Development and analysis of a commercial hair treatment

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Popular science summary

Development and analysis of a commercial hair mask

Our degree project has been to develop and analyse a hair mask. We tried out different “recipes” for the hair mask in the lab, and then used different methods to evaluate our recipes to find out which one is the best.

Like many other industries, the hair cosmetic industry is facing a change where customers are asking for a new type of environmentally friendly cosmetic products. Because of this, we wanted to develop a hair mask with mostly organic and natural ingredients. Our degree project was done in cooperation with a Swedish cosmetic company named Rapsodine, who provided ingredients and guidance in the process of developing a hair mask.

We tried out two types of recipes, a classic one (which is similar to other hair masks on the market) and a more experimental one. For both types of hair mask, we tried out different versions where we made small changes to the proportions of the ingredients. The goal was to find the best version for both types (classic and experimental) and to then compare them to each other.

To find out which of the different recipes actually worked best, we did a number of different tests and evaluations:

- We photographed and rated the hair masks based on five different characteristics and compared their photographs and points
- We did a customer evaluation where six people got to try three of the hair masks at home in the shower and then give them points
- We measured how well the hair could withstand compression before and after being treated with the hair masks
- We looked at the hair masks in a microscope and compared them
- We looked at hair treated with the hair masks in a microscope, before and after the treatment
- We analysed the particles in the hair masks: both their sizes, their particle size distributions and how they change with time
- We measured the pH-values of the hair masks

After our evaluation, we concluded that the classic recipe resulted in a better hair mask while the experimental recipe still needed a lot of work. We also found which classic recipe and which experimental recipe was most successful.

Introduction

Like many other industries, the hair cosmetic industry is facing a transformation where customers are inquiring for a new type of environmentally friendly cosmetic products. skriva mer

The master thesis was done in collaboration with the Swedish cosmetics company Rapsodine, which manufactures organic cosmetic products containing canola oil. Rapsodine provided guidance and expertise regarding the hair mask development process, as well as the ingredients used in the formulations.

1.1 Aim

To align with this readjustment, the aim of this master thesis is develop an hair mask with a high percentage of natural and organic ingredients. Further, the aim is to investigate and evaluate the properties and qualities of the developed hair mask emulsions using both qualitative analysis methods and sensory evaluation. Due to time restrictions, focus was put on the surfactant/oil/water ratio of the hair mask and other parts of hair mask formulation was neglected. A large part of the project process has been to identify suitable analysis and evaluation methods. Therefore, a final aim of this master thesis is to evaluate how well the different analysis and evaluation methods actually provided useful and significant information regarding the emulsions.

1.2 Project description

In order to accomplish the aims described above, a project plan was established. The first step was to perform a literature study to acquire deeper understanding of hair structure, emulsions, hair damage and hair mask ingredients. When the literature study was finished, a period of laboratory work began where different emulsion formulations, both classic and pickering, were developed. After formulating different series of emulsions, they were photographed and evaluated based a number of sensory properties. Further, their pH was measured. Based on the sensory evaluation, the most promising emulsions were identified and chosen for further analysis. Compression strength analysis, laser diffraction and microscopy was used to gain better understanding of the chosen emulsions improvement of the hair quality, stability and small-scale structure. Lastly, the most promising emulsions were given to be evaluated by a test panel which used the hair masks at home in the shower and then answered an user survey regarding their experience.

Background

2.1 The structure of hair and the hair masks function

The human body, apart from the soles, palms and lips, is covered in hair follicles producing either thick terminal and or fine vellus hair. The human hair has multiple functions such as heat regulation and protection of the underlying skin. Hair is also an important factor in sexual recognition and attractiveness [1], therefore it is of particular interest in the personal care and cosmetic industry. Understanding the growth and chemical composition of hair is a necessity for development of effective hair cosmetic products. The structure of hair and the hair masks function will thus be covered in the following sections.

2.1.1 Keratin

Hair is essentially composed of the group of fibrous proteins called keratins. Keratin is a strong and compact biomaterial formed in epithelial and epidermal cells, keratinocytes. One essential and characteristic property of keratin is the high content of the amino acid cysteine. Cysteine contains sulfur which enables it to covalently cross-link between the polypeptide chains by creating -S-S- bonds. The disulphide bond is a key factor in the mechanical stability of hair and also the chemical inertness that keratin possesses. Another important property of keratin is its ability to form network structures between intermediate filaments (IFs) in the endoplasmic regions of the hair. Description of intermediate filaments will be covered in the sections below.

Keratin can be classified as “soft” or “hard” depending on the degree of cysteine content. Soft keratin contains less cysteine and thus fewer strengthening disulfide bonds, it’s mostly found in the outermost layer of the skin. Hard keratin has a higher sulfur content and forms densely packed structures together with the IFs.

Hard keratin is the variant found in hair and nails.

Another way of classifying keratin is based on its secondary structure. Depending on the amino acid constitution, the protein can fold into two types of conformations, helicles (α-keratin) or beta sheets (β-keratin). Only α-keratin is present in mammalians and there are approximately 30 different variants.

Keratin's molecular structure is highly related to the mechanical and physical properties of keratin. Altering the structure via chemical or physical hair treatments can thus severely affect the mechanical and structural integrity of the hair. [2]

2.1.2 Structural components

Hair is a complex material composed of multiple morphological units acting as one. Keratin is the major component and constitutes approximately 65 to 95 percent of the mass content, the proportion depends on the moisture content. The remaining components includes water, lipids, minerals and pigments (4). The mammalian hair shaft is divided into three structural regions: the cuticle, cortex and medulla.

The cuticle is the outer, protecting layer and consists of overlapping cuticle cells. The cells are approximately 0.5 μm thick and 45-60 μm in length and the cuticle normally consists of 6-8 cell layers. (3) The cuticle cells contain five structural layers. The first, outermost layer is a lipid layer containing 18-methyl eicosanoic acid (18-MEA) and free lipids. The lipid layer enfolds the hair fiber and is responsible for the hair's hydrophobicity. (5) The second layer is a proteinaceous cell membrane, the epicuticle. The epicuticle is followed by three heavily cross-linked protein layers: the A-layer, the exocuticle or B-layer and the endocuticle. The A-layer and exocuticle are both cystine rich containing approximately 30 and 15-20 weight percent respectively, the endocuticle contains 3 percent cystine. [2]

The intercellular matter is called the cell membrane complex (CMC) and consists of cell membranes and adhesive material. The CMC binds covalently between cuticle cells, cortical cells and cuticle-cortical cells. [3]

The cortex is suited beneath the cuticle and constitutes 70-90 percent of the hair fiber mass; it contains cortical cells surrounded by CMC. The cortical cells include several structural sub units: macrofilaments, intermediate filaments (IFs) and protofilaments. Each cortical cell is built of 5-8 macrofibrils and each macrofibril contains 500-800 IFs. The IFs are surrounded by an amorphous, cystine rich matrix known to be relatively hydrophilic in nature due to the high content of non-ordered protein. The intermediate filaments are composed of α-helice coiled keratin. The α-helices arrange tetramers which are connected longitudinal, end to end to form the protofilament structures. The IFs are highly crystalline and hydrophobic.

The medulla is considered as one of the three main structural regions of the hair fiber but is nonetheless often absent. It is only found in coarser hair and can be present continuously or fragmented. The medulla is suited in the core of the hair fiber and consists of loosely packed medullary cells, it's often considered as a void. It is inaccessible to chemical treatments and considered to have a negligible impact on the chemical and mechanical properties of hair. [2]

2.1.3 Hair damage

The natural appearance of newly formed hair displays a smooth, scale surface. As the hair is exposed to stress of various kinds, the structure is subsequently damaged, a process which is referred to as weathering. [4] Damaged hair entails poor manageability, dryness, brittle-ness, loss of shine and decreased strength (fiber breakage).[5] Solutions to avoid or depress weathering is therefore of great concern within the hair cosmetic industry.

There are multiple ways of how weathering may occur and accelerate. These are environmental factors such as sunlight and humidity, physical actions such as brushing or toweling, heat treatments such as blow drying or heating tool styling or finally, chemical treatments which can be anything from shampoo to hair dying or chemical perming.[2] The path of chemical and physical alterations of the hair fiber is not completely understood but some confirmed chemical degradation pathways has been identified. [4]
The fiber breakdown differs depending on the source of the stress. Bleaching of hair is often performed with dihydrogen oxide at a pH of 9-11. The high pH primary induces oxidation of the disulphide bonds in cystine. As much of the hair is cystine rich, bleaching weakens the hair fiber structure even at deeper located structural layers. Studies have shown that 15-25 percent of the hair fiber disulphide bonds are degraded during a bleaching process. Other alkaline treatments such as alkaline straighteners are performed at pH 13 or above. These treatments induce several reactions throughout the fiber structure; alkaline hydrolysis of the peptide and amide bonds in the protein, beta-elimination of cystine and hydrolysis of the ester and thioester groups in the cell membrane complex. The latter results

in removal of the 18-MEA layer at the hair shaft surface and also weakens the cellular matrix complex bonding between cuticle and cortical cells. Breakage of peptide, amide and disulphide bonds within the protein rich regions makes the α-helical chain to unfold and reorganize the crystalline protein into more amorphous.

Permanent waving is often completed with acidic compound glycerolmonothioglycolate (GMT). This process involves cleavage of the disulphide bonds in keratin and cysteine residues are formed. The hair fibers are rearranged into the desired shape before an alkaline neutralizer is added, during this process the disulphide bonds are reformed but it's well known that the re-oxidation process is incomplete and that many di-sulfide bonds remain cleaved.

Heat damage primarily involved two damaging chemical pathways; degradation of the amino acid tryptophan and removal of surface lipids. [2]

Hair damage increases the permeability of the fibrous structure and leads to increased, adverse absorption of water. The absorption implies swelling and enables water molecules to plasticize hair fiber components. Increased water content deteriorates the hair's tensile properties and more easily leads to hair fiber fracture. [5]

Visually, weathering takes the form of jagged edges of the cuticle scales, partial or complete scale erosion and longitudinal splitting of the fibers either into two cylindrical components or into several fibrous elements. [4]

2.1.4 Hair mask function

Hair treatments (hair masks) are used as a supplement to hair conditioners to partly repair damaged hair, moisturize the hair and improve the quality and sensory properties of the hair. While conditioners are used the improve the softness of the hair after shampooing, treatments are expected to have repairing qualities and to moisturize the hair more deeply. This is mainly achieved in two ways. Firstly, treatments are often left on longer before being rinsed off compared to conditioners. Secondly, treatments have an higher content of moisturizing and repairing ingredients. [6]

Hair that is subjected to damage is left with negatively charged residue on the hair fiber surface. Since hair fibers mainly consist of dead cells which can not be replaced, damaged hair cuticle has no biological way to repair. To combat this problem, conditioning hair treatments are used to temporally cover and fill the damaged parts of the cuticle. Cationic surfactants in hair treatments interact with the anionic residues and are deposited onto the fibers. This reduces their static electricity and friction between hair fibers. Further, fatty alcohols (such as cetearyl alcohol) bind to the hair which lubricates the hair and makes combing easier. Bleached or damaged hair is more negatively charged, which increases the affinity of the cationic surfactant to the hair fibers, making the hair treatment have even more effect.

The hair shaft pH is 3.7 and the hair scalp pH is 5.5. Due to the function of hair treatments, the pH of the treatments is of importance. A hair product with a pH higher than 3.7 increases the negativity of the electric network of the hair, which as mentioned above causes increased friction and static electricity. Further, a pH higher than 5.5 could cause irritation of the scalp. [7] [8] [9]

2.2 Hair mask formulation

Formulation is the term used to describe the total composition of ingredients in a cosmetic or pharmaceutical product. The system of ingredients (the formulation) enables delivery of essential substances to the target area and together make up the final product. The formulation is important to ensure that the product fulfill its function, and in addition, its sense and appearance to a large extent constitutes the customers over all perception of the product. The creation of an appealing formulation is thus important for a succeeding outcome in cosmetic product development. The following segment will cover how the hair mask for this project is composed and how to the adjust the characteristics of the formulation.

2.2.1 Emulsions

Hair masks are most commonly created as two phase, dispersed systems called emulsions. Emulsification is widely used in cosmetic product development since it enables combination of efficacious oily and aqueous components into a single system and facilitates the delivery of the active ingredients to the body. [6] The hair mask emulsion consists of one oil phase and one water phase. Depending on which phase is dispersed in the other, they are classified either as water in oil emulsions (W/O) or oil in water emulsions (O/W). The choice of the dispersed phase highly affects the form and the sensory characteristics of the hair mask. O/W emulsions give a light, non sticky and moisturized feeling and W/O a dense, oily feeling.[10] The hair masks developed for this project are exclusively O/W emulsions. The homogenization procedure and amount of surfactant determines the droplet size which in turn affects the hair masks appearance. One way to classify emulsions are by the degree of dispersion:

- **Macroemulsions:** Particle diameter $>$ 500 nm.

- **Microemulsions:** Particle diameter < 500 nm.
- **Nanoemulsions:** 20 nm $<$ Particle diameter $<$ 200 nm.

?? In addition to different degree degrees of dispersion, micro and nanoemulsions are also differentiated based on their stability, functional and physiochemical characteristics. The most important distinction is that microemulsions are thermodynamically stable systems while nanoemulsions are thermodynamically unstable. [11]
The emulsions appearance is affected due to light scattering by the oil droplets. The light is scattered by dispersed particles with a diameter larger than the incoming light wave. The visible light waveband is 390-750 nm. Thus for macroemulsions, the entire visible light spectrum is scattered when passing the dispersed phase, resulting in a white appearance. Micro- and nano-emulsions are semi-transparent. **??**

2.2.2 Emulsifiers

Emulsions are thermodynamically unstable systems. The two phase system degrades over time due to several physicochemical mechanisms:

- Creaming - The separation due to differences in density between the droplets and the surrounding medium.
- Ostwald ripening - The dissolution of smaller droplets or particles into larger ones.
- Coalescense - When droplets merge together and lose their identity.
- Flocculation - The aggregation of droplets.

Additives, broadly referred to as *emulsifiers*, are thus required to stabilize the system. Emulsifiers are compounds that act in the interface between the dispersed and the continuous phase in order to counteract the degradation of emulsions. [12] [10]

Surfactants

The most commonly used emulsifiers are surfactants. Surfactants are amphiphilic molecules which allows them to adsorb to the surface of the dispersed particles and interact with the continuous phase simultaneously. The adsorption of surfactants lower surface tension and stabilizes the system from destabilization. Beyond

the purpose of stabilizing the emulsion, surfactants are used to adjust rheological properties and sensory characteristics of the formulation.[13]
The hydrophobic part of surfactants is referred to as the "tail" and usually consists of a hydrocarbon chain of variable length. To achieve greater lathering and detergency effect, a shorter chain length is preferable. A longer tail length contributes to better oil affinitivity and is thus preferable in emulsification. [6] The surfactant tail is usually between 8-18 carbons long. [13] The hydrophilic part of the surfactants is referred to as the head group, this part widely differs between surfactant types and is what determines the nature of the surfactant. Surfactants are often classified depending on the ionization state of the head group. The four surfactant classes are:

- Anionic
- Cationic
- Amphoteric
- Nonionic

Anionic surfactants have a negatively charged hydrophilic part. They are most often used in cleansers and soaps, due to their high cleansing power, wetting agent properties and excellent lather characteristics. However, anionic surfactants are known to be irritants to the skin. Some examples of common anionic surfactants are sodium laureth sulfate, sodium lauroyl methyl isethionate or sodium methyl lauroyl taurate.

Cationic surfactants have a positively charged hydrophilic part (often qarternary ammonium) which neutralizes the negative charge on the surface of damaged hair. The antistatic effect give the hair a flexible and smooth surface and because of this, they are common ingredients in conditioners. Depending on the counter ions and the amount of alkyl groups in the head group, cationic surfactants have varying emulsifying effects but several variants are used as emulsifiers with great stabilizing and thickening performance. Similar to anionic surfactants, cationic surfactants are irritating to the skin. Commonly used cationic surfactants are benzalkonium chloride, trimetrhylalkylammonium chloride or cetylpyridinium cetrimoniumchloride.[6]

Amphoteric/zwitterionic surfactants can act both anionic or cationic depending on the pH, and is a less aggressive group of surfactants compared to anionic and cationic surfactants. They are commonly used in liquid cleansers, body washes, schampoos and shaving products in combination with anionic and cationic surfactants to enhance mildness.

Nonionic surfactants are not electrically charged. Due to this, they have comparatively weaker lather properties and cleansing power. However, they are the most gentle and least skin irritating type of surfactant. They are most often used as emulsifiers and solubilizers in cosmetics.

Solid particles

The application of solid particles as emulsifiers has not yet been used commercially to any large extent, but research shows promising prospects for the technique. [14] Several types of particles have been demonstrated to effectively serve as pickering emusifiers. Silica, clay, hydroxyapatite (Hap) and starch are a few examples. A further description of solid particles as emulsifiers is covered in section 2.2.3.

2.2.3 Pickering emulsions

An upcoming approach to meet the increasing demand of natural products is the use of pickering emulsions. In pickering emulsions, traditional surfactants are replaced by emulsifying agents in the form of solid particles. [15] The emulsification technique has received relatively little commercial attention, but research shows promising prospects regarding improved stabilization, lowered toxicity and lowered environmental pollution compared to traditional emulsions[16].
Just as in case for surfactant based emulsions, the solid particles stabilizes the system by attaching to the surface of the dispersed droplets. Unlike surfactants (which are in equilibrium between the phases and constantly adsorb and dissolve), the solid particles attachment is irreversible and give rise to improved stability. Emulsion stability arises due to the formation of a steric barrier between the phases, as thus preventing the dispersed droplets to approach each other.
The choice of particle type is crucial for effective stabilisation and also which type of emulsion that is formed. Whether a O/W emulsion or W/O emulsion is formed is determined by the wettability at the phase boundary of the solid particles. The phase which the solid particle have the highest affinity for is the one becoming the continuous phase. Thus, hydrophilic particles are used to stabilize O/W emulsions and hydrophobic particles are used to stabilize W/O emulsions. Particles which are fully wetted by one of the phases will be dispersed and can not form emulsions, it's thus often required to modify the particles in order to make them more amphiphilic. [17] [16] Given the aim of this degree project; to create

a sustainable cosmetic hair mask with the highest proportion of natural ingredients as possible, a pickering emulsion containing starch particles was developed and evaluated. Along with the starch particles, a thickening agent is needed to manufacture a pickering emulsion with suitable rheological properties. For this project, two different thickening agents are used: polyacrylic acid and hydroxypropyl methylcellulose.

2.2.4 Emulsion stabilization

Emulsification implies creation of a large amount of small droplets dispersed into a continuous phase of a liquid with a different polarity. The process is nonspontaneous and requires energy input. Emulsification entails an increase in the total surface area of the dispersed phase and an increase in the surface tension at the phase boundaries. The resulting emulsion is thermodynamically unstable which means it will reverse into its original, separated state over time if no surfactants or other stabilizing agents are added. Due to the amphiphilic properties of surfactants, surface tension is decreased when surfactants adsorb to the interface between the dispersed and continuous phase. The surfactants also form an energy barrier between the dispersed droplets by steric or electrostatic repulsion. The energy barrier exceeds the natural Wan der Waal attraction and thus stabilizes the system.

The stabilizing effect to a large extent depends on the chemical structure of the surfactant applied. The HLB concept was developed 1949 and is a useful tool to determine which surfactants are suitable to create a stable emulsion with the desired form. HLB stands for Hydrophilic-Lipophilic Balance and is a measure of the degree of hydrophilicity in relation to the total molar mass of non ionic surfactants.[10] There are several methods to calculate a surfactant's HLB number, it can be calculated by incrementation according to equation 2.1. Values of increments can be found in other literature. [18]

$$HLB = 7 + hydrophilic\ groups + hydrophobic\ groups \tag{2.1}$$

HLB is a scale that ranges from 0-20 and indicates the surfactant's relative affinity for the oil and water phase. Surfactants with a large proportion of hydrophilic groups with a HLB number between 10-18 are suitable for the creation of O/W emulsions. The opposite occurs for preparation of W/O emulsions and in this case, HLB should be between 3-8. Other than stabilizing emulsions by reducing the surface tension, surfactants form a layer at the surface of the dispersed particles that contributes with other stabilizing effects. For instance, non ionic surfactants in O/W emulsions create spatial obstacles and hydrogen bonds with the surrounding water. This prevents the oil droplets from approaching each other.

Ionic surfactants form an electrically charged sphere around the particles, causing them to repel one another. To optimize the stability of the emulsion, several surfactants with different oil/water affinity characteristics are commonly used for one formulation. This is since it facilitates a larger amount of surfactant molecules to orient on the surface of the particles, another way to achieve this is to increase the proportion of the emulsifier phase. [10]

Beyond the choice of emulsifier and emulsifier concentration, other factors such as temperature, pH, droplet size and droplet distribution affect the stability. Increased temperature destabilizes the emulsion since increased temperature means increased thermal energy in the system and therefore also increased frequency of droplet collisions. Also, the viscosity decreases which results in faster drop coalescence and surfactant-film drainage. Smaller droplet size and high droplet distribution have a positive effect on emulsion stability. The droplet distribution is in general higher when the droplets are smaller, smaller droplets leads to increased viscosity and thus increased stability. Smaller droplets also coalesce to a lesser degree due to their size. pH can affect the stability significantly since it can change the electrostatic force of the stabilizing surfactant film. [19][20]

2.2.5 Micelle formation and viscosity adjustment

Surfactants put into solution self-assembles into different types of organized structures. The most familiar form of aggregation in aqueous solution is micelle: a globular structure where the hydrophilic head groups are pointing out in the solution and the hydrophobic tails inwards to the globe center, thus shielded from the polar environment. Micelles are formed spontaneously when the concentration and temperature is sufficient, the concentration and temperature required differs depending on surfactant type. The minimal concentration required to form micelles is called Critical Micelle Concentration (CMC) and the minimal temperature is referred to as the Krafft point. [21] Some surfactants are able to grow and create elongated structures called Wormlike micelles (WLM). These aggregates behave much like polymers, they are semi-flexible and entangle above a critical concentration, resulting in the solution being viscoelastic. WLMs are therefore often used as emulsion thickening agents. [22]

A viscous emulsion is preferable for cosmetic hair treatments, it's thus necessary to add a surfactant which form WLM and/or to add other thickening agents. Other factors which are influencing the viscosity are:

- The viscosity of the continuous phase
- The volume of the dispersed phase
- The mean particle size and the particle size distribution

- The concentration of the emulsifier

2.2.6 Sensory characteristics adjustments

Laboratory evaluation of hair masks characteristics such as rheological and stability properties is important for development and optimization of the hair masks quality, but does not always reflect the users actual impression of the product. Labeling characteristics with descriptive words facilitates the work with designing and optimizing the sensory impression of the hair mask. [6] The vocabulary used for this project can be seen in Table 2.1.

Table 2.1: Vocabulary used for sensory characteristics evaluation.

	Major expression	Further descriptions
Positive characteristics	Moisturizing	Oily, heavy, moist
	Smooth	Slippy, flowing
	Cohesiveness	Adhering, coated, compact
Negative characteristics	Dry	Hard, tight, stiff, course, dull
	Light	Thin, airy, eviscerated

The most contributing factor to the outcome of the hair masks sensory characteristics are the alkyl groups of the oil and surfactants, and the choice of cationic surfactant is of particular importance when developing rinse off conditioning products. [6] For this project, only three sets of ingredients will be used in the formulation development. One set for classic emulsions and two for pickering emulsions. For this reason, sensory characteristics will be adjusted based on the added amount oil and surfactant, and the relation between them. The alterations of oil and surfactant concentration mainly affect the viscosity of the formulation, but some effects in other sensory characteristics mentioned in 2.1 also follows. The content of oil and surfactants will be altered in four ways for classic emulsions and pickering emulsions.

Classic Emulsions:

- Oil concentration
- Surfactant concentration
- Alteration in Oil/surfactant ratio
- Alteration in Oil phase/Water phase ratio

Pickering emulsions:

- Starch particle concentration
- Carbopol concentration
- Cellulose thickener concentration
- Carbopol + Cellulose thickener concentration

2.3 Ingredients

2.3.1 Classic emulsion

A formulation proposal for the conditioning hair treatment was given by Rapsodine to use as a starting point, see Table 2.2. The ingredients are described below. The same ingredients were used throughout the formulation process of the classic emulsions, with only the proportions between them changing.

Table 2.2: The ingredients used to formulate the classical emulsions. [23]

Ingredient name	Type	Function
Aqua	Water	Solvent
Cetearyl Alcohol	Nonionic surfactant	Conditioning, viscosity controlling
Behenamidopropyl Dimethylamine	Cationic surfactant	Emulsifying, antistatic
Cetearyl Glucoside	Nonionic surfactant	Emulsifying
Lactic Acid	Acid, pH regulator	Humectant, Buffering
Aloe Barbadensis Leaf Juice	—	Soothing, humectant
Panthenol	Reconstructor	Humectant, antistatic
Glycerin	Polyols	Humectant
Butyrospermum Parkii (Shea) Butter	Emollient/Wax	Superfatting agent, viscosity controlling
Canola Oil	Oil	Emollient
Simmondsia Chinensis Seed (Jojoba) Oil	Oil	Emollient
Sodium Benzoate	Preservative	Preservative
Potassium Sorbate	Preservative	Preservative
Apple Vingear Extract	Acid, extract	Antistatic, antioxidant

Aqua

Aqua (water) is used as a solvent in cosmetic products - it is probably the single most common ingredient in all of cosmetics. Both regular (tap) water and purified water is used with the purpose of solving water-soluble ingredients. In some cosmetics, functional water such as water from hot springs is used instead, however no effects have been shown that would make these types of water better suited for cosmetic use. [24]

Cetearyl Alcohol

Cetearyl Alcohol is a fatty alcohol mixture of predominantly cetyl alcohols and stearyl alcohols. It is an nonionic surfactant frequently used in hair conditioners and hair treatments as an emulsion stabilizing and viscosity increasing ingredient. It is suitable for both oil-in-water emulsions and water-in-oil emulsions.

Behenamidopropyl Dimethylamine

Behenamidopropyl Dimethylamine (BAPDMA) or AMIDET® APA-22 (trade name) is a biodegradable, cationic surfactant. It is synthesized at high temperature from behenic acid and dimethylaminopropylamine. Behenic acid is a fatty acid derived from rapeseed oil. When using Behenamidopropyl Dimethylamine in cosmetic hair rinse formulations, it is recommended to keep the pH-level around 4. Behenamidopropyl Dimethylamine is suitable for cosmetic products with a natural and organic profile thanks to its eco-toxicological properties such as low aquatic toxicity and rapid aerobic and anaerobic degradation. [25]

Cetearyl Glucoside

Cetearyl Glucoside is a kind of alkyl glucoside. It is a biodegradable nonionic, mild surfactant, used in hair treatments for its emulsifying effect. Cetearyl Glucoside is synthesized from glucose and fatty alcohols, often derived from coconut, rapeseed and palm oil. Alkyl glucosides are known to be skin sensitizing and can cause contact allergy. This is however more common when used on the skin and in leave-on products such as sunscreen. [26] [27]

Lactic Acid

Lactic acid is an organic acid, commonly used in the cosmetic industry to regulate the pH-level. It is generally considered safe for personal use and can be produced through chemical synthesis or fermentation with a number of different substrates. [28]

Aloe Barbadensis Leaf Juice

Aloe Barbadensis Leaf Juice (Aloe Vera gel) is a versatile and frequently used base ingredient in natural cosmetics. Over 75 active components have been recognized in Aloe Vera gel, such as protein, enzymes and vitamins. It is however not known which of these account for the healing, calming and moisturizing effect of the plant gel. [29]

Panthenol

Panthenol (D-panthenol) is the alcohol and provitamin of pantothenic acid (vitamin B5). In hair treatments, it is used for its lubricating and antistatic properties - it binds easily to the hair and coats it. Further, it improves strength, shine and detangling. [30]

Glycerin

Glycerin, also known as propantriol or glycerol, has a variery of application in cosmetic, food and chemical industry. Glycerin also accurs naturally in the body. It is used to moisturize the hair and to stabilize the hair treatment formulation. [31]

Butyrospermum Parkii (Shea) Butter

Butyrospermum Parkii Butter, also known as shea butter, consist mainly of fatty acids such as oleic acid, stearic acid, linoleic acid, palmitic acid, linolenic acid and arachidic acid. It has known antiinflammatory and antioxidant properties, as well as viscosity and refatting properties, making it suitable for many types of cosmetics. [32] [33]

Canola Oil

Canola oil is the name of oil from the genus Brassica, more specifically Brassica napus, Brassica rapa and Brassica juncea. It is a modified version of rapeseed oil - canola oil has a erucic acid content below 2 percent. This is mainly due to the health risks associated with oral intake of erucic acid, but has influenced the rapeseed-growing industry to such an extent that canola oil is the dominating oil compared to rapeseed oil in the cosmetic industry as well. The canola oil used by Rapsodine is made from Brassica napus. The use of vegetable oils in conditioning hair treatments has been shown to increase softness, strength and resistance to breakage. [34] [35]

Simmondsia Chinensis Seed (Jojoba) Oil

Simmondsia Chinensis Seed Oil, or jojoba oil, is actually not an oil or fat but a liquid wax. It contains wax esters, fatty acids, alcohols, hydrocarbons, sterols and vitamins. It is widely used in the cosmetics industry due to its emollient and antioxidant effect. [36]

Sodium Benzoate

Sodium Benzoate is the sodium salt of benzoic acid. In acid formulations, sodium benzoate converts to benzoic acid, which has antimicrobial effect and is effective against fungi and yeast. Sodium benzoate is not skin sensitizing, shows rapid biodegradability and low aquatic toxicity.[37]

Potassium Sorbate

Potassium sorbate is the salt of sorbic acid and converts to sorbic acid in acid formulations, similar to sodium benzoate and benzoic acid. Sorbic acid has documented inhibitory effect for the growth of a variety of bacteria, yeasts and moulds. Potassium sorbate is considered safe for human use and has rapid biodegradability, however the eco-toxicologial profile is still incomplete. [38] [39]

Apple Vinegar Extract

Apple Vinegar is vinegar produced from fermentation of apples and contains acetic acid and malic acid, as well as polyphenols and other acids in low concentrations. It has known antimicrobial and antioxidant properties. [40]

2.3.2 Pickering emulsion

For the pickering emulsions, the cationic surfactant (Behenamidopropyl Dimethylamine) and the nonionic surfactants (Cetearyl Alcohol Cetearyl Glucoside) were exchanged for xstarch particles and thickening agents (Polyacrylic acid Hydroxypropyl methylcellulose). The other ingredients were kept the same.

Starch particles

Starch is a plant based compound that can be obtained from various botanic resources [41], it is biodegradable and Generally Recognized As Safe (GRAS). The native starch particles are unsuitable as emulsifiers since they appear in a broad size range and have insufficient hydrophobicity, modification is thus necessary before the particles are applied as emulsifiers.[15] The starch particles used for this project will be isolated from quinoa grains and chemically modified by OSA (2.9 percent) to obtain higher hydrophobicity which will be required. [42]

Polyacrylic acid

(Carbomer/Carbopol/C10-30 Alkyl Acrylate Crosspolymer) is a commonly used polymer in the cosmetics industry. It thickens aqueous solutions by forming a networked microgel structure. [43]

Hydroxypropyl methylcellulose (HPMC)

HPMC is another widely used viscoelastic polymer in the cosmetics industry. It is based on cellulose, and is hydrophilic and biodegradable. It swells in aqueous solutions, making the viscosity of the solution increase. [44]

2.4 Laboratory equipment

In order to formulate the emulsions and to evaluate the rheological properties of them, the laboratory equipment described below was used.

2.4.1 Homogenisator

In the emulsification process, surfactants reduce the interfacial tension which allows the liquid to be dispersed into smaller particles. However, a supply of energy is needed in order to break the shear forces and comminute the droplets. For this purpose, an homogenization machine is used - it uses mechanical agitation to disperse a liquid into smaller particles. The homogenization machine used was an high-speed shearing homogenization machine. [45]

2.4.2 Particle size analyser

The particle size distribution of the emulsions is analyzed by using laser diffraction spectroscopy (Malvern Mastersizer 2000) and the measurement principle of Mie scattering. In laser diffraction spectroscopy, laser light is directed towards particles and the angular variation in intensity of scattered light is measured. The angular variation of the scattered light is determined by the particle size. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at larger angles. Using the Mie solutions to Maxwell's equations, particle size and particle size distribution. [46]

2.4.3 Microscope

Microscopy was performed as a complement to laser diffraction in order to determine the emulsions average particle size and stability. The assay was also used for visual inspection of the emulsion structure and to observe the amount of unnecessary emulsifier, free in solution.

Microscopy was also performed on damaged hair shafts to evaluate the hair quality before and after treatment.

2.4.4 Texture analyser

In order to test the compression strength of the hair, a texture analyser (TA-XT2i Texture Analyser) was used and a compression test was performed. The texture analyser takes measurements of force while either compressing or stretching an object or material. In this case, a strand of hair was compressed over a probe with edges - and the force needed to break the hair was measured.

Method

3.1 Hair treatment formulation

Hair treatment formulation was performed in two steps. The first step was equal for both classic and pickering emulsions, while the second step was customized for the different emulsion types. In the first step, ingredients were weighted and divided into two separate beakers, one containing the ingredients for the dispersed phase and another with the ingredients for the continuous phase. Ingredients included for each phase can be seen in Table 3.1 and Table 3.2.

Table 3.1: Ingredients in the continuous and dispersed phase for the classic emulsions.

Continuous phase	Dispersed phase
Aqua	Cetearyl Alcohol
Lactic Acid	Cetearyl Glucoside
Panthenol	Behenamidopropyl Dimethylamine
Glycerin	Jojoba Oil
Apple Vinegar Extract	Canola Oil
Sodium Benzoate	Perfume
Potassium Sorbate	
Aloe Barbadensis Leaf Juice	

The second step included heating and mixing of the phases. For the classic emulsion, each phase was heated to a temperature of 70 ° C - at which the dispersed phase was melted. The dispersed phase was slowly poured into the continuous phase while the continuous phase was being homogenized. The mixture was homogenized for 60 seconds. For pickering emulsions, both phases were heated to 45 ° C. The quinoa starch was added to the continuous phase and the dispersed phase was then slowly poured into the continuous phase during magnetic stirring. The mixture was then homogenized for 60 seconds.

Table 3.2: Ingredients in the continuous and dispersed phase for the pickering emulsions.

Continuous phase	Dispersed phase
Aqua	Carbopol/Carbomer
Lactic Acid	Hydroxipropylmetylcellulose
Panthenol	Q-80 modified quinoa starch
Glycerin	Jojoba Oil
Apple Vinegar Extract	Canola Oil
Sodium Benzoate	Perfume
Potassium Sorbate	
Aloe Barbadensis Leaf Juice	

Emulsions were prepared in four series for classic emulsions and pickering emulsions respectively. In each serie, a specific parameter was varied to study the impact on the formulations characteristics. Formulation series for classic and pickering emulsions are listed in table 3.3. In addition, classic formulations nr 9 and 11 was formulated without preservatives. Lists of all emulsions created, their serie belonging and ingredient concentration in mass % can be seen in table 3.4 and 3.5.

Table 3.3: Parameter changed in each serie of formulations.

Classic emulsion	Pickering emulsion
Oil concentration	Starch particle concentration
Surfactant concentration	Carbopol concentration
Oil/Surfactant ratio	HPMC concentration
Oil phase/Water phase ratio	Carbopol + HPMC concentration

Table 3.4: Formulation series, oil concentration (mass %), surfactant concentration (mass %), Oil/Surfactant ratio and Oil phase/Water phase ratio of all classic emulsions formulated.

Number	**Serie**	**Oil**	**Surfactant**	**O/S**	**O/W**
1 (original recipe)		0.9	10.2	0.09	0.13
13	Oil	4.5	10.2	0.49	0.18
2	Oil	9	10.2	0.68	0.25
8	Oil	10.8	10.2	0.98	0.27
10	Surfactant	0.9	5.1	0.17	0.07
5	Surfactant	0.9	7.9	0.11	0.10
6	Surfactant	0.9	13.3	0.07	0.17
12	O/W	1.35	15.3	0.09	0.21
4	O/W	2.7	29.6	0.09	0.49
3	O/W	4.5	51	0.09	1.27
7	O/W + O/S	4.5	18.9	0.25	0.30
14	O/W + O/S	6.3	15.3	0.41	0.28
15	O/W + O/S	7.5	20.9	0.36	0.41
9	No preservatives	0.9	10.2	0.09	0.13
11	No preservatives	1.35	15.3	0.09	0.21

Table 3.5: Formulation series, oil concentration (mass %), starch particle concentration (mass %), Carbopol concentration (mass %) and HPMC concentration (mass %) of all pickering emulsions formulated.

