve 20 cm x 10 cm glass plates. The side in contact with the sample of the glass plate is coated with a thin layer of white cellulose. When the oil permeates the sample, it absorbs into the cellulose. Coloured oils are used to enhance the detection limit. The cameras below are set to register tiny, coloured oil spots. To avoid false detections from, for example, hand movements in the oven, the first detection of oil needs a second detection to confirm the permeation and end the test. Cameras are also set to take forced pictures at certain time points of the test. The first picture is taken when the test has been on for 10 minutes, and the second after 120 minutes. Also, the first detection of oil permeation and the test end pictures are taken. All pictures and

timestamps are saved to a specific folder on the computer. The test can be left running overnight or for weekends and needs no supervision after the test is on.

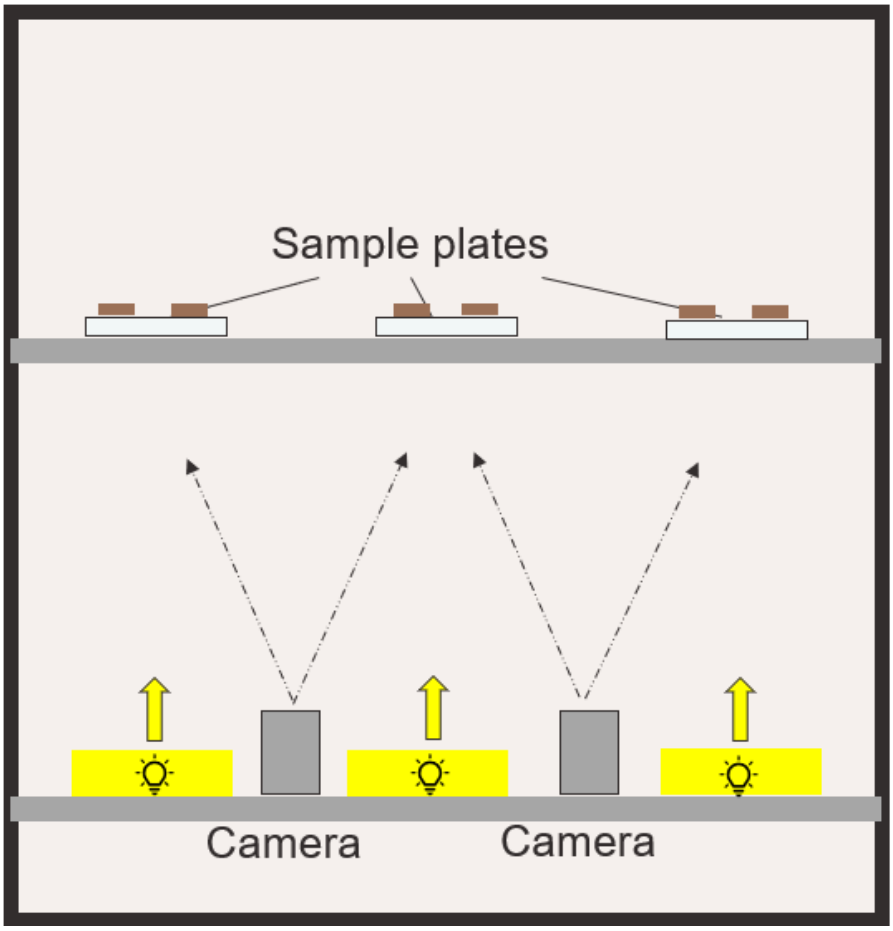

Figure 7. Schematic of the automated test.

The automated oil permeation detection test was originally designed for paper samples, and the molded fiber tests were allowed to be done along with the paper sample tests. The situation resulted in some limitations for the conditions. Molded fiber samples measurement temperatures were planned to be 5°C, 23°C, and 60°C, but for the paper test measurements, the inside temperature was set to 40°C permanently. The starting temperature of the oils was estimated to create sufficiently different conditions. Additionally, for the 5°C tests also the samples were also cooled with the oil to create fridge-like conditions and to study how that affects the permeation times. All the samples and oils are listed in Table 7.

Table 7. Oil test plan.

Temperature		Number of measurements				
		Canola oil	Olive oil	Soybean oil	Palm oil	Salad dressing
5°C	KP1	-	3	-	-	3
	KP2	7	7	7	7	5
	KP3	7	7	7	7	5
23°C	KP1	3		-	-	3
	KP2	7	7	7	7	5
	KP3	7	7	7	7	5
60°C	KP1	-	-	-	3	-
	KP2	5	5	5	5	-
	KP3	5	5	5	5	-

Colored oils were prepared a few days in advance to ensure proper mixing of the dye. Sudan IV red dye was chosen based on its good oil solubility. Oversaturated solutions of each oil were prepared to ensure the color was deep enough for the camera to detect.

Precut and selected representative samples were checked visually to be suitable for tests. A cut layout of the sheet was planned to maximize the number of samples, and it is shown in Figure 8. Samples cut from the corner of the hand sheet usually had some uneven corners that would not affect the measuring area but needed to be removed to make the samples as flat as possible. The sample must be fully in contact with the cellulose surface of the glass plate to ensure accurate detection of the absorbed oil. The measurement side of the samples was the smoother top side without the wire mesh marks.

700 µl of colored oil used in the test requires a mold to hold it in the measuring area. Rubber molds were cut into a square shape, and round areas were die-cut in the middle where the oil is added. The molds were carefully greased on

the bottom side with a high vacuum grease, and placed in the middle of the sample, grease side facing the sample. Samples with the rubber mold attached to them were placed on the glass plates. Two metal rings, with a combined weight of 75 grams, were placed on the rubber mold to provide extra support by pressing the rubber mold and keeping the grease attachment tight. Each plate could hold three samples, but only two were used because of the weight limitation caused by the additional metal rings. Sample preparations 1-3, step-by-step pictures are shown in Figure 8. Glass plates and samples were placed in the oven on the shelf, and the necessary sample data were entered into the computer. The proper amount of oil was added to both of the samples, and then the test was started from the program.

