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Development of an Innovative Handwashing Product – the Paper Syndet

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CASTELBEL
PORTO

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Abstract

Human civilisation has always shown the need to evolve, and so everything we create must also grow to meet that need. The skin care industry is included in this evolution, which has shown it by developing different kinds of products and the new benefits they bring to consumers and the environment – one of the main ones being the assurance of better health through cleanliness.

This is what the present work was focused on – offering an innovative handwashing product to respond to the society demands, while keeping the Castelbel ideals of an eco-friendly and luxury invention.

With that, the *Paper Soap* concept was born. The product had some key requirements such as an efficient and mild performance, while at the same time keeping its form and only dissolving when in contact with water. And so, the experimental conditions and the chosen ingredients required to develop a product were first tested at a laboratory scale. The work proceeded to the Polyvinyl Alcohol testing, determining which one of the samples performed better. This was followed by the Surfactant assessment of which would be the better replacement for Sodium Lauryl Sulfate used in the original formulation. The analysis of the results was preceded by a scale up to a pilot scale, highlighting the differences between the two stages, which would allow for the development of a final product with the characteristics acceptable for commercialisation.

Given the time limitation for the realisation of this dissertation, it was only possible to complete a few of the preliminary quality tests this type of product requires. The quality tests include the pH assessment and comparison to the skin pH; stability analysis in a controlled environment; and finally, in an effort to obtain a broader sense of how the product would be perceived, a small questionnaire to potential consumers was undertaken. Finally, everything needs packaging to be commercialised – an important detail in the cosmetic and personal care products industry - so some suggestions using eco-friendly packaging, keeping to the Castelbel brand identity, were presented.

As so, an eco-friendly and safe syndet with an efficient but mild cleaning action forming a pleasant lather, in an innovative, easy to use and portable format, was produced.

Keywords (theme): Paper syndet; innovative syndet; eco-friendly; product design.

Resumo

A civilização humana sempre demonstrou a necessidade de evoluir e, portanto, tudo o que cria tem a necessidade de desenvolver-se para cobrir essa necessidade. A indústria de sabão e de *syndets* está incluída nesta evolução. É possível comprová-lo pelo desenvolvimento de diferentes tipos de produtos e de novos benefícios para o consumidor e ambiente – um dos principais sendo o de assegurar uma melhor limpeza.

E é neste contexto que a presente dissertação se foca – na contribuição de um produto inovador de limpeza de mãos que responde aos pedidos da sociedade, mas sempre mantendo os ideais da Castelbel de criar uma invenção que seja *eco-friendly*, mas com um toque de luxo.

Neste trabalho, o conceito do Sabão de papel foi desenvolvido. Começou por se testar diferentes condições experimentais à escala laboratoial, bem como os compostos necessários para o desenvolvimento de um produto que apresente uma lavagem eficiente, mas suave na pele, mas que mantenha a sua forma e estrutura, só se dissolvendo quando em contacto com a água. Procedeu-se à testagem ao Polivinil álcool, determinando qual das amostras apresentava um melhor desempenho e, logo a seguir, à testagem de qual dos surfactantes seria o melhor substituto do Lauril sulfato de sódio, usado na formulação original. Após análise dos resultados precede-se um *scale-up* para a escala piloto, permitindo a produção de um produto final com propriedades aceitáveis para a sua comercialização.

Atendendo à limitação de tempo para a realização desta dissertação, foram realizados apenas alguns dos testes de qualidade necessários realizar a este tipo de produtos. Os testes de qualidade realizados incluíam a determinação do pH do produto que deve ser compatível pH da pele; análise de estabilidade do produto num ambiente controlado; e por fim, de forma a concluir sobre a aceitação do produto (papel de *syndet*), realizou-se um pequeno questionário a um pequeno grupo de potencias consumidores. Por fim, como a embalagem, na área da cosmética, é um requisito muito importante para a comercialização d qualquer produto, foram propostas algumas sugestões de embalagens *eco-friendly* e dentro da imagem de marca da Castelbel são apresentadas.

Assim sendo, foi possível obter um *syndet eco-friendly* com uma ação de limpeza eficiente, mas suave na pele, formando uma espuma agradável, num formato inovador, fácil de utilizar e portátil.

Palavras chave: Sabonete de papel; *syndet* inovador; *eco-friendly*; design de produto.

Declaration

I hereby declare, under word of honour, that this work is original and that all non-original contributions are indicated, and due reference is given to the author and source

Porto, 08 de fevereiro de 2021

(Maria Estanque Brogueira)

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Notation and Glossary

A_u	Area of the paper sheet	cm^2
A_s	Area of the unity of product	cm^2
E	Mass percentage of oxyethylene	%
h	Height	cm
l	Length	cm
w	Width	cm
ω	Rotational speed	rpm

List of Acronyms

CMC	Critical Micelle Concentration
DEA	Diethanolamine
EDTA	Ethylenediaminetetraacetic Acid
PVA	Polyvinyl Alcohol
SC	Stratum Corneum
SCI	Sodium Cocoyl Isethionate
HLB	Hydrophilic Lipophilic Balance
SLS	Sodium Lauryl Sulfate

1 Introduction

1.1 Framing and presentation of the work

In a society where the need to evolve is always present, brands must match the rhythm of change. In the light of recent world events, there is a more apparent concern and need to have clean hands, making the consumer look for easier-to-use products, while never forgetting the innovative, high-quality and aesthetic factors. From this, Castelbel, a cosmetics company, began the process of developing a new product, creating the *paper syndet*, presented in this work. Syndets, the fusion of the words "synthetic" and "detergent", have a similar function to soaps – to clean. However, their formulations are quite different. Soap implies a saponification reaction, and syndets do not. Syndets are composed of synthetic surfactants and allow the addition of other components such as emollients and skin nutrients. These characteristics make syndets milder cleansers. The "paper" structure was attained with the help of a film-forming agent and a thickener, added to a standard liquid syndet formulation, followed by the use of a specific drying machine, the JF – 600 Drum Dryers, to produce the final product.

The aim of this work was to develop a paper syndet, making it ready for market, while at the same time furthering the current knowledge of these types of products. There are not many studies regarding this sort of innovative formulation. Firstly, the formulation was assessed and altered in a laboratory scale, mimicking the drying process through two methodologies. Then, the formulation was tested in a pilot-scale production. With no previous information regarding the equipment, the operational parameters had to be defined. Various parameters such as texture, structure, performance, lather, and mildness were assessed throughout the assays, until the product matched the standard. Afterwards, some preliminary evaluations were performed, such as quality tests, to build a data base regarding the products quality and the assessment of the product by possible consumers, through a questionnaire. Finally, having collected the data needed, it was possible to propose packaging and estimate pricing values.

1.2 Presentation of the company

Castelbel is a 100 % Portuguese held company, founded in 1999, which specializes in manufacturing luxury fragranced products, both for the home and body. The name of the brand comes from a fusion between the words “Castêlo” – where the company was founded - and “Beleza” – what the brand wants the customer to feel while using their products.

It started with only one customer from one country and one type of product. Now, besides selling hundreds of products with an equal variety to Portugal, its home country, Castelbel is also selling to 55 different countries - an export rate of 80 % instead of the original 100%. As of 2019, 6.5 million soaps have been produced, which in combination with the rest of the products sold, amounts to 13.6 million euros in turnover. This number has been growing since the foundation of the company. In terms of product variety, the Company ranges from soaps to candles, hand and body creams to solid shampoo. Every single product has its own scent and carefully thought-out packaging. Keeping the motto "affordable luxury" while maintaining a balance between old traditions and new experience, mixed with the dedication that is put into the creation of the new product and the attention that is put into its details, being it the scent, the colour of the wrap or of how the bow is tied, is what sets Castelbel apart in this market.

1.3 Contribution of the author to the work

Going from the challenge set by Castelbel, based on the idea of a new handwashing product, it was possible to start developing the product design. After having idealised the product it was necessary to assess possible formulations and to perform the assays leading to the best product. Then a scale up of the process was performed, to obtain an almost finalised product. Finally, preliminary quality tests and the assessment of the satisfaction by possible consumers, with a questionnaire, were performed in order to suggest some packaging options. This work is meant to develop a paper syndet product, assess it, and ready it for the market.

1.4 Organisation of the thesis

The development of an innovative handwashing product, denominated paper syndet, will be assessed in different conditions. The first chapter identifies the issue the new product is meant to solve, as well as the main goals of the work. The second section is dedicated to a brief introduction of cosmetics and syndets, the product design, the function of some components presented in the work and the legislation to which these products are subjected. The formulation and the required alterations, along with two different production methodologies and the equipment used throughout the work are explained in chapter three. The methodologies for quality, pH and quantitative assessment are also described. The fourth chapter analyses the different assays, in various stages and conditions, showcasing the suggested final product. Afterwards, the discussion correspondent conclusions will be described in chapter five. Finally, in chapter six, the fulfilment of the initially proposed goals is evaluated, with a future perspective presentation to be developed.

2 Context and State of the art

2.1 Development of products and technologies throughout time

Cosmetics are nothing new to the world, they are something as ancient as the grand civilisations we hear about in history, such as the Egyptians, Greeks and Romans (Chaudhri & Jain, 2009). The word itself is derived from both the ancient Greek "*kosm tikos*" and the Latin from ancient Rome "*cosmetae*" (Chaudhri & Jain, 2009; Halla et al., 2018; Peet, 1952). Eventually they developed into different cosmetics, with different proposes. A more updated and official definition of cosmetics, given by the Council of European Union in 2009 " *'cosmetic product' means any substance or mixture intended to be placed in contact with the external parts of the human body … with a view exclusively or mainly to cleaning them, perfuming them, changing their appearance, protecting them, keeping them in good condition or correcting body odours*" (Couteau & Coiffard, 2010). Cosmetics can fall into a few classifying categories, according to the fields and duration of application, use, function, target consumer and form of preparation, which is one of the most important categories (Guerra et al., 2018; Halla et al., 2018). From those different categories stated by Halla et al. (2018), this work will be focusing on the first category of cosmetics for personal cleansing, mainly regarding soaps and detergents.

The most easily identified product of this category is soap. The process of saponification was only published in 1775 and only then became known to the public (Abbas et al., 2004; Peet, 1952). Soap, in its pure nature, is a natural surfactant, a tensioactive agent, which primal function is to clean and to produce foam (Abbas et al., 2004). Soap production is done through the alkaline hydrolysis of glycerides, designated by *saponification*, meaning oils/fats are transformed into glycerine and soap. While several saponification reactions exist, the end product is the same, however they will depend on the side chain (R) (Abbas et al., 2004; Contarini & Waldman, 2008; George & Raymond, 2016). One example is presented in Figure 1.

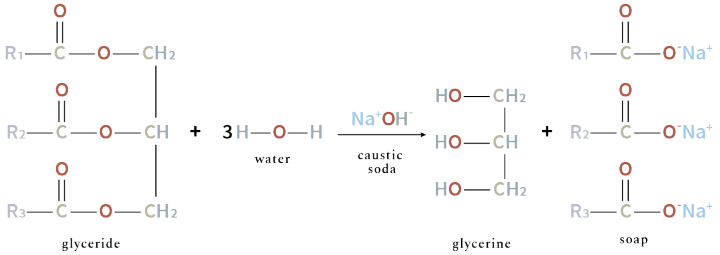


Figure 1 - Reaction of saponification.

The reaction presupposes the breaking of bonds in the triglyceride molecule through the alkaline hydrolysis, which will form four molecules, one alcohol (glycerine) and three

of soap (the sodium salts and the fatty acids). The type of fat/oil chosen, and the length of its carbon chain determine the different properties of the soap that is attained – reactions involving sodium hydroxide originate harder soaps and those using potassium hydroxide create softer soaps (Contarini & Waldman, 2008).

Soap is clearly a synonym of cleanliness. However, cleansers and their properties (both chemical and physical) have improved through the times (Hollstein & Spitz, 1982; Peet, 1952). Cleansers were created to remove dirt, sweat and oily materials from the skin, but in a way that is just limited to the removal of unwelcomed materials and to the minimisation of damage to the skin (Ananthapadmanabhan et al., 2004, 2019).

However, consumers were no longer satisfied by the simple cleansing action of these products. As so, there was a rise in the need for more beneficial products. Benefits such as pleasant fragrances, sustainable ingredients (when possible), antibacterial action and moisturising benefits, while also becoming gentler and milder cleansers (Abbas et al., 2004; George & Raymond, 2016). There was nothing on the market that met the requirements, so there was the need to create a different product, one that offered the possibility of change. The answer to this gap, emerged with the development of the *syndet* – synthetic detergent – which presents (mostly) no soap in their composition, but synthetic surfactants (Ananthapadmanabhan et al., 2019; Solodkin et al., 2006; Zoller, 2008). This invention is the basis for most of the personal care and even laundry products, amounting to a total of 60 % of the global production (Coiffard & Couteau, 2020).

Soap vs Syndets

Soap and syndets are utilised for the same end, despite all of their differences in formulation, performance and even appearance. Besides these two options, there is also their combination, named Combo bars, with a 10% of soap base (Zoller, 2008). To better understand the differences between the three types of cleansers, their characteristics will be presented in the Table A.1 in Annex A. It was possible to deduce that the milder cleansing action of syndets was the reason for their growth, even if they cost more to produce. Also, syndets do not damage the skin as much as soaps, due to the similarity with the skin pH and to added components such as emollients or skin nutrients, creating added benefits and value (Abbas et al., 2004; Ananthapadmanabhan et al., 2004, 2019; Solodkin et al., 2006). However, the soap cleansers are still a strong contestant, being easier and more economic to manufacture (Hollstein & Spitz, 1982). For reference and comparison, the remaining types of cleanser can be located in Annex A – Cleansers Formulation.

2.1.2 Syndets

Syndets can be found in two main different physical forms: bar or liquid. Liquid syndets were one of the evolutionary steps on the cleansers' history and were considered a ground-breaking development in the industry, during the 1940s (Ananthapadmanabhan et al., 2004; Draelos, 2018; Nix, 2000). Even though bar cleansers are still widely used, liquids are promptly gaining popularity for two main reasons: *convenience of use* and *control of infections* (Draelos, 2018; Lai, 2006; Nix, 2000; Qing, 2011).

As much as syndets were an evolutionary step in the cleanser industry they are not perfect. A key fact to remember is that syndets can still be irritating to the skin, even with their improved mildness (Abbas et al., 2004). However, it is possible to adjust and alter the formulation, adapting and improving it. There are 3 strategies that can be applied to achieve a mild product – minimise the damage made to the skin, compensate for the damage in case it cannot be eliminated and provide extra positive benefits (Ananthapadmanabhan et al., 2019). These strategies and their aftereffects are presented in Figure 2.

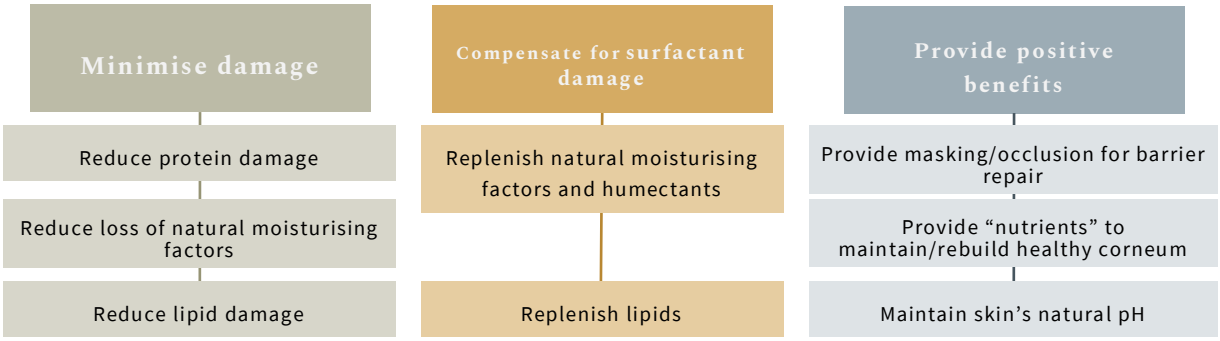


Figure 2 - Strategies to obtain a mild and moisturising cleanser and their aftereffects adapted from (Ananthapadmanabhan et al., 2019).

2.1.3 The need to evolve – the creation of paper syndet

Worldwide consumers are evermore focused on health and beauty and the related benefits (Carvalho et al., 2016). The progression through time can be translated into the evolution of 5 factors that became a requisite for the consumers: *Basic cleansing*; *Improved cleansing conditions,* marked by the development of more efficient surfactants; *Safety for the user and the environment*, the use of skin compatibility, safety and eco-friendliness products were starting to be considered by both consumers and developers alike; *Mildness*, users often chose a product with a milder action; *Additional benefits,* enhanced cosmetic benefits by the work of additives (S. Showell, 2006). This evolution of products was set in motion by the change of requirements and need, which has given the consumer a certain power over decisions regarding products, such as how they look and feel and how much they cost (S. Showell, 2006; Yao et al., 2019).

Eco-friendly, natural and environmentally safe products are much preferred, which adds an extra preoccupation to the developers (George & Raymond, 2016; Yao et al., 2019). So, manufacturers have to keep creating to fulfil the requirements. In order to meet the challenging requirements of creating an innovative cleansing product, these, Figure 3, are the factors the developer should take into consideration (S. Showell, 2006). The **paper syndet**, a cleanser with an innovative format, was developed due to the need of evolution.

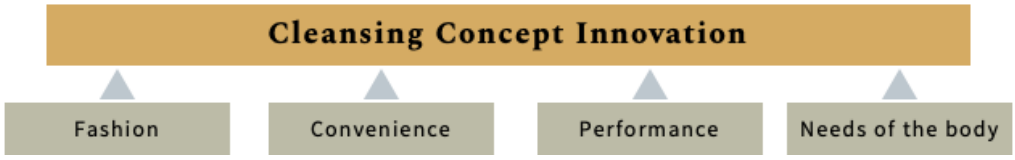


Figure 3 - Factors considered to create an innovative cleansing product adapted from (S. Showell, 2006).

2.2 Product design

This subchapter represents the idea that the creation of a proper product has to answer to a balance between the needs of the consumer, manufacturer and market. (S. Showell, 2006). Product design is the fastest and most efficient way to create a product based on that balance, which conveys input from marketing, research, sale, production and engineering all in cohesion (Cussler & Moggridge, 2012). This process is defined by four phases, all of them answering to a specific question the company and the developers should make, which will be represented in the following Figure 4 (Cussler & Moggridge, 2012):

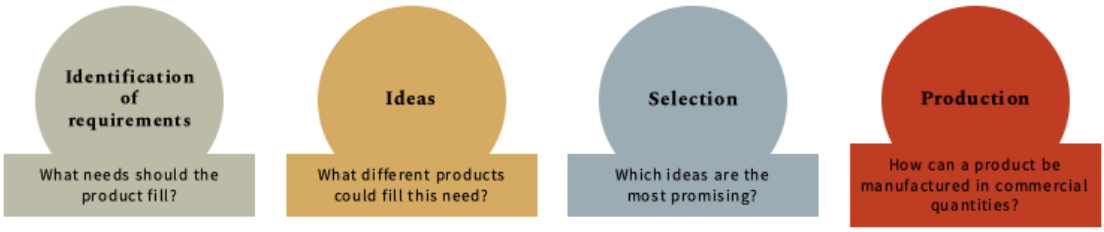


Figure 4 - Phases of product design and its questions adapted from (Cussler & Moggridge, 2012).

Creating a product based on the process of product design follows a seven-step process presented in Figure 5.

Figure 5 - Process of product design adapted from (Cussler & Moggridge, 2012).

Paper-syndet design

Having identified how the process of product design operates, it is possible to apply it to the product being developed – the **paper syndet**.

During the search to create a new product Castelbel took into consideration the previous factors (as seen in Figure 3) and it was then decided to focus on *Convenience*, feeling that this was what the market lacked, which required a solution – a convenient hand washing product. Even though liquid syndets are considered the most popular form for personal care products, they still have disadvantages. Some of those issues relate to packaging, difficulties in storage and transportation, added shipping and storage costs and also problems in terms of convenience; it is a harder to control the dosage of the product (Robert et al., 2014). With the intention of solving this prominent problem the new product had to bring more than just convenience. The product had to be mild to the skin, leaving the consumer with the feeling of a luxury product, while presenting an affordable price and keeping the brand identity, plus it should be eco-friendly if possible and safe to use (Lai, 2006; Yao et al., 2019) . The paper syndet is not a commonly used format. However, it might solve the identified problem, being cost efficient, portable, effective and an easy to use product (Qing, 2011). Recurring to the literature about the needs for a good cleanser, found in Lai (2006), and to the prior information Castelbel had assembled, it was possible to assemble the following list of requirements (Table 21). Without the presence of these requirements the product could not be considered valid.

