



# Treball Final de Grau

**Development of several multipurpose facial creams (BB, CC, DD creams) and the process for their manufacture.**

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*Pasa menos tiempo frente al espejo y más tiempo sintiéndote increíble.*

Frédéric Fekkai

En primer lugar, me gustaría dar las gracias a mi tutora Alicia Maestro Garriga por su gran ayuda, dedicación y apoyo en todo momento.

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## **SUMMARY**

Cosmetics are formulated products that have emerged to meet the needs of society. In its synthesis process there are some stages with greater relevance such as product conceptualization, definition of quality criteria, ingredient's selection and design or selection of the equipment for its manufacture.

Cosmetics were already used by Egyptians, Greeks and Romans, but it was not until the nineteenth century, and specially until the twentieth century, when cosmetic industry experienced a significant growth that reflected the importance given to the personal image. This great change has led to the development of different improved cosmetic creams that make us look better and feel more confident about ourselves. In addition, it should be noted that, over time, society seeks to reduce their steps in its daily beauty routine, so that more and more creams have ingredients with different objectives that make the same cream have different functions in the same application. Because of this, three types of multipurpose facial creams have been developed: BB, CC and DD creams. The first is intended for young skin that wants to keep their skin hydrated, the second for more mature skin with a touch of anti-aging and the last for more mature skin with greater sun protection and anti-aging effects.

The design of the manufacturing process has been carried out for an annual production of 4,000 kg of cream. For this production, some ingredients have been formulated, with their approximate quantities and quality criteria, and a process diagram with the necessary equipment.

**Keywords:** Formulated product development, cosmetics, make-up, facial cream, emulsion, process synthesis.



## RESUMEN

Los cosméticos son productos formulados que han surgido para satisfacer las necesidades de la sociedad. En su proceso de síntesis hay etapas con una mayor relevancia como la conceptualización del producto, la definición de los criterios de calidad, la selección de los ingredientes y el diseño y selección de los equipos para su fabricación.

Los cosméticos ya fueron utilizados por los egipcios, griegos y romanos, pero no fue hasta el siglo XIX, y especialmente hasta el siglo XX, cuando la industria cosmética experimentó un crecimiento importante que reflejó la importancia que se da a la imagen personal. Este gran cambio ha provocado el desarrollo de diferentes cremas cosméticas mejoradas que hacen vernos mejor y sentirnos más seguros de nosotros mismos. Además, cabe destacar que, con el paso del tiempo, la sociedad busca reducir los pasos en su rutina diaria de belleza, por lo que cada vez más las cremas llevan ingredientes con diferentes objetivos que hacen que una misma crema tenga diferentes funciones en una misma aplicación sobre la piel. Debido a esto, se han desarrollado tres tipos de cremas multifuncionales faciales: BB, CC y DD creams. La primera va destinada a pieles jóvenes que quieren mantener hidratada su piel, la segunda a pieles más maduras con un toque de anti-edad y la última a pieles más maduras con mayor protección solar y efectos antienvjecimiento.

El diseño del proceso de fabricación se ha llevado a cabo para una producción anual de 4,000 kg de crema. Para esta producción se han formulado unos ingredientes, con sus cantidades aproximadas y sus criterios de calidad, y un diagrama de proceso con los equipos necesarios.

**Palabras clave:** Desarrollo de productos formulados, cosméticos, maquillaje, crema facial, emulsión, síntesis de procesos.



# 1. INTRODUCTION

The cosmetic term, from the Greek word *Kosmetikós*, with meaning of good order or ornament, was created in the 17th century, although its first use took place in Ancient Egypt, about 4000 BC, when Romans and ancient Greeks began using these substances. During their round, cosmetics have acquired different functions depending on the influence of cultural and religious traditions (*González and Bravo, 2017*).

In Europe, from Renaissance to the beginning of the 20th century, the use of cosmetics was only intended for those women who worked in cabarets, those who worked in prostitution and rich women. During that time, there was a big difference between the upper class and the lower class because the latter was engaged in outdoor agricultural work, so, because of sun exposure their skins darkened and deviated from the tonality of European upper class. This class with greater wealth had the pleasure of not working under the sun and could afford to stay at home all day so they presented a paler face. This difference in status caused many European men and women to try to clear their skins using white powders or white lead paint that could contain arsenic. They wanted to maintain the typical European pale face and appear more aristocratic (*González and Bravo, 2017*).

European Regulation 1223/2009 (*Garrote, 2019*) defines cosmetic products as: *any substance or mixture intended to be put in contact with the superficial parts of the human body (epidermis, hair and hair system, nails, lips and external genital organs) or with teeth and the oral mucous membranes, with the exclusive or main purpose of cleaning them, perfuming them, modifying their appearance, protecting them, keeping them in good condition or correcting body odors*. On the one hand, cosmetic term includes makeup, deodorants, shampoos, shower gels..., everything that is applied topically. On the other hand, any product that can be inhaled, ingested or injected into the human body and/or has as its objective the cure of any disease is not included in the cosmetic concept.

In addition, according to Ullman's Encyclopedia of Industrial Chemistry (*Wiley-VCH, 2002*), cosmetics, depending on their use, can be divided into different groups: hair care, skin care and

maintenance, odour improvement, cleansing, hair removal, care and maintenance of mucous membranes and decorative cosmetics. This work will be focused exclusively on skin care and maintenance.

With the time, and after having come a long way, cosmetics have been developing different applications that can go from highlighting eyes with shadow palettes to hide undesirable pores; in any case, the main purpose of cosmetics is to improve beauty of humans, specially the face, and achieve a younger and healthier appearance. These have experienced great growth in the global market because of society's concern for its image and for enhancing beauty, so they have become a fundamental part of humans' live, specially of women.

Until the end of twentieth century, cosmetics were divided into different creams (nourishing, moisturizing, solar, anti-wrinkle ...) which each performed its function separately:

- **Nourishing creams** are responsible for providing lipids and maintain the protective barrier function of the skin. Lipids are fillers located between the cells that are responsible for retaining water in the skin (*F.Martel, 2010*).
- **Moisturizers** are the ones that provide water to the skin in order to keep it hydrated. They protect the skin from external factors such as wind, cold, sun ... (*Hernández-Barrera et al., 2011*).
- **Sun creams** have the function of preventing the penetration of ultraviolet rays into the skin. No cream of this type offers 100% protection but there are different intensities, from an SPF (Sun Protection Factor) of 15 to 50 plus (*Mejía et al., 2013*).
- **Wrinkle creams** are responsible for exfoliating the skin in order to remove dead cells present in the skin's superficial layer and give it a healthy appearance and a natural glow (*Muneerah et al., 2018*).

With passage of time and new trends, these creams came together and formed a single cream that would do all the functions described above. These multipurpose creams are those known as BB, CC and DD Cream depending on the main function desired.

Until recently, women lost a considerable time of good morning putting products in order to protect or hide imperfections of their faces, in addition to the large number of products that accumulated in their cosmetic counters. But all this changed when multipurpose creams offering

an all-in-one solution for your multi-step skin care in the morning. All creams discussed above were united in a same cream that did all the joint functions.

The first marketing term, which was BB cream, was developed in 1960 by the German scientist and dermatologist Christine Schrammek (*Sennebogen, 2012*). This cream was created to protect, conceal spots, calm and moisturize her patients' skin after medicine treatments such as surgery and facial peels.

Although its beginnings date back to the 60s, it was not until 1980 when, thanks to Korean cosmetics of South Korea and Japan, it became popular and made the leap to the big market by promoting some Asian actresses who went from being applied seven facial products daily to be applied all in one with a perfect result: obtain a porcelain skin. At that time, because the cream was intended specially for Asian skins, the variety of tones was very scarce, so it was hardly sent to other countries; but all this changed when American cosmetics introduced this type of cream into their markets and expanded the variety of skin tones in 2012. After causing a furore in Korea and some countries in Asia, this boom was subsequently moved to Europe and, finally, to Western countries. Although American beauty came from Asian, both brands differed in the variety of skin tones and their functionality on the skin. On the one hand, the Asian market developed thicker creams since they wanted perfect skin with the same skin tone, while the US market developed a purer cream because they wanted a healthy and free appearance with a radiant finish (*Sennebogen, 2012*).

After the appearance of these BB creams, intended for young skin to moisturize, other descending creams of these appeared, CC and DD creams. These new creams included improvements such as increased hydration, sun protection and anti-aging effect, which made them intended for more mature skin that wanted to get perfect skin.

### **BB CREAM**

BB Creams, named *Blemish Balm Cream* or *Beauty Balm Cream*, are creams intended for young people who start in the world of makeup. These 3-in-1 multipurpose facial creams (foundation, primer and moisturizer) reduce these 3 steps by 1, so, they are an all-in-one daily basis. These creams have as main functions: moisturize, protect the skin from the sun, illuminate, hide imperfections and smooth the skin. These creams act mainly on the upper layer of the skin (epidermis).

### **CC CREAM**

CC Creams, named *Colour Correcting Cream* or *Complexion Corrector Cream*, are creams arising from the evolution of BB Creams, so they have greater advantages than these. CC Creams, closer to a makeup base, are intended for more mature skin with the main objective of correcting anti-aging effects and imperfections and hide skin spots and redness, in addition to protecting the skin from sun damage with greater extent than previous. The effects of aging are usually the result of factors such as excessive exposure to the sun that leads to precipitated aging of the skin, which is known as photoaging. These creams are more straightforward, have a lighter body and texture and have a more powerful coverage than BB Creams.

### **DD CREAM**

DD Creams, named *Daily Defense Cream* or *Dynamic Do-All Cream*, are a combination of BB and CC Creams intended for mature skin because, unlike the previous ones, these ones contain antioxidants that act as a protective barrier of the skin preventing damage and aging of the skin in order to keep it young. It is impossible to stop aging, but what can be done is to slow it down with certain active ingredients that make aging more pleasant. In addition to this, other important functions of these creams are to moisturize, protect the skin (they are multipurpose facial creams with greater sun protection, can reach 50 SPF), unify the skin tone and hide imperfections and redness.

Thanks to its fast effectiveness, more and more, these creams have been making room in the routine of many women.

With this innovative concept, women stopped having thousands of cosmetics at the time of makeup and went on to have a single cosmetic that did all the functionalities they wanted in the same application.

The greatest concern of society today is to look good with oneself and improve their self-esteem to obtain greater social, warlike or loving success.

The objectives of the work and the development of the creams discussed will be presented next.



## 2. JUSTIFICATION AND OBJECTIVES

As already mentioned, BB, CC and DD creams appeared and revolutionized society as they also reduced the routine time of putting on makeup, they also expanded the space in the cosmetic counters of many women. These new all-in-one products, which allowed different functions to be made in one application, were a boom and allowed people to look younger, healthier and happier in less time.

The main objective of this work is the development of a variety of formulated multipurpose facial creams (BB, CC, DD creams). This work will include all stages in product development, from product conceptualization to the process synthesis for product manufacturing, including the design and/or the selection of equipment for their manufacture. The development will be divided into the 5 steps necessary for the formulation process:

**Product conceptualization:** In this first chapter, a market analysis will be carried out through encyclopaedias, patents, scientific articles and books to detect possible customer requirements or market trends, that is, to know the needs of the consumer and place products to be developed in an advantageous way over existing products. In addition to the needs, a study of the products to be developed will be carried out defining the functionality, form of application, structure and packaging.

**Identification of product quality factors:** After conceptualizing the products defining their main characteristics, will be identified the quality factors that will ensure the good functionality of products. These factors have the technical objective of covering the needs described above and are quantified by the quality indexes that will ensure the quality of the product developed.

**Selection of ingredients:** In order to the products to exercise their main functions, active components, excipients or application vehicles and additives that optimize the quality indices defined above will be selected. The structure is already defined, an emulsion; the type of surfactant will be defined.

**Process synthesis for product manufacturing:** Knowing the ingredients, the operations necessary for the manufacture of the desired products will be selected. Once the operations are known, a process of obtaining the product will be synthesized.

**Designing and selection of equipment:** In this last chapter, a production will be selected and for this production the necessary equipment will be designed and selected.

### 3. PRODUCT CONCEPTUALIZATION

As mentioned previously and according to *Wibowo (2002)*, the first step in developing a new product is conceptualization or definition of this. It is important to know consumer needs in order to try to satisfy them and maintain effective and optimal manufacturing processes in time and costs.

The first aspect to consider is the definition of the physical form of the product. According to *Wibowo 2001*, depending on the end use, products are delivered in different physical forms. The division made in the article "*Product-Centered Processing: Manufacture of Chemical-Based Consumer Products*" (*Wibowo, 2002*) classifies creams, which are formed by immiscible liquids and possible small solid particles, in the physical form of semi-solids: typical form of the products that are applied in the human body.

The product conceptualization is divided into three important sections:

**Market trends and consumer needs:** in this section a study of market trends will be made to know consumer needs through the following steps:

- Interviewing customers
- Interpreting their needs
- Translating needs into product specifications

**Product functionality:** here the main functions of the different creams developed will be defined.

**Product packaging:** in this last section of conceptualization, the form in which the product will be delivered will be selected.

