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Development of functional facial creams and their manufacturing process

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SUMMARY

Formulated product development starts from needs emerged from the society which are after transformed into commercial products. During the process some stages are taken into account such as product conceptualization, quality criteria, ingredient's selection and manufacturing process design.

Since people started being more self-concerned about skin diseases and the desire of always be well-looking, cosmetic creams have made an impact in cosmetic industry. The consumption of creams which improve our appearance and make us feel more self-confident is increasing every year. That is the reason why two different facial creams have been developed. One consists on an anti-aging method in order to reduce fine lines and wrinkles, and the other cream is an exfoliating and moisturizing cream which removes the dead skin cells and keeps skin hydrated. For both of them, a recipe has been proposed with its required quantity. They differ in function, aroma and texture but both are oil-in-water emulsions.

The design of a manufacturing process has been carried out with a production of approximately 10,000 kg of each facial cream per year. Approximate formulas with its ingredients and the required quantity, a production quantity and a process flowsheet with the necessary equipment and packaging design for each cream have been proposed.

Keywords: Product development, manufacturing process design, facial creams, emulsion, moisturizing, anti-aging.

RESUMEN

El desarrollo de un producto surge a partir de las necesidades de la sociedad y que posteriormente son transformadas en productos comerciales. Durante el proceso hay ciertas etapas que cobran un peso significativo como la conceptualización del producto, el criterio de calidad, la selección de ingredientes y el diseño del proceso de fabricación.

La gente cada vez empieza a ser más consciente de los problemas relacionados con la piel y del deseo de siempre lucir bien. Debido a esto, las cremas cosméticas han marcado un gran impacto en la industria cosmética. Año tras año, el consumo de cremas que mejoran nuestra apariencia y nos hacen sentir mas seguros de nosotros mismos crece cada vez más. Por esta razón, dos cremas faciales han sido desarrolladas. Una de ellas se trata de una crema anti-edad cuya función reside en reducir las arrugas provocadas por diferentes factores. La otra es una crema exfoliante e hidratante que elimina las células muertas de la piel y, además, la mantiene hidratada. Para ambas cremas, se ha propuesto una formulación con los distintos ingredientes y su cantidad aproximada. Se trata de cremas basadas en una emulsión aceite-en-agua, pero difieren en su función, aroma y textura.

El diseño del proceso de fabricación se ha llevado a cabo pensando para una producción anual de 10,000 kg de cada crema. Se ha propuesto una fórmula con unos ingredientes y una cantidad aproximada, una cantidad de producción, un diagrama de proceso con los equipos necesarios y un diseño del envasado junto con una distribución de las cargas durante el año para ambas cremas.

Palabras clave: Cremas faciales, hidratante, proceso de fabricación, emulsión, desarrollo del producto, exfoliante, anti-edad.

1. JUSTIFICATION AND OBJECTIVES

Humans have used various substances to modify their appearance or to accentuate their strong points for at least 10,000 years. Kohl was used by women in Ancient Egypt, a substance containing powdered galena (lead sulphide, PbS) to darken their eyelids. By 3,000 B.C. Chinese people started to colour their fingernails with gum arabic, gelatine, beeswax and egg. The colours used represented social class. What's more, Greek women used poisonous lead carbonate ($PbCO_3$) to reach a pale aspect.

In Europe, the higher class a person was, the paler was his/her skin. So, the highest classed of the European society frequently had the lightest-looking skin. As a consequence, European people often used to lighten their skin directly or used white powder on their skin to look more aristocratic. They used a large variety of products such as white lead paint, which, as if the toxic lead was not bad enough, it also contained arsenic.

As *Ullmann's Encyclopedia of Industrial Chemistry* defines: "Cosmetics have been defined as products created by the cosmetic industry and marketed directly to consumers. They represent a large group of consumer products designed to improve the health, cleanliness and physical appearance of the external parts of our body".

The function of cosmetics in society is often understated. Products such as soaps, shampoos and toothpastes, are an indispensable part of basic hygiene. Other products like sun care creams and rash lotions, play an important role in protecting the skin and health. Even make-up products contribute to society by raising self-confidence and improving the well-being of consumers. As said before, cosmetics have been and always will be an integral part of human society.

Nowadays, humans are self-concerned and love to look good and younger, feel great, be healthy and enhance our body features when possible. We try to cover or minimize any imperfection such as scars or wrinkles. The desire of achieving youth and beauty has never been more evident, it has become a societal norm proving that appearance is increasingly valued in much the same way as health. Looking good is considered to be a requirement for a happy and successful lifestyle.

Cosmetics can be grouped by product use into the following categories according to *Ullmann's Encyclopedia of Industrial Chemistry*: skin care and maintenance, cleansing, odour improvement, hair removal, hair care, decorative cosmetics and care and maintenance of mucous membranes. During this work, we will focus on the skin care and maintenance cosmetics. They include products that soften, hydrate (moisturizers), tone, protect (sunscreens) and repair.

To understand how these products work, it is important to understand the structure of the skin. It basically consists of three layers: epidermis, dermis and hypodermis. Epidermis, which is the surface part, consists of several layers. In the deepest of these, melanin or cutaneous pigment is produced, while in the dermis and hypodermis, new cells are formed which replace the dead ones. Skin and its structure will be described in more detail in the following chapters.

On the whole, in this work the main objective is the development of functional facial creams. This development includes since product conceptualization to production process basic design through the five stages that constitute the following chapters in this work:

Conceptualization of the product: In this chapter, a study of possible products to be developed will be carried out so that they can be conceptualized by defining the functionality, the mode of application and the product packaging. This study is carried out by looking for facial cream products already existing, comparing them, and studying patents, documents and encyclopedias in order to choose good products to develop and the production scale.

Establishment of quality factors: Once the products to develop have been conceptualized by defining their basic characteristics, the establishment of quality factors will be done. It consists on describing the principal factors that will ensure the product quality from the point of view of consumer acceptance and defining the quality indexes that allow quantifying compliance with quality factors.

Selection of structure and ingredients: As it is about creams, the structure is defined: it will be an emulsion. In this section, the type of emulsion, the volumetric fraction of the dispersed phase and the drop size will be defined. For achieving the functionality of each cream, active ingredients, the product delivery vehicle and the additives that will allow satisfying the quality index previously defined will be selected.

Synthesis of the manufacturing process: The plant must allow the manufacture of the products. The operations to be carried out, which will be assigned to the necessary equipment, will be selected.

Sizing and selection of equipment: In this chapter, the necessary equipment for the production scale will be dimensioned and the possible suppliers for the necessary equipment will be selected.

2. PRODUCT CONCEPTUALIZATION

Clearly, the first step when developing a product is conceptualizing the product and this is remarked by *Wibowo (2002).* At this point, it is interesting to take into consideration what consumers are asking or expecting for. It's important to satisfy consumer needs but it is also significant to succeed in the development of the product by innovating and being creative. That's the reason why making a research of actual market trends will be the very first step into conceptualizing the product.

One case that will be studied is a moisturizing and exfoliating cream. Most moisturizers (in fact, almost all cosmetic products in general) work exclusively on the cells in the epidermis layer. According to *Tiwari (2017),* the objective is to prevent the moisture from leaving the skin surface and/or refill its water content. Moisturizers basically provide a quantity of water that pass through the cell membranes to rehydrate and refill the external layers of dead skin cells. At the same time, exfoliating properties can help remove dead skin cells and uncover fresh and more youthful skin. Thus, at the same time we are removing our dead cells, we are hydrating our skin.

The other case will be an anti-aging cream. Skin aging is a complex process associated with significant changes in skin structure and its chemistry. It is a natural consequence of chronological aging (simply getting older) and is stimulated by external factors, such as an excessive UV irradiation from sun exposure which leads to photoaging (premature skin aging). Skin changes associated with photoaging are age spots, skin dryness, wrinkles and a general loss of skin elasticity, softness, smoothness and firmness.

2.1. MARKET TRENDS

As reported by *Wibowo (2002),* consumer needs and market trends need to be captured since will help us to understand the different formulas that industries have for the same product and the consumers' demand.

Most common forms of skin care products are emulsions, which consist of mixtures of two immiscible liquids that are stabilized against separation. There are two main types of emulsions, oil in water (O/W) where oil is present as the dispersed phase and water as the continuous phase, or, on the contrary, water in oil (W/O) where water is present as the dispersed phase and oil as the continuous phase.

O/W emulsions are the most usual formulated due to the fact that O/W emulsions tend to feel less greasy and its cost of formulation is lower because of a higher content in water. Moreover, they are used for common moisturizers, hand and body lotions.

Among typical trends of consumer wants and needs are the expectations that a product would last longer or cost less, a product small-sized, easy to carry when travelling or be safer as well as more environmentally friendly *(Wibowo, 2001).*

Consumers also tend to like a product that combines several complementary ingredients. For example, in our case, a cream that moisturizes and exfoliates at the same time. According to *Wess (1981),* some creams contain abrasive materials for removing dead skin cells. Such creams often leave the consumer's skin dry after its use and, in the case of the removal of dry skin, they can irritate the skin. Consequently, it requires a supplemental application of a moisturizing cream. That is the reason why the development of a two-in-one facial cream is more attractive to the consumers when buying a facial cream.

Referring to the product safety, it should be considered that the product should not contain toxic or allergenic ingredients, should not contain dangerous chemical for little children and it should contain more natural ingredients *(Wibowo, 2001).* For instance, it has been recently discovered that parabens used in cosmetic can stimulate the growth of certain types of breast cancer cells. Therefore, consumers will search for products not containing these compounds.

According to *Kumar (2005),* concern for healthy products came up with increased environmental awareness. Recently, users have been searching for creams containing as natural ingredients as possible. As a response, cosmetic industry has focused on developing creams that contain fewer chemical components and more natural ingredients such as proteins, vitamins and botanical ingredients. For this reason, enterprises compete to be the manufacturers of the best well-known cream in the market. In the following table we find a summary of the most common natural ingredients used in different types of creams after making a study in the cosmeceutical market.

Table 2.1. Natural ingredients in facial creams found in the market.

Ingredient	**Action**	**Market preparations**
Aloe-vera	Softens skin	Aloe-vera gel
Calendula	Softens skins, promotes cell formation	Calendula cream
Neem oil	Anti-microbial	Himalaya neem face wash
Rosemary extract	Antioxidant	L'oreal body cream
Cucumber	Antioxidant, refreshing	Everyouth cream

Finally, it is also important to consider in the product development the legal and environmental issues. The typical trends in this case are focused on being preferable that the product would be biodegradable and be more environmentally friendly. In the same way, consumers tend to search for creams not tested on animals before due to the amount of movements against animal testing before introducing it into the cosmeceutical market. According to *Kumar (2005),* numerous companies promoted cosmetic lines that were developed without animal testing. One example was *SafeBrands Inc*., which banned the use of animal testing in the development of their products.

These are the common market trends that had made an impact in cosmeceutical market, but in each type of cream there are different market trends that are important to talk about and to take into consideration.

Exfoliating and moisturizing cream

According to *Kraft et al. (2005),* there is a big number of moisturizers available on the actual market and consumer demand for these products is increasing as years go by. These products range from simple products that provide basic moisturization to luxury therapeutics with different benefits.

Nowadays, facial cleansing takes part in many of consumers' life. It is true that cleansing means to remove dead skin, dirt and sebaceous oil among other things, but it is also useful to prepare skin for moisturizers or other type of treatments. Moreover, facial cleansing is involved in maintaining the psychological health of consumers by contributing in the feeling of looking renewal and with a sense of rejuvenation *(Barlage et al., 2016).*

Consumers, since long time ago, tend to take more care of their face by cleansing and keeping moisturized than other parts of their body. That is the reason why many companies have developed many different formulas and cleansing forms that benefit different facial skin types.

Anti-aging cream

According to *Clarys et al. (2009)* the most common trends in consumers when asking for an anti-aging cream are an interest for “Green and Natural”, what means a substitution of cosmetic surgery and skin injections for more natural and less invasive cosmetics without the side effects.

Several studies show that it is possible to delay skin aging and to improve skin conditions by administrating selected nutritional supplements. Nutrition is the most important source of supplying antioxidants. What’s more, the most known antioxidants are vitamin C, vitamin E, carotenoids and even the trace elements copper and selenium.