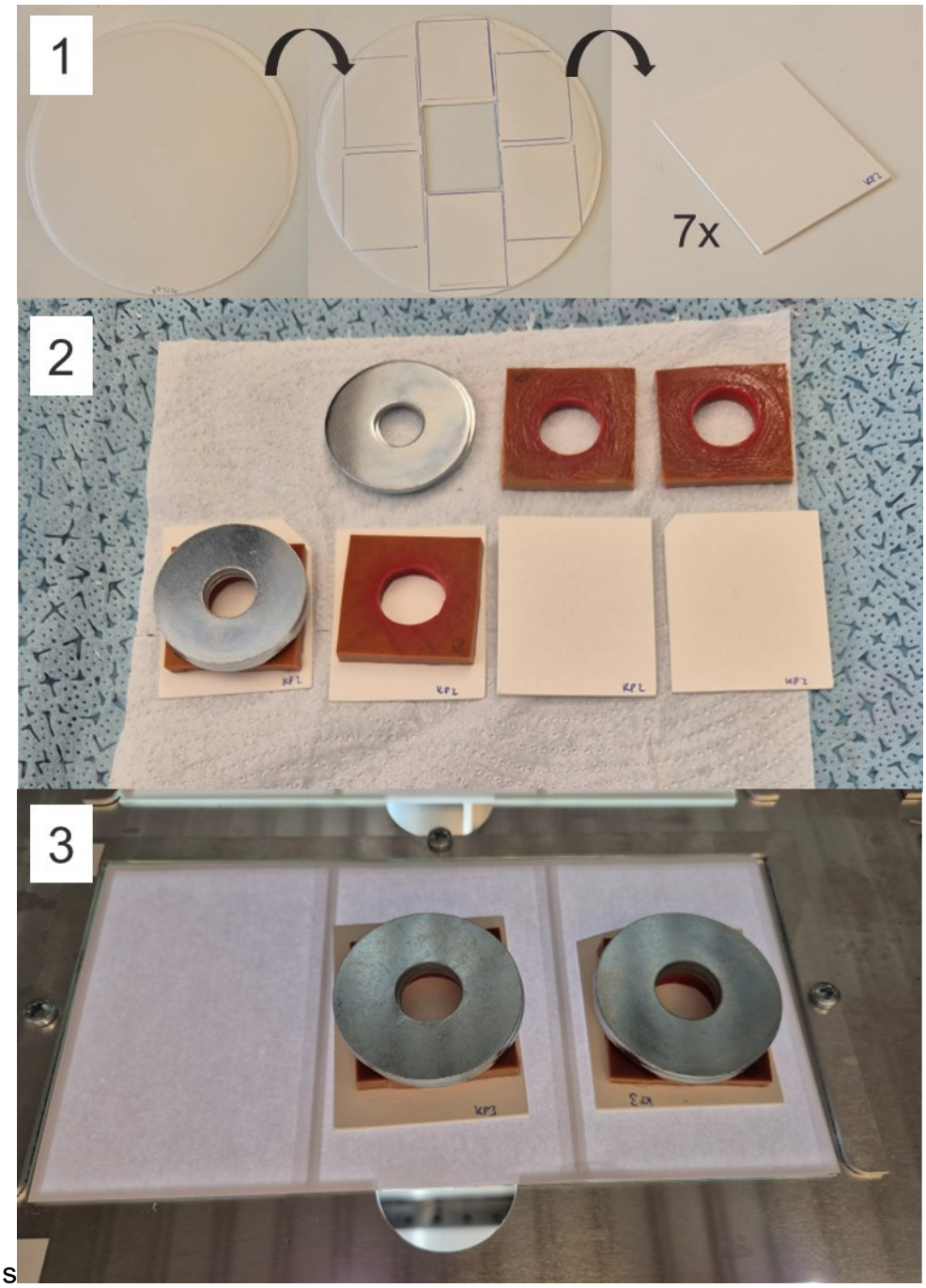

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Figure 8. Sample preparation steps. 1:Cutting diagram of the hand sheet. 2: Rubber molds are attached to the samples. 3: Samples on the plates are ready for oil addition and measurement

Oil temperature changes over time towards room temperature, so the test should be done as quickly as possible. To speed up testing, each oil was divided into two bottles so that one was ready in the oven or fridge, while the other was being used. An insulated container was used to keep oils at the

correct temperatures when carrying them to the test location. No more than two plates were started using the same oil bottle to prevent the oil temperature from changing too much. Palm oil turned completely solid at 5°C, making pipetting it impossible. 700 µl of liquid palm oil was weighed 5 times using an analytical scale. The average of the 5 results was approximately 0.6 g, and that amount was used for the 5°C tests.

5.4 Oil Viscosity Measurement

Viscosity is dependent on the oil temperature and composition. To put it simply, the higher the temperature rises, the less viscous the oil is. Viscosity might correlate with the oil permeation results, so it was necessary to measure it.

Viscosity profiles were measured for all oils at different temperatures. Measurements were conducted using Anton Paar Modular Compact Rheometer 302 with a double gap measuring system. Shear rates were set to measure 0.1 [1/s] – 100 [1/s] with 22 interval datapoints. Chosen measuring temperatures were 5°C, 23°C, 40°C, 60°C and 80°C. Measurements were performed in order from highest to lowest, starting with the highest temperature. The profiles of all measurements were reviewed, and a suitable shear rate was picked. Profiles seemed stable and constant between 1[1/s] and 100[1/s]. The viscosity value of the oil was selected at a shear rate of 10[1/s].

6 Results

6.1 Properties of the Hand Sheets

The average physical measurement results of the hand sheets are presented in Table 8. Grammages of the samples KP1, KP2, and KP3 are very close to the target of 400 g/m^2. One hand sheet of the KP3 group was discarded due to an error in pouring less furnish than required to the RK-sheet former. Grammage of

the discarded sheet was around 10% less than the rest. Otherwise, the target grammage was reached, and the hand sheet production was successful. Detailed measurements are listed in Appendix 1.

Table 8. Average results of grammage, thickness, density, and bulk for KP1, KP2, and KP3.

	Grammage	Thickness	Density	Bulk
Sample	g/m^2	µm	g/cm^3	cm^3/g
KP1	401	574	0.70	1.43
KP2	402	579	0.69	1.44
KP3	401	576	0.70	1.44

Roughness results had variation. Both KP2 and KP3 roughness seem to be less than KP1. There were no significant differences in air permeability between the samples. Average results are shown in Table 9.

Table 9. Average results of Bendtsen Roughness and Bendtsen & Gurley Air Permeation.

	Bendtsen - Roughness TS	Air Permeability Bendtsen	Air Permeability Gurley
Sample	ml/min	ml/min	s
KP1	2430	243	49.5
KP2	2050	246	48.9
KP3	1880	244	49.3

6.2 Oil Permeation Test Results and Interpretation

Hand sheets produced manually sometimes have defects. As shown in Figure 9, at 15 minutes from the start of the test, there is a single spot in the middle of the test area. Formation of the sheet varies even if every step in the production of the sheet is conducted carefully. There might also be some small particles from the equipment and the machinery that can remain unnoticed until the visual check of the finished sheet. Small particles can act as a quick pathway for oil and may affect the permeation time results. It is also important to keep in mind that sometimes the additives can just perform badly and tend to spread unevenly, creating also defectives like pinholes. In the sample shown in Figure 9, a pinhole is visible through which oil has permeated.