### 3.1. MARKET TRENDS AND CONSUMER NEEDS

The European cosmetic industry investigates consumer preferences and needs in order to innovate those products and bring to the market a new improved product. Innovation, a constant process that includes investigation of consumer behaviour, beauty aspirations, new formulations, skin studies and improvements in sustainable development, can take more than 5 years because, every year, a quarter of all cosmetic products on the market are improved or are completely new. Because of all this, the cosmetics industry has a great role in product development (*Cosmetics Europe, 2016*).

The main consumer trends can be divided into three aspects: consumer wants and needs, product safety and legal and environmental issues. In the first one, consumer wants and needs, it is said that the product should be smaller in size, easy to carry when travelling, last longer and/or cost less, its use should be a pleasurable experience and functions should be combined in one product (*Wibowo, 2002*). Currently, according to the consumer's last preference, most cosmetic products have become mixtures of various ingredients with defined structure since they are more attractive to them.

Regarding to the product safety, it should not contain toxic or allergenic ingredients and dangerous chemicals for little children, and it should contain more natural ingredients (*Wibowo 2002*). Presently, most beauty companies are adding natural ingredients to their products, although they continue using some synthetic chemical products because these ingredients are what give the product its long shelf life and obtaining natural ingredients quickly and economically is not always possible.

Finally, environmental and legal issues are less documented in relation to the dangers that these health products can cause. According to *Wibowo (2002)*, it is preferable that the product would be biodegradable and be more environmentally friendly. Packaging is the main cause of environmental impact, so all those skin care packages must be created with materials that are not degraded by products that have been designed to contain (*Cesta, 2018*).

Creams are usually emulsions, a heterogeneous unstable dispersed system formed by two immiscible liquids, in which one of the phases is dispersed in the other. The liquid in which the droplets are dispersed is named continuous or external phase, while the liquid present as droplets is called dispersed or internal phase. Moreover, some solid particles can be suspended in the continuous phase. The preparation process is called emulsification and the agents necessary for

its preparation, stabilization and properties modification are emulsifiers. Emulsifiers are known as surfactants, emulsion promoters and stabilizers (*Wiley-VCH, 2002*).

The two most common types of emulsions in cosmetics are:

**Water in oil (W/O):** water is the dispersed phase and oil is the continuous phase. The surfactant should be lipophilic.

**Oil in water (O/W):** oil is the dispersed phase and water is the continuous phase. The surfactant should be hydrophilic.

In addition to these two types of emulsions, there is a third type, less usual for its instability and complexity, known as multiple emulsion which can be water-in-oil-in-water (W/O/W) or oil-in-water-in-oil (O/W/O) emulsion (*Wiley-VCH, 2002*).

The most common type of emulsion for skin care products is O/W emulsion since there is a higher proportion of water, so its formulation is much cheaper, and they are usually less greasy than the other type of emulsion.

Emulsions are thermodynamically unstable, so their phases, sooner or later, will tend to macroscopic separation. There are two types of emulsions depending on the droplet size:

**Macro-emulsions:** emulsions with a droplet size between 1 and 100  $\mu\text{m}$  in diameter (*Morales, 2009*).

**Nano-emulsions:** emulsions with a droplet size of 20 to 200 nm in diameter and a bluish-translucent colour. (*Chavda, 2019*).

On the other hand, micro emulsions are also known but, although they contain the noun emulsion, they are not emulsions since they are thermodynamically stable systems formed by particles of a few nm. Due to this size, micro emulsions are considered an intermediate between micelles and very small droplet emulsions (*Wiley-VCH, 2002*).

The rheological behaviour of creams is determined by the microstructure of the product which is also determined by the rheology of continuous phase, the volumetric fraction of phases and the drop size distribution of the dispersed phase.

The microstructure of a product also affects other performance indices such as softness, opacity and stability. An emulsion, intended for cosmetic or pharmaceutical purposes, with a

droplet size between 1  $\mu\text{m}$  and 20  $\mu\text{m}$  has greater softness, stability and may become transparent (Wibowo, 2001).

The type of emulsion not only depends on the volumetric phase of each phase and the particle diameter, but also influences the preparation. Depending on the order and the way the phases are added, there is an area known as ambivalence area in which both a water-in-oil emulsion and an oil-in-water emulsion can be obtained (Wibowo, 2001).

In conclusion, the properties of the final product, such as microstructure of the product and appearance, are determined by different aspects such as formulation, cutting speeds, order of addition of the phases, preparation conditions, desired drop size and fraction phase volume. So, the emulsions that form the developed creams are going to be O/W macro-emulsions with a drop size close to 20  $\mu\text{m}$ .

## 3.2. PRODUCT FUNCTIONALITY

After having done the market research and knowing market trends, the second step in product conceptualization is to define the main function or functions of the facial creams developed in this work.

Being a product that is applied to the skin, it is interesting first to make a brief explanation of some topics to understand some concepts later.

### 3.2.1. Skin

The cosmetics developed in this work are applied to the skin, so before going deeper into some aspects, it is interesting to have some knowledge of the surface on which the product is applied.

The skin, a barrier between humans' bodies and environment, is the largest organ of human body since it represents approximately 15% of adult body weight and covers an area of 2  $\text{m}^2$  (Kolarsick *et al.*, 2010). This organ is formed by three sub-layers and has four main functions:

- Moisture and prevent the loss of vital components for the body, specially water
- Thermoregulation: body temperature regulation
- Sense of touch

- Protection against chemical, biological and physical agents

It is also important to know the structure of the skin. This organ is subdivided into three sub-layers that act as a barrier to the environment and allow the transmission of sensory information: epidermis, dermis and hypodermis (*Figure 3.1*) (*Afriat, 2010*). *Appendix 1: Layers of skin* contains more information about each layer.

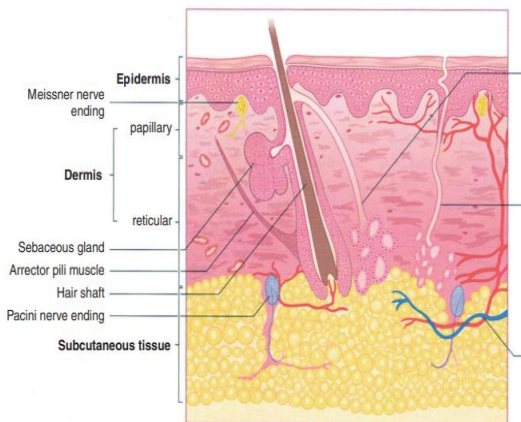


Figure 3.1. Cross-section of skin  
(*Kolarsick et al., 2010*).

### 3.2.2. UVA/UVB

Photobiology is the science which studies the effects of solar rays on humans since these rays reach the earth's surface and can cause harmful effects on the skin of humans, such as photoaging, photodermatitis, immunosuppression, and even, they can cause serious health problems such as cancer.

The Sun emits electromagnetic radiation, the set of which is called electromagnetic spectrum, characterized by their frequency and wavelength. Most radiations are retained by ozone, but some reach the earth's surface and do so in the following proportion:

- Visible light: 50%
- Infrared: 40%
- Ultraviolet: 10%

This last radiation, which reaches the earth's surface in a small proportion, is responsible for light dermatoses, a skin rash caused by sun exposure.

The energy of these radiations is inversely proportional to their wavelength, so those of shorter wavelengths will be the most energetic. Ultraviolet radiation is classified into 3 energy bands (*Lipsky and German, 2019*):

**UVC:** (200 - 290) nm → the most harmful and luckily absorbed by oxygen and the ozone layer.

**UVB:** (290 – 320) nm → composes 5% of ultraviolet radiation that reaches the earth's surface (*Bernard et al., 2019*). Can damage the DNA of cells and is the main causes of burns and skin cancers.

**UVA:** (320 – 400) nm → composes 95% of ultraviolet radiation that reaches the earth's surface (*Bernard et al., 2019*). Rays associated with long-term damage such as wrinkles and have a role, but less important, in the onset of cancer.

Humans have a natural protection on their skin against ultraviolet light that protects them from the damage they can cause to the skin. Protection mechanisms act in two different ways:

**Absorbing radiation:** they stand out at the epidermal level. Some examples are: urocanic acid, DNA, RNA, tryptophan and melanin.

**By deflecting/reflecting radiation:** they stand out at the dermis level and, among them, blood haemoglobin, tissue bilirubin and fat beta-carotene stand out.

Each person presents different effects on the skin with the same sun exposure since not everyone tolerates harmful effects of ultraviolet radiation in the same way, mainly since the content of melanin is different for each person. Increasingly, tanning is becoming a mark of social well-being and a symbol of beauty, but concern about the effects of sunrays is still present, so many cosmetic products are currently including sunscreens to reduce the effects of the sun on the skin. This situation deviates greatly from that of the late twentieth century in which people liked to get brown and did not take any precaution for harmful effects of the sun.

There are two types of filters according to their composition: chemical filters (aromatic organic molecules that absorb radiation energy wavelength ultraviolet spectrum) and physical filters (inorganic pigments that reflect or deflect radiation forming an opaque barrier that acts as a screen, so they offer greater protection against solar radiation).



### 3.2.3. Creams

BB, CC and DD creams are considered an intermediate cosmetic between a moisturizer and a makeup base because they are all-in-one creams that aim to care, moisturize and uniformize skin tone, correct imperfections, provide lighter coverage respect other bases and protect the skin from sun's rays. But all these benefits will disappear once these creams are no longer applied since these types of creams only act on the most superficial layer of the skin, the epidermis, so skin will return to its initial state when they stop applying.

These creams have different shades of colour according to the skin prototype since not everyone has the same skin tone. The tones they present are:

- Low: aimed at people with a paler skin
- Medium: aimed at people with a medium skin tone, neither too pale nor too dark
- High: aimed at people with a darker skin tone, with greater melanin

It has been decided that the creams to be developed in this work will have same colour tone, although, later, in the manufacturing process, the 3 colour tones for each type of cream will be considered. The decided tone has been the medium tone since it is the most common in Spanish people.

First, and before explaining the main functions of creams, it is important to define the concept of sun protection factor (SPF). The SPF, introduced in 1962 because of the importance that was given to the protection of skin from the sun, is a number that indicates the time that a person can be exposed to the sun without having risk of burns (*Surber and Reinau, 2015*).

In this section there will be explained the main functionalities of the creams developed in this work:

#### **BB CREAM**

The BB Creams to be developed are intended for young girls with young skin without or with slight imperfections. The main objective of these creams is to moisturize and refresh the skin while offering a touch of colour and natural luminosity.

Moisturizers have become a primary cosmetic for basic skin care since they are responsible for restoring the protective barrier function of the most superficial layer of the skin, the epidermis, increasing the water content in this outermost layer.

The epidermal water content is important for skin plasticity (*Kraft, 2005*) since water originates in the deeper epidermal layers and is transported upwards in order to hydrate the cells in the stratum corneum, although it is finally lost by evaporation.

Moisturizers have the functions of restoring the skin barrier, increasing the water content, reducing transepidermal water loss (TEWL), repairing the capacity of lipid barriers and maintaining the integrity and appearance of the skin acting as moisturizers, emollients and occlusives (*Kraft, 2005*).

There is currently a great dilemma with the following terms: humectants, emollients and occlusive, since there is a lot of confusion because of the absence of an exact and precise definition for each of them. After an exhaustive search they have been defined as follows:

**Emollients:** lipids and oils responsible for moisturizing and improving the lubrication of the skin getting better the flexibility and softness of the skin (*Kraft, 2005*). These substances, lipophilic in nature and with a chemical structure of alcohol or ester, are responsible for the final touch of the skin once applied since they are responsible for providing softness, elasticity and occlusion. There are different types of emollients (*BOT, 2019*).

**Occlusive:** they create a hydrophobic barrier on the skin that reduces TEWL. The main limitations are smell, allergenicity and fatty sensation (*Kraft, 2005*). They are substances which are added to the oil phase in order to increase water retention by forming an occlusive film on the skin's surface. They offer a hydrated, soft and flexible stratum corneum (*BOT, 2019*).

**Moisturizers:** hygroscopic substances that retain water in the stratum corneum and improve water absorption from the dermis to the epidermis. These substances prevent the drying of cosmetic products thanks to their hygroscopicity and facilitate the solubility of active ingredients in the aqueous phase (*BOT, 2019*).

In addition, BB Cream incorporates sunscreens that protect skin from sunrays with a minimum sun protection factor, SPF of 15, and additives that will give coverage to the cream and allow an attractive tone for the skin's face.

### **CC CREAM**

The CC Creams have more functions than the previous ones since they are an evolution and improvement of the latter. Unlike the previous ones, these creams are intended for more mature skin with greater imperfections such as pimples, spots, redness and wrinkles so they will present different functions to those mentioned above (*Marin, 2013*).

The CC Cream, in addition to allowing the same benefits as the previous creams, improves the appearance of skin thanks to the formulation of anti-aging ingredients that uniform the skin. These benefits, so that they are seen in long term, must be linked to a continuous use of the creams since as it has been commented, these creams act in the most superficial layer of the skin, so the disuse of these creams causes the disappearance of its benefits (*Martin, 2019*).

Like the previous creams, these also have a hydration function, but unlike the previous ones, in this case, the hydration factor is greater since these creams are aimed at more mature skin that have a greater loss of hydration. In addition, these creams offer greater coverage, so they have more similarity to a makeup base.

These creams have more sun protection than the previous ones, an SPF of 30, since they are aimed at more mature skins that need more sun protection.