Moreover, hyaluronic acid is very common because of its different roles in skin repairment, diagnosis of cancer, anti-inflammatory and immunomodulation among others. According to *Abbas (2018)*, hyaluronic acid has been widely employed as one of the most important components of the cosmetic products and for skin rejuvenation. Especially, it is important to highlight its anti-aging, face rejuvenating and anti-wrinkle properties.

2.2. PRODUCT FUNCTIONALITY

In this chapter, after making a research about the different market trends that rule the cosmeceutical market, the next step towards product conceptualization is defining the principal functionality of both creams. But first of all, it is interesting to have a little knowledge of our skin and its characteristics.

The skin is a vital organ for human life which plays a complex set of functions and covers the entire body surface *(Ribeiro et al., 2017).* Two important functions of the skin lie in maintaining the hydric balance of the organism and forming an effective mechanical barrier against external factors. The skin consists of three main layers: epidermis (0,1 to 1 mm) on the outside, followed by the dermis (0.3 to 3 mm) and the hypodermis consisting of fatty tissues.

The epidermis provides the barrier function by protecting from the entry of microorganisms and maintaining our body water content. Moreover, it is composed of four different layers including the stratum corneum (SC), which is formed of dead cells. The SC is the responsible of keeping this barrier function (Pott, 2006). In Figure 2.1, a schematic representation of the epidermis is shown together with the SC in more detail.

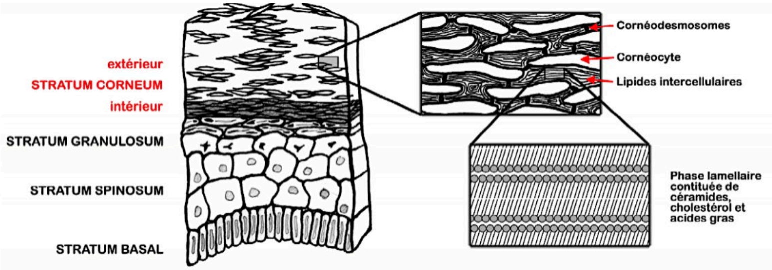

Figure 2.1. Different layers of the epidermis and the stratum corneum (Pott, 2006).

Exfoliating and moisturizing cream

As the name implies, it moisturizes and exfoliates so it will have to be carried out by two key ingredients.

On one hand, according to the article *Moisturizers: what they are and a practical approach to product selection* written by *Kraft et al. (2005),* moisturizers are a key component of basic skin care especially when there is a change of the epidermal barrier and when the water content in the epidermis decreases.

They are used to repair the barrier function of the epidermis, to cover small fissures in the skin, provide a protective film and increase the content of water of the epidermis. They contain diverse combinations of emollients, occlusives and humectants to achieve their beneficial effects, what makes possible a large variety of formulations available. Especially, moisturizers designed for face application are typically non-greasy, noncomedogenic emollients, with an emphasis on skin feel and aesthetics with maximal skin benefits.

Emollients (usually lipids and oils) soften the skin and contribute to improve the flexibility and softness resulting in a hydrated and improved appearance of the skin. There are different types of emollients such as astringent, dry, fatting and protective emollients.

Occlusives create a hydrophobic barrier over the skin in order to reduce TEWL (transepidermal water loss). They have the most pronounced effect when applied to slightly moistened skin. It makes the stratum corneum to turn out to be more hydrated than the underlying epidermis, making it suppler and softer. Their main limitations are odour, the potential allergenicity and the greasy feel associated.

Humectants are added to reduce the rate of dry-out. On one hand, they improve water absorption from the dermis into the epidermis, and on the other hand, in humid conditions they help the SC to absorb water from the external environment. The most effective humectant is glycerol but there are other common substances with humectant properties, for instance, hyaluronic acid, urea, propylene glycol, gelatine or honey.

On the other hand, an exfoliant is used in order to remove dead skin cells. This makes the skin look softer and with a youthful appearance, as well as allowing the living layers to grow and stay healthy.

Exfoliating creams commonly use an abrasive compound to remove dead skin cells from the skin which its friction is generated by the direct interaction of exfoliating particles. Common abrasives particles include powders from ground apricot seeds, crushed walnut shells, coconut shells, almond seeds and shells, various solid polymer powders and various inorganic particles such as sand, salt, alumina and silica among others. *(U.S. Pat. No. 2007/0020302 A1*).

According to *U.S. Pat. No. 2005/0169868*, apart from physical exfoliation, there is chemical exfoliation too. Some chemical compounds are alpha hydroxy acetic acid (AHA), beta hydroxy acetic acid (BHA), retinoic acid and enzymes. The difference between chemical and physical exfoliation is that chemical exfoliation acts on the bond of the squames while on the contrary physical exfoliation acts removing dead skin cells where the bond of the squames has already broken down.

Anti-aging cream

The anti-aging cream will have its main function on fighting against physiological alterations and progressive changes in each skin layer as well as changes in skin appearance, especially, on the sun-exposed skin areas.

Nowadays, skin health and beauty are considered one of the principal factors representing overall “well-being” and the perception of health in humans. That is the reason why several anti-aging strategies have been developed during the last years.

In agreement with *Abbas (2018),* aging is one of the very first factors which contributes to the formation of wrinkles due to the loss of skin elasticity, collagen reduction and elastin. All of these factors make skin become thinner and with a less smooth countenance. These morphologic changes associated with aged skin result in cutaneous laxity and wrinkle appearance on the face. The loss of thickness in aging skin starts with a thinning of the epidermis junction. Atrophy mainly occurs in the dermis because of morphological alterations related to aged skin.

Skin aging is a complex biological process influenced by combinations of intrinsic factors such as genetics, hormone or metabolic processes and extrinsic factors, for instance, chronic light exposure, pollution, chemicals and toxins. These factors go along with structural and physiological alterations and progressive changes in skin layers together with changes in skin appearance.

During one’s entire life, skin changes in its appearance and structure not only due to chronological and intrinsic processes but also due to various extrinsic factors, such as gravity, exposure to ultraviolet rays, and high levels of pollution. Moreover, lifestyle factors have important effects on skin aging, for instance, diet, smoking, diseases or stress.

With chronological aging, all the cells of the skin begin to produce an excessive number of free radicals (FR), which are unstable molecules of oxygen that in ideal circumstances are removed by antioxidants within the skin cells occurring naturally. The FR that are generated produce damage to the cellular membranes, proteins and the DNA. These free radicals may break down a protein substance in the conjunctive tissue (collagen) and liberate chemical substances that originate inflammation at the skin. Combining these cellular and molecular events lead to the skin aging and wrinkling.

According to *Ganceviciene et al. (2012),* there are two principal groups of agents that can be used as anti-aging cream components: antioxidants and cell regulators. Antioxidants reduce collagen degradation through reducing the concentration of FR in the tissues. Some examples of antioxidants are vitamins, polyphenols and flavonoids. Cell regulators, such as retinols, and peptides, have direct effects on metabolism of collagen and influence collagen production. Vitamins C, B3, and E are the most important antioxidants due to their ability to penetrate the skin because of their small molecular weight.

2.3. PRODUCT PACKAGING

Finally, the last part of the product conceptualization is defining the product packaging. The packaging can influence on consumer perception of the product, for this reason, it should be considered carefully (Wibowo, 2002).

The overall look of a cosmetic product is one of the most important factors of its market attraction added to its quality. Cosmetics packaging should be easy to brand, it should also allow the name for the product, the brand and other important information such composition or usage instructions to be printed and read as required.

What is more, packaging decision is only influenced by the overall product form and its choice may put additional limitations on the product. For instance, choosing a flip-cap bottle for a viscous product leads to the requirement that the product can flow through the small hole on the cap with ease.

According to *Shivsharan et al. (2014)* from DSTSM's College of Pharmacy, the best packaging solution for creams are plastic bottles with screw caps because of their robust body and affordable price. They can come in different types of shapes and sizes to set the specific requirements of the manufacturer.

Some advantages of plastic containers that are mentioned are:

- Low in cost.
- Light in weight.
- Pleasant to touch.
- Odourless and inert to most chemicals
- Able to retain their shape throughout their use.
- Durable.

Although these are usually round or a tube-like shape, plastic bottles can come in different styles. PP (polypropylene) plastic containers can acquire different shapes to satisfy the style or marketing of the cosmetic to be distributed. Jars and pots are commonly used for face creams made of PP or acrylic plastic. It is true that acrylic plastic is not as affordable as PP plastic, but it is a synthetic material that is typically clear, durable, light weight and does not produce any residue. It is ideal for cosmetic products because it allows longer shelf-life for the cosmetic product and it is more affordable as compared to glass.

For these two types of facial creams that will be studied in this work, the best packaging solution will be a screw cap bottle shaped as a jar made of acrylic plastic. These containers will have a maximum volume of 50 mL due to its casual use and ease of handling.

Figure 2.2. Different packaging shapes made of acrylic plastic.

3. ESTABLISHMENT OF QUALITY FACTORS

According to *Wibowo (2001),* the next step towards is to identify the desired performance in terms of quality factors. The satisfaction of the consumer is not only influenced by the ability of the product to perform a certain function, but also by other issues such as convenience of use, sensation and product durability.

While the primary concern is always functionality, quality factors such as the ease of application, stability and appearance are often crucial in consumer satisfaction. For instance, an important consideration of consumer perception when a moisturizing cream is applied to the skin, apart from being smooth or oily, over and above protecting the skin from dryness. Therefore, cream texture will take part in consumer's choice. What is more, a cosmetic product should be stable for over a year, taking into account possible changes in external temperature, humidity, and the presence of direct sunlight.

The final and most important goal, on one hand, of the moisturizing and exfoliating cream is to moisturize facial skin while we are exfoliating it, and on the other hand, for the anti-aging cream the most important is to stop the degradation of the skin primary structural constituents, such as collagen and elastin, to prevent the formation of wrinkles. However, there are other quality factors that should be considered and are important to keep in mind.

In terms of functional quality factors, the consumer expects a cosmetic cream to protect and clean parts of the body, to provide a protective coating and cause adhesion to a surface. In both cases that will be studied in this work, quality factors related to functionality are based on protecting and cleaning consumer's face, providing a protective coating and causing adhesion to the face surface. One case of consumers expectation of a cream would be to be absorbed quickly and provide immediate hydration.

For example, some functional quality factors of what an ideal moisturizer should be are (*Kraft et al. (2005):*

- Effective: By hydrating the SC, reduces and prevents TEWL.
- An emollient: makes skin smooth and reduces TEWL.
- Cosmetically acceptable.
- Moisturizing to sensitive skin: fragrance free, non-sensitizing, hypoallergenic.
- Affordable and long-lasting.
- Absorbed rapidly and providing immediate hydration.

3.1. SENSORIAL QUALITY FACTORS

It is significant to take account of sensorial quality factors that will make both creams more competitive. It is good that both creams feel smooth, do not feel oily, do not cause irritation and appear opaque.

In the case of the exfoliating and moisturizing cream, the exfoliating particles can cause irritation depending on the type of consumer's skin. We can distinguish between:

- Dry skin: lack of the right quantity of water in the most superficial layer of the skin (epidermis).
- Sensitive skin: Skin sensitive to emotional changes and environmental factors.
- Oily skin: Skin with an over production of sebum.
- Normal skin: well-balanced skin (not too oily and not too dry).
- Normal-to-oily skin: Skin can be dry or normal in some areas and oily in others such as nose, forehead and chine.

After deciding that it will be a physical exfoliation, the cream will be for all types of skins, but it is not recommended for oily skin. It is true that exfoliating a dry skin can cause irritation, but it is beneficious for these types of skin to apply and exfoliating cream that apart from removing dead cells, provides hydration. As it's our case, people with dry skin will have no problem in applying this facial cream.

On the contrary, for consumers with oily skin it is not surely recommended. If we provide more hydration to a skin that has an over production of sebum, probably, we will not get the effect we expected.

After each use, it is preferably to remove the cream using warm water.

Fragrance

No matter how effective a cosmetic may be that nobody will want to use it if it smells disagreeable. Consequently, smell is one of the key factors when deciding to buy a product.