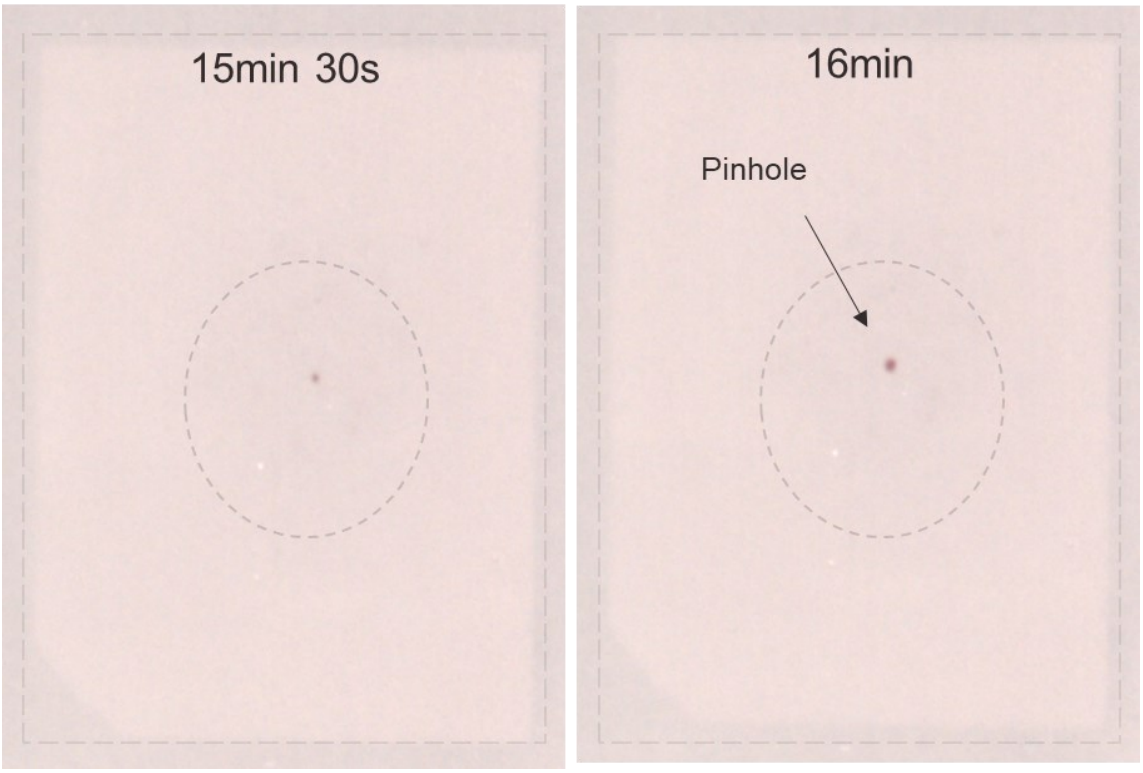


Figure 9. Example of a sample with fast oil permeation through a single pinhole.

Typically sample with a permeation point from a single pinhole just shows the weakest spot and is not very representative. A sample with a pinhole usually has a faster permeation time than replica samples. At least several replicate

samples are needed to get reliable information about the OGR capabilities. A good example of a well-formed hand sheet is shown in Figure 10. Oil permeation is happening evenly in many different spots. The result usually reflects the oil resistance of the sample well. Figures 9, 10, and 11 are pictures that the automated camera had taken during testing. The left side is the first detection recognized, and the right side is when the test ended, and a picture was also taken. The intensity of the spot color is slightly increased, as can be seen from the pictures.

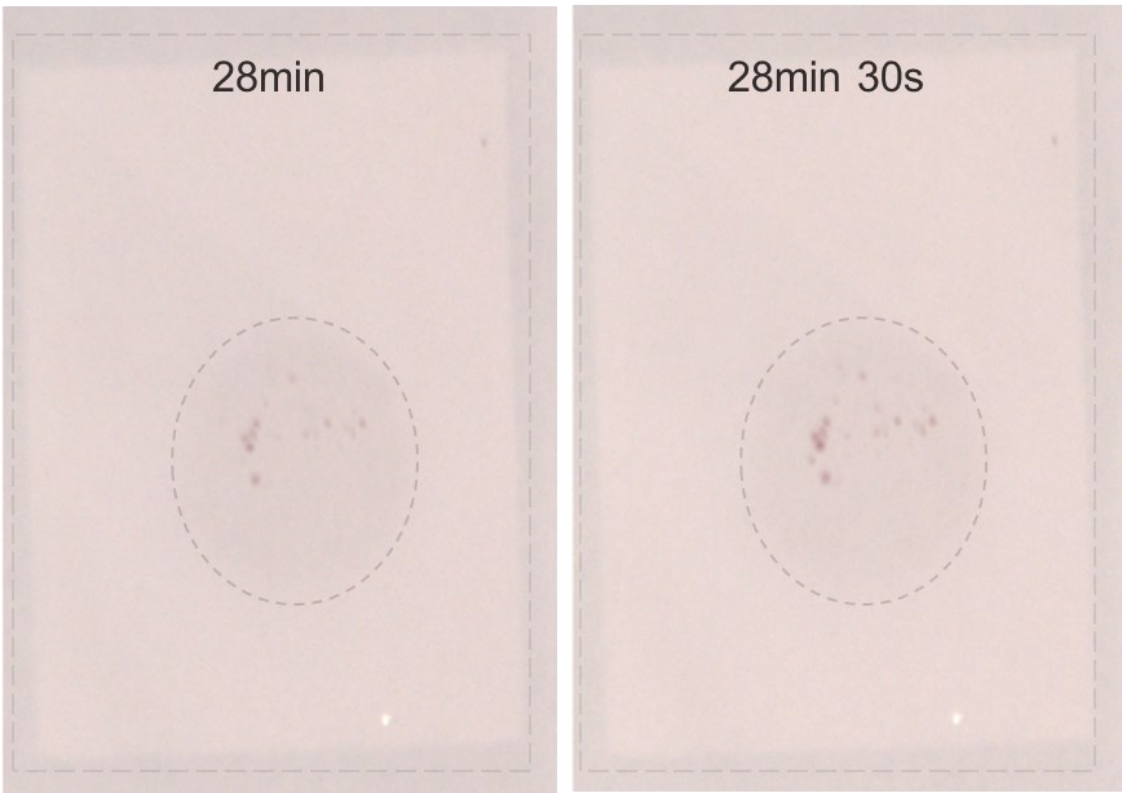


Figure 10. Example of a sample where oil is evenly permeated. Formation of the sample is good.

Reference samples were also tested to point out how fast the oil would go through without any OGR-chemicals. Figure 11 shows a time-lapse of a reference sample. The colored oil is through in seconds, and after 10 minutes, most of the oil is spread, covering a large part of the sample.