This formulation increases hydration while camouflaging imperfections in mature skin such as blackheads, pores, dark circles and wrinkles. The purpose of these creams is to achieve a bright, flexible and radiant skin.

These creams have a great use in the period of pregnancy of women since, during this period, between 50% and 70% of women have a skin disorder that causes the appearance of dark spots on the skin, specifically in the facial face (*Vázquez, 2013*).

This cutaneous alteration is caused, for example, by the hormonal change that women suffer during pregnancy, resulting in hyperpigmentation, specially if exposition to sun is important (*Azcona, 2006*). The most common type of hyperpigmentation is melasma, irregular dark spots that manifest on the facial face. These spots may slowly disappear after the birth of the baby, or they may remain, an event that occurs in 30% of women (*Vázquez, 2013*). One of the main treatments for melasma, according to AEDV (*Spanish Academy of Dermatology*), is the use of makeups that have built-in sunscreens since sun exposure favours the appearance of these spots, and the disuse of treatments that irritate the facial face (*Vázquez, 2013*).

## **DD CREAM**

DD Creams are intended for mature skin. The main difference of these creams regarding the previous ones is that they contain antioxidant ingredients with anti-wrinkle properties that prevent damage and aging of the skin.

Currently, presenting healthy and perfect skin is one of the most important aspects for society, so, in recent years, anti-aging creams have been developed to prevent a deteriorated facial appearance. These creams fight against the progressive and apparent changes of the facial parts of skin exposed to the sun.

Aging is a change in the skin related to environmental factors, genetics, nutrition, hormonal processes and other factors such as lifestyle. Of all the factors, the most important is sun exposure since there is a large difference between parts of the body exposed to the sun with protected areas (*MedlinePlus, 1997*).

Aging is one of the first wrinkle formation factors due to changes in connective tissue that reduce the resistance and elasticity of the skin, as well as a reduction in collagen and elastin (*Abbas et al., 2018*). With aging there is a thinning of the outermost layer of the skin, the epidermis, which causes the skin to appear thinner, pale and translucent.

Chronological aging is caused by the appearance of free radicals. They are unstable oxygen molecules that produce modifications in DNA. DNA damage is related to aging.

DD Cream, like the previous ones, have a sun protection factor incorporated. In this case, the protection factor is higher: SPF of 50. These creams are intended for consumers with an age from 40 years forward, those skins that have greater imperfections and need more sun protection to maintain a good appearance (*Sierra, 2015*).

## **3.3. PRODUCT PACKAGING**

The last step, but not least, of product conceptualization, is the definition of its packaging. Packaging, which includes both the delivery of the key ingredients and the product, is an important aspect in conceptualization because it may affect consumer's perception of the product (*Wibowo, 2002*).

Increasingly, ecological packaging is having greater weight in purchasing decisions. An environmentally friendly packaging based on the three Rs (reuse, recycle, reduce) enhances

shelf-appeal and highlights the product of the competition. This packaging is what is known today as "green packaging" (*SpecialChem, 2018*).

The two most important aspects for the attractiveness of a product in the market are quality and general appearance of it. Within the latter is the packaging that is an important part in brand and marketing of cosmetic products. Both the material and the shape, colour and durability of the packaging have a great role when it comes to marketing a product. In addition, packaging should be easy to brand as it must have printed the name of the product, as brand and other important information such as composition, instructions and necessary warnings (*Shivsharan and Shaikh, 2014*).

There is a wide variety of packaging materials according to the cosmetic product, although the most used are glass, metals and plastics. The latter can be flexible (tubes) or non-flexible (jars). The following table (*Table 3.1*) shows different types of packaging, with the material, according to the consumer product in question (*SpecialChem, 2019*).

Table 3.1. Typical packaging for Chemical-Based Consumer Products (*Wibowo, 2002*).

Type of Packaging	Composite	Tablet/capsule	Powder/Granule	Cream/Paste
<i>Wrapping</i>				
Carton box	✓	✓	✓	
Paper/Plastic wrap	✓	✓		
Aluminum foil	✓	✓		
<i>Bag (Paper/plastic)</i>				
Rescalable bag		✓	✓	
Sealed bag/sachet		✓	✓	✓
<i>Bottle (Glass/Plastic)</i>				
Screw cap		✓	✓	✓
Flip cap				
Slit orifice				
Pump top				
<i>Tube (Metal/Plastic)</i>				
Collapsible Tube				✓
Squeezeable tube				✓

According to the previous table and the *SpecialChem* article *Trends in Cosmetic Packaging* (*SpecialChem, 2007*), the best packaging for this type of creams is collapsible tube for its price, its portability and its ease of use and carry. Within this type of packaging, different shapes will be formulated, from square to more round, and sizes, from 25 mL to 250 mL, depending on consumer's preference. Also, the tube must allow the product to come out, but not to come into to avoid possible contamination so the closure which is a critical component in package will be screw cap.

The material for the chosen packaging should be selected according to the use and needs of the cosmetic, therefore, it should give a good appearance and be compatible with the product. The most used material for cosmetic containers is plastic and some of its advantages are:

- Ease with which they can be formed
- High quality
- Wide variety of designs presented
- Resistant to breakage, so they offer safety to consumers
- Low in cost and light in weight
- Inert to many chemicals
- Pleasant to touch

Plastic containers are made from different polymers with specific additives (colors, stabilizers, antioxidants...), but the most common type of plastic is polypropylene (PP) since it can easily acquire different shapes and satisfy the consumer with their preferred styles. However, these can also be made of PET or a high-end acrylic plastic, transparent and glass-like acrylic plastic, although with greater resistance to breakage (*SpecialChem, 2015*).

For these three types of multipurpose facial creams developed in this work it has been decided that the best packaging is screw cap collapsible polypropylene tube for its resistance to breakage because many women always carry these creams on top. These containers will have a volume of 40 mL for easy handling and transport (*Figure 3.2*).



Figure 3.2. Collapsible polypropylene tube  
(*SpecialChem, 2015*)

### 3.4. PRODUCT CONCEPTUALIZATION SUMMARY

The creams to be developed will be O/W macro-emulsions since, as mentioned above, they provide less fatty sensation and are cheaper in general because of their greater proportion of water. In addition, 80% of emulsions that go on the market are oil in water emulsions (*Kontogeorgis and Kiil, 2016*). These emulsions will have a droplet size close to 20  $\mu\text{m}$  because they offer greater stability. They will be formed by mixtures of ingredients that will provide the cream with different functions since consumers prefer those products manufactured by different ingredients that combine all their functions in the same product. In addition, these ingredients will be as natural as possible since they do not harm the environment and are better accepted by consumer.

The following table (*Table 3.2*) shows the main functions and characteristics of the creams developed in this work. The examples developed in this work have medium colour tone, but in the manufacturing process the three possible colour tones will be considered.

Table 3.2. Functions and characteristics of each cream.

FUNCTIONALITY	
<b>BB CREAMS</b>	<ul style="list-style-type: none"> <li>- SPF: 15</li> <li>- Refresh</li> <li>- Moisturizing</li> <li>- Tone: Medium</li> <li>- Young skin</li> </ul>
<b>CC CREAMS</b>	<ul style="list-style-type: none"> <li>- SPF: 30</li> <li>- Refresh</li> <li>- Moisturizing</li> <li>- Tone: Medium</li> <li>- More coverage</li> <li>- Intermediate skin</li> </ul>
<b>DD CREAMS</b>	<ul style="list-style-type: none"> <li>- SPF: 50</li> <li>- Moisturizing</li> <li>- Tone: medium</li> <li>- Mature skin</li> <li>- Anti-aging factors: antioxidants</li> </ul>

The table below (*Table 3.3*) shows the main characteristics of the cream packaging and the main information that all cream packaging is printed on.

Table 3.3. Packaging design and printing.

<b>PACKAGING FOR ALL CREAMS</b>	
<b>Design</b>	<b>Printed</b>
<ul style="list-style-type: none"> <li>- Collapsible tube of polypropylene</li> <li style="padding-left: 20px;">- Size: 40 ml</li> <li>- Manageable</li> <li>- Easy to carry</li> <li style="padding-left: 20px;">- Durable</li> <li>- Economical packaging</li> <li style="padding-left: 20px;">- Attractive</li> </ul>	<ul style="list-style-type: none"> <li>- Name of the cream (BB, CC or DD Cream)</li> <li style="padding-left: 20px;">- Brand</li> <li style="padding-left: 20px;">- Instructions for use</li> <li>- Type of tonality: low/ medium/ high</li> <li style="padding-left: 20px;">- Warnings of possible dangers</li> <li style="padding-left: 20px;">- Composition</li> <li style="padding-left: 20px;">- Fabrication place</li> <li style="padding-left: 20px;">- Tube size</li> </ul>



## 4. IDENTIFICATION OF PRODUCT QUALITY FACTORS

After having conceptualized the product knowing consumer needs, market trends and product functions, it is necessary to define the quality factors that will allow correct product functionality. Product quality is the central theme and is closely linked to the manufacturing process (*Wibowo, 2002*).

According to *Wibowo (2001)*, although functionality is always the first important aspect when defining a product, there are some quality factors such as ease of application, stability and appearance that are crucial in consumer satisfaction. For example, in the purpose of the cream there are some important considerations like texture of the product, stability of the product, since it must be stable for more than a year, or more commonly 24 months, considering possible changes in external temperature, humidity and presence of direct sunlight and the convenient use; all these aspects will influence consumer's choice.

Quality factors will change depending on product form or delivery system. For instance, in this case, rheology is going to be an important factor to consider since this work is focused on emulsions. Most of these quality factors aim to meet consumer needs and maintain the functions of the products. They are usually qualitative factors, so quality indexes are defined to measure the performance of the product through these factors while reflecting the level of satisfaction of the use of the product.

There are different ways of measuring according to the quality factors that are going to be studied. On the one hand, sensorial quality factors are measured using an arbitrary scale related to the properties of materials and structure. Instead, mechanical quality factors are quantified using physical properties such as tensile strength, melting point and viscosity. On the other hand, there are also some that are measured by dimensionless numbers (*Wibowo, 2001*).

The main quality factors will be linked to the main functions of creams. It should be borne in mind that, for a band, BB Cream moisturizes while offering colour to the skin in order to cover

slight imperfections and protects the skin with a sun protection factor 15 from harmful effects of the sun. On the other hand, CC Cream aims to hide redness and spots while moisturizing, covering and protecting the skin from sunlight with a SPF 30. Finally, DD Cream prevents the appearance of wrinkles thanks to the presence of antioxidant ingredients while protecting the skin from sunlight with a SPF 50 and offering coverage. In addition to these quality factors, there are others which should be specially careful.

#### 4.1. FUNCTIONAL QUALITY FACTORS

The first quality factors to be explained will be the functional quality factors that, as the name itself indicates, are related to the main function of the product.

In response to the product developed, a cream is expected to protect the parts of the body where it is applied, to provide protective or decorative coverage and, in addition, cause an adhesion to a surface that in this case will be the skin of the face. In the studied cases in this work, the most important functional quality factors are the protection of facial features while offering both protective and decorative coating as they offer ranges of colours to match skin tone. In addition, consumer expects the cream to be absorbed quickly and provide its function immediately.

**Visual appearance:** factors related to the brightness and colour of the product on the package and specially on the skin. To this type of creams, colouring agents are added to fulfil one of its functions, offering a tone coverage like that of the skin tone where it is applied. These colouring agents can be classified into dyes or pigments. The most used in cosmetic products are dyes since they are synthetic organic and soluble in both water and oil (*Knowlton and Pearce, 1993*). As already mentioned, the three types of creams will have three different shades, but the three creams developed as examples in this work will have the same colour tone: medium tone.

These quality factors, like decorative coating and colour tone, can be measured by developing questionnaires aimed at consumers in which they are asked about the satisfaction they have had when applying and using these products.

The sun protection quality factor, related to SPF (Sun Protection Factor), can be measured in two ways. In the past, the most common method was the test known as “in vivo” which allowed to measure the degree of sensitivity that skin had against sun exposure. The test consisted of the sun exposure of individuals in some parts of the body, usually the back since it was the part of

the body less exposed to the sun during the year (Costa *et al.*, 2007). In this test the SPF was determined through an experiment in which DEM of the skin without sun protection and DEM of the skin with protection were obtained, and were related as follows [1] (Costa *et al.*, 2007):

$$SPF = \frac{DEM \text{ protected skin}}{DEM \text{ unprotected skin}} \quad [1]$$

However, this method was being replaced by a more current and effective one. Currently, the SPF measurement method is the use of the UV transmittance analyzer. This powerful device has the function of calculating sun protection value for both UVA and UVB. This is achieved by measuring radiation transmittance through a sample of product (solar, cosmetic ...) in a region of wavelength from 250 nm to 450 nm. This equipment is widely used by the cosmetic industry as it serves as a research, development and quality control (Labsphere, 2008).

## 4.2. RHEOLOGICAL QUALITY FACTORS

Rheological quality factors are very important when working with emulsions. It is essential to relate the selected requirements to the rheological requirements since they are the ones that will allow a good application on the surface where the product is going to be applied.

For one band, the viscosity of the product affects energy consumption and mixing time and, on the other band, the fluidity of the product when applying it plays a great role in consumer perception because consumers look for creams that can be poured and spread easily on the skin (Wibowo, 2001).