Table 1 - Consumer requirements identified for the new Castelbel product.

Effective cleanser	Mildness
Competitive cost	Appealing aesthetics
Portable	Skin care benefits and post use feeling
Environmentally friendly	Skin compatibility
Easy to use	Safe
Sufficient lather	High quality

To create an easier task of evaluating the needs, they were divided into the following categories: *product performance*, *product perception*, *commercialisation* and *legislation*. The requirements and respective metrics and ranking are presented in the Tables A.1-4 in Appendix A, followed, in the same Annex, by the respective explanation.

2.3 Function of chosen components

The main components for the developed product during this work will be presented in more detail in further chapters. However, they fall under the categories such as surfactants, thickeners, moisturisers, solvents, film-forming agents, fragrances and colouring agents. A short introduction and explanation of these categories will be given in this subchapter.

2.3.1 Surfactants

Surfactants, or **surf**ace-**act**ive **agents**, are considered one of the most versatile products in the chemical industry, appearing in a considerable variation of products. These are components capable of adsorbing onto surfaces of a system and altering the free energy of said surfaces. This is referred to as modifying the interfacial tension (reduced) (Cullum, 1994; Rosen & Kunjappu, 2012; S. Showell, 2006). Surfactant molecules present two prominent parts: one lyophilic and another lyophobic, represented in Figure 6. In each case, the solvent is either water or oil, changing the prefix to "hydro" and to "lipo", respectively (Rosen & Kunjappu, 2012). The lyophobic part is represented by a hydrocarbon chain, with an average of 12-18 carbons, which could include an aromatic ring (Cullum, 1994).

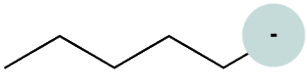
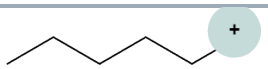
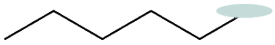
Figure 6 – Structure of a surfactant adapted from (Rosen & Kunjappu, 2012)

Surfactants can be divided into two classes – natural and synthetic (Abbas et al., 2004). An equally important aspect of surfactant characterisation is their division according to their hydrophilic part or polar group (Cullum, 1994; Rosen & Kunjappu, 2012). Some common examples and the correspondent chemical structure are presented **in Table 2**.

The anionic polar group bear a negative charge. Anionic surfactants are known for producing lather (white foam produced by detergents and soaps). They can be used in soaps, hair, or skincare detergents and in toothpastes. **The cationic** polar group bear a positive charge. As so, these surfactants are known to adsorb strongly to solid surfaces, but are usually more expensive than anionic or non-ionic surfactants and show poor detergency (to enhance the cleaning power of a liquid) (Rosen & Kunjappu, 2012). They can be found in hair conditioners, softeners for textiles and herbicides. The **non-ionic** hydrophilic part is uncharged, making them compatible with the remaining categories of surfactants and can be made resistant to hard water. They do not produce much foam, and can even be used as antifoaming agents (Chang, 2016; Rosen & Kunjappu, 2012). As so, non-ionic surfactants are used for laundry detergents, cleaning agents, cosmetics and even herbicides (Cretier et al., 2000). The **amphoteric/zwitterionic** hydrophilic part represents a balance between both a positive and a negative charge, which makes them compatible with the remaining

surfactants. Skin and eye irritation are less apparent in a product that uses amphoteric surfactants.

Table 2 - Surfactants commonly used in liquid hand soap and their correspondent chemical structure adapted from (Friedman, 2016; Lai, 2006; S. Showell, 2006)

Type of surfactant	Example	Schematic representation
Anionic	Sodium lauryl sulfate (SLS)	
	Sodium lauryl ether sulfate (SLES)	
	Sodium cocoyl isethionate (SCI)	
	Sodium lauryl sarcosinate (sarkosyl)	
Cationic	Quaternary monoalkylammonium chloride	
Non-ionic	Alkyl polyglycoside	
Amphoteric	Cocoamidopropyl betaine	

Some factors should be noted to compare surfactants of the same group,: *the hydrophobic group length* may affect the *solubility* of the surfactant in organic solvents, it can increase the melting point of the surfactant and the tendency to aggregate (micelles); *branching* may decrease the biodegradability and *unsaturation* may affect the oxidation, the colour of unsaturated compounds, both may even increase thermal instability and solubility of the surfactant in water or organic solvents; the presence of an *aromatic nucleus* decreases the biodegradability, while increasing the *adsorption* to polar surfaces (Rosen & Kunjappu, 2012).

Besides the general division according to the charge of the hydrophilic group, the level of this group will influence the action of the surfactant and its purpose. The strongest hydrophilic polar group is sulfate ($-SO_4^-$) and the weakest one is the hydroxyl group (OH). The stronger hydrophilic groups can be applied for washing, usually, as detergents. On the other hand, the weaker groups can be antifoaming agents (Chang, 2016; Lai, 2006). The position on the hydrophilic chain of the polar group also matters. If it is positioned on either of the ends, the surfactant will be a strong detergent, otherwise the surfactant will be a good wetting and penetrating agent, as is stated in Chang (2016).

Ionic surfactants are commonly hydrophilic surfactants, but non-ionic surfactants can be either hydro or lipophilic, depending on the balance of the strength. The relative balance between the hydrophilic-lipophilic parts, is usually designated with HLB, which indicates the capability to attract water of the hydrophilic group and oil in the case of the lipophilic group (Nakama, 2017). There are many different paths to calculate the HLB, most created

by Griffin in 1949 (Nakama, 2017; Pasquali et al., 2008). The HLB can be determined by recurring to Equation 1. It regards polyoxyethylene alkyl ethers and polyoxyethylene esters, considering the ethylene oxide chains as the hydrophilic group, using its mass percentage (E) and dividing it by 5 to obtain the HLB (Pasquali et al., 2008).

$$HLB = \frac{E}{5} \quad (1)$$

HLB is merely an indicator, as so it may be used as a reference intended to determine which surfactant is better to different applications (but it is not a "bulletproof" method when creating a formulation). In Table 3, the intervals of the HLB value, the solubility in water and respective applications for non-ionic surfactants are presented.

Table 3 - HLB values interval and respective water solubility and application.

HLB value	Solubility in water	Applications
0 - 2	Non-soluble	Antifoam agent
3 - 6	Less dispersible	Emulsifier W/O
7 - 9	Dispersible	Wetting agents
8 - 18	Dispersible	Emulsifiers O/W
13 - 15	Soluble	Detergents
15 - 18	Soluble	Solubilising

Surfactants are usually added to solutions. How they interact with the surfaces (while mixed) depends on a particular point in the concentration in the solvent. The surface becomes saturated, creating a monolayer, which then makes the molecules to start to create agglomerates. These agglomerates form a molecular cluster named micelle (shown in Figure 7.b) and occurs due to the hydrophobic properties of the hydrocarbon chains. The concentration in which the micelles begin to form is known as the critical micelle concentration (CMC). When the concentration of surfactant reaches the CMC point the surface tension stops decreasing (Chang, 2016; Nakama, 2017).

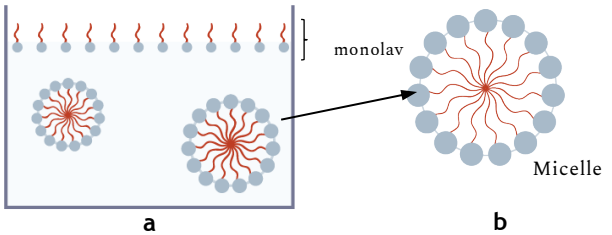


Figure 7 – a- formation of micelles; b- micelle structure. Adapted from (Chang, 2016)

Interaction with the skin

The skin is a complex structure, with an external part denominated stratum corneum (SC), with the main objective to protect humans from environmental aggression. The protection is only efficient when the SC, a hydrolipidic film, is properly moisturised (Barel et al., 2009; Casanova & Santos, 2016). Now considering the surfactants used in syndets, as

previously stated, are designed to remove dirt and other impurities to clean the skin (Ananthapadmanabhan et al., 2004), it is possible to observe that the surfactants can interact with the skin using different mechanisms. The surfactants interact with the skin by binding themselves to the proteins on the SC and then denaturing the proteins, solubilising or disorganising the intercellular lipids, penetrating the epidermal lipid barrier and, lastly, by interacting with the living cells. All these methods can be irritating to the skin, however the interaction of the surfactants with the skin proteins and lipids is the one that could be damaging to the skin. By binding themselves to the skin proteins, the surfactants will reduce the protein ability to hold water to the skin, lowering the levels of the skin hydration. The decrease of natural moisturising on the SC might lead to changes in the skin viscoelasticity, causing an after-wash tightness. In the long run, the continuous use of harsh cleansers can lead to dry skin, itch or barrier damage (Ananthapadmanabhan et al., 2004).

2.3.2 Thickener

These types of agents have an important influence not only on fundamental properties, but also on the aesthetics required by the consumers (Lai, 2006). Thickening materials are mainly used in industries that deal with aqueous-based products in need of an increased viscosity, to have easy handling or dispensing (Bhattacharyya, 1989; Lehmann et al., 2008). It is expected that thickening agents provide the targeted viscosity at the lowest quantity possible, in order to minimise the cost, yet having compatibility with the remaining ingredients (Bhattacharyya, 1989; Lai, 2006). The thickener should not be chosen without a proper analysis of the rheology or flow of the system, which, as stated in K. Lai (2006) “is intimately bound up the nature of the formulation, as well as its intended delivery system and use”.

The main way to classify these agents is in *organic* and *inorganic* thickeners. In terms of the inorganic thickeners, there are 2 major categories used in cosmetics and personal care products, salts and clays, also known as electrolytes. Organic thickeners are divided into natural thickeners (such as starch), modified natural thickeners (e.g.: propyl cellulose) and finally synthetic polymers (polyamides) (Lai, 2006; Lehmann et al., 2008).

2.3.3 Moisturisers

Moisturisers are an essential part of most skin-related products, since cleaning is not the only demand consumers have of these products (Lai, 2006). The main objective of the moisturiser is to increase the water content of the skin. Moisturisers are divided into two primary categories (Fluhr, 2020; Nolan & Marmur, 2012).

Emollients, hydrophobic agents, create an occlusive film on the surface of the skin, forming an obstacle to water loss, and thus reducing the evaporation of the water from the

SC. Their nature allows the prevention of dehydration, through the preservation of fat/grease on the skin, and the improvement of the skin's smoothness and softness. Emollients are components such as dimethicone (silicone) or essential fatty acids, such as linoleic acid. **Humectants** raise the water content of the skin by attracting more water molecules to the epidermis, both from layers under the skin and the environment. The capability to attract and hold water molecules derives from the hydrophilic groups, more specifically the hydroxyl groups. They are usually paired with occlusives (which prevent water loss). Some common humectants are hydroxy acids, propylene glycol and urea.

2.3.4 Solvent

Choosing what type of solvent to use depends on each formulation, the contemplated application and cost of production. Water is usually the preferred solvent for cleaning products, being non-toxic, with lower cost and better for the environment in terms of ecology (S. Showell, 2006). But obviously, water it is not always the ideal choice of solvent, being replaced by, for example, ethanol. Solvents have the goal to homogenize the solution and improve the performance of cleaning products, having a leading role in enhancing the power of the surfactants (Zoller, 2008).

2.3.5 Fragrances

Fragrances are a mixture of natural (animal or plants) and synthetic products that give an aroma to products (Fráter et al., 1998). The function of these components is entirely dedicated to creating and keeping the aesthetics of the product, but they also help to mask some unpleasant odours. It is one of the only added ingredients which has a percentage less or equal to 1 % (Lai, 2006; S. Showell, 2006). These characteristics, seeing as the scent can be a differentiating factor, improve the likeability and possible purchase of the product, propelling the success of the product (S. Showell, 2006). It should be noted that adding a fragrance to a product is no easy task since it can be: *unstable* due to pH or other reactive ingredients; *challenging to solubilise*, it can alter the viscosity or even create a phase separation; *hard to mask foulness* (if it exists) of the product due to its strength(S. Showell, 2006; Tchakalova & Fieber, 2012).

2.3.6 Colouring agents

Colouring agents are responsible for colouring the product or a body part where the product is applied. They are connected to the aesthetic of the product, heavily influencing the consumer, spiking interest and having a major role in the marketing of the product. Personal care products contain circa 0.01 to 0.3 % in colouring agents (Guerra et al., 2018; Lai, 2006).

There are several ways to characterise them by structure, source, colour, application, and solubility. The most common characterisation is by solubility, which divides colouring agents into dyes and pigments. *Dyes* are hydro and oil-soluble, being synthetic organic compounds or from natural sources, and can be found in both skincare care products and personal care products. *Pigments*, in the other hand, are insoluble and so they stain in their particulate form (Guerra et al., 2018; Weisz et al., 2018).

The favoured colouring agents tend to be synthetic dyes, due to their persisting properties, such as brightness, superior stability to pH, light and heat alterations, and to their lower production costs (Guerra et al., 2018). However, this perception is altering because consumers are starting to demand more eco-friendly and safe health products (Yusuf et al., 2017).

2.3.7 Other Components

There are other additives that can be introduced to the formulation depending on the end application or the need of the formulation itself. However, since they were not used during this work, there was no need for an extensive description. Some of these other components are preservatives, antioxidant and chelating agents and film-forming.

2.4 Legislation

In Europe, both soaps and syndets are considered cosmetics. These products, according to the European Union (EU) legislation, fall under the definition of Article 2 of Regulation (EC) No. 1223/2009. To comply with the definition, no claim of any healing or preventing characteristic of either human or animal diseases can be made, meaning the product cannot be considered a medicinal product. Additionally, according to the Portuguese legislation, cosmetic products have to follow the rules stated in Decree Law no. 296/98, 25 of September. This decree aims to regulate the production, commercialisation, labelling and marketing of cosmetic products. It also dictates that cosmetic products can be placed on the market without the need of prior authorization if the product has a correct and complete label. Identification of the production place, quality, and safety information regarding human health, of the product's objective and finally the designation of the components and ingredients in the product are all required factors.

3 Materials and Methods

3.1 Reagents

3.1.1 Surfactants

There are many types of applications for different surfactants, but sometimes also for the same surfactant. To produce the paper syndet, four surfactants were tested. All of the selected surfactants are commonly used in personal care products, specifically in liquid handwashing ones (Lai, 2006). The main surfactant had the function of cleanser, foaming agent and emulsifier, when the secondary surfactant should act as a lather booster and a co-emulsifier.

Sodium Lauryl Sulfate

TENSOPOL A794 - SLS, 93 % ($CH_3(CH_2)_{11}SO_4Na$, CAS 85586-07-8). Ordered from KLK Oleo. The first surfactant tested was the Sodium Lauryl Sulfate, known as *SLS* o SDS, commonly used in household cleaning products and in personal care products (Bondi et al., 2015; Lai, 2006). However, SLS has been classified as a harsher surfactant, sometimes damaging the skin barrier. However, from the literature it was possible to determine that it will not be harmful to humans or the environment if the product is properly formulated. Nonetheless, the decision to replace SLS was maintained. There are milder surfactants, which will not damage or dry the skin as much, even leaving the skin feel soft and silky. Plus, SLS has a somewhat negative reputation with consumers, due to some media misconception (Ananthapadmanabhan et al., 2019; Bondi et al., 2015).

Sodium Cocoyl Isethionate

Pureact I-80 P. Sodium Cocoyl Isethionate standard ($C_{14}H_{27}O_2SO_3Na$, CAS; 61789-31-0). Bought from Innospec. Sodium Cocoyl Isethionate, known as *SCI*, is a very mild and vegan anionic surfactant, derived from fatty acids of coconut oil and isethionic acid, that is used in syndet products. Besides the common function of surfactants it also presents soft emollient properties, soothing the skin and reducing possible irritation (Rieger & Rhein, 1997; Sun et al., 2003). In comparison to SLS, SCI is a much milder option, due to the fact that the micelles are unable to perturb the skin barrier (Ghosh & Blankschtein, 2007). That was one of the reasons it was chosen as a substitute. It is also known for its high, rich and velvety lather production that will not cause the skin do feel dry (dehydration) (Sun et al., 2003). It is a cost efficient product and is, also, compatible with the other co-surfactant and foaming agent - cocomide DEA (Masuda et al., 2004; Sun et al., 2003).

Sodium Lauroyl Sarcosinate

Two different samples were tested: Eversoft S-12D and Oramix L 30 ($C_{15}H_{28}NNaO_3$, CAS: 137-16-6), provided by RNM and Seppic, respectively. *Sarkosyl*, as it is also known, is an anionic surfactant derived from the amino acid sarcosine. It has all the good features of SLS, while adding a gentle action and soft moisturising action. As so, this surfactant is known for a soft but thorough clean and for giving a boosting shine to the surface it is applied to (Rieger & Rhein, 1997). It is a surfactant that is listed in the *Handbook of Green Chemicals* which means it is seen as a good to the environment surfactant in many ways (Ash & Ash, 2004).

The chemical structure of the surfactants is presented in the following Figure 8.

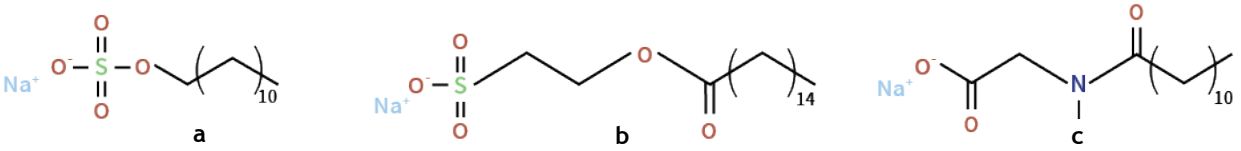


Figure 8 - Comparison of the chemical structure of the three tested surfactants. (a) SLS; (b) SCI; (c) Sarkosyl.

Cocamide DEA

Cocamide DEA standard ($C_{17}H_{35}O_3N$, CAS: 68603-42-9). Ordered from RNM. Cocamide DEA is a diethanolamide formed from the mixture of coconut oil fatty acids and diethanolamide. It is considered a common secondary surfactant which main function is to boost lather, however it will serve as a co-emulsifier (Lai, 2006).

3.1.2 Film forming

Polyvinyl Alcohol, partially saponified grades, (86.7-89.0 % hydrolysed, $(C_2H_3OH)_xOCOCH_3$, CAS: 9002-89-5), attained from Kuraray Poval. The elected film-forming was Polyvinyl Alcohol (PVA) which is a water-soluble synthetic polymer. It will act both as a film-forming and a co-emulsifier. It also gave the final product a more plastic structure (Mehta, 2018). Five different PVAs, from the same provider, were tested: PVA 4 - 88, 18 - 88, 22 - 88, 26 - 88 and 40 – 88. The first two digits correspond to the number of monomers (molar mass) and consequently the viscosity of the product. The last digits represent the hydrolysis degree of the ester molecule.

3.1.3 Moisturiser

Glycerine standard ($C_3H_9(OH)_3$, CAS: 56-81-5), provided by Consorima. Glycerine is a water soluble and hygroscopic glycol. This reagent was chosen due to its moisturising characteristics, a natural humectant, and because it would improve the skin's softness (Jungermann & Sonntag O.V, 1991). Glycerine has also been known to act as a preservative to the formulation, if its concentration is quite high (Barel et al., 2009).

3.1.4 Thickener

Maizena, Corn starch standard, ($C_{27}H_{37}O_9(OH)_{11}$ CAS: 9005-25-8), bought from Continente. Corn starch is a natural organic polymer, chosen as the thickener, viscosity controller. Starches have the ability to, when added to bath or hand washers, help restore the skin barrier, which helps create the feeling of skin on the after washing (Barel et al., 2009).