Perfumes are closely related with cosmetic products, since it is very popular to add perfumes to cosmetics so as to make consumers feel good and clean. What's more, perfumes are sometimes added to cosmetics to cover undesirable odours that are caused by other cosmetic ingredients. It is true that fragrance improves the overall aesthetic qualities of cream, especially one that is applied to the face.

Fragrance is a component of creams and especially of facial moisturizers that is frequently dismissed as a potential irritant that is not necessary. In terms of cosmetics, the *Scientific Committee on Consumer Safety (SCCS)* raised the problem of fragrance allergens implicated in a high number of skin sensitization cases. Although, this though is becoming increasingly old-fashioned as the science supporting its proper use and evaluation is improved.

In table 3.1 we can see the perfume content depending on the cosmetic product. In this case, as facial creams are skin care products, the perfume content for both products should be around 0.01 and 0.5 %.

Table 3.1. Perfume contents usually found in cosmetic products.

Cosmetic product	**Content (%)**
Parfum	12 - 18
Skin care products	0.01 – 0.5
Hair care products	0.01 - 1
Bath preparations	0.1 - 3
Toothpastes	0.5 - 1

According to *Knowlton et al. (1993),* perfume oils are usually added to creams at levels of between 0.2 % and 0.5 %. Moreover, referring to *Díez (1998),* facial creams should contain between 0.1 and 0.2 % of perfume content. Facial creams are often white in colour and perfume raw materials which are highly coloured, or can discolour with time, should be avoided.

Referring to *Martini et al. (2006),* there are some raw materials which represent those fragrance families. For example, table 3.2 shows the comparation between some natural and synthetic raw materials related to perfumes.

Table 3.2. Comparation between natural and synthetic raw materials of fragrances.

Family	Natural raw material	Synthetic raw material
Parfum	Essence of Bergamonte	Linalool
Aromatic	Essence of Lavender	Linalyl acetate
Floral	Essence of Rose Otto	Phenethyl alcohol
Oriental	Absolute of vanilla	Ethylvanillin

According to this, it has been decided that both creams will have different aromas. The anti-aging cream will possess oriental aroma and the exfoliating and moisturizing cream will have a floral aroma.

Colour

Colouring agents are added to cosmetics in order to provide colour to cosmetics. There are two types: colorants (dyes) or pigments. For skin care products, colorants are used to colour cosmetic products. They are synthetic and organic colorant agents that are soluble both water and oil.

Cream coloration is not always recommended, that is the reason why creams are often conserved in white colour. In this work, both creams will be white-coloured because there's no necessity of colouring and both will be still well-looking.

3.2. RHEOLOGY

Moreover, rheological quality factors are also important to take into consideration. What most of consumers search when they are looking for the best cosmetic cream are creams that can be poured and spread easily when rubbed on the skin *(Wibowo, 2001).*

It is also important to take into account the fact that the cream should not flow readily under gravity but should be easy to stir and should not flow by itself but can be squeezed out of the container. For example, it should have low viscosity at high shear, flowing readily when rubbed on the skin. Moreover, its viscosity should increase meaningfully at low shear rates, so that it does not spill easily. That is to say, it is desirable that the cream presents a pseudoplastic rheological behaviour (Figure 3.1).

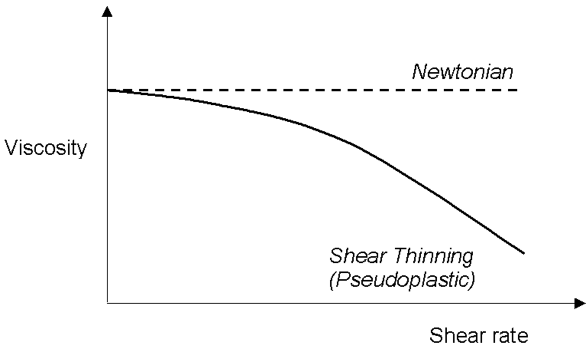

Figure 3.1. Viscosity of pseudoplastic behaviour as function of shear rate (Vlachopoulos et al., 2003).

According to *Wibowo (2001),* cosmetic emulsions assessed as good by a group of panellists have been found to have a low viscosity (about 0.025 Pa·s) when applied to the skin at high shear rate (5000 s^{-1} for creams). At very low shear rates, the viscosity can be as high as 1000 Pa·s.

Emulsion viscosity depends on overall ten entry parameters: shear rate, time, viscosity of continuous and dispersed phase, density of both phases, particle radius, concentration of particles, thermal energy, and interfacial tension.

High values of The Hydrophilic–Lipophilic Balance (HLB) (greater than 10) are characteristic of hydrophilic surfactants, with low HLB values (1-10) given to lipophilic surfactants. To concentrate the emulsifier at the oil/water interface it is desirable to maintain a good hydrophilic and lipophilic balance. Concretely, an O/W emulsion should have a surfactant with an HLB above 7 to promote its formation *(Laba, 1993).*

Table 3.3. HLB range depending on application (Tadros, 2013).

HLB range	Application
3 -6	W/O emulsifier
7 - 9	Wetting agent
8 -18	O/W emulsifier
13 - 15	Detergent
15 -18	Solubilizer

Finally, due to the fact that both creams should be applied in the same way, they should have similar rheological behaviour. Although, the abrasive function of the exfoliating and moisturizing cream makes having a higher viscosity than the anti-aging cream.

3.3. EMULSION STABILITY

As mentioned in the previous chapter of this work, both the moisturizing and exfoliating cream and the anti-aging will be O/W (oil in water) emulsions.

In agreement with *Sherman (1983)* an emulsion is a colloidal dispersion comprising two immiscible liquids. It is usually a two-phase system where one phase is dispersed in the other as microscopic droplets. We can differentiate two types of emulsions depending on the properties of the dispersed and continuous phase. On one hand, if oil is dispersed in water, the emulsion is known as oil in water (O/W). On the other hand, if water is dispersed in oil, then it is known as an emulsion water in oil (W/O). Consequently, both facial creams will be oil in water emulsions. According to Block (1988), the structure of most emulsions is based on a series of droplets dispersed in a continuous phase, with the particle size ranging between 1 and 100 μm.

Emulsions are unstable systems and require an emulsifying agent or emulsifier. These emulsifying agents are surface active agents that are added to the emulsions in order to stabilize both phases, what means, they are responsible for keeping both phases together for an extended period of time. Emulsifiers have a general form with one nonpolar hydrocarbon end and one polar end.

According to *Moravkova (2013),* the presence of an emulsifier shows two favourable inputs:

- A decrease in interfacial tension between both liquids
- A stabilization of the dispersed phase against coalescence.

Choosing the best emulsifier represents one of the final steps in the process of development of a new cosmetic lotion. Emulsifiers that are more soluble in water usually form oil-in-water emulsions, while emulsifiers that are more soluble in oil will form water-in-oil emulsions.

When talking about the stability of an emulsion, it occurs that there are processes that can make an emulsion unstable:

- Flocculation: The particles or droplets of the dispersed phase aggregate together due to attractive forces. It occurs when there is not sufficient repulsion to keep the droplets apart to distances where the van der Waals attraction is weak.
- Coalescence: Droplets of discontinuous phase join to form a larger droplet, increasing the average particle size over time.
- Ostwald Ripening: It is the change of non-homogeneous structure over time. With emulsions smaller droplets have larger solubility when compared with the larger ones (due to curvature effects). With time, the smaller droplets disappear, and their molecules diffuse to the bulk and become deposited on the larger droplets. Moreover, the droplet size distribution turns to larger values.
- Gravitational separation (creaming and sedimentation): Consists on the migration of the dispersed phase of an emulsion caused by the action of gravity and buoyancy without varying droplet size.

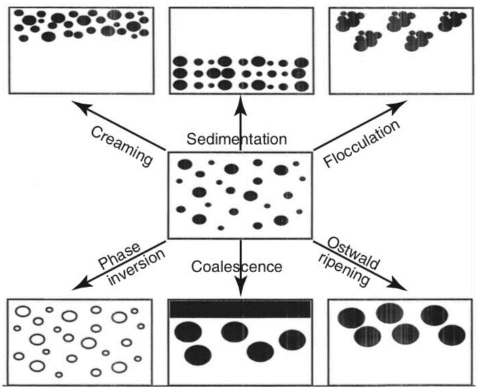

Figure 3.2. Representation of the different breakdown process in emulsions (Tadros, 2013).

Moreover, according to *U.S. Pat. No. 6,126,948*, despite the presence of emulsifying agents, some of these emulsions show a lack of stability over time reflected by a phase separation between the aqueous and oily phases of the emulsion. Therefore, in order to avoid instability, it is often necessary to include thickening agents. They are added to the emulsion and their main function is to create, within the aqueous phase, a gelled matrix which serves to set the globules of the fatty phase, providing for mechanical maintenance of the entire emulsion. Then, thickeners are used to control the viscosity and the rheology of the emulsion and help in maintaining the stability of the emulsion. The viscosity of a cream is specially settled by the thickener used and the viscosity of the external phase.

Shelf life is also very important when talking about creams. In agreement with *Laba (1993),* it is defined as the length of time before the properties of the emulsion become consumer unacceptable. Commercially available cosmetic products must have shelf life of several years to include a time allowance for distribution and storage and allow the consumer time to use the product fully. Stabilization of the processes achieved by increasing the viscosity of the continuous phase or by decreasing the particle size including a homogenization step.

Exfoliating and moisturizing cream

The exfoliating and moisturizing cream should exfoliate at the same time that moisturizes the skin. Usually, exfoliators can irritate consumer's skin and it is good to prevent dryness and irritation by moisturizing at the same time consumer's skin.

Due to the fact that the face is one of the most sensitive areas of our body, consumers demand for a cream that reduces dryness, enhance dull appearance, smooths and softens the skin. Moreover, these expectations should be followed by a moisturizer with pleasant sensory quality factors.

This cream should have small particles in order to exfoliate. There can be different sizes of these particles. According to *U.S. Pat. No. 5,658,577,* there are exfoliant particles which size is under 75 microns, a small size which is not perceptible to the consumer. It has been found that exfoliants of a concrete particle size range gives to the user a better exfoliation and moisturization simultaneously.

For *Massaro et al. (2005),* an effective size of exfoliant particles for exfoliating the skin where at least about 80% by weight of the particles have a particle size from about 100 microns to about 1000 microns.

In contrast, according to *Li (2013),* facial creams can contain scrub particles of equal or different size and equal or different shape. A study was made in order to discover which size and shape of scrub particles was the most effective. Three kinds of particles were used: scrub particles A were 100 μm size and irregular shape, B had an oval shape with two different sizes of scrub particles (125 and 200 μm), and scrub particles C size were 250 μm and uniform. After making the study and testing the three different creams, the conclusion was that the surface roughness values decreased after exfoliating the skin, but the surface roughness decreased more and the skin became smoother with scrub particles type B.

Moreover, if the exfoliating facial cream has two different sizes of scrub particles it removes more dead skin cells, which contributes to the moisturizing ingredients to a better penetration into the skin, led to a more hydrophilic skin surface and a better skin hydration.

In conclusion, scrub particles in this work will have same shape but two different sizes: 125 and 200 μm due to its effectiveness and with a medium hardness.

Anti-aging cream

The reason why an anti-aging cream will be developed is that it is necessary to stop or reduce the degradation of the primary structural constituents of the skin (collagen or elastin) in order to prevent the formation of wrinkles.

There are two main groups of agents that can be used as anti-aging cream components: the antioxidants and the cell regulators. L-ascorbic acid (vitamin C), due to its properties of being water-soluble and heat-labile, in concentrations between 5 and 15% was proven to have a skin anti-aging effect by inducing the production of collagen, as well as enzymes important for the production of collagen.

3.4. QUALITY FACTORS SUMMARY

In this point we summarize the quality factors for both facial creams related with fragrance, colour, emulsion stability, rheological behaviour and prerequisites or conditions set by us.

Table 3.4. Quality factors summary of the exfoliating and moisturizing cream.

EXFOLIATING AND MOISTURIZING CREAM	
Sensorial quality factors	- Floral fragrance - White coloured - Absorbed rapidly and providing immediate hydration - Removed with warm water - Physical exfoliation
Rheological quality factors	- Pseudoplastic rheological behaviour - Higher viscosity - Not flows readily under gravity but easy to stir
Emulsion stability quality factors	- O/W emulsion - Particle size between 1 and 100 μm - Shelf life of several years
Other quality factors	- 2 size of exfoliating particles

Table 3.5. Quality factors summary of the anti-aging cream.