It is important that the creams have a pseudoplastic behaviour: they must have a low viscosity at high shear, so they flow easily when rubbed on the skin, and the viscosity should increase at low shear so they do not pour easily as if they were a liquid, that is, they should not flow under gravity, but they should be easy to pour. In addition, they should give a uniform coating when applied to a surface.

To offer some viscosity values that would have to have this type of creams, it has been decided to do an experiment. A BB Cream accepted by the market has been taken and its rheological quality factors have been measured. A sample of cream has been placed on the rheometer, working at a temperature of 24 degrees, and the variation of its viscosity has been

observed at different shear rates over time (*Figure 4.1*). In blue the data of the experiment are represented, both in logarithmic scale, and in red the trend line is represented.

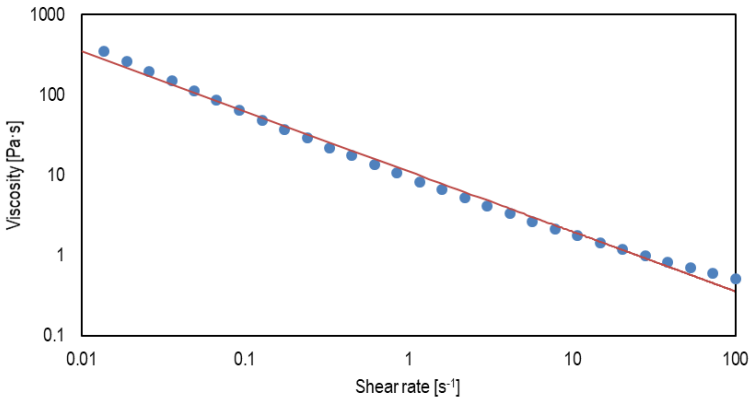


Figure 4.1. Viscosity variation at different shear rates.

The line obtained is what is known as rheogram, which represents the equilibrium viscosity vs. shear rate. This behaviour can be done through the Ostwald-de Waele model, or also known as the power law which has the expression [2]:

$$\tau = K \cdot \gamma^n \quad [2]$$

K is the consistency index and n the behaviour index. Representing shear stress vs. shear rate as indicated by the expression, it is obtained that the creams developed in this work must have a **K** of  $(11 \pm 2)$  Pa·s and a **n** of  $(0.25 \pm 0.05)$ .

For creams, being non-Newtonian fluids, it is important to know the viscosity at the actual shear rates experienced. Some examples found in the article by *Wibowo (2001)* are (*Table 4.1*):

Table 4.1. Typical shear rates in processing and application of creams (*Wibowo, 2001*)

Action	Shear rate $\gamma$ (s <sup>-1</sup> )
Suspending pigment or active ingredients	$10^{-3} - 10^{-1}$
Extrusion from a bottle or tube	$10^3 - 10^1$
Topical application of lotions/creams	$10^2 - 10^4$

The viscosity that must be presented by the creams developed in this work must have values like those represented in *Figure 4.1*. On the other hand, a group of panelists have found that cosmetic emulsions considered good have a low viscosity of approximately 0.025 Pa·s when

applied to the skin at a high shear rate of approximately  $5,000 \text{ s}^{-1}$  for creams. At very low shear rates the viscosity can be as high as  $1,000 \text{ Pa}\cdot\text{s}$  (Wibowo, 2001).

In addition, the minimum yield stress that must be made in the creams developed to obtain a good application is  $(6 \pm 1) \text{ Pa}$  approximately. This value can be obtained from *Figure 4.2* through the experiment developed. As it can be seen in the figure, from that value the shear rate increases rapidly, so it causes a decrease in viscosity and a good application on the skin.

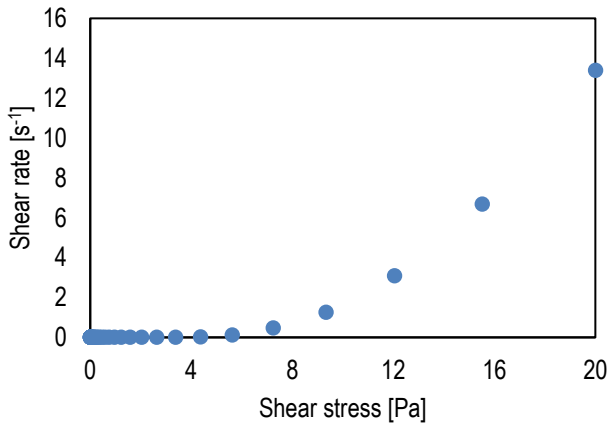


Figure 4.2. Determination of yield stress.

In conclusion, all creams are applied in the same way on the skin so they must have a similar rheological behaviour and the main properties are (*Table 2: Examples of Typical Quality Factors for Creams and Pastes, Wibowo, 2001*):

- The cream should be easy to pour (performance index: viscosity at application shear rate)
- Ease of spread on the skin when applying (performance index: viscosity at application shear rate)
- The cream should present obstacles to flow under gravity and should be easy to stir (performance index: yield value)
- The cream should offer uniform coverage when applied to the skin (performance index: minimum thickness at which even coating is observed)
- The cream must be able to be removed from the packaging, but must not flow by itself (performance index: yield value)

### 4.3. EMULSION STABILITY QUALITY FACTORS

As already mentioned in product conceptualization section, the creams to be developed in this work are emulsions, dispersed systems formed by two immiscible liquid phases in which one phase is dispersed within the other in the form of tiny drops that may have different sizes.

Two types of emulsions are distinguished depending on the properties of the phases. These two emulsions can be water in oil (W/O: water disperses in oil) or oil in water (O/W: oil disperses in water). In this case, the creams developed in this work are oil-in-water emulsions (O/W).

Emulsions are unstable systems because of differences in phase properties. To stabilize emulsions, the presence of a third component, an emulsifier or emulsifying agent, which is related to both phases is required. The main functions of emulsifiers are to keep both phases together for a long period of time, facilitate emulsification and promote emulsion stability.

Emulsifiers, which are formed by a polar and a non-polar end, produce water-in-oil emulsions if they are more soluble in oil; however, they produce oil-in-water emulsions if they are more soluble in water. The mechanism to stabilize an emulsion is complex since it can vary from one system to another, but there are two very important and common aspects in all systems (*Chang, 2016*):

- Low interfacial tension between liquids facilitates the formation of the emulsion and improves the stability of the interfacial area linked to the emulsion.
- Strong interfacial film: films adsorbed on the periphery of the droplets cause emulsion stability.

A very important aspect to try to stop the destabilization process is that the drops do not adhere when they collide with each other. There are different ways to destabilize emulsions:

- Gravitational separation
- Flocculation or coagulation
- Coalescence
- Ostwald ripening

More information on each type of destabilization mechanism is found in *Appendix 2*.

Shelf life is a very important aspect when working with creams because they are products that must present a shelf life of several years to allow consumers. Losing stability is synonymous with losing the properties for its good use and application, therefore, for this kind of products, high

stability is required since consumer must have time to consume the entire product without losing its properties.

Other stabilization mechanism, apart from the addition of a third component, is the reduction of the droplets including a homogenization step.

#### 4.4. SENSORIAL QUALITY FACTORS

Sensory quality factors are very important when dealing with creams since they are products applied to the skin and, therefore, related to the five senses of human body. According to L. Rigano (*Rigano and Bonfigli, 2009*), sensory aspects always have a great dominant role in human being since the senses are continuously alert.

Good sensory quality factors will attract consumer attention and make these creams the most competitive in the market. Some of the sensory factors that make creams the most competitive are absence of irritation when applying the cream on the skin, opaque appearance, soft touch and not feeling oily (*Wibowo, 2001*).

**Smell:** type and intensity of the perfume of the product. According to Knowlton (*Knowlton and Pearce, 1993*), skin care products should have a perfume ratio between 0.01% and 0.5%. Within these products, facial creams must have between 0.1% and 0.2% perfume (*Diez, 1998*). In conclusion, each cream will have a different smell. It has been decided that (*Knowlton and Pearce, 1993*):

- BB Cream: white lotus perfume
- CC Cream: balsamic aroma with a vanilla character
- DD Cream: herbal aroma (odour of eucalyptus)

There are three types of responses to sensory stimuli: hedonistic, intensive and qualitative. In this case, for the first type of stimulus, which are more related to the pleasantness of use, research is done on the type of consumer; while, for the other answers which are intensive and qualitative, they are measured with trained panelists since the senses are the best measuring instruments (*Rigano and Bonfigli, 2009*).

## 4.5. PHYSICOCHEMICAL QUALITY FACTORS

Emulsions are unstable systems since their phases tend to separate. To avoid this problem, emulsifying agents are added to favour the union of the phases and make the emulsion stable. Because of this factor, one of the main physicochemical quality factors is that the cream is stable for a certain period, that is, that its phases are held together during the use of creams and fight against destabilization mechanisms. This quality factor can be measured by the duration of the stability of the cream (*Wibowo, 2002*).

The shelf life of a product differs slightly from the expiration date. While the first relates to the quality of the product in a specific period, the second is related to the quality and safety of the product at a specific moment (*Hulisz, 2013*).

One technique to measure the stability of products is laser diffraction. With this technique you can count the number of individual drops along the time and control the stability of the product. Laser diffraction works even when none of the destabilization's mechanisms discussed above can be observed (*Kontogeorgis and Kiel, 2016*).

Another very important aspect of creams is the ability to change phase when applied to the skin. Creams should be able to absorb quickly when applied to the skin and form a uniform covering. Considering that creams have been defined as semi-solid, when they come into contact with the skin, they should have a more liquid character in order to expand more easily (*Wibowo, 2002*).



## 4.6. QUALITY FACTORS AND INDICES SUMMARY

In this section, the quality factors and indices explained for each type of cream are summarized (Table 4.2).

Table 4.2. Quality factors and indices summary.

QUALITY FACTORS AND INDICES SUMMARY	
Functional quality factors	<ul style="list-style-type: none"> <li>- Colour corrector: appears opaque – beige colour</li> <li>- Covering power               <ul style="list-style-type: none"> <li>• CC and DD more coverage</li> </ul> </li> <li>- Sun protection: SPF 15/30/50</li> <li>- Antiaging: adequate antioxidants' %</li> <li>- Moisturizing</li> </ul>
Sensorial quality factors	<ul style="list-style-type: none"> <li>- Smell               <ul style="list-style-type: none"> <li>• BB: White lotus perfume</li> <li>• CC: Balsamic aroma with a vanilla character</li> <li>• DD: Herbal aroma (odour of eucalyptus)</li> </ul> </li> <li>- Does not cause irritation and does not feel oily</li> <li>- Ease of spreading and quickly absorption</li> </ul>
Physicochemical quality factors	<ul style="list-style-type: none"> <li>- Shelf life (3 years)</li> <li>- Life once opened (1 year)</li> <li>- Ability to change phase</li> <li>- Hygroscopicity</li> </ul>
Rheological quality factors	<ul style="list-style-type: none"> <li>- Pseudoplastic behaviour</li> <li>- Uniform coverage</li> <li>- Minimum yield stress: 6 Pa</li> <li>- Does not flow readily under gravity but easy to stir</li> </ul>
Emulsion stability quality factors	<ul style="list-style-type: none"> <li>- O/W emulsion</li> <li>- Droplet size: 20 <math>\mu\text{m}</math></li> <li>- Emulsifiers soluble in water</li> </ul>



## 5. SELECTION OF INGREDIENTS

After defining the main characteristics of the product, ingredients and their quantities will be selected. The selection of the ingredients is made based on the ability to perform a certain function.

The formulation of a product is defined by some important steps (*Wibowo, 2001*):

- Selection of active ingredients
- Selection of excipients or delivery vehicle (type of emulsion)
- Selection of a surfactant (if necessary)

The formulation of each cream will be elaborated from studies of different formulations, patents and encyclopaedias.

The first step in formulating a product is the selection of active ingredients, which are potential aspirants that will achieve the main functionality of the product. In addition to these, other types of ingredients are also necessary to meet secondary requirements such as appearance and ease of application (*Wibowo, 2001*).

Two important heuristics found in *Wibowo (2001)* are the selection of multifunctional ingredients, which serve more than a single function, and imperishable ingredients, that do not perish, whenever possible.

Then, after selecting the active ingredients, product delivery vehicle will be chosen. In this case, for the products developed, the most appropriate delivery vehicle is water, in the structure of an emulsion.

Following the selection of the ingredients, the type of emulsion must be defined. As already mentioned in previous sections and according to the heuristics of the article *Wibowo (2001)*, creams will be O/W emulsions since they should not feel greasy.

If two or more immiscible phases are involved, next step is the selection of a suitable surfactant or emulsifier. The emulsifier is responsible for keeping the emulsion stable by

dispersing a liquid phase in another liquid phase or solid particles in a liquid phase so that they are held together.

Normally, a mixture of surfactants is used instead of just one. Depending on the quantities and initial placement of the surfactants between the continuous phase and the dispersed phase, emulsions with different properties will be formed (*Bancroft, 1913*).