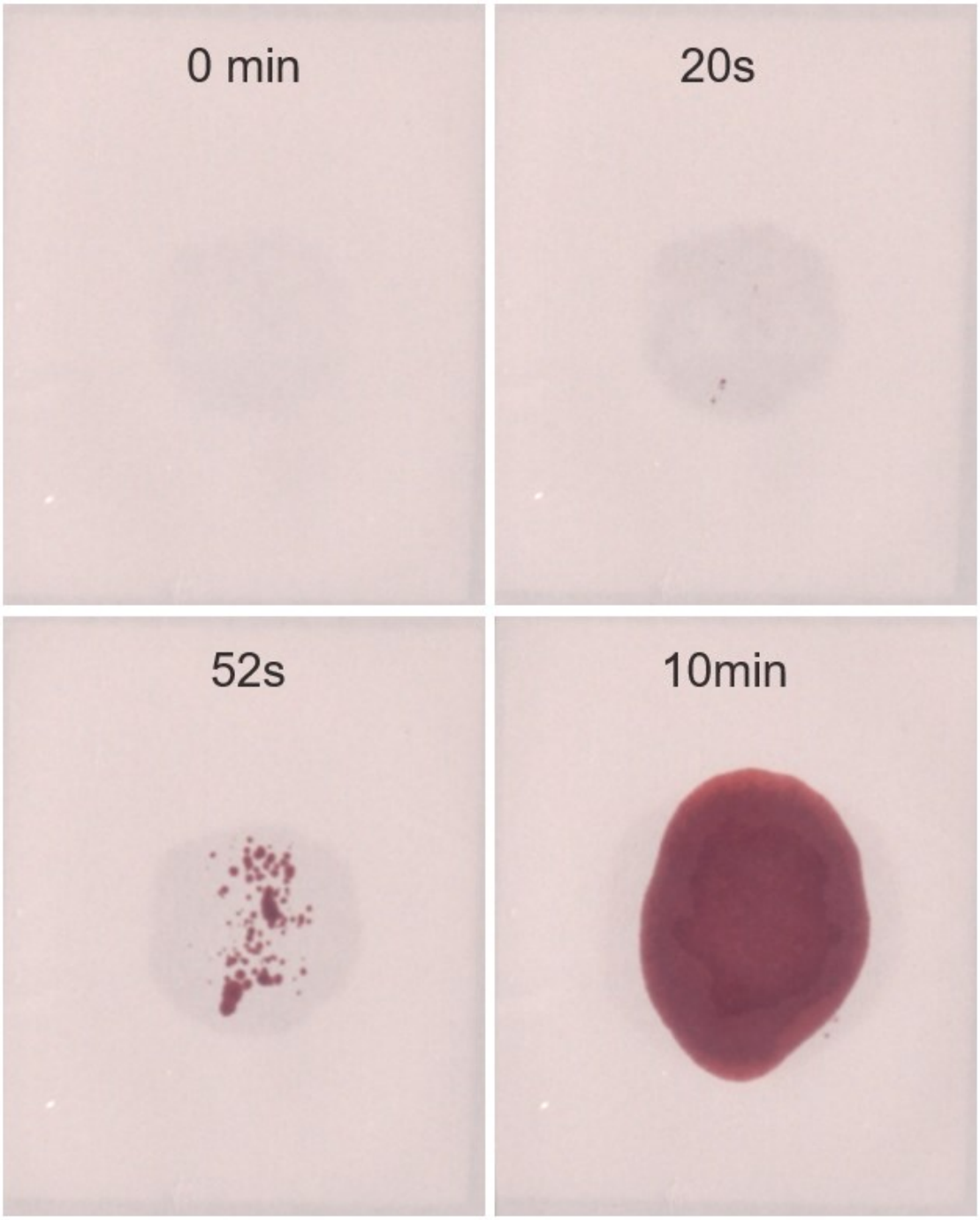


Figure 11. A time-lapse of a sample without OGR chemicals.

Figures 9, 10, and 11 demonstrate how the test results vary when testing hand sheets. When developing products, it is valuable to know the true potential of the sample. On the other hand, many underwhelming results may indicate problems in the process. Therefore, it is important to know if the direction is right. The development phase can take a long time, and using less time and fewer resources is beneficial for the business. With that in mind, it was decided

that at temperatures of 5°C and 23°C, seven samples were tested for each oil, and the 2 worst results were discarded due to the high rate of pinholes in the samples. Even though the samples cut from the sheets were also mixed for each KP so that there would be random samples for the tests rather than tests fixed to a certain sheet, there were outlier results here and there. There were some obviously defective samples with only few minutes of oil resistance. If time had not been limiting the practical work of the thesis, more samples would have been made to get more accurate results. All oil permeation times are shown in Appendix 2.

A commercial product made from vegetable oil was also tested to see the results compared to pure oils. A salad dressing was chosen to be tested in the same way as the oils. The salad dressing was also dyed red using food coloring. The average oil permeation times for the oils and salad dressing are shown in Table 10. Salad dressing oil permeation times were significantly higher compared to pure oils. Pure vegetable oils went through samples in 20-30 minutes, but when the salad dressing was used, the samples lasted 3-4 hours. Even without any OGR chemicals in KP1, the permeation times of salad dressing were on average around 40 minutes. Partly this can be explained by that the salad dressing changed from liquid to solid at the testing temperature of 40 °C. Salad dressing ingredients listed on the label were mostly rapeseed oil, water, vinegar, and sugar. All in all, usually commercial products behave differently from pure oils and are a good reminder that MFPs should also be tested in practice with real-life applications.

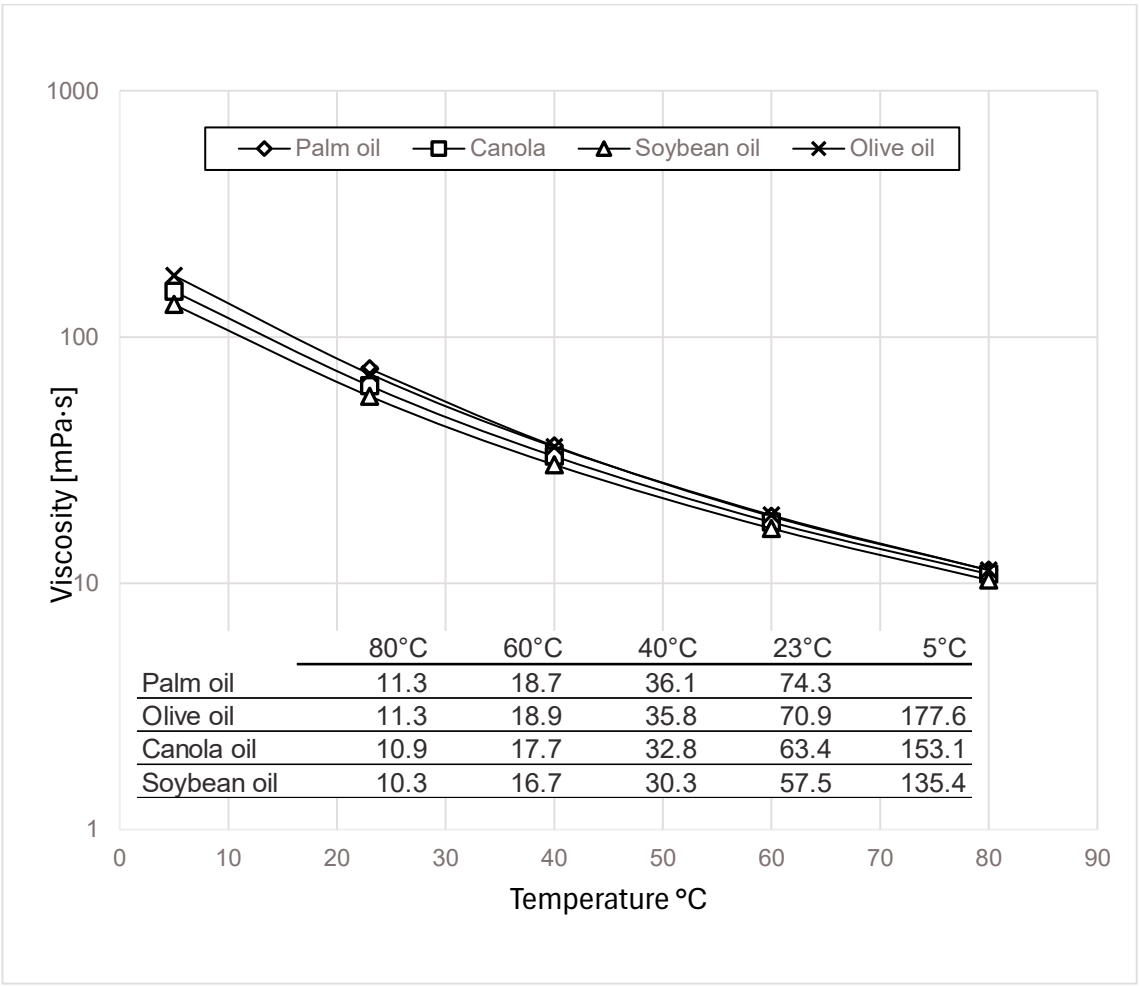
Table 10. Average oil permeation times for KP1, KP2, and KP3.