3.2 Operational Procedures for Paper Syndet Production

The development of the "paper syndet" is, as stated previously, not a commonly studied matter, which demands a carefully planned out course of action. The objective was to take the reference process and the formulation and develop an improved product, making it more appealing to the target consumer. The work was divided into three main experimental stages - two at a laboratory scale and another at a pilot scale. The planned strategy is represented in Figure 9. The experimental stages were preceded by research regarding the components and product resorting to literature, to information given by the providers and the technical sheets of the components. There will be three main stages, which are consequently divided into smaller steps: 1. PVA: Selection of PVA and Adjustments 1; 2. Surfactant: Selection of the best surfactant and Adjustments 2; 3. Final Product: Pilot testing and possible Adjustments.

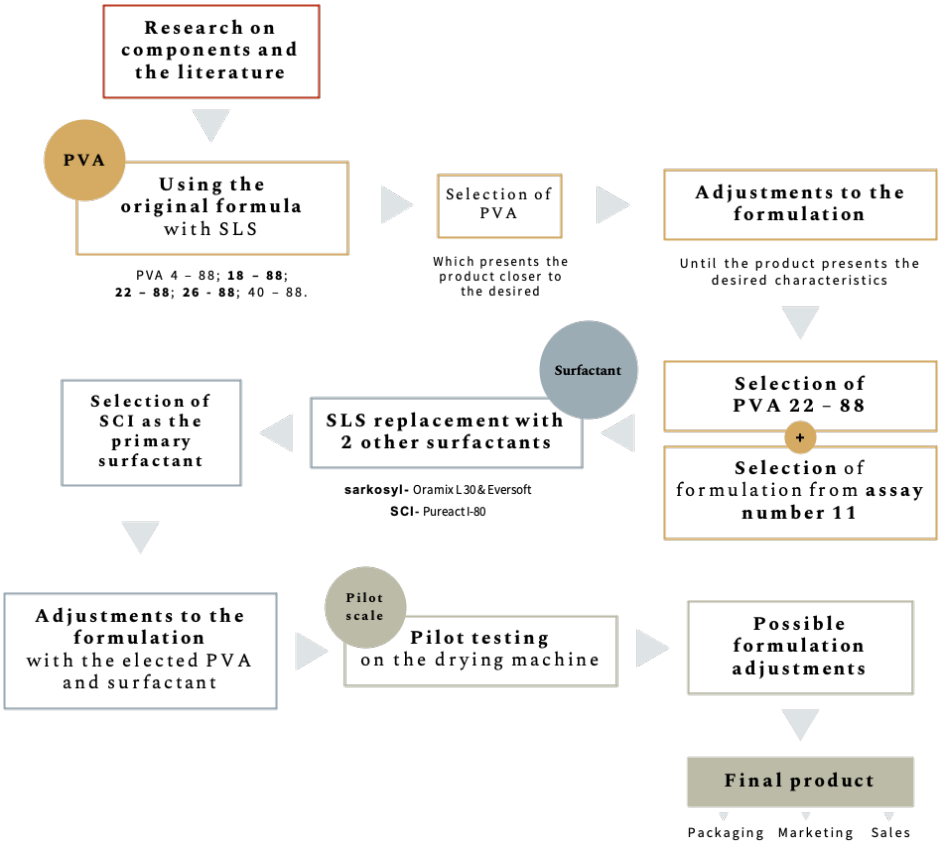


Figure 9 - Schematics of experimental stages of paper-soap production.

3.2.1 Generic Formulation

The general liquid soap formulation was thought to have served as the basis for the paper syndet. Nevertheless, the formulation lacked the components responsible for creating the desired "paper-like" structure, requiring additional components.

The formulation used in the present work (presented in Table 5) will be specifically based on the one sent by the provider (shown in Table C.1 in the annexe C.1 - Reference formulation). However, changes were applied during the laboratory work, in terms of the ingredient percentage, PVA and surfactant type. The specific alterations will be explained in chapter 4. Results and Discussion.

Table 4 – Used Generic formulation function, reagent and respective percentage.

Number	Function	Reagents	Percentage (%)
1	Solvent	Water	60
2	Film-forming	PVA	9
3	Moisturiser /Emollient	Glycerine	9
4	Surfactant	SLS, sarkosyl & SCI	8
5	Emulsifier	Cocamide DEA*	5.8
6	Thickener/filler	Corn starch	8
7	Fragrance	-	0.2
8	Pigment	-	as needed

*Cocamide diethanolamine can also be known as cocamide DEA for short.

It was possible to corroborate the list of ingredients given by the provider with two registered patents, one Chinese and one American. In the Chinese patent, CN101063066B, it was stated that "a kind of instant paper-shaped perfumed soap" had a membrane-forming agent, surfactants, softener, antiseptic and germicide agents, alcohol, and pigment, fragrance and water (Qing, 2011). Meanwhile in the American patent, with the number US 8765170 B2, it was specified that the personal care article in question had a surfactant (23 – 75%), a polymer soluble in water (10 – 50%), and optionally a plasticizer (1 – 15%) (Robert et al., 2014). The American patent also confirmed how the final product should look like a "flexible porous dissolvable solid structure" (Robert et al., 2014). There was the need to note the slightly distinctive samples of the film-forming agent and the three different surfactants tested by the author. The type of fragrance and the quantity and colour of pigments added varied. It was only added in an assorted way to simplify each assay's identification during the present work. However, Castelbel, in the future, will be able to control and change these characteristics.

3.2.2 Laboratory Scale Production

Method

The laboratory assays followed a slightly altered version from the original method (Annexe A.2. – Reference Laboratory Method) due to some adjustments to the equipment. The generalised production of the paper syndet is presented in the following Figure 10. Only the temperature of work would be shown since both the mixing speed and the time varied with the scale worked on.

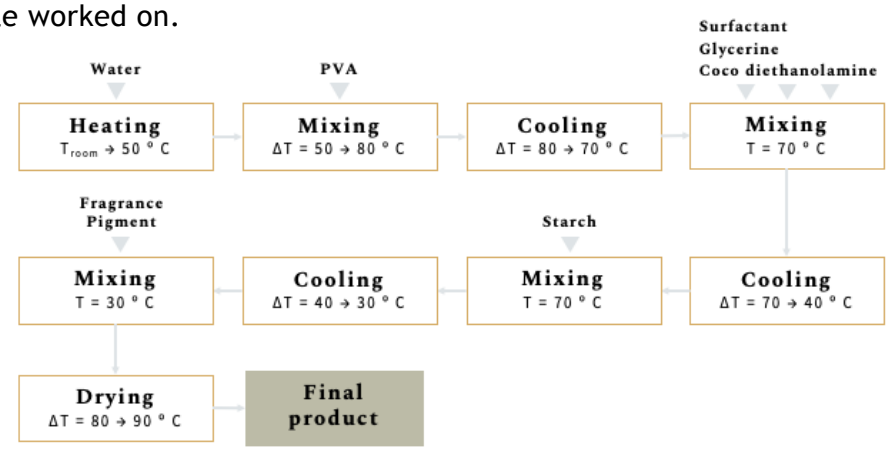


Figure 10 - Process diagram of production for paper soap.

Paper-syndet production began with the weighing of all ingredients on a scale with an uncertainty of ± 0.001. The next step was the heating – through a magnetic heating agitator (VELP Scientifica) - of water for about 5 minutes until it reached 50 ºC. At that point, the PVA was added, maintaining the heating and a continuous mixing at 100 rpm until the temperature reached 80 ºC. Having reached the determined temperature (never surpassing ± 5 ºC) the PVA would start to melt. This step would be considered completed when the mixture appeared transparent, and all the product had melted – taking about 30 to 45 minutes. The continuous mixing in this step was rather essential to speed up the process. Some of the used equipment is shown in Figure B.1 (Appendix B).

Following the PVA dissolution, the mixture needed to cool down to 70 ºC, and be kept at this temperature (tolerating a variation of ± 2 ºC) for the next steps. Then the addition of the chosen surfactant could begin. This step required some caution because the surfactants could cause some irritation to the respiratory tract, so a gradual addition to the mixture was recommended. If the assay used SLS, the product, which came in white ivory needles, should be turned to powder, using a ceramic mortar, for easier mixing. For the other two surfactants, there was no requirement for this step; one was in powder form, and the other presented a liquid appearance.

After mixing in the surfactant and the mixture presented a uniform appearance, the glycerine, and the coco diethanolamide could be added. The agitation, up until this point, was created by a magnetic agitator at a constant speed of 100 rpm.

The second cooling step represented the mixture cooling from 70 ºC to 40 ºC - only then could the corn starch be added. The magnet could be removed, since the mixture would be too thick for it to work correctly. From this point onward, the mixing would be done manually. To advance to the third cooling step (40 ºC to 30 ºC), the mixture should be soft and smooth, presenting an increase in viscosity. At 30 ºC, it would be safe to add both the fragrance and the pigments, without compromising the components and guaranteeing a homogenous blend. As previously stated, many different combinations of fragrance and pigments were added to promote the recognition of the assays. The entirety of these steps took about 80 minutes to complete.

The final step involved the drying of the product. Two methodologies were tested, presented in Figure B.2 – Annex B, to replicate the drying machine. The first one was through the heating plate, where the product was put in direct contact with it, similar to the workings of a pan, allowing the water to evaporate. The second method was performed through a lab oven (VWR Drying Oven). The first option, the drying process through the heating plate, never attained satisfactory results even though it was the swiftest option. These results were believed to be related to the direct contact with high temperatures, which was not gentle enough to create a gradual evaporation of the water. The second method proved more successful in terms of an effective final product, that appeared, felt and worked closer to what was expected. The product was placed in a silicone cast placed inside the oven, between 80 ºC to 90 ºC. This method was gentler and allowed the evaporation of the water to happen in a gradual and uniform way, without reaching the plasticising point of the PVA. The downside to this method was the extended waiting that was necessary - around 2 hours. The time varied with the quantity and the thickness of the mixture placed in the silicone case. The method presented can then be scaled up to bigger productions (excluding the drying step) and adapted to the available equipment shown in the next sub-sub chapter 3.2.2.

PVA Testing

The first section of the practical work involved using the general formulation (displayed in Table 4) with SLS, while experimenting with different PVAs, to define which one would appropriate. This decision was made based on the end result using SLS serving as a guide (reference product), as there was a lack of information regarding the PVA characteristics. The features of the different PVAs are presented in the following Table 6 and was handed over by the PVA provider.

This step was divided into smaller sections. It began with testing all the PVAs with the formulation established as the basis, then a selection of the best results. With the elected PVAs, it was possible to enforce alterations to attain the desired appearance, texture, and

efficiency. These alterations were grounded on the varying quantities of water, PVA and corn starch in the formulation, in an assortment of ways.

Table 5 - Characteristics of the distinct PVA of partially saponified grades.

PVA Classification	Viscosity (mPa·s)	Degree of hydrolysis (mol%)	pH
4 - 88	3.5 – 4.5	86.7 – 88.7	4.5 – 7.0
18 - 88	16.5 – 19.5	86.7 – 88.7	4.5 – 7.0
22 - 88	20.5 – 24.5	87.0 – 89.0	5.0 – 7.0
26 - 88	24.5 – 27.5	86.7 – 88.7	4.5 – 7.0
40 - 88	38.0 – 42.0	87.0 – 89.0	5.0 – 7.0

This step covered the assays 1 through 11. And it should be noted that the first method of drying in the laboratory was only used in 6 assays.

SLS: surfactant testing

The second stage of the work, in its roots, was quite similar to the first one. It had the objective of verifying which surfactant would be the better option and was divided into smaller segments: assays using the different surfactants, but with a formulation in common. Afterwards, there was the selection of the better option and subsequently, the alteration of the formulation, to establish the best choice for the product.

In this case, instead of the first formulation, the one used was based on the PVA testing results. The formulation that presented better results was elected. All three samples of the surfactants were tested, and then the alterations were only applied to the promising ones. The characteristics of the surfactants are on the following Table 6. All the chosen samples were biodegradable and environmentally friendly. For easier identification, from this point on, the surfactants tested will be designated by their shortened commercial name Pureact, Oramix and Eversoft. This stage comprehended the assay 12 to 18.

Table 6 - Characteristics of the surfactant samples.

Classification	Surfactant	Appearance	pH
Pureact I-80	SCI	Fine white powder	4.7 – 6.0
ORAMIX L 30	Sarkosyl	Light yellow liquid	8.0
Eversoft S-12D	Sarkosyl	Colourless liquid	8.0

3.2.3 Pilot Scale Production

Drying Machine

The pilot-scale stage was categorised by the scale-up of the process's dimensions and the use of a specific drying process. The drying process was carried out by a drying machine denominated JF – 600 Drum Dryers, which helped to create the soft and paper-like structure.

In the following Figure 13, both the machine in a stationary, dynamic state and the control panel are presented.

a b c

Figure 11 - Drying machine used in the work. (a) front view; (b) back view; (c) control panel.

The drying machine was composed of a motor, a control panel, five rollers (cylinders) working independently from each other and a feeding tray. There were three static rollers, which were named static 1, static 2 and static 3. The former helped create a smooth and uniform product layer and both the latter rollers helped to wind the product. There were also three dynamic ones, denominated as main, feeding, and winding rollers. The process of how the machine operates is demonstrated in the following schematics (Figure 12). The operation manual will be presented in Annex B - Operation manual.

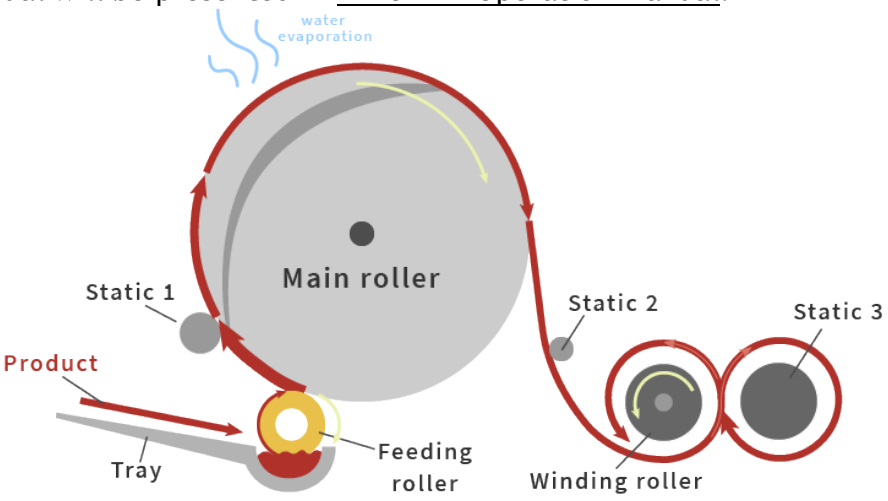


Figure 12 - Schematics of how the machine operates.

Method

The process for the pilot testing remained mostly the same as previously presented. Some of the equipment was replaced with similar options, but with a bigger capacity. Besides the drying step, the biggest differences were the time the process took (for example: to produce a mixture of 8 kg it took roughly three and a half hours) and the agitation of the mixture was fulfilled mostly manually. The use of a hand blender was

needed to achieve a smooth liquid mixture with no agglomerates. Some of the specific equipment used for the scale up process is presented in Figure B.3 (Appendix B).

For the drying step, a more industrial drying machine was used. The process began with turning on the device and then turning on the heating and setting the desired temperature. Afterwards, the rest of the green buttons were pressed, they were linked to the three dynamic rollers turning them on. Finally, having set the speeds, it was possible to press the remaining switches on and the button "RUN" (activate the main roller). Control panel showed in Figure 11.

The rollers' characteristics, temperature, and rotational speed (ω) could be adapted according to the formulation's requirement, depending on the type of ingredients and the liquid mixture's viscosity. However, the following conditions were the suggested ones by the provider: Temperature = 80 – 90 ºC; ω of feeding roller = 10 rpm; ω of winding roller = 20 rpm; ω of main roller = 60 rpm (maximum speed). Afterwards, the product was added to the feeding tray, which set the connection feeding roller to the main roller. The product should start to dry when it comes into contact with the heated main roller and would be ready to be removed when it had completed ¾ of the main roller's perimeter. At that point, with extreme caution, the tip of the product should be lifted and connected to the winding roller. This process will create various sheets that can be collected at the end of the process.

Pilot testing

For the pilot testing, with which the final product would be obtained, a smaller trial was performed during the laboratory stage. This trial test was done to comprehend how the drying machine operated. It was completed recurring to the information presented in the previous method. The conditions at which the main roller performed were at a controlled temperature of 82 ºC and rotational speed of 60 rpm. This assay was conducted using the formulation from assay 9. After completing the laboratory testing stage, a formulation was chosen to carry on the work to the pilot scale. The selected formulation was that of assay 18 (Table C.4). These final tests in the drying machine were completed using the same method as the trial test.

3.3 Quantitative Evaluation

The resulting products were quantitatively evaluated between each assay, regarding different parameters: liquid mixture, appearance, structure and performance. Each of the main parameters was divided into small attributes. This evaluation consisted in assigning each one of the attributes a value, by setting a scale of 0 to 10, and then giving the parameters an average value. The evaluation of the products' appearance was *subjective*.

It was determined in correlation to the sample sent by the provider (reference sample – assay 0) and what was thought to be the agreeable texture and structure of the final product. The parameters and attributes are presented in the following Table 7.

Table 7 - Parameters and respective metrics for the qualitative evaluation of the products.

Liquid Mixture	Performance
Viscosity: Measures the viscosity of the liquid mixture 0 – the mixture is too liquid (runny) 5 - the mixture is too thick 10 – the mixture has an ideal viscosity	*Aroma*: measures the intensity of fragrance the product leaves on the skin after being used. 0 – too intense; 10 – pleasant reminiscent scent
	Dissolution: measures the quality of the dissolution of the product when in contact with water. 0 – does not dissolve; 10 – dissolves completely and immediately
	Foaming duration: measures the time between the dissolution of the product and the creation of foam. 0 – a considerable amount of time: 10 – instantly
	Lather: measures the dispersion of the foam. 0 – no foam; 10 – pleasant foam quantity
	Mildness: measures the after feeling of the product on the skin of the user. 0 – dryness feeling (not mild); 10 – soft feeling (mild product)

Appearance	
Texture	**Structure**
Oil or Colour release: measures the release of colour and the presence of an oily residue derived from the fragrance (due to some error in the formulation). 0 – Release of either colour or oil; 10 – No release	*Elasticity*: determines if the product presents an elastic structure. 0 – inelastic (inflexible); 10 – flexible
Plastic finish: determines if a plastic layer was formed in the resulting product. 0 – completely plastic; 10 – no plastic layer	*Paper structure*: determines if the product has a plastic finish or a softer like paper one. 0 – completely plastic; 10 – paper like structure
Smoothness: determines the finish of the surface. 0 – porous surface; 10 – smooth surface	*Tear resistance*: determines how well a material can withstand the effects of tearing. [1] 0 – high resistance; 10 – medium resistance
Uniformity: measures the uniformity of the product's texture. 0 – different textures; 10 – uniform texture	

[1] In the terms of tear resistance, the 10 ranking characterizes the medium resistance, as in it will stay compact, however when tested it will give without too much use of force, similar to paper.

3.4 pH protocol

One of the most noteworthy characteristics of a cosmetic or personal care product is the compatibility with the skin pH. The natural pH of the SC (stratum corneum) is around 4.5 to 5.0. Therefore, the product has to present a similar or neutral value of pH to not disrupt the skin (Ananthapadmanabhan et al., 2019).

A pH meter with an electrode was used to monitor the pH of the final product. This procedure began with the calibration of the electrode with three different buffers – 4.01, 7 and 10.01. Afterwards, a dilution of 1 g of product in 100 g of water was performed (1:100 solution). With this solution, it was possible to complete three measurements and then the

final pH would be calculated through the average of the values. In this work, the pH was determined using two different solutions. The first methodology used the final dried product and dissolved it in water. On the other hand, the second one used the liquid mixture which was then mixed with water. From these two methodologies it would be possible to determine if the drying process altered the chemical properties of the product.

3.5 Product control

3.5.1 Stability control

Due to time limitation, it was not possible to conduct quality tests with the proper duration. However, it was decided to conduct a ten-day quality control appraisal to see what, or if, alterations would be visible. The performed tests included: Stability test under different *temperature intervals*; Stability test under a *high degree of humidity*; *Product assessment* by the target market, through a questionnaire.