Many factors must be considered to achieve the desired emulsion. Some of the main criteria for selecting emulsifiers are (*Wibowo, 2001*):

- The emulsifier or emulsifiers should favour the formation of the desired emulsion.
- The emulsion must be stable for an enough period under various conditions
- The emulsifier and the other components must be compatible so as not to cause side reactions.
- The emulsifier should not be dangerous for the client, for health or for the environment and its cost should be as low as possible.

Another important factor to describe the emulsifier is the hydrophilic-lipophilic balance number (HLB). This number was first defined by Griffin in 1949 for nonionic emulsifiers as % hydrophilic part of the molecule divided by 5. Therefore, it could theoretically move in the range 0-20, with low values for hydrophobic surfactants and high values for hydrophilic ones. Later, this concept was extended to ionic surfactants and, due to the strong hydrophilicity of charges, values higher than 20 can be obtained.

Depending on the HLB value of the emulsifier, it will have some applications or others. The creams developed in this work, being O/W emulsions, must have a mixture of surfactants with a resulting HLB between 8 and 18 (*Tadros, 2013*).

As just mentioned, one of the most important factors is to obtain the desired emulsion. To achieve this, it is important to keep in mind that for a stable emulsion the continuous phase must be the one in which the surfactants chosen are more soluble (*Bancroft, 1913*). Therefore, if the creams developed in this work are O/W emulsions, the surfactants to be used will be hydrophilic, as said above with HLB 8-18.

There are different types of hydrophilic surfactants:

- Cationic surfactants can irritate the eyes so they are not an ideal choice for facial creams (*Wibowo, 2001*).

- Amphoteric are also a bad choice since they are unusual in cosmetic products (Salvador and Chisvert, 2007).

- Anionic surfactants are sensitive to low pH values (Salvador and Chisvert, 2007).

- Nonionic surfactants are ingredients of cosmetic products capable of performing different functions (Salvador and Chisvert, 2018).

After the above, one of the most suitable options for cosmetic products is a mixture of non-ionic surfactants. However, the use of mixtures of different types of surfactants (anionic-amphoteric, nonionic-amphoteric) is also acceptable.

## 5.1. BB CREAM

The main function of BB Cream is to hydrate, while offering light coverage and protecting from the sun. Hydration is given by three components: emollients, humectants and occlusives.

### Emollients

The emollients are lipids and oils that improve flexibility and softness of the skin while moisturizing it since they are easy to spread on the skin and provide an occlusion that improves the sublayer of the epidermis, the stratum corneum (Sethi et al., 2017). A good emollient should be chosen that provides a perception to the consumer of maximum softness in the skin.

The most used emollients in cosmetics are acids and long-chain fatty alcohols that generate benefits in the skin barrier. There are different types of emollients (Table 5.1).

Table 5.1. Examples of emollients (Sethi et al., 2017).

Type of emollients	Substances
Astringent emollients	Cyclomethicone, dimethicone, isopropyl myristate, octyl octanoate
Dry emollients	Decyl oleate, isopropyl palmitate, isostearyl alcohol
Fatting emollients	Castor oil, glyceryl stearate, jojoba oil, octyl stearate, propylene glycol
Protective emollients	Diisopropyl dilinoleate, isopropyl, isostearate

The hydration action of emollients should be visible after 30 minutes - 1 hour and should last for about 4 hours, as they aim to increase the water content in the epidermis.

The emollient chosen for this cream is glyceryl stearate since it is respectful with the skin and helps to stabilize oil-in-water emulsions by reducing the surface tension of substances (*Specialchem*, CAS Number 31566-31-1).

According to Rawlings et al. (2004), the percentage of emollients for facial products should be compressed between 2% and 15% by weight, so it has been decided that the percentage of emollients in this cream is 12%.

### Humectants

Humectants, also with emollient properties, are hygroscopic substances that attract water from the dermis to the epidermis and from the environment in humid conditions. Some examples of humectants are shown in the following table (Table 5.2), but the most effective and used in cosmetics is glycerin, also known as glycerol, thanks to its multiple applications (Sethi et al., 2017).

Table 5.2. Examples of humectants (Sethi et al., 2017).

Humectants	
- Glycerin	- Panthenol
- Urea	- Hyaluronic acid
- Honey	- Propylene glycol
- Sorbitol	- Butylene glycol

Glycerol is an ideal substance for facial hydration since it has no side effects and, in addition, when combined with occlusive agents produces synergistic relief of dry skin (Sethi et al., 2017).

Most moisturizers have a mixture of emollients, lipids and moisturizers incorporated. The main humectant that is most effective when formulated with lipids is glycerol. In the following image (Figure 5.1) the difference can be observed: it is shown that a mixture of lipids is less effective than when combined with glycerol. The image compares the effect of a cream that contains 1% phospholipids, 2% cholesterol and 1% stearic acid (◆) with another cream that contains the same plus 1% glycerol (■).

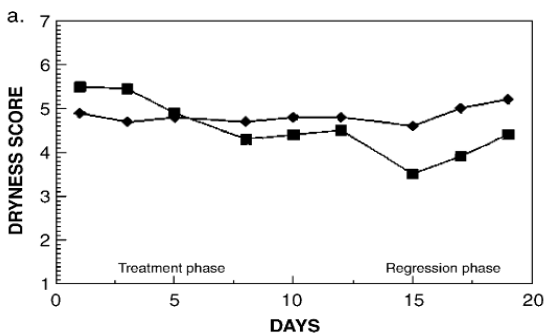


Figure 5.1. Comparison (Rawlings *et al.*, 2004).

According to an article by Wibowo (2001), the maximum amount of glycerin in creams is 20%, but the percentage of glycerin in facial products must be compressed between 2% and 10% by weight (Rawlings *et al.*, 2004). Also, according to *US Pat. No. 2004/0180026 A1*, glycerol concentration must be between 4.9% and 10.7%, so, also, according to the formulation *JZ8-159* (Hallstar, 2019) and as mentioned above, the percentage decided for this cream is 5%.

### Occlusives

Occlusive substances are those that form a barrier on the surface of the skin to prevent water evaporation, so they block TEWL in the stratum corneum. These substances are more effective when combined with humectants.

There are a wide variety of occlusives as shown in the following table (Table 5.3), but not all of them are recommended for facial cosmetic products.

Table 5.3. Examples of occlusives (Sethi *et al.*, 2017).

Class of occlusive	Examples
Hydrocarbons	Petrolatum, paraffin, mineral oil, squalene...
Fatty acids	Lanolin acid, stearic acid
Fatty alcohols	Cetyl alcohol, stearyl alcohol, lanolin
Phospholipids	Lecithin
Polyhydric alcohols	Propylene glycol
Sterols	Cholesterol
Vegetable waxes	Carnauba, candelilla
Wax esters	Beeswax, lanolin, stearyl stearate

Many occlusive agents are mineral oils derived from petroleum, so the two least recommended agents for cosmetic products are petrolatum and lanolin. For a band, petrolatum, although it is an occlusive agent that reduces water loss by 98%, while other occlusives only achieve 20-30%, has a harmful aesthetic and is not acceptable because its greasiness. On the other hand, lanolin is not recommended because of its smell and sensitivity problems, particularly allergenicity (*Sethi et al., 2017*).

Substances derived from silicones are the most used as occlusive agents. According to the formulation of *HouseHold 866-511-6529*, the occlusive chosen for these creams is going to be dimethicone. As in the previous case, the ideal percentage of occlusives for facial products should be between 2% and 15% (*Rawlings et al., 2004*). The percentage chosen according to *US Pat. No. 2012/0082695 A1* is 4%.

### **Preservatives**

According to MicrochemLaboratory, preservatives are antimicrobial ingredients that are added to cosmetic products to reduce or avoid the number of microbial contaminants while maintaining the microbiological safety of the products by preventing the growth of microbes.

These types of substances are added to cosmetic products since all beauty products are composed of biodegradable ingredients which microbes can easily break down. Antioxidants are also used as preservatives as they protect the alteration of the product caused by oxygen.

All cosmetic products must take preservatives to high levels as this can ensure good product quality. That a cosmetic product decomposes is linked with an unpleasant perception on the part of the consumer, while it becomes an insecure product towards its health.

The choice of preservative is very important since it is a very critical aspect, so according to Bombeli (*2012*), the ideal preservative should have the following characteristics:

- Odorless, colorless and safe
- Effective and stable over a wide pH range
- Compatible to always be activated with any type of ingredients and useful in both hot and cold phases
- Soluble in water and liquid and effective at low proportions



- Extensive activity against bacteria and fungi
- Economic

Next, the five most common types of preservatives will be presented with some examples (Table 5.4):

Table 5.4. Examples of preservatives (*MicrochemLaboratory*).

Preservatives	Examples
Parabens	Germaben II, Methylparaben, Propylparaben, Butylparaben
Formaldehyde	Germall Plus, DMDM Hydantoin, Imadiazolidinyl Urea, Diazolidinyl Urea
Isothiazolinones	Kathon
Phenoxyethanol	Optiphen, Optiphen Plus
Organic acids	Benzoic acid/ sodium benzoate, sorbic acid/ potassium sorbate, anisic acid, levulinic acid

Preservatives can be divided into two groups: antioxidant and antimicrobial preservatives. The most widely used antimicrobial preservatives in cosmetic products have been parabens. They are used in a wide variety of products (shampoos, deodorants, scrubs ...), everything and this, for a few years they have entered a great discussion since they have found estrogenic effects of parabens on human body. These effects cause fertility problems in men and cause the appearance of breast cancer in women (*Garner et al., 2014*). These effects caused parabens to be unwanted ingredients by consumers for their cosmetic products.

According to *Schuelke company*, the best preservative for its work efficiency in a wide temperature and pH range is Euxyl® PE 9010 formed by phenoxyethanol (90%) and ethylhexylglycerin (10%) (*Safety Data Sheet LotionCrafter*).

According to *Rastogi (Rastogi et al., 2000)*, the maximum concentration of this preservative in cosmetics is 1%, so this cream will have a preservative composition of 0.5%. Of this 0.5%, 0.45% is phenoxyethanol and 0.05% ethylhexylglycerin.

### Thickeners

Thickeners are substances that are added to cosmetic products to improve rheological properties of the product, increase the viscosity, improve the stability and change the appearance of the product. These ingredients are very important for cosmetic products since they maintain a

good appearance of the product and, subsequently, provide a good application on the skin (*Laba, 2001*).

Depending on the viscosity range of the product, a thickener or another will be chosen as there are more efficient thickeners at certain viscosity ranges. In addition, the selection of the thickener will also be influenced by the phase with which it should be more compatible, whether oil or water. Considering what is explained in the section on rheological quality factors, knowing that the creams have a low viscosity at high shear rates and high viscosity at low shear rates, and that the continuous phase is water it has been decided that the most suitable thickener for this cream is going to be xanthan gum. This thickener has also been chosen since it is a substance of natural origin (*Moravkova and Filip, 2014*).

Cosmetic products that have xanthan gum incorporated as a single thickener have a higher stabilization performance compared to those that have a set of thickeners formulated (*Russ and Sebök, 2017*).

According to *Wibowo (2001)*, the percentage of thickener in creams should be between 0.1% and 2%, and according to *Rawlings (Rawlings et al., 2004)*, the percentage of thickeners for facial cosmetic products should be between 0% and 4%. With all this, and according to *US Pat. No. 10,058,503 B2* and *US Pat. No. 5,079,003*, a daily facial cream must have a composition of xanthan gum between 0.2% and 0.5%, so with the example presented in the *US Pat. No. 10,058,503 B2*, this cream will carry 0.5% of xanthan gum.

### **UV filter**

Sunscreens are substances that protect the skin from sunlight. These filters can reflect and absorb the two types of ultraviolet rays, A and B. The addition of these substances in cosmetic products serves to prevent the appearance of spots, wrinkles, even signs of skin cancer (*Marín and Del Pozo, 2005*).

One of the main sunscreens is going to be titanium dioxide, since it has both sun protection and colour contribution functions (*Pigments and dyes section*). Considering that this cream has a low sun protection factor, its percentage of sun protection will be included in the following section considering its other function of coverage.

The sunscreen chosen for this cream is known as PARSOL® TX, which incorporates three substances: titanium dioxide, silica and dimethicone. Two of these substances are multifunctional

substances capable of exercising this function while giving colour (titanium dioxide) and maintaining skin hydration (dimethicone).

PARSOL® TX, formed by a titanium dioxide core coated with a double layer of silica and dimethicone, is an inorganic sunscreen that offers great sun protection and is approved by all regulations. This substance is formed by 85% titanium dioxide, 10% silica and 5% dimethicone (*Sunjin, 2016*).

According to *US Pat. No. 6,146,617*, the percentage of sunscreens in non-solar cosmetic creams must be between 5% and 25%, depending on the protection factor desired for the cream. Desiring a sun protection factor of 15, a percentage of PARSOL® TX of 5% has been decided considering that part of the ingredients will perform two functions at the same time. Of this 5%, 4.2% is titanium dioxide, 0.5% silica and 0.3% dimethicone.

### **Pigments and dyes**

The substances responsible for giving coverage to the cream are solid particles dispersed in the emulsion known as colorants or pigments. The *US Pat. No. 2018/0263884 A1* provides new surface treatment methods in which inorganic pigments are combined with titanium dioxide and the result is a more stable and uniform distribution.