Number	Serie	Oil	Starch	Carbopol	HPMC
1	None	27	10	0.1	0
2	None	5	1	1	0
3	None	27	15	0.5	0
4	None	27	15	1	0
5	None	27	10	0.5	0
6	Carbopol	27	5.4	0.1	0
7	Carbopol	27	5.4	0.5	0
8	Carbopol	27	5.4	1	0
9	HPMC	27	5.4	0	0.1
10	HPMC	27	5.4	0	0.5
11	HPMC	27	5.4	0	1
12	HPMC	27	5.4	0	2
13	HPMC	27	5.4	0	3
14	Starch	27	7	0	3
15	Starch	27	10	0	3
16	Carbopol + HPMC	27	5.4	0.5	2.5
17	Carbopol + HPMC	27	5.4	0.5	3.5
18	Carbopol + HPMC	27	5.4	1	3.5
19	Oil	15	5.4	1	3.5
20	Oil	20	5.4	1	3.5
21	Oil	10	5.4	1	3.5

When the emulsions were created, two pictures were taken of each formulation. One picture were taken on the emulsions clicked out on a piece of glass. The other pictures shows the formulation smeared out.

3.2 Sensory characteristics evaluation

Evaluation of each formulations sensory characteristics was performed in order to select a few formulations with appealing properties to evaluate further.
Sensory characteristics were evaluated by simply touching and looking at the final formulations. All formulations were evaluated by the same two persons. The hair masks were then rated with a grading system containing five categories: Moisturizing, smoothness, cohesiveness, dryness and lightness. A more detailed explanation of what was considered within each category can be found in section 2.2.5, table 2.1.Three categories were considered positive for the sensory impression of

the hair masks: moisturizing, smoothness and cohesiveness, and two were considered negative: dryness and lightness. [6] The formulations was graded -1,0 or 1 in each category depending on if the formulation was considered to possess the property or not. -1 meant it did not posses the property, 0 was neither or and +1 meant it did possess the property. The given points were summarized according to equation 3.1.

$$P_p - P_n = Total \tag{3.1}$$

P_p = Points from positive categories
P_n = Points from negative categories

3.3 pH measurements

pH measurements were performed with pH paper to ensure an accurate pH in the range of 2.5-4.5. Measurements were performed on all formulations.

3.4 Laser diffraction

Laser diffraction spectroscopy was performed to determine the particle size distribution and evaluate the stability of the hair treatment emulsions. A Malvern Mastersizer 2000 instrument was used and the user manual was followed during the measurement procedure. The analysis was performed in duplicates of the selected formulations from sensory characteristics evaluation (section 4.1). Each sample was measured three times. No samples was diluted before added to the dispersion unit.

In order to evaluate stability, laser diffraction was performed in rounds of two. First measurement was performed 0-24 h after formulation and the second was performed approximately 5 weeks after formulation.

3.5 Microscopy

Microscopy images was taken on single samples of all formulations selected in sensory characteristics evaluation (section 4.1). Microscopy was performed in rounds of two to evaluate the emulsion stability, 24 hours after formulation and approximately 5 weeks after formulation. Three pictures at magnification x20 and x10 was taken at classic and pickering emulsions respectively. The second largest particle in each picture was measured, and a mean particle size value was calculated. Pictures at magnification x5, x10 and x20 was taken on both classic and pickering emulsions for visual comparison between the samples. All samples were diluted with a factor of 10 before microscopy was performed. Microscope model Olympus BX50 was used and the software JImage was used for particle size measurements. The complete collection of microscopy images can be found in Appendix C.

3.6 Microscopy on hair

Three damaged hair strains were selected, one hair strain for each formulation evaluated. Three microscopy images were taken 0-7 cm from the hair strains end before treatment, severely damaged spots were especially prioritized. The hair treatment formulation was added to the hair and left for 1-2 minutes before it was rinsed off. The hair was left to dry for 10 minutes before three new pictures were taken on the same area, after treatment.

3.7 Compression strength analysis

The texture analyser was calibrated and appropriate settings were selected. Tests were done on natural hair, both bleached and unbleached. The hair strands were rinsed, covered with hair treatment formulation for 60 seconds and then rinsed again. After being left to dry, the hair strains were compression tested. 13 measurements were done with each emulsion.

The data from the compression tests was processed and analysed. The peak force (hardness) was chosen to be especially relevant to analyse - since it shows how resilient the hair is to outside mechanical compression before breaking. Outliers in the data set were detected and removed using Grubbs test (two-sided, ($p<0.05$)).

3.8 User survey

The two classic emulsions (nr. C13 and nr. C14) and the one pickering emulsion (nr. P18) with the most promising results were sent out for consumer evaluation. The chosen emulsions were given to a panel of six hair mask users, along with a survey to fill out after at-home use of the hair masks. The hair mask users had used the hair mask 1-2 times before filling out the evaluating survey anonymously. In the survey, the users were asked to rate the hair mask on a scale of 1-5 in different parts of the hair mask using process:

- Before application
- During application
- During rinsing
- After use

The user survey also included open questions:

- What did you think was positive about the hair mask?
- What improvement opportunities do you see with the hair mask?
- Do you have any other comment about the hair mask?

These questions were however voluntary and were therefore not answered by all panel participants.

Results

4.1 Formulation development and sensory characteristics evaluation

Classic emulsions

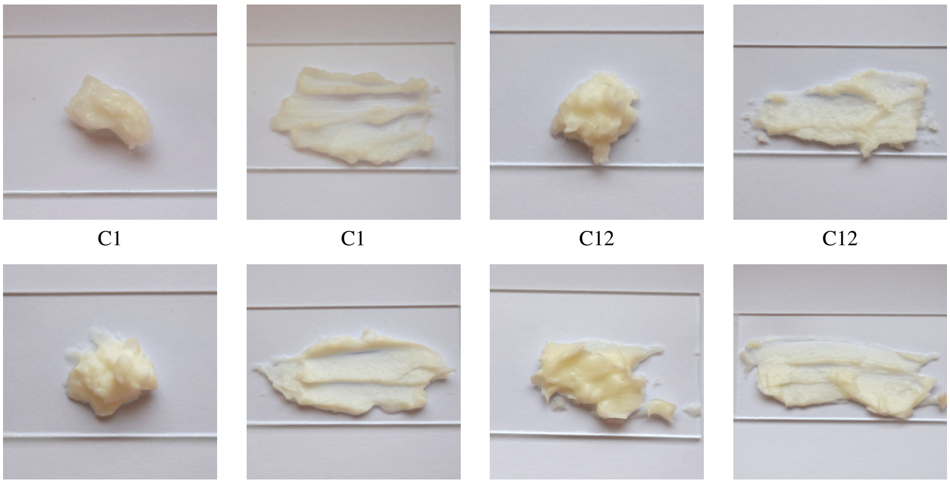
Classic emulsion overall gained high scores in the sensory characteristics evaluation. Properties such as thickness, cohesiveness and moisturizing sense were all varying to a wide extent, while properties such as slippyness and smoothness were changed to a lesser degree. Large differences could be seen in the formulations appearance, some formulations were gel-like, almost semi-transparent, white/gray and shiny while others presented a clear, compact color of white/yellow. Formulations within the earliest mentioned category were 9, 10, 11. Formulation 1 looked to be in between and the remaining formulations were within the latest mentioned category but deviated to a small degree in terms of color shade and shine. Formulation 9,10 and 11 were also comparably rinny and loose, while formulation 3 and 4 possessed significant thickness and cohesiveness. Other differences seen were formulation 1 and 10 being sufficiently viscous, but felt thin and airy when being smeared on the skin.

The formulations which gained the most points during sensory characteristics evaluation were 8, 12, 13 and 14. Of these, formulations 12, 13 and 14 were selected for further investigation. Formulation 1 was also picked as a reference.

Pickering emulsions

A compact, cream-like formulation was not achieved in any of the pickering hair treatments created. Compared to classic emulsions, pickering emulsions possessed a looser, more gel-like sense. P1, P3, P5, P6, and P7 showed graininess and seemed to separate immediately. Formulation P4 separated during the addition of starch particles and were thus discarded from the formulation results. P2 and P21 were completely liquid while P13, P17, P18, P19 and P20 showed similar and sufficient cohesiveness. Pickering emulsions were all shiny and possessed a non-translucent color tone of green/yellow.

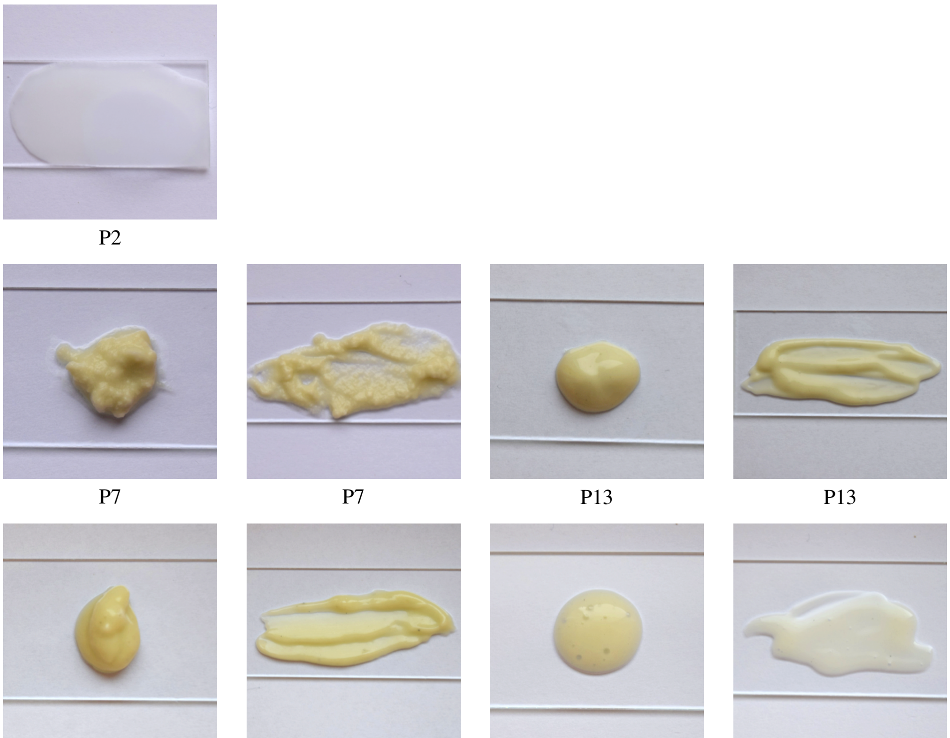
From sensory characteristics evaluation, P13 and P18 gained the highest scores and were selected for further analysis and evaluation. P2, P21 and P7 were also selected as references.



C1 C1 C12 C12

C13 C13 C14 C14

Figure 4.1: Two pictures of respective classic formulation selected in sensory characteristics evaluation. One picture shows the formulation clicked out on a piece of glass, the other picture shows the formulation smeared out.



P2

P7 P7 P13 P13

P18 P18 P21 P21

Figure 4.2: Two pictures of respective pickering formulation selected in sensory characteristics evaluation. One picture shows the formulation clicked out on a piece of glass, the other picture shows the formulation smeared out.

The total points from the sensory evaluation of the classic emulsions and pickering emulsions are presented in Table 4.1 and 4.2. Further descriptions of each emulsion, as well as more details regarding the scoring of points, can be found in Appendix B.

Table 4.1: Summary of the sensory evaluation of the classic emulsions. The total points of the evaluations are presented. The formulation numbers correspond to the photographs presented in Appendix A.

Formulation	Total points
1	2
2	4
3	1
4	2
5	5
6	4
7	4
8	5
9	4
10	2
11	3
12	5
13	5
14	5

Table 4.2: Summary of the sensory evaluation of the pickering emulsions. The total points of the evaluations are presented. The formulation numbers correspond to the photographs presented in Appendix A.

Formulation	Total points
P1	-1
P2	-
P3	3
P5	4
P6	3
P7	4
P8	-
P9	3
P10	1
P11	3
P12	2
P13	4
P14	3
P16	4
P17	3
P18	4
P19	3
P20	3
P21	4

4.2 pH-measurement

pH-measurement was done on all formulations with pH-paper, results are presented in Table 4.3 and Table 4.4.

Table 4.3: The pH-value of the classic emulsions. The values marked with asterisks are uncertain, due to difficulties with the measurements.

Formulation	pH
1	4.5
2	4.5
3	5*
4	6*
5	4.5
6	5
7	4.5
8	4,5
9	4
10	4
11	4.5
12	4
13	4
14	4.5

Table 4.4: The pH-value of the pickering emulsions.

Formulation	pH
P1	3
P2	2.5
P3	3
P5	3
P6	3
P7	3
P8	3
P9	3
P10	4
P11	3
P12	3.5
P13	4
P14	3
P16	3.5
P17	3.5
P18	4
P19	3.5
P20	4
P21	3

4.3 Laser diffraction

The following section includes results from laser diffraction measurements on the hair treatment emulsions selected in sensory characteristics evaluation (section 4.1). Duplicates were measured for all formulations except P2 and P13. Particle size distribution graphs for all conducted measurements can be found in Appendix E.

Classic emulsions

Particle size distribution curves generally include several peaks in a wide spectrum. By comparing laser diffraction graphs with mean particle size from microscopy results, peaks apparent in the interval of 5-20 μm were selected as the most accurate for emulsion droplet size estimations. Detection signals outside the selected peak spectrum were excluded since these peaks are likely to originate from micelles, WLM or liquid crystalline structures. Values of D_{10}, D_{50} and D_{90} included in Table 4.5 were read from the particle size distribution graphs and are mean values of the three conducted measurements of each sample. Full distribution graphs of all samples can be seen in Appendix E.

Pickering emulsions

Particle size distributions of pickering emulsions generally include the largest volume fraction of particles in the interval of 10-100 um. All sample measurements also include a small peak in the interval 0.1-2 μm. Values of D10, D50 and D90 were obtained directly from Mastersizer 2000 software (see Table 4.6), and no adjustments were done regarding exclusion of irrelevant signals.

Table 4.5: D_{10}, D_{50} and D_{90} values obtained from laser diffraction measurements of classic emulsions.

Sample	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)	D_{90} / D_{10}
C1.1 (24h)	2.5	6.2	10.3	4.1
C1.1 (5 weeks)	5.1	10.6	43	8.43
C1.2 (24h)	6.6	24.3	63.3	9.6
C1.2 (5 weeks)	8.7	34.7	87	10
C12.1 (24h)	8.7	56.7	111.7	12.9
C12.1 (5 weeks)	4.7	16	57.7	12.1
C12.2 (24h)	2.9	8.1	10.4	3.6
C12.2 (5 weeks)	3.2	10.0	18.9	5.9
C13.1 (24h)	2.4	6.9	11.3	4.7
C13.1 (5 weeks)	4.2	10.3	44.7	10.6
C13.2 (24h)	8	62.7	250	31.2
C13.2 (5 weeks)	3.3	7.4	19.3	5.9
C14.1 (24h)	12	35.3	53.3	4.44
C14.1 (5 weeks)	2.3	8.5	77	33.5
C14.2 (24h)	2.9	6.3	16.3	5.6
C14.2 (5 weeks)	4.3	14	54	12.6

Table 4.6: D_{10}, D_{50} and D_{90} values obtained from laser diffraction measurements of pickering emulsions.

Sample	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)	D_{90} / D_{10}
P2.1 (24h)	2.1	19.5	42.1	20.0
P2.1 (5 weeks)	5.9	29.0	64.9	11.0
P7.1 (24h)	24.5	59.1	122.7	5.0
P7.1 (5 weeks)	47.2	107.9	236.1	5.0
P7.2 (24h)	4.3	27.9	56.8	13.2
P7.2 (5 weeks)	17.8	49.7	109.5	6.2
P13.1 (24h)	1.5	9.2	110.2	73.5
P13.1 (5 weeks)	2.2	17.1	54.4	24.7
P18.1 (24h)	7.3	21.9	55.2	7.7
P18.1 (5 weeks)	4.4	32.5	67.4	15.3
P18.2 (24h)	1.6	14.5	217.7	136.1
P18.2 (5 weeks)	6.4	29.8	69.1	10.8
P21.1 (24h)	2.5	10.9	47.9	19.2
P21.1 (5 weeks)	6.4	25.5	110.8	17.3
P21.2 (24h)	3.7	240.2	370.1	100.0
P21.2 (5 weeks)	14.8	39.9	81.7	5.5

4.4 Microscopy

The following section includes results of emulsion particle size estimations obtained from microscopy. Microscopy was performed to investigate the emulsion structure and emulsion stability. All formulations selected in sensory characteristics evaluation (section 4.1) was analyzed. The full collection of microscopy images can be found in Appendix C.

Classic emulsions

Microscopy images of classic emulsions show a low concentration of emulsion oil droplets where the droplet size ranges between 1.8-6.4 μm. Smaller particles can be glimpsed but are hard to distinguish due to insufficient resolution at higher magnification degree. The droplet size distributions are even and no significantly large droplets could be observed for any of the analyzed formulations, however, larger "cloud-like" structures are present in all observed samples. The cloud structures can be described as darker, blurred fields and constitute a large proportion of the image area.

No notable changes could be seen while comparing microscopy images taken 24 hours after formulation with images taken 5 weeks after formulation. The average particle size slightly decreased for all formulations. Microscopy images of classic formulation C1 state an example of the typical classic emulsion results in figure 4.3, an emulsion oil droplet is marked in red. Cloud structures are marked and seen in figure 4.4.

Table 4.7: Particle diameter for classic emulsions measured 24 hours after formulation. Values are obtained from the second largest particle in each picture.

Formulation	**D^1(μm)**	**D^2(μm)**	**D^3(μm)**	**Mean value** (μm)
C1	2.8	3.1	3.5	3.1
C12	3.2	4.0	3.3	3.5
C13	4.0	6.4	5.4	5.2
C14	3.7	4.6	5.3	4.5

Table 4.8: Particle diameter for classic emulsions measured 5 weeks after formulation. Values are obtained from the second largest particle in each picture.

Formulation	**D^1(μm)**	**D^2(μm)**	**D^3(μm)**	**Mean value** (μm)
C1	1.9	2.2	2.5	2.2
C12	2.3	1.5	3.1	2.3
C13	2.2	1.9	2.5	2.2
C14	2.2	1.9	2.2	2.1

Pickering emulsions

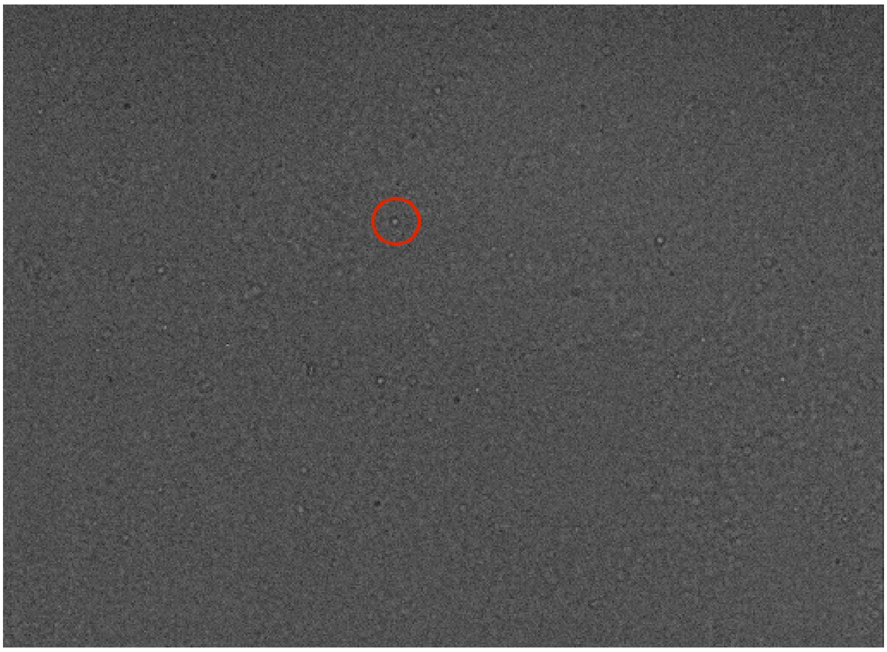
Pickering emulsion's microscopy results obtained 24 hours after formulation indicate wide particle size distributions and an average particle diameter in the spectrum of 11-43 μm. Particle size estimates obtained 5 weeks after formulation were in the interval of 22-62 μm. Observations of increased average particle size apply to all formulations except P2. Flocculation can be seen to occur for all formulations already 24 hours after formulation and increased dramatically in 5 weeks. Formulation P13 state an example of the typical pickering emulsion microscopy result, this can be seen in figure 4.5.

Table 4.9: Particle diameter for pickering emulsions measured 24 hours after formulation. Values are obtained from the second largest particle in each picture.

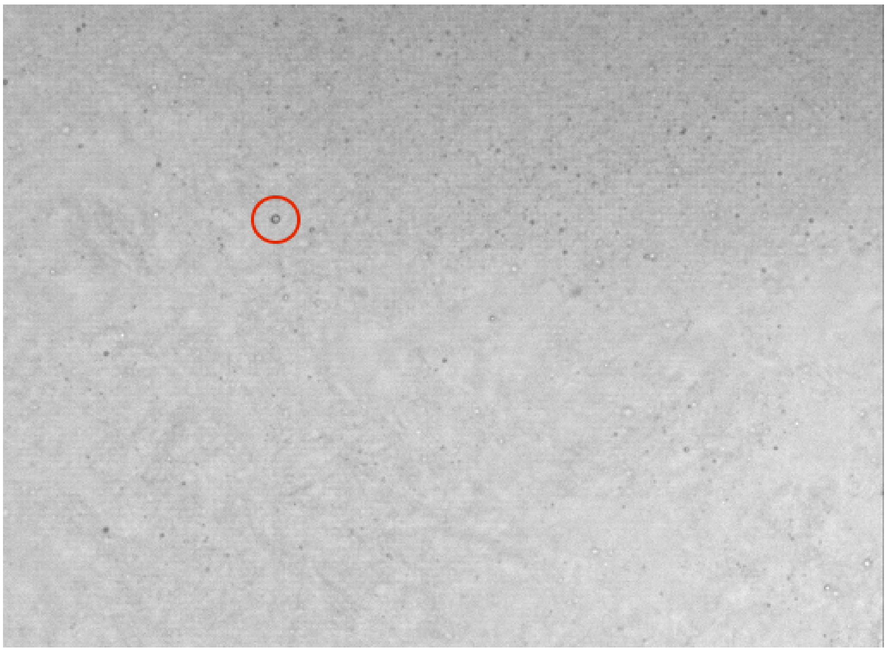
Formulation	**D^1(μm)**	**D^2(μm)**	**D^3(μm)**	**Mean value** (μm)
P2	38	49	43	43
P7	26	27	15	23
P13	16	27	17	20
P18	31	22	18	24
P21	7	14	12	11

Table 4.10: Particle diameter for pickering emulsions measured 5 weeks after formulation. Values are obtained from the second largest particle in each picture.

Formulation	**D^1(μm)**	**D^2(μm)**	**D^3(μm)**	**Mean value** (μm)
P2	28	33	44	35
P7	17	51	26	31
P13	23	19	25	22
P18	17	41	99	52
P21	27	19	16	62

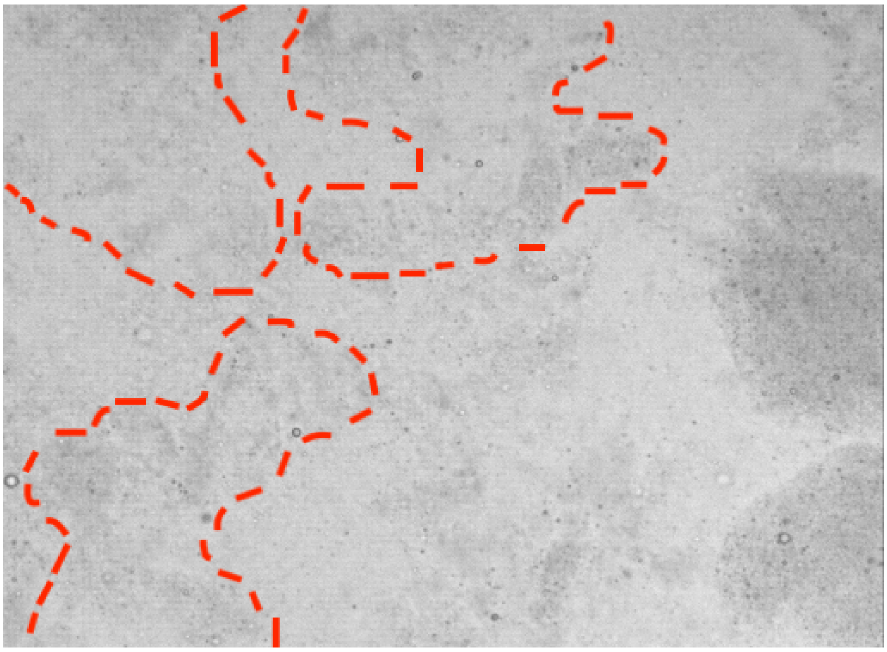


(a) 24 hours



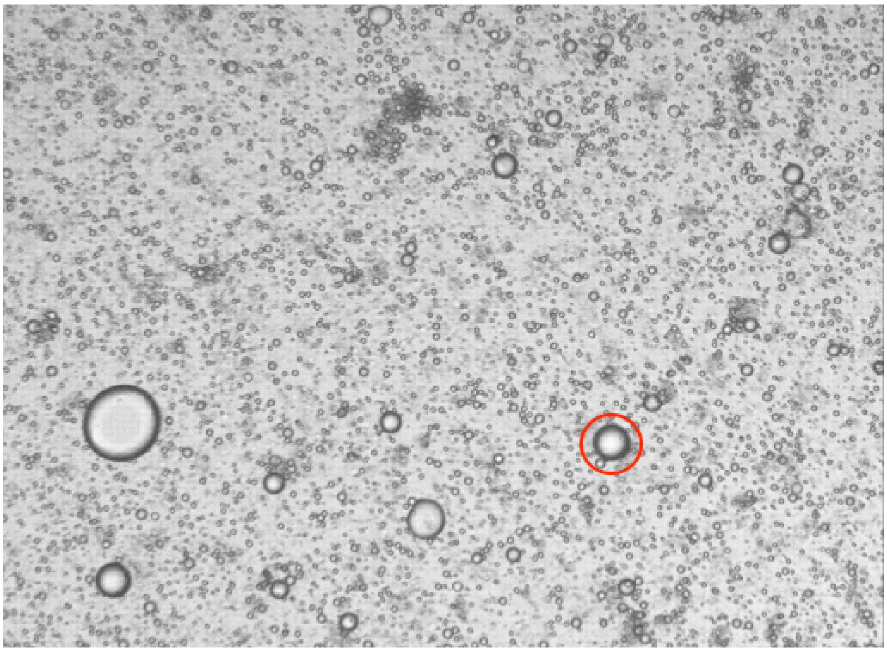
(b) 5 weeks

Figure 4.3: Microscope images of classic emulsion C1 at magnification degree x20. Pictures was taken 24h (a) and 5 weeks (b) after formulation. An example of a dispersed oil droplet is marked in red.

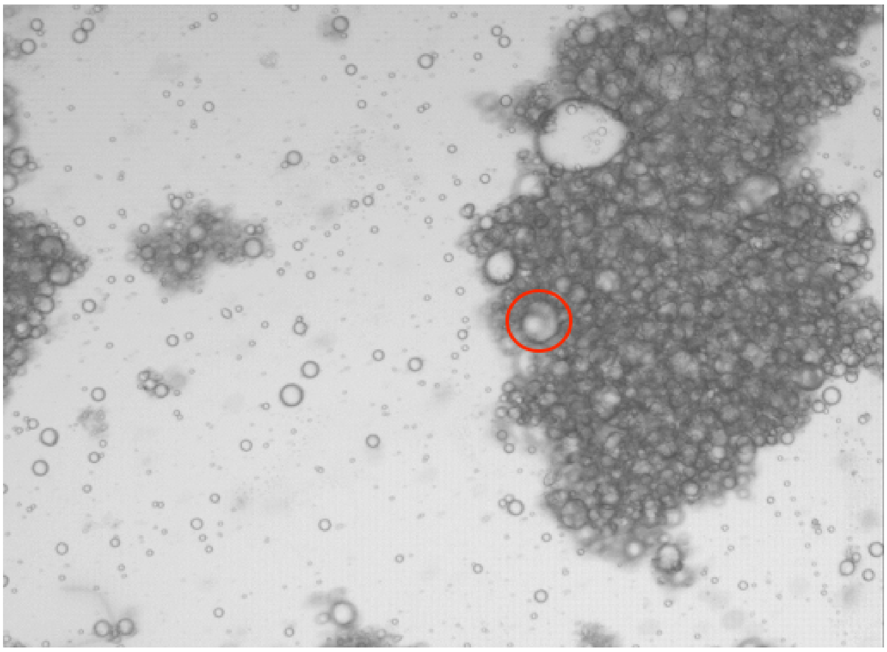


(a) Cloud structure

Figure 4.4: Microscope images of classic emulsion C13 (5 weeks) at magnification degree x20. Examples of cloud structures are marked in red.



(a) 24 hours



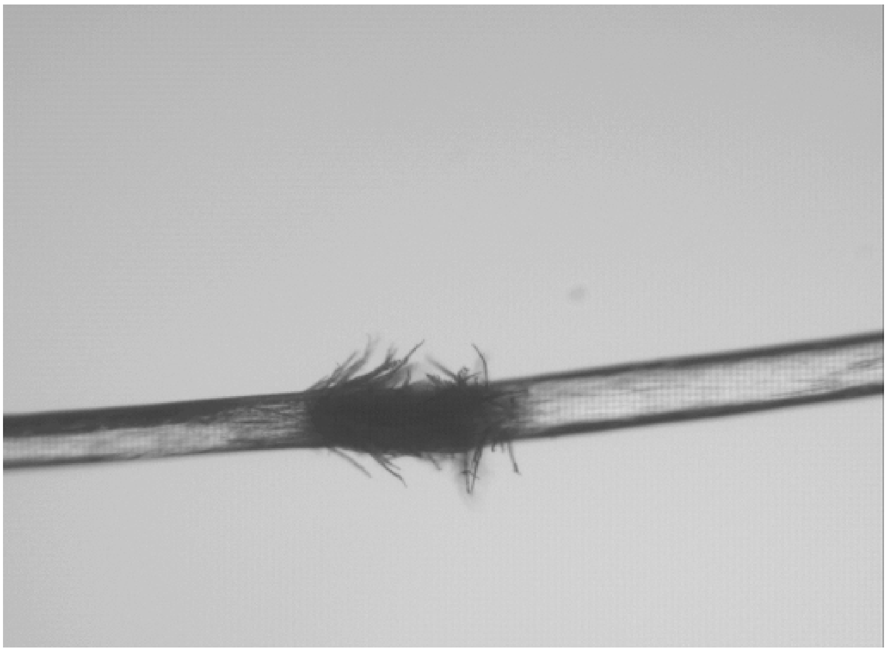
(b) 5 weeks

Figure 4.5: Microscope images of pickering emulsion P13 at magnification degree x10. Pictures was taken 24h (a) and 5 weeks (b) after formulation. An example of a dispersed oil droplet is marked in red.

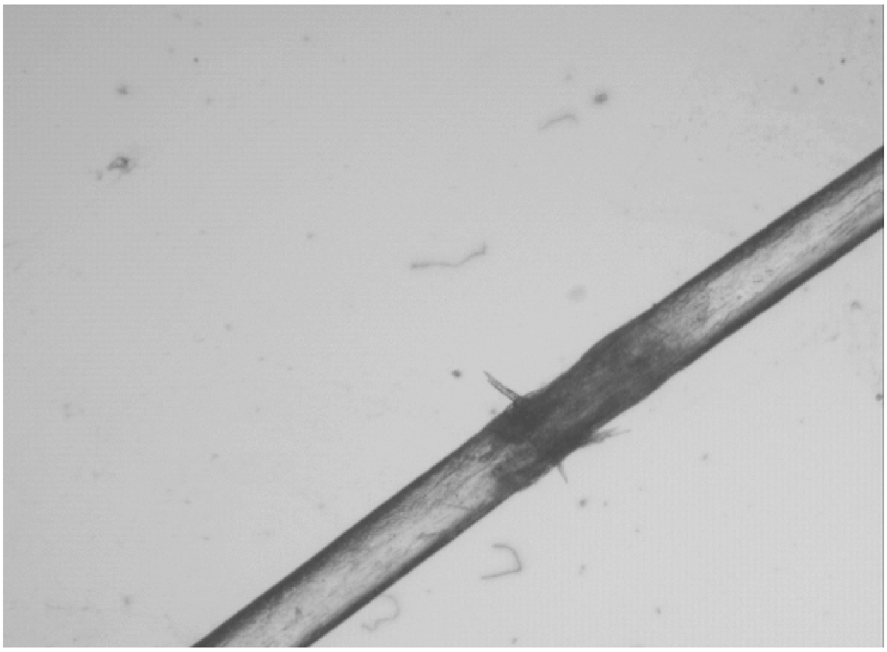
4.5 Microscopy hair

The following section includes microscopy images taken on hair tips before and after hair treatment with formulation C13, C14 and P18. All pictures taken can be found in Appendix D.

All formulations could be seen to patch together protruding fibrous elements and enclose the hair tip.