Both the stability tests indicated how the product would react when in contact with different than ideal conditions. These tests would allow the developer to determine the packaging characteristics to prevent the product from altering its state, reproducing transport and storage conditions, but also environmental changes that could happen on a day-to-day basis.

The temperature intervals corresponded to 50 - 52 ºC high temperature test and 4.3 – 5.4 ºC (low temperature). For each of these tests only one sample was used. Alternatively, the humidity stability test examined three groups of samples placed in three large beakers: one single sample, three samples but separated by waxed paper and finally six samples all stacked together. Besides the initial observation of the product's condition, information was collected in two situations: after four days and by the end of the trial (ten days).

3.5.2 Product assessment

The product assessment was performed with the help of an online questionnaire, although one must try the product before answering any questions. The questionnaire was designed to present to the target consumer the idea of this innovative product and to see how the consumer perceives the characteristics of the product. Two samples were given to each consumer to test, one produced by the author of this thesis and the other one was the reference product used, which is already being commercialised. The consumer had no knowledge of which was which.

4 Results and discussion

4.1 Laboratory testing

In order to do a proper analysis of the resulting products from each assay, there were a few factors that were taken into consideration. These factors will be detailed to facilitate reading the results in Table 8. Exemplification of the products are presented in Figure 13.

Figure 13 – The resulting dried products of all the assays.

The first column of Table 8, following the assay number, presents the colouring chosen for each assay. This was done exclusively for rapid identification and had no impact on the performance. The second section in the table defines the various details that were altered throughout the work. During the initial stages, the focus was on the type of component being tested, so it was only logical that each assay was presented with the respective PVA and Surfactant. The drying process had a considerable impact on each assay's outcome, so it was important to distinguish which methodology was selected. The last column of this section was dedicated to formulation's changes, seeing as it had significant impact on the improvement. However, the variations do not encompass all the components used. It affects only the ratio of water, PVA and corn starch. This ratio allows the detection of small modifications in some of the products' parameters, such as the texture and the dissolution when in contact with water. Refer to the next subchapters for a more detailed analysis. The third section was dedicated to a set of parameters used for electing the "chosen" formulation. The parameters are the following:

- Structure and texture - *Appearance*;
- Product's dissolution with water, lather formation and mildness - *Performance*.

The appearance and performance were derivative from the quantitative assessment performed throughout the work. These parameters were given a metric, using a scale of 0 to 10, that is dependent on the specific features' evaluation. More details on this will be given in the section 4.1.3. Quantitative evaluation. The methodology has been explained previously, in subchapter 3.3. Quantitative Evaluation.

The products' performance should be acknowledged as the leading parameter to validate the resulting product, seconded by the close analysis of the structure and texture. The visual appearance of the product, despite altering the perception, did not compromise the product itself. Ergo there were various acceptable combinations that could be chosen. These results will be explained in the following subchapters.

Table 8 - Summarised description of the various assays done throughout the project.

Assay	Colour	PVA	Surfactant	Drying process	Percentage ratio[1] H_2O:PVA:Starch	Appearance		Performance[2]
						Texture	Structure	
0	Light blue	-	SLS	-	-	-		10
1	Orange	4 -88	SLS	Heating plate	59:9:8	0	0	0
2	-	40-88			59:9:8	-	-	-
3	Teal	18-88			59:9:8	2	1	0
4	Dark blue	26-88			59:9:8	2	1	0
5	Light purple	18-88			62:6:8	1	1	0
6	Yellow	18 -88			60:6:10	2	2	1 (starchy)
7	Light blue	26-88		Drying oven	62:6:8	3	3	3
8	Red	26-88			62:5:9	8	6	5 (starchy)
9	Light green	26-88			62.5:5:8.5	9	7	7
10	Coral	22-88			59:9:8	8	7	7
11	Blue	22-88			62.5:5:8.5	8	5	10
12	Lavender	22-88	Pureact	Drying oven	62.5:5:8.5	4	5	5
13	Red	22-88	Oramix		62.5:5:8.5	1	0	0
14	Mint green	22-88	Eversoft		62.5:5:8.5	1	0	0
15	Light yellow	22-88	Pureact		62:5:9	8	8	8 (starchy)
16	Sage green + pink	22-88			62.3:5:8.7[3]	9	8	9
17	Light blue	22-88			62.8:5.1:8.6[3]	7	7	10
18	**Pastel pink**	**22-88**	**Pureact**	Drying oven	**62.86:5:8.62[3]**	**10**	**10**	**10**

[1] in the alteration enforced in the formulation are all concerning the percentage of water, PVA and corn starch. The original ration is 59:9:8.

[2] the performance, or the capability of the product to dissolve when in contact with water.

[3] change in the percentage of fragrance in these assays.

4.1.1 PVA testing: stage 1

As it was stated previously, performance was the elected parameter to determine the efficiency of the formulation. The assessment associated with each assay is presented in the following Figure 14 (assay 1 through 11). The graphic should be read, for example, assay 10 had a performance (yellow) of 7, the product dissolved but not in an efficient way, and presented a texture of 6 and a structure of 5.

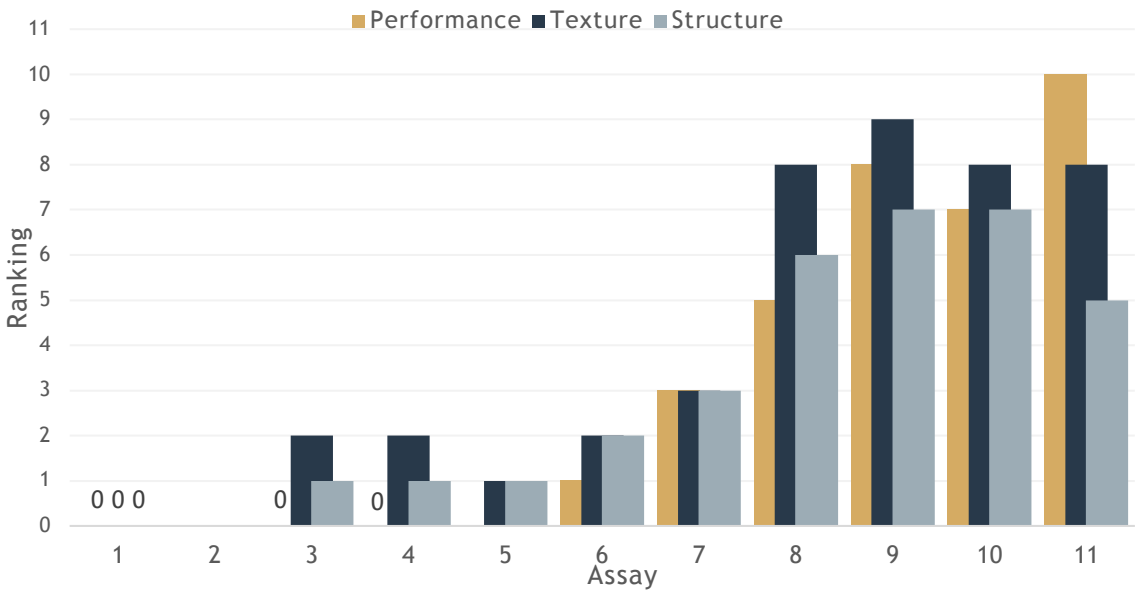


Figure 14 - Graphic representation of assay 1 through 11 evaluation.

Through a preliminary analysis of the data, it was possible to determine that the assays 1- 5 could be discarded instantly because of the non-existent performance. The same principle could be applied to assay 6 since it had a subpar performance. The results using the heating plate were not favourable, as a plastic layer had been formed over all the products, both in assays with the original or altered formulations. The cause of this was theorised to be the overheating of the surface in direct contact, enabling a reaction on the film-forming component (PVA) which has negative repercussions on the performance (prevents dissolution). This equipment could be assumed to be the wrong choice for the laboratory process. Several attempts to control the outcome, such as varying the temperature or altering the duration of contact, proved ineffective in improving the results.

Continuing the analysis of Figure 14, it was possible to observe on assay 7 to 9 the tangible improvements in performance and appearance. This enhancement was, initially, due to increasing the water quantity, while at the same time decreasing the percentage of PVA (a cut of 9 % to 6 %). In spite of improvements, the pre-identified issue with the plastic film, formed by the PVA, was still present at this stage (assay 7, presented two different surfaces), even if thinner. To try and eliminate this drawback, it was assumed that the reduction (-1 %) of PVA and the increment of water would be adequate. The lack of structure with this alteration was observed, so, it was further decided to alter the corn starch's percentage. This decision presented considerable progress in terms of appearance (soft and uniform texture with a solid texture). At the same time, it confirmed what had already been hypothesised in assay 6; that the corn starch's increment had its limitations. If the limit of what was thought to be 9 %, was surpassed, the product would not dissolve completely, forming a starchy mixture in the palm of the user (assay 8). The formulation of

assay 9 showcased an intermediate solution, in which the quantity of PVA was deflated, and the remnant percentage was divided between water and corn starch.

Interestingly, these experiments revealed that none of the PVAs used might have presented the ideal characteristics for producing a viable product. The supposition that the drying oven would be a valid option for the laboratory drying process, was further proved by these experiments. It was also determined that the quantity and thickness of the product placed in the cast influences the time it takes to dry completely. All the dried products of the assays are presented in Figure 15.

Figure 15 – Photography of the resulting products of assay 1 through 9.

Electing to use the same alterations proven efficient in previous trials, the new PVA sample (22 – 88) was tested in assays 10 and 11. Assay 11 presented a maximum in terms of performance, pointing to the strong probability of the PVA 22 – 88 as the ideal choice. The product resulting is shown in Figure 16. Comparing the new PVA sample (22 – 88) to the previous better option (PVA 26 – 88), the former presents a liquid mixture with lower viscosity and a product that dissolves with more efficiency. The smaller number of monomers that the PVA 22 – 88 possessed, which correlates, to *lower molecular weight*, might be one possible explanation for the observed changes. The lower molecular weight decreased the *polymer's viscosity* and *increased water solubility* (Nair, 1998).

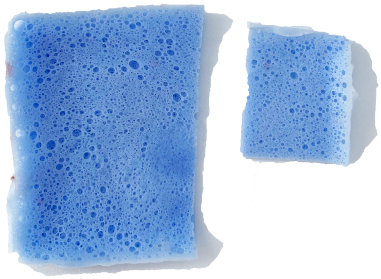


Figure 16 - Resulting products of assays 11.

One prevailing attribute in all of the assays was the feeling of slightly dried skin (not feeling hydrated) afterwards. This should not be alarming, although, it should be taken into consideration. It was caused by the harsher stripping action of the SLS. With the desired consistency, appearance, and performance acquired, it was possible to advance to the next stage using the assay 11 formulation.

4.1.2 Surfactant testing: stage 2

The testing of three different surfactant samples, two of them presenting the same active ingredient, was the main focus of this stage.

It was possible to detect obvious differences, created by the presence of a considerable plastic layer covering the product of assays 13 and 14. The plastic layer rendered the performance of the products non-existent, both marked as 0 on the scale. By contrast, the subsequent product of assay 12 presented dual appearance (two different textures). A softer and "spongy" texture on one of the sides and a more rubbery and plastic texture on the side touching the cast – which weighed on the performance of the product, being evaluated with a 5. Products presented in Figure 17.

These trials, along with the ones from the previous stage, highlighted the correlation between a plastic layer development and a poor performance – the plastic film would not allow for a proper dissolution when in contact with water. With this analysis, the results were quite straightforward allowing the determination of which surfactant would be the better option – Pureact with the active surfactant being SCI.

Figure 17 - Resulting products of assay 12, 13 and 14 – from left to right respectively.

With SCI selected as the SLS substitute, the following steps were similar to stage 1. The qualitative assessment of assays 15 to 18 is presented in Figure 18.

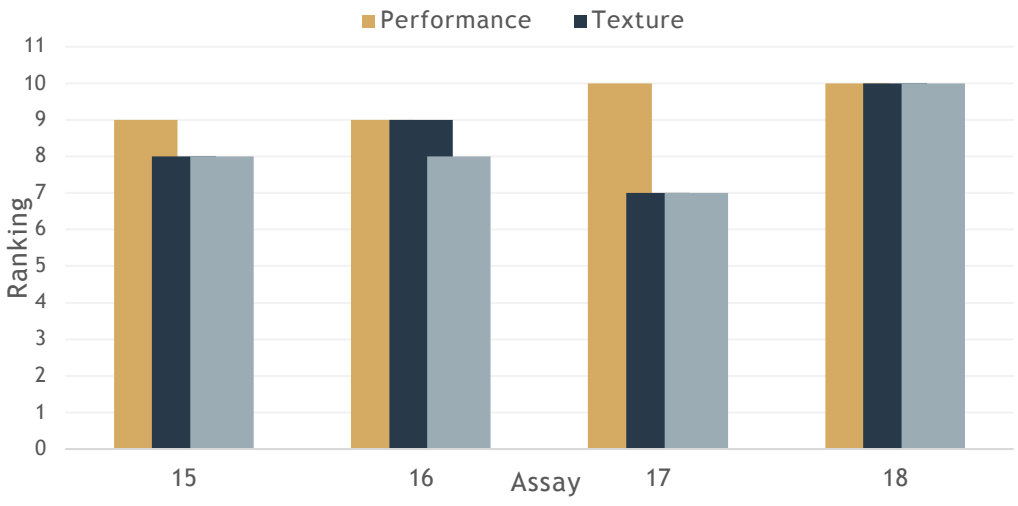


Figure 18 - Graphic representation of the results of assay 15 to 18.

Having identified the faults and the respective solutions in the previous assays, a more uniform assessment was expected and confirmed. Assays like number 8 revealed that increasing the quantity of corn starch would counter the plastic and uneven texture presented in some products (assay 12). This type of change was applied during assay 15 - the quantity of corn starch is 9 % - which resulted in the improved appearance parameters, although the performance suffered slightly – the product did dissolve but created a starchy mixture. This finding proved that the percentage of corn starch, with the elected surfactant and PVA, should be below 9 %, contradicting the hypothesis that it was a viable percentage. Assay 16 presented a very similar result to assay 15, with only a small decrease in the corn starch, although the mixture did not dissolve into a starchy mixture. Throughout the project, there was the identification of the fragrance's influence in product assessment (aroma attribute). The required quantity depends on type and composition, and in this project 1 % originated a product with an overwhelming scent (not what was desired), which resulted in the alteration to 0.5 %, during assay 16. One common attribute of assay 15 and 16 was the soft feeling the product showcased. Not necessarily negative, but the idea of a more structured product, not so easily teared, was proposed by one of the thesis advisors. To achieve what was asked, the main alteration regarded the quantity of PVA, an alteration that had not been applied since assay 8. The change ended up increasing the plastic layer and structure, even with a small increment (+0.1 %), which reflected the relation of PVA and a more plastic appearance, expected from the former drawn observations. Regardless, with this specific alteration, it was not possible to achieve the required product – the product had more structure but lost its smoothness and uniformity. The suggested solution was to lower the percentage of PVA, to the former value (5 %), that allowed a uniform texture. To compensate for the expected loss of the sturdy structure, the quantity of corn starch was slightly increased (from 8.6 % to 8.62 %).

This alteration proved to be effective. A slightly sturdier product was obtained, while maintaining a softness to the touch, which was believed to be an important asset for marketing. In the end, the decision was addressed from both a chemical and a marketing perspective. This formulation presented a perfect performance (in terms of what was required) and a balance between structure and texture. The resulting outcome from assays 15 to 18 is presented in Figure 19.

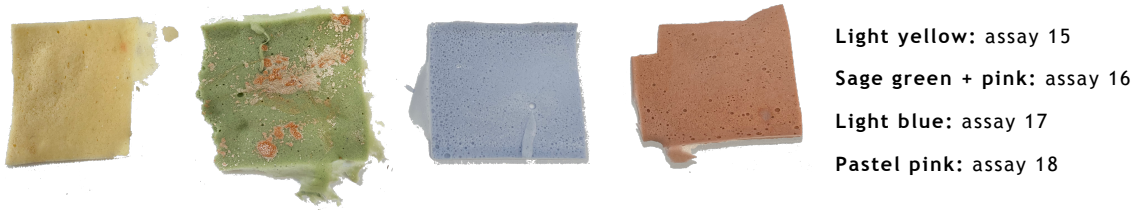


Figure 19 – Photography of the resulting dried products of the final laboratory assays.

In essence, the laboratory trials and assays offered compelling evidence for the importance of choosing the correct ingredient. For example, the PVA's different characteristics influenced highly the: *Appearance* of both liquid mixture and dried product, which were determined by the viscosity of the PVA and were influenced by ratio previously mentioned; and the *Performance*, due to the PVA higher hydrolysis degree. Similarly, corn starch also had a significant impact on the final product, by offering it some *structure*. It could help reduce the plastic film on the product, but if in excess, it would jeopardise the products' dissolution, creating a starchy mixture on the user's hand. The choosing of the surfactant mostly affects the after washing feeling, the mildness to the skin, and the formed lather. However, it also influences the appearance, as was observed in assays 12, 13 and 14.

4.1.3 Quantitative evaluation

The previous appearance and performance parameters, presented in Table 8, can be divided into individual features that detail the product. The various features and corresponding rankings for all the assays are presented in Appendix D, along with the final rank given to the parameters of the resulting product, which will be critical for the progression analysis of the project. However, to understand the improvement of each performed sections of the laboratory work, the three assays with the better performance in the three different steps – PVA selection, Surfactant selection and Final adjustments – were elected. Their evaluation is presented in Table D.4 and the improvement is presented in graphic form in Figure 20. Although, these results would indicate that the product is not ideal yet, it is possible to argue with that analysis. Some of the features such as the "plastic finish", in which the 10 would indicate a total paper-like structure, was a deliberate choice to leave it with a minor sensation of plastic.

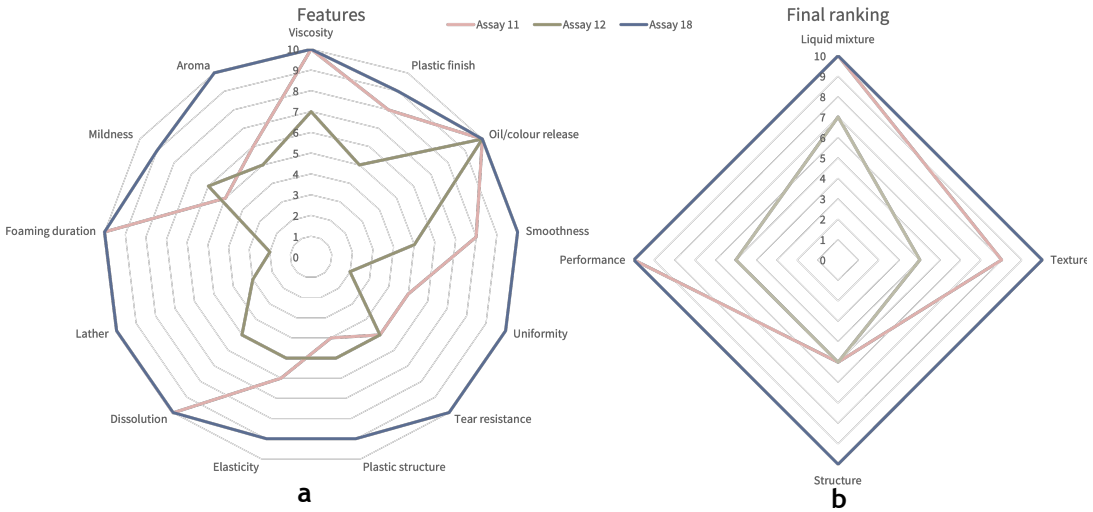


Figure 20 - Graphic representative of the qualitative evaluation of assays 11,12 and 18. (a) all of the features; (b) final rankings of each parameter.

4.2 Pilot testing

4.2.1 Trial assay

One identified limitation of this work was the shortage in information regarding the drying equipment. The only information, besides the operation manual given by the provider, was the temperature at which the reference product was fabricated (between 80 ºC to 90 ºC). To further the data gathered, an earlier trial was performed. During the trial, neither the PVA nor the SLS substitute had been selected. It was performed using the formulation of *assay 9*, which already presented a high performance and what was thought to be an almost ideal texture and appearance. The process followed the protocol presented in 3.2.3. Method. This trial allowed the solidification of some suppositions regarding the process, that had been previously noted. For example, step 2, PVA dissolution in water, needs a continuous stirring to help reduce the procedure's time and will create a more homogenous liquid mixture.