Inorganic pigments are iron oxide yellow, iron oxide red and iron oxide black. The different composition of each pigment with titanium dioxide will result in creams with different coverages. Titanium dioxide is used as a dye, although it also has properties as a sunscreen. This substance can colour the cosmetic product while protecting the skin from sunlight (UV filter).

According to *US Pat. No. 2018/0263884 A1*, the maximum advisable percentage of iron oxide yellow is 7.6%, that of iron oxide red is 3% and iron oxide black is 1.4%. With this information it has been decided that the composition is as follows:

- Iron oxide yellow (CI 77492), Triethoxycaprylsilane: 1.1%
- Iron oxide red (CI 77491), Triethoxycaprylsilane: 0.4%
- Iron oxide black (CI 77499), Triethoxycaprylsilane: 0.1%
- Titanium dioxide (CI 77891), Triethoxycaprylsilane: 1%

## Fragrances

The perfume chosen is Perfume Lotus Blanc RS 67561. In facial cosmetic products the percentage of perfume must be between 0.0% and 0.5%. The percentage chosen is 0.2% (Rawlings et al., 2004).

## Surfactant

Two types of surfactants have been chosen for BB Cream:

- Polyethylene glycol-100 Stearate: non-ionic emulsifying agent of vegetal origin with an HLB value of 18.8.
- Glyceryl stearate: non-ionic emulsifying agent of low HLB value: 3.8.

These two types of emulsifiers have been selected since the combination of both results in greater stability of O/W emulsions. Also, the combination of these two emulsifiers is the main basis for many cosmetic products.

Although there are sources that say polyethylene glycols are harmful to human health, the article "Safety assessment on polyethylene glycols (PEGs) and their derivatives as used in cosmetic products" quotes: "In general, it is concluded that the PEGs covered in this analysis are safe for use in cosmetics under the current intended conditions of use. Considering all available information, it can be assumed that these compounds, as currently used in cosmetic preparations, will not present a risk to human health" (Fruijtier-Polloth, 2005).

According to formulation JZ7-133 (HallstarBeauty), the percentage of use of polyethylene glycol should be between 1% and 3%, instead, the percentage of use of glyceryl stearate should be between 1% and 5%. In conclusion, polyethylene glycol will have 1.3% and glyceryl stearate 1.5%.

## Formulation summary

The following table (Table 5.5) summarizes the formulation of BB Cream.

Table 5.5. BB Cream formulation.

Substance	Percentage (%)
Water	67.4
Glyceryl stearate	13.5
Titanium dioxide	5.2
Glycerin	5.0

Substance	Percentage (%)
Dimethicone	4.3
Polyethylene glycol	1.3
Iron oxide yellow	1.1
Xanthan gum	0.5
Silica	0.5
Phenoxyethanol	0.45
Iron oxide red	0.4
Perfume Lotus Blanc RS 67561	0.2
Iron oxide black	0.1
Ethylhexylglycerin	0.05

## 5.2. CC CREAM

CC Creams are creams with greater moisturizing power and greater coverage and sun protection.

### Emollients

The emollient chosen for this cream is known as Dedraflow HCO. This emollient is formed by hydrogenated coconut oil, a vegetable oil made up of 90% saturated acids. It is a fatty substance of vegetable origin and with wide uses in cosmetics.

The choice of this natural emollient is due to its multifunctional functionality as it can act as an emollient, while moisturizing and lubricating. Its stability, water retention capacity and antimicrobial properties make it one of the emollient agents most accepted by consumers to prevent skin cracking. In addition to offering the product a good ability to spread on the skin, it leaves a shiny and smooth skin after application (*Vera, 2018*). Adding to all this, it is also able to protect skin from the sun.

According to *SpecialChem* emollient datasheet, the composition of this emollient in facial creams should be between 5% and 10%, so it has been decided that this cream will have 8% Dedraflow HCO.

To offer greater hydration and considering the compatibility with the previous emollient, cream will also include Biochemica ® Aloe Butter, an extract of *Aloe barbadensis* that incorporates a

coconut fat fraction. This extract is totally natural, miscible in oil and has uses in both creams and lotions, body balms and bar soaps.

Biochemica © Aloe Butter is a solid butter that melts when it comes in contact with the skin. It acts against the dryness of the skin helping to maintain good hydration after sun exposure, together with other aggressive elements.

Its use in cosmetic products ranges between 1% and 8% (*Hallstar, formulation: CPD074-11-08H*), so, when combined with another emollient, it has been decided to choose 1%.

### **Humectants**

The moisturizer chosen for this cream is going to be glycerin again due to its multiple functions and excellent characteristics.

The percentage decided for this cream, according to the percentages mentioned in the previous section of humectants of BB Cream, is 9% since these creams require a greater hydration factor.

### **Occlusives**

The occlusive selected for this cream is the propylene glycol for its great versatility since it can act as a humectant, prevent the growth of bacteria and increase the elasticity of the skin.

The recommended percentage of occlusives in facial products should be between 2% and 15% (*Rawlings et al., 2004*). According to the formulation *CPD074-11-08H (HallstarBeauty)*, it has been decided to put 3%.

### **Preservatives**

The preservative chosen for this cream is sodium benzoate which can act as a preservative, anti-corrosive agent and masking and fragrance ingredient, but predominantly as a preservative.

According to Nair (*2001*), the concentration should be between 0.5% and 1%. The percentage chosen is 0.5%.

### **Thickeners**

The thickener chosen for this cream will be the same as that of BB Cream: xanthan gum. The proportion of thickener is 0.5%.

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## UV filter

The sunscreen chosen for this cream is, on the one hand, diethylaminohydroxybenzoyl hexyl benzoate because of its photostability and its compatibility with cosmetic ingredients and other sunscreens. It is a filter capable of absorbing UVA radiation, the main responsible for the appearance of wrinkles and spots.

According to the regulations of the European Union and the formulation *CPD074-11-08H (HallstarBeauty)*, since 2005 this ingredient has a maximum composition in creams of 10%, so this cream will carry 3%.

On the other hand, the other sunscreen chosen is octocrylene because it is capable of absorbing both UVA and UVB radiation. This filter protects the skin from direct DNA damage and penetrates the skin acting as a photosensitizer.

This filter is considered safe when used in a maximum concentration of 10% in cosmetic products (*Berardesca et al., 2019*). According to the formulation *CPD074-11-08H (HallstarBeauty)*, the percentage chosen for this cream is 5%.

Finally, a third sunscreen is included in the formulation to present a greater protective factor. Ethylhexyl methoxycinnamate is a sunscreen that absorbs, reflects or disperses ultraviolet rays and protects the skin from possible sunburn and other harmful effects.

The European Union cosmetics directive sets a maximum permissible concentration of 10% of this filter in cosmetic products with solar factor. In this cream the chosen percentage is 4%.

## Pigments and dyes

As previously mentioned, all three types of creams will have the same colour tone, but this type of cream offers greater coverage power, so that a greater proportion of titanium dioxide will be present.

- Iron oxide yellow (CI 77492), Triethoxycaprylsilane: 1.1%
- Iron oxide red (CI 77491), Triethoxycaprylsilane: 0.4%
- Iron oxide black (CI 77499), Triethoxycaprylsilane: 0.1%
- Titanium dioxide (CI 77891), Triethoxycaprylsilane: 3%

## Fragrances

The perfume chosen to obtain the desired aroma is Vanilla Fragrance RR79226 with a percentage of 0.2%.

## Surfactant

The surfactant chosen for this cream is cetearyl olivate/sorbitan olivate, also known as Olivem® 1000. Olivem is a non-ionic self-emulsifier formed by fatty acids with chemical composition like that of the skin. This compound has been chosen for its wide variety of properties, both aging and anti-redness, as a sunscreen and moisturizer.

According to *HallstarBeauty*, the recommended percentage of use for this ingredient in cosmetic products, with O/W emulsion and as the sole emulsifier, should be between 1.5% - 4%. In this cream a percentage of 3% has been chosen (*Formulation: CPD074-11-08H*).

## Formulation summary

The following table (*Table 5.6*) summarizes the formulation of CC Cream.

Table 5.6. CC Cream formulation.

Substance	Percentage (%)
Water	58.2
Glycerin	9
Dedraflow HCO (Hydrogenated coconut oil)	8
Octocrylene	5
Ethylhexyl methoxycinnamate	4
Propylene glycol	3
Titanium dioxide	3
Diethylaminohydroxybenzoyl hexyl benzoate	3
Cetearyl olivate /Sorbitan olivate	3
Iron oxide yellow	1.1
Biochemica ® Aloe Butter (Aloe barbadensis)	1
Xanthan gum	0.5
Sodium benzoate	0.5
Iron oxide red	0.4
Vanilla Fragrance RR79226	0.2
Iron oxide black	0.1



### 5.3. DD CREAM

DD Creams are creams intended for very mature skin that have already lost elasticity, flexibility, shine and softness.

#### Emollients

The emollient chosen for this cream is collagen. Collagen is a natural rejuvenating protein molecule present in the skin. One of its main functions is always to keep the skin hydrated. The presence of this substance in the cream is beneficial since it offers elasticity, resistance and extensibility to the dermis, which causes a lower appearance of wrinkles (*SpecialChem, CLR Collagen*). In addition to its moisturizing functions, it also acts as an anti-aging agent and anti-wrinkle agent since it always keeps the skin smooth and stretched.

According to *SpecialChem, CLR Collagen*, the common use of collagen ranges between 0.5% and 1%, the latter being the most predominant. So, a weight percentage of 1% collagen has been decided.

Caprylic/capric triglyceride has also been chosen as an emollient. This multifunctional ingredient of natural origin can act as an emollient, as a preservative, antioxidant and stabilizer. Its main benefits are to repair the skin's superficial layer and resist water loss (Watson, 2019).

According to formulation Perfect DD Cream SPF 20 (*Review Personal Care*) a percentage of caprylic/capric triglyceride of 13% has been chosen.

#### Humectants

The humectants chosen for this cream are glycerin together with hyaluronic acid. Glycerin is one of the main and most effective moisturizers.

Wrinkles, loss of elasticity and skin fat and decreased collagen and elastin are the main signs of aging. These signs produce a thinner and less smooth appearance. These factors usually appear mainly on the face and neck between the ages of 40 and 50. Over the years, the concentration of hyaluronic acid decreases, which leads to dehydration and loss of elasticity.

Research has shown the potential anti-wrinkle factor of the formulations that include hyaluronic acid, since it can retain water with a percentage of up to a thousand times its weight.

The incorporation of hyaluronic acid in the creams allows the hydration of the skin and the firmness and smoothness of the skin by lubrication of the collagen fibbers (*Macías et al., 2015*).

The percentage of glycerin in facial products must be compressed between 2% and 10% by weight (Rawlings *et al.*, 2004). The percentage of glycerin in this cream is 4%.

The percentage of hyaluronic acid is 0.5%.

### **Occlusives**

The occlusive chosen for these creams is dimethicone.

The percentage of occlusives for facial products should be between 2% and 15% (Rawlings *et al.*, 2004). The percentage chosen according to *US Pat. No. 2012/0082695 A1* and the *formulation 15.177.01B C152 (Essential DD Cream, LucasMeyer)* is 3%.

### **Preservatives**

The preservative chosen is DMDM Hydantoin. Its percentage in the cream according to *US Pat. No. 10,058,503 B2* is 0.5%.

Vitamins are also considered antioxidant agents since they prevent the appearance of wrinkles. In this case, vitamin E has been chosen as an antioxidant thanks to its great antioxidant power against free radicals and its ability to improve the skin.

This vitamin is present in nature and helps skin to prevent the signs of aging while calming that dehydrated and sensitive skin and protects the scars present in the most superficial layer of the skin. Also, although by itself it is not a sunscreen, there are studies that show that the application of vitamin E makes it more prepared against the harmful effects of the sun.

Vitamin C will be added to this vitamin since both together are able to lighten the dark spots of the skin and increase its antioxidant power.

In accordance with *U.S. Patent No. Pat. 3,996,193* the concentration of vitamin E should be between 0.5% and 5%, so the percentage chosen is 0.5%. In the case of vitamin C, it is the same, its percentage is 0.5%.

### **Thickeners**

The thickener chosen for this cream is xanthan gum. The proportion of xanthan gum is 0.5%.

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## UV filter

These creams, unlike the previous ones, have greater sun protection. In this case, being creams that are also intended to reduce the appearance of wrinkles, biological sunscreens will be combined with vitamins of type A, C and E to enhance sun protection.

The sunscreen chosen for this cream is known as Solaveil™ XT-40W formed by titanium dioxide, aqua, polyglyceryl-2 caprate, sucrose stearate, simmondsia chinensis (jojoba) seed oil, stearic acid, alumina, glyceryl caprylate and squalene. This filter offers exceptional protection against UVA rays with high UVB efficiency. When this filter is combined with Venuceane™, which protects against infrared and free radicals, both can offer ample protection against the sun and its anti-aging effects as they protect the generation of free radicals.

Venuceane™, formed by thermus thermophilus ferment and glycerin is the most effective active ingredient to fight against photoaging.

According to the *formulation Perfect DD Cream SPF 20 (Review Personal Care)* and *Daily Defense (DD) Cream (Croda)* the percentage chosen for Solaveil™ XT-40W is 15% and for Venuceane™ it is 3%.