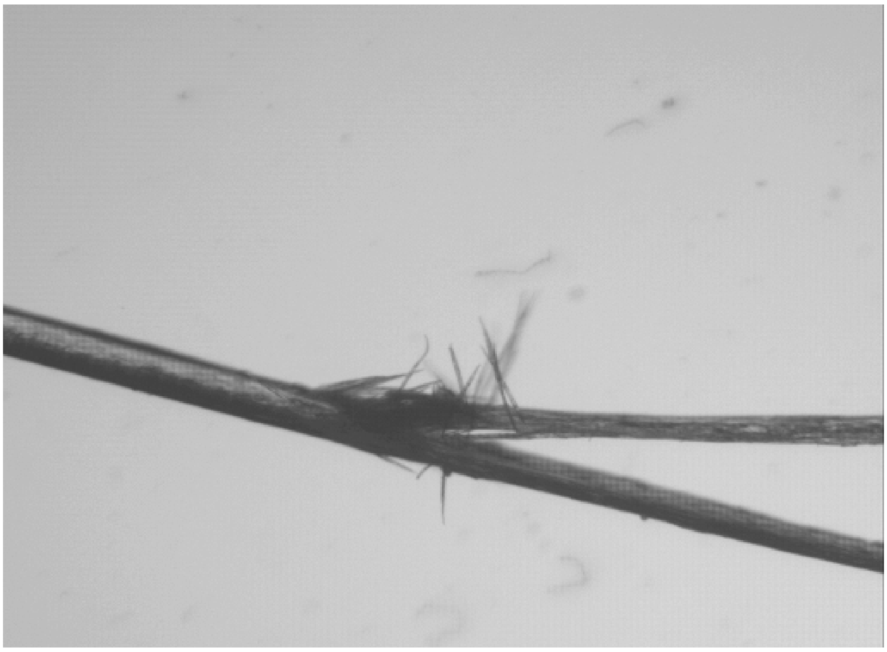


(a) Before treatment

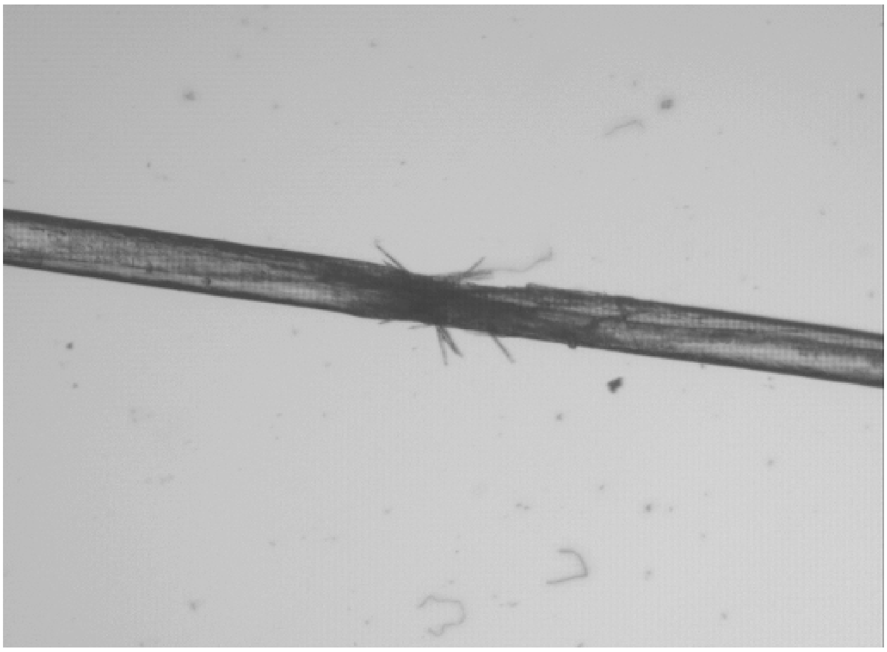


(b) After treatment

Figure 4.6: Microscopy images taken on a damaged hair strain before and after treatment with formulation C13

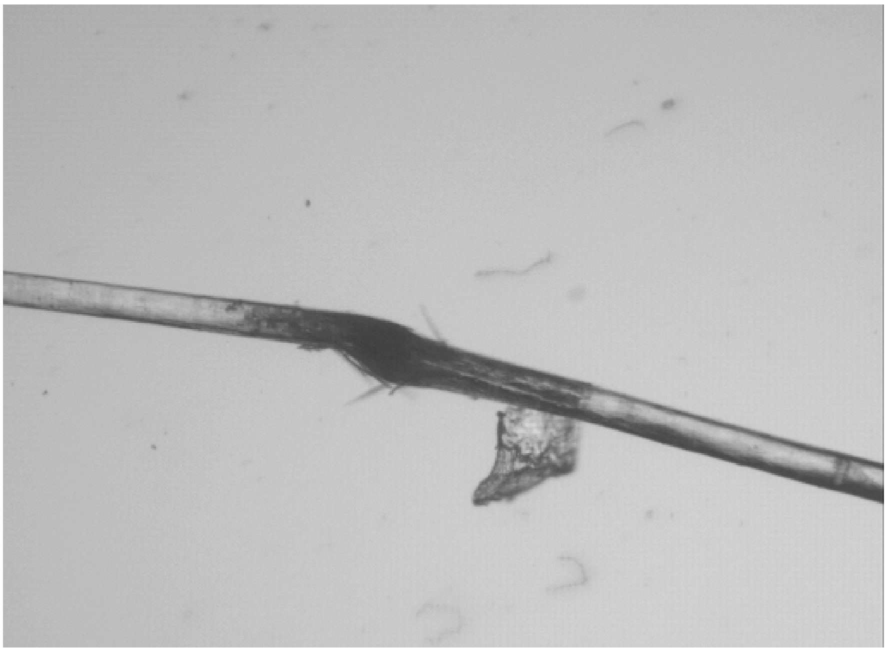


(a) Before treatment

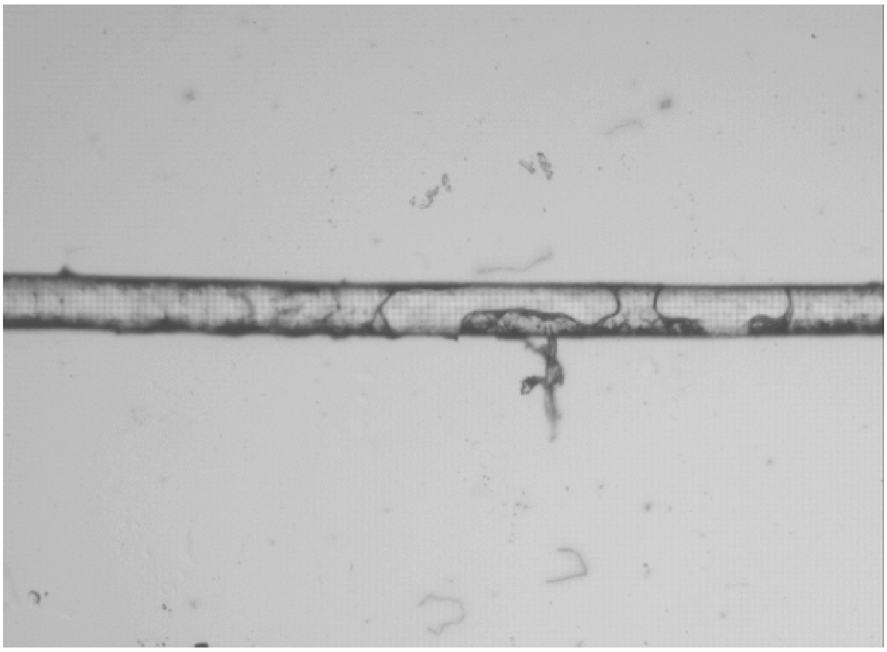


(b) After treatment

Figure 4.7: Microscopy images taken on a damaged hair strain before and after treatment with formulation C14



(a) Before treatment



(b) After treatment

Figure 4.8: Microscopy images taken on a damaged hair strain before and after treatment with formulation P18

4.6 Compression test

The peak force (hardness) results are presented in Table 4.11 and Table 4.12 with the outliers removed. An ANOVA-test resulted in p-value=0.39 for the bleached hair and p-value=0.81 for the unbleached hair.

Table 4.11: Hardness of bleached hair in (N). Standard deviation (σ) and mean average are presented below.

	Untreated hair	Emulsion nr. 1	Emulsion nr. 12	Emulsion nr. 13	Emulsion nr. 14
	30,4	26,9	27,2	23,9	24,1
	20,2	22,2	22,6	20,2	21,5
	24,1	21,5	24,3	22,0	19,3
	33,2	32,3	24,2	21,9	21,5
	21,4	21,8	23,8	21,0	24,0
	22,8	22,9	23,4	24,5	23,6
	25,3	21,3	21,1	26,7	21,2
	24,4	26,0	28,2	23,7	26,6
	21,3	28,7	25,4	23,3	23,6
	21,0	26,1	25,0	29,8	19,0
	21,5	22,2	26,2	23,5	25,5
	21,7	20,2	25,0	27,1	21,0
	22,4	21,1	27,5	25,7	22,7
σ	3,72	3,48	1,95	2,58	2,18
Average	23,80	24,09	24,91	24,10	22,57

Table 4.12: Hardness of unbleached hair in (N). Standard deviation (σ) and mean average are presented below.

	Untreated hair	Emulsion nr. 1	Emulsion nr. 12	Emulsion nr. 13	Emulsion nr. 14
	20,8	22,0	22,0	19,8	23,6
	21,6	21,3	25,9	20,6	27,4
	19,9	26,2	22,7	25,1	20,1
	27,3	21,8	22,1	25,6	18,3
	21,3	20,9	20,5	21,2	19,5
	26,6	21,0	26,3	19,0	20,9
	28,8	20,7	30,4	23,0	23,6
	20,1	23,7	19,3	21,8	23,6
	23,6	25,3	26,7	19,8	29,0
	18,7	20,9	19,3	27,1	20,7
	18,9	19,6	20,8	23,1	25,6
	21,3	19,6	22,7	22,1	22,9
	19,8	23,4	22,3	23,4	24,4
σ	3,21	1,97	3,15	2,36	3,00
Average	22,21	22,03	23,15	22,41	23,05

4.7 User survey

The results from the user survey are presented in Table 4.13. The mean value for each emulsion in each part of the hair mask using process is presented and the total points are summarized.

The total points show that emulsion nr. C13 and emulsion nr. C14 were given similar points while emulsion nr. P18 was given significantly lower points. The difference in points is especially evident after use, meaning that there is less difference in quality in the shower.

Table 4.13: Mean value points from the user survey. The formulation numbers correspond to the photographs presented in Appendix A.

	Nr. C13	Nr. C14	Nr. P18
Before application	3.5	3.3	2.8
During application	3.3	3.0	2.5
During rinsing	3.4	3.7	3.2
After use (dry hair)	4.1	4.0	1.7
Total points:	14.3	14	10.2

Discussion

5.1 Formulation development and sensory characteristics evaluation

Classic emulsions

Formulation C9, C10 and C11 possessed a distinctive appearance in terms of semi transparency, more shine and grayer color compared to other classic emulsions. By analyzing distinctive things within the recipes of these formulations (Table 3.4), low oil content seems to be one factor causing the typical appearance. The only difference between recipe C1 and C9 is exclusion of preservatives, the same applied to formulation C11 and C12. Since formulations C1 and 12 are less transparent and express a more yellow color tone, exclusion of preservatives seems to affect the emulsion appearance to a high degree. Particle distribution is known to affect the emulsion transparency since visible light isn't scattered by particles with a diameter below the visible light wave band. None of formulations C9,C10 and C11 were analyzed by laser diffraction or microscopy, thus no conclusions can be drawn regarding particle distributions and appearance of the formulations concerned.

For all formulations, the viscosity of the continuous phase is constant and the particle distribution is not varying to any significant degree. Thus, the concentration of surfactant and volume fraction of the dispersed phase are the factors affecting the thickness perception. Formulations C3 and C4 were significantly thick and cohesive which is a consequence of high surfactant concentration (29.6 and 51 m% respectively), thus also increased O/W ratio (0.49, 1.27 respectively). The opposite is seen to apply for formulation 10 which is loose and less cream-like. For comparison, formulation 10 has a surfactant content of 5.1 m% and an O/W ratio of 0.07 (Table 3.4). Preservatives also seem to affect the thickness sensory characteristics since formulation C9 and C11 were more runny than formulation C1 and C12 where preservatives were included.

Pickering emulsions

Formulation P4 separated during the addition of starch particles while P3, P5, P6 and P7 were successfully homogenized but all exhibited high graniness and fast separation. There are two aspects of why this may occur. Solid starch particles constitute an excessive part of the volume which prevents sufficient mixing of the two phases during homogenisation and thus entails immediate separation. Another reason is that the formulations contained insufficient concentration of polymer which entailed low viscosity and thus fast separation. The choice of polymer is also considerable for the formulation result. Recipes of formulation P7 and P10 were the same but P7 contained solely Carbopol as the thickening agent while P10 contained HPMC. P10 exhibited quite low viscosity but no graniness. Since P13 and P18 gained the highest score in sensory characteristics evaluation, HPMC or a combination of Carbopol+HPMC seemed to be the best option for optimization of sensory characteristics. Overall, pickering emulsions showed less appealing sensory characteristics compared to classic emulsions and thus lower scores in the sensory evaluation.

5.2 pH

As presented in Table 4.3, all classic emulsions (with exception of nr. C3 and nr. C4) have a pH-value around pH 4, which is the suitable value when using Behenamidopropyl Dimethylamine as a cationic surfactant. Due to the high viscosity of emulsion nr. C3 and emulsion nr. C4, the pH-measurement of them was difficult and their pH-values are therefore uncertain.

The pickering emulsions all exhibit pH-values below 5.5, making their pH satisfactory with regard to protecting the scalp from irritation. Further, they exhibit lower pH than the classic emulsions meaning that their lubricating and static electricity reducing properties are expected to be better.

5.3 Emulsion stability and particle size distribution

Classic emulsions

A general observation of laser diffraction distributions obtained 24 hours after formulation is a population of particles in the size spectrum of 6.2-62.7 μm. The average size of this particle population is relatively aligned with the diameter measurements of small, circular structures obtained from microscopy images, which have an average particle size between 3.1-5.3 μm. Even though the mean particle size of the population concerned is larger in laser diffraction results, the population is most likely the same as the one observed in microscopy, and thus most likely emulsion oil droplets. Cloud structures can be seen to occupy big parts of the microscopy image area and are thus hard to define and determine the size of. From what can be seen, they estimated to be in the size spectrum of 50-150 μm, which correlates well with the peak obtained in the largest size spectrum in laser diffraction results. The cloud structures are seen as brighter, blurry segments and the molecular structure can not be studied in any greater detail. Since they occupy a large volume fraction of the formulation, they are likely to be either WLM micelles or liquid crystalline structures formed by cetearyl alcohol (Montanov 68 BM) and Behenamidopropyl Dimethylamine (Amidet APA-22). These structures are what generates the high viscosity obtained in all classic formulations created.

Particle distributions of duplicates performed 24 hours after formulation deviate greatly in all formulation measurement series. Sample C1.2, C12.1, C13.2, C14.1 (24h) and C1.2 (5 weeks) has D50 values 20-45 μm larger than the other samples within the series. The same samples express wider distributions, which indicates that the peaks may have been overlapped by intensity signals obtained from cloud structures. Since no remarkably large particles could be observed during microscopy analysis, the largest proportion of emulsion droplets are most likely to be smaller than results from laser diffraction indicate.

By comparing results 24 hours after formulation with results 5 weeks after formulation, no significant differences could be seen in microscopy. When excluding samples with overlapped emulsion droplet peaks, laser diffraction indicates a small increase in the D50 value for all formulations. Overall obtained stability of all classic emulsions analyzed can thus be stated. The preserved stability is reasonable considering the large volume fraction of liquid crystalline structures seen, and the high viscosity all classic formulations possess.

Pickering emulsions

Microscopy observations performed 24 hours after formulation indicate large emulsion oil droplets with average size estimations between 22-62 μm. Laser diffraction particle size distributions generally include one peak in the spectrum of 10-100 μm. The peak obtained in this spectrum is thus likely to arise from emulsion oil droplets. Small, dark spots can be seen throughout the images, both surrounding emulsion droplets and free in solution. These dark clusters are likely to be accumulated starch. Laser diffraction signal in the spectrum of 0.1-2 μm is seen in all particle distribution graphs and is likely to arise from free starch particles.

All pickering emulsions are stated as non stable. Flocculation is seen to occur 24 hours after formulation, and increased significantly in 5 weeks for all formulations. The steric hinder obtained from surface adsorbed starch is likely to provide protection for coalescence but does not hinder the emulsion droplets to form flocks. No elongated polymer structures could be seen during microscopy, and the viscosity of pickering emulsions were significantly lower for pickering emulsion than classic emulsions. Stability could be obtained by increasing the polymer concentration and thus the viscosity.

5.4 Microscopy on hair before and after treatment

All hair treatments were seen to improve the hair quality by enclosing the hair shaft and patching together protruding fibrous elements. No significant difference could be seen regarding hair treatment effect while comparing the formulations, but pictures taken after treatment of formulation P18 shows a clear attachment of product on the surface of the hair shaft. The distinctive surface layer seen in figure 4.8 b is likely to be oil. The oil phase of formulation P18 is significantly larger than in formulation C13 and C14.

5.5 Compression

The results from the compression tests show that there is no significant difference between the hardness of untreated hair or hair treated with any of the hair treatments, neither for the bleached nor the unbleached natural hair.

However, this could be due to the fact that the hair treatments only were left on the hair for 60 seconds. Further tests could be done with the treatment left on the hair for 2 minutes, 5 minutes or 10 minutes so evaluate if longer exposure to the hair treatment has any significant effect.

Further, it could be argued that a tensile test where the hair is pulled apart would have been more appropriate since this is a more common stress that hair is naturally exposed to. The reason for the choice of a compression test instead of a tensile test is simply due to difficulties with setting up the apparatus in a way that allowed for an tensile test. With more suitable probe equipment, a tensile test on the hair would have been ideal.

5.6 User survey

As mentioned, emulsion nr. C13 and emulsion nr. C14 were given similar points while emulsion nr. P18 was given significantly lower points. These results are consistent with the sensory evaluation where the pickering emulsions were given lower points in general, and emulsion nr. P18 has lower points than emulsion nr. C13 and emulsion nr C14.

While the points for emulsion nr. C13 and emulsion nr. C14 are quite similar, there are some differences to be found in the comments regarding the emulsions. Nr. C13 was described as more light and giving the hair a more soft feeling, while nr. C14 was stated to be more heavy. This could be explained by the higher oil content and higher surfactant content in emulsion nr. C14.

For the classic emulsions, the points are quite consistent throughout the entire user experience - with somewhat higher points after use/in dry hair. Therefore, further development of the hair mask should focus on improving the in-shower feeling.

With the pickering emulsion, it is the other way around. Most improvement is needed for the after use-feeling. Comments submitted regarding emulsion nr. P18 reveal that users found the hair mask heavy and difficult to rinse out, and that the hair felt somewhat oily and weighted down even after drying. This could be due to the significantly higher oil content in the pickering emulsion, compared to the classic emulsions.

Further, it would have been interesting to compare how the emulsions with high points and low points in the sensory evaluations were rated in a user survey, in order to investigate whether the emulsions with high points in the sensory evaluation actually were better hair masks according to consumers.

Lastly, it should be pointed out that not many reliable conclusions can be drawn from the survey since only six users have participated. Further research could be done with a bigger panel and more extensive survey questions, in order to get a better basis for understanding the consumer assessment of the hair masks and to understand what qualities lie behind the given points.

5.7 Evaluation of methods

One part of the project was to develop a method for analysing and evaluating the hair mask emulsions and their properties. It was not entirely clear which methods were suitable for the purpose of this project, since there is no given protocol for hair mask analysis. Below an evaluation of the different methods used is presented.

Sensory evaluation was performed on all emulsions, along with the emulsions being photographed. The method was useful for gaining a overview of all emulsions and their differences. Even more thorough evaluations would have been more helpful for drawing conclusions regarding the emulsions properties.

pH measurement was done using pH paper, which was somewhat problematic with the most high-viscose emulsions. Nonetheless, the method was simple and provided relevant information. The pH-value of the solutions provides knowledge regarding the emulsions qualities as a hair mask, as described in section 2.1.4.

Laser diffraction and microscopy in combination provided relevant information regarding the particle size distribution, particle population behavior and changes in the distribution over time. It was especially advantageous to perform laser diffraction and microscopy in combination, to see how the results correlate to and confirm each other.

Microscopy of hair strands was done before and after use of hair mask as a complement to other methods used for evaluating the effect of the hair mask on hair, to explore if the hair mask has any visual effect on the visible hair damage. This method showed great promise and could be used much more extensively if it had not been for the time constraint.

Compression tests were done to study the hardness of the hair strands. Although it being a relevant parameter to measure, a tensile test would have been much more appropriate since it more closely resembles the stress that hair is exposed to in the real world,

User survey was chosen due to it being time efficient. The method provided information regarding how the hair masks are perceived by consumers, and would have been even more useful with a bigger panel of at-home users. However, a interview with consumers would probably have been even more valuable to gain insight regarding which qualities consumers seek in a hair mask.

Other analysis methods that would have been relevant to include are:

Viscosity measurement in order to have comparative values of the viscosity of the different emulsions. This would have been useful for drawing conclusions regarding how changes in the formulation affects the emulsions properties.

Water content measurement in order to investigate any changes to water content before and after use of the hair masks, which would give information regarding the tensile properties of the hair in relation to hair mask use.

Conclusion

Many conclusions can be drawn from the project, both general conclusions and from each step of the process.

The formulation and sensory evaluation revealed that the classic emulsions showed promising qualities and are well suited to continue developing into a finished product. The pickering emulsions were less successful and need more work before being finished.

Laser diffraction and microscopy showed destabilization in the form of flocculation for all pickering emulsions, while classic emulsions obtained their stability.

Microscopy of the hair showed some promising results but must be used more methodically before any conclusions can be drawn.

The compression test showed no significant effect for the hair treated with hair mask, neither for the bleached hair nor the unbleached hair.

The user survey displayed that the classic emulsions was more appreciated by the consumers than the pickering emulsions, both in the shower and after use, which could be explained by the high oil content of the pickering emulsion.

Further, it can be concluded that different methods can be used for different purposes when analysing and evaluating hair mask emulsions, and with more time an even more complete analysis could be done. The methods used could be improved and more analysis methods could be added to the process.

Other than the suggestions for further research already mentioned, the next step of developing the hair mask would be to research other ingredient groups such as preservatives or oil types. The stability of the formulation should also be more thoroughly evaluated - over longer time periods and in other higher temperature environments.

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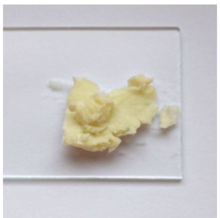
Appendix

7.1 Appendix A - Photographs of emulsions


1


1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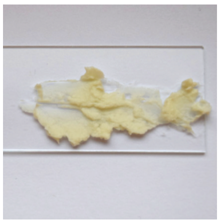
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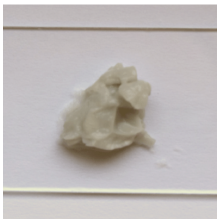
3


3


4


4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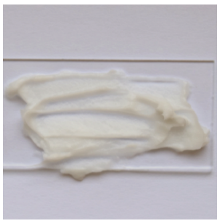
5


6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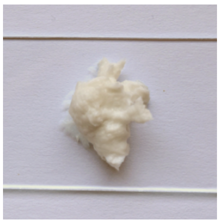
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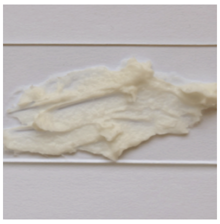
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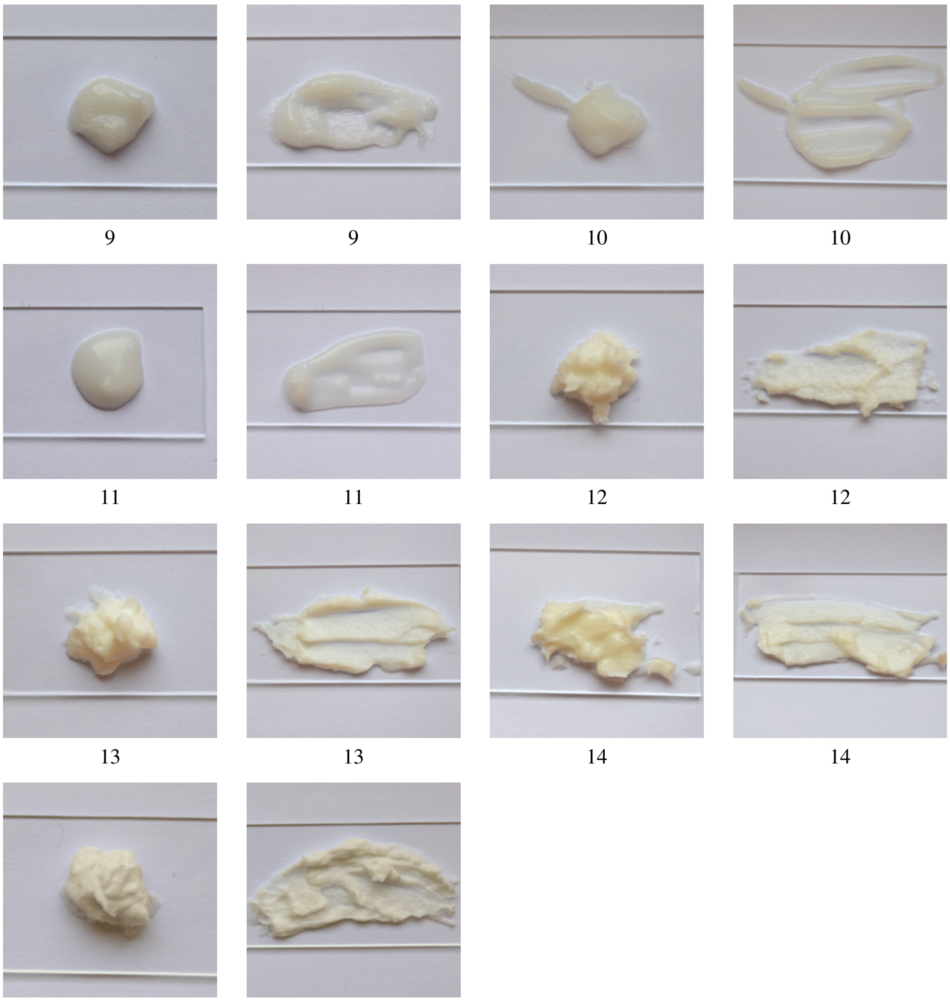
8


8



9 9 10 10

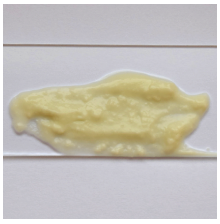
11 11 12 12

13 13 14 14

15 15

Figure 7.1: Classic formulations


P1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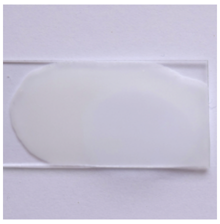
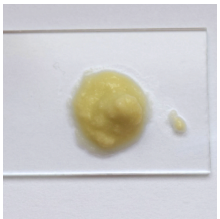

P2


P3


P3


P5


P5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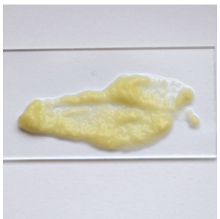
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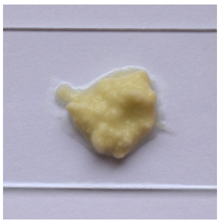
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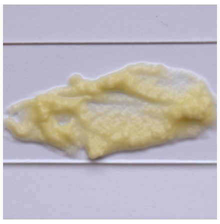
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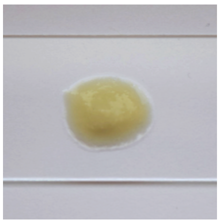
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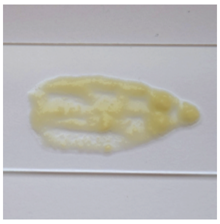

P8

P9 P9 P10 P10

P11 P11 P12 P12

P13 P13 P14 P14

P16 P16 P17 P17

P18 P18 P19 P19

P20 P20 P21 P21

Figure 7.2: Pickering formulations

7.2 Appendix B - Sensory evaluation

Table 7.1: Sensory descriptions of classical emulsions.

Number	Description
1	A little transparent, binds together well, white, shiny.
2	White, not transparent, binds together nicely.
3	Hard, stiff, grey, crumbs.
4	Hard, compact, grainy, dirtwhite, matte.
5	White, a little transparent and shiny, no graininess.
6	Binds together well, white, shiny, not grainy.
7	Grainy, white, thick.
8	Dirtwhite, quite thick, binds together well, not so shiny.
9	Semi-transparent, shiny, gel-like, binds together well, airy, foaming.
10	Semi-transparent, gel-like, quite loose.
11	Semi-transparent, loose, somewhat gel-like, shiny.
12	White, some graininess, a little transparent and shiny.
13	White, quite shiny, not transparent, no graininess.
14	White, shiny, not transparent, not grainy.

Table 7.2: Sensory evaluation of emulsion nr. 1.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	0
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	1

Table 7.3: Sensory evaluation of emulsion nr. 2.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	0

Table 7.4: Sensory evaluation of emulsion nr. 3.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	-1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	1
Light	Thin, airy, eviscerated	-1

Table 7.5: Sensory evaluation of emulsion nr. 4.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	0
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	1
Light	Thin, airy, eviscerated	-1

Table 7.6: Sensory evaluation of emulsion nr. 5.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.7: Sensory evaluation of emulsion nr. 6.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	0

Table 7.8: Sensory evaluation of emulsion nr. 7.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	0
Light	Thin, airy, eviscerated	-1

Table 7.9: Sensory evaluation of emulsion nr. 8.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.10: Sensory evaluation of emulsion nr. 9.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	0

Table 7.11: Sensory evaluation of emulsion nr. 10.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	1

Table 7.12: Sensory evaluation of emulsion nr. 11.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	0

Table 7.13: Sensory evaluation of emulsion nr. 12.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.14: Sensory evaluation of emulsion nr. 13.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.15: Sensory evaluation of emulsion nr. 14.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.16: Sensory descriptions of pickering emulsions.

Number	Description
P1	Light green/yellow colour, no graininess, not transparent, shiny
P2	Light yellow, completely liquid.
P3	Light grey, quite grainy.
P5	Grey, quite loose, light, little graininess, not so shiny.
P6	Very grainy, has separated, light green
P7	Grainy, light green, light.
P8	Loose, has separated, grainy, green/grey.
P9	Has separated, light yellow, loose, some air bubbles.
P10	Yellow, shiny, quite transparent, no graininess.
P11	Yellow, quite loose but bind together, gel-like, feels light, shiny.
P12	Grey, shiny, loose formulation with some adhering abilities.
P13	Light green, gel-like cream, binds together well, not compact, not grainy, quite shiny.
P14	Dark grey/green, slimy, does not bind together, shiny, not grainy.
P16	Quite loose, green-ish, airy.
P17	Binds together well, light yellow, shiny but too loose.
P18	Light-green, black dots, gel-like, binds together well, shiny.
P19	Grey, slimy, a bit loose, shiny.
P20	Light yellow, slimy, quite compact, black dots, some air bubbles, not grainy.
P21	Light green/grey, not grainy, a lot of air bubbles, quite loose.

Table 7.17: Sensory evaluation of emulsion nr. P1.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	-1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	-1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	1

Table 7.18: Sensory evaluation of emulsion nr. P2.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	-
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	-
Dry	Hard, tight, stiff, course, dull	-
Light	Thin, airy, eviscerated	-

Table 7.19: Sensory evaluation of emulsion nr. P3.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	1

Table 7.20: Sensory evaluation of emulsion nr. P5.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.21: Sensory evaluation of emulsion nr. P6.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	-1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.22: Sensory evaluation of emulsion nr. P7.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	1
Light	Thin, airy, eviscerated	1

Table 7.23: Sensory evaluation of emulsion nr. P8.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	-
Smooth	Slippy, flowing	-
Cohesiveness	Adhering, coated, compact	-
Dry	Hard, tight, stiff, course, dull	-
Light	Thin, airy, eviscerated	-

Table 7.24: Sensory evaluation of emulsion nr. P9.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	-1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.25: Sensory evaluation of emulsion nr. P10.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	0
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	-1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	0

Table 7.26: Sensory evaluation of emulsion nr. P11.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	0
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.27: Sensory evaluation of emulsion nr. P12.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	-1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.28: Sensory evaluation of emulsion nr. P13.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.29: Sensory evaluation of emulsion nr. P14.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	-1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.30: Sensory evaluation of emulsion nr. P16.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.31: Sensory evaluation of emulsion nr. P17.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	0
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.32: Sensory evaluation of emulsion nr. P18.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	0
Cohesiveness	Adhering, coated, compact	1
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.33: Sensory evaluation of emulsion nr. P19.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	0
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.34: Sensory evaluation of emulsion nr. P20.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	0
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

Table 7.35: Sensory evaluation of emulsion nr. P21.

Major expression	Further description	Points (-1, 0, 1)
Moisturizing	Oily, heavy, moist	1
Smooth	Slippy, flowing	1
Cohesiveness	Adhering, coated, compact	0
Dry	Hard, tight, stiff, course, dull	-1
Light	Thin, airy, eviscerated	-1

7.3 Appendix C - Microscopy images

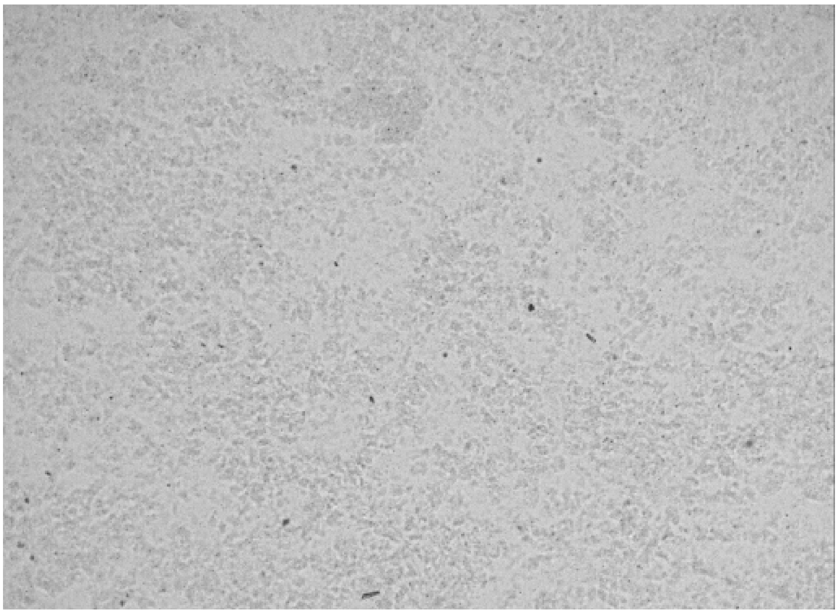


Figure 7.3: C1 (24h). Magnification x5.

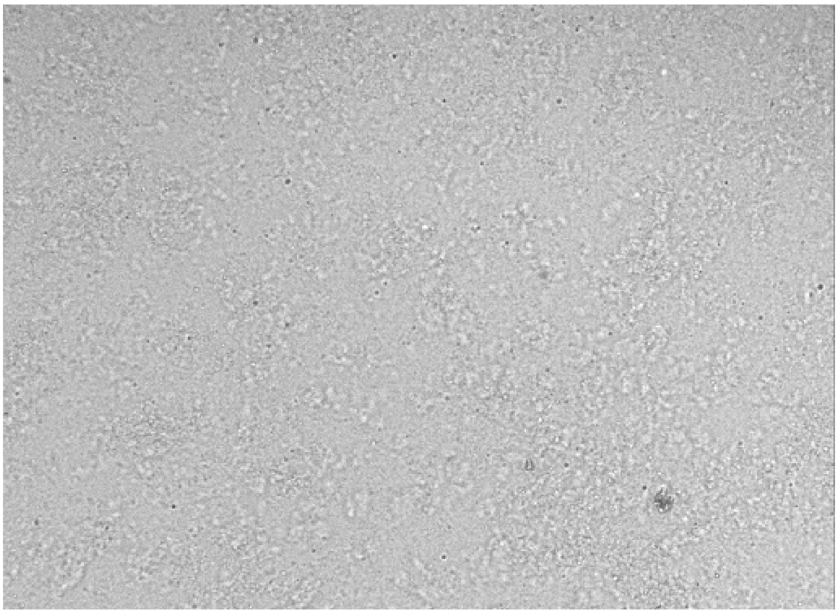


Figure 7.4: C1 (24h). Magnification x10.

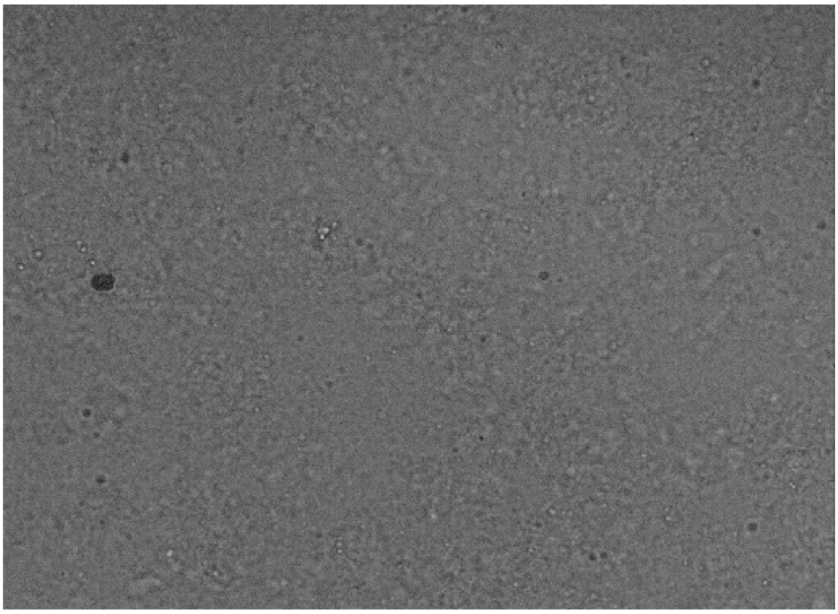


Figure 7.5: C1 (24h). Magnification x20. Picture 1.