Regarding the process, it was a straightforward trial. The product dried almost instantly when in contact with the main roller (evaporation). As soon as it reached the other side of the roller, it was set and ready to be removed from the equipment. The only limitation identified was the insufficient mass of liquid mixture. The quantity prepared (3kg) was not enough to understand the full workings of the equipment, since it was not even enough to fill the feeding tray. The trial product presented the following features. A thin sheet of syndet and then a soft but somewhat oily and humid feeling - initially, it was supposed the cause was the high percentage of fragrance, but it was disregarded. Interestingly, through observation and analysis of the product and process, it was determined that the product required a post-production drying period. The displayed humid sensation was reduced through time spent in a drying room. The minimum time logged in which alterations were noted was 16 h. It is suggested at least a period of 24 h of drying, but in a room with a low degree of humidity. In Figure 21 a visual representation of the equipment at work during the trial and the final product are shown.

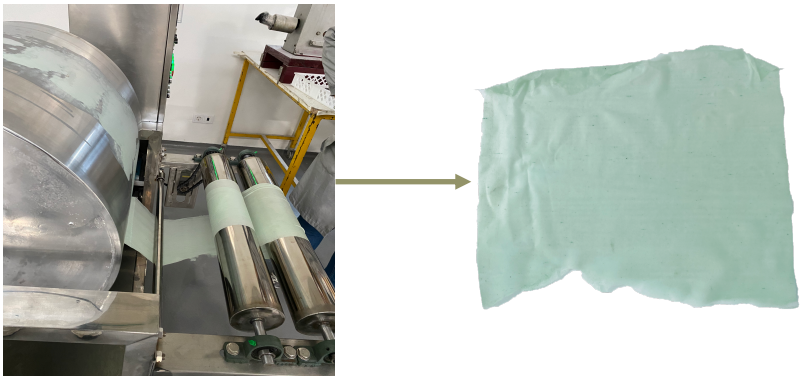


Figure 21 - Equipment at work and final product - Trial assay.

With this trial it was possible to gather the following information, regarding the operational parameters of the equipment (Table 9).

Table 9 - Operational parameters for the trial assay.

Temperature (°C)	ω (rpm)		
	Feeding roller	Winding roller	Main roller
82	10	40	60

4.2.2 Final assay

Having completed the laboratory testing and having evaluated the formulation of assay 18 it was possible to proceed to the final trial stage. Three factors from the trial stage should be considered: a higher quantity of liquid mixture must be prepared; a thicker sheet of product should be produced, the former one was not approved; and finally, the period, of at least 24 h, of post-production drying.

The preparation of the liquid mixture was performed using the same methodology as the previous assay. This was a method that performs well, despite its length. A more elegant and simplified solution would be to use an industrial mixer with a heating system. With the liquid mixture prepared (8 kg) and with the equipment already set to the operational parameters presented in Table 9, it was possible to deposit the liquid mixture in the feeding tray and begin the process. In contrast to the earlier stated assay, the product did not dry instantly. Initially, it was thought to be a positive factor because it would allow an increase to the thickness of the product (the feeding roll would be continuously adding product). But soon, by observation of the process, it was evident that the operational parameters were not adequate to the new formulation in use.

The first error detected in the process was allowing the accumulation of the product in the main roller. The product should not have completed more than one loop in the system. To build on the thickness, the equipment required an enhanced speed of the feeding roller and at the same time an adjustment to the static 1 roller. The modification to the speed allowed the build-up of the product, which would then through the static 1 roller. The main function of this roller is to homogenise the product, both in thickness and in uniformity. So, by withdrawing the static 1 roller from the main roller, we were able to increase the thickness of the product. However, there is a limit to the thickness of the product; if the static 1 roller was too distant from the main roller it would work properly.

Inevitably, there was some accumulation on the main roller's surface, however it was not drying, as was supposed. The feeding system was halted, to stop the product from accumulating. The water on the product continued to not evaporate successfully. This change suggested that, not only the thickness of the product influences the time required

to dry (as stated previously), but also the temperature at which the machine should be operated. It was hypothesised that the formulation might require a higher temperature.

To solve this setback, the first parameter to be altered was the temperature of the main roller. It was done by increasing in increments of 10 degrees Celsius and waiting about 10 minutes, allowing the equipment to stabilise and for the product to react to the change. The final temperature was 150 ºC, due to some noticeable improvements. This decision might be unreliable, as there were no literature references to validate this alteration, and it was used only in one assay. Besides the temperature change, both the main and winding roller were slowed down. This alteration was introduced to allow a longer drying period time, before being removed from the equipment. With these final adjustments the water on the product, still placed on the main roller, was able to evaporate. It was removed with the help of the winding roller. The experiment, with the adjusted parameters and with a reconnected feeding roller, was then repeated following the same continuous loop presented in Figure 12. The alterations can be found in Table 10.

Table 10 - Operational parameters used during the final assay.

Temperature (°C)	ω (rpm)		
	Feeding roller	Winding roller	Main roller
150	80	20	9

During this stage it was possible to detect two wasteful situations. The first one was observed during the feeding stage, the higher speed of the feeding roller made the tray began to overflow. This issue was not detected on the previous assay using this machine. It is suggested a more gradual feeding method, when handling higher quantities. The second wasteful situation had been already identified. Some product was left at the bottom of the feeding tray, which was not possible to use unless more volume of product could be added.

In the following Figure 22 the equipment used during this assay is presented.

Figure 22 - Dying equipment at work during and resulting product – Final assay.

By the end of this assay, it was possible to attain a final product with acceptable characteristics. The product presented a remarkably smooth texture (feeling) and a soft paper-like structure, with a slight resistance to tearing. The product dissolved immediately with the water and the lather was very pleasant. The product was very mild, when compared to the samples with SLS, and left the skin feeling smooth and clean. These characteristics were translated to a quantitative evaluation by the author and the advisors presented in the following Figure 23. The referent values are in Table D.6 in Appendix D. There were two limitations to the validation of this analysis. This analysis was done after 24 h, and it had been hypothesised a longer drying period post-production. And finally, due to small adjustments made to the equipment, not all the sheets were uniform.

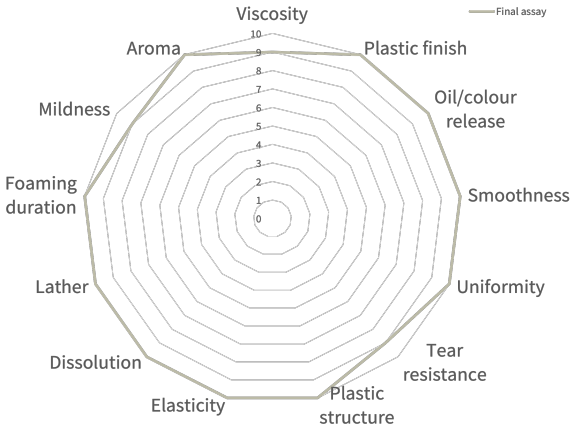


Figure 23 - Graphic representative of the qualitative evaluation of the product of the final assay.

The final assay and its resulting product were not without faults, as stated throughout this section. Unfortunately, it was not possible to perform another essay with the proper corrections, due to time limitations. The first required correction regarded the confirmation at which temperature the equipment should operate. The product still showcased a slightly humid sensation to the touch, giving the product an “unfinished” feeling. It was only eliminated after a period of 24 h hours, in a dry and heated room. The second correction regarded the method of using the drying equipment. The final assay served as a sort of trial, which resulted in some alterations to the state and parameters of the equipment. Despite the limitations of this assay, and consequently the imperfect result, it was possible to attain a functional, uniform and aesthetically pleasing sample product (with the added post-production drying time).

4.3 Quality control

Strong evidence of the product's susceptibility to the environment's humidity was detected throughout the present work. The nature of the developed product was to dissolve with water, in an efficient and almost instant way. Consequently, the resistance to different environments such as transportation, storage or the day-to-day basis of the consumer had to be tested. As explained in 3.5 Quality Control, three quality tests were performed. All of them were performed during a time span of 10 days. This evaluation helped to further the knowledge of the product, allowing the team to design the packaging.

Up until the fourth day the products exhibited no visible or tangible alterations. The alterations perceived on the fourth day and the tenth day were similar. There were no visible alterations, but the tests were not long enough to allow valid conclusions. The sample being tested under high temperature displayed a faintly more rigid structure and an enhancement of the fragrance's intensity. The sample under low temperature showed no alteration. Finally, the samples under humidity assessment featured a moister sensation to the touch, which was expected. The samples separated by wax paper, as expected, were not glued together. An unforeseen but satisfactory result was found in the samples that were stacked together. They did display added moisture to the texture, which lead them to slightly stick, however their separation was easy to execute. Initially, it was thought that in case of extreme humidity, the product might degrade, making the sheets stick to each other. However, it was possible to assume that the product might not need a separator, such as waxed paper.

One surprising factor of the formulation in analysis was the lack of a conservative or an antioxidant. These components help extend the shelf life of the product and prevent contamination, and so are usually found in cosmetics. Moreover, the paper syndet should follow the same rule, even in some liquid syndets these components can be found. This factor could prove a limitation to the commercialisation of the product. To determine the need of these components, some additional tests such as microbial and water activity should be performed.

4.4 Product assessment

In an effort to obtain a broader sense of how the product would be perceived, besides the opinion of the mentors and of the author, a small *questionary* was undertaken with a group of thirty people, for a period of about two weeks. The questionnaire requested the previous use of the developed and reference products in order to compare them, so, due to the present movement restrictions, it was not possible to gather more answers. Of the study population, the majority were female, and it was an equal major percentage (30 %) of 18-

25 and 26-35 year olds. The aim of this questionnaire was to understand if the possible consumers would be interested in the paper syndet product and how they perceived it. Of the study population 80 % answered that they would be interested in a new product and just over half of the participants (53.3 %) indicated their preference for recyclable packaging, with one use.

Regarding the comparison between the products, 63.3 % of the population preferred the developed over the reference product regarding texture and structure of the products. A small minority responded that the developed product did not produce enough lather or rinse off efficiently. It was believed that these answers might be explained by the non-uniformity of the samples, and the need to dry the product under more correct parameters. Also, both samples had the same measurements of 1.5 x 4 cm, which confirmed that the presented dimensions might not produce the ideal quantity of lather for the consumers. No one claimed the product irritated the skin. When the subjects were questioned if there was anything they would improve in the product, the majority requested different smells, such as lavender, and a variation of colours. Finally, **76.7 %** of the participants preferred the developed product, which was a very positive assessment for the work developed. Of the 30 subjects who completed the questionnaire, fifteen rated that they liked the product (4 in a scale of 1 to 5) and twelve answered they loved the product. But only eight people answered they would definitely buy the product. The majority of the population was willing to pay 1 to 5 euros for a product with fifty applications. The answers to the questionnaire are presented in detail and in graphic form in Appendix C – Questionnaire.

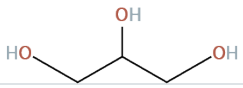
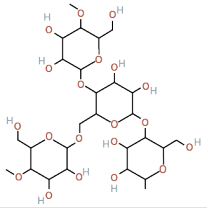
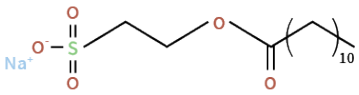
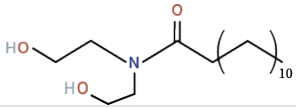
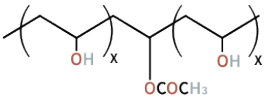
4.5 Final Product

4.5.1 Formulation

This work was mostly based on the theory proposed by S. Showell (2006) of formulations being a compromise between easy handling, processing properties, acceptable consumer aesthetics and, of course, economic requirements. The resulting product and the incurring decisions are in line with this theory. The final formulation chosen for the product is shown in the following Table 11. In the pilot testing stage, the product presented the following characteristics: the most striking smooth texture, that feels curiously silky. Regarding the structure, it presented a more delicate one than the reference's product, definitely not plastic. Performance was ranked at 10/10, a mild action leaving a soft and creamy feeling on the hands. The product presented a neutral pH in both performed tests. Even so, the solution using an already dried product proved to have a slightly higher pH than the pH test using the liquid mixture (7.171 > 7.010). The pH presented the expected

value according to Johnson & Ananthapadmanabha (2010). This neutral pH contributed to the mildness of the product, not disturbing the SC and its lipids.

Table 11 - Final formulation, with reagents and respective chemical structure.

Number	Function	Reagents	Percentage (%)	Chemical structure
1	Solvent	Water	62.86	
2	Moisturiser/Emollient	Glycerine	9.00	
3	Thickener/filler	Corn starch	8.64	
4	Main Surfactant	SCI	8.00	
5	Emulsifier	Cocamide DEA	6.00	
6	Film-forming	PVA	5.00	
7	Fragrance	-	0.50	-
8	Pigment	-	as needed	-

For marketing, packaging, and pricing purposes there was a need to determine the ideal dimension for a single use of the product. Interestingly there were many possible options depending on the consumer's preference. To the consumer's eyes there is a correlation between the quantity of lather and how efficient the product is at cleaning, even if it is inherently not real (Redding, 2012). With this in mind, several combinations for a rectangular shaped sample were tried out. The results are presented in Table 12.

Table 12 - Dimensions for trial samples. x - indicates no foam; o - quantity of foam.

w (cm) \ *l* (cm)	1	1.5	2
2	x	x	x
3	x	x	o
4	x	o	oo
5	o	oo	ooo

The proposed dimensions for a commercialised product were 1.5 (*w*) x 5 (*l*) cm and 2 x 4 cm. The dimension 2 x 5 cm could also be acceptable. The lather becomes slightly bigger, less condensed, but it is a matter of preference. Even though it was not assessed, the dimension 1.5 x 6 cm, would also be acceptable. With this information and the idealised

number of fifty sheets per package, it was possible to advance to the packaging development.

4.5.2 Packaging

Packaging is quite an important detail for the development of cosmetic products. As stated in Topoyan & Bulut (2008) not only is packaging commonly regarded as protection, but it can convey information, add value and draw attention. The main requirements for the developed product were aesthetic visuals, coherence with the brand's image, high-quality finish, innovative, sustainable, easy to use and compact (portable). In terms of mock-ups three main ideas were presented. The first one was based on a more common idea of a rectangular "wallet" packaging, where the product would be displayed on both sides of the box. The second proposal was a bit more unconventional. The suggestion regarded a compact cylinder box, having the product in the form of a spiral. To avoid the overuse of the product, the proposed solution was to add a perforation at the correct length. Finally, the last mock-up would be with a rectangular box with a lid with the round corners, to have a more pleasing visual and holding sensation. The product would be cut into a rectangular shape but could be presented in two distinct ways. All the mock-ups present a limitation regarding the (possible) need to separate each sheet from each other. Due to the lack of knowledge concerning the behaviour of the product it still had to be considered. There are varied solutions to this issue. The schematics for each mock-up and the correspondent dimensions are presented in Figure 24.

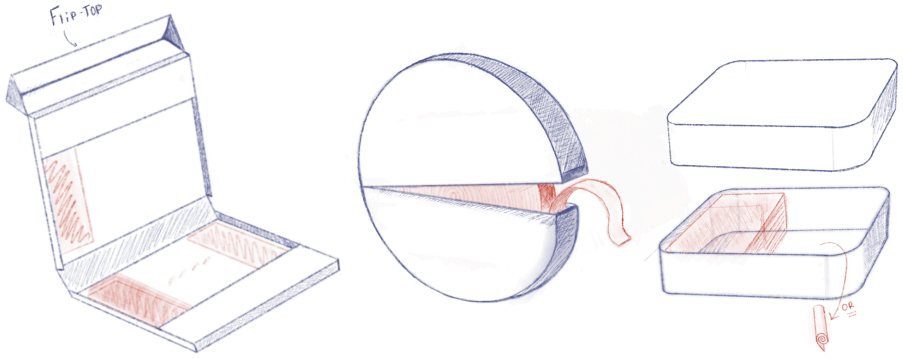


Figure 24 - Schematics for the three packaging solutions proposed.

Regarding the use of materials for the packaging, it was proposed the use of impermeable cork. The main reason was the waterproof characteristic, isolating the product from the elements. Cork is an eco-friendly option - natural, biodegradable and recyclable. This study revealed the possibility of using recycled cork(*Escolha e Recicle a Cortiça - Green Cork*, n.d.). It would be a more expensive option than the usual materials. Before the ending of this work, Castelbel had not yet decided which packaging would be selected.

4.5.3 Market and Pricing

The sector of cosmetics and personal care products has been considered one of the most competing and demanding markets. Besides performance and aesthetical features, the consumers choose a product based on the price. To determine the price of a product, a few elements had to be taken into account: how much the consumer was willing to pay, the competition's prices, production cost and packaging price (Bygrave & Zacharakis, 2011; Luong et al., 2017). In this case, the target market possessed a medium to high purchasing power, allowing the brand to implement *higher prices*. It was possible to identify fifteen products from competitive brands, with various prices and types of packaging. However, it was not possible to make a direct comparison between the developed product and its possible competitors. The calculations and the compilation can be found in Appendix F. The fact that both the pricing extremes showcased the same number of products was a striking observation to emerge from the data comparison. However, the Castelbel product would be considered to integrate the group of products with prices superior to 3.00 €. There were six possible competitors in the study, of which four could be disregarded due to the type of product. All were marketed as paper syndets/soaps, although most used a specific cellulose based paper submerged in a solution with the desired formulation - different product. The remaining products were from the brands Fomin and Mathilde M. Both brands showcased a particular attention to the product's perception. However, the Mathilde M product was developed to be a homeware/decor product and not a portable one. Therefore, the only product that would be a direct competitor in this study was Fomin, which presented both a visually pleasing, easy-to-use packaging (cardboard) and a concept product with added value (they plant a tree with each purchase). There are some differences in the formulation of the products, however it was believed that they could be equated. This analysis was performed in order to collect data on the rival market, furthering the collected data. The **economic analysis** will be mainly influenced by production cost, which includes raw material, equipment and labour cost, plus packaging and marketing cost. The cost of raw materials and the respective percentage on the formulation were used to predict the cost of producing 1 kg of product, which was estimated to be **3.06 €**. The price per package of the product would be approximately **0.04 €.** Having asked for a quotation for different packages, it was stipulated that the average price of a package should be around 2.05 €. It was then possible to the suggest that the final price (estimative) should be 3.42 **€**. The method utilised to determine this value is explained in Appendix G – Economic analysis.

5 Conclusion

In this work an innovative product, denominated paper syndet, was developed through assays and product assessments divided into two different stages – laboratory scale and pilot scale production. The main difference between these two stages, besides the scale, was the use of different equipment for the drying step. The "paper like" appearance and structure were possible due to the use of the machine denominated JF – 600 Drum Dryers, which was used in the pilot scale production.

The main objective of the first stage was the improvement, through alteration, of the original reference formulation. The work was initiated with a series of assays, to determine which sample of Polyvinyl Alcohol (PVA) would perform better. There were five different PVA samples all presenting a degree of hydrolysis between 86.7 % and 89.9 %. During this stage the surfactant used was Sodium Lauryl Sulfate (SLS), seeing as it was the surfactant used in the reference product. It was determined that the chosen PVA would be 22 – 88, with a viscosity of 20.5 – 24.5 mPa.s, a degree of hydrolysis of 87.0 - 89.0 mol% and a pH between 5.0 and 7.0. However, using the quantities determined by the reference formulation the resulting product did not present the appearance and performance desired, so some alterations were required. The evidence from this laboratory stage points toward the use of a formulation with the ratio of water:PVA:corn starch of 62.5:5:8.5, which showcased a softer texture. Having managed to determine the (supposed) ideal formulation, it was possible to test milder surfactants to replace the SLS, in order to decrease the harshness of the product to the skin. Two different surfactants, from three samples, were tested – Sodium Cocoyl Isethionate (SCI) and Sodium Lauroyl Sarcosinate (sarkosyl). The selected surfactant was the SCI. Although from the resulting product, it was determined that the formulation still required some changes to reach the standard previously stipulated. This was followed by a new series of assays, altering once more the determined ratio of water, PVA and corn starch, until the product was approved. The final formulation presented a ratio of 62.86:5:8.6. It should be noted that formulations were chosen based on a quantitative evaluation of set parameters, that defined the performance and the appearance.