ANTIAGING CREAM	
Sensorial quality factors	- Vanilla fragrance. - White coloured. - Absorbed rapidly and providing immediate hydration.
Rheological quality factors	- Pseudoplastic rheological behaviour. - Lower viscosity. - Not flows readily under gravity but easy to stir.
Emulsion stability quality factors	- O/W emulsion. - Particle size between 1 and 100 μm. - Shelf life of several years.
Other quality factors	- Use of antioxidant components.

4. SELECTION OF INGREDIENTS AND STRUCTURE

In this chapter, after defining the parameters and characteristics of both facial creams and stablishing the quality criteria, ingredients and structure will be selected as well as its quantity. The selection of ingredients and its quantity have been defined by analysing different formulas, articles and what is popular and most well-seen in the cosmetical market.

Consequently, two formulas will be proposed, one formula for each type of facial cream. The formulas suggested are tried to include natural ingredients and are tried to be innovative.

4.1. SELECTION OF INGREDIENTS

The very first step is identifing the active ingredients to meet the functionality requirements. In the same way, to meet the secondary requirements, such as appearence and ease of application, other ingredients are also needed in the product.

According to *Wibowo (2001),* some heuristics for the selection of ingredients are found:

- Multifunctional ingredients are preferred. What means selecting an ingredient, when possible, that can serve for more than one function.
- Favor the use of imperishable ingredients whenever possible.

4.1.1. Active ingredients

EXFOLIATING AND MOISTURIZING CREAM

In attemp to achieve the moisturizing function**,** according to *Kraft et al. (2005)* moisturizers contain varying combinations of emollients, occlusives and humectants to achieve their beneficial effects, and there is a large variety of formulations available, as said previously in chapter 2.

Emollients

Emollients are agents, mainly lipids and oils, which hydrate and improve skin appearance by contributing to skin softness and smoothness. Refering to *Appa (2016),* the objective of an emollient not only is to replace the absent natural lipids of the skin in the space between the corneocytes in the SC, but also smoothing of skin by changing the appearance of the skin and providing occlusion to attenuate TEWL and intensify moisturization.

Long chain saturated fatty acids and fatty alcohols are commonly used in cosmetic formulations. They possede positive effects on the skin barrier and on permeability. Some examples are stearic, linoleic, linolenic, oleic, and lauric, which can be found in palm oil, coconut oil, and wool fat.

Table 4.1. Examples of emollients (Kraft et al., 2005).

Type of emollients	**Substances**
Astringent emollients	Cyclomethicone, dimethicone, isopropyl myristate, octyl octanoate
Dry emollients	Decyl oleate, isopropyl palmitate, isostearyl alcohol
Fatting emollients	Glyceryl stearate, jojoba oil, octyl stearate, propylene glycol
Protective emollients	Diisopropyl dilinoleate, isopropyl isostearate
Protein Rejuvenators	Collagen, elastin, keratin

In attempt to choose the best emollient for our exfoliating and moisturizing cream*, De Polo (1998)* shows a sensorial analysis based on different emolients. This analysis studied four propierties for different emollients: ease of spread, skin feeling, after -10 min.- feeling and softeness. Each of one was marked with a number from 1 to 10:

- Ease of spreading: A high mark indicates a product which spreads easily.
- Skin feeling after application: A low mark indicates a dry emollient. A mark around 5 means a product with a rich feeling.
- The after-10 min.- feeling: A high mark indicates a product which leaves an oily and greasy feeling. Products marked with 1 to 2 are almost imperceptible.
- Softness: A high mark indicates a product related to skin care. A low mark indicates a product with a short residence time on the skin after applied.

Avocado oil, glyceryl triisostearate and mineral oil show low values of spreading, which means that the product spreads with difficulty. What's more, although they have high marks when talking about softness, they leave an oily and waxy feeling after applying to the skin. On the contrary, isoproyl mystrate and isostearate and octyl stearate have high values of spreading, however, they have low marks in softness which is not beneficial in our case. Jojoba oil and isostearyl isostearate acceptable values for each property.

Refering to *Sandha (2009),* due to the fact of being chemically close to human sebum, jojoba oil can provide the natural balance of the skin by forming a non greasy film that holds moisture in while controlling the flow of sebum. Moreover, it provides excellent spread ability as shown in the study made by *De Polo (1998).* The characteristic of being hypoallergenic and pure makes jojoba oil an ideal emollient for any type of skin.

Finally, the emollient choosen is jojoba oil.

According to *U.S Pat. No. 201724609 A1*, the preferred amount of emollient comprised from 3% to 60% and preferably ranging between 10 to 30%. Spetially, Rawlings et al. (2004) stablishes that in the case of face care products the quantity of emollients and occlusives range between 2 to 15%. Thus, jojoba oil will be in an amount of 10% by weight.

Humectants

Humectants are organic compounds soluble in water that can absorb a large number of water molecules. They are a key component in maintaining skin hydration. Some examples of humectants presented in table 4.2 are: glycerin, socium lactate and urea. According to *Appa (2016)* and *Kraft et al. (2005),* the most common and effective humectant is glycerol, aslo known as glycerin, due to its universal applications and its effects on multiple and different objectives.

Table 4.2. Examples of humectants (Kraft et al., 2005).

Humectants	
· Gelatin	· Glycerin
· Honey	· Hyaluronic acid
· Panthenol	· Propylene glycol
· Sodium and ammonium lactate	· Sodium pyrrolidine carboxylic acid
· Sorbital	· Urea

Its chemical structure brings together the stability of three carbon atoms with three water-seeking oxygen atoms in an anisotropic molecule that is perfectly designed for use in skin and hair moisturizers.

According to *Appa (2016),* glycerin is a prime candidate for facial moisturization because it acts on many different parameters with almost without side-effects. Moreover, when glycerol is combined with occlusive agents, there is a synergistic alleviation of dry skin.

In *U.S. Pat. No. 2015/080346 A1* and *U.S. Pat. No. 2017/0367954*, glycerin is used in concentration of 5 % by weight. According to *Wibowo (2001)* typical amount for glycerin is up to 20%. Moreover, *Rawlings et al. (2004)* remarks that the range of percentage contact of glycerin is 2 to 10% in face care products, and especially the ideal percentage is 5 %.

Occlusives

Kraft (2005) remarks that combining humectants and occlusive agents intensifies the water-holding capacity of the skin. Occlusive agents are basically used with the function of complementing the water-attracting nature of humectants in order to maintain epidermal water content and preserve the barrier function of the SC. They attenuate TEWL by creating a hydrophobic barrier over the SC and its interstitial areas.

There is an extensive list of occlusive agents, as table 4.3 includes. Although, not all of them are recommended for using in facial formulations. It is the case of petrolatum and lanolin. On one hand, in accordance with *Appa (2016),* lanolin is not recommended due to its odour and high allergenicity. On the other hand, petrolatum, first most common occlusive in moisturizers, is known to be an effective occlusive agent but it suffers from an unfavourable aesthetic.

Table 4.3. Some examples of occlusive (Kraft et al., 2005).

Type of occlusives	Substances
Fatty acids	Lanolin acid, stearic acid
Fatty alcohols	Cetyl alcohol, lanolin alcohol, stearyl alcohol
Hydrocarbon oils/waxes	caprylic/capric triglyceride, mineral oil, petrolatum, silicone derivates (cyclomethicone, dimethicone), squalene
Polyhydric alcohols	Propylene glycol
Sterols	Cholesterol
Wax esters	Beeswax, lanolin, stearyl stearate.

According to *Appa (2016),* silicone derivates have been used in moisturizers due to its occlusive properties apart from intensifying the aesthetic quality by adding a dry touch. In consonance with *Nolan et al. (2012),* dimethicone, a silicone derivate, is the second most common occlusive. Its main characteristics are based on being hypoallergenic, colourless, odourless and non-acnegenic.

U.S. Pat. No. 8,293,291 and *U.S. Pat. No. 2009/104174 A1* include dimethicone 3% in their cosmetic formulations. What's more, a *renewing face cream* formulated by *Lucasmeyer cosmetics* also included dimethicone in 3% by weight

The occlusive agent chosen will be dimethicone.

Exfoliating particles

In order to exfoliate the epidermis, the abrasive substances will be insoluble particles with its size and shape. As defined in chapter 3, exfoliating particles will have same shape (oval shape) and two different sizes: 125 and 200 μm.

According to *U.S. Pat. No. 2005/0169868* there is a wide range of different exfoliating particles used in cosmetic. For instance: olive stone, almond meal, walnut shell power, jade power, aluminium oxide, polyurethane, silica, etc.

In order to choose the right option, a research through patents and actual market trends has been done. As reported by *U.S. Pat. No. 6,432,430 B1*, a good option is walnut shell power. It is a natural ingredient made of walnut husks that has the best characteristic of being hypoallergenic. It is added to the facial cream in a quantity range of 5 to 7% concentration by weigh.

Other formulas include sea salt, dead sea salt (*U.S. Pat. No. 7,101,578 B1*), precious or semi-precious stones (*U.S. Pat. No. 2007/0020302 A1*). Synthetic exfoliating particles are dismissed. Natural exfoliating particles are preferred. Precious stones are discarded due to its price.

Finally, exfoliating particles will be made of walnut shell powder. It is well known for facial exfoliators for being natural and hypoallergenic.

ANTI-AGING CREAM

According to *Balistreri et al. (2017),* as years go by skin becomes more fragile, thinner and loses its natural elasticity.

Moisturizers play a role in treating and augmenting therapy for the aging face. Consequently, the antiaging cream formulation will be focused on choosing ingredients in order to moisturize the skin and prevent skin aging.

Emollients

Collagen is known as a protein rejuvenators emollient *(Kraft et al., 2005).* Referring to *Juncan et al. (2016)***,** collagen is a natural component of the skin which its presence in cream formulation is beneficial. Its function lies on stimulating young neo-fibrillogenesis and collagen ensure the permanent maintenance of skin hydration.

According to *Savary et al. (2016),* collagen is usually incorporated between 0.2 and 2% by weight. Moreover, it is remarked that collagen is included at 1% by weight in cosmetic creams.

Collagen will be chosen.

Moreover, shea butter is usually used as a great emollient. It is a fat extracted from the nut of the African shea tree. It has highly moisturizing and emollient properties due to shea butter's semi-solid characteristics, providing an ease of spreading when it is applied to the skin. It is often used in products such as hand creams, facial moisturizers or shampoos among others.

According to *U.S Pat. No. 2013/189207 A1*, shea butter is added in an amount ranging from 0.2 to 35%. *Juncan et al. (2016)* establishes an anti-aging cosmetic cream adding shea butter in a concentration of 2%.

As shown in figure 4.1, properties of jojoba oil and isostearyl isostearate are near to each other. Thus, for the anti-aging cream isostearyl isostearate will be chosen as a protective emollient. According to *Make it laboratory* the recommended concentrations for lotions and creams are between 3 and 6%. In line with *Rawlings (2004),* as typical amount of emollients and occlusive is between 2 and 15%, the concentration chosen of isostearyl isostearate will be 5%.

Humectants

Glycerin will be again chosen due to its excellent properties.

With age the epidermal concentration of hyaluronic acid decreases progressively. In women aged 19 to 47 years it is 0.03 %; in over 60-year-olds it is reduced to 0.015 %; and in 70-year-olds even only 0.007 %. Simultaneously during aging, a reduction and structural changes of collagen fibers occur. All factors lead to dehydration and loss of elasticity.

Hyaluronic acid will be chosen as a humectant too.

Abbas et al. (2018) made a study in order to analyse the anti-wrinkle efficacy of HA depending on its molecular weight. 76 females aged between 30 and 60 with wrinkles were applied with 0,1% by weight of HA cream formulation containing different molecular weights of HA twice a day for a period of 60 days. The results concluded that low molecular weight of HA was associated with significant reduction of wrinkle depth, which may be due to better penetration abilities of LMW-HA (Low Molecular Weight Hyaluronan).