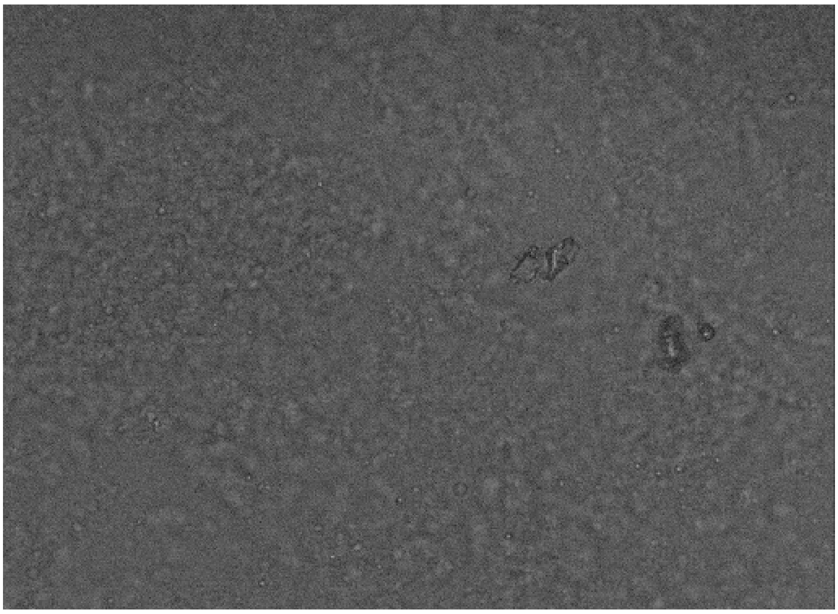


Figure 7.6: C1 (24h). Magnification x20. Picture 2.

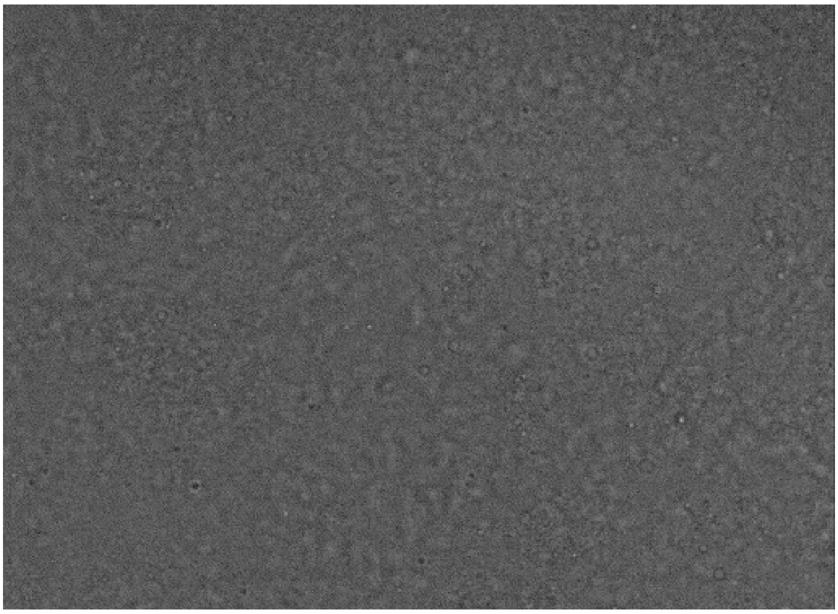


Figure 7.7: C1 (24h). Magnification x20. Picture 3.

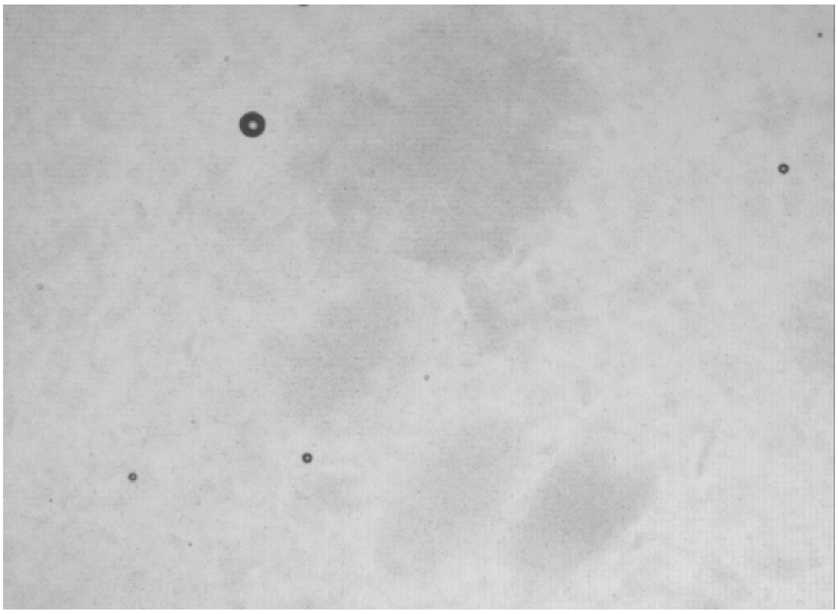


Figure 7.8: C1 (5 weeks). Magnification x5.

Figure 7.9: C1 (5 weeks). Magnification x10.

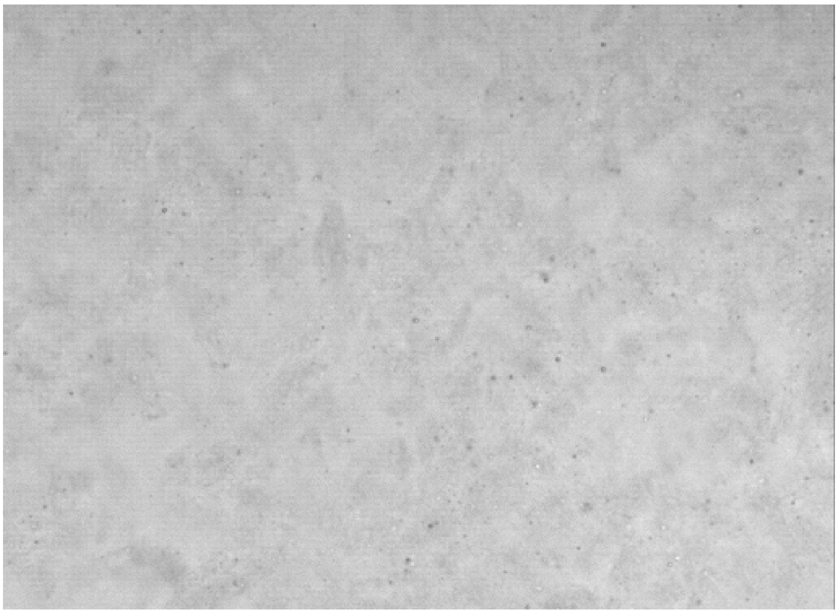


Figure 7.10: C1 (5 weeks). Magnification x20. Picture 1.

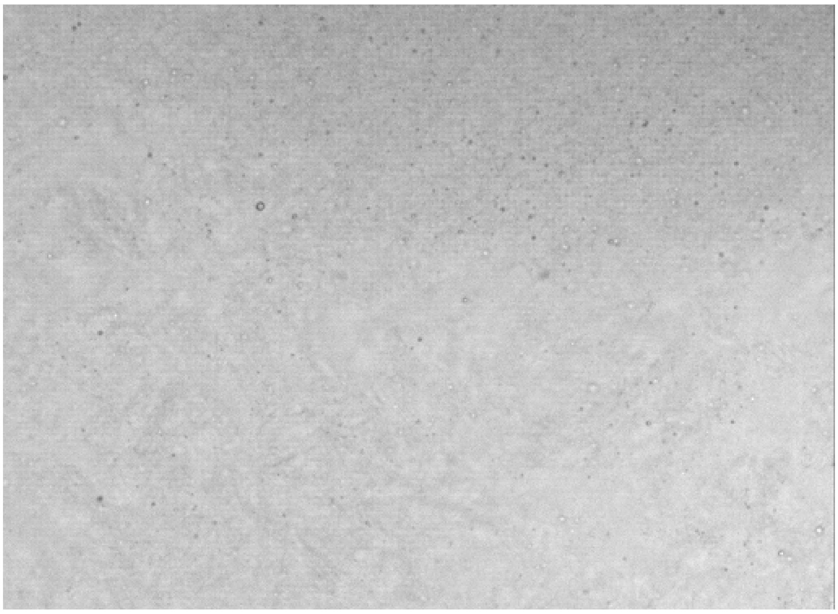


Figure 7.11: C1 (5 weeks). Magnification x20. Picture 2.

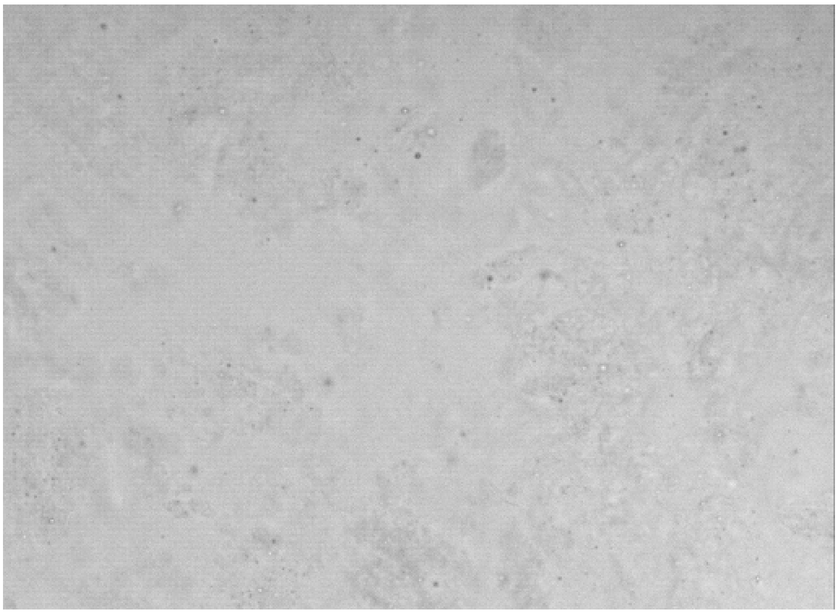


Figure 7.12: C1 (5 weeks). Magnification x20. Picture 3.

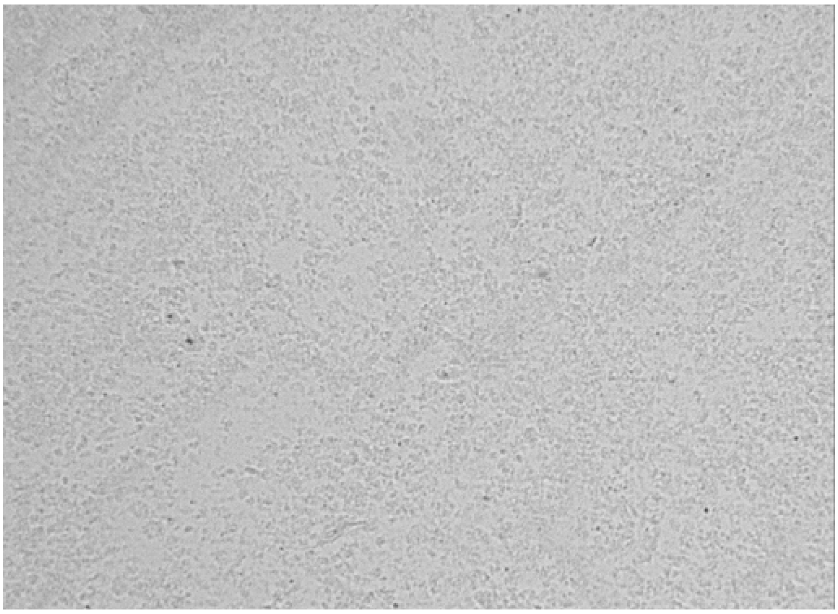


Figure 7.13: C12 (24h). Magnification x5.

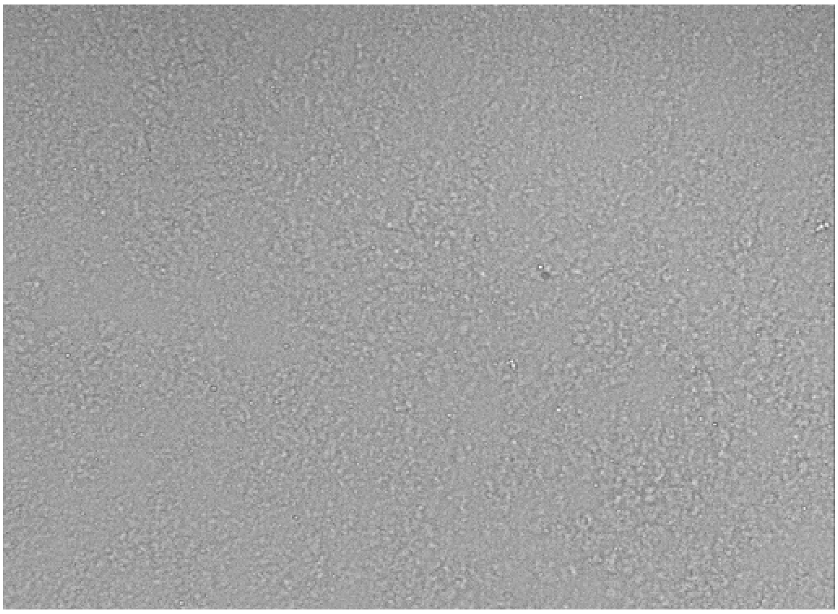


Figure 7.14: C12 (24h). Magnification x10.

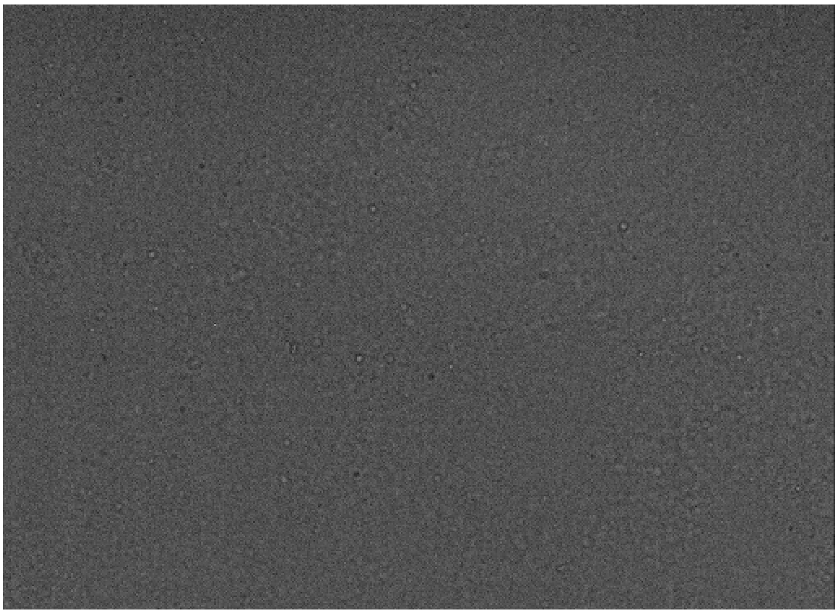


Figure 7.15: C12 (24h). Magnification x20. Picture 1.

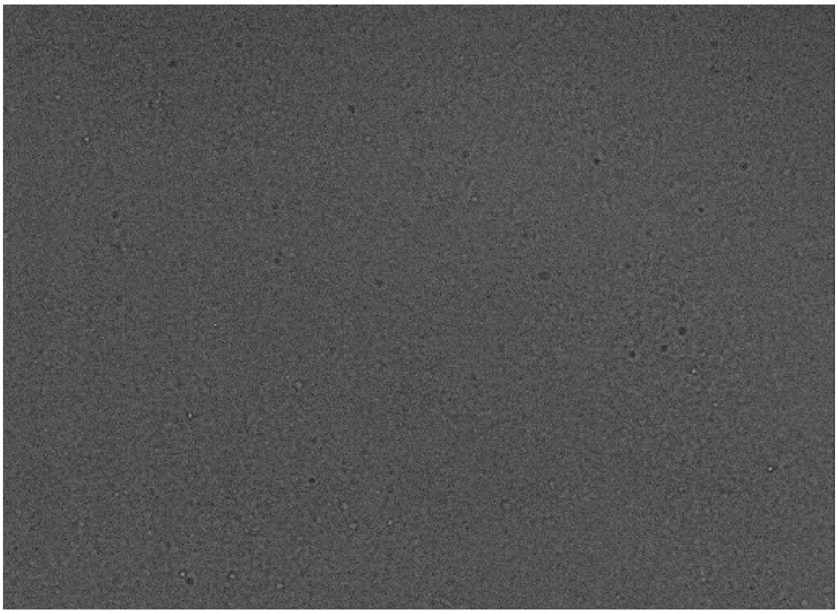


Figure 7.16: C12 (24h). Magnification x20. Picture 2.

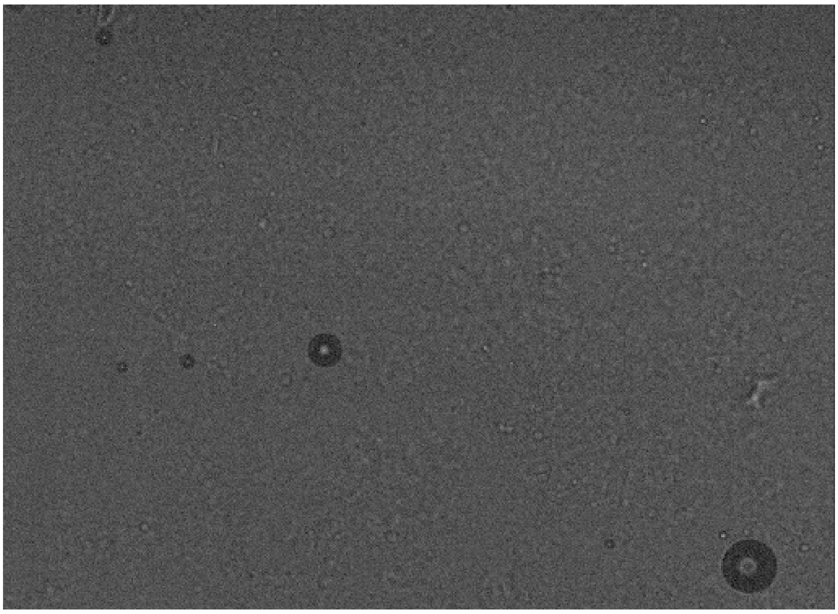


Figure 7.17: C12 (24h). Magnification x20. Picture 3.

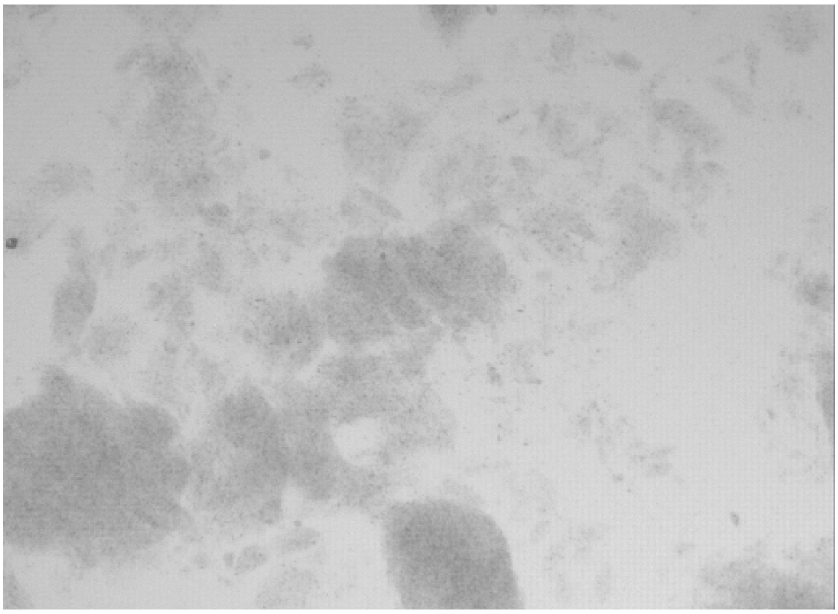


Figure 7.18: C12 (5 weeks). Magnification x5.

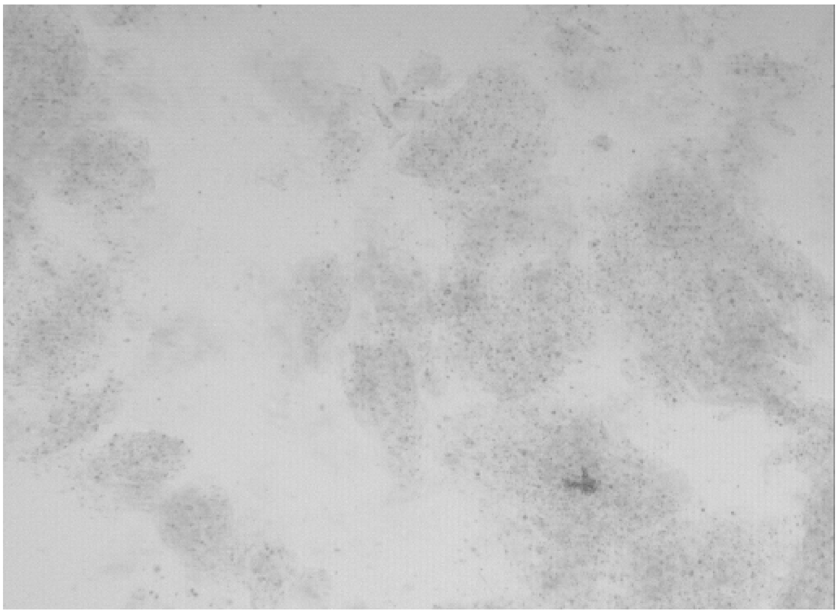


Figure 7.19: C12 (5 weeks). Magnification x10.

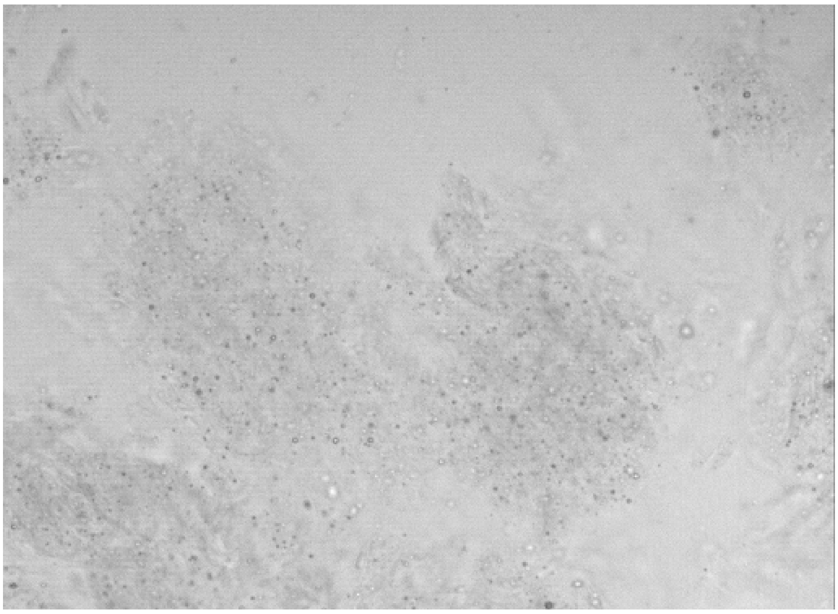


Figure 7.20: C12 (5 weeks). Magnification x20. Picture 1.

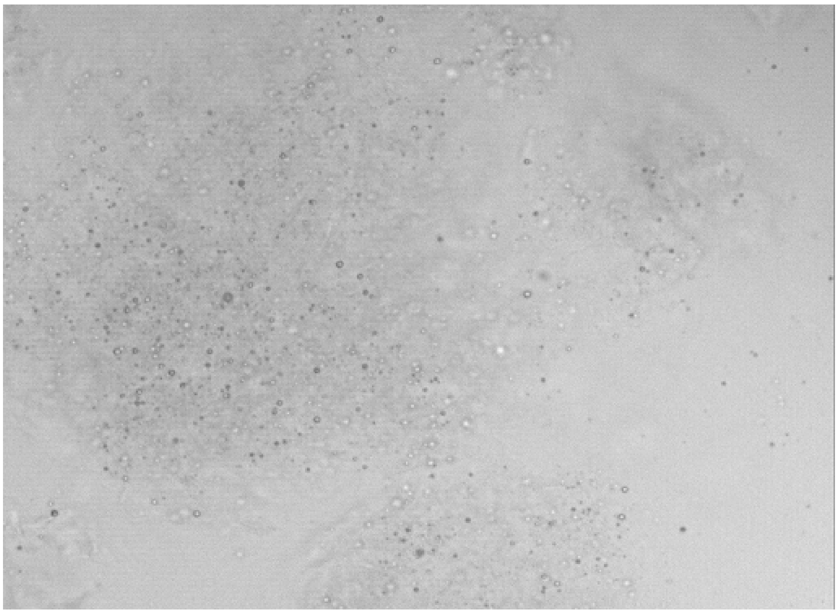


Figure 7.21: C12 (5 weeks). Magnification x20. Picture 2.

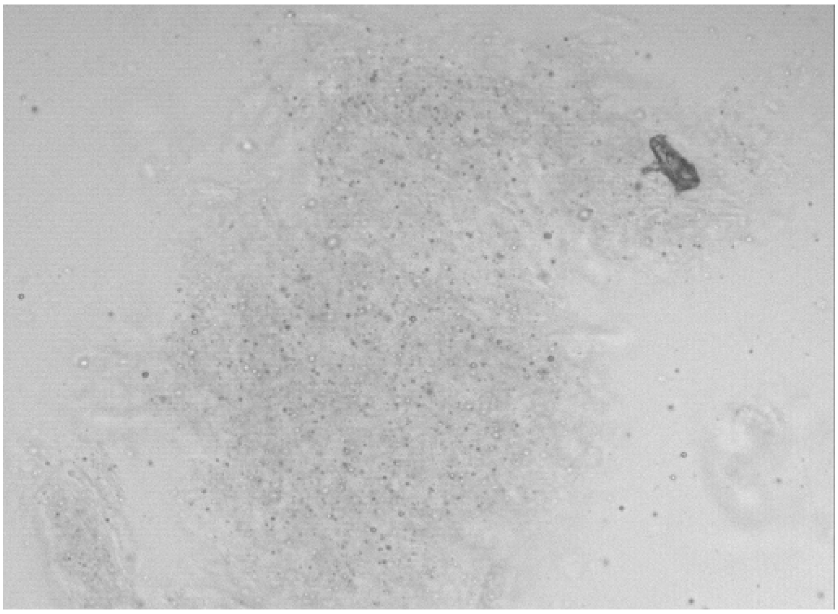


Figure 7.22: C12 (5 weeks). Magnification x20. Picture 3.

Figure 7.23: C13 (24h). Magnification x5.

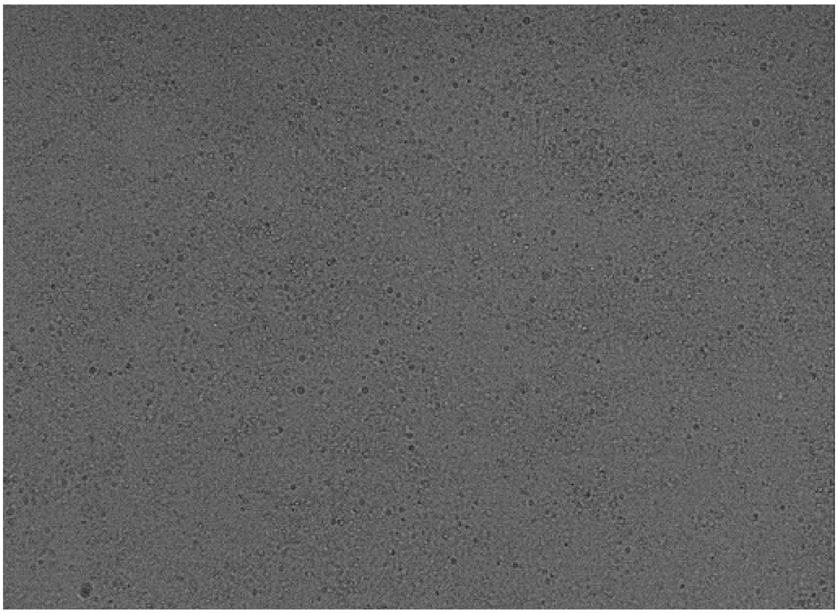


Figure 7.24: C13 (24h). Magnification x10.

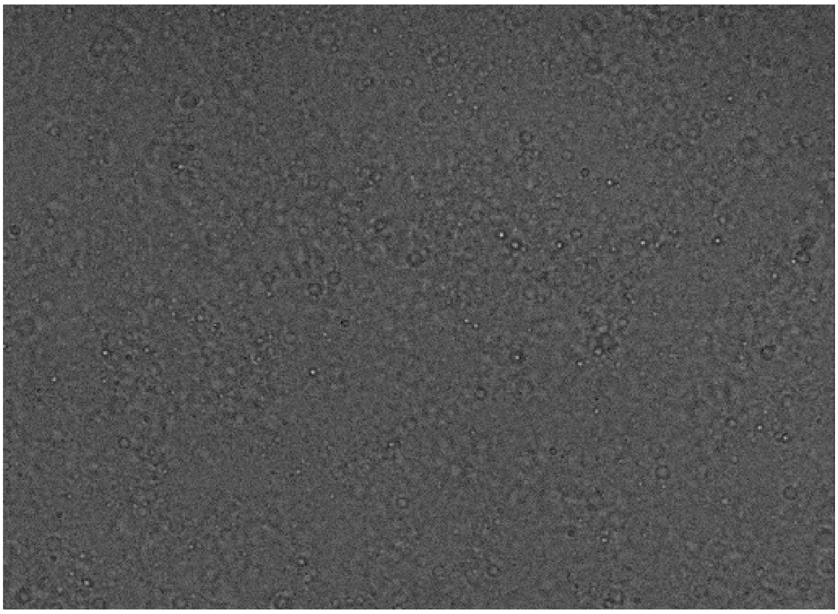


Figure 7.25: C13 (24h). Magnification x20. Picture 1.

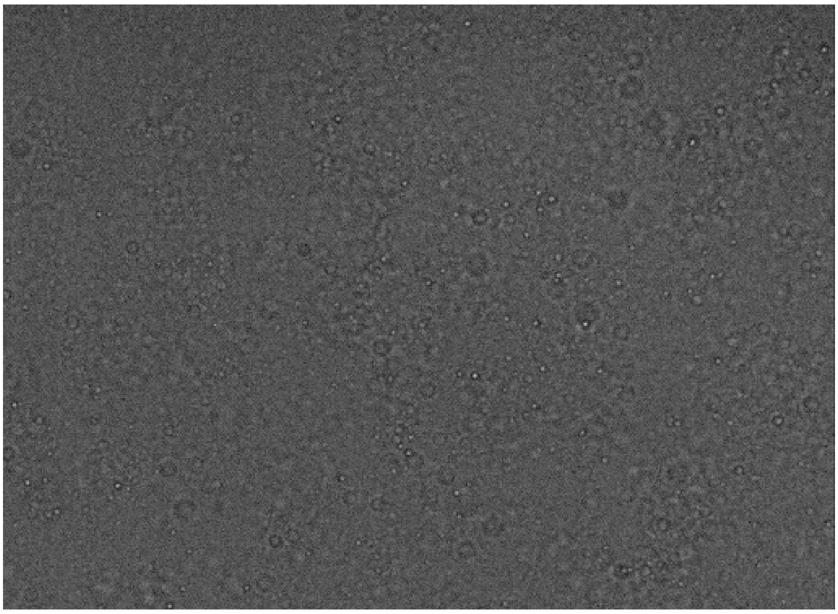


Figure 7.26: C13 (24h). Magnification x20. Picture 2.

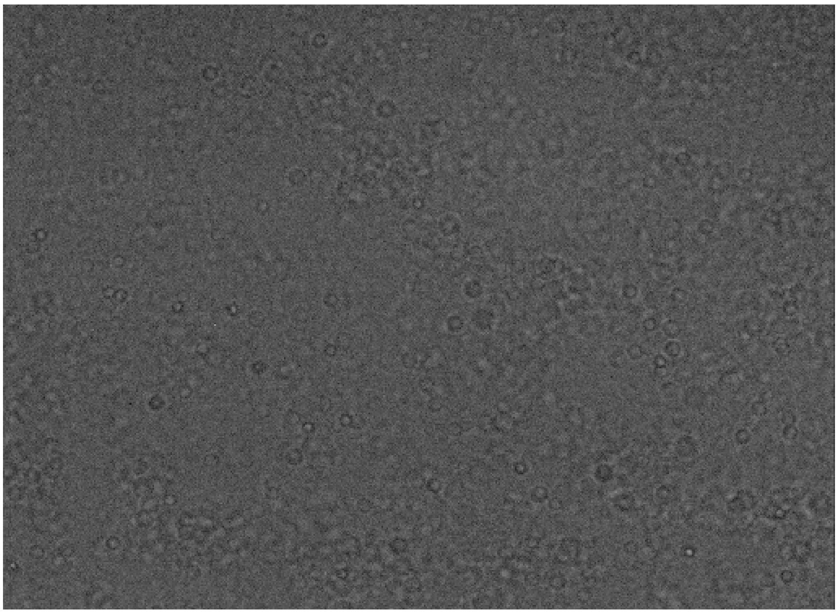


Figure 7.27: C13 (24h). Magnification x20. Picture 3.

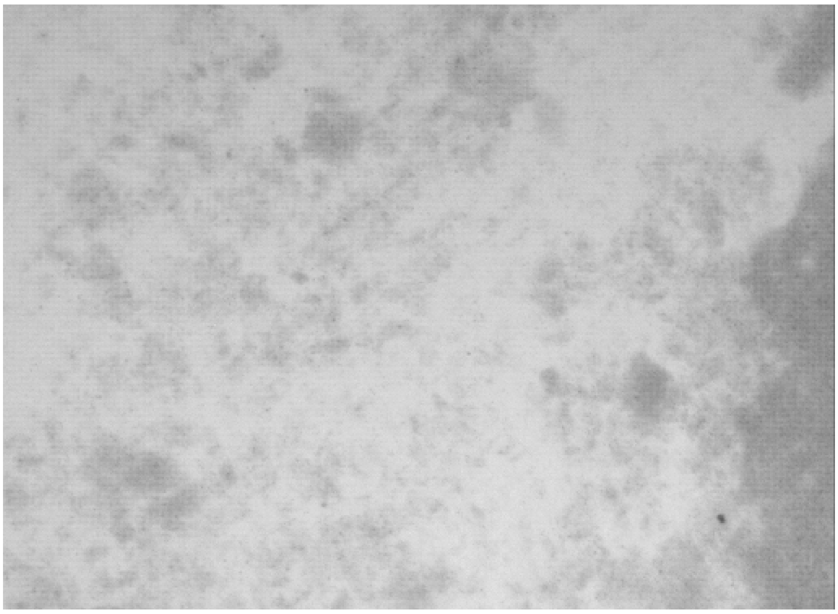


Figure 7.28: C13 (5 weeks). Magnification x5.

Figure 7.29: C13 (5 weeks). Magnification x10.

Figure 7.30: C13 (5 weeks). Magnification x20. Picture 1.

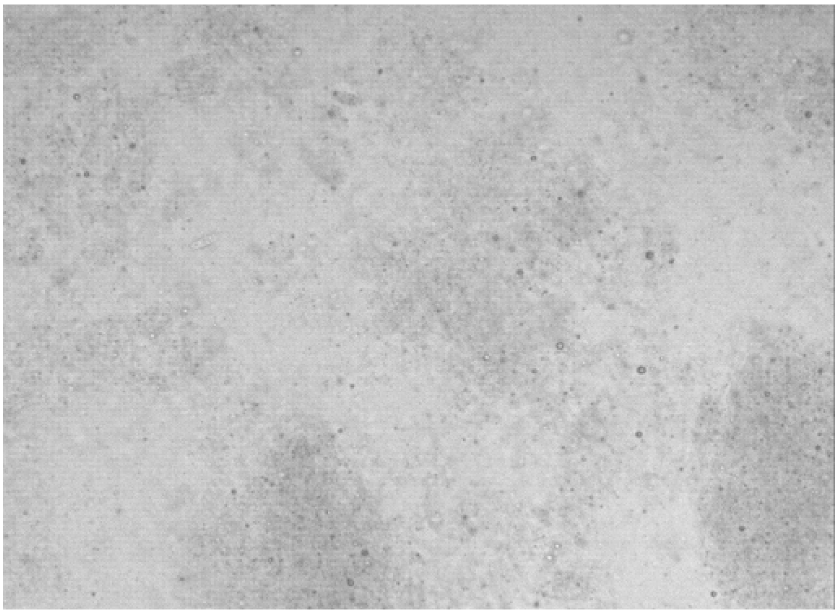


Figure 7.31: C13 (5 weeks). Magnification x20. Picture 2.

Figure 7.32: C13 (5 weeks). Magnification x20. Picture 3.