	Average oil permeation times (min)		
KP1	5°C	23°C	60°C
Canola oil	-	0.7	-
Olive oil	1	-	-
Soybean oil	-	-	-
Palm oil	-	-	1
Salad dressing	45	38	-
KP2	5°C	23°C	60°C
Canola oil	28.2	24.4	21.2
Olive oil	20	28.4	28.2
Soybean oil	20.6	28.2	22.6
Palm oil	40.4	30.2	35.6
Salad dressing	271	225	-
KP3	5°C	23°C	60°C
Canola oil	23	26.4	26
Olive oil	23.2	30.4	30.6
Soybean oil	12.8	28.4	24.6
Palm oil	39.8	34.6	32.2
Salad dressing	319	246	-

6.3 Viscosity Results

Viscosity profiles were measured for all the oils at five different temperatures. The only exception was palm oil, which turned semi-solid below room temperature, making it impossible to get reliable results. All of the oil profiles

seemed reliable between shear rates of 1[1/s] and 100[1/s]. Results were collected from the reliable point 10[1/s], and a visual graph was drawn as seen in Figure 12. Oils are behaving similarly to each other, viscosity decreases when the temperature rises. At almost every temperature, the order of viscosity from highest to lowest was palm oil, olive oil, canola oil, and soybean oil.



	80°C	60°C	40°C	23°C	5°C
Palm oil	11.3	18.7	36.1	74.3	
Olive oil	11.3	18.9	35.8	70.9	177.6
Canola oil	10.9	17.7	32.8	63.4	153.1
Soybean oil	10.3	16.7	30.3	57.5	135.4

Figure 12. Viscosity curves of all oils.

6.4 Statistical Observations

One interesting question was raised during the experiment planning for the chemical additives to the furnishing process. The question was whether the order in which the OGR and AKD additives were added affected the performance of hand sheets. Once all the oil tests were completed, the first thing to determine was whether there were any differences in the KP2 and KP3

samples' permeation times. As shown in Figure 13, there were minor differences in permeation times of KP2 and KP3 between different temperatures. To ensure the reliability of the analysis, a separate comparison was conducted at each temperature.

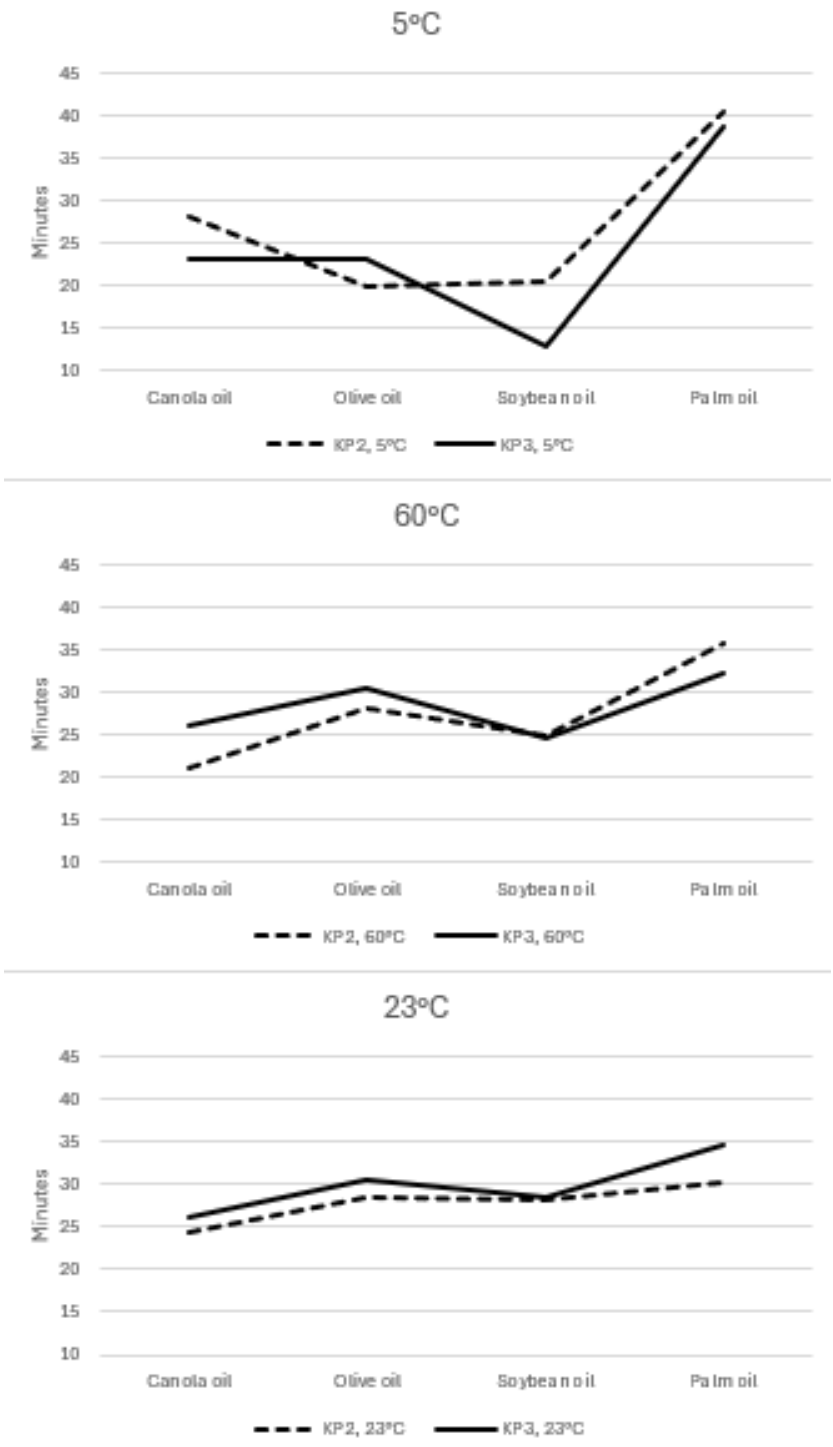


Figure 13. Comparison of average oil permeation times for KP2 and KP3 at each temperature.