## Pigments and dyes

This cream has the same colour tone as the previous ones, but offers greater coverage power so the titanium dioxide percentage' increases:

- Iron oxide yellow (CI 77492), Triethoxycaprylsilane: 1.1%
- Iron oxide red (CI 77491), Triethoxycaprylsilane: 0.4%
- Iron oxide black (CI 77499), Triethoxycaprylsilane: 0.1%
- Titanium dioxide (CI 77891), Triethoxycaprylsilane: 4%

## Fragrances

The perfume chosen to obtain the herbal aroma with odour of eucalyptus is the natural raw material essence of Rose Otto that will give a floral and herbal aroma. The percentage of the ingredient is 0.2%.

## Surfactants

The emulsifier chosen for DD Cream is Heliocel<sup>™</sup>, which is a natural compound formed by glyceryl stearate citrate, polyglyceryl-3 stearate and hydrogenated lecithin. This compound is an oil-in-water emulsifier with moisturizing functions. According to the technical sheet of this compound in SpecialChem, the level of use must be between 3% and 6%, so 4% has been decided on this cream. The second emulsifier is known as NatraGem<sup>™</sup> E145 and is formed by polyglyceryl-4, laurate/succinate and water. This non-ionic emulsifier acts as an emulsifier O/W and is 100% natural and very versatile. According to formulation *Perfect DD Cream SPF 20 (Croda, Personal care)*, a composition of 2.5% has been chosen.

### Formulation summary

The following table (Table 5.7) summarizes the formulation of DD Cream.

Table 5.7. DD Cream formulation.

Substance	Percentage (%)
Water	46.2
Solaveil <sup>™</sup> XT-40W	15
Caprylic/Capric triglyceride	13
Heliocel <sup>™</sup>	4
Glycerin	4
Titanium dioxide	4
Venuceane <sup>™</sup>	3
Dimethicone	3
Polyglyceryl-4, laurate/succinate	2.5
Iron oxide yellow	1.1
Collagen	1
Hyaluronic acid	0.5
Vitamin C	0.5
Vitamin E	0.5
Xanthan gum	0.5
DMDM Hydantoin	0.5
Iron oxide red	0.4
Rose Otto	0.2
Iron oxide black	0.1

## 6. SYNTHESIS OF PROCESS FOR PRODUCT MANUFACTURING

The next step, after deciding the ingredients and the structure of the product, is the design of the manufacturing process. Design selection is determined by three steps (*Wibowo, 2001*):

- Process flowsheet capable of designing the product with the desired microstructure
- Equipment selection
- Order of addition of the ingredients and operating conditions of the equipment

To start this section, it is necessary to have a total production of these multi-purpose creams. A total of 100,000 buyers will be assumed and from there the production, in kg/year, of each type of cream will be calculated considering that each one has three different shades of colour.

As it mentioned, assuming a total of 100,000 buyers, they have been distributed in the purchase of the different creams as follows (*Table 6.1*):

Table 6.1. Consumers for each cream

Cream	Consumers
BB Cream	50,000
CC Cream	40,000
DD Cream	10,000
TOTAL:	100,000

As already mentioned, each cream has three possible colour tones: low, medium and high. The classification of each shade for each cream according to the defined buyers is as follows (*Table 6.2*).

Table 6.2. Classification of colour tones.

CREAM	TONE	Consumers/tone	Total consumers
BB CREAM	Low	15,000	50,000
	Medium	25,000	
	High	10,000	
CC CREAM	Low	15,000	40,000
	Medium	20,000	
	High	5,000	
DD CREAM	Low	2,500	10,000
	Medium	5,000	
	High	2,500	

Depending on the cream, it is intended for an age range of women or another. Each type of cream and tone have different consumers since the use is not the same.

Knowing that these facial creams are for daily use and the packaging is 40 mL in size, it has been assumed that each consumer spends an average of 1 cream per year. With all this, the amount produced per year is 4,000,000 mL, which is the same, 4,000 L. Knowing the ingredients and its amounts, the density of the three types of creams can deduce. The density of all creams is around 1.01 kg/L, so, rounding, it has been decided to take the same density value for all creams of 1 kg/L (*Mujica et al., 2010*). With all these data it is concluded that the production of these three creams for a year is 4,000 kg. These 4,000 kg are divided into the three types of creams and the different shades as follows. (*Table 6.3*):

Table 6.3. Production of each cream.

CREAM	TONE	Kg/year	Kg/year
BB CREAM	Low	600	2,000
	Medium	1,000	
	High	400	
CC CREAM	Low	600	1,600
	Medium	800	
	High	200	
DD CREAM	Low	100	400
	Medium	200	
	High	100	

The manufacture of these multi-purpose creams, being new high-performance products that seek to be incorporated into the market, will be carried out in a batch process. This type of process is a manufacturing process in which different operations of a recognizable product are carried out in a short period of time. Most of the industries where chemical processes are carried out use this type of manufacturing method, together with the semi-continuous process (Vinson, 2008).

The method of manufacturing chosen is the batch process since it is a process with greater flexibility, it allows to change the production of a cream to another cream in less time and with greater ease, in addition to having lower costs (Tan et al., 2006).

In this section a process flowsheet will be developed with the manufacturing process for the three creams developed. Since each cream has some main functions, the manufacturing processes will have the same procedural basis.

It has been decided that the total desired production will be produced in batches of 100 kg. If the 4,000 kg per year are produced in batches of approximately 100 kg, a total of 40 batches are obtained. These 40 lots will be divided into three four-month periods of four months each one. The distribution of the lots will be done uniformly in three four-month periods, but it can be modified since it will depend on the demand that exists at that time. The plant will not only be focused on the production of these creams but will also produce other cosmetic products, as it will be free for long periods of time. After all that has been explained, 14 lots will be made in the first four-month period and 13 lots in both the second and third quarters. To produce the desired 100 kg of each batch, the corresponding amount of each ingredient will be added in an agitated vessel with a capacity of 150 L.

Batches of the same type of cream (BB, CC or DD) are to be manufactured together and the order of manufacture will be from the lowest tone to the highest tone. This decision is due to the fact that in the same type of cream there is no need to clean between, and when passing from the lowest to the highest tone, the only thing that differs is the composition of the particles that give the cream a darker tone.

Next, the manufacturing processes will be designed.

## 6.1. SYNTHESIS OF FLOWSHEET

The cream manufacturing process usually consists of five units' operations (*Wibowo, 2002*): pre-treatment, mixing, structure formation or homogenization, post-treatment and packaging or filling. The process flowsheet for the creams developed in this work is as follows (*Figure 6.1*):

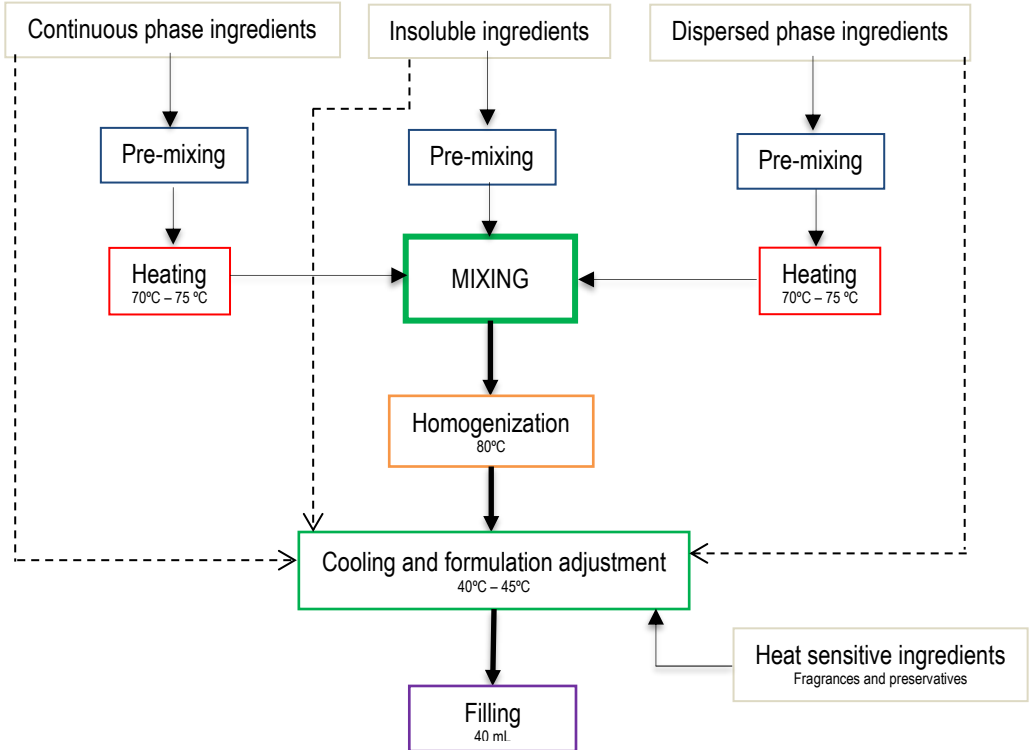


Figure 6.1. General structure of creams manufacturing process.

As can be seen in the figure, the first unit operation is a pre-mixing in which the continuous phase ingredients, those that are soluble in water, and the dispersed phase ingredients, those that are soluble in oil, are mixed separately. In addition, if there are insoluble solid ingredients, they could need a pre-treatment like drying, humidification, mixing, reduction of the particle size (*Wibowo, 2001*). Next, the two phases, together with the insoluble ingredients, are mixed by dispersing the oil phase in drops in the aqueous phase in order to form a pre-emulsion with a large droplet size. After mixing, a homogenization step is necessary to reduce the droplet size. All these mixing operations are carried out at high temperature, while before reaching



homogenization, the temperature is lowered, and a viscous product is obtained. If the product obtained does not keep to the specifications, a formulation adjustment (colour, viscosity) will be made by adding the necessary phase to obtain the appropriate properties of the product. The last step is the packaging of the product in a 40 mL package for sale.

This flowsheet explains the manufacturing process of the creams developed in this work since it is very similar between them.

In order to obtain the desired emulsion, O/W emulsion, the order of addition of ingredients and the placement of surfactants are very important (*Bancroft, 1913*). On this occasion surfactants will be mixed with ingredients of the aqueous phase, both those water-soluble ingredients and the thickeners present in the formulation (*Wibowo, 2001*).

The first phase added to the initial equipment, before mixing with other phases, will be the continuous phase. Wanting to obtain an O/W emulsion and avoiding phase inversion, the oil phase will slowly be added to the aqueous phase present in the initial vessel.

First, before starting the mixing, both ingredients of the aqueous phase and those of the oily phase are heated separately to reduce viscosity and facilitate mixing. Both phases are heated to a temperature of approximately between 70°C - 75°C.

Once hot, insoluble particles are slowly added to the dispersed phase without the need for any pre-treatment since the particles are already the right size to be used. Shake well until the mixture is homogeneous. Once insoluble particles have dispersed, add this phase slowly in the aqueous phase to obtain an O/W emulsion (*Hallstar Beauty*).

Then, heat the mixture to 80°C and homogenize in order to reduce the droplet size and obtain a homogeneous mixture (*Wibowo, 2001*).

After homogenization, cool the mixture below 40°C - 45°C. Once the mixture is below this temperature, add the preservatives to the mixture together with the fragrance. A cooling stage is included because these ingredients are heat sensitive (*Wibowo, 2001*). Then, stir the mixture until uniform and allow it to reach the desired room temperature. A sample of the product is taken and if it does not meet the regulatory specifications, a formulation adjustment is made.

Finally, package the cream in 40 mL polypropylene folding tubes.

### BB Cream

The following table (Table 6.4) classifies the ingredients.

Table 6.4. Classification of the ingredients in the phases.

Continuous-phase ingredients	Insoluble ingredients	Dispersed-phase ingredients
<ul style="list-style-type: none"> <li>- Water</li> <li>- Glycerin</li> <li>- Xanthan gum</li> <li>- Polyethylene glycol</li> </ul>	<ul style="list-style-type: none"> <li>- Titanium dioxide</li> <li>- Iron oxide yellow</li> <li>- Iron oxide red</li> <li>- Iron oxide black</li> </ul>	<ul style="list-style-type: none"> <li>- Dimethicone</li> <li>- Glyceryl stearate</li> <li>- Silica</li> </ul>

### CC Cream

The following table (Table 6.5) classifies the ingredients.

Table 6.5. Classification of the ingredients in the phases.

Continuous-phase ingredients	Insoluble ingredients	Dispersed-phase ingredients
<ul style="list-style-type: none"> <li>- Water</li> <li>- Glycerin</li> <li>- Xanthan gum</li> <li>- Propylene glycol</li> <li>- Cetearyl olivate /Sorbitan olivate</li> </ul>	<ul style="list-style-type: none"> <li>- Titanium dioxide</li> <li>- Iron oxide yellow</li> <li>- Iron oxide red</li> <li>- Iron oxide black</li> </ul>	<ul style="list-style-type: none"> <li>- Dedraflow HCO (Hydrogenated coconut oil)</li> <li>- Octocrylene</li> <li>- Ethylhexyl methoxycinnamate</li> <li>-Diethylaminohydroxybenzoyl hexyl benzoate</li> <li>- Biochemica ® Aloe Butter (Aloe barbadensis)</li> </ul>

### DD Cream

The following table (Table 6.6) classifies the ingredients.

Table 6.6. Classification of the ingredients in the phases.