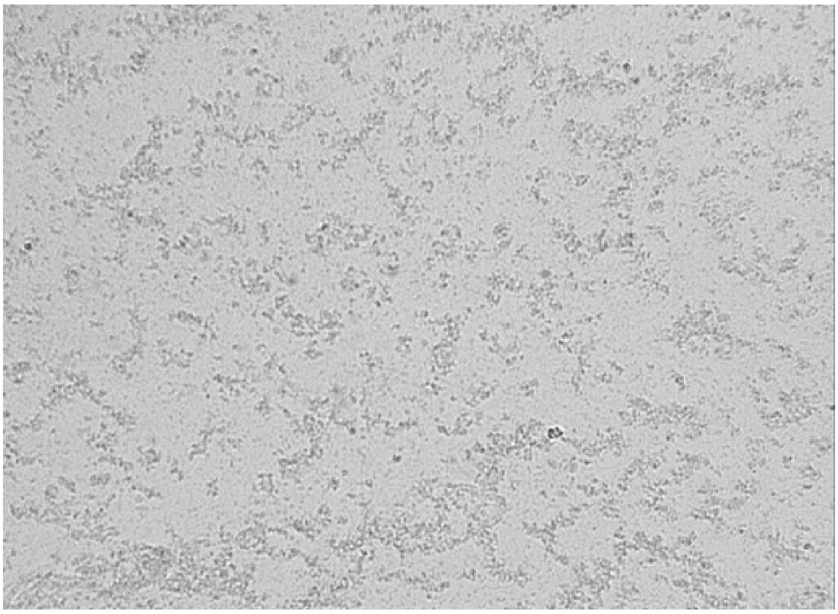


Figure 7.33: C14 (24h). Magnification x5.

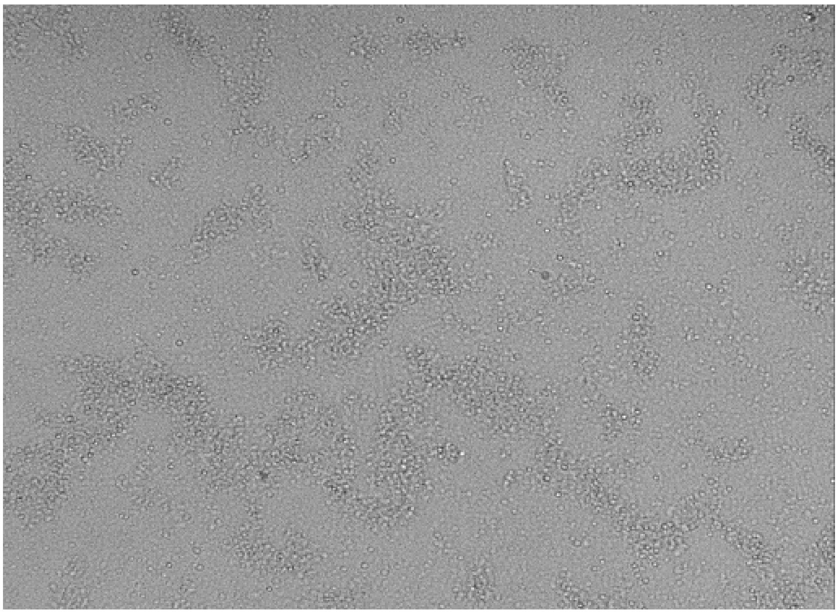


Figure 7.34: C14 (24h). Magnification x10.

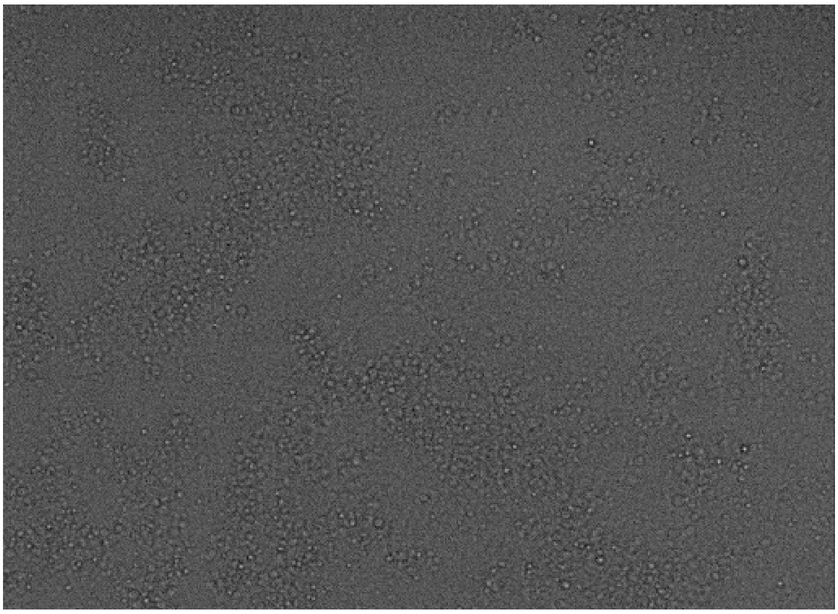


Figure 7.35: C14 (24h). Magnification x20. Picture 1.

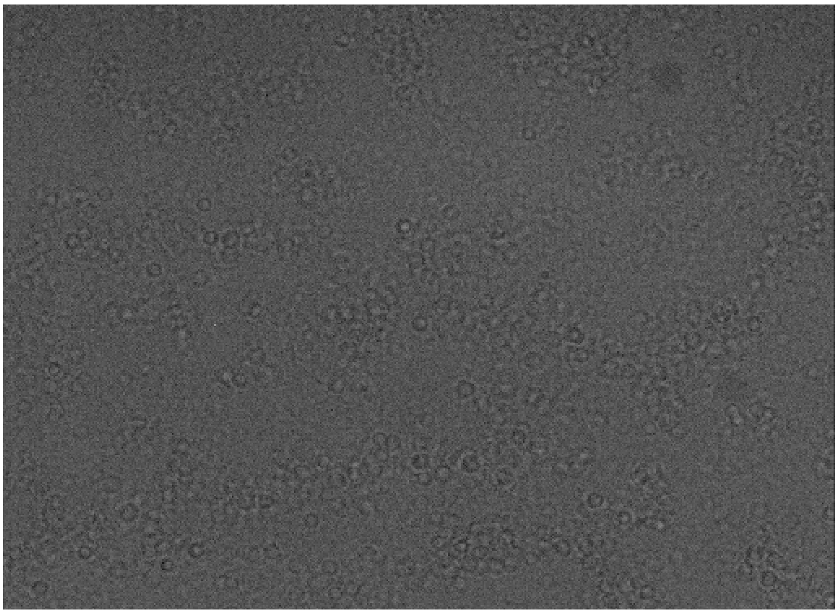


Figure 7.36: C14 (24h). Magnification x20. Picture 2.

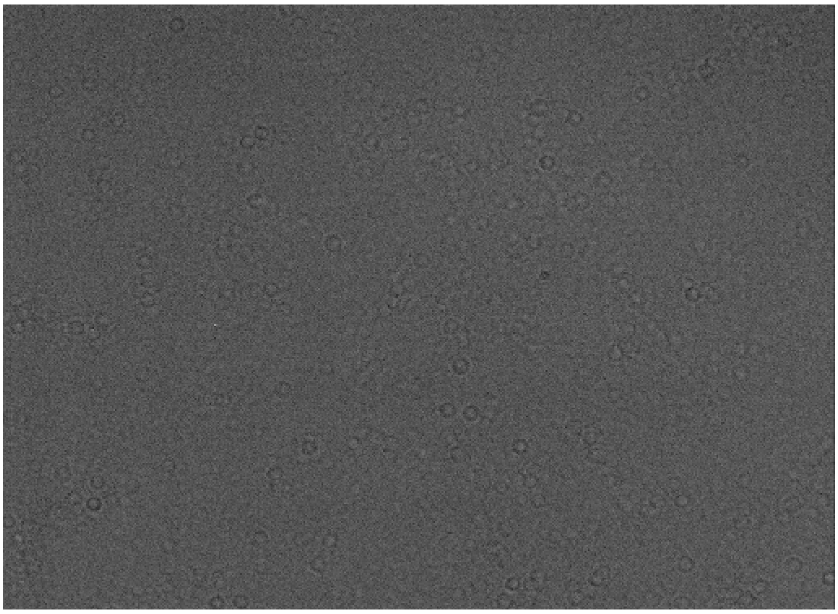


Figure 7.37: C14 (24h). Magnification x20. Picture 3.

Figure 7.38: C14 (5 weeks). Magnification x5.

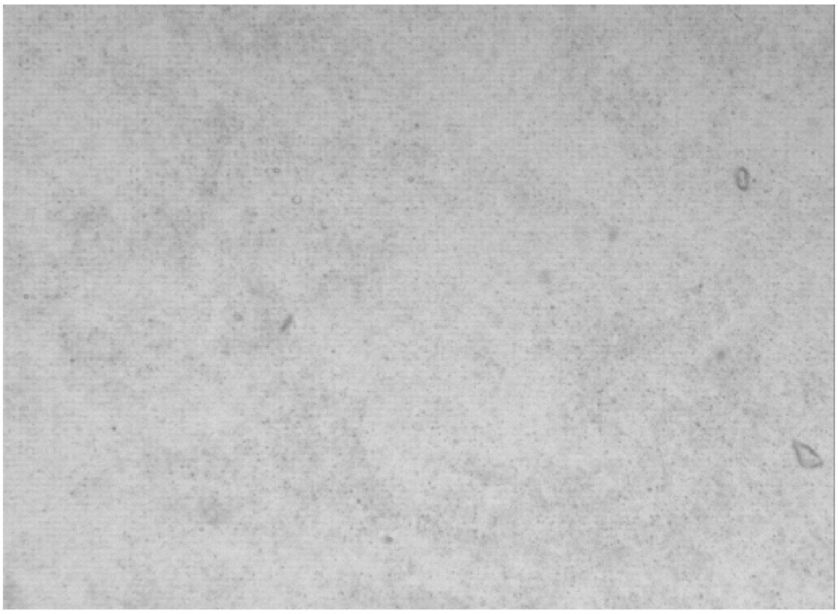


Figure 7.39: C14 (5 weeks). Magnification x10.

Figure 7.40: C14 (5 weeks). Magnification x20. Picture 1.

Figure 7.41: C14 (5 weeks). Magnification x20. Picture 2.

Figure 7.42: C14 (5 weeks). Magnification x20. Picture 3.

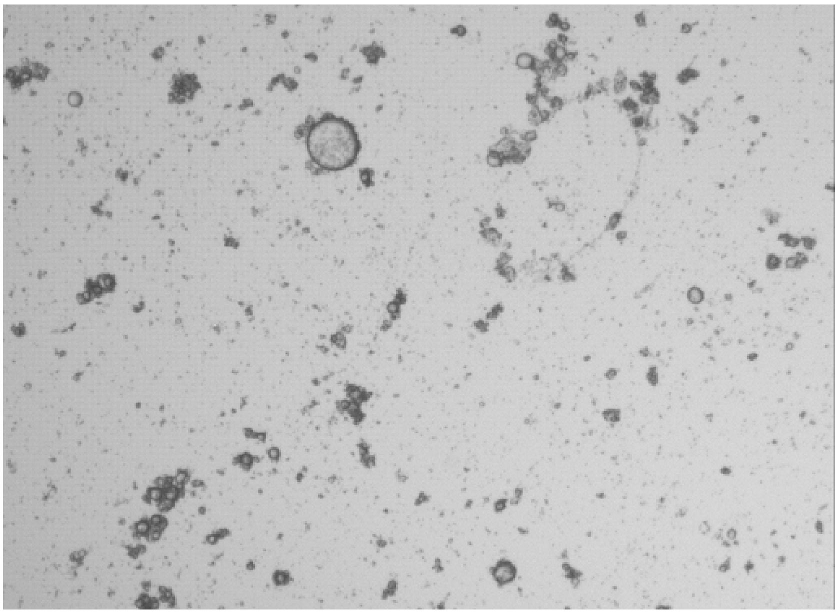


Figure 7.43: P2 (24h). Magnification x5.

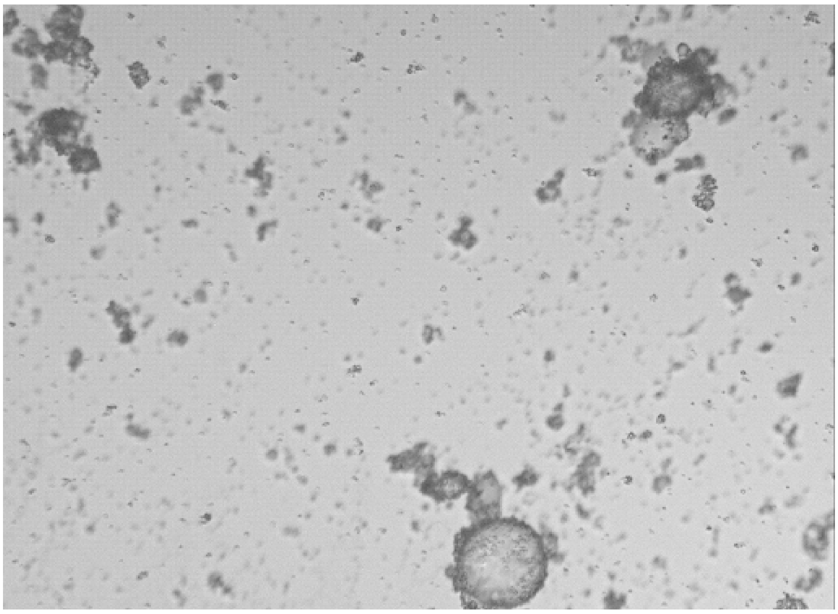


Figure 7.44: P2 (24h). Magnification x10. Picture 1.

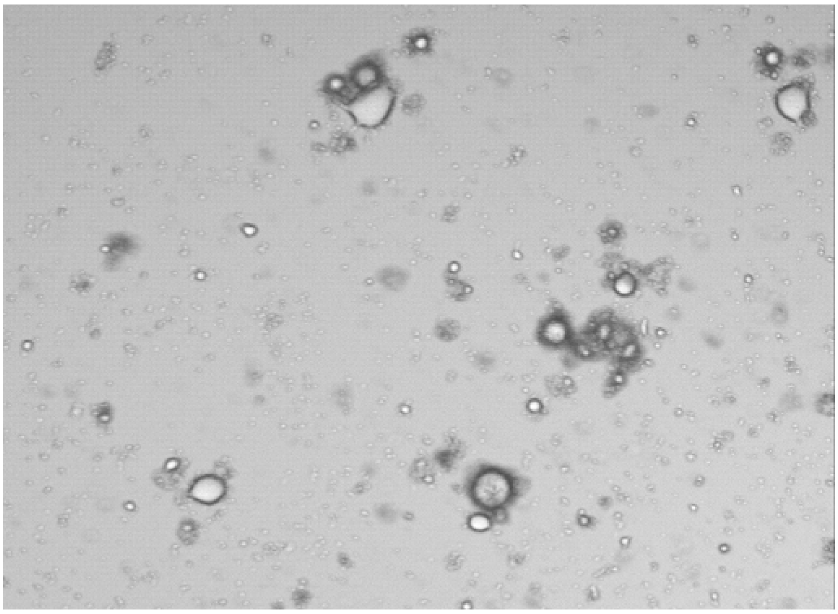


Figure 7.45: P2 (24h). Magnification x10. Picture 2

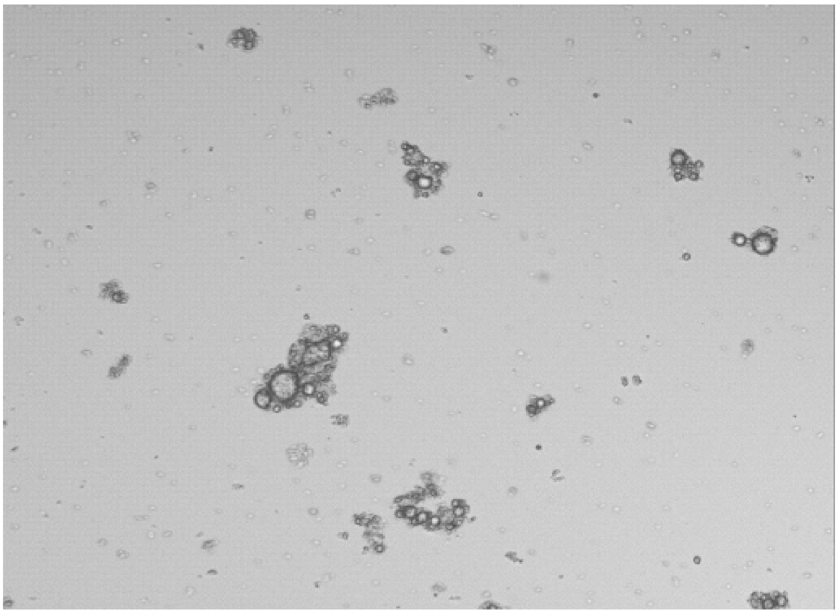


Figure 7.46: P2 (24h). Magnification x10. Picture 3.

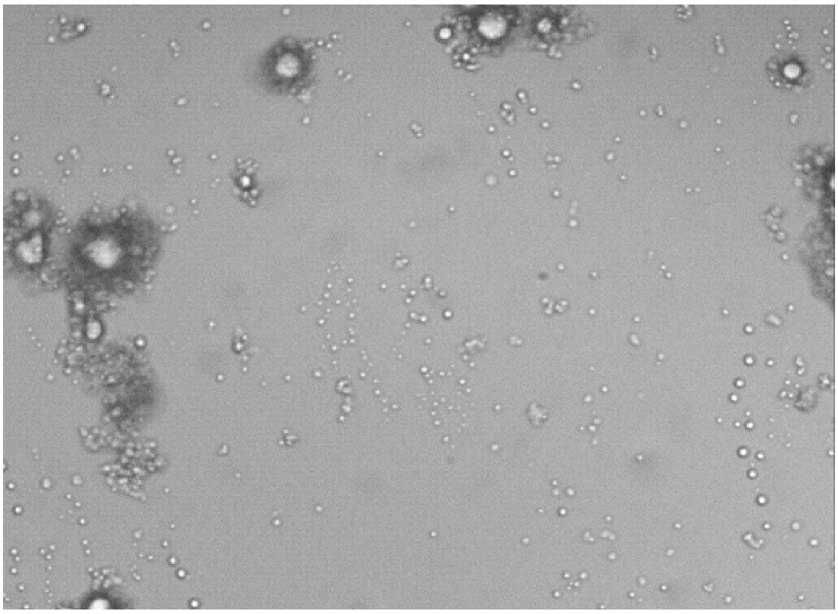


Figure 7.47: P2 (24h). Magnification x20.

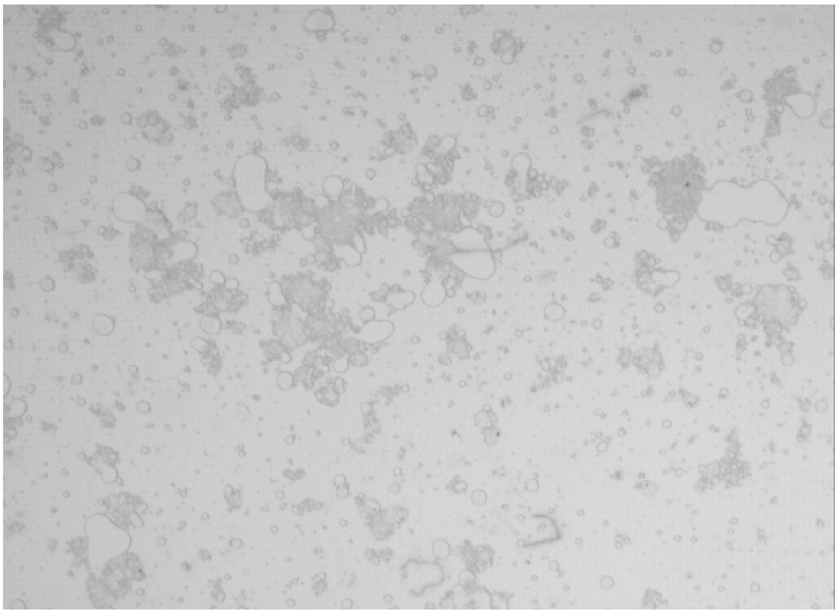


Figure 7.48: P2 (5 weeks). Magnification x5.

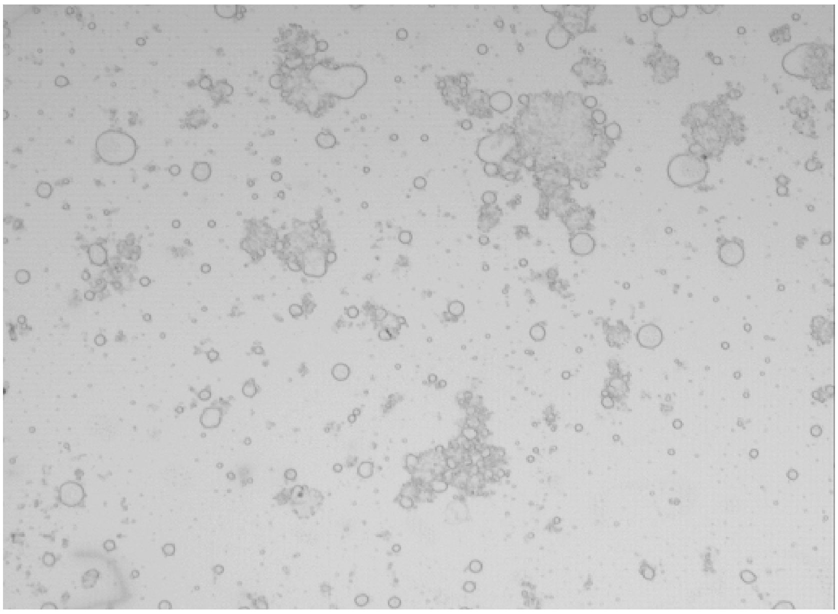


Figure 7.49: P2 (5 weeks). Magnification x10. Picture 1.

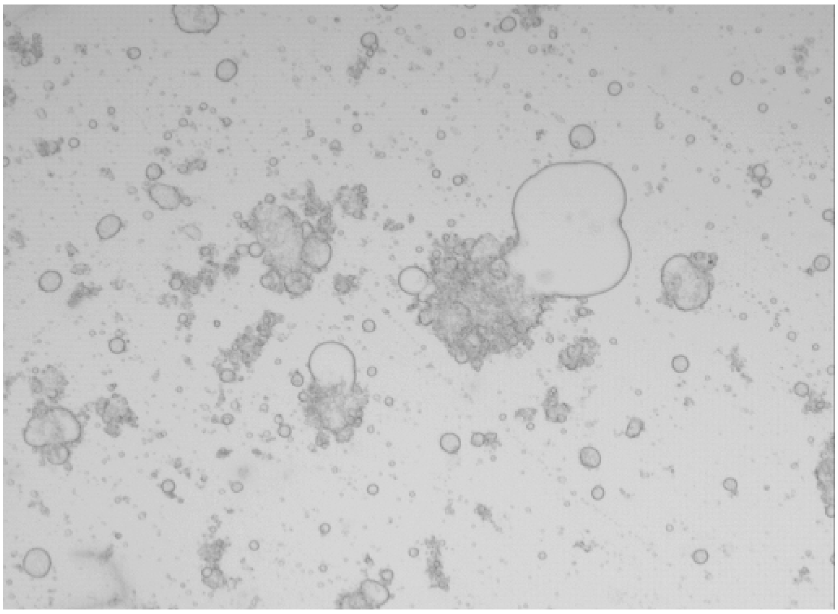


Figure 7.50: P2 (5 weeks). Magnification x10. Picture 2

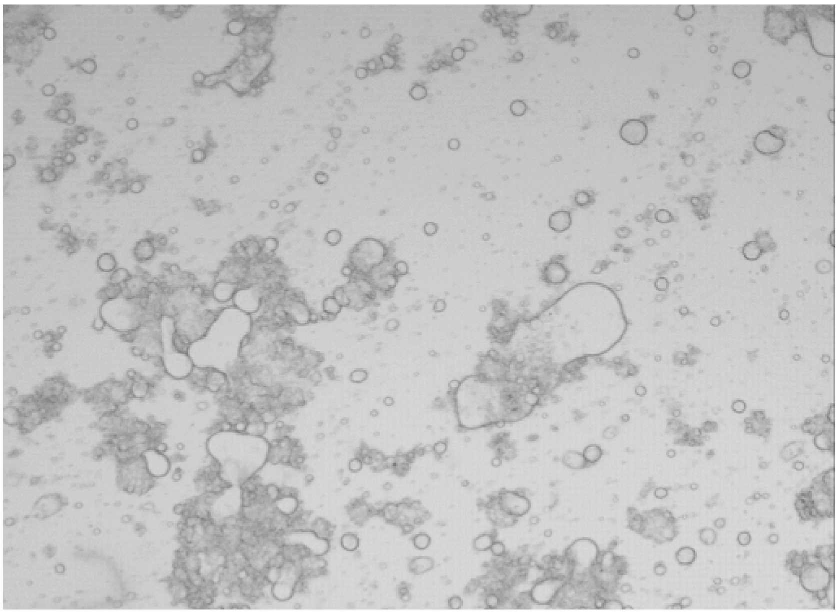


Figure 7.51: P2 (5 weeks). Magnification x10. Picture 3.

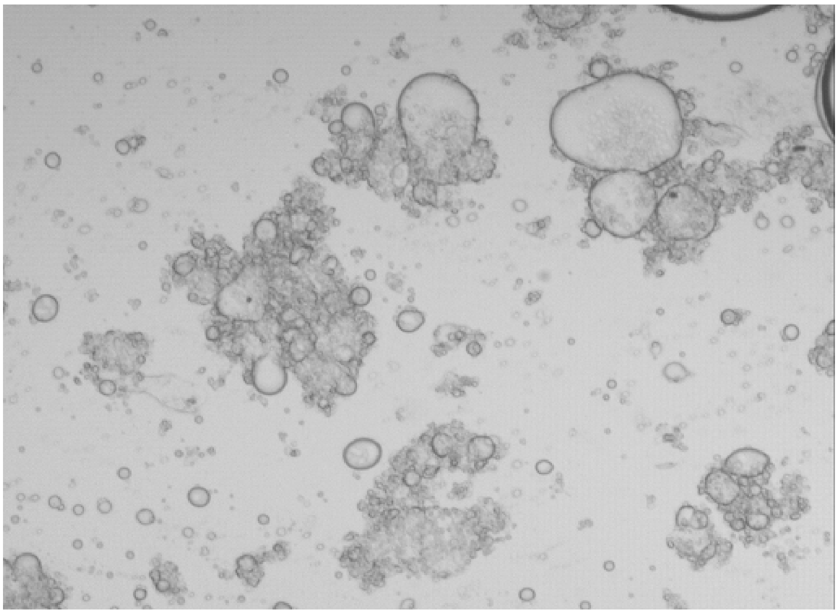


Figure 7.52: P2 (5 weeks). Magnification x20.

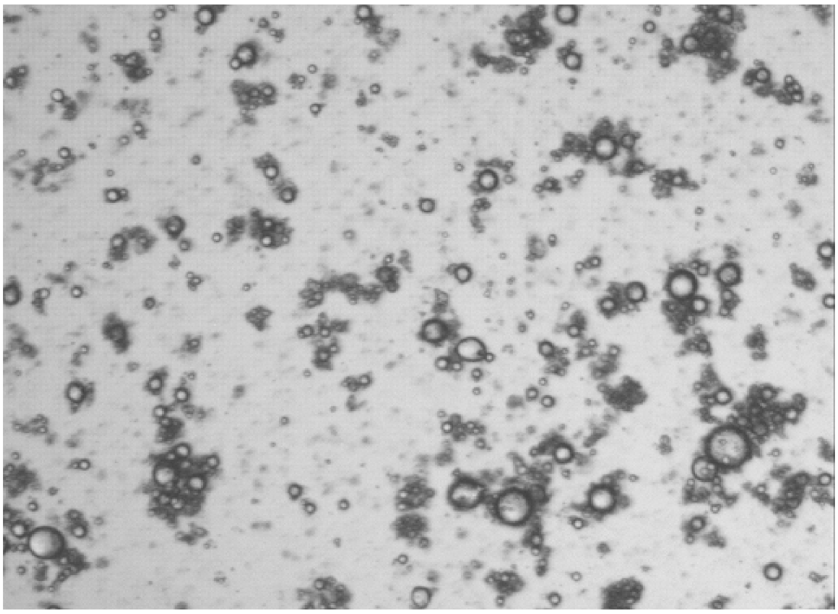


Figure 7.53: P7 (24 h). Magnification x5.

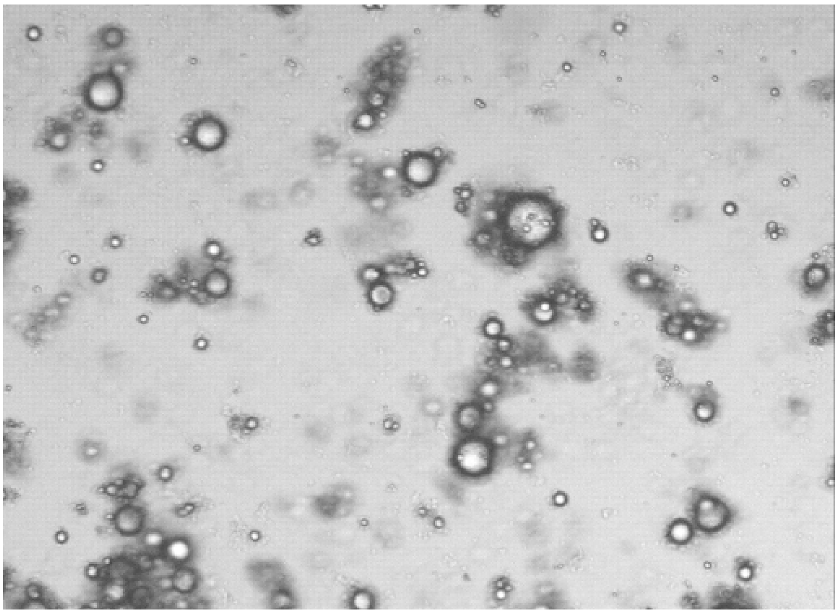


Figure 7.54: P7 (24h). Magnification x10. Picture 1.

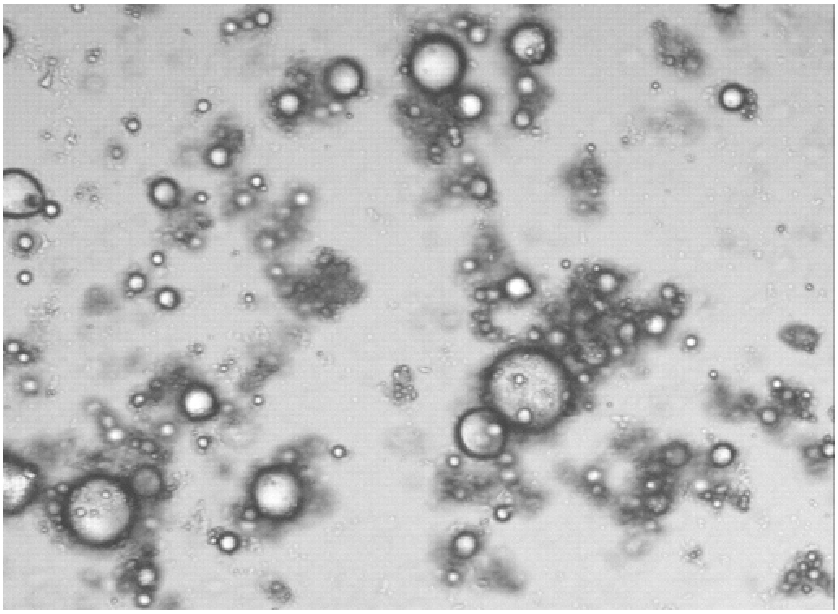


Figure 7.55: P7. Magnification x10. Picture 2.

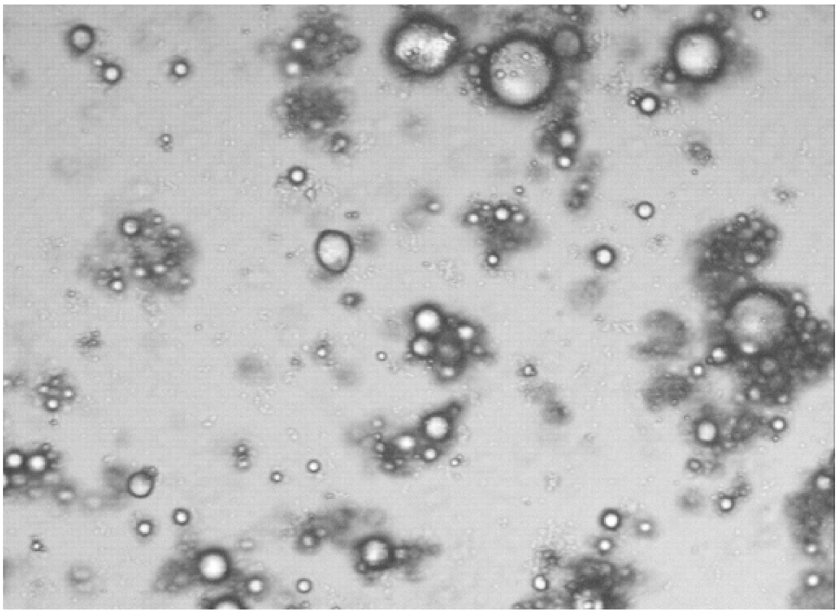


Figure 7.56: P7. Magnification x10. Picture 3.

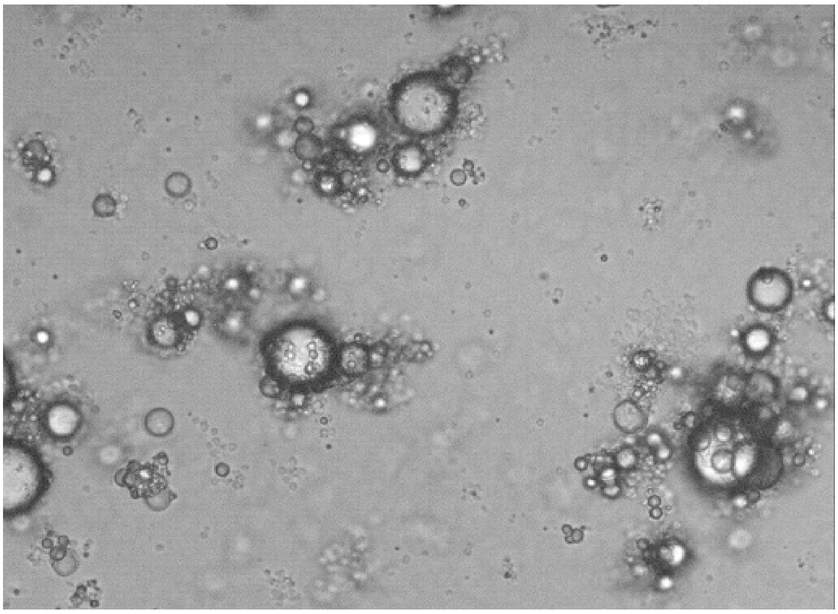


Figure 7.57: P7 (24h). Magnification x20.

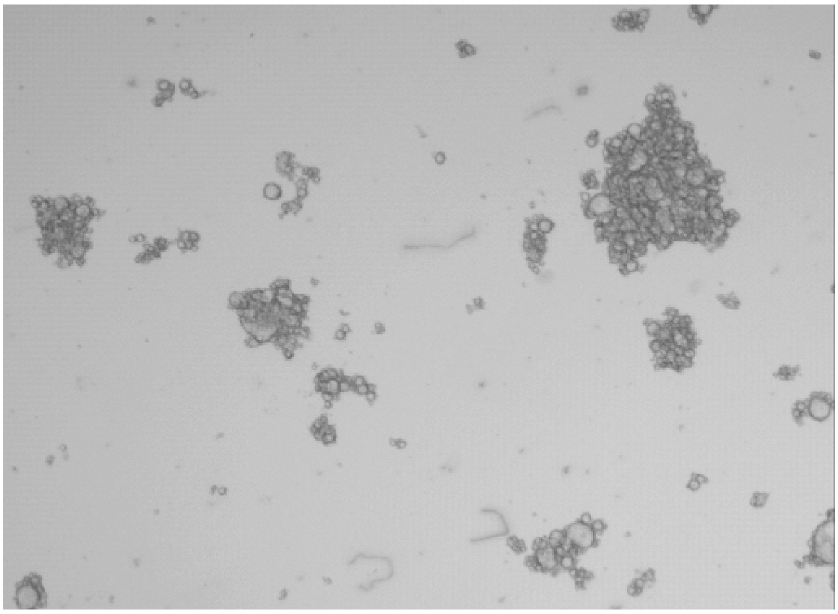


Figure 7.58: P7 (5 weeks). Magnification x5.