To select the most suitable statistical method, it was first necessary to identify all variables influencing the outcome. If the statistical analysis was done separately at each temperature, then two factors with different levels were influencing the measurements. The first factor was the mixing order of additives, which had two levels: KP2 and KP3. The second factor was the type of oil used, having four levels: olive, palm, soybean, and canola. Two-way ANOVA with replication could be used to investigate whether either or both of the factors had a significant influence on the permeation times and if there was interaction between them. Two-way ANOVA with replication was conducted using the Excel data analytics tool. Samples per row were set to 5, and the significance level $\alpha = 0.05$. Results summaries for each temperature are shown in Table 11.

The p-value tells whether the between-group effect is significant. In other words, if p > significance level (α), the effect is statistically insignificant. Firstly, the p-value of the interaction was examined from the result summary. The p(interaction) > α in all temperatures indicates that the factors were not dependent. For all the temperatures, p(mixing order) > α, which meant that there were no significant differences in which order additives were mixed. However, the p(type of oil) < α, which tells that there were differences in results depending on the oil used. Full data of the ANOVA reports are shown in Appendix 3.

Table 11. Two-way ANOVA with replication results summaries of all the oil permeations of temperatures 5°C, 23°C, and 60°C.

5°C

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Mixing order	67.6	1	67.60	1.524	0.226	4.149
Oil type	3048	3	1016	22.90	0.000	2.901
Interaction	178.6	3	59.53	1.342	0.278	2.901
Within	1420	32	44.36			
Total	4714	39				

23°C

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Mixing order	42.03	1	42.03	2.68	0.111	4.149
Oil type	265.3	3	88.43	5.64	0.003	2.901
Interaction	22.88	3	7.625	0.49	0.694	2.901
Within	501.6	32	15.68			
Total	831.8	39				

60°C

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Mixing order	7.225	1	7.225	0.445	0.510	4.149
Oil type	663.5	3	221.2	13.61	0.000	2.901
Interaction	94.07	3	3.36	1.930	0.145	2.901
Within	520	32	16.25			
Total	1285	39				

Since the only difference between samples of KP2 and KP3 was the mixing order of the two additives, and that did not statistically impact oil permeation times of any oil at any temperature, KP2 and KP3 samples were treated as a single experimental group in further evaluations. In this way, the sample size increased from 5 to 10, making the following conclusions statistically stronger. Average permeation times of the merged samples are shown in Table 12.

Table 12. Average oil permeation times (min) and intervals at 95% confidence level.

	Average permeation time (min) and interval at 95% CI.		
	5°C	23°C	60°C
Canola oil	25.6 ± 7.8	25.2 ± 2.5	23.6 ± 2.5
Olive oil	21.6 ± 2.9	29.4 ± 2.9	29.4 ± 2.6
Soybean oil	16.7 ± 6.0	28.3 ± 2.3	24.8 ± 2.4
Palm oil	40.1 ± 4.2	32.4 ± 3.5	33.9 ± 4.1

Temperature results from merged samples showed that average permeation times at 23°C and 60°C for any oil did not show much difference. Their mutual order remained the same at 23°C and 60°C test temperatures. Results from 5°C temperature differ, though. The permeation times of olive oil and soybean oil decreased noticeably, while canola oil permeation times were unaffected. In contrast, palm oil permeation times were longer at higher temperatures. ~~A~~s shown in Figure 14, there is an indication of interactions likely caused by different temperatures.

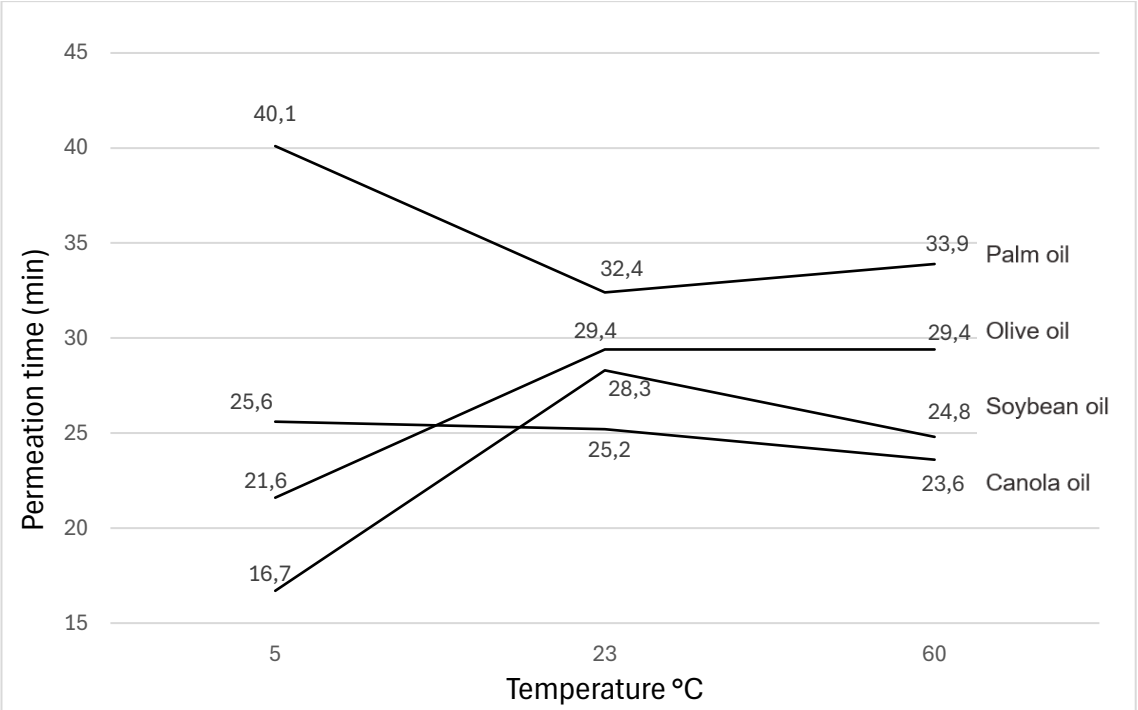


Figure 14. Average oil permeation times at different temperatures.

Since the average permeation times for all the oils seemed to be very similar between 23°C and 60°C, a comparison was made using Two-way ANOVA with replication. Factors that influenced the outcome were temperature and oil types. Samples per row were set to 10, and the significance level α = 0.05. To see how the results would change, another Two-way ANOVA with replication, including the 5°C samples, was also conducted. The p(interactions) < α for ANOVA including all the temperatures, which meant that both of the factors were significant. Further interpretation could be made for inspecting the MS-values. Comparing the MS values for both factors indicates that the oil type value (891.4) was much higher than the temperature value (83.31), thus signaling that temperature was dominant of the two. The results from comparing just the 23°C and 60°C differ though. The p(interaction) > α indicated that the factors were not dependent. P(temperature) > α meant that there was no significant difference in temperatures. However, p(oil type) < α indicated that the type of oil used was significant. Summary results for both Two-way ANOVAs with replications are shown in Table 13, and full data are shown in Appendix 4.