Continuous-phase ingredients	Insoluble ingredients	Dispersed-phase ingredients
<ul style="list-style-type: none"> <li>- Water</li> <li>- Xanthan gum</li> <li>- Hyaluronic acid</li> <li>- Glycerin</li> <li>- Polyglyceryl-4, laurate/succinate</li> <li>- Glyceryl stearate citrate, polyglyceryl-3 stearate and hydrogenated lecithin</li> <li>- Solaveil™ XT-40W</li> <li>- Vitamin C</li> <li>- Venuceane™</li> </ul>	<ul style="list-style-type: none"> <li>- Iron oxide yellow</li> <li>- Iron oxide red</li> <li>- Iron oxide black</li> <li>- Titanium dioxide</li> </ul>	<ul style="list-style-type: none"> <li>- Collagen</li> <li>- Dimethicone</li> <li>- Vitamin E</li> <li>- Caprylic/Capric triglyceride</li> </ul>

## 6.2. SELECTION OF EQUIPMENT UNITS

The equipment selection, related to the flowsheet, is made according to the energy consumption and capacity of the equipment. Always try to opt for an equipment that has a wide range of capacity while consuming little energy (*Wibowo, 2001*).

### Pre-mixing, mixing and formulation adjustment

For the operations of pre-mixing, pre-emulsification and dispersion of solids in liquid, the best equipment according to its capacity and energy consumption is an agitated vessel, as show the following figure (*Figure 6.2*).

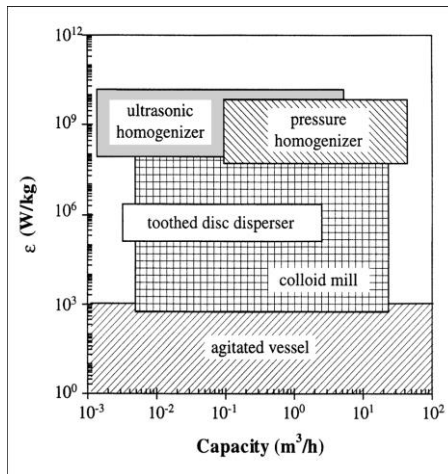


Figure 6.2. Typical capacity and energy consumption for selected emulsification units (*Wibowo, 2001*).

When selecting the most appropriate agitator, the following parameters must be considered: type of flow, viscosity of the fluid and applications dependent on the speed. There are three types of agitators related to the existing flow models, however, the two most usual agitators for turbulent flow are the radial and axial agitators (*Castillo, 2013*).

According to an Inoxpa article, axial flow agitators are suitable for mixtures of immiscible liquids and solutions of solids in liquids since they prevent solids from being deposited in the bottom. The most common are marine propeller, gamma and lineflux. With the type of flow, vortices can be formed, if this phenomenon occurs deflectors are placed in the tank. Also, to maintain the phases at the desired temperature, shirts or heat transfer surfaces are placed.

Bachiller designs a wide range of agitated vessels with heating system and following ASME regulations. Stainless steel reactors with a capacity of up to 300,000 L that work in cycles and cover a temperature from -50°C to 400°C. Considering that working with lots of 100 kg, the oil phase tank will have a capacity of 50 L and the continuous phase tank of 150 L. The third tank, located at the outlet of the homogenizer, will also have a capacity of 150 L. The first two tanks where the pre-mixing of phases takes place will have a jacket as an exchanger, instead, the last one will have a half-round exchanger incorporated. In Appendix 3 there is a picture of the type of tank and agitator.

### Homogenization

The homogenization stage is necessary to reduce the droplet size. There are three possible devices for this stage: colloid mill, pressure homogenizer and ultrasonic homogenizer.

The selected equipment is the pressure homogenizer since, although they require more energy, they produce smaller droplets and are usually combined with agitated vessels. In addition, according to the article by Wibowo (2002), the best equipment for the homogenization operation is the pressure homogenizer or the ultrasonic homogenizer.

This equipment, which is formed by a compression block and a homogenization valve, allows to obtain a homogeneous, stable emulsion with small droplet sizes and a better dispersion of the active ingredients than that obtained in other types of homogenizers, so it results in a product of higher quality and efficiency.

In this equipment, the feed enters a high pressure of between a range of 3-20 MPa and then is passed through a narrow valve that reduces the droplet size due to the turbulence that occurs within the equipment (Figure 6.3) (Wibowo, 2001).

It has been decided to choose a GEA homogenizer since it reduces the size of the particles on a nanometric scale with a homogeneous dimensional distribution because the product is forced with high pressure through the valve with dynamic effects such as shear, turbulence, impact ... among others (GEA, *engineering for a better world*). The most effective homogenizers are Ariete homogenizers. Of all the existing models, it has been decided to choose Ariete 5400 homogenizer since it is the most powerful and versatile (GEA, *engineering for a better world*).

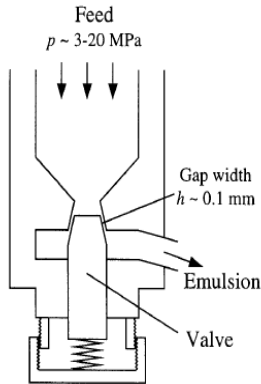


Figure 6.3. Diagram of a pressure homogenizer (Wibowo, 2001).

### Filling

After cooling and verifying that the product keeps to the properties of the specification sheet, the finished product is sent for packaging through a lobular pump. In addition, the installation can incorporate a SIL PIG product recovery system. The product will be sent to pack (Inoxpa).

## 6.3. PRODUCTION TIME

This section will estimate and represent the production time of a batch.

1. Filling the tank with hydrophilic components: 10 minutes
2. Mixing of hydrophilic components: 4 hours (*xanthan gum (solid) is added slowly for complete hydration and prevention of the appearance of lumps without hydrating*)
3. While the 4 hours are completed, filling the lipophilic components tank: 10 minutes
4. Mixing of lipophilic components: 15 minutes
5. Addition of pigments in the lipophilic components tank: 5 minutes
6. Mixing of lipophilic components + pigments: 10 minutes
7. After 4 hours and all well mixed, slowly add the mixture from step 6 to the hydrophilic components tank: 30 minutes
8. Stir the entire mixture: 1 hour

9. Pass the final mixture obtained by the homogenizer: 30 minutes
10. Once the mixture is in the formulation adjustment tank, cool it: 1 hour
11. Adding heat sensitive ingredients: 10 minutes
12. Continue cooling with stirring to room temperature: 2 hours
13. Take a sample to check product specifications: 30 minutes
  - a. If there is a need to make adjustments: + 1 hour
  - b. If there is no need to make adjustments: + 0 hours
14. Product packaging: 2 hours

Batches can overlap because when the ingredient tanks are empty, the next batch can start, so the cycle time is reduced. In addition, cleaning time between a change of cream and another (not tone) will be 20 minutes.

Everything explained above can be seen in the following diagram (Figure 6.4).

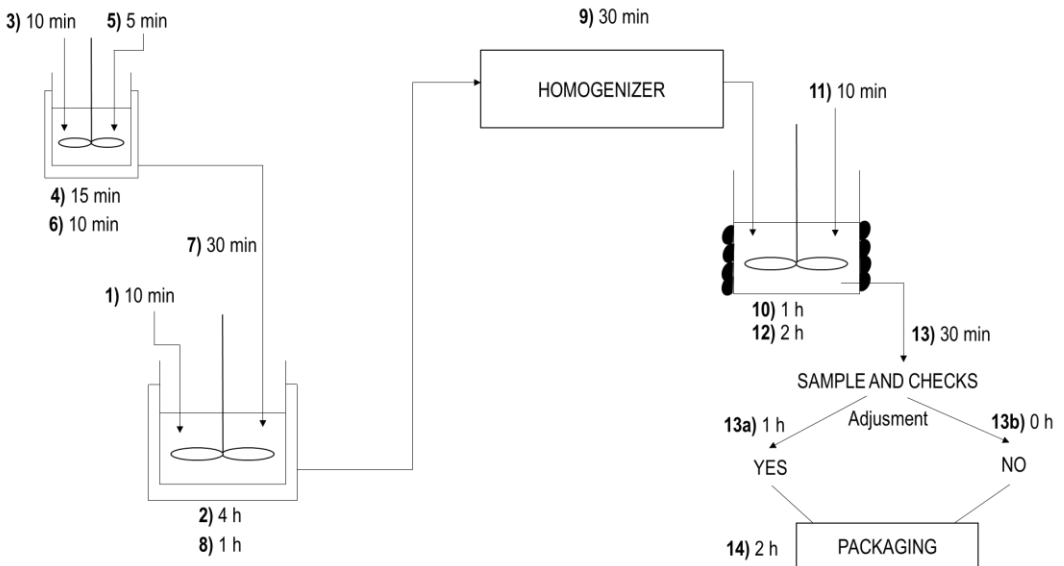


Figure 6.4. Production time representation.

## **7. CONCLUSIONS**

Three multifunctional facial creams, known as BB, CC and DD Cream, have been developed considering the main needs of consumers and market trends. From each, with different main functions, both its factors and quality indices, its ingredients and quantities of each and its delivery vehicle have been selected. In addition, the manufacturing process has been designed and the equipment selected.

The delivery vehicle for this product is an O/W emulsion with a droplet size of 20  $\mu\text{m}$ . The emulsion, consisting of active ingredients, surfactants and additives (pigments, fragrances ...), has a low viscosity when applied to the skin, a shelf life of 3 years and 12 months once opened and a minimum yield stress of approximately 6 Pa. In addition, although the creams developed in this work present a medium colour tone, in the design of the manufacturing process the three possible colour tones are considered: low, medium and high.

The selected ingredients are as natural as possible, multifunctional, not harmful to human health or environment, or irritating to the skin. They have been selected doing studies on market trends, patents, articles and encyclopaedias. The main differences between creams are their main functions, their smell and their coverage power.

A total annual production of 4,000 kg of cream has been decided. Each cream has a different production since they are intended for different types of consumers. The manufacturing process selected is by batches of approximately 100 kg of each type of cream of the same tone divided into three four-month periods, although depending on demand there may be changes in production. The selected equipment consists of agitated vessels for pre-mixing and mixing operations and a homogenizer to obtain the desired emulsion.





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# APPENDICES



## APPENDIX 1: LAYERS OF SKIN

### Epidermis

The epidermis, the outermost layer of the skin with a thickness of 0.1 to 1 mm, is formed primarily by two types of cells: keratinocytes (95% of epidermis cells), with the function of synthesizing keratin, protein with a large protective role, and dendritic cells. In a smaller proportion, 1-2% of epidermis cells, are cells called melanocytes that produce the pigment known as melanin (*Kolarsick et al., 2010*).

This layer has the functions of maintaining the proportion of water in human body and acts as a protective barrier against microorganisms. Epidermis is composed of four layers, starting with the outermost: horny cell layer or stratum corneum, granular cell layer or stratum granulosum, prickled cell layer or spinous layer and basal cell layer or stratum basale (*Jones and Bartlett, 2009*).

### Dermis

The dermis, separated from the epidermis by the basement membrane, is a layer of connective tissue with a thickness of 0.3 to 3 mm formed by three components: collagens, elastic fibres and cells. The first two components provide resistance, elasticity and extensibility to this layer. When the skin is stretched, collagen, thanks to its high tensile strength, prevents tearing and elastic fibres allow the unstretched state. As time goes by, the dermis becomes thinner and loses elasticity (*Blackwell, 2007*).

### Hypodermis

The hypodermis, the thicker and deeper layer of the skin, contains around 50% of the body fat responsible for producing energy and protecting and cushioning the body from injury or shock. It is formed by loose connective tissue and elastin and its main cells are fibroblasts, macrophages and adipocytes. The latter are fat cells responsible for storing energy as fat (*Montagna, 1974*).





## APPENDIX 2: DESTABILIZATION MECHANISMS

**Gravitational separation:** reversible destabilization mechanisms caused by phase separation due to a density difference. There are two types of gravitational separations: sedimentation and creaming. This sedimentation can be normal (settling up) or reverse (settling down) (*Kontogeorgis and Kiil, 2016*).

**Flocculation or coagulation:** destabilization mechanisms that are consistent in the aggregation of particles of the dispersed phase due to attractive forces. These mechanisms end up forming agglomerates due to the absence of repulsive forces that maintain a distance with which the particles are kept separate. Flocculation is a reversible mechanism while coagulation is an irreversible mechanism. This mechanism normally takes place in oil in water emulsions (*Kontogeorgis and Kiil, 2016*).

**Coalescence:** irreversible mechanism in which the drops, by random movement, form a new drop with a different shape than it originally had (*Kontogeorgis and Kiil, 2016*). Mechanism very similar to the previous one, but as the main difference, no agglomerates are formed here. This phenomenon can produce a complete phase separation.

**Ostwald ripening:** destabilization mechanism that describes the change of a structure over time. This phenomenon, very common in suspensions and emulsions, consists in the growth of large particles at the expense of small ones. This happens because the larger particles have more energy than the small ones since the molecules that are on the surface are less stable than those that are inside the particle. This mechanism normally takes place in water in oil emulsions (*Kontogeorgis and Kiil, 2016*).



## APPENDIX 3: SELECTION OF EQUIPMENT UNITS

Photograph of the type of tank and agitator model chosen.