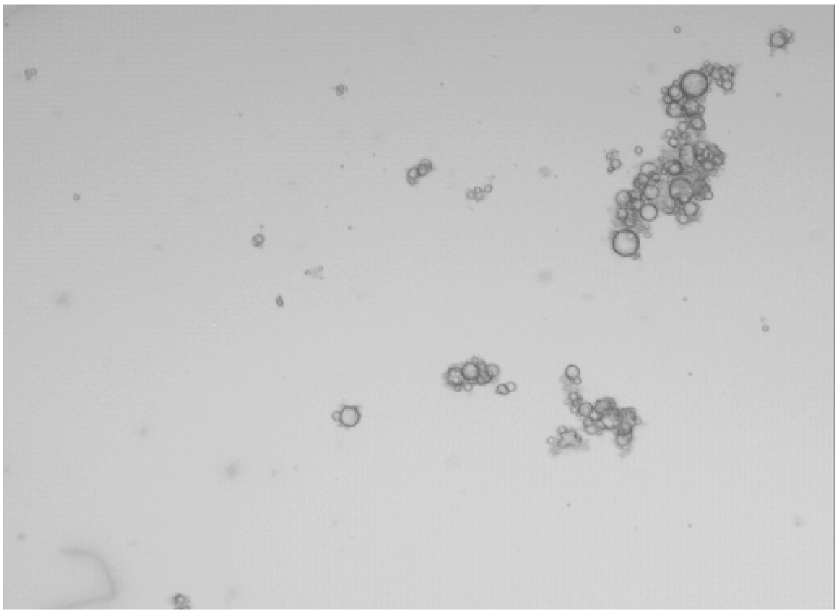


Figure 7.59: P7 (5 weeks). Magnification x10. Picture 1.

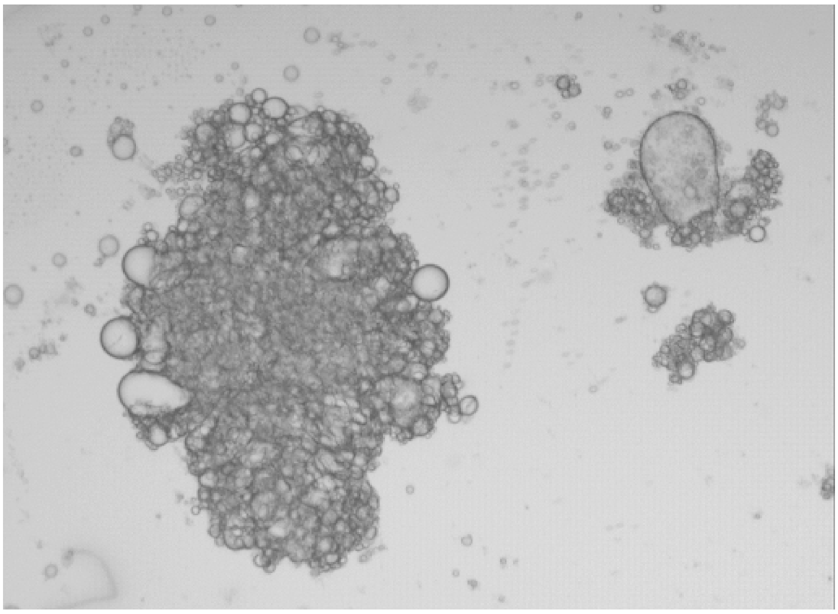


Figure 7.60: P7 (5 weeks). Magnification x10. Picture 2.

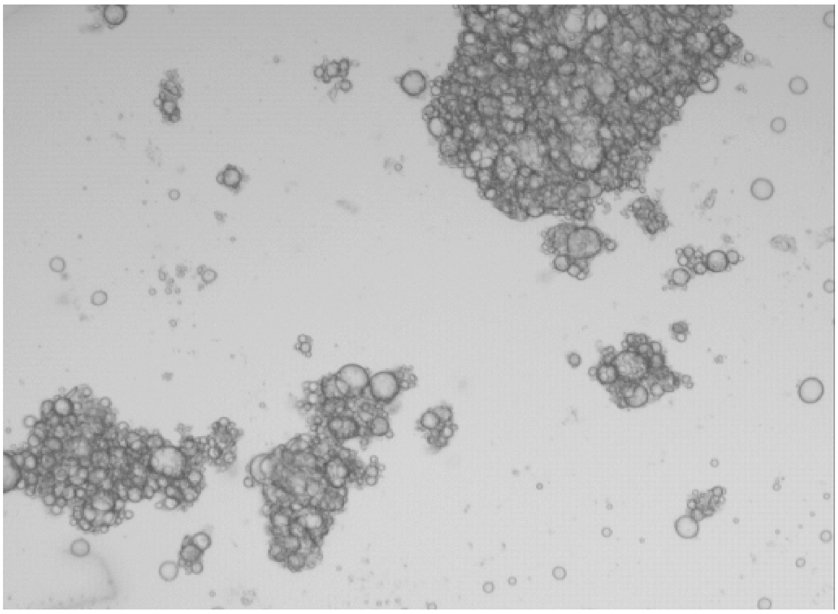


Figure 7.61: P7 (5 weeks). Magnification x10. Picture 3.

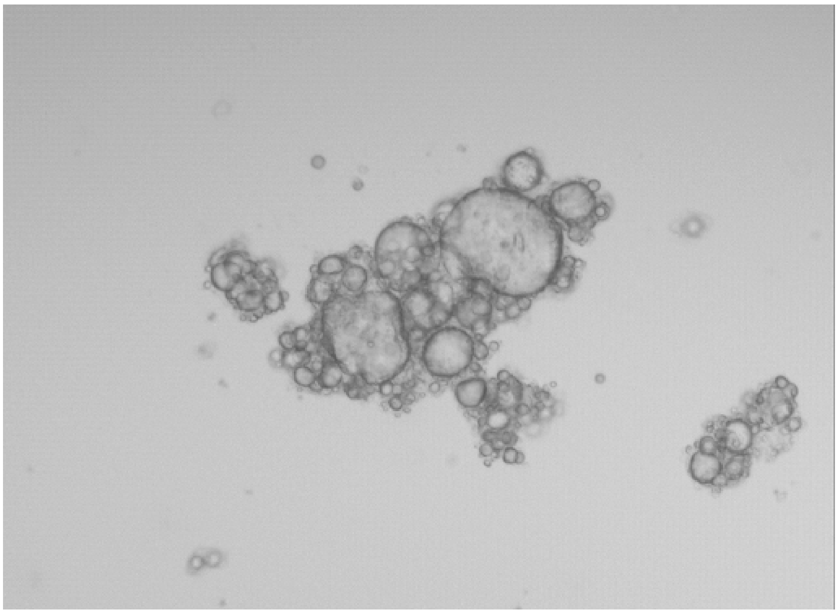


Figure 7.62: P7 (5 weeks). Magnification x20.

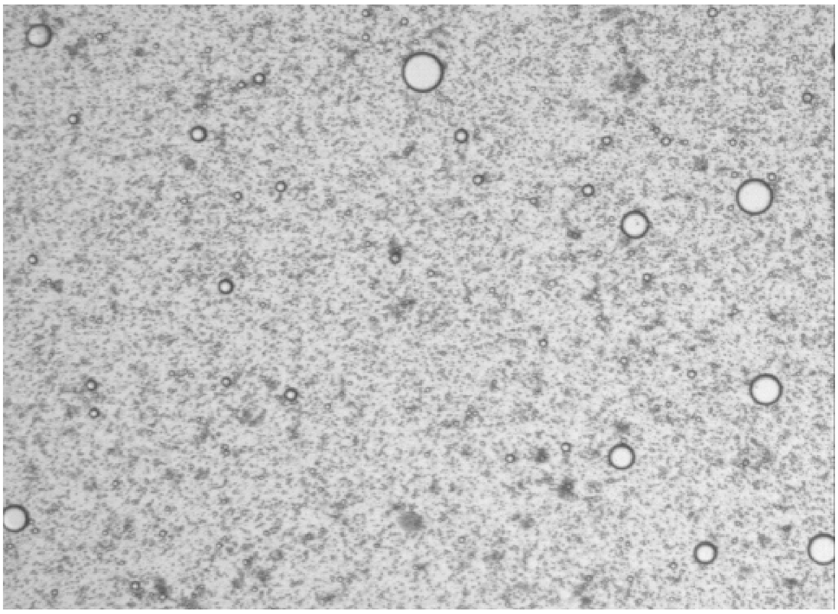


Figure 7.63: P13 (24h). Magnification x5.

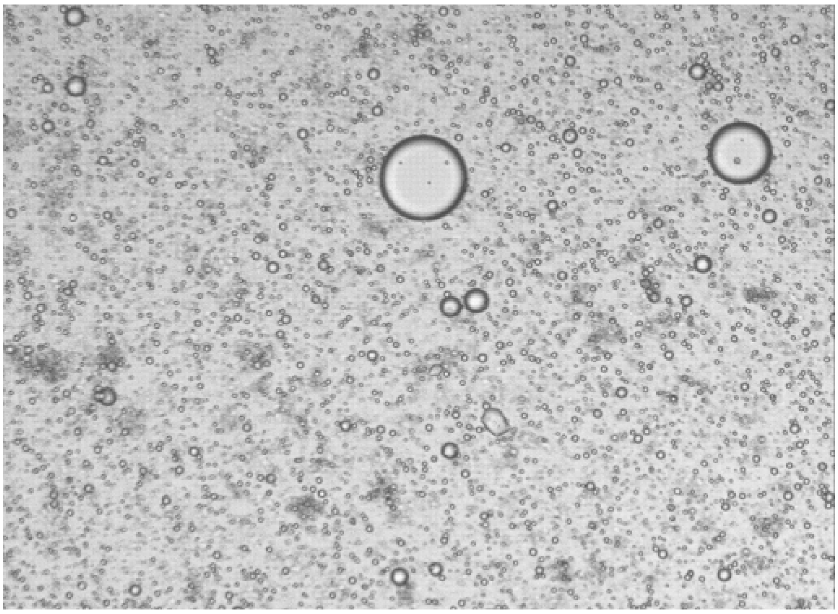


Figure 7.64: P13 (24h). Magnification x10. Picture 1.

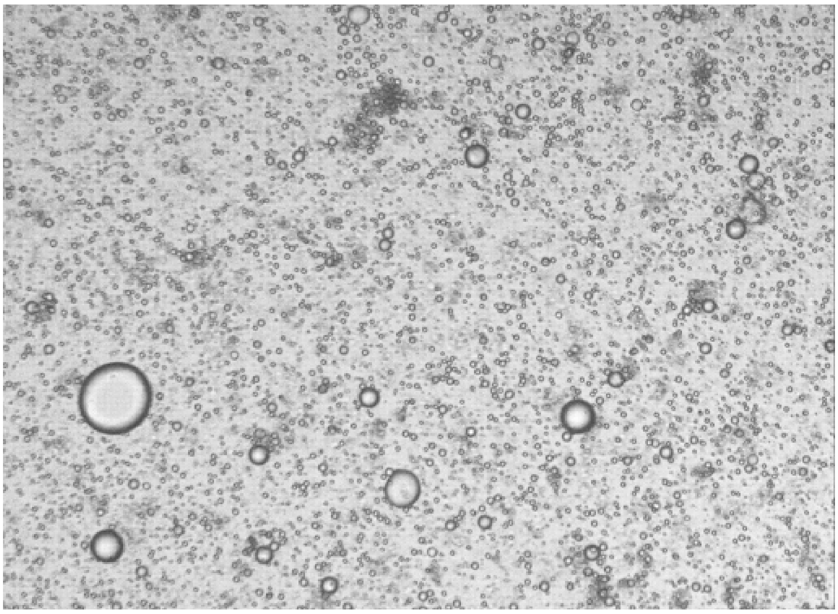


Figure 7.65: P13 (24h). Magnification x10. Picture 2.

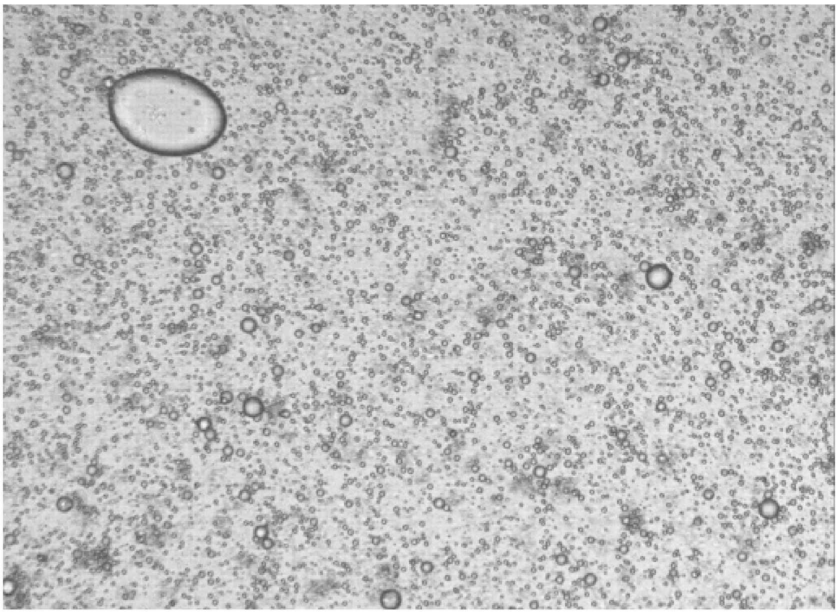


Figure 7.66: P13 (24h). Magnification x10. Picture 3.

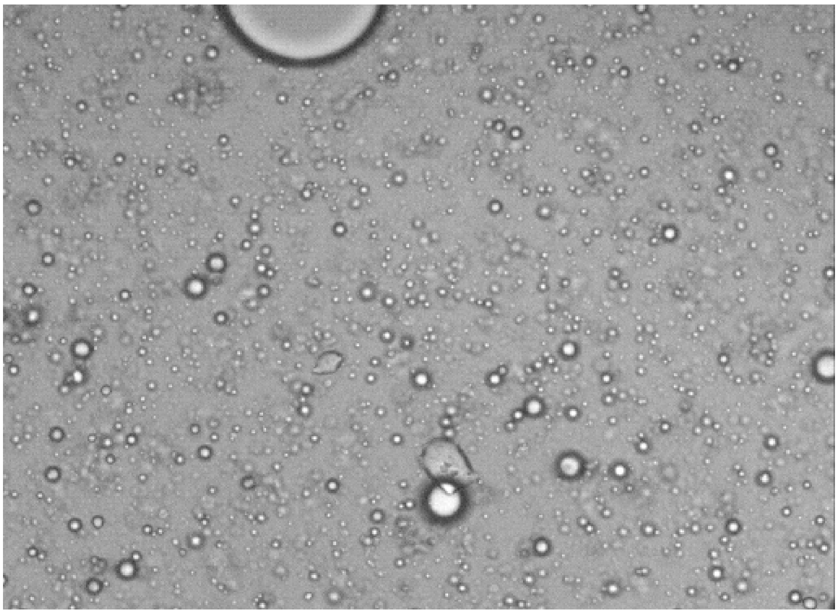


Figure 7.67: P13 (24h). Magnification x20.

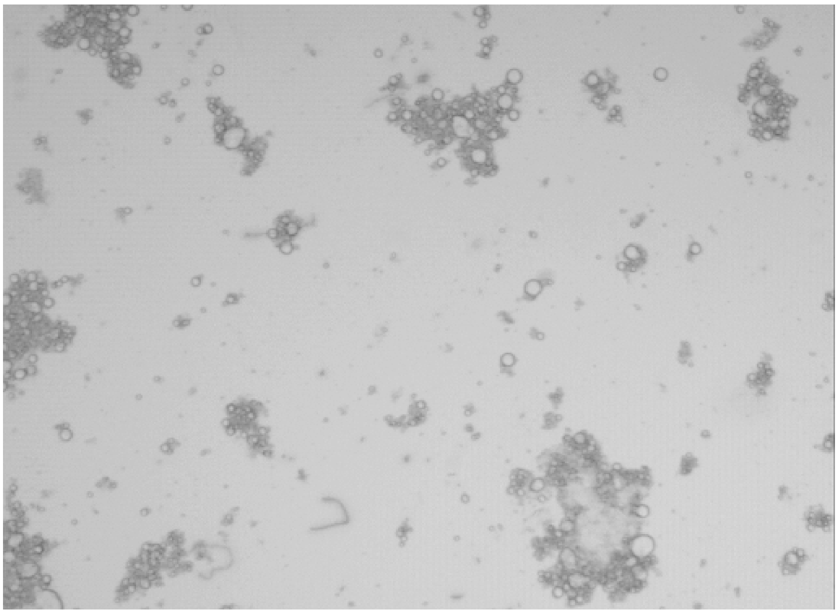


Figure 7.68: P13 (5 weeks). Magnification x5.

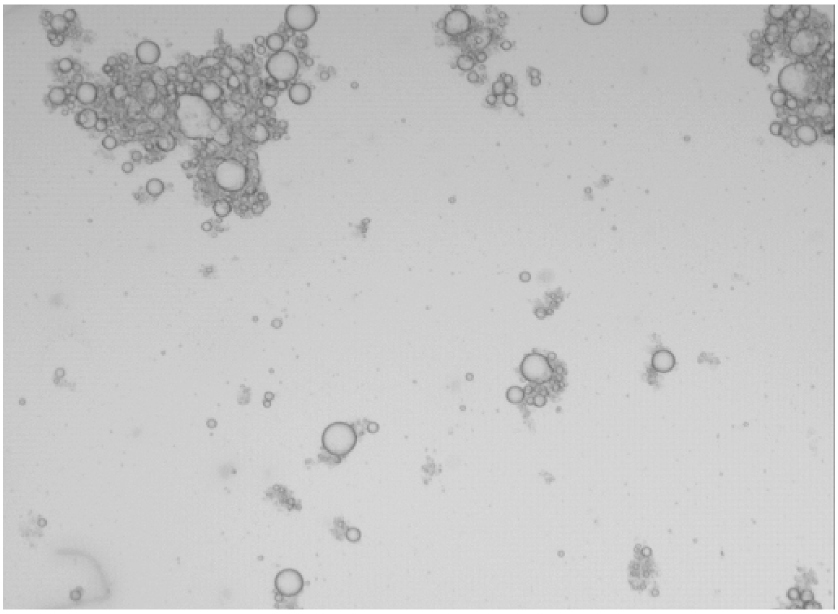


Figure 7.69: P13 (5 weeks). Magnification x10. Picture 1.

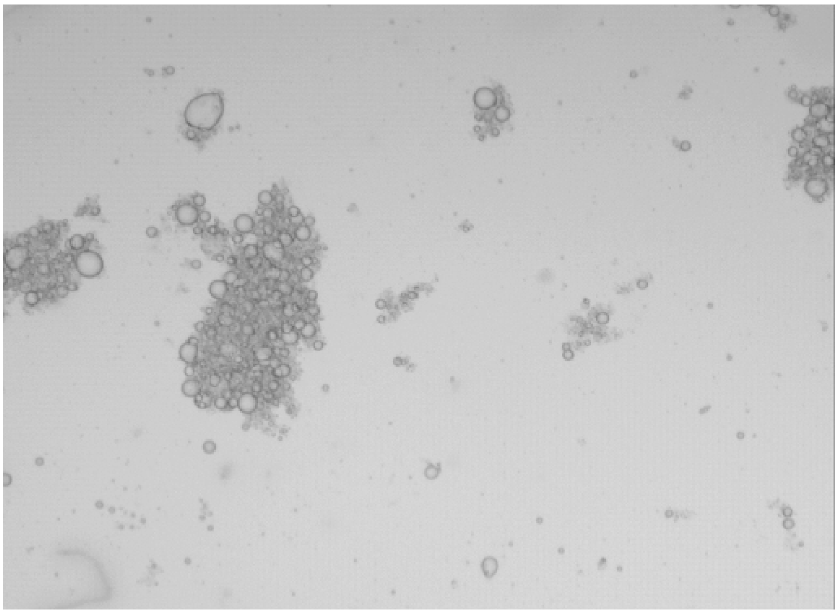


Figure 7.70: P13 (5 weeks). Magnification x10. Picture 2.

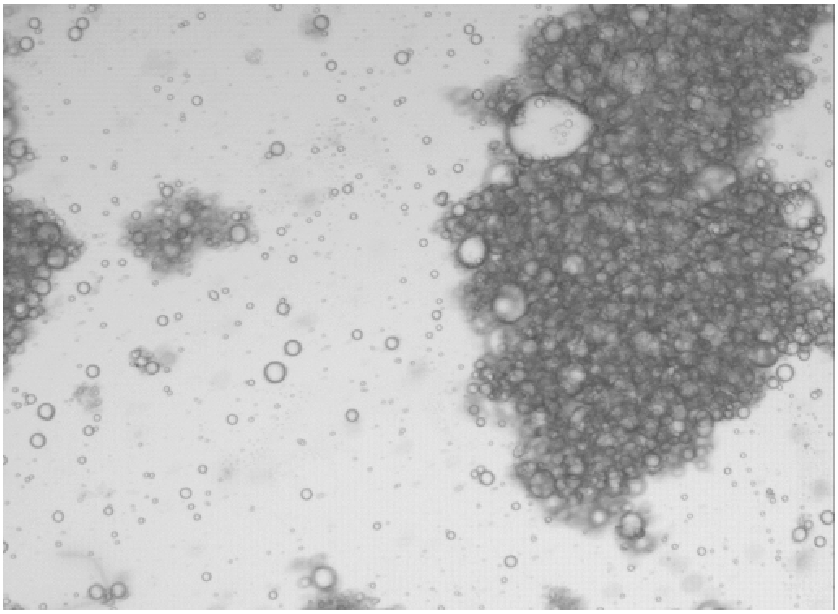


Figure 7.71: P13 (5 weeks). Magnification x10. Picture 3.

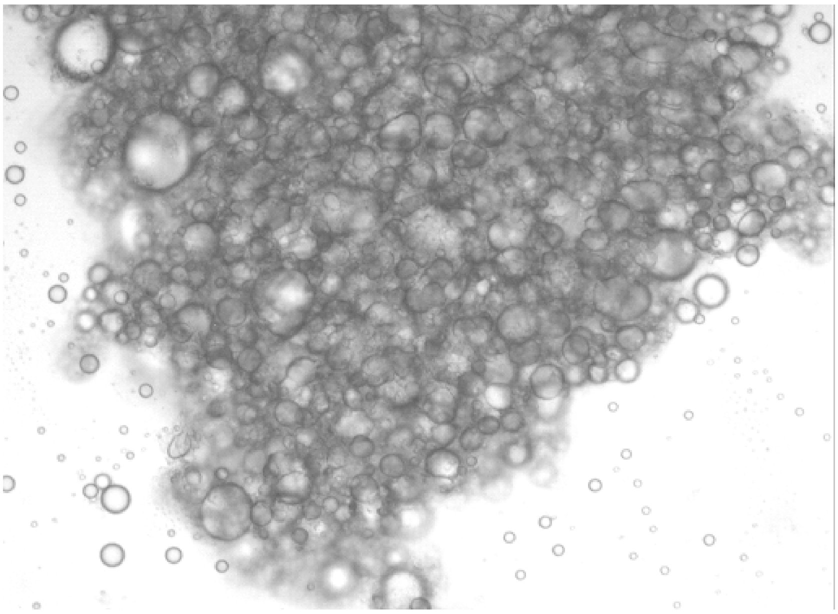


Figure 7.72: P13 (5 weeks). Magnification x20.

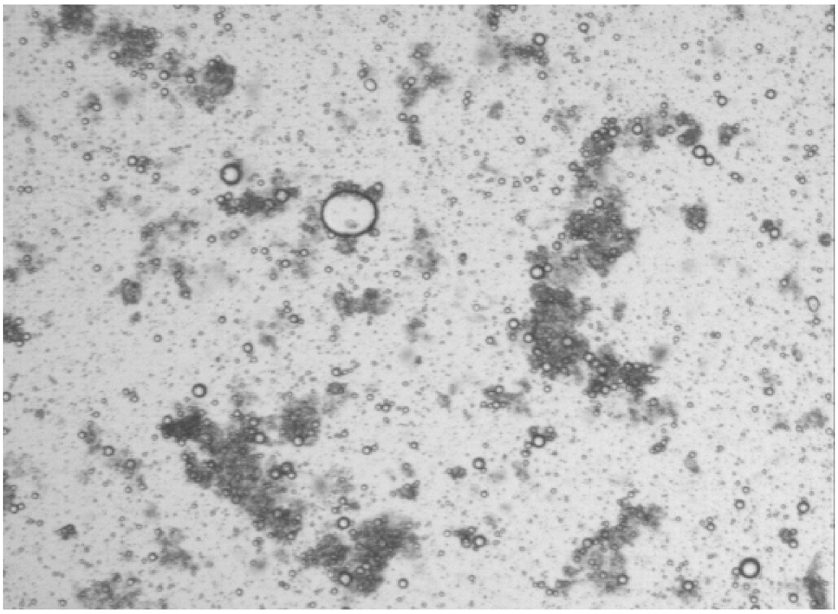


Figure 7.73: P18 (24h). Magnification x5.

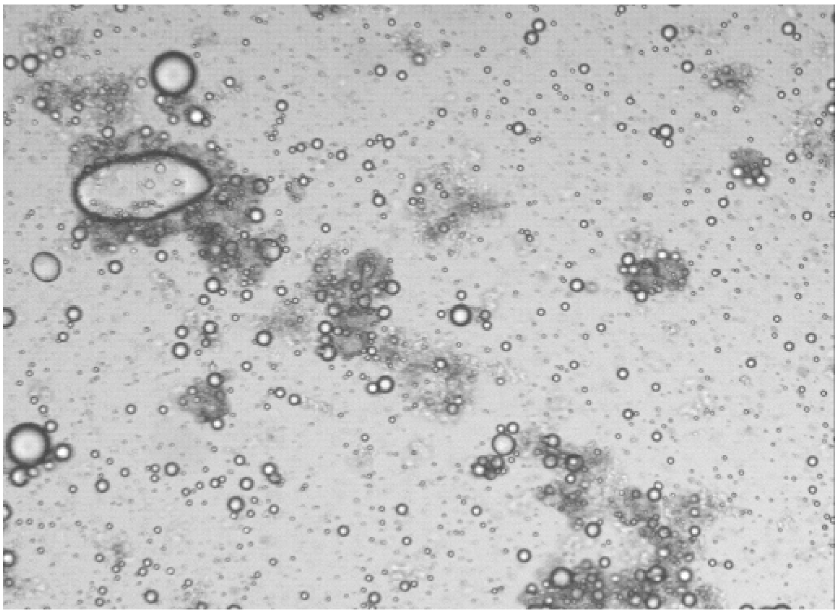


Figure 7.74: P18 (24h). Magnification x10. Picture 1.

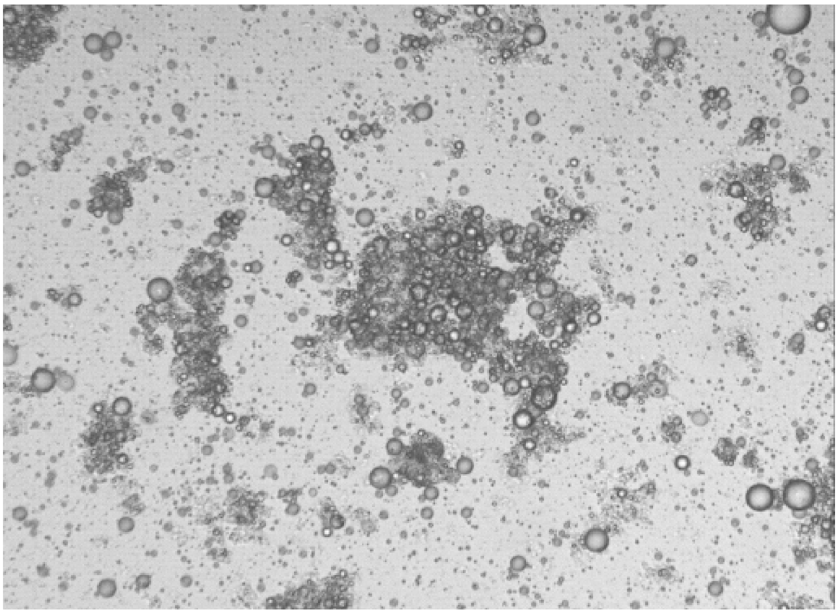


Figure 7.75: P18 (24h). Magnification x10. Picture 2.

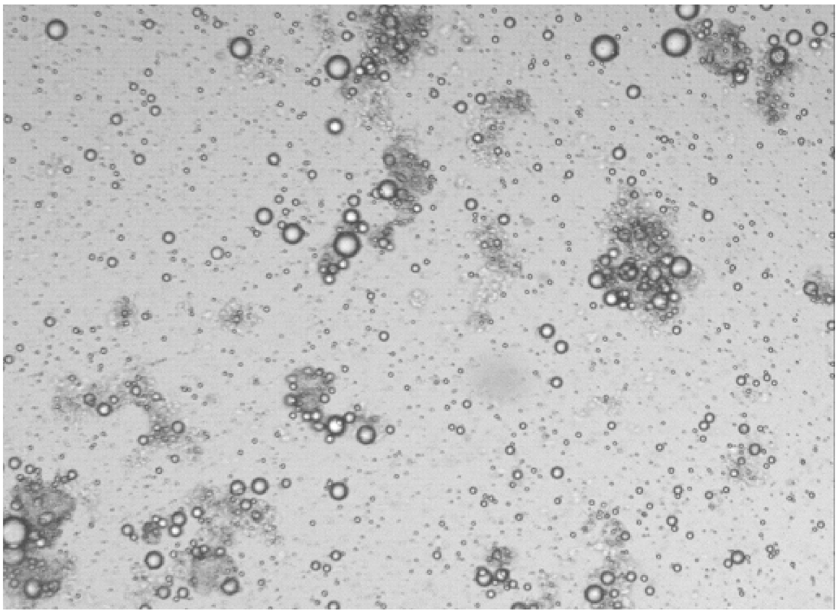


Figure 7.76: P18 (24h). Magnification x10. Picture 3.

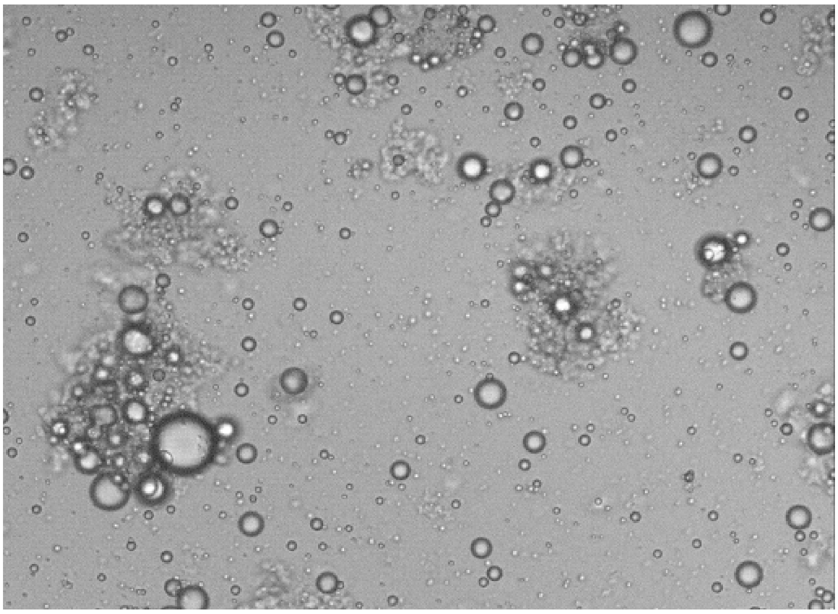


Figure 7.77: P18 (24h). Magnification x20.

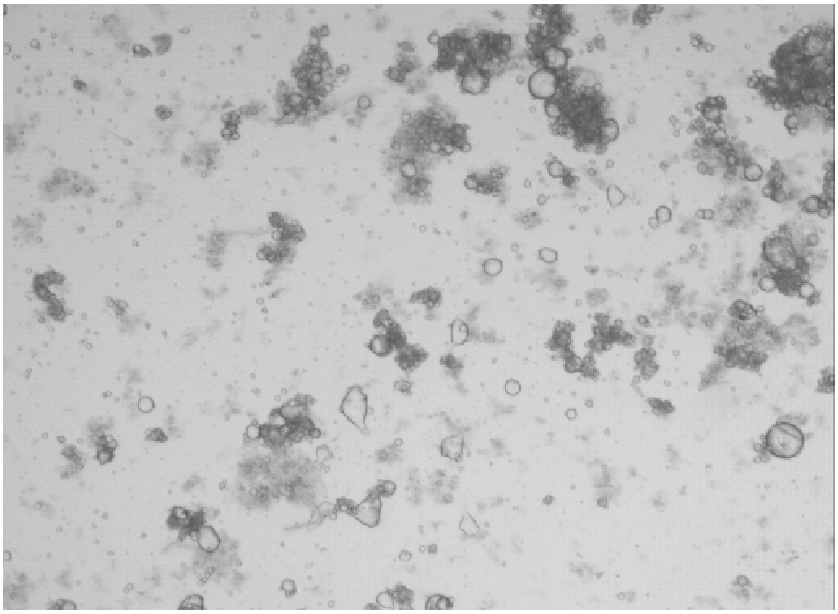


Figure 7.78: P18 (5 weeks). Magnification x5.

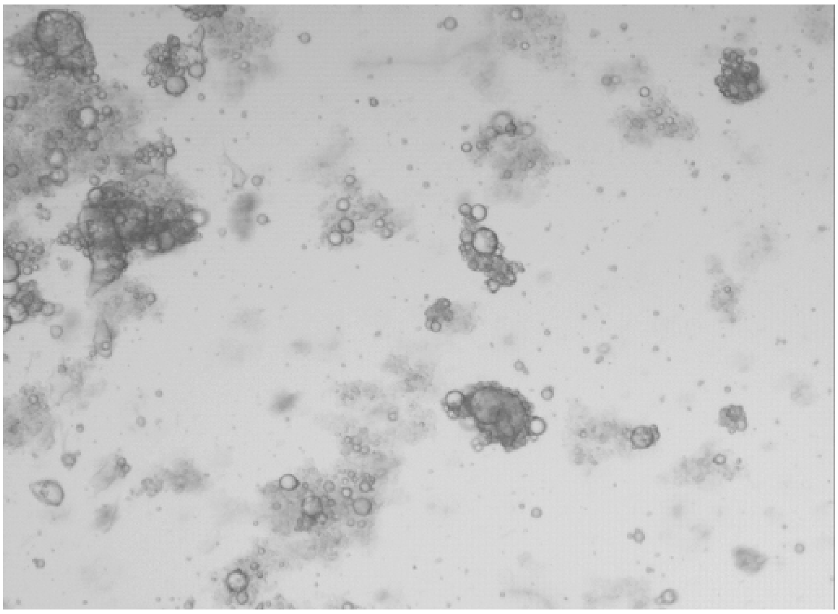


Figure 7.79: P18 (5 weeks). Magnification x10. Picture 1.

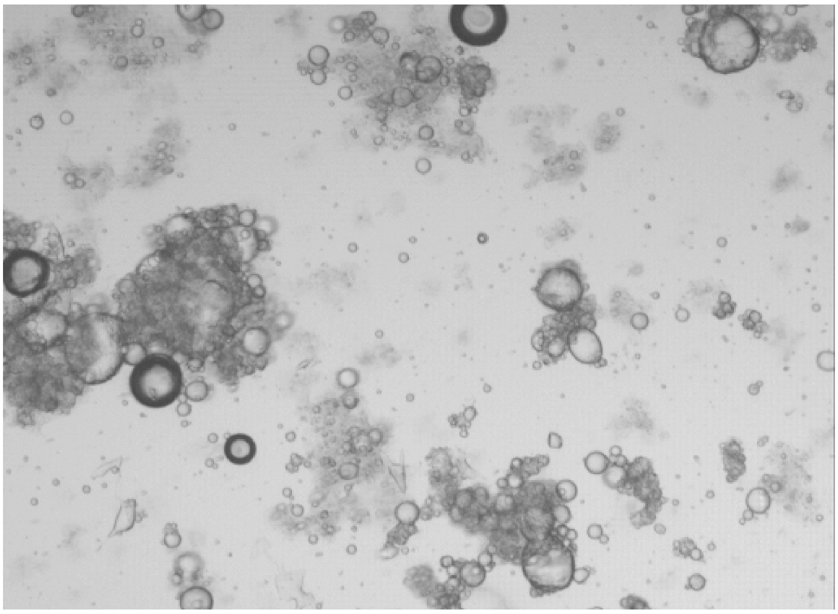


Figure 7.80: P18 (5 weeks). Magnification x10. Picture 2.