It was then possible to advance to the pilot scale production. Initially, a first trial assay, using a formulation which was not yet ideal but presented a good performance, was completed to understand the workings of the equipment and the respective operational parameters. However, when performing the final assay with the chosen formulation, the operational parameters had to be altered – the feeding roller speed was increased, to allow the build-up of the thickness of the product; the temperature of the main roller required

an increase to 150 ºC, form the original 82 ºC, and both remaining dynamic rollers were slowed down to a minimum to allow the product to dry properly.

Finally, with the resulting product from the final assay, a product assessment was performed in order to validate it. The validation was achieved through a quality analysis and an evaluation of the product by possible consumers, with a questionnaire. The quality tests were performed during a ten-day period and considered the stability of the product in high and low temperatures and also in an environment with high humidity. The results were rather satisfactory, with no compromising alterations being detected. It was also the questionnaire that indicated that 90 % of the study population liked the product, which is a very positive outcome, but only 73.4 % would buy the product. Most of the population was willing to pay up to 5 € for a product with fifty applications. The population outlined that they would like a variety of perfumes and colours in future developments.

With this data collected it was possible to construct a suggestion for three different types of packaging and the ideal dimension for a single use of the product, and also to estimate the pricing of the product. The packaging which appeared to be innovative and draw the attention of the customer was the cylinder option. Regarding the price, it was estimated that the price could amount to 3.42 €, but by the conclusion of this work, Castelbel had not yet made the final decision regarding these factors.

The work was not without limitations. Due to a limited time period, it was not possible to consolidate the knowledge from the pilot scale production. It was believed that the temperature could be increased to minimise the already small feeling of humidity the product presented, disappearing only with a rather long post-production drying period - at least three to four days in a dry and slightly heated room. The product also required further quality control to validate the product, such as tests of water and microbiological activity, and more stability tests in a broader range of temperature and humidity levels for a longer period of time. These tests would allow to determine if the product required the adding of conservative agents.

Despite these limitations, it was possible to develop an innovative solution in the form of a paper syndet - an easy to use and portable hand washing product, with the added values of a mild and soft cleansing - while still maintaining the brand’s initial requirements, such as an aesthetic and high-quality appearance, eco-friendly and cost-effective characteristics. This work is believed to also provide a framework for future developments in this area and this type of product.

6 Assessment of the work done

6.1 Objectives Achieved

The main goal of this dissertation was the development of an innovative product. The development included two different stages – the laboratory and the pilot stage. During the first stage the main objective was the improvement and alteration of the original formulation, through the assessment of many performed assays in order to detect which combination would provide the desired features. The second stage consisted of a larger scale pilot, using the proper drying equipment, determining the operational parameters and seeing what type of product would be attained. Afterwards, to validate the product, some small quality control tests would be performed, along with a questionnaire to possible consumers. The work was concluded with the development of suggestions for packaging and pricing. The results for the quality control testing were satisfactory for the small time period and the product seemed to have caused a good impression on the possible consumers, having highlighted the softness of the product and its remarkable performance.

Thus, it is possible to say that the goal of developing a new product was achieved with satisfactory results. Additionally, it is believed that this study can be used as a framework for future alterations or similar developments, since there is not much information on the literature.

However, the situation is still incomplete. The process, during the final assay of the pilot stage, served as a trial to collect information on how the chosen formulation would react to the drying equipment. It is believed that the used temperature was not high enough, because the required post-production drying period was too long, which was unexpected. So, some testing should be done regarding this limitation. Another was the impossibility to perform proper quality control evaluation, due to the time limitation. And finally, it was not possible to test out the packaging with the product to see if there were any details that needed adjusting.

6.2 Future work

Besides the need for future testing using the drying equipment to consolidate the attained results, and the need to perform water and microbiological activity testing to establish the need to add conservatives and/or antioxidants, it is believed that this project has the possibility to expand. Firstly, the product could be expanded beyond just one line. This idea came from several answers to the questionnaire which asked for different fragrances, which would lead to the production of colourful products which could attract

more customers. The product can be developed to present more value through added benefits to the skin (moisturising action, aroma therapy, exfoliating, etc.). However, it could be developed to act beyond a hand washing product to possibly a body or face cleanser, or even to a conditioner and shampoo. There are numerous possibilities that ae believed to be worth studying, considering the solution the type of product could represent.

6.3 Final Assessment

This work helped Castelbel accelerate the research and development stage of the paper syndet, generating some value to the company.

In terms of personal experience, it was a very enriching one in all fields academic, professional and personal. It was possible to integrate the team of workers at Castelbel and learn how the industry works and how it feels to work at a company. Besides, it has been this author's aspiration to develop a product in the cosmetic industry, so to have a guided and such satisfactory first experience was extremely beneficial.

By the end of this work, it was possible to obtain an innovative product, which is easy to use, portable, eco-friendly, while still maintaining the brand's identity. The product is also quite inexpensive to produce. It is believed the paper syndet will draw some attention and curiosity, due to its ground-breaking nature.

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Annex A – Cleansers formulation

Before comparing the formulation of each type of cleanser, the fundamental differences between soaps and syndets should be understood. That comparison is presented in Table A.1.

Table A.1 - Comparison between the characteristics of 3 different cleansers: soap, combo and syndet. (Friedman, 2016; Zoller, 2008).

Characteristics	Types of cleanser		
	Soap	Combo	Syndet
Function	Cleansing	Cleansing	Cleaning
pH[1]	8.5 – 11	7.5 – 10	5.5 – 7
Composition	Alkaline	Slightly alkaline	Alkaline free Plasticiser or binders
Hydrophilic groups	Shorter	Mix	Longer
Hydrophobic groups	Longer	Mix	Shorter
Performance under Hard water	Scum[2]	Rinsible	Rinsible
Solubility	Lower	-	Higher
Lather	Low	Medium	Higher
Wear rate	High	Medium	Low
Mildness	Not mild	Potential mild	Mild
Slough (in bar products)	Low	Medium	Higher
Cost	Low	Medium	High

[1] Skin pH is between 6.5 and 5 (Friedman, 2016)

[2] white or grey filmy layer that covers the surfaces around our showers, bathtubs, and sinks

A cosmetic or personal care product formulation is made with the intention of satisfying the requirements of the consumers , which have been growing, not only in numbers per product, but also in difficulty to achieve (Lai, 2006). As it was assured by K. Lai (2006),a good product is the result of the careful selection and balance of all the ingredients present in the product. The more common elements in personal care products, in between the existing diversity, according to S. Showell (2006) are surfactants; dispersing polymers; builders and chelants; bleaching systems; solvents; and performance-enhancing minors. As so, modern detergents, nowadays, are comprised of a broad variety of ingredients (usually 20 +) which vary with the purpose of the product, its target consumer and, also, its type (soap or syndet) (S. Showell, 2006). It was possible to hypothesise that there is a multitude of options that lead to a somewhat equal product. As so, there is a need to narrow down the immense list of possible ingredients to use.

The chronological order of "evolution", as stated previously, was soap bar –syndet bar - liquid syndet – new form syndets. So, it could be suggested that the liquid syndet is the closest in terms of formulation to this new form of syndet. For a better understanding of the selected formulation for the new syndet and for future reference, an example of a liquid syndet detailed by Lai (2006) will be presented in Table A.2.

Table A.2 - Liquid hand soap and body wash composition, adapted from (Lai, 2006).

Functional ingredients	Percentage (%)
Surfactants	10 - 40
Emollients	1 – 30
Rheology modifiers	1 – 5
Preservatives	< 1.0
Fragrances	0.3 – 1.5
Colouring agents	< 0.1
Other additives	0 – 3
Water	balance

In the following Tables A.3 and A.4, the functional ingredients, some examples, and the percentage of a bar of soap and a syndet bar, respectively, will be shown. In the table correspondent to the syndet bar, the percentages and the presence of the ingredient will be shown according to the type of customer that the product is meant for.

Table A.3 - Bar soap formulation adapted from (Abbas et al., 2004).

Functional ingredients	examples	Percentage (%)
Surfactants	Natural soap, synthetic surfactants	30 – 87
Water	-	5 – 30
Colourants	Different colours	0.001 – 0.1
Preservatives		
Antioxidants	BHT[1]	0.005 – 0.01
Sequestering agent	EDTA[2]	0.005 – 0.1
Whitening agent	Titanium dioxide	0.1 – 2.0
Processing aids	Sodium Chloride	0.3 – 2.0
Fillers	Starch, clays, etc.	1.0 – 20.0
Fragrance	-	0 – 1.5
Speciality ingredients		
Lather boosters	Co-surfactants – SLES[3]	1.0 – 5.0
Antibacterial agents	Tricloson	0.1 – 1.0
Moisturizers/skin feel agents	Polyols, lanolin, etc.	0 - 30

1 – butyl hydroxy toluene; 2 – ethylene diamine tetraacetic acid; 3 – sodium lauryl ether sulfate.

The choosing of both examples was not at random, since the bar soap is the "original" form of the product that is being developed in this project and the syndet bar has the same

base as the product, and the liquid hand wash has the most similar composition to the product, being that the idea is to produce a liquid hand wash, with a bit more structure, and then evaporate the water.

Table A.4 - Typical formulas of syndet bars.1

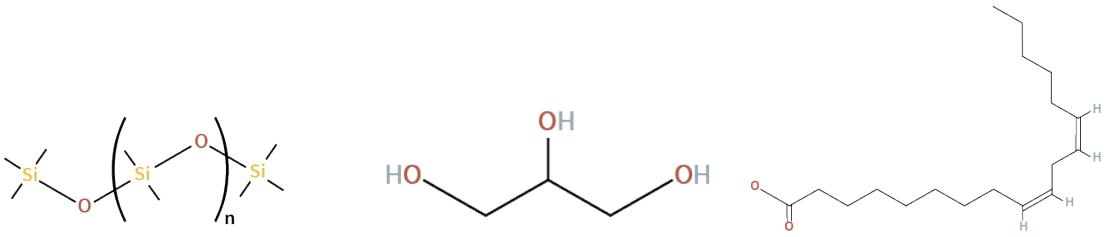
Functional ingredients	Percentage (%)			
	Dry skin	Oily skin	Delicate skin	Sensitive skin
Synthetic surfactant	95 - 96	96 - 97	95 - 96	95 - 96
Water	1 - 2	1 - 2	1 - 2	1 - 2
Perfume	0.3	0.25	0.27	-
Plasticiser	*	-	-	-
Emollient	*	*	*	*
Preservative	-	*	-	-
Antibacterial agent	-	-	-	*
Dye	*	*	*	-

1 (*) product has the ingredient; (-) product does not have the ingredient adapted from (Zoller, 2008)

The different options evolved with the need of creating different products to meet the new requirements and needs of the consumers, be it for the need of better efficiency in different types of skins, to add different characteristics to the product, to create a milder effect, to improve the quality of the lather or to mask the off-odours, and these are just a few examples (Friedman, 2016).

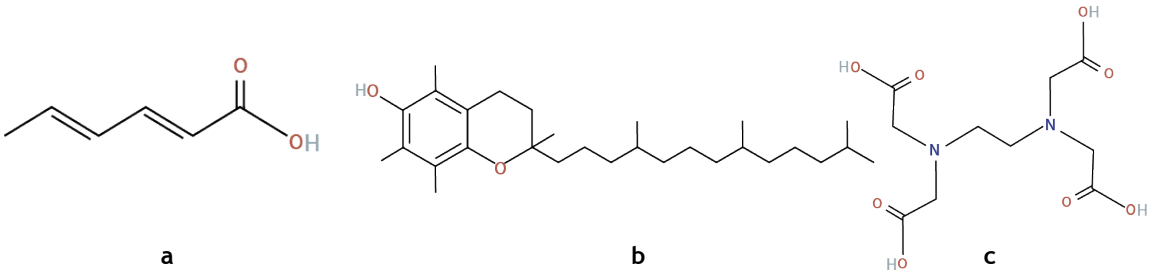
Annex B - Chemical structures

In order to improve the workings of the type of components used in syndets, some examples of components, and the respective chemical structures are presented.



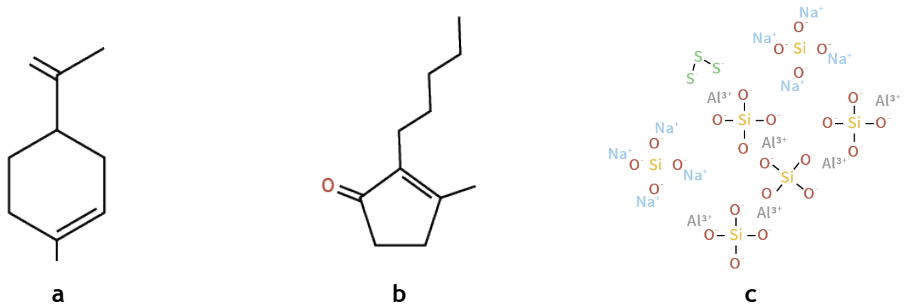
a b c

Figure B.1 - (a) Chemical structure of dimethicone, an occlusive moisturiser. (b) Chemical structure of glycerine a moisturiser. (c) Chemical structure of linoleic acid, an emollient.



a b c

Figure B.2 - a) Chemical structure of sorbic acid a stabiliser. (b) Chemical structure of antioxidants examples: vitamin E. (c) Chemical structure of EDTA, a chelating agent.



a b c

Figure B.3 - Chemical structure of some fragrance examples: (a) limonene (cyclic terpene); (b) dihydrojasmone (ketone). (c) Chemical structure of colouring agents – ultramarine blue C-29 CI77007.

Annex C - Product development

C.1 Reference formulation

To initiate this project, there was the need to have a base for it and seeing as it is not information easily found in the literature, due to the product not being familiar in the market. So, in favour of the project, the company responsible for producing the dryer used sent the information needed.

Table C.1 - Formulation composition sent by the provider.

Raw materials	Percentage (%)
Water	60
PVA	9
Medicinal glycerine	9
K12	8
6501 detergent	5.8 – 6
Edible starch	8
Fragrance	0.2
Edible pigment	as needed

PVA stands for polyvinyl alcohol, *K12* corresponds to Sodium Dodecyl Sulfate, known as SLS, and 6501 detergent is coconut diethanolamine.

C.2 Reference Laboratory Method

The resume of the protocol, in which this work was based, is as following, for 100 g of product: 1. Put water into the mixing pot, open mixer and set mixer to 15 rpm, and heat the water until 50 degrees Celsius. When the temperature is reached, add the PVA, adjust the mixer to 25 rpm (open homogeniser and work it for 1-2 minutes). Melt PVA to a transparent mixture and with no precipitation, until 80 ºC is reached - finish step and change mixer speed to 15 rpm; 2. PVA melt finished, into the cooling water for about 20 mins (5 jets of cold water). The temperature should decrease to 70 ºC; 3. While the temperature dropped, the mixer speed was changed to 5 – 10 rpm, and then the K12 (surfactant) was added (mixer speed was increased again to 15 rpm); 4. When the K12 has finished mixing add the medical glycerine; 5. Once entirely mixed, add 6501 detergent (emulsifier) mixing it thoroughly; 6. Then decrease the temperature to about 40 ºC. Next, add the starch and slowly increase the stirring speed; 7. Flavour and pigment can be added as needed. Remark: if the final mixture is too wet – add starch; if the final mix is too dry – add water.

Annex D – Operation MANUAL

JF-600

Drum dryers
(laundry equipment)

Operation instruction

Guangzhou Jinfeng Light Industry Machinery Co.,Ltd
Tel：+86-20-86070290 Fax：+86-20-36206862
Email: aftersales@china-jfmachiney.com

1.Assembly diagram

2.Overview

JF-02 series drum dryer is a new product developed by our company, are widely used in sticky, paste materials production processes.Worked in the food, chemical, pharmaceutical and other industries. Good performance.

3.Working principle

JF-02 series drum dryer working principle is to make liquids or muddy object item in steam or other hot heating surface to form films. drum turn around was in the process of drying is completed. use spatula scrape. revealing the surface again with the raw material contact drying form a thin film.

4.Structure introduction

JF-600 series drum dryer composed by a rollers, racks, the charging device (top feeding or bottom pre-pregs), drive, Hopper, charge transfer device and so on.

5.Feature

①With steam or thermal oil as the heat source. larger heating area. efficiency. uniform heating

②Drying rate fast. wall of wet material membrane on heat and mass transfer process from the inside outward in the same direction. temperature gradient greater. keeps the material membrane surface high evaporation intensity

③The drying time is short. about 7~30s. suitable for heat-sensitive materials

④Operation is simple. easy maintenance

6.Technical parameter

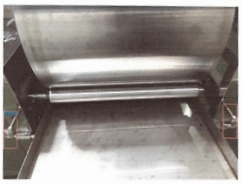
Model	Roller diameter	Effective heating area M²	Power（kw）	Machine size（mm）	Machine weight（kg）
JF-600	Φ600X600	1.4	1.1	1330X1180X1920	250

Note: this form of technical parameters for electric heating

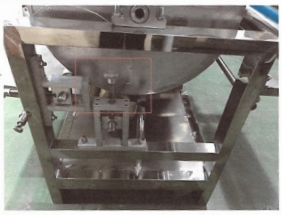
7. Installation and test

JF-02 drum dryer is made of uniset, the main machine must be installed in a solid level on the ground, placed smoothly. No need to be fixed. Before installation, according to the site, the equipment in place and install the draught fan and pat wet pipe position.

1. The speed regulating device of feeding drum： is used to adjust the speed of feeding drum
2. Rolling drum speed control device： is used to adjust rolling drum speed
3. Main drum temperature gauge device ： is used for regulating main drum temperature
4. Main roller speed regulating device： is used to adjust the speed of the main drum
5. Main drum switch device： is used to control the opening of the main drum
6. Rolling drum switch：is used to control drum start-up
7. Feeding drum switch：is used to control the start of the feeding drum
8. Heating switch： is used to control the heating start of the main drum



As shown in the red box device is scraper adjustment, is used to adjust the thickness of the laundry sheet, when rotating clockwise at the same time, the laundry sheet becomes thinner and thinner, when turning counterclockwise at the same time, the laundry sheet becomes thicker and thicker.



as shown in the figure, the red box device is the feeding drum clearance adjustment, two at the same time to clockwise twist, the gap is getting larger and larger, two at the same time to counterclockwise twist, the gap is getting smaller and smaller.

3, The electric use 380V 50Hz three-phase five-wire cable, the operation of electronic control box and the main machine must have a good wiring.

8.Care and maintenance

JF-600 drum dryer consists of cylinder, frame, transmission and exhaust system components, the material is generally all stainless steel, stainless steel and carbon steel material contact for production, to ensure the normal operation of the equipment in the best condition, usually have to do maintenance work.

1, often check the gear reducer lubrication oil condition, forbid the gear reducer to work under the condition of no oil, detailed reducer maintenance, see manufacturer's instruction manual

2, often to the transmission gears and bearings with grease, avoid oil free state transmission, shorten the service life, generally three months to check again.

3, the surface of the cylinder must be kept smooth and clean. After use, it should be cleaned and cleaned. No hard objects should be used during cleaning. The surface of the cylinder can be scratched. Only towels and various kinds of cotton cloth can be used to scrub it

4, often check whether the connecting bolts are firm and reliable, to prevent bolt loosening, equipment production accidents

5, to ensure that the control of electrical boxes clean and dry, maintenance must have professional electrical operations, often check the electrical control box grounding wire is good, to prevent leakage accidents

6, stop using the equipment must ensure that equipment surface clean and dry, to prevent damp surface rust.

7, electricity, with wiring see electrical principle diagram.

9.Debugging

Try to carefully check whether all transmission connecting parts are reliable machine, check whether the lack of reducer oil, each component fixing bolt is loose, the electrical console is normal, the heating is normal, all the check to make sure the normal rear can carry out the following.

1, the power supply is switched on, start the drive motor, speed slowly, observe whether it is normal to drum;

2, observe the rotation of the main drum and the driving parts, whether it is flexible or not. If there is any case, stop the inspection immediately and find out the reason before it can be switched on again;

3, when the material has dried up, first turn off the main engine heating source, stop the cylinder rotation, and wait until the cylinder is cooled to 50 degrees Celsius, then clean the cylinder and chat trough with clean water, then cut off the power supply.

Appendix A - Product Design

A.1 Process

In order to better understand the Figure 5 in 2.2 Product Design, it was decided to expose its explanations in this Annex.