Occlusives

From the list of the most common occlusives mentioned in table 4.3, squalane provides a geriatric type activity on wrinkled skin, reducing fine lines. Squalane is derived from squalene, and is the more stable form of this molecule, as well as being colourless and odourless.

Squalane will be chosen. Referring to *U.S Pat. No. 5,079,003,* squalene is used between the range of 0 to 1 % by weight.

4.1.2. Additives

EXFOLIATING AND MOISTURIZING CREAM

Preservatives

As reported by the *European Union Cosmetics Directive 76/768/EEC* preservatives are: "*Substances which may be added to cosmetic products for the primary purpose of inhibiting the development of micro-organisms in such products".* Moreover, Cosmetic Art remarked in 1976: *"Cosmetics are used for aesthetic reasons and should carry no health risks to the consumer".* For that reason, most of them need preservative agents.

According to *Polati (2007),* preservatives can be divided into 2 categories: antimicrobial and antioxidant preservatives.

On one hand, adding antimicrobial preservatives into cosmetic products is essential in order to prevent bacterial or fungal growth and all formulations containing water must include them. If microbial contamination occurs, it can cause changes in the viscosity and the odour of the cream and what's more, it can put the consumer at risk.

For *Polati (2007)* an ideal preservative should be:

- Effective at low levels.
- Tasteless, odourless and colourless.
- Effective against bacteria and fungi.
- Effective and stable in the pH range between 2.5 and 10.5.
- Cost-effective.
- Useable in hot and cold phases.

In table 4.4 few examples of common antimicrobial preservatives substances are shown.

Table 4.4. Some examples of antimicrobial preservatives.

Type of antimicrobial preservatives	**Substances**
Organic acid and their salts and esters	Sorbic acid, salicylic acid, benzoic acid and its salts.
Aldehydes	Formaldehyde
Phenols and derivates	Phenol, chlorophene, triclosan
Alcohols and derivates	Phenoxyethanol, dichlorobenzyl alcohol, benzyl alcohol
Imizadole and derivates	Climbazole, DMDM hydantoin, imidazolindinyl urea

According to *Kerdudo et al. (2015),* the most antimicrobial preservatives used are parabens, methylchloroisothiazolinone/methylisothiazolinone (MCIT/MIT) or phenoxyethanol. Since different studies exposed that concentration of parabens were found in human breast tumours, such as *Darbre (2004)* did, they became non-desirable ingredients for cosmetic consumers.

A. *Kunicka-Styczynska et al. (2009)* proposed using commercial essential oils as preservatives instead of synthetic preservatives. Essential oils studied were: lavender, tea tree and lemon oils in O/W. He concluded that combining two essential oils enhances the activity of the preservatives but the possibility of interactions between oils, synthetic preservative and the other components of the cosmetic cream makes it essential to test the proposed preservative system in specific formulations.

Sirilun et al. (2017) remarks that formaldehyde-releasing compounds, such as imidazolindinyl urea, have potential skin irritation properties among the consumers. Some cosmetic industries are focusing on preservative-free products, which are products without the presence of any antimicrobial substance. Nevertheless, preservative-free products usually contain ingredients with antimicrobial activity which are not yet identified as preservatives by the European Scientific Committee. Thus, referring to these products as self-preserving cosmetics is better than preservative-free products.

According to *Lebreux (2018),* one of the most suitable alternatives of parabens is phenoxyethanol. It works in a wide pH ranges and has a large spectrum activity against many pathogens. What's more, is stable and compatible with other preservatives. The maximum concentration allowed in cosmetics in Europe is up to 1%. *Lebreux (2017)* affirms that the mean concentration of phenoxyethanol was 0.46% and only 25% of the products had concentrations greater than 0.6%. Thus, phenoxyethanol will be use at a concentration of 0.4% by weight.

On the other hand, antioxidants are important too. In line with *Polati (2007),* antioxidants are ingredients that inhibit reactions promoted by oxygen, consequently the oxidation of the different compounds included in the cream is avoided. The most commonly used are butylated hydroxytoluene (BHT) and butylated hydroxyanisole (BHA) although it has been discovered that they are not beneficial at all. According to an article published by Lanigan and *Yamarik (2002),* BHT can damage lung tissues when it is applied to the skin and, what's more, BHA cause the underdevelopment of the reproductive system as reported by *Jeong et al. (2005).* BHA and BHT will be discarded.

Some examples of antioxidants commonly used are citric acid, gallic acid, glycolic acid, thiotic (or lipoic) acid and its derivates dihydrolipoic acid and nordihydroguaiaretic. Vitamins are often included to cosmetic formulations as antioxidants. Vitamin A (retinol), Vitamin E, Vitamin C and vitamin derivates such as retinyl palmitate, ascorbyl palmitate, magnesium ascorbyl, retinyl acetate are some examples of vitamins used.

In particular, according to *Casas (2007)* vitamin E is used as an antioxidant in cosmetic formulations. Its function lies in softening the skin and moderating dry skin conditions.

Natural oxidants are considered. *Polati (2007)* reports that the antioxidant action of natural oxidants is lower than the synthetic ones although synthetic antioxidants have more secondary effects.

Citric acid is included and widely used in cosmetic and especially in facial creams. *U.S Pat. No. 6,432,430 B1* and *U.S Pat. No. 8,293,291* are examples of formulas in which citric acid is included. Citric acid will be chosen as antioxidant compound. *U.S Pat. No. 6,432,430 B1* suggests including it in a range between 1 and 3%. Moreover, *Fiume et al. (2011)* remarks that citric acid is used in concentrations up to 4%.

Thickeners

Thickeners are used to control de viscosity and improve the rheological properties of stability flow and feel of the cream. They provide flexibility and there is a wide range of thickening agents.

According to *Laba (2001),* there are several ways to thicken creams:

- Adding solids.

- Increasing the internal phase ratio.
- Homogenizing.
- Adding thickeners

In this case, thickeners will be included to the formula. For instance, depending on the viscosity range, a certain thickener can be chosen. Figure 4.1 represents various thickening agents depending on the viscosity range.

De Polo (1998) suggests that in order to improve the thermal and electrolyte stability of O/W emulsions is necessary to use a hydrocolloid-thickener (long chain polymers). Two examples are xanthan gum or carbomer. Furthermore, consistency can also be regulated by adding polar, hydrophilic ingredients such as cetyl alcohol, cetearyl alcohol or glyceryl stearate.

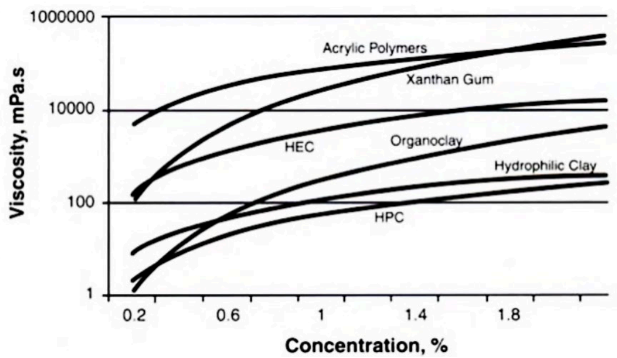

Figure 4.1. Viscosity as function of different thickeners concentration

Moreover, *Laba (1993)* remarks that an emulsion containing gums continued to hydrate and thicken the emulsion even after 45 days. What's more, a comparison of three different thickeners (xanthan gum, sodium carboxymethylcellulose and methylcellulose) about their effectiveness in retarding creaming showed that xanthan gum was the most effective and methylcellulose the worst.

Taking into account that cosmetic emulsions tend to have a low viscosity when applied to the skin at high shear rate and at very low shear rates, the viscosity can be as high as 1,000 Pa·s, we can conclude that the appropriate thickener for our facial cream will be xanthan gum. In figure

4.2 it is observed that a low shear rates xanthan gum shows a very high viscosity and at high shear rates, a low viscosity.

Xanthan gum will be chosen.

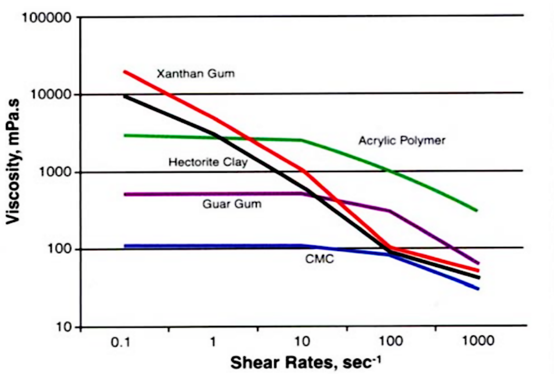

Figure 4.2. Shear flow viscosity of different thickeners as function of the viscosity. (D. Laba 2001).

According to *De Polo (1998),* the concentrations typically used are between 0.3 and 1%. Referring to *CP Kelko* solutions of xanthan gum at 1% or higher concentration appear gel-like at rest. At levels of 0.1 to 0.3%, same characteristics are perceived. From xanthan gum 0.5% (Figure 4.3), it covers the range of viscosity between 10^6 (at low shear rates) and 1 mPa·s (at high shear rates). Moreover, referring to the study made by *Krstonosic et al. (2015),* if the concentration of xanthan gum was between the range of 0.01 to 0.2 %, droplets flocculation occurs. What's more, under 0.08% creaming is enhanced and there's a face separation in the emulsion.

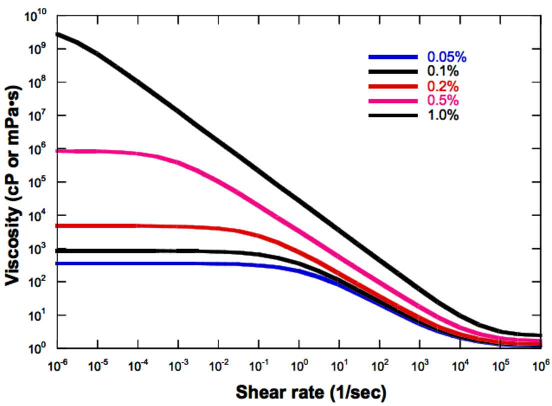

Figure 4.3. Shear rate as function of the viscosity depending on the concentration of xanthan gum (CP Kelko).

For these reasons, the concentration of xanthan gum chosen will be between 0.5% and 1%. Since the viscosity of this facial cream is required to be higher, xanthan gum 0.8% will be added.

Fragrances

In attempt to achieve the floral aroma for the moisturizing and exfoliating cream, the main raw material is phenylethyl alcohol (PEA). According to *Sirilun et al. (2017),* is a naturally occurring aromatic compound that is found in various flowers and is commonly used due to its flowery odor.

PEA will be chosen.

Cream formula summary

Table 4.5 Exfoliating and moisturizing cream formula.

Ingredient	% by weight
Water	72.6
Jojoba oil	10
Glycerin	5
Dimethicone	3
Walnut shell powder	6
Xanthan gum	0.8
Citric acid	2
PEA	0.2
Phenoxyethanol	0.4

ANTI-AGING CREAM

Preservatives

For the anti-aging cream, the antimicrobial preservative that will be choosen is DMDM Hydantoin. It is an odourless white and crystalline substance that it is usually used as a preservative agent in cosmetics and personal care products. This antimicrobial preservative is a formaldehyde donor, that is to a say that during the shelf-life of the cosmetic product, it realeases small quantities of formaldehyde.

According to the *Cosmetics Directive of the European Union* the maximum concentration allowed of DMDM hydantoin as a preservative agent in personal care produts is 0.6%.

Vitamins are excellent ingredients with antioxidant properties often used in anti-aging strategies.

Vitamin B3, also known as niamacide, not only reduces the appearance of wrinkes and fine lines but also improves reduction in sebaceus lipids and pore size, what means, improving skin texture and appearance. In a study made by *Bisset et al. (2017),* compared the tan per cent of reduction in total wrinkle lenght as funtion of time (figure 4.4). He conlcuded that the presence of niacinamide in 5% by wheigh, reduced the appearance of wrinkles. *Ganceviciene et al. (2012)* affirms that niamacide is used in 5% concentration as an anti-aging component.