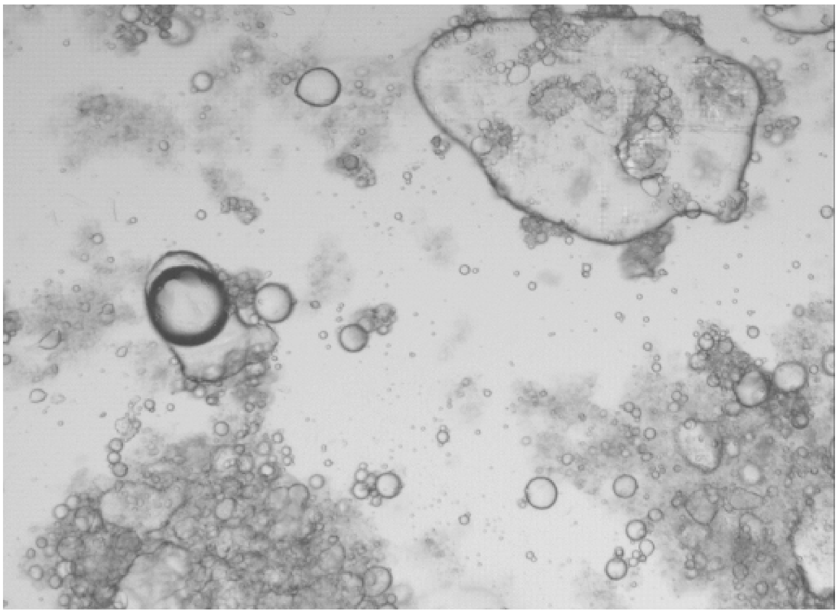


Figure 7.81: P18 (5 weeks). Magnification x10. Picture 3.

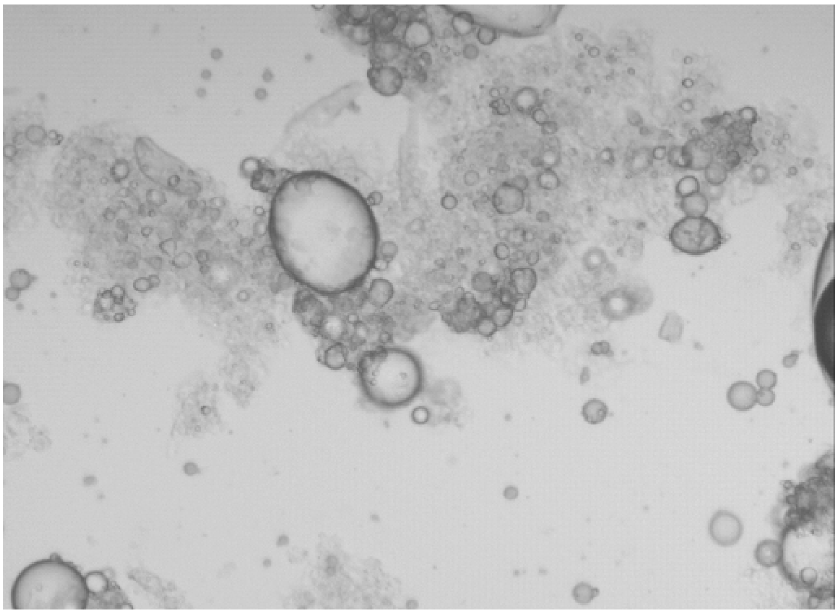


Figure 7.82: P18 (5 weeks). Magnification x20.

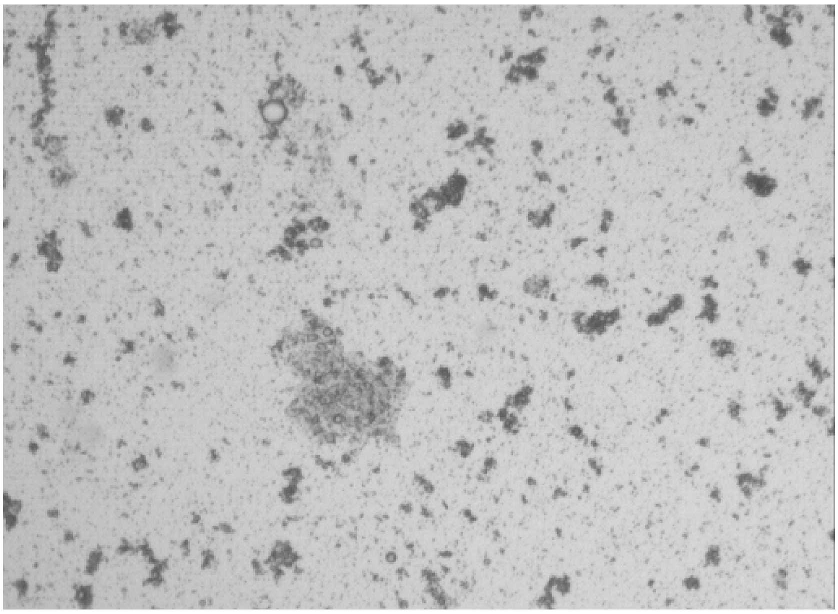


Figure 7.83: P21 (24h). Magnification x5.

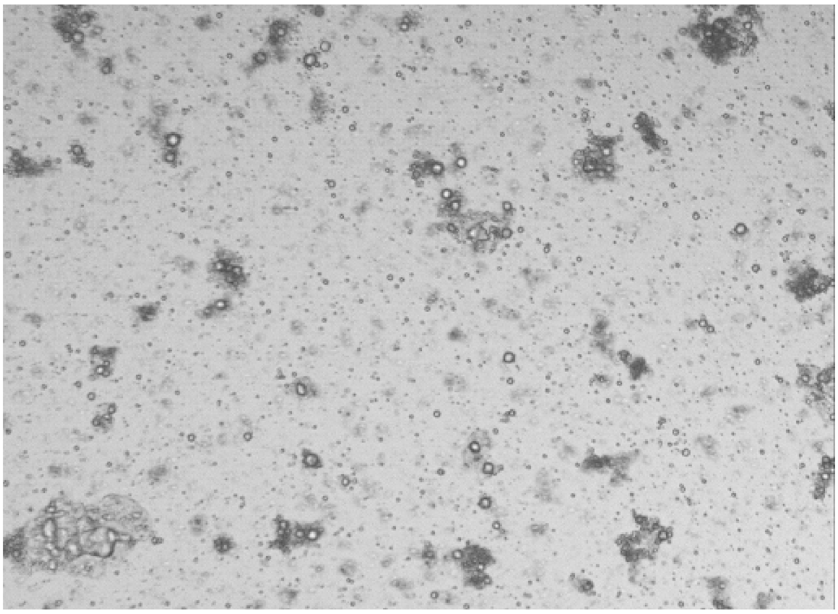


Figure 7.84: P21 (24h). Magnification x10. Picture 1.

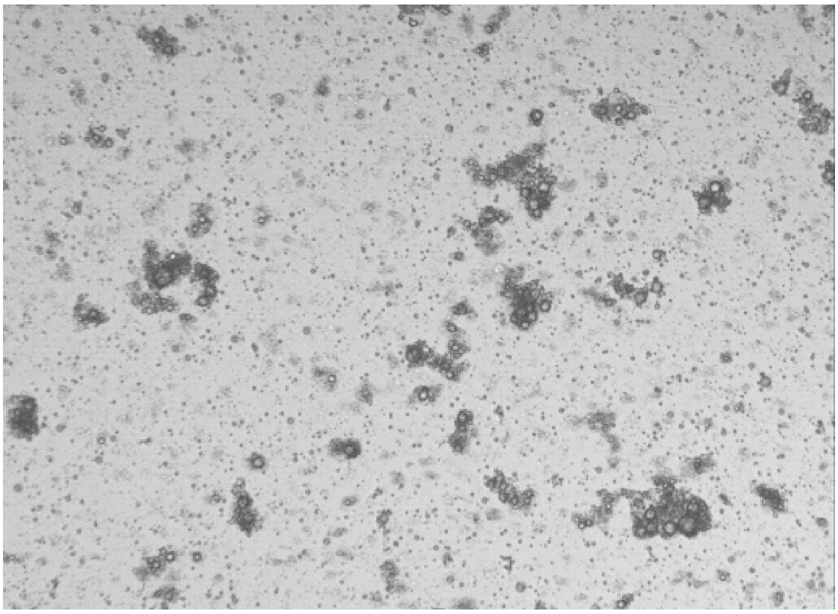


Figure 7.85: P21 (24h). Magnification x10. Picture 2.

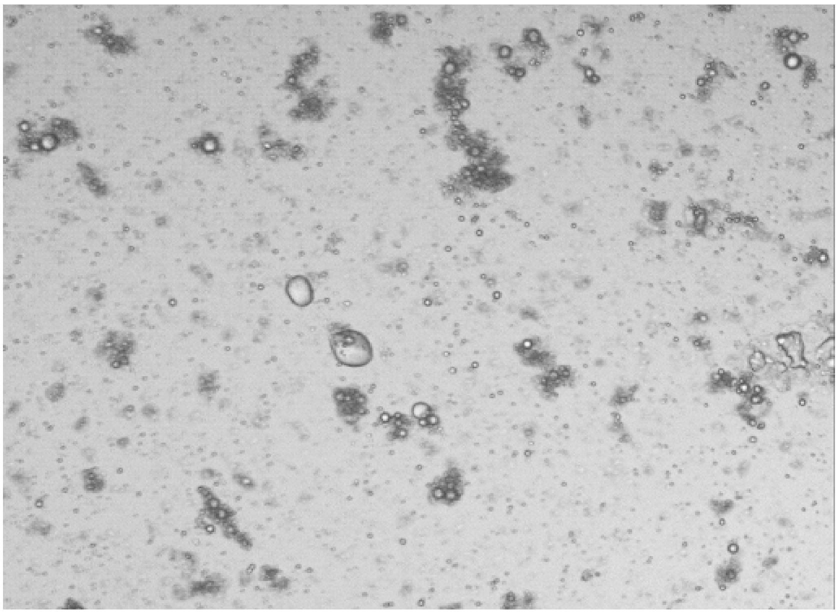


Figure 7.86: P21 (24h). Magnification x10. Picture 3.

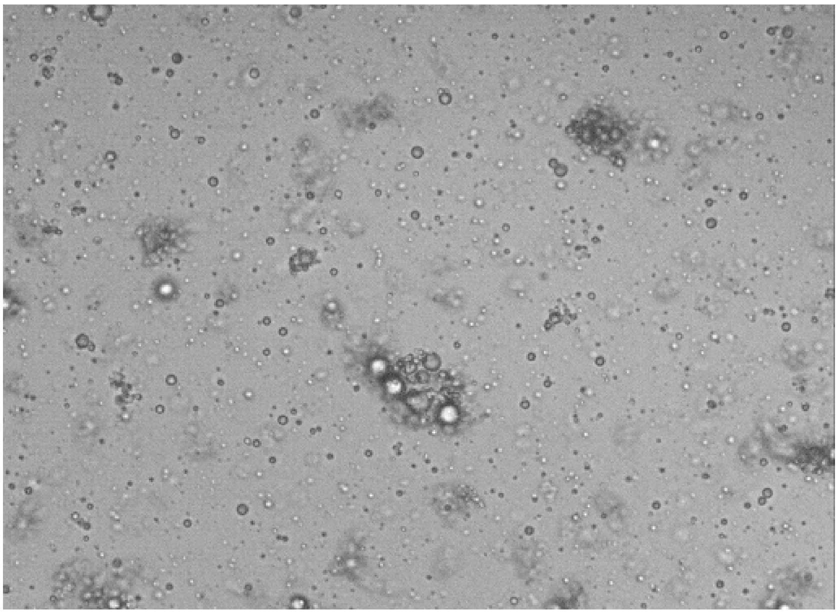


Figure 7.87: P21 (24h). Magnification x20.

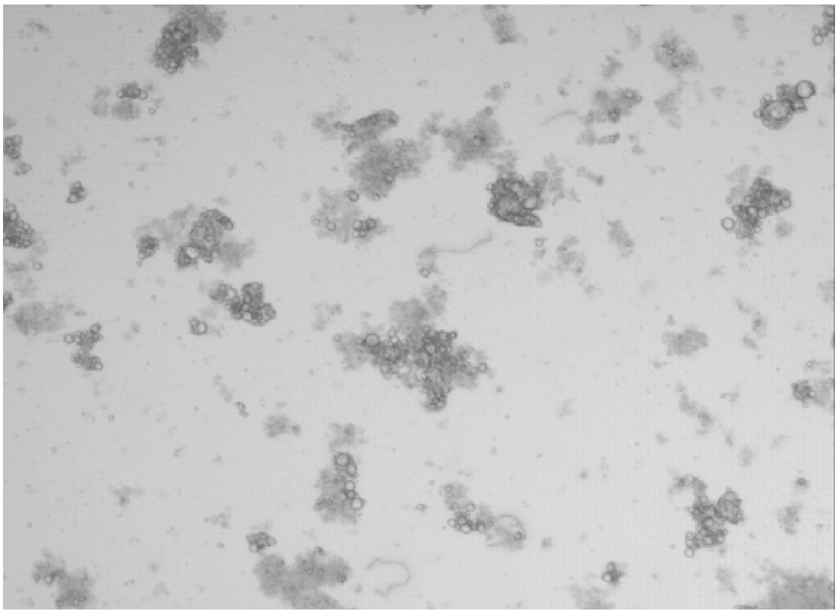


Figure 7.88: P21 (5 weeks). Magnification x5.

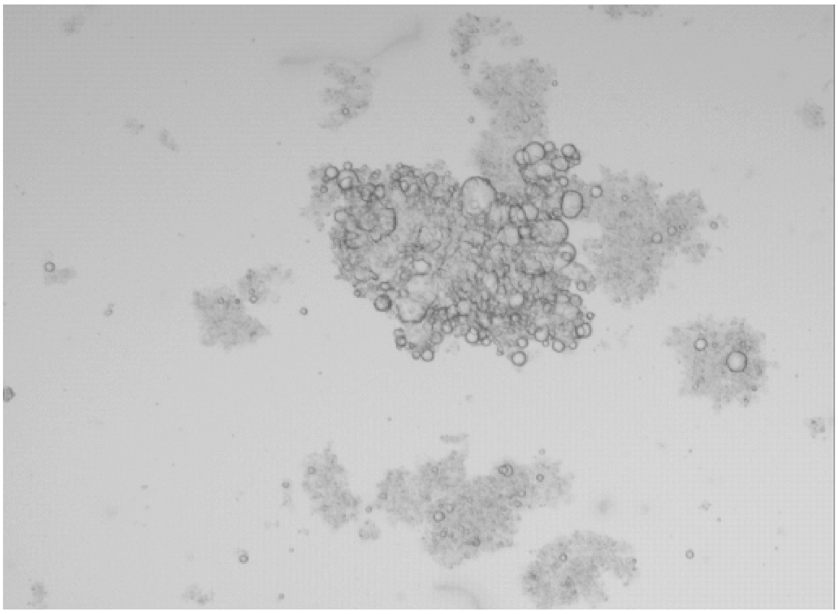


Figure 7.89: P21 (5 weeks). Magnification x10. Picture 1.

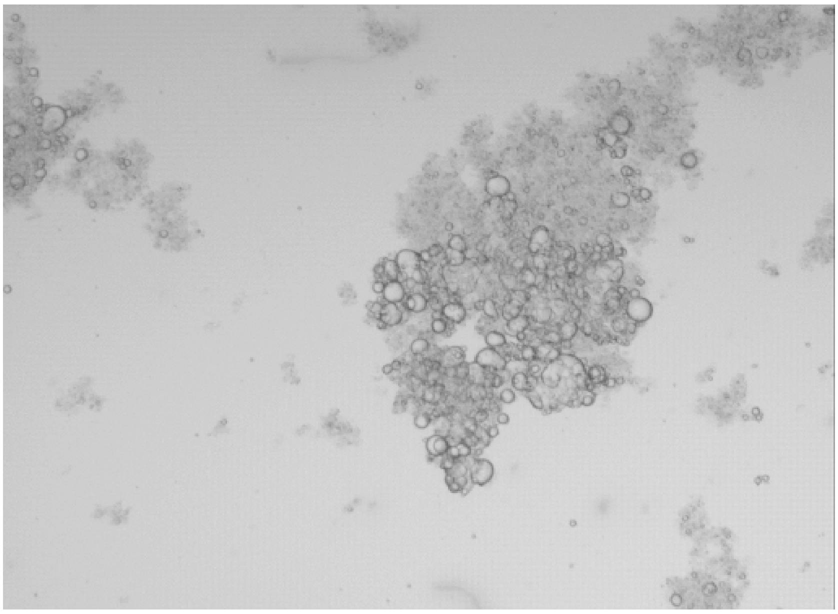


Figure 7.90: P21 (5 weeks). Magnification x10. Picture 2.

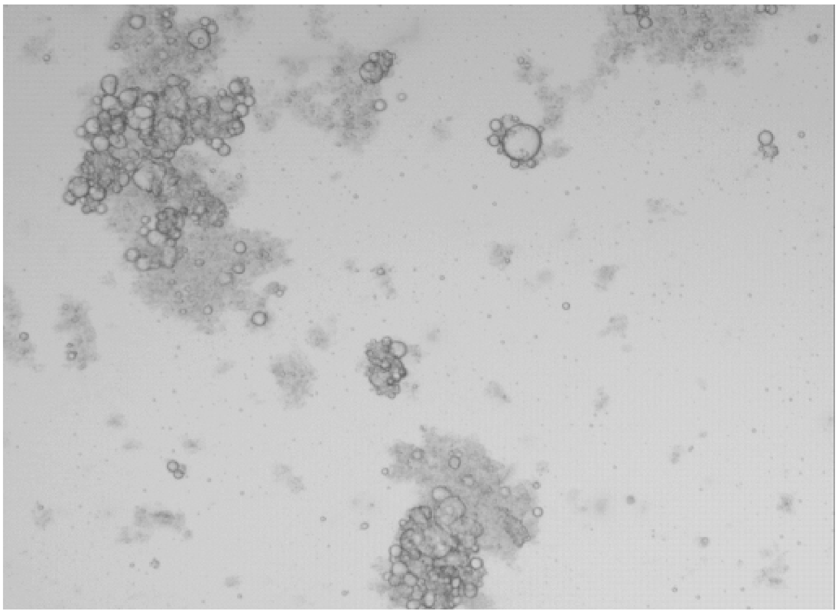


Figure 7.91: P21 (5 weeks). Magnification x10. Picture 3.

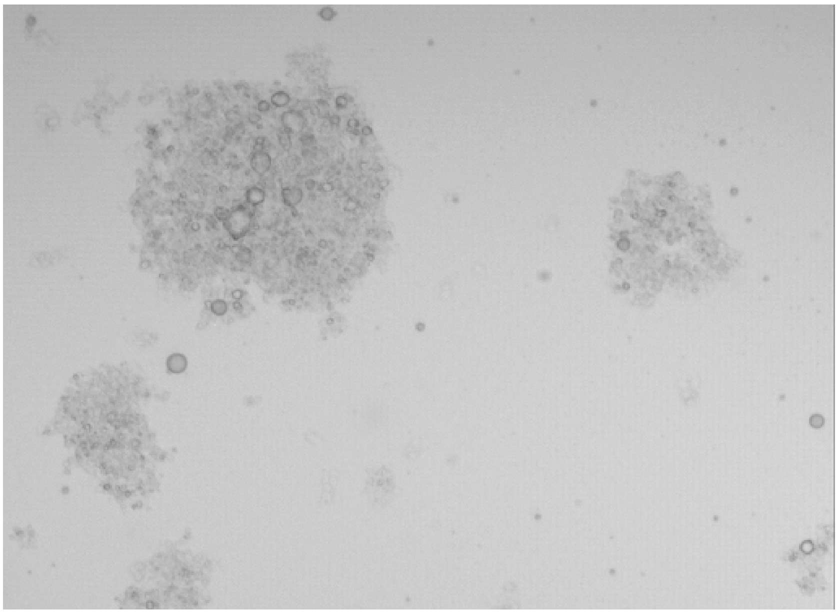


Figure 7.92: P21 (5 weeks). Magnification x20.

7.4 Appendix D - Microscopy images of hair

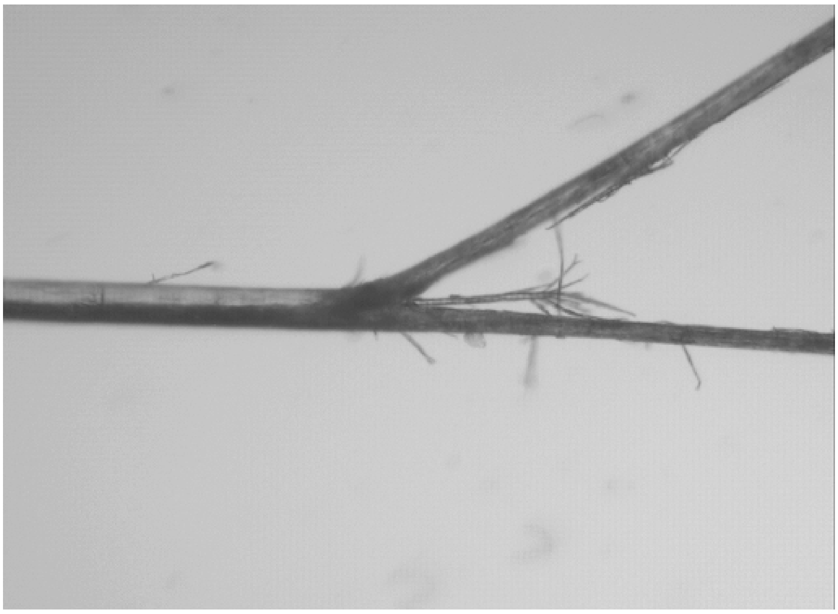


Figure 7.93: Hair shaft before treatment with formulation C13. Magnification x5. Picture 1.

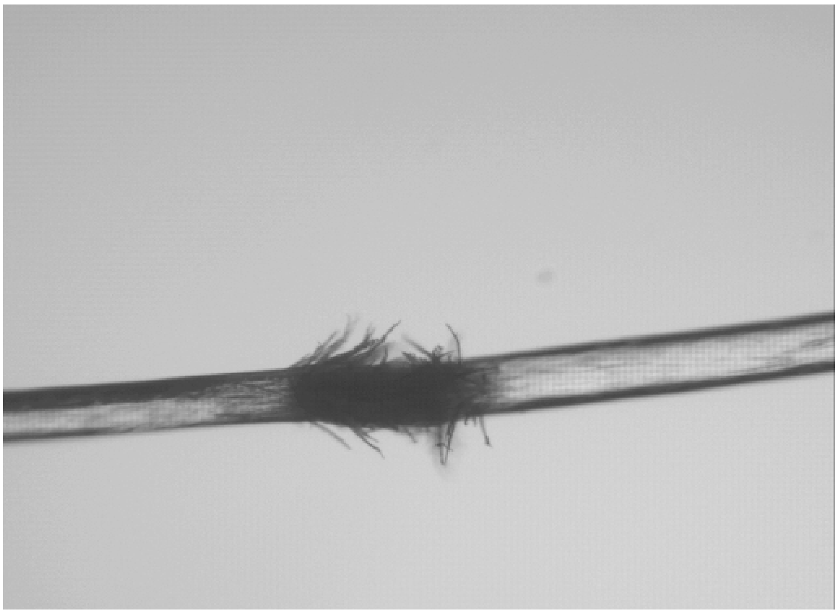


Figure 7.94: Hair shaft before treatment with formulation C13. Magnification x5. Picture 2.

Figure 7.95: Hair shaft before treatment with formulation C13. Magnification x5. Picture 3.

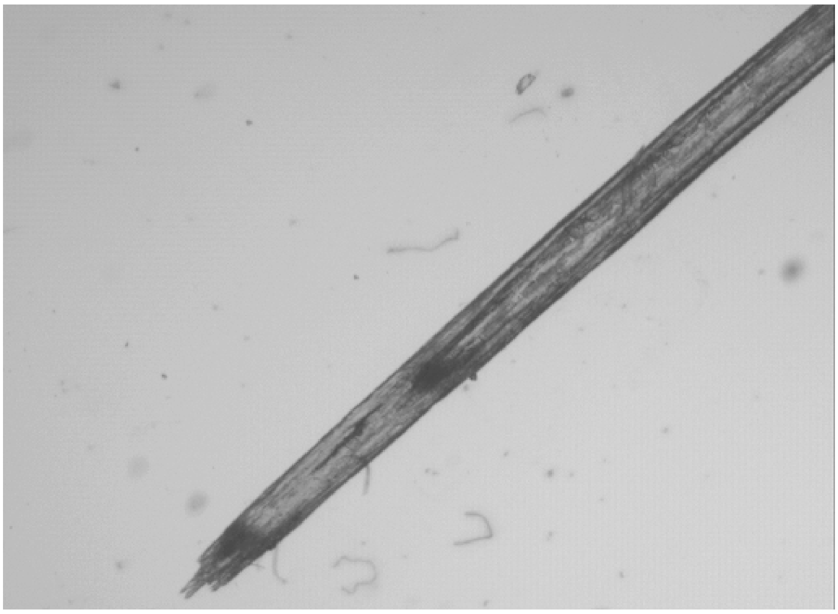


Figure 7.96: Hair shaft after treatment with formulation C13. Magnification x5. Picture 1.

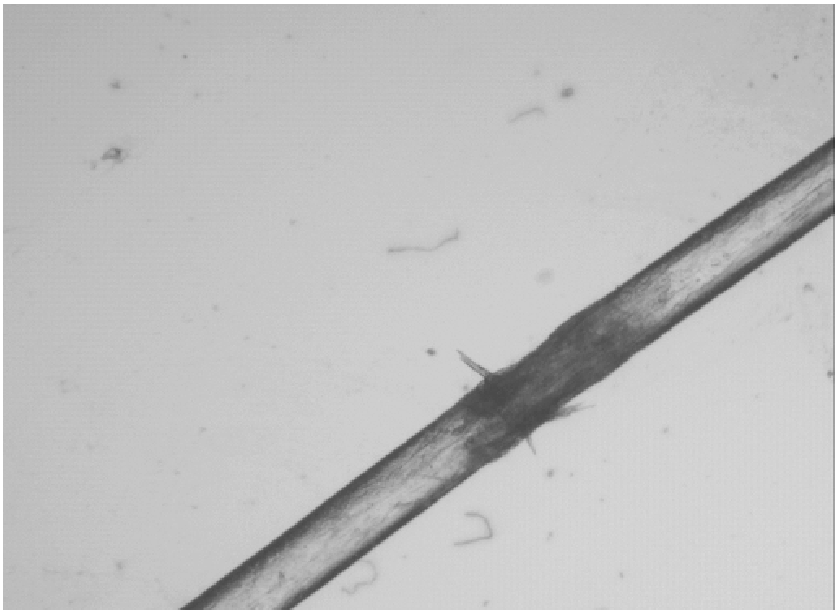


Figure 7.97: Hair shaft after treatment with formulation C13. Magnification x5. Picture 2.

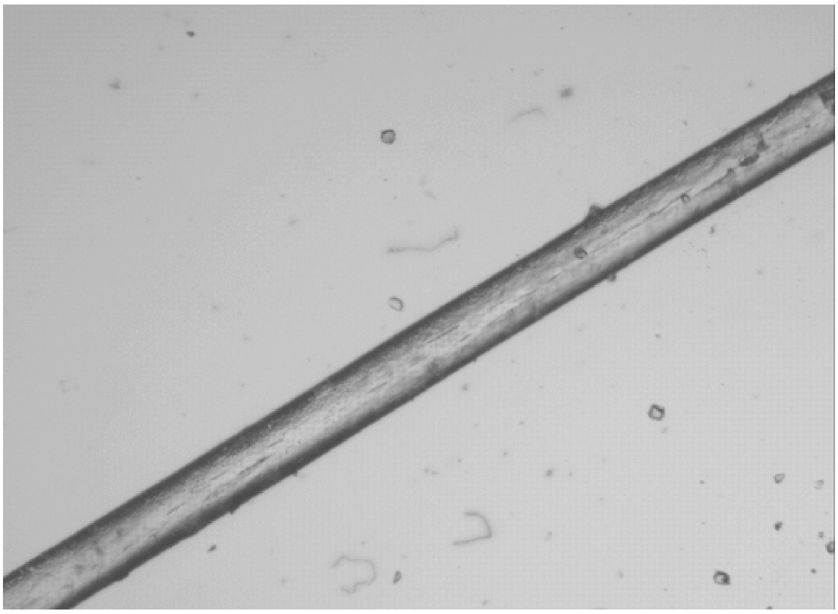


Figure 7.98: Hair shaft after treatment with formulation C13. Magnification x5. Picture 3.

Figure 7.99: Hair shaft before treatment with formulation C14. Magnification x5. Picture 1.

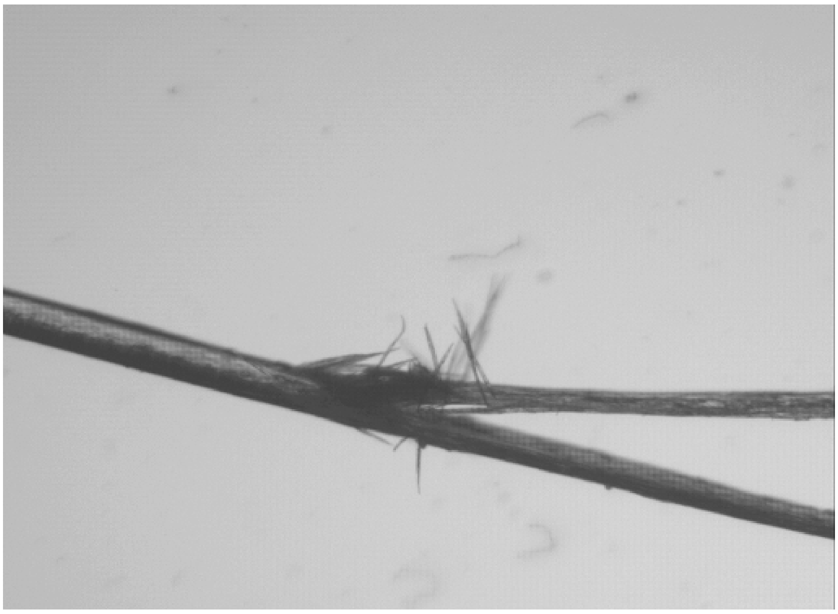


Figure 7.100: Hair shaft before treatment with formulation C14. Magnification x5. Picture 2.

Figure 7.101: Hair shaft before treatment with formulation C14. Magnification x5. Picture 3.

Figure 7.102: Hair shaft after treatment with formulation C14. Magnification x5. Picture 1.

Figure 7.103: Hair shaft after treatment with formulation C14. Magnification x5. Picture 2.

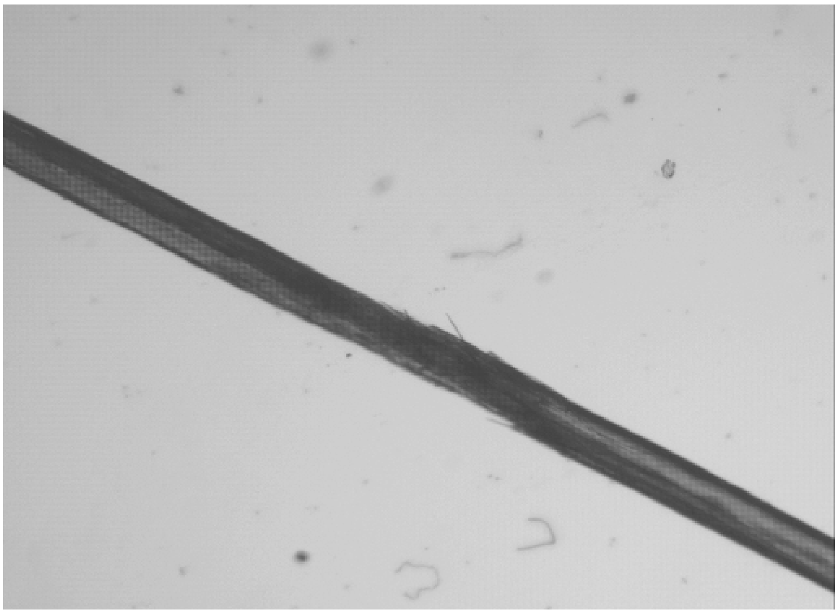


Figure 7.104: Hair shaft after treatment with formulation C14. Magnification x5. Picture 3.

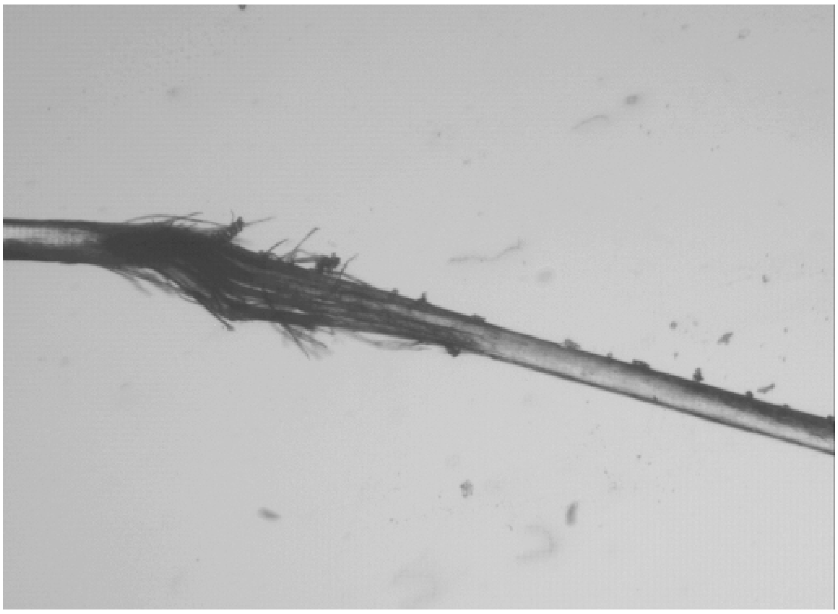


Figure 7.105: Hair shaft before treatment with formulation P18. Magnification x5. Picture 1.

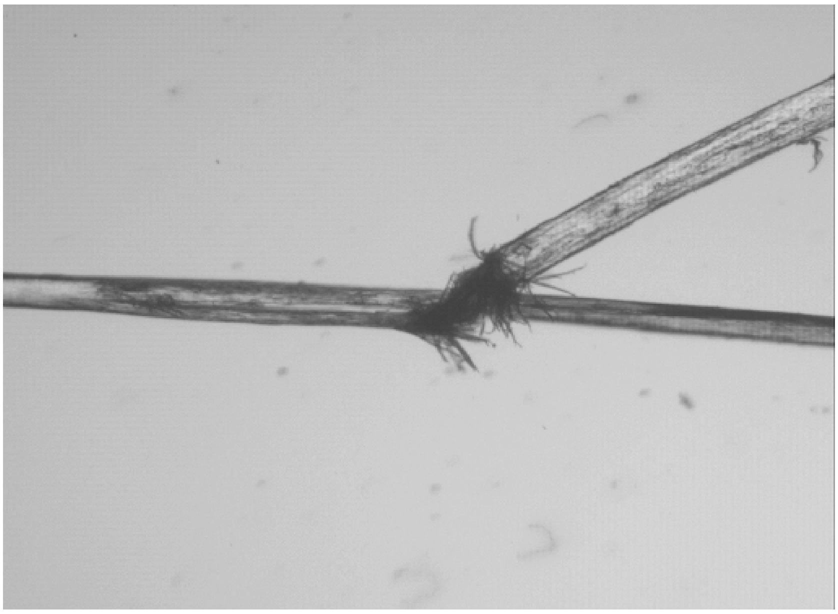


Figure 7.106: Hair shaft before treatment with formulation P18. Magnification x5. Picture 2.