Table 13. Result summaries of two ANOVAS. One with all temperature groups compared, and another with 23°C and 60°C compared.

Comparison including sample groups of 5°C, 23°C, and 60°C.

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Temperature	166.6	2	83.31	3.153	0.047	3.080
Oil type	2674	3	891.4	33.74	0.000	2.689
Interaction	1303	6	217.1	8.217	0.000	2.184
Within	2854	108	26.42			
Total	6997	119				

Comparison including sample groups of 23°C and 60°C.

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Temperature	16.2	1	16.2	0.982	0.325	3.974
Oil type	859.7	3	286.6	17.37	0.000	2.732
Interaction	69.1	3	23.03	1.396	0.251	2.732
Within	1188	72	16.50			
Total	2133	79				

7 Conclusions

Keeping in mind that the sample size was relatively low for the conclusions to be statistically strongly justified, some conclusions could still be deduced cautiously. The mixing order of the chemical additives in the furnish caused no statistical differences in the oil permeation times. The oil penetration times of the samples tested with 5°C olive oil and soybean oil were lower than those tested with hotter oils. This result was not consistent with the hypothesis that as

the viscosity of the oil increases, the permeation time also increases. The viscosities of the oils decreased when the temperature rose, as expected. The number of double bonds in oils seemed to affect the viscosity by reducing it the more there were. However, the viscosity results of the different oils were relatively close to each other. Statistical analyses also indicated that either there were no differences whether the oil tests were conducted at a temperature of 23°C or 60°C, or that the conditions were too similar to make notable differences. Especially when using small amounts of oil, the temperature can move from both starting temperatures towards the oven conditions equally quickly.

Based on the results of this thesis and experience from the practical work, it is recommended to use canola oil in the tests. Canola oil is a good, inexpensive, general-purpose oil that consists of an average ratio of unsaturated and saturated fatty acids among the oils. There is also a lot of room to improve the test conditions for future testing. In particular, improved temperature control and a larger sample size would increase the accuracy and reliability of the results.

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Appendix 1. Physical Measurements of the Hand Sheets

Bendtsen roughness topside, air permeability Bendtsen & Gurley, and deviations.

Measurement	Bendtsen - Roughness TS	St.dev.	Air Permeability Bendtsen	St.dev.	Air Permeability Gurley	St.dev.
Number of measurements	4		4		4	
Standard	ISO 8791-2		ISO 5636-3		ISO 5636-5	
Sample:	ml/min	ml/min	ml/min	ml/min	s	s
KP1	2189	474	227	12,7	53	3,0
KP1 / 2	2123	630	249	8,2	48	1,6
KP1 / 3	2979	199	252	18,1	48	3,4
KP2	2130	668	245	15,9	49	3,2
KP2 / 2	2429	351	245	12,4	49	2,4
KP2 / 3	2007	320	226	5,6	53	1,4
KP2 / 4	1873	180	247	7,5	49	1,4
KP2 / 5	1610	235	230	19,3	52	4,6
KP2 / 6	1636	425	240	17,5	50	3,8
KP2 / 7	2384	389	273	16,4	44	2,6
KP2 / 8	1783	574	245	8,3	49	1,6
KP2 / 9	2091	813	251	26,7	48	5,7
KP2 / 10	2522	1178	255	18,6	47	3,4
KP2 / 11	1831	228	240	13,6	50	2,8
KP2 / 12	2098	483	242	15,3	50	3,1
KP2 / 13	2232	359	256	14,4	47	2,6
KP3	2477	828	263	15,9	46	2,7
KP3 / 2	2016	241	244	23,9	49	5,0
KP3 / 3	1394	351	256	20,0	47	3,9
KP3 / 4	2074	823	247	13,6	49	2,6
KP3 / 5	1840	539	232	13,5	52	3,2
KP3 / 6	1683	332	302	18,4	40	2,4
KP3 / 7	2081	726	235	16,1	51	3,4
KP3 / 8	1896	578	248	14,7	48	2,8
KP3 / 9	1717	153	237	11,8	51	2,6
KP3 / 10	1780	321	233	25,3	52	6,2
KP3 / 11	1694	261	226	22,6	53	5,7
KP3 / 12	1714	294	249	22,9	48	4,8
KP3 / 13	2051	438	261	18,1	46	3,3
KP3 / 14	1751	621	242	23,5	50	5,0

Appendix 1

Weight, thickness, density, and bulk of the hand sheets.

Measurement	Grammage	Thickness	St.dev.	Density	Bulk
Number of measurements	2	5			
Standard	ISO 536	ISO 534		ISO 534	ISO 534
Sample:	g/m2	µm	µm	g/cm3	cm3/g
KP1	410,0	586	20,9	0,700	1,429
KP1 / 2	396,4	569	31,1	0,697	1,435
KP1 / 3	397,7	568	23,2	0,700	1,428
KP2	404,6	583	27,5	0,694	1,442
KP2 / 2	403,8	579	20,6	0,697	1,434
KP2 / 3	403,2	578	24,7	0,697	1,434
KP2 / 4	404,0	589	21,8	0,686	1,457
KP2 / 5	402,1	584	24,0	0,688	1,453
KP2 / 6	404,1	579	24,8	0,697	1,434
KP2 / 7	403,3	569	29,0	0,708	1,412
KP2 / 8	402,1	572	26,6	0,704	1,421
KP2 / 9	402,8	584	27,3	0,690	1,449
KP2 / 10	401,3	583	11,8	0,688	1,453
KP2 / 11	399,2	571	17,8	0,699	1,430
KP2 / 12	398,8	579	15,3	0,688	1,453
KP2 / 13	392,8	575	15,8	0,683	1,464
KP3	402,0	578	29,6	0,695	1,438
KP3 / 2	403,0	582	17,0	0,692	1,445
KP3 / 3	401,0	581	17,7	0,690	1,449
KP3 / 4	401,1	569	24,8	0,705	1,418
KP3 / 5	400,4	579	25,4	0,692	1,445
KP3 / 6	357,3	543	15,0	0,658	1,519
KP3 / 7	401,9	573	28,9	0,702	1,425
KP3 / 8	402,2	582	15,9	0,691	1,446
KP3 / 9	402,4	581	23,9	0,693	1,444
KP3 / 10	400,2	573	21,8	0,699	1,431
KP3 / 11	400,8	568	26,9	0,706	1,417
KP3 / 12	399,4	576	32,8	0,693	1,442
KP3 / 13	398,6	573	12,0	0,696	1,437
KP3 / 14	395,6	567	19,2	0,697	1,434

Appendix 2. Oil Permeation Times of Each Temperature

Oil permeation results (min) at a temperature of 5°C.

5°C				
KP1				
	Olive oil			Salad dressing
	1			43
	1			41
	1			51
KP2				
Canola oil	Olive oil	Soybean oil	Palm oil	Salad dressing.
14	23	3	15	231
20	10	11	45	285
17	16	3	47	242
37	14	30	43	271
39	23	11	41	327
28	5	26	22	
3	24	25	26	
KP3				
Canola oil	Olive oil	Soybean oil	Palm oil	Salad dressing
24	19	9	34	229
18	12	6	37	186
7	27	6	38	293
30	24	19	39	285
26	21	10	37	602
4	7	4	42	
17	25	20	43	

Appendix 2

Oil permeation results (min) at a temperature of 23°C.