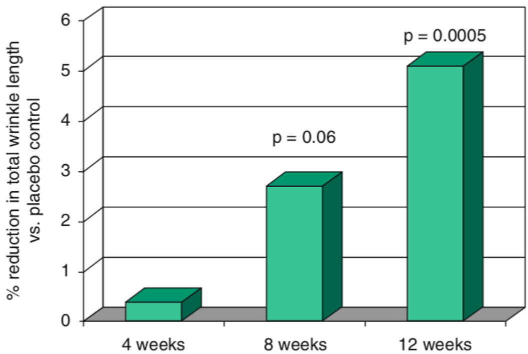

Figure 4.4. Tan per cent of reduction in total wrinkle length as function of time (Bisset et al. 2017).

What's more, he concluded that niamacide also improves the skin surface texture. Results are shown in figure 4.5.

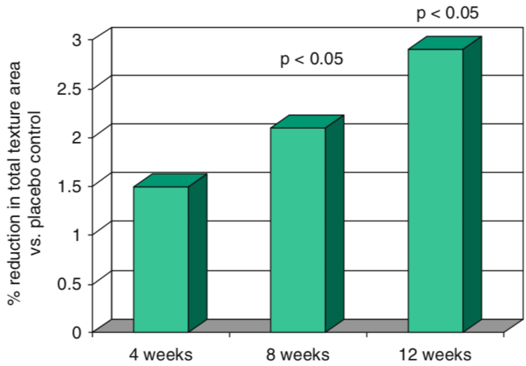

Figure 4.5: Tan per cent of reduction in total texture area as function of time (Bisset et al. 2017).

Vitamin E is usually used as an antioxidant in cosmetic formulation. It is abundant in the skin, especially in the SC, which is delivered there by sebum. Its functions lie on softening the skin and alleviating dry skin. According to *Casas (2007),* there are esterified forms which have a superior stability such as vitamin E acetate (tocopheryl acetate).

For moisturizing preparations, creams and lotions the concentration of use is ranging between 0.001 to 25% by weight (Cosmetic, Toiletry, and Fragrance Association). Moreover, referring to *U.S. No. Pat. 5,153,230,* formulations with vitamin E acetate at concentrations of 0.5 to 5% seem to be effective.

Finally, vitamin C (ascorbic acid) is found in citrus fruits, strawberries or mango among others. This vitamin increases the natural protection of the skin against UV radiation, reduces the amount of UV damage cells and protects against damage by reactive oxygen radicals. In this manner, *Burke (2007)* affirms that improving vitamin C levels in the skin is very important and delivery topically as a cosmetic cream can be really beneficial.

It is important for the production of collagen, although according to *Bisset (2017),* its stability is a challenge and it can lead to unwanted effects of skin irritation. Thus, formulations containing pure vitamin C have short shelf life *(Burke, 2007). Casas (2007)* remarks that some derivates of vitamin C have the advantage of being stable in cosmetic formulations and avoid the undesired efects of vitamin C. The most common vitamin C derivates are salts of scrobic acid such as sodium or magenisum ascorbate, sodium or magnesium ascorbyl phosphate, ascorbyl glucoside and ascorbyl palmitate.

In line with this, the most useful vitamin C derivate is sodium ascorbyl phosphate (SAP). It is easily soluble in water to concentrations to the limit of 50% by wheight. Moreover, it is a white powder that can remain stable in aqueous solutions. Its functions lie on preventing the formation of free radicals and increasing the firmness of the skin. SAP at a concentration of 3% by weight produces firmer skin *Casas (2007).*

Vitamin E acetate, B3 (niamacide) and SAP will be chosen as antioxidants.

Thickeners

Same thickener of the exfoliating and moisturizing cream will be used for the anti-aging cream. Xantham gum will be chosen.

Since the moisturizing and exfoliating cream viscosity is required to be higher, xanthan gum 0.5 % will be added.

Fragrances

In order to provide to the anti-aging cream the vanilla fragance desired, Vanilla Fragrance RR79226 will be added.

Cream formula summary

Table 4.6 Anti-aging cream formula

Ingredient	% by weight
Water	76.1
Isostearyl isostearate	5
Glycerin	5
Hyaluronic acid	0.1
Collagen	1
Shea butter	2
DMDM hydantoin	0.6
Vitamin B3	5
Vitamin E	0.5
SAP	3
Squalane	1
Xantham gum	0.5
Vanilla fragrance RR79226	0.2

4.2. SELECTION OF PRODUCT DELIVERY VEHICLE

The next step after selecting the ingredients is deciding the product delivery vehicle. According to *Wibowo (2001),* emulsion is the best option. Both creams will be an O/W emulsion as defined in previous chapters. The structure of most emulsions is based on a series of droplets which are dispersed in a continuous phase. The particle size of droplets is between 1 and 100 μm. If two or more immiscible phases are involved, the next stage is the selection of a suitable surfactant or emulsifier.

Laba (1993) remarks that the presence of an emulsifier as a stabilizer extends the inevitable separation of the phases and frequently is the major contributor to the flow characteristics of the entire system. In general, its structure is based on one nonpolar hydrocarbon end and one polar end.

Furthermore, the initial location of the surfactant (oil or water phase) is important for determining the type of emulsion that is formed. Referring to *Laba (1993),* depending on the initial localization of the surfactant, the final emulsion can have different physical properties.

Eccleston (nee Saunders) proposed that combinations of emulsifiers interact in the aqueous phase to form lamellar or crystalline structures, stabilizing and contributing to the consistency of the emulsion.

According to *Wibowo (2001),* there are some criteria for selecting the right emulsifiers:

- The combination of emulsifiers must stimulate the formation of an emulsion of the desired type.
- The final emulsion must be stable for a sufficiently long period of time and under various conditions that it may experience during processing, storage and application.
- The emulsifier must be compatible with other components in the system, such that there would not be side reactions occurring in the mixture.
- The emulsifier must not be hazardous to the costumer’s health or to the environment.
- The cost of the emulsifier must be as low as possible.

In agreement with *Wibowo (2001),* the most important point is achieving the desired emulsion type. The most common and widely used guideline for predicting emulsion type is that the continuous phase is the one in which the surfactant is more soluble. Moreover, according to *Prieto-Blanco et al. (2007),* non-ionic surfactants emulsify cosmetic formulations such as milks, creams, and lotions. Therefore, as our emulsion type is O/W, a non-ionic surfactant will be chosen.

What’s more, it is important to take into consideration health and safety issues. Cationic surfactants can irritate the eyes and for that reason, they are not appropriate for facial creams. Cationic surfactants will be discarded.

Anionic emulsifiers are sensitive at low pH values and electrolytes. Anionic surfactants will be discarded.

In accordance with *De Polo (1998),* the most widely used method of characterizing surfactants is the HLB (Hydrophilic Lipophilic Balance) system, as mentioned in chapter 3. The HLB value of an emulsifier quantifies its hydrophilic and hydrophobic tendencies. A low HLB value indicates a hydrophobic tendency, whereas a high value of HLB indicates hydrophobic tendency. *Wibowo (2001*) establishes that an HLB between 6 and 17 is usually suitable for making O/W emulsions.

A mixture of different emulsifiers, one with low HLB and the other with a high HLB value, provides better stability than a single emulsifier with the right HLB *(Cheng et al., 2008).*

Referring to *Polygon*, among all the emulsifiers and mixtures of them, the emulsion will be formed by glyceryl stearate alcohol and ceteareth-20. This mixture is an O/W emulsifier used in stable lotions. Its HLB is approximately 12, which is between the ranges of HLB values in O/W emulsions.

According to *Cosmetics Ingredients Database*, this mixture of emulsifiers is recommended for skin care, especially for facial care and facial cleansing among others. Moreover, *The derm review* remarks that ceteareth-20 can function as a surfactant and emulsifier in different skin care products including facial moisturizers and anti-aging treatments.

In order to determinate de quantity of emulsifiers in both formulas, *convergent cosmetics* suggest calculating the HLB of the mixture:

$$HLB = HLB_1 \cdot x + \ HLB_1 \cdot (1 - x) \qquad \text{(eq.1)}$$

Where:

- HLB_1: HLB (glyceryl stearate) = 3.8.
- HLB_2: HLB (ceteareth-20) = 15.2.
- *x:* percentage of glyceryl stearate in the oil phase.

If an HLB of 12 is required, the quantity in terms of percentage should be:

$$12 = 3.8 \cdot x + \ 15.2 \cdot (1 - x)$$

$$x = 0.28 \ \cong 0.3$$

So, the quantity of glyceryl stearate in the oil phase will be of 30% and the rest is ceteareth-20. The next step is to know the quantity of each ingredient in the cosmetic formula. In line with *convergent cosmetics*, the typical oil phase may contain 1 to 2 % of each emulsifier. Therefore, if we start with 2 % of total emulsifier:

$$100\% \rightarrow 2\%$$

$$30\% \rightarrow X$$

Calculating this, x= 0.6 %, where x is the quantity in percentage of glyceryl stearate in the cosmetic formula. Thus, 1.4 % will be the quantity defined of ceteareth-20.

4.3. SELECTION OF PRODUCT STRUCTURE

Finally, the selection of product structure is the last step toward the selection of ingredients and structure.

According to *Nienow et al. (1997),* in the case of creams and lotions microstructure can be created by *structurants* such as surfactants, polymers and crystals. In this manner, the cosmetic formula also includes active ingredients, which are the ingredients that provide the efficacy required to the product. Nevertheless, the properties of the final product not only are determined by the formulation, but processing conditions such as shear rates or orders of addition play an important role in defining product's microstructure, appearance and properties.

Wibowo (2001) remarks that it is necessary to estimate the viscosity at the actual shear rate. In table 4.8 are shown the estimated shear rate of typical application of creams.

Table 4.8. Common shear rates in processing and application of creams and pastes (Wibowo, 2001).

Action	**Shear rate γ (s^{-1})**
Suspending pigment or active ingredients	$10^{-3} – 10^{-1}$
Pouring from a bottle	$5 \cdot 10^{1}$ - 10^{2}
Extrusion from a bottle or tube	10^{1} - 10^{3}
Topical application of lotions/creams	$10^{2} – 10^{4}$
Application by brush	$5 \cdot 10^{3}$ - 10^{4}
Roller milling	10^{3} - 10^{4}
Forcing through homogenizing valve	10^{3} - 10^{5}
Colloid milling	10^{5} - 10^{6}

Moreover, microstructure also effects smoothness, stability and opacity. Referring to *Wibowo (2001),* an emulsion containing droplets about a range between 1 to 20 μm is more stable, smoother and may be transparent. Thus, cosmetic emulsion will have a droplet size of approximately 20 μm.

5. SYNTHESIS OF THE MANUFACTURING PROCESS

Once determined the ingredients and the product microstructure, the final step is designing the manufacturing process. According to *Wibowo (2001),* the objective is to design a process flowsheet that can produce both facial creams with the desired microstructure.

Process design establishes a series of chemical and physical operations, operating conditions, equipment needed to guarantee the functioning of the plant and materials of constructions of all process equipment, line sizes and principal instrumentation. Thus, it is shortened by a process flowsheet, materia and energy balances and a set of equipment specifications *(Couper et al., 2010).* This work will be focused not only in the process flowsheet and equipment selection but designing a quantity production for both facial creams. The rest of sections mentioned are beyond our scope.

The manufacturing process for both facial creams will be carried out as a batch process. A batch process is the process in which a sequence of operations are executed over a period of time on a separate, identifiable item or parcel of material *(Sharratt, 1997).* A wide range of chemical process industries such as pharmaceuticals, agrochemicals, perfumes and many more still maintain batch processing as their principal method of manufacture.

Batch process for both facial creams will be the right option because batch production has the flexibility to produce a variety of different products variations or different products apart from having lower capital costs. In this work the product will be a facial cream with two different compositions depending on its function *(Graphic products Editorial Staff).*

These products are destined to a group of consumers of around 100,000 people. It has been supposed that each one consumes 2 creams of each type per year. That is to say, an amount of 10,000,000mL produced per year of each cream, considering that one cream is packaged in a bottle of 50mL. Knowing the density of the ingredients that compound both creams and their respective quantity, we can approximate the final density of both creams. Both densities are close to 1 kg/L, so we will suppose that both creams will have a density of 1 kg/L. Therefore, the production is thought to be of 10,000 kg per year, or what is the same, 10 tonnes per year of each cream.