The process begins with the identification and the understanding of the customer needs, which will be considered the "spark" of the product development. Customers are seen as both the ones who will buy and use the product. Determining the requirements of the customers, involves three phases in itself: interviewing the customers, interpreting and then translating the requirements into product specifications. (Cussler & Moggridge, 2012) Once the specifications for the product have been established, the second phase can commence – the idea generating. Only one brilliant idea is needed in order to create an efficient and distinguished product. However, to do that a high volume of alternative ideas have to be designed. Ideas come from various sources such as the *customers*, every bit of information given by this group will be analysed and taken into consideration, the *competitors*, the *consultants* and the the *inner design team* (through their brainstorming sessions). After being organised into different categories, the ideas will be filtered and selected in accordance to different types of criteria. (Cussler & Moggridge, 2012) Thus, initiating the third phase – the selection of the idea. This selection is supposed to elect the best ideas for further development. The criteria in which the selection is based goes from chemistry and engineering elements to more subjective criteria. After the selection of the best ideas, they should be put to the test, to determine possible adjustments. Finally, the manufacture downstream development should be thought out, with a patent if needed. In the middle of this phase there should be a revision of the product specifications, taking into consideration the chosen specific solution and the values it represents. (Cussler & Moggridge, 2012)

A.2 Requirement assessment and metrics

To initiate the process, the correlation between both the need and the specification had to be made. Afterwards both a rank and a metric were assigned to each specification, creating an easier and more precise way to guarantee the final product resonates with the identified needs. The metrics correspond to the unit measure of specification, it can be both quantitative and qualitative. The ranking allows the specifications to be evaluated according to their importance to the process of product design. The rank will be categorised as: 5 – mandatory; 4 – priority; and 3 - recommended (Cussler & Moggridge, 2012).

Product performance

Product performance presents the needs that define functional aspects of the product and are presented in Table A.1.

Table A.1 - Requirements, product specifications, metrics and rank of product performance.

	Requirements	Product specifications	Metric	Rank
1	**Dirt and impurities removal**	Cleanser activity	Subjective	5
2	**Durable**	Time	Days	4
3	**Easy removal of lather**	Water solubility	g / mL	4
4	**Efficient dissolution when in contact with water**	Water solubility	g / mL	5
5	**Good skin adherence**	Viscosity	Pa.s	4
6	**Mildness**	Type of surfactant	subjective	5
7	**Non-irritating to the skin**	Concentration	$mg_{surfactant}$ / $g_{product}$	5
8	**Skin moisturisation**	Concentration of moisturisers	$mg_{moisturiser}$ / $g_{product}$	5
9	**Smooth-feel sensation**	Type of surfactant	subjective	3

The idea of the product classifies as a proper and functional cleanser, but one that brings innovation to the market. Ergo, its cleanser activity would have to be ranked at 5, it could not be irritating to the skin, otherwise it could be dismissed by consumers. In terms of the extra benefits, it was thought to be of importance the mildness of the syndet and the added moisturisation effect, both ranked with 5. The efficient and quick dissolution when in contact with water was also mandatory.

Product perception

In the cosmetic world, product perception is an important factor, because cosmetics are chosen initially by how they are perceived, how they look and feel, so the needs regarding this factor are presented in Table A.2.

Table A.2 - Requirements, product specifications, metrics and rank of product perception.

#	Requirements	Product specifications	Metric	Rank
10	**High quality**	-	subjective	5
11	**Pleasant fragrance**	Fragrance	Subjective	5
12	**Smooth lathering**	Superficial tension	Pa	4
13	**Smooth paper like texture**	-	subjective	3
14	**Solid consistency**	Friction	N	4
15	**Thin sheet**	Thickness	mm	3
16	**Variety of colour**	Colour power	A	4

High quality is a need imposed by the brand Castelbel, being as it is one of the brand ideals – to create luxury products in an ethical, but affordable way – so it was ranked with

5. The other rank 5 of this category would fall to the category of pleasant fragrance, seeing as it is a handwasher, consumers tend to enjoy a mild and pleasant smell. Fragrance-free products are usually associated with medical or baby products, as they sometimes are considered allergens, so in order to construct a high-end product the fragrance plays a very important part (Barel et al., 2009). It would be important in this case to mention one od the needs that are ranked 4 – smooth lathering is an important specification, because consumers usually assume (wrongly) that a large quantity of lather indicates a better cleansing action, so a smooth and in a nice quantity will make the consumer feel more fulfilled (Redding, 2012).

Commercialisation

This section goes hand-in-hand with the product perception in terms of how successful a product shall be in regard to selling. The needs of commercialisation are presented in Table A.3.

Table A.3 - Requirements, product specifications, metrics and rank for commercialisation.

#	Requirements	Product specifications	Metric	Rank
17	**Aesthetically pleasing packaging**	Marketing	Subjective	5
18	**Competitive cost**	Price	Euros (€)	5
19	**Easy to use**	Packaging	-	5
20	**Eco-friendly**	Packaging and formulation	-	5
21	**Small packaging (portable)**	Volume	mL	5
22	**Variety of colour and fragrances**	Marketing	Subjective	4

Small, portable, and easy to use packaging are the essence for the product, since its existence is based on solving a convenience issue of having a handwasher product on the go or in a public place. So, both these needs received a ranking of 5.

A competitive cost is imperative in today's market (ranked 5), in order to not be consumed by other cheaper products that may offer some variation of the same characteristics. At the same time it must show a respectable price, to build trust in consumers and to align with the Castelbel identity – affordable luxury products. Consumers are starting to pay more for creditable and eco-friendly products, so if the product shows the characteristics to be valuable to the consumers, the price can be increased, hence, the ranking of 5 in eco-friendly characteristics (Yao et al., 2019). These eco-friendly characteristics, both in packaging and in formulation, are presented, not only, to appeal to the consumer, but because it is a company worry – Castelbel is starting to think "greener" in order to help the environment.

Aesthetically pleasing packaging is a maximum (5) qualified requirement, implemented by both the brand and the cosmetic market it will be commercialised in. Also,

aesthetically pleasing packaging and a variety of both colours and fragrances are strategies of marketing, to appeal and call to as many consumers as possible. The cosmetic and the personal care product market is known for its beautifully and well thought out packages. Also, one of Castelbel unique selling propositions – the attention to the detail and the creative process with the intention of creating beautiful products.

Legislation

All the needs presented in this category (in Table A.4) have to present a rank of 5, otherwise, it is more likely that the product will not be approved. The product must be safe for both the user and the environment, being compatible with its contact surface (the skin) and never exceeding the legal limits of the components' concentration. The label needs to present these legal limits regarding the countries where the product will be commercialised, and it also needs to present the expiration date of the product after being opened.

Table A.4 - Requirements, product specifications, metrics and rank for legislation.

#	Requirements	Product specifications	Metric	Rank
23	**Dermatologically compatible with skin**	-	-	5
24	**Expiration date**	Time	Months	5
25	**Legal limit for components**	concentration	$mg_{surfactant}$ /$g_{product}$	5
26	**Label**	-	-	5
27	**Nontoxic**	-	-	5

Appendix B - Equipment used throughout the work

Equipment used during laboratorial scale stage

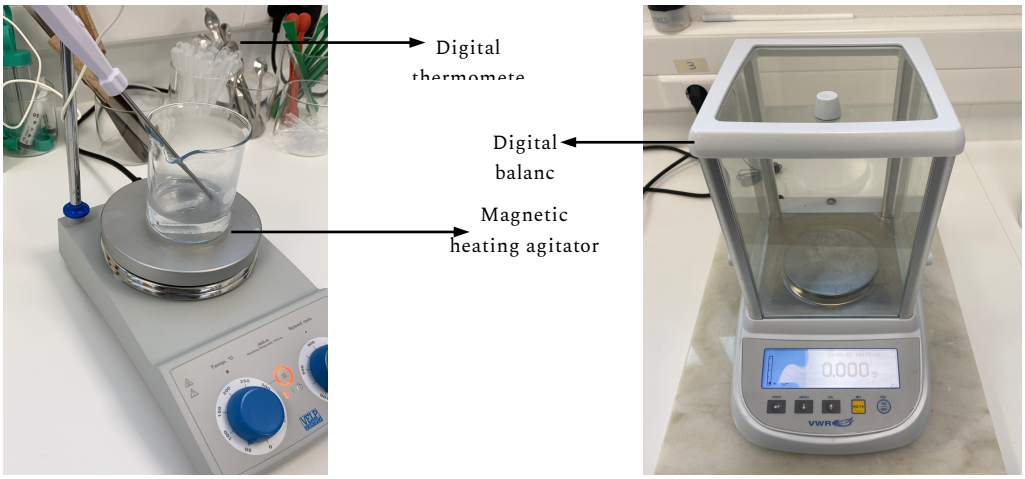
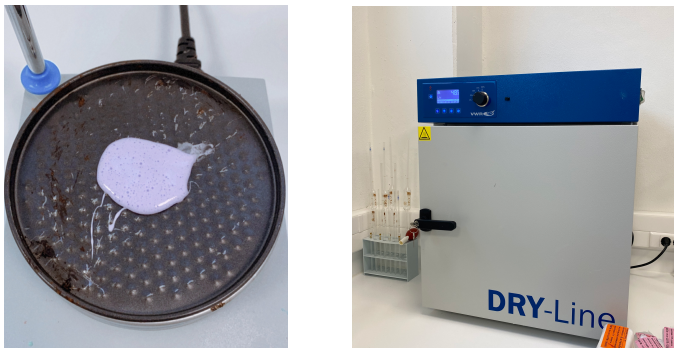


Figure B.1 - Laboratory set up with the magnetic heating agitator, digital thermometer and balance.



a **b**

Figure B.2 – (a) the first drying method used - heating plate (with product from assay 5). (b) the drying oven used throughout the project.

Equipment used during the pilot scale stage

Figure B.3 - Equipment used during the scale-up stage.

Appendix C - Performed laboratory assays

C.1 PVA Testing

Table C.1 - Results obtained from PVA testing step with the first formulation.

Assay		1	2	3	4[1]
Type of PVA		4 - 88	40 - 88	18 - 88	26 - 88
Reagents	%	g			
Water	59	59.520	59.045	59.041	29.556
PVA	9	9.048	9.034	9.030	4.519
SLS	8	8.019	-	8.048	4.005
Glycerine	9	9.554	-	9.043	4.525
Coco Diethanolamide	6	5.981	-	6.023	3.073
Corn Starch	8	8.065	-	8.032	4.028
Fragrance	1	1.035	-	1.015	0.503
Pigment	-	0.015	-	0.080	0.056
Total	100	101.237	-	100.312	50.27

[1] to optimise the use of the samples, not all of the assays produce 100 g of the liquid mixture.

C.2 Adjustments to the formulation

Table C.2 - Results obtained from adjustments to the formulation of step 1.[1]

Assay	5		6		7		8		9		10		11	
Type of PVA	18 - 88		18 - 88		26 - 88		26 - 88		26 - 88		22 - 88		22 - 88	
Reagents	%	g	%	g	%	g	%	g	%	g	%	g	%	g
Water	62.00	31.080	60.00	30.070	62.00	31.046	62.00	31.116	62.50	15.660	62.50	31.323	62.50	62.529
PVA	6.00	3.041	6.00	3.021	6.00	3.129	5.00	2.543	5.00	1.259	5.00	2.502	5.00	5.026
SLS	8.00	4.059	8.00	4.014	8.00	4.038	8.00	4.013	8.00	2.054	8.00	4.032	8.00	8.084
Glycerine	9.00	4.530	9.00	4.518	9.00	4.577	9.00	4.507	9.00	2.266	9.00	4.537	9.00	9.072
Coco Diethanolamide	6.00	3.038	6.00	3.085	6.00	3.006	6.00	3.06	6.00	1.5	6.00	3.068	6.00	6.069
Corn Starch	8.00	4.088	10.00	5.110	8.00	4.045	9.00	4.521	8.50	2.148	9.00	4.469	8.50	8.511
Fragrance	1.00	0.498	1.00	0.505	1.00	0.523	1.00	0.418	1.00	0.163	1.00	0.453	1.00	0.325
Pigment	-	0.084	-	1.044	-	0.020	-	0.011	-	0.009	-	0.033	-	0.072
Total	100.00	50.42	100.00	51.37	100.00	50.38	100.00	50.19	100.00	25.06	100.00	50.417	100.00	99.69

[1]The colours at the bottom of the table represent the colours representative of the assays (colour of the liquid mixture and dried product)

C.3 Surfactant testing

Table C.3 - Results obtained from testing the different surfactant samples.

Assay		12	13	14
Type of Surfactant		Pureact (SCI)	Oramix (sarkosyl)	Eversoft (sarkosyl)
Reagents			g	
Water	**62.50**	31.252	31.271	31.265
PVA	**5.00**	2.501	2.501	2.517
Surfactant	8.00	4.052	4.07	4.032
Glycerine	9.00	4.559	4.533	4.537
Coco Diethanolamide	6.00	3.074	3.078	3.068
Corn Starch	**8.50**	4.251	4.255	4.254
Fragrance	1.00	0.196	0.190	0.200
Pigment	-	0.047	0.423	0.033
Total	100.00	49.932	50.321	49.91

[1]The colours at the bottom of the table represent the colours representative of the assays (colour of the liquid mixture and dried product)

C.4 Adjustments to the formulation

Table C.4 - Results obtained from the alterations to the formulation with the chosen surfactant.

Assay	15		16		17		18	
Reagents	%	g	%	g	%	g	%	g
Water	62.00	31.038	62.30	31.168	62.80	31.422	62.86	31.341
PVA	5.00	2.540	5.00	2.510	5.10	2.553	5.00	2.502
Pureact (SCI)	8.00	4.088	8.00	4.025	8.00	4.084	8.00	4.015
Glycerine	9.00	4.594	9.00	4.536	9.00	4.513	9.00	4.513
Coco Diethanolamide	6.00	3.044	6.00	3.021	6.00	3.052	6.00	3.028
Corn Starch	9.00	4.515	8.70	4.355	8.60	4.313	8.64	4.324
Fragrance	1.00	0.545	1.00	0.240	0.50	0.259	0.50	0.217
Pigment	-	0.014	-	0.111	-	0.023	-	0.403
Total	100.00	50.38	100.00	49.97	100.00	50.22	100.00	50.34

[1]The colours at the bottom of the table represent the colours representative of the assays (colour of the liquid mixture and dried product)

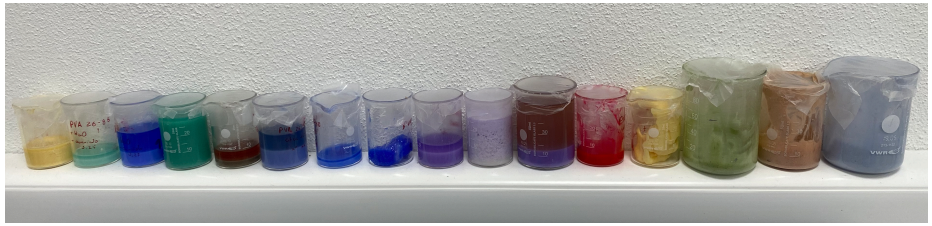


Figure C.1 - Some examples of liquid mixtures assays

Appendix D – Quantitative and Qualitative evaluation

D.1 Resulting products from stage 1 – laboratory scale

Quantitative evaluation

The entire qualitative evaluation of the laboratory assays is presented in the following tables D.1, D.2 and D.3. It is worthwhile noting that the final ranking is not a simple average of the given ranking to the features; dissolution has a higher weight in determining if the product valid than, for example, the aroma. Also, the feature "oil or colour release" will only be considered when it differs from 10, the norm has been assumed to not release neither oil nor colour.

Table D.1 - Quantitative evaluation of assay 1 to 11.

Parameters		Features	Assays										
			1	2	3	4	5	6	7	8	9	10	11
Liquid mixture		Viscosity	0	5	5	4	5	6	6	7	9	9	10
Appearance	*Texture*	Plastic finish	0	-	0	0	0	2	3	5	8	8	8
		Oil/colour release	10	-	10	10	10	10	10	3 [3]	10	10	10
		Smoothness	0	-	4	4	2	2	4	8	9	8	8
		Uniformity	0	-	3	3	2	2	3	8	9	9	5
		Final ranking	0	-	2	2	1	2	3	7	9	8	8
	Structure	Tear resistance	[1]	-	0	1	2	2	3	6	8	7	5
		Plastic structure	0	-	0	0	0	2	2	4	6	8	4
		Elasticity	0	-	0	2	1	2	3	4	6	7	6
		Final ranking	0	-	1	1	1	2	3	5	7	7	5
Performance		Dissolution	0	-	0	0	0	1 [2]	2	6 [2]	8	7	10
		Lather	0	-	0	0	0	1	1	4	7	8	10
		Foaming duration	-	-	-	-	-	0	0	3	7	8	10
		Mildness	-	-	-	-	-	1	0	3	3	5	5
		Aroma	-	-	-	-	-	0	1	5	7	6	6
		Final ranking	**0**	**-**	**0**	**0**	**0**	**1**	**3**	**5**	**7**	**7**	**10**

[1] the product had no cohesion, crumbles were formed.

[2] the product dissolved, but not completely forming a starchy mixture left on the hand.

[3] colour release, it is caused by the chosen pigment. The red pigment has been found to have this effect. It should not compromise the analysis of the product at a high level.

Mildness in this particular stage of the work, will not be considered to influence the performance a great deal, since the used surfactant SLS will be replaced with an alternative.

Table D.2 - Quantitative evaluation of assay 12, 13 and 14.

Parameters		Features	Assays		
			12	13	14
Liquid mixture		Viscosity	7	1	1
Appearance	*Texture*	Plastic finish	5	0	0
		Oil/colour release	10	10	10
		Smoothness	5	4	4
		Uniformity	2	0	0
		Final ranking	4	1	1
	Structure	Tear resistance	5	0	0
		Plastic structure	5	0	0
		Elasticity	5	0	0
		Final ranking	5	0	0
Performance		Dissolution	5	0	0
		Lather	3	0	0
		Foaming duration	2	-	-
		Mildness	6	-	-
		Aroma	5	-	-
		Final ranking	**5**	**0**	**0**

Table D.3 - Quantitative evaluation of the assays 15 through 18.

Parameters		Features	Assays			
			15	16	17	18
Liquid mixture		Viscosity	5	7	9	10
Appearance	*Texture*	Plastic finish	8	8	6	9
		Oil/colour release	10	5 [2]	10	10
		Smoothness	8	10	6	10
		Uniformity	8	10	7	10
		Final ranking	8	9	7	10
	Structure	Tear resistance	9	5	8	10
		Plastic structure	10	6	6	9
		Elasticity	5	8	7	9
		Final ranking	8	8	7	10
Performance		Dissolution	8 [1]	9	9	10
		Lather	9	9	9	10
		Foaming duration	9	9	9	10
		Mildness	9	9	9	9
		Aroma	7	10	10	10
		Final ranking	**8**	**9**	**9**	**10**

[1] the product dissolved, but not completely forming a starchy mixture left on the hand.

[2] oily feeling during the first few hours of the product, seemed to dry off.

In the table D.4 the qualitative evaluation of the best assays from each section is presented.

Table D.4 - Quantitative evaluation regarding the best assays from each stage.

Parameters		Features	Assays		
			11	12	18
Liquid mixture		Viscosity	10	7	10
Appearance	*Texture*	Plastic finish	8	5	9
		Oil/colour release	10	10	10
		Smoothness	8	5	10
		Uniformity	5	3	10
		Final ranking	8	4	10
	Structure	Tear resistance	5	5	10
		Plastic structure	4	5	9
		Elasticity	6	5	9
		Final ranking	5	5	0
Performance		Dissolution	10	5	10
		Lather	10	3	10
		Foaming duration	10	2	10
		Mildness	5	6	9
		Aroma	6	5	10
		Final ranking	**10**	**5**	**10**

Qualitative evaluation of the dried products.

Table D.5 - Qualitative evaluation of products.