23°C				
KP1				
Canola oil				Salad dressing
0				50
1				17
1				47
KP2				
Canola oil	Olive oil	Soybean oil	Palm oil	Salad dressing
16	29	32	5	199
19	30	26	34	235
18	24	26	16	235
23	23	31	33	238
27	35	26	28	220
26	24	14	22	
27	5	24	34	
KP3				
Canola oil	Olive oil	Soybean oil	Palm oil	Salad dressing
21	27	19	26	271
27	24	19	29	253
25	16	32	36	233
31	35	32	27	261
23	30	29	34	212
26	27	24	34	
4	33	25	40	

Appendix 2

Oil permeation results (min) at a temperature of 60°C.

60°C			
KP1			
			Palm oil
			1
			1
			1
KP2			
Canola oil	Olive oil	Soybean oil	Palm oil
16	29	29	31
23	33	24	33
23	32	24	34
22	23	21	47
22	24	15	33
KP3			
Canola oil	Olive oil	Soybean oil	Palm oil
26	33	24	36
28	32	28	31
28	27	20	35
23	31	29	35
25	30	22	24

Appendix 3. ANOVA Reports of KP2 and KP3 Comparisons

KP2 and KP3 comparison at 5°C.

Anova: Two-Factor With Replication 5°C

SUMMARY	Rypsiöljy	oliiviöljy	soijap.öljy	palmuöljy	Total
kp2					
Count	5	5	5	5	20
Sum	141	100	103	202	546
Average	28,2	20	20,6	40,4	27,3
Variance	96,7	21,5	80,3	69,8	127,6947
kp3					
Count	5	5	5	5	20
Sum	115	116	64	199	494
Average	23	23,2	12,8	39,8	24,7
Variance	30	10,2	39,7	6,7	116,8526
Total					
Count	10	10	10	10	
Sum	256	216	167	401	
Average	25,6	21,6	16,7	40,1	
Variance	63,82222	16,93333	70,23333	34,1	

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Sample	67,6	1	67,6	1,52381	0,226032	4,149097
Columns	3048,2	3	1016,067	22,90373	4,17E-08	2,90112
Interaction	178,6	3	59,53333	1,341974	0,278124	2,90112
Within	1419,6	32	44,3625			
Total	4714	39				

KP2 and KP3 comparison at 23°C.

Anova: Two-Factor With Replication 23°C

SUMMARY	Rypsiöljy	oliiviöljy	soijap.öljy	palmuöljy	Total
kp2					
Count	5	5	5	5	20
Sum	122	142	141	151	556
Average	24,4	28,4	28,2	30,2	27,8
Variance	11,8	21,3	9,2	27,2	19,32632
kp3					
Count	5	5	5	5	20
Sum	130	152	142	173	597
Average	26	30,4	28,4	34,6	29,85
Variance	13	12,8	14,3	15,8	22,23947
Total					
Count	10	10	10	10	
Sum	252	294	283	324	
Average	25,2	29,4	28,3	32,4	
Variance	11,73333	16,26667	10,45556	24,48889	

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Sample	42,025	1	42,025	2,681021	0,111349	4,149097
Columns	265,275	3	88,425	5,641148	0,00321	2,90112
Interaction	22,875	3	7,625	0,486443	0,694099	2,90112
Within	501,6	32	15,675			
Total	831,775	39				

Appendix 3

KP2 and KP3 comparison at 60°C.

Anova: Two-Factor With Replication

SUMMARY	Rypsiöljy	oliiviöljy	soijap.öljy	palmuöljy	Total
kp2					
Count	5	5	5	5	20
Sum	106	141	125	178	550
Average	21,2	28,2	25	35,6	27,5
Variance	8,7	20,7	9,5	41,8	46,47368
kp3					
Count	5	5	5	5	20
Sum	130	153	123	161	567
Average	26	30,6	24,6	32,2	28,35
Variance	4,5	5,3	14,8	24,7	20,76579
Total					
Count	10	10	10	10	
Sum	236	294	248	339	
Average	23,6	29,4	24,8	33,9	
Variance	12,26667	13,15556	10,84444	32,76667	

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Sample	7,225	1	7,225	0,444615	0,509683	4,149097
Columns	663,475	3	221,1583	13,60974	6,82E-06	2,90112
Interaction	94,075	3	31,35833	1,929744	0,14461	2,90112
Within	520	32	16,25			
Total	1284,775	39				

Appendix 4. ANOVA Reports of Comparisons Between Temperatures

Comparison between all variables.

5°C, 23°C, and 60°C

Anova: Two-Factor With Replication

SUMMARY	Rypsiöljy	oliiviöljy	soijap.öljy	palmuöljy	Total
5°C					
Count	10	10	10	10	40
Sum	256	216	167	401	1040
Average	25,6	21,6	16,7	40,1	26
Variance	63,82222	16,93333	70,23333	34,1	120,8718
23°C					
Count	10	10	10	10	40
Sum	252	294	283	324	1153
Average	25,2	29,4	28,3	32,4	28,825
Variance	11,73333	16,26667	10,45556	24,48889	21,32756
60°C					
Count	10	10	10	10	40
Sum	236	294	248	339	1117
Average	23,6	29,4	24,8	33,9	27,925
Variance	12,26667	13,15556	10,84444	32,76667	32,94295
Total					
Count	30	30	30	30	
Sum	744	804	698	1064	
Average	24,8	26,8	23,26667	35,46667	
Variance	28,02759	28,37241	52,82299	39,84368	

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Sample	166,6167	2	83,30833	3,152965	0,046684	3,080387
Columns	2674,233	3	891,4111	33,73717	1,83E-15	2,688691
Interaction	1302,717	6	217,1194	8,217304	2,48E-07	2,183657
Within	2853,6	108	26,42222			
Total	6997,167	119				

Comparison between all permeation times of at temperature 23°C and 60°C.

5°C, 23°C

Anova: Two-Factor With Replication

SUMMARY	Rypsiöljy	oliiviöljy	soijap.öljy	palmuöljy	Total
23°C					
Count	10	10	10	10	40
Sum	252	294	283	324	1153
Average	25,2	29,4	28,3	32,4	28,825
Variance	11,73333	16,26667	10,45556	24,48889	21,32756
60°C					
Count	10	10	10	10	40
Sum	236	294	248	339	1117
Average	23,6	29,4	24,8	33,9	27,925
Variance	12,26667	13,15556	10,84444	32,76667	32,94295
Total					
Count	20	20	20	20	
Sum	488	588	531	663	
Average	24,4	29,4	26,55	33,15	
Variance	12,04211	13,93684	13,31316	27,71316	

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Sample	16,2	1	16,2	0,981983	0,325028	3,973897
Columns	859,65	3	286,55	17,36959	1,39E-08	2,731807
Interaction	69,1	3	23,03333	1,396195	0,250944	2,731807
Within	1187,8	72	16,49722			
Total	2132,75	79				