Due to the fact that they are high added-value products, we can stablish batches of 200 to 500 kg. So, if the desired production is about 10,000 kg by batches of around 400 kg, the number of batches per year will be 25. In order to reduce stock, the number of batches per year will be distributed into four trimesters of three months each one. In each trimester will be manufactured 6 batches of each cream except the last one that will have 7. That is an approximated quantity because the number of batches will depend on the demand. The plant is not only focused on manufacturing these two facial creams but this is beyond our scope.

It is to say that batches of 400 kg are supposed to start manufacturing, but it can be modified depending on the demand too. In order to produce these batches, the quantity of each ingredient (expressed in mass percentage) will be adapted to kilos and introduced to each agitated vessel where the biggest one will have a capacity of 500 L.

Two manufacturing process will be designed, one for the exfoliating and moisturizing cream and the other for the anti-aging cream since they will have little differences in the process flowsheet. The only difference will be the addition of a pre-mixing unit for the insoluble particles of the exfoliating cream. When producing the anti-aging cream, the same equipment will be used except for the pre-mixing unit of insoluble solids since the anti-aging cream does not contain any insoluble particles.

5.1. PROCESS FLOWSHEET

According to *Wibowo (2001)* the flowsheet for creams and pastes manufacturing process usually consists of only a few unit operations. A generic flowsheet will be as Figure 5.1

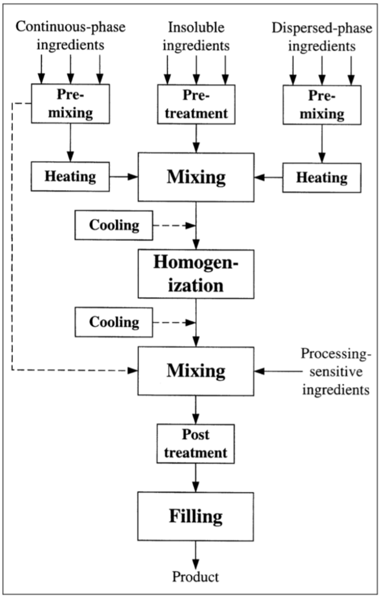

Figure 5.1. General manufacturing process for creams and pastes (Wibowo, 2001).

In pre-mixing units, the continuous-phase ingredients (water soluble ingredients) and the dispersed-phase ingredients (oil soluble ingredients) are mixed separately. If there are insoluble solids existing, they should be mixed separately too. After it, both phases (including insoluble ingredients) are mixed where the oil phase is dispersed as droplets in the water phase in order to form a pre-emulsion with relatively large droplet size. A homogenization step is necessary since the droplet size must be less than 100 μm and placed before cooling since it is desired to reduce the droplet size as much as possible, but still have a viscous final *product (Bornfriend, 1978).*

Two different process flowsheets will be proposed, one for the exfoliating and moisturizing cream and the other for the anti-aging cream.

5.1.1. Exfoliating and moisturizing cream

For the exfoliating and moisturizing cream, the process flowsheet is described in figure 5.2. An additional pre-mixing step is included in order to mix the exfoliating particles with different sizes. There is no pre-treatment step included since there is not the necessity of making a size reduction, exfoliating particles are ready to be used and well sized and shaped. Before mixing, continuous and dispersed phase ingredients are heated in order to facilitate mixing and minimize viscosity *(Wibowo, 2001).*

Emulsification step is executed mixing continuous, dispersed phases and the exfoliating particles with a posterior homogenization in order to reduce droplet size. A posterior cooling will be included in order to reduce the temperature to add heat-sensitive ingredients such as preservatives or fragrances *(Cheng et al,2009).*

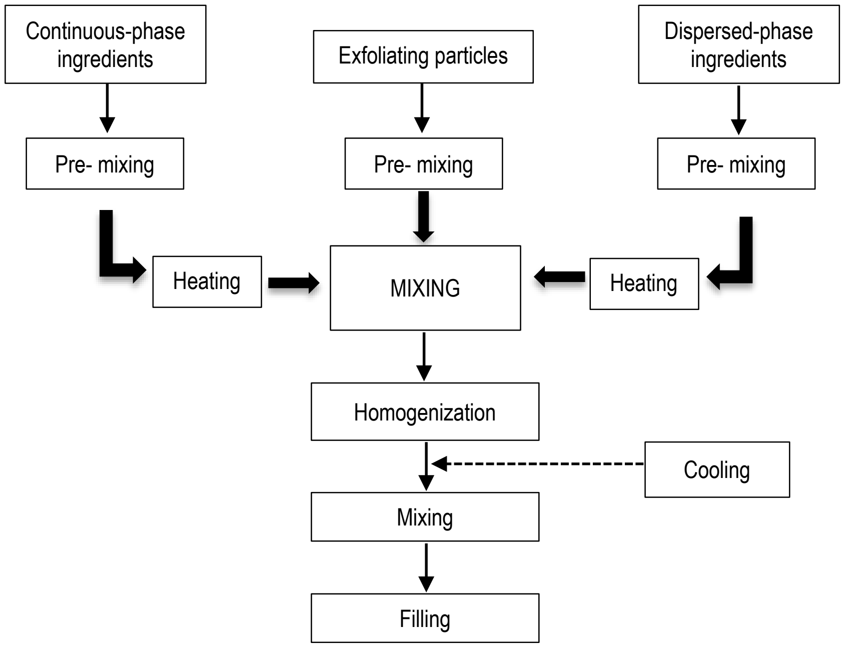

Figure 5.2 Process flowsheet for the exfoliating and moisturizing cream.

Pre-mixing / heating

Emulsifiers can be incorporated into the emulsion in different ways but if the purpose is to obtain an O/W emulsion, the best is to place all the surfactants in the aqueous phase *(Lin, 1967).* So, emulsifiers will be pre-mixed with the aqueous-phase ingredients. The continuous phase may include all water-soluble ingredients, including thickeners (xanthan gum). In addition, according to *Wibowo (2001),* if there is a thickener involved, it should be placed in the continuous phase. Increasing the viscosity of the aqueous phase produces more resistance to the movement of the oil phase, making the droplets to remain separate and not coalesce *(Labba, 2001).* Moreover, increasing the viscosity of the continuous phase will avoid phase inversion of the emulsion *(Norato et al., 1998).* Table 5.1 establishes the water-soluble and oil-soluble ingredients and the exfoliating particles forming the exfoliating and moisturizing cream.

Continuous and dispersed phase are heated up to a temperature usually between 70 and 80ºC to ensure that the oil phase is completely melted *(Grossmann, 2007).* Moreover, heating is important in order to facilitate mixing and minimize viscosity. The oil phase will be heated at 70ºC. The aqueous phase is heated at the same temperature.

Table 5.1. Continuous and dispersed phase and insoluble ingredients of the exfoliating cream.

Continuous phase ingredients	**Dispersed phase ingredients**	**Insoluble ingredients**
Water	Dimethicone	Walnut shell powder
Xanthan gum	Jojoba oil	
Citric acid		
Glycerin		
Ceteareth-20		
Glyceryl monostearate		

Mixing

The oil phase, aqueous phase and the exfoliating particles are mixed in order to form and emulsion. According to *Wilkinson et al. (1982),* if a vessel is initially filled with one phase prior to the addition of the second one, this initial phase will be the continuous one. With the objective of forming O/W emulsions, the oil phase is added to the aqueous phase while the mixture is stirred vigorously. Phase inversion is a spontaneous process to take into account although is not often encountered in cosmetic production provided that the production chemist is aware of the conditions under which it can occur. In order to prevent phase inversion, that is to say converting to a W/O emulsion, the oil phase is added slowly to the continuous phase to prevent droplets to coalesce.

Another technique is a procedure known as phase-inversion temperature. It basically consists on determining whether emulsions are W/O or O/W depending on the temperature. Increasing or lowering the temperature makes a variation on the HLB of the surfactant making it more hydrophobic. Moreover, according to the *International Federation of Societies of Cosmetic Chemists (IFSCC) (1997*), cooling below the PIT produces the formation of an O/W emulsion, whereas heating above the PIT makes a W/O emulsion. Thus, the emulsion should be heated, leading to a W/O emulsion, while mixing and then cooled in order to form an O/W emulsion with a reduction of the particle size. With this emulsion procedure, the homogenization step is not necessary since the required droplet size is achieved.

Figure 5.3 represents a typical PIT diagram where in the PIT range there is a zone where three phases are formed at lower levels of emulsifier concentration. At high surfactant levels, microemulsions may be formed.

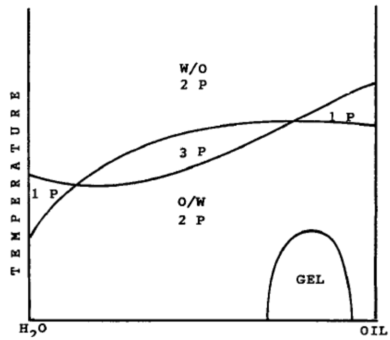

Figure 5.3 Process flowsheet for the exfoliating and moisturizing cream.

PIT method can be good alternative, but experimental procedures may be necessary to define initial and final temperatures, cooling speed and polish quantity of surfactants that are needed. All options are considered but the best option in order to produce an emulsion will be adding the oil phase to the water phase slowly while stirring.

Homogenization

The homogenization step is included in order to reduce the final droplet size to 1-20 μm (for cosmetic applications) of the emulsion *(Wibowo, 2001).* The process up to this stage is formed at 70 ºC.

Cooling and mixing

The cream is cooled back at room temperature and preservatives (phenoxyethanol) and fragrances (PEA) are added in a mixing step after temperature is dropped to about 40-50ºC since they are heat-sensitive ingredients *(Wibowo, 2001).* Stirring is usually stopped when temperature reaches a value of 35ºC and rarely continues under 25ºC.

Filling

After including all ingredients and mixing, finally the cream will be packaged in 50 ml acrylic plastic bottles, as mentioned in chapter 2.

5.1.2. Anti-aging cream

The process flowsheet of the anti-aging cream is similar to the other facial cream. The only difference is that in the anti-aging cream the pre-mixing step of insoluble solids will not be included.

The process flowsheet of the anti-aging cream is described in figure 5.4.

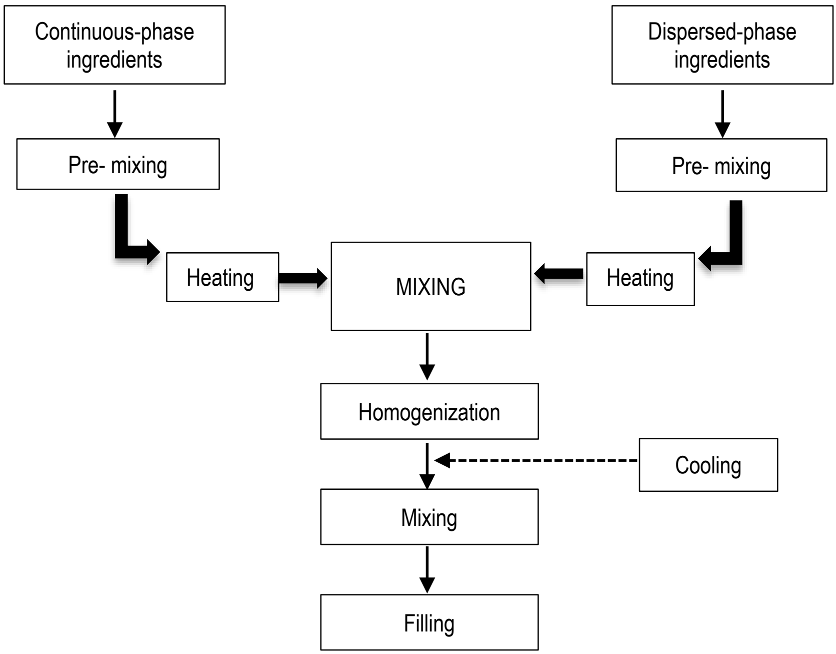

Figure 5.4. Process flowsheet for the anti-aging cream.

Pre-mixing / heating

Continuous and dispersed-phase ingredients are pre-mix separately and heated up to 70ºC. Emulsifiers are incorporated to the aqueous phase along with xanthan gum. In table 5.2 ingredients are divided depending if they are water or oil soluble.