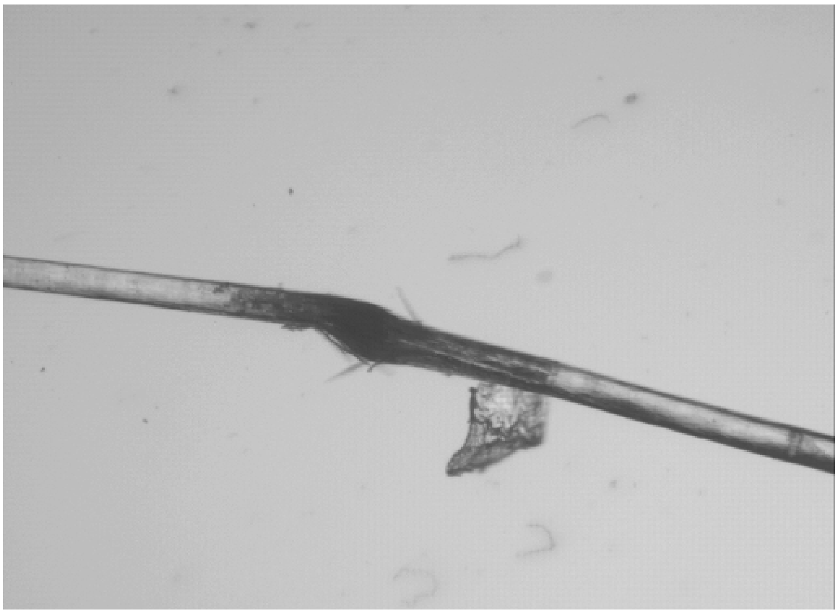


Figure 7.107: Hair shaft before treatment with formulation P18. Magnification x5. Picture 3.

Figure 7.108: Hair shaft after treatment with formulation P18. Magnification x5. Picture 1.

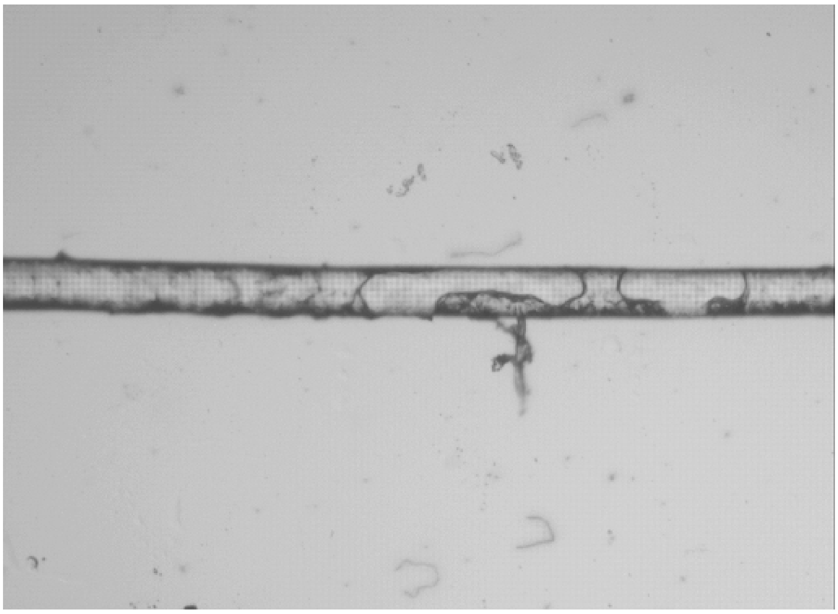


Figure 7.109: Hair shaft after treatment with formulation P18. Magnification x5. Picture 2.

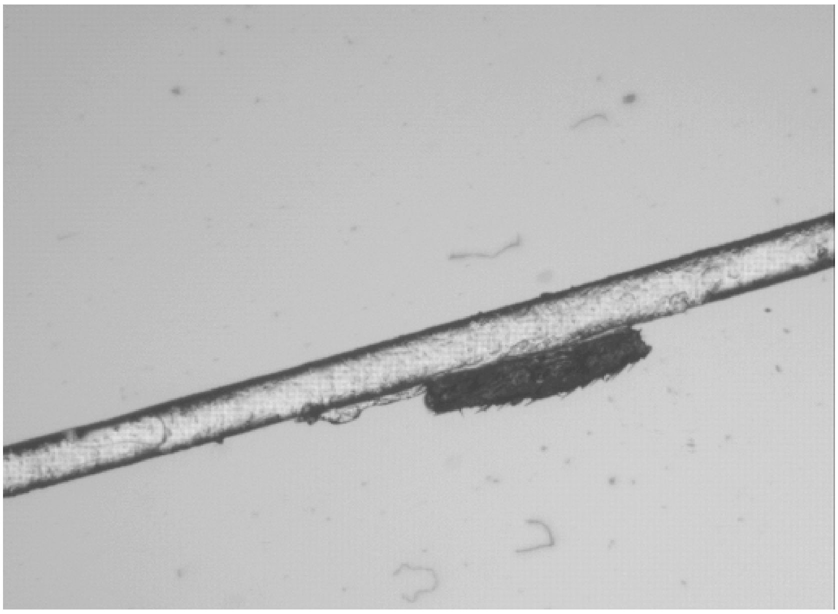
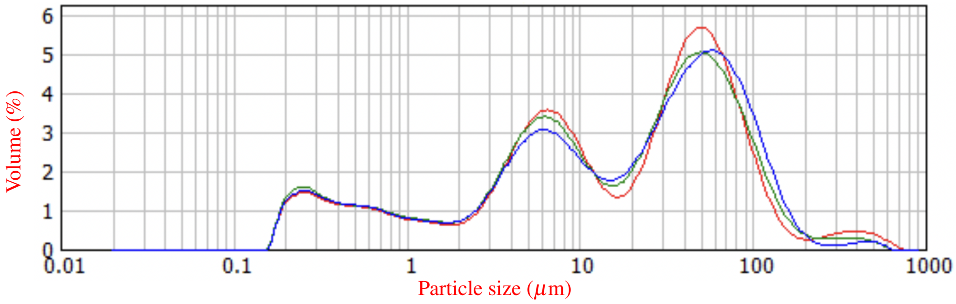


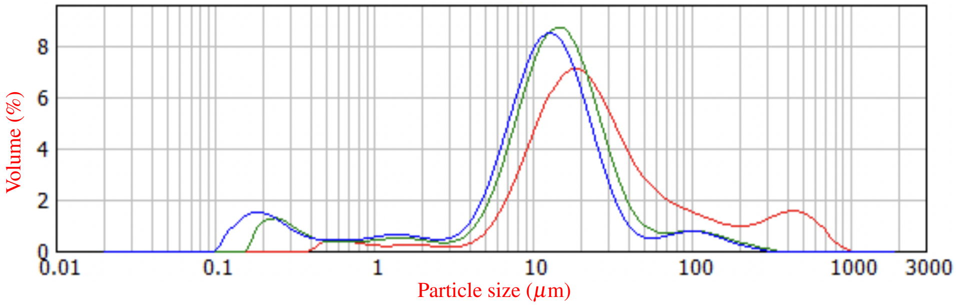
Figure 7.110: Hair shaft after treatment with formulation P18. Magnification x5. Picture 3.

7.5 Appendix E - Particle size distribution graphs

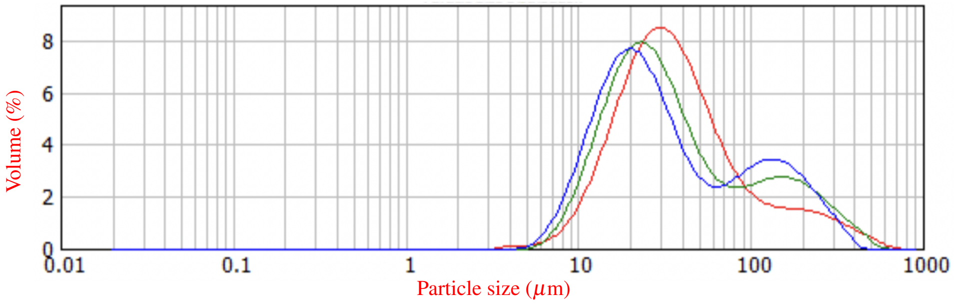
C1.1 (24h)



C1.1 (5 weeks)



C1.2 (24h)



C1.2 (5 weeks)

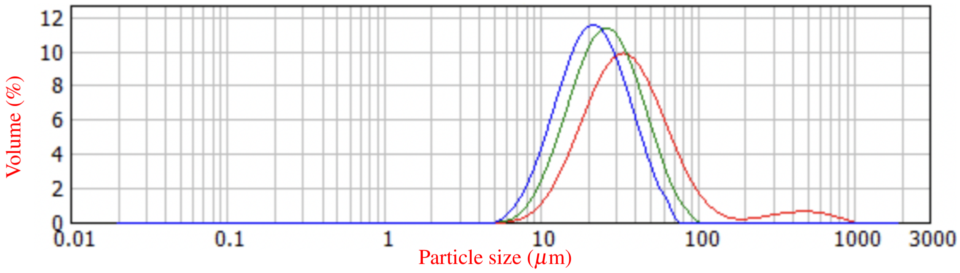


Figure 7.111: Particle size distribution graphs of formulation C1. Red graph shows measurement 1, green graph measurement 2 and blue graph measurement 3.

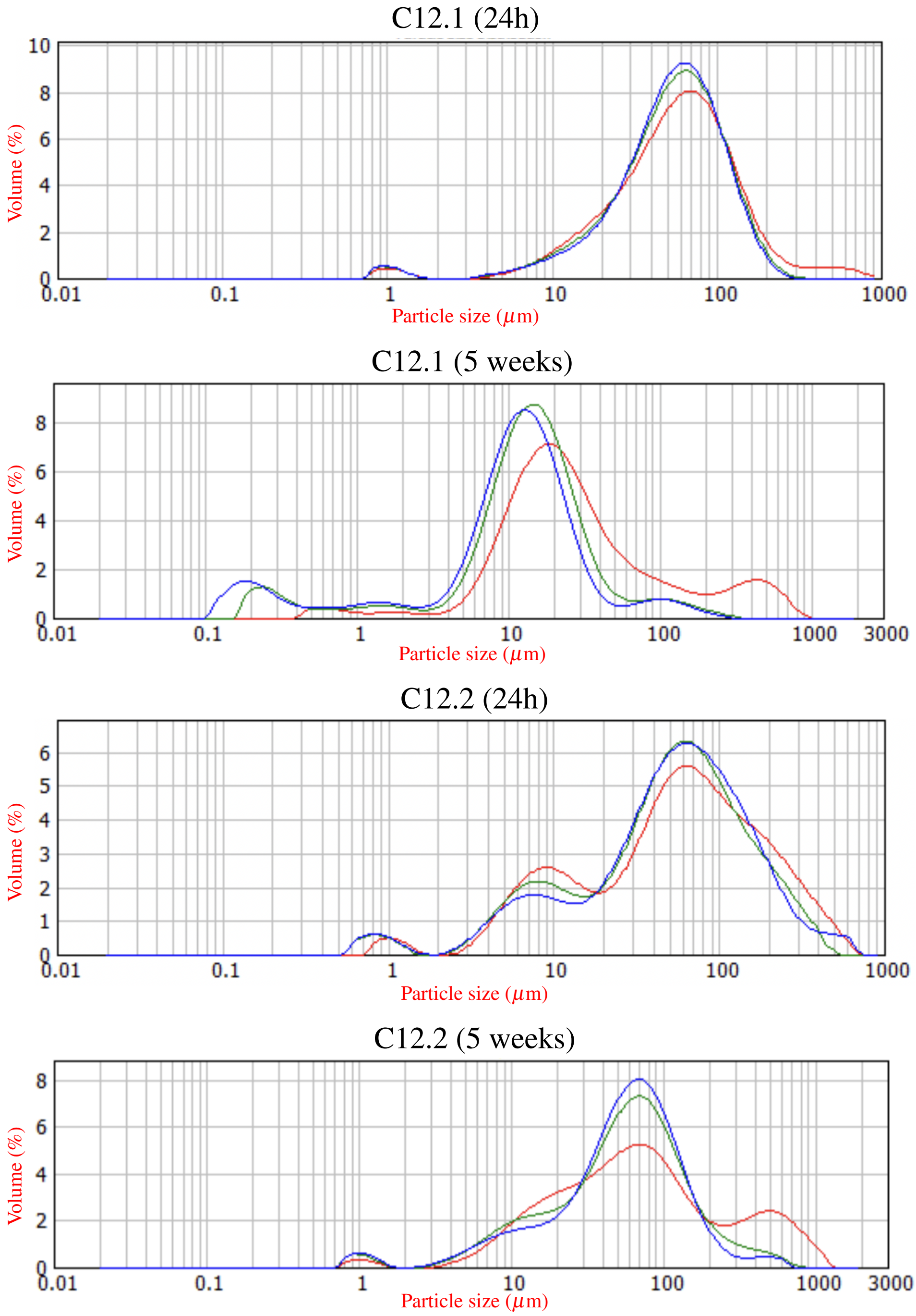
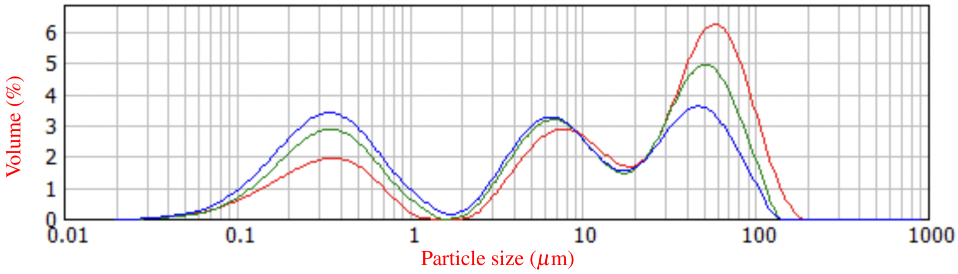
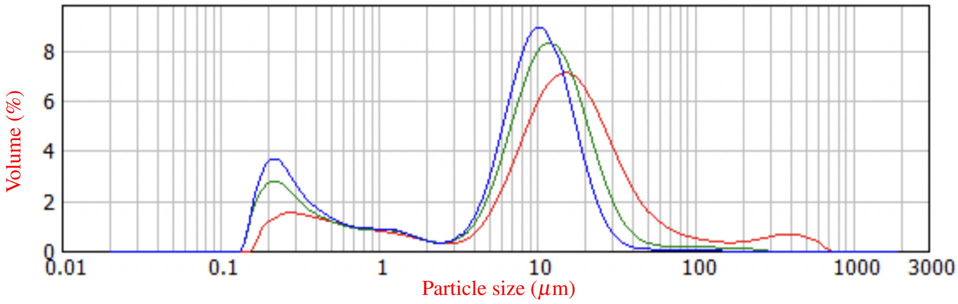


Figure 7.112: Particle size distributions of formulation C12. Red graph shows measurement 1, green graph measurement 2 and blue graph measurement 3.

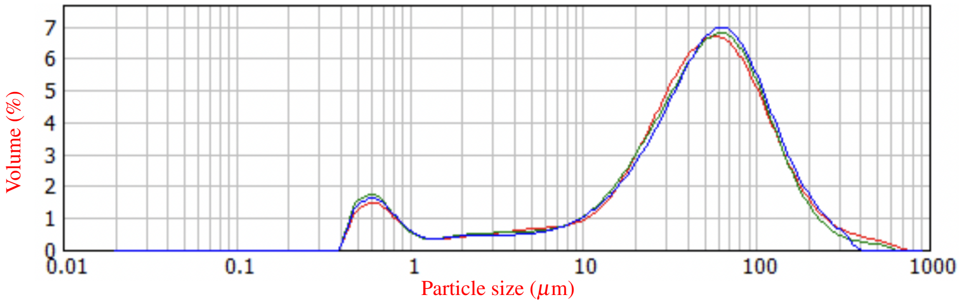
C13.1 (24h)



C13.1 (5 weeks)



C13.2 (24h)



C13.2 (5 weeks)

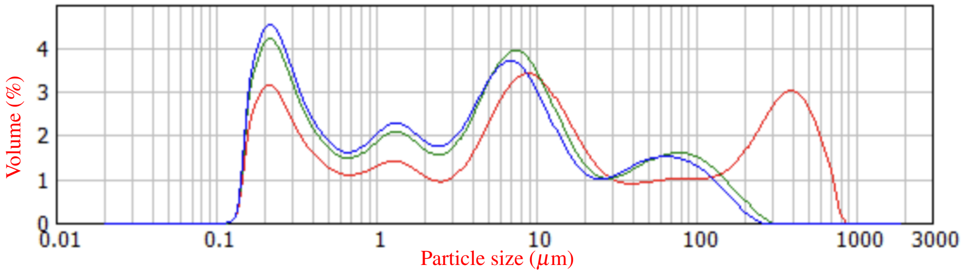


Figure 7.113: Particle size distributions of formulation C13. Red graph shows measurement 1, green graph measurement 2 and blue graph measurement 3.

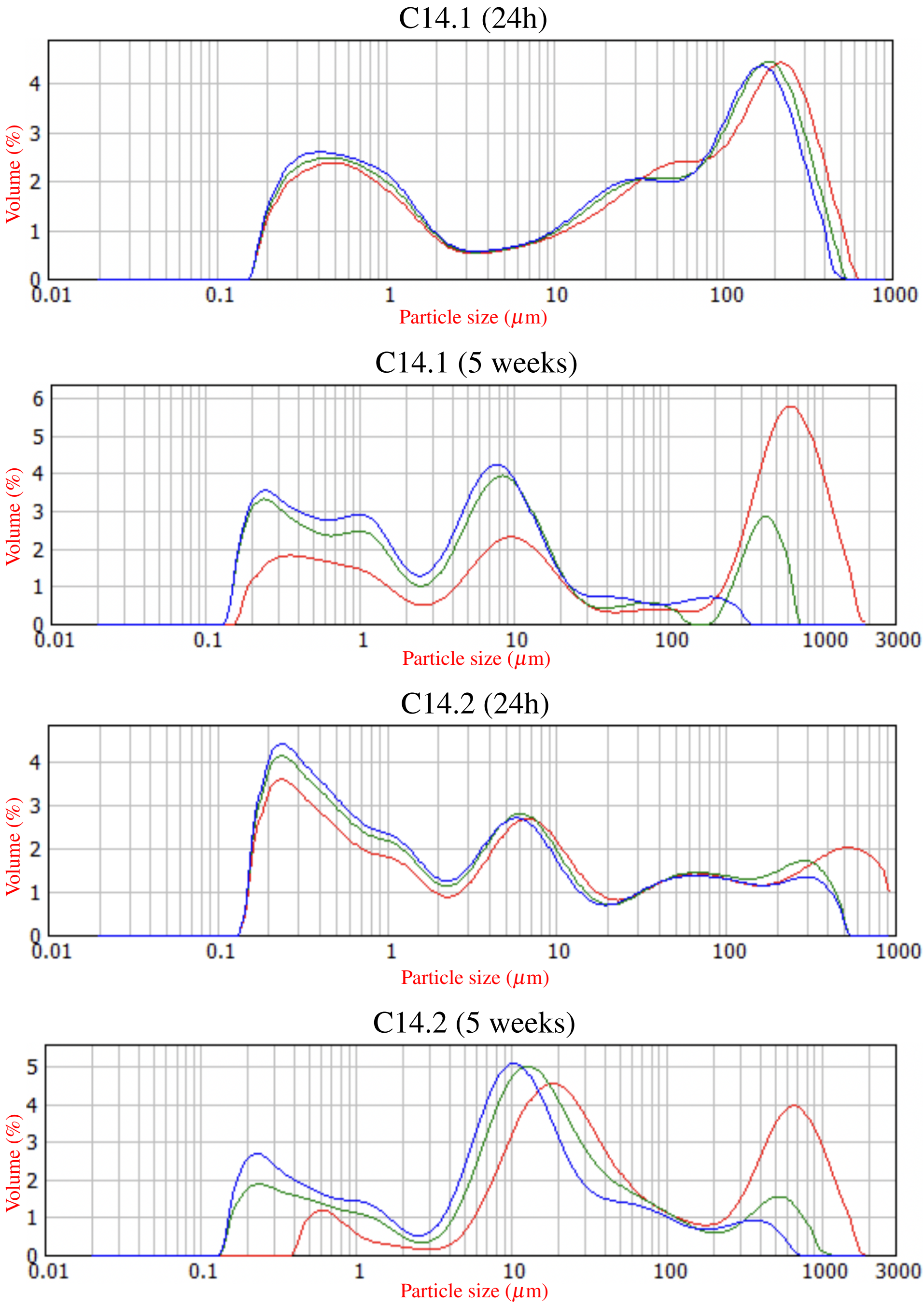


Figure 7.114: Particle size distributions of formulation C14. Red graph shows measurement 1, blue graph measurement 2 and green graph measurement 3.

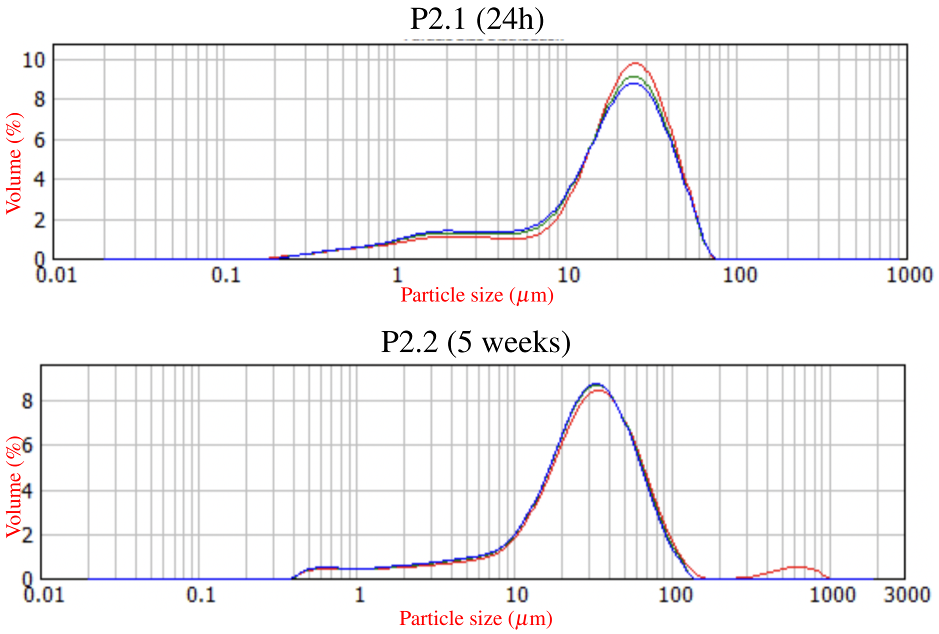
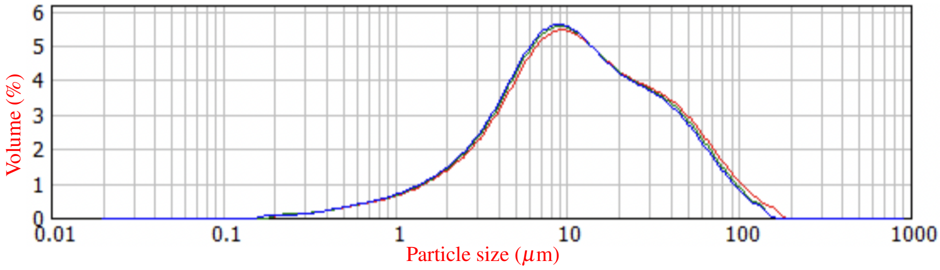
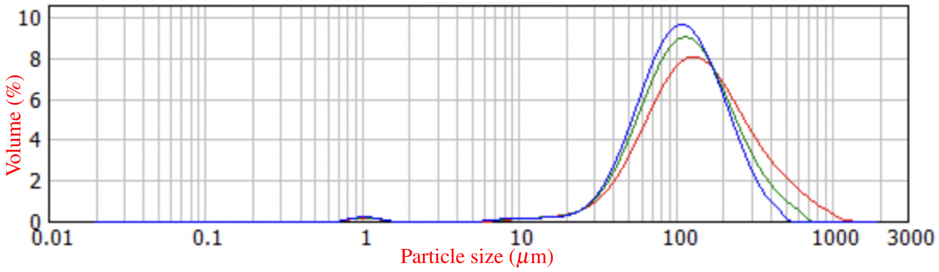


Figure 7.115: Particle size distributions of formulation P2. Red graph shows measurement 1, green graph measurement 2 and blue graph measurement 3.

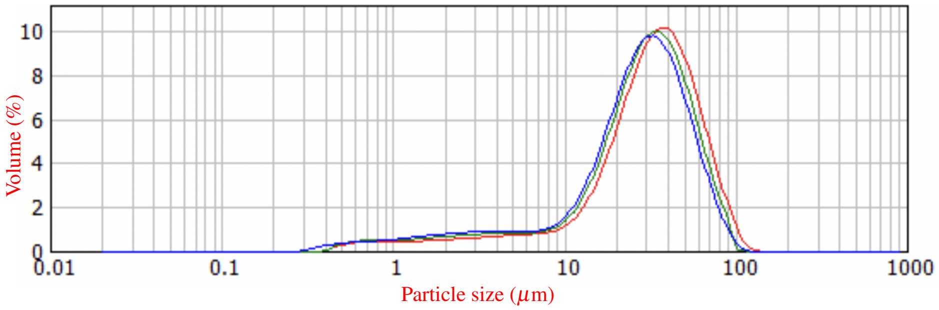
P7.1 (24h)



P7.1 (5 weeks)



P7.2 (24h)



P7.2 (5 weeks)

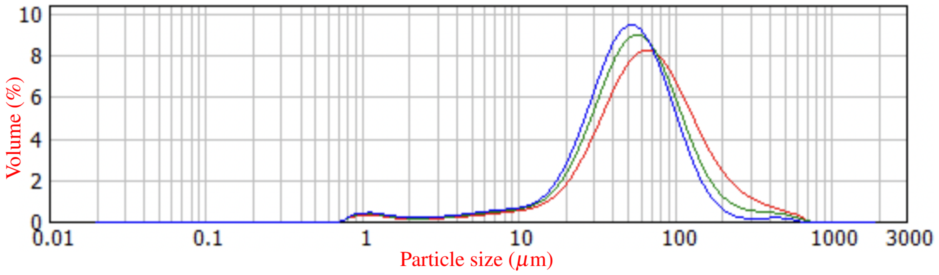
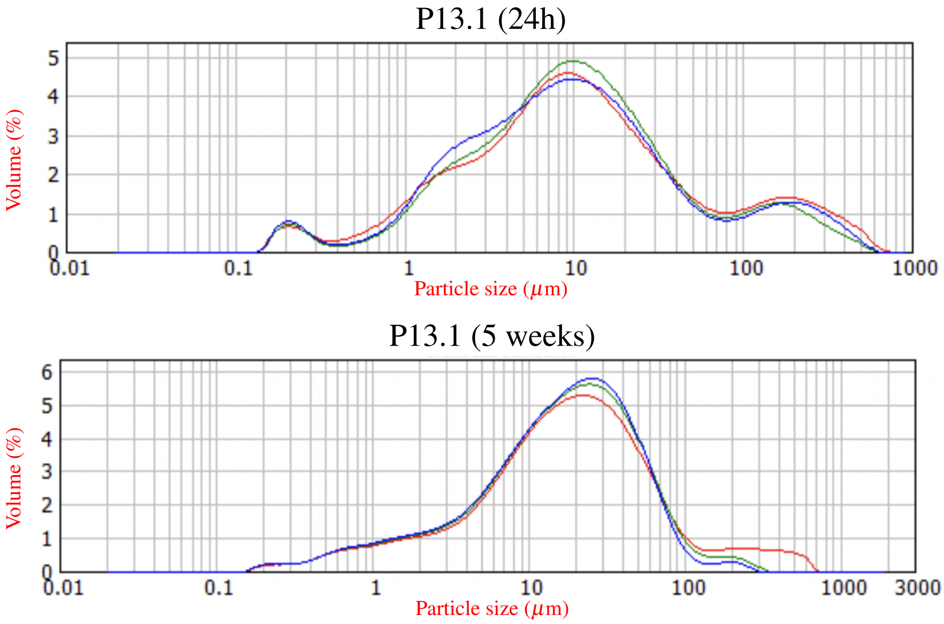


Figure 7.116: Particle size distributions of formulation P7. Red graph shows measurement 1, green graph measurement 2 and blue graph measurement 3.



P13.1 (24h)

P13.1 (5 weeks)

Figure 7.117: Particle size distributions of formulation P13. Red graph shows measurement 1, blue graph measurement 2 and green graph measurement 3.

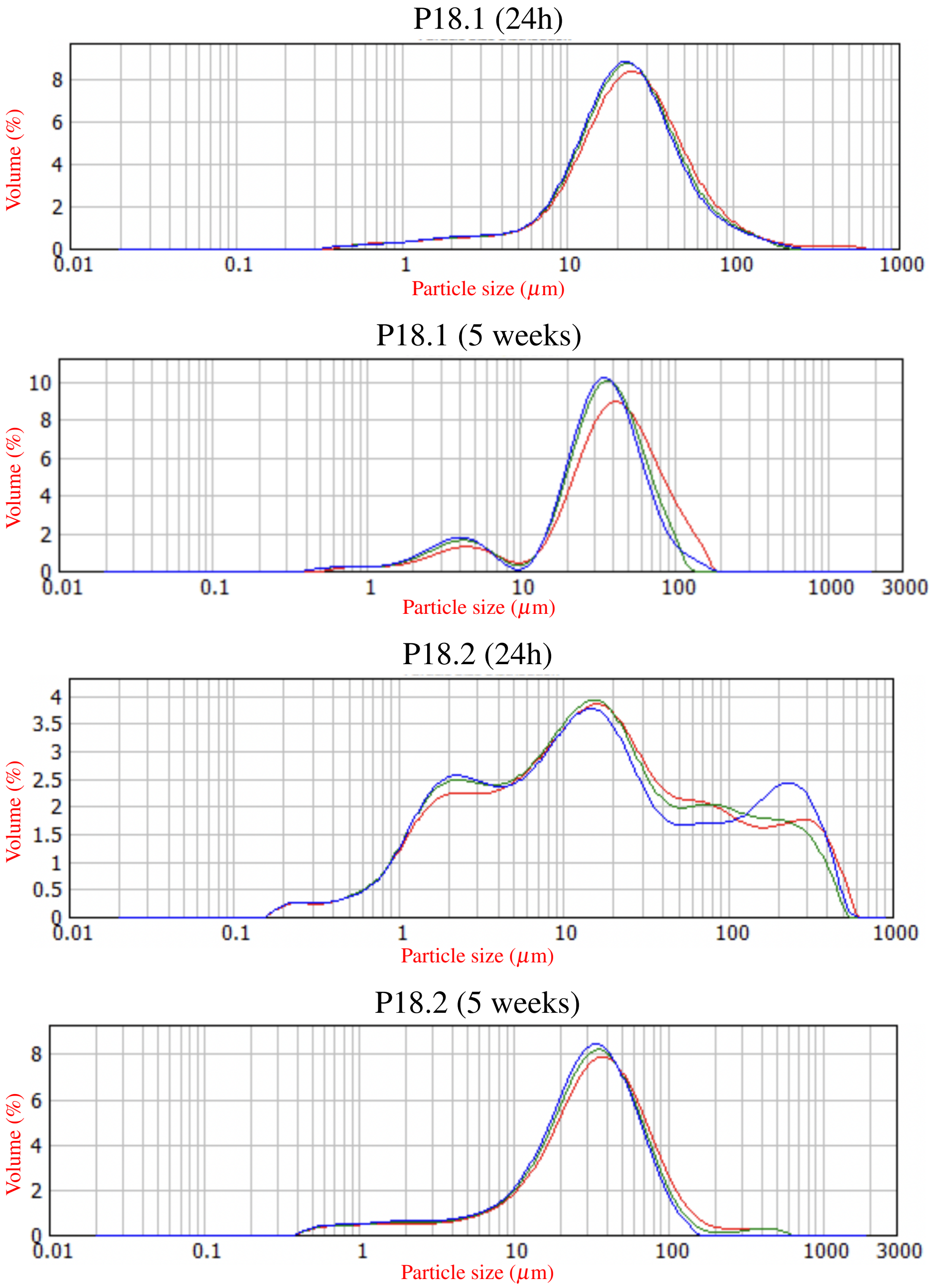


Figure 7.118: Particle size distributions of formulation P18. Red graph shows measurement 1, green graph measurement 2 and blue graph measurement 3.

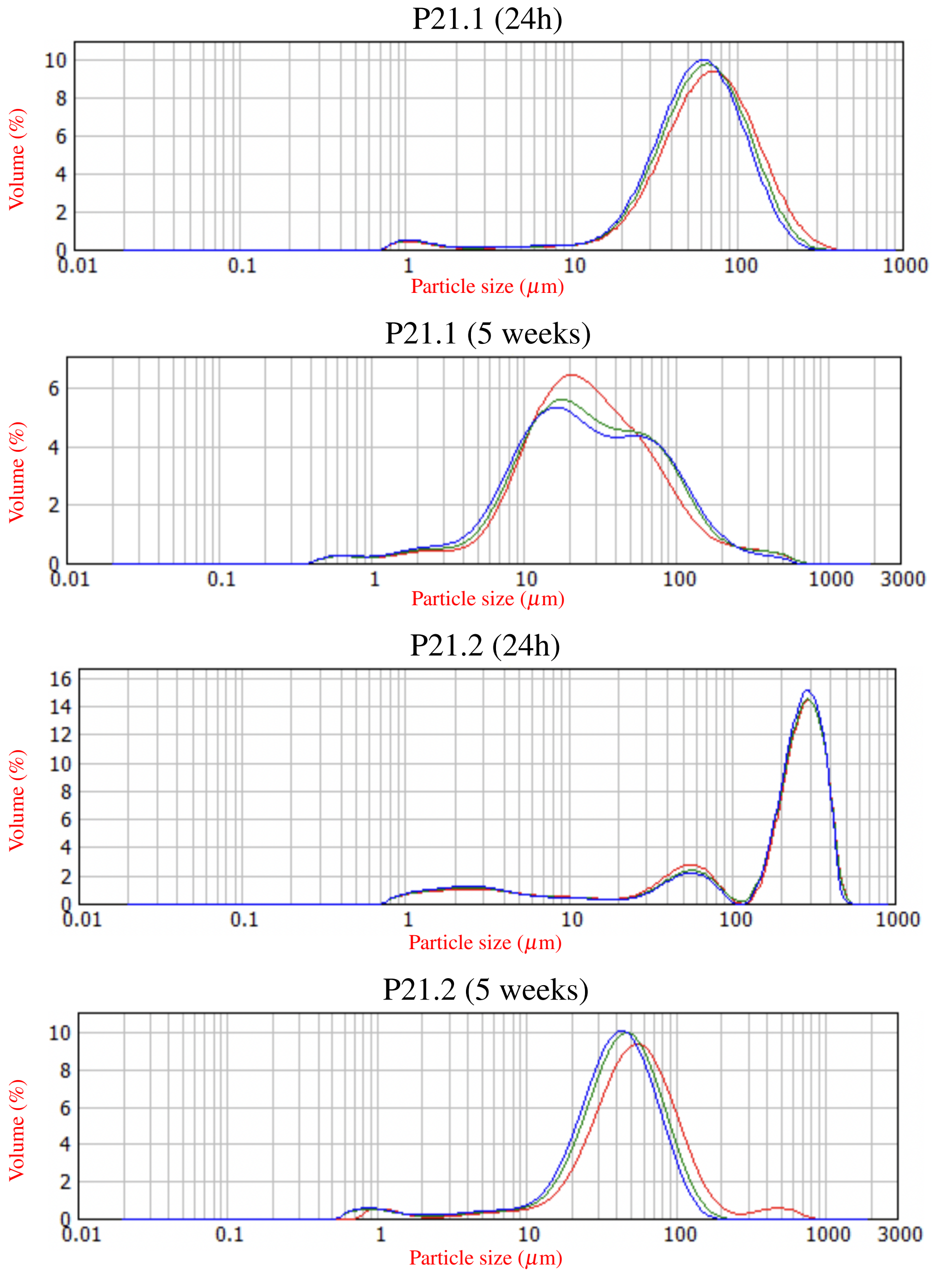


Figure 7.119: Particle size distributions of formulation P21. Red graph shows measurement 1, blue graph measurement 2 and green graph measurement 3.