Assay	Appearance	Assay	Appearance
1	Soft and paper like	10	Soft, paper structure, but thin plastic film
2	Plastic crumbles	11	Soft & smooth; Paper structure
3	-	12	Soft & smooth; Paper structure
4	Plastic but with an elastic feeling	13	Paper feeling ; Thin plastic film
5	Less plastic film	14	Completely plastic; Shiny appearance
6	Plastic film	15	Completely plastic; Shiny appearance
7	Plastic film	16	Very soft & smooth; Fragile paper like
8	Thinner plastic film; softer feeling	17	Very soft & smooth; Fragile paper like
9	Thinner plastic film	18	Soft & smooth; Fragile paper like

D.2 Resulting products from stage 2 – pilot scale

Quantitative evaluation of the dried products.

Table D.6 - Quantitative evaluation of the final product.

Parameters		Features	Final Assay
Liquid mixture		Viscosity	9
Appearance	*Texture*	Plastic finish	10
		Oil/colour release	10
		Smoothness	10
		Uniformity	10[1]
		Final ranking	10
	Structure	Tear resistance	9
		Plastic structure	10
		Elasticity	10
		Final ranking	10
Performance		Dissolution	10
		Lather	10
		Foaming duration	10
		Mildness	9
		Aroma	10
		Final ranking	**10**

[1] even though, the sheets were not completely uniform in relation to each other, the sheets were uniform throughout itself

Appendix E - Questionnaire

To better understand the answers to the questionnaire the graphics and the data collected from it will be presented.

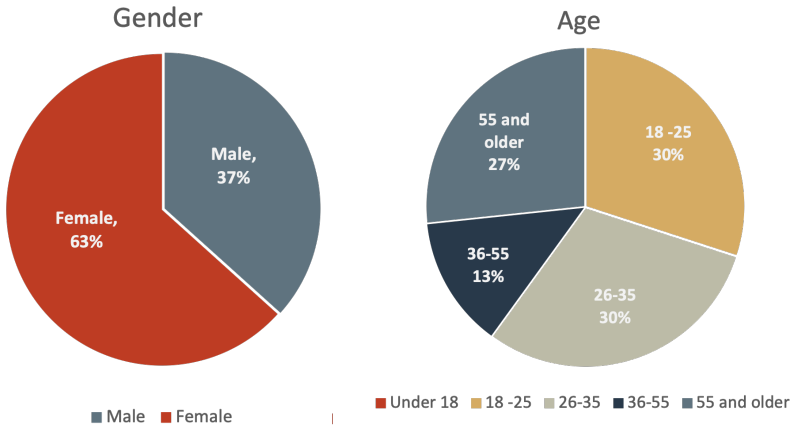


Figure E.1 - a) Gender. (b) Age.

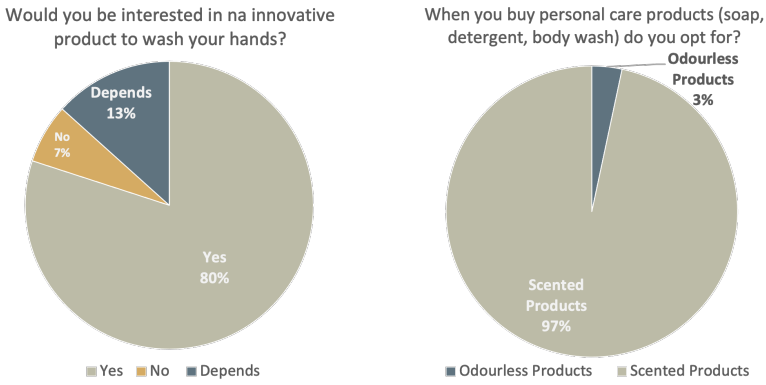


Figure E.2 - Fourth and Fifth question of the questionnaire, regarding willingness and preferences.

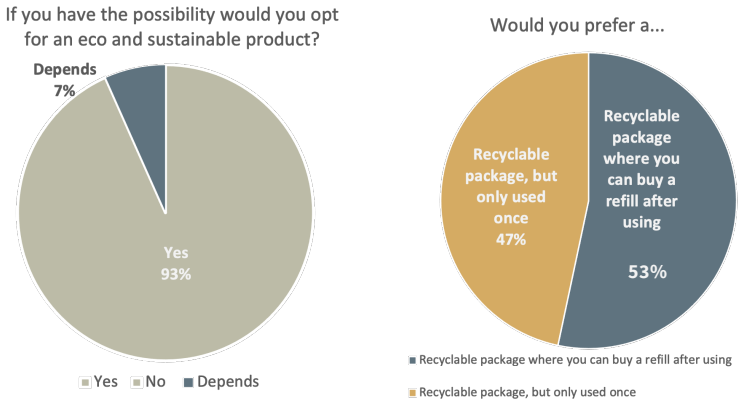


Figure E.3 - Questions regarding eco-friendly and sustainable products.

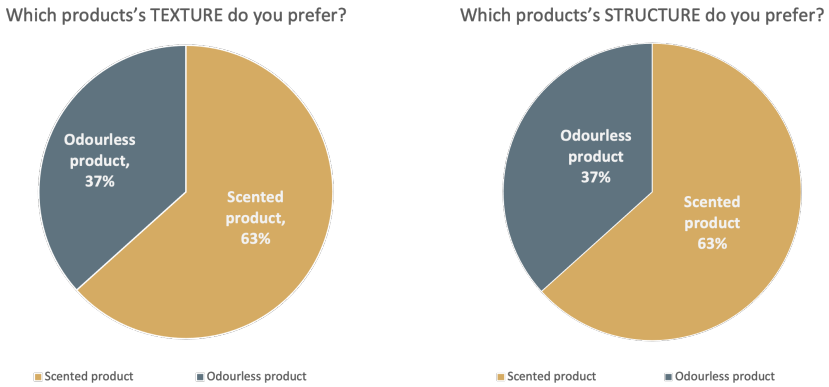


Figure E.4 - Texture and Structure preferences.

Table E.1 - Opinion of the study population regarding structure and texture.

Structure		Texture	
É mais macio	O produto com perfume não fez espuma.	É mais firme	leveza e toque
Desfaz-se mais rapidamente	a lavagem é mais suave, faz mais espuma	Produtos sem fragrância, dá-me sempre a sensação que não lavam. Claro que lavam, mas fica essa sensação.	A textura é mais macia e desfaz-se com mais facilidade, tornando-se por isso mais hidratante.
It was easier to grab and use.	Because the scented ones, gives a feeling of comfort and cleanliness.	Very soft	I think it is more pleasant.
pareceu mais macio	It's softer	Mais fácil de dissolver	Apesar do sem cheiro ser mais fácil de pegar, gosto mais da estrutura flexível
A textura da folha com perfume era demasiado pegajosa. Em embalagem será mais difícil de separar	A fragrância é mais durável e perdura nas mãos	Mais rigido. O sem odor parece que posso rasgar sem querer. O com odor parece mais resistente.	It´s less soft
Desfaz-se melhor que o azul	It was softer	Only because it smells good.	leveza e toque
Dissolve mais facilmente na água e fez mais espuma.	I like the perfume that´s in the hands	Because it was easier to melt	I think it is more pleasant.
Mais agradável ao toque	O produto com perfume não fez espuma.	Tive a impressão que agarra melhor à pele.	A textura é mais macia e desfaz-se com mais facilidade, tornando-se por isso mais hidratante.
They behave similarly, reason why I prefer the one with scent		Apesar de gostar também da estrutura do produto com cheiro, a outra parece me mais fácil de pegar por ser mais resistente, mas obviamente que dependerá do tipo de embalagem em que o produto será comercializado	Apesar do sem cheiro ser mais fácil de pegar, gosto mais da estrutura flexível

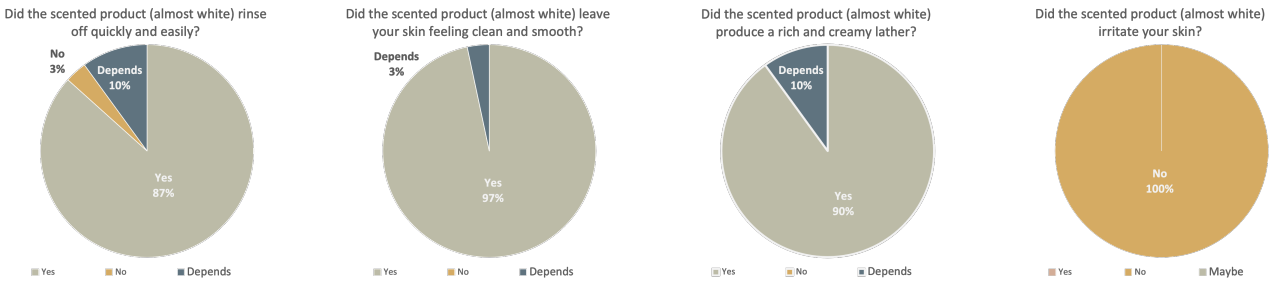


Figure E.5 - Preference regarding performance.

Table E.2 - Opinion regarding performance.

Rinsing	Cleanliness	Lather
Demorou mais tempo a dissolver-se, tive que fazer mais fricção.	When washing my hands I did not think about it	Precisa fazer um pouco mais de espuma
Demorou um bocado a começar a sair e depois saiu bem e rapidamente		
Não fez espuma.		Precisa fazer um pouco mais de espuma
It took a lot of time to dissolve		

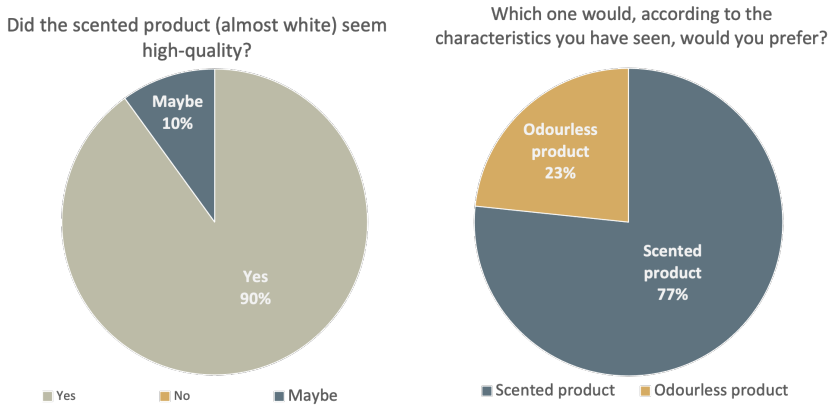


Figure E.6 - Answers regarding the perception and preference of the product.

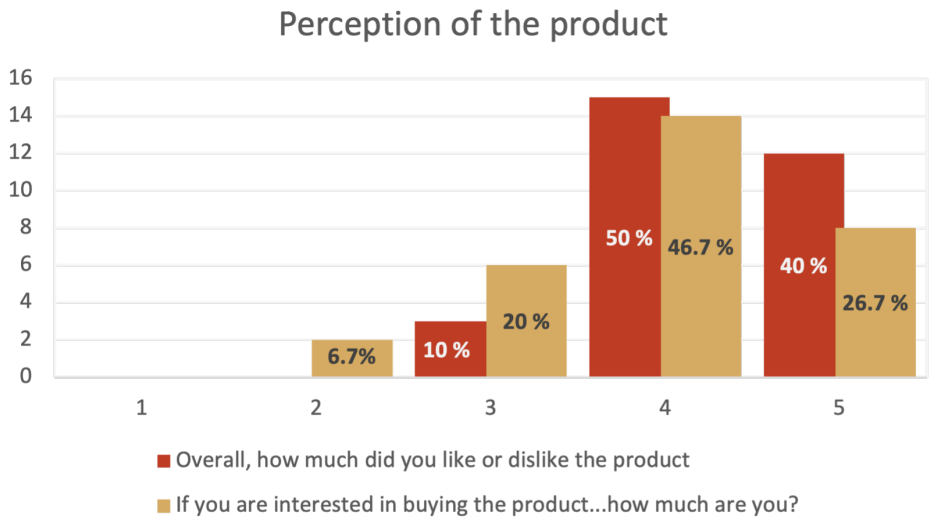


Figure E.7 - Perception and preference of the developed product.

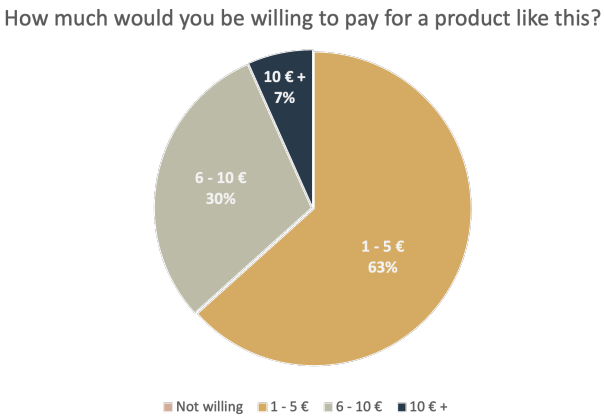


Figure E.8 - How much the study population is willing to pay for the developed product.

Table E.3 - Suggestion given by some elements of the study population regarding possible improvements or changes.

Suggestion for improvements or changes
A estrutura
gostei mais de lavar as mãos com o produto sem fragrância. Porque me pareceu mais macio/ suave... gostei do branco, mas tendo o outro para comparar gostai mais.
Apenas fazer um pouco mais de espuma
Cheiro a lavanda!
Gosto bastante da sensação cremosa e suave que ficam as mãos depois de usar. Também gostei do cheiro por ser simples e nada muito exagerado. Em termos de cor e forma, não me faz grande diferença.
Due to hand shape, I would prefer a round, square or similar shape
Gostaria de ver o produto com outras fragrâncias e cores, penso que seria ainda mais apelativo ao consumidor. apesar de preferir uma estrutura mais rígida, gosto bastante da sensação macia do produto, parece um produto muito suave que irá ser bom para as minhas mãos
I would prefer Sea Salt Fragrance.
Cheiro um pouco mais intenso
Cores mais vivas e com outros diferentes
Using attractive perfumes like Sea Shore

Appendix F - Market pricing

All the data was gathered from the Amazon website, with the search of "paper soap products". The Table F.1 represents the various products and respective prices, and the Figure F.1 is the compiled information.

Table F.1 - Competitors pricing.

Brand name	Number of sheets	Price (€)	Price / sheet (€)	Price / 50 sheets (€)
Akudy	**100**	**7.5**	**0.075**	3.75
Borsali	250	7.58	0.03	1.52
Coleman	50	7.43	0.149	7.43
FaSoLa	100	4	0.04	2.00
Fomin [1]	**100**	**6.59**	0.066	**3.30**
Heyna Q	120	8.34	0.07	3.48
Jidue	300	8.43	0.028	1.41
Kisser	200	8.1	0.041	2.02
Macellani	270	15.5	0.048	2.42
Mathilde M [1]	**70**	**9.51**	0.136	**6.79**
Mistic Cool	300	8.43	0.028	1.40
Rechiato	800	16.02	0.02	1.00
Sankin	300	8.43	0.028	1.40
Wishdiam	400	10.12	0.025	1.27
ZDU	80	5.06	0.063	3.16

[1] these two products are the closest in the market, regarding the developed product

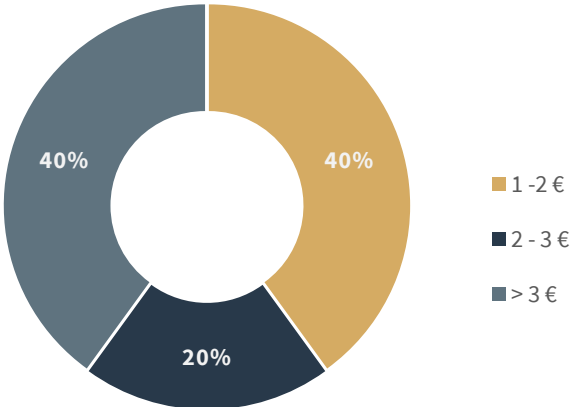


Figure F.1 - Percentage of price interval, per 50 sheets.

Appendix G - Economic analysis

G.1 Production cost

Table G.1 - Economic analysis of the product regards the raw materials.

Reagents	Percentage (%)	Reagent price (€/kg)	Price for 1 kg of product (€/kg) [2]
Water	62.86	1.60	1.006
PVA	5.00	3.00	0.150
SCI	8.00	11.87	0.950
Glycerine	9.00	0.90	0.081
Cocamide DEA	6.00	1.60	0.096
Corn starch	8.64	4.98	0.430
Fragrance	0.50	21.00	0.105
Pigment	as needed [1]	100.00	0.249
TOTAL	100	-	3.06 [3]

[1] value used was estimated using the various assays performed during this work. It was determined that for 1 kg to obtain a light colour 2.40 g of pigment might be needed.

[2] this value is attained by multiplying the percentage per 1 kg, determining the quantity of each product that is used in 1kg. Then by using the quantity reagent and multiplying it per price/kg (respective) and finally summing each value.

[3] this value obtained was the sum of the price per kg of the reagents.

G.2 Number of packages per kg of product

Afterwards it was assessed the number of packages produced by using 1 kg of product. The number of syndet units per package (fifty) had to be defined and the dimensions of each unit (2 x 4 cm). With this relation, the area of each sheet (Figure G.1), the area of each unity and the relation of 8 kg product to 60 packages, it was possible to estimate the number of *7.5 packages* per one kilogram of product. The process used was as following.

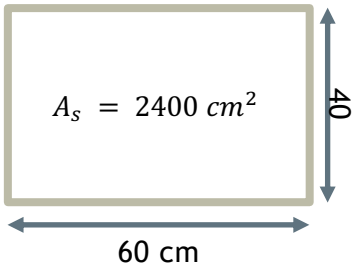


Figure G.1 - Representation of paper syndet sheet and its measurements.

Form the characteristics of the equipment it was possible to determine that the main roller is 80 cm in diameter and 60 cm. However, the sheets, when removed from the winding roller, are cut in half, forming rectangular sheets with 40 (*w*) x 60 (*l*) cm. Knowing that the chosen dimension to the unity of the product was 2 (*w*) x 4 (*l*) cm and that each package will have fifty applications it was possible to calculate the area correspondent to the product of one packaging.

$$A_u = 50 \times (2 \times 4) = 400\ cm^2 \quad \text{(G.1)}$$

From the final assay of the pilot testing, it was possible to obtain about 10 sheets from a total of 8 kg. However, it should be noted that there was a waste of about 1 to 2 kg of the liquid mixture (errors in the process and product accumulation). Therefore, these calculations are just suppositions. Each sheet will have, approximately, an area (A_s) of 2400 cm^2. With that this author was able to determine (equation G.2) how many packages for the 10 sheets, the 8 kg, it would be possible to obtain.

$$\frac{A_s}{A_u} = \frac{2400 \times 10}{400} = 60\ packages \tag{G.2}$$

With this value and creating a proportion between 8 kg and 1kg it was possible to determine that 7.5 packages could be attained from the production of 1 kg of product.

Having attained the weight of 50 units of the product with the defined measurements, amounting to 14 g, and having calculated the value of how much it costs to produce 1 kg of product, it is possible to determine how much will cost per package recurring to equation G.4.

$$€/package\ = 3.06\ \times\ \left({}^{14}/_{1000}\right) = 0.0428\ \approx 0.04\ € \tag{G.4}$$

G.3 Final price

To attain the real price of the product the information regarding how much product would be produced in a year, the price of manual labour for that production and, finally, the price of packaging would be needed. However, at this point of the work it was not possible to establish any of these factors. It was advised to try and estimate the value. Generally, the value of the manual labour is assumed to be 25 - 50 % the cost of production per package. The products cost is too low for a valid estimative, so the price of the packaging will be used. Since the manufacturing process was quite lengthy and laborious the manual labour estimative was 50 % of the packaging cost. Plus, there is a need to consider the safety factor of 10 %. Two external factors were considered: the average competitive price determined by the study, which would amount to 4.62 €; and the answers to the questionnaire, which showed that more than half of possible consumers are not willing to pay more than 5.00 € for the product. Having done a quick survey of the market for possible cork packaging prices and having asked for a quotation, it was possible to hypothesise the value of **2.05 €** per package. The calculation is presented in Equation G.5 and G.6.

$$0.04 + 2.05 + 2.05 \times 0.5 = 3.11\ € \tag{G.5}$$

$$3.11\ \times\ 1.1 = 3.421\ \approx \mathbf{3.42}\ € \xrightarrow{{}^{1}/_{50}} \mathbf{0.07}\ €\ per\ unity \tag{G.6}$$

Therefore, the suggested basis for the final price (estimative) should be **3.42 €**.