Table 5.2 Continuous and dispersed phase and insoluble ingredients of the exfoliating cream.

Continuous phase ingredients	**Dispersed phase ingredients**
Water	Isostearyl isostearate
Hyaluronic acid	Vitamin E
Niamacide (B3)	Squalane
SAP (Vitamin C)	Shea butter
Xanthan gum	Collagen
Ceteareth-20	
Glyceryl monostearate	
Glycerin	

Mixing

In this stage, following the same procedure as in the other facial cream, continuous and dispersed phase are mixed together at 70ºC.

Homogenization

In order to reduce particle size (1-20 μm), a homogenization step at a temperature of 70ºC is included.

Cooling and mixing

After cooling to a temperature of 40-50ºC, DMDM hydantoin and vanilla fragrance are added in a mixing step.

Filling

After including all ingredients and mixing, finally the cream will be packaged in 50 ml acrylic plastic bottles.

5.2. SELECTION OF EQUIPMENT UNITS

Pre-mixing and mixing

According to *Wibowo (2001)* an agitated vessel, in turbulent shear, is usually used for pre-mixing, mixing steps and dispersion of solids into liquids. As shown in figure 5.4, agitated vessel is the equipment unit which offers a wide range of capacity and has low energy consumption.

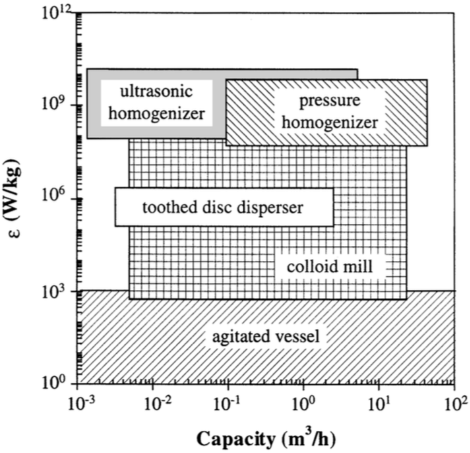

Figure 5.4 Typical capacity and energy consumption for selected emulsification units (Wibowo, 2001).

Standard reactor designs are considered sufficient for most process since it is desired minimizing costs and simplifying the design *(Jakobsen, 2008).* In agitated vessels there are various parameters and design variables than can be estimated such as agitation speed, mixing time, drop size distribution, breakup and coalesce or phase inversion among others.

Drop breakup and coalescence are two basic phenomena when talking about turbulent liquid-liquid mixing. Breakage and coalescence can reach a dynamic equilibrium with no changes in drop size distribution when steady-state dispersion is achieved. But if there is no equilibrium, with a minimum change in agitation speed or in concentration of the oil phase coalescence will increase over breakage. Moreover, higher volume fractions of the oil phase also produce an intensification in the interaction rate between droplets and a following increase of both phenomena *(Ghotli et al., 2013).*

On one hand, the occurrence of droplet breakup depends on the balance between the disruptive forces generated by the equipment, which tend to pull the droplet apart, and the interfacial forces, which tend to keep it intact. Breakage occurs when the ratio between them, the Weber number N_{We}, exceeds a critical value which depends on the viscosity ratio of the dispersed and continuous phase. In turbulent flow, droplet disruption can be represented by the local turbulence energy dissipation rate ε. With this and the ratio $\tau_{adsorption} / \tau_{collision}$ (Eq. 1, Eq.2) the necessary shear rate or ε to produce and emulsion with a certain droplet size can be estimated *(Wibowo, 2001).*

$$\tau_{\text{collision}} = \frac{d_p^{2/3}}{15\ \phi\ \varepsilon^{1/3}} \qquad \text{(eq. 2)}$$

$$\tau_{\text{adsorption}} = \frac{10\ \Gamma\ \mu^{\frac{1}{2}}}{d_p\ m_s\ \rho_c^{\frac{1}{2}}\ \varepsilon^{1/2}} \qquad \text{(eq. 3)}$$

On the other hand, phase inversion can be generated not only if the impeller speed is increased but volume fraction of the dispersed phase too. *Kato et al. (1991)* suggested that the aqueous phase became continuous when the agitation speed was less than 400 r.p.m and when it was over 1050 r.p.m., the phase with higher viscosity became dispersed.

When selecting the type of impeller there are some parameters to take into consideration: viscosity of the fluid, system flow regime and operating conditions. There are basically two types of impellers for turbulent flow: radial and axial impellers. Radial flow impellers create two circulating loops where between them mixing takes place. On the contrary, in axial flow impellers the fluid flows along the bottom of the vessel and rises up to form a single loop *(Jakobsen, 2008; Paul et al., 2004).*

For pre-mixing, axial flow impellers are used in mixing miscible liquids at low viscosity and high speed *(Paul et al.,2004).* Some examples are marine propeller and pitched blade turbine. In the case of turbulent mixing, solid body rotation phenomena and central surface vortices may take place. In order to avoid these two phenomena, baffles are placed in the tank wall. Moreover, heat transfer surfaces such as helical coils, harps coils or platecoils are placed inside the vessel and jackets in order to make the vessel wall and bottom head heat transfer surfaces.

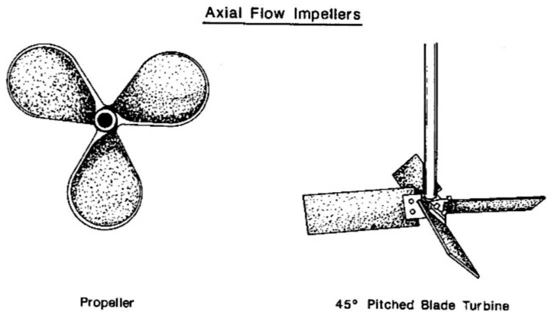

Figure 5.5 Typical axial flow impellers

The company *GMM Pfaudler* offers a DIN Reactor AE which consists of a two-piece design with a standard agitator system based on an impeller-type agitator with baffle *(DIN Reactor AE Catalogue).* A standard jacket is provided supporting a maximum design temperature of 200ºC. Both reactors, for the aqueous and oil phase, will be chosen. With batches of 400 kg, the agitated vessel of the oil phase will have a volume of 63 L and the aqueous phase of 500 L.

In the case of pre-mixing the exfoliating solid particles (insoluble solids), a mixer for powder should be chosen. *Bachiller Barcelona* offers such a wide range of equipment units where the vertical conical screw mixer is the chosen one (Figure 5.6). It consists on a vertical conical recipient with a mixing system based on a vertical screw working through the entire volume of the cone, forcing an upward flow of the product. It I characterized for having a short mixing time (maximum 10 minutes), capacities from 100 to 50,000 litters, batch-wise operation and very easy to clean. For this work, the volume required will be 100 L.

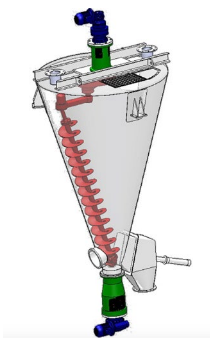

Figure 5.6 Solid pre-mixing equipment for the exfoliating particles (Bachiller Barcelona)

The next step towards pre-mixing is pre-emulsification or mixing. This step is produced at low shear conditions and turbulent flow in which both phases are mixed and form a pre-emulsion with a droplet size of 100 μm *(Butler, 2000).* For this conditions hydrofoil or axial flow impellers are suitable (Figure 5.7). According to *Ghotli et al. (2013)* turbine impellers are a suitable option for dispersion of immiscible liquids, even these that are quite viscous. Since it is desired to produce batches of 400kg, the mixing vessel, where both phases are mixed, will present a capacity of about 500 L.

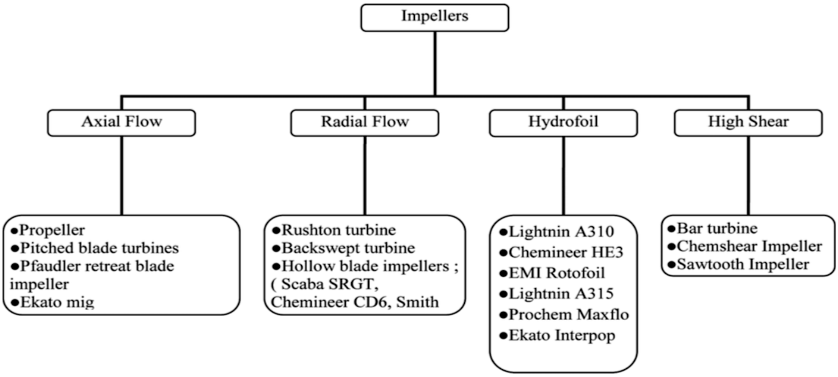

Figure 5.7. Common impeller types (Ghotli et al., 2013)

Homogenization

As described previously, the emulsion needs an emulsification device in order to reduce the droplet size. There are three main equipment units for high shear homogenization step: colloid mill, pressure homogenizer and ultrasonic homogenizer. Colloid mill is a preferred high-shear mixer compared to the others since requires less energy consumption. It usually employs a rotor having rotor blades which move at high speed relative to a stator formed by fixed walls of a mixing cavity.

In colloid mills, the degree of shear produced depends on the rotational speed, rotor radius and the gap width between the stator and the rotor. The rotation speed can vary between 1,000 and 10,000 r.p.m. and the rotor-stator gap between 0,05 to 0,5 mm *(U.S. Pat. No. 3,635,834).*

In order to choose the required homogenizer, the company *Bachiller Barcelona* offers such a wide variety of equipment for emulsification. In concrete, the *Bachmix Fixed* wich consists of a high shear rotator stator agitator (Figure 5.8). It is useful for capacities from 500 to 20,000 L, operates at atmospheric pressure and optionally up to 3 bar, it has a heating (cooling jacket operating with hot water, vapor or thermal oil. What's more, it holds an integrated inlet of solids and liquids which can be useful for both creams. For this current work, the volume required will be of 500 L.

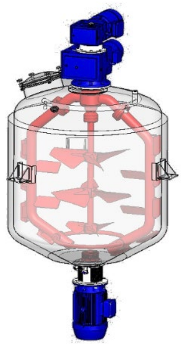

Figure 5.8. Bachmix Fixed for homogenization (Bachiller Barcelona).

Cooling and mixing

After cooling, heat-sensitive ingredients are added in a mixing unit without stop stirring. The final mixing unit will be characterized as the pre-mixing and mixing steps with a volume of 500 L.

6. CONCLUSIONS

Two type of facial creams have been developed by establishing their quality factors, selecting the suitable ingredients and the product delivery vehicle and finally, designing their manufacturing process and selection of equipment. The two facial creams developed are an exfoliating and moisturizing cream and an anti-aging cream. The main differences between each other remain on their functionality, aroma and texture.

Once it is determined the product delivery vehicle as an O/W emulsion, suitable surfactants and other additives have been selected. The selection criteria of surfactants, thickeners and other additives such as preservatives or fragrances was based on typical market trends and studying different articles. Same surfactants were chosen for both creams since the characteristics and objectives are similar.

The exfoliating and moisturizing cream presents a floral fragrance, low viscosity when applied to the skin and no phase separation for a long time. The function of exfoliating the skin is carried out by exfoliating particles of walnut shell powder. The anti-aging cream presents a vanilla fragrance, low viscosity when applied to the skin and no phase separation for a long time. Both creams were intended to include as many natural ingredients as possible and to avoid irritating and harmful ingredients for the skin or human health.

It has been decided to work as a batch process with an approximated production of 400 kg of each facial cream with a total of 10,000kg per year for each facial cream. Changes in production were considered since it depends on the demand. To carry out the manufacturing process, agitated vessels were chosen for pre-mixing and mixing steps and a homogenizer to create a final stable emulsion. Same equipment was thought to be used for both facial creams

Table 6.1 summarizes all the ingredients chosen depending if they are active or additive ingredients and the emulsifiers.

Table 6.1. Composition for each facial cream

		Exfoliating and moisturizing cream	Anti-aging cream
Active ingredients	Continuous phase	Water, citric acid, glycerin	Water, hyaluronic acid, niamacide, SAP, glycerin
	Insoluble solids	Walnut shell powder	
	Dispersed phase	Jojoba oil, dimethicone	Isostearyl isostearate, vitamin E, squalane, shea butter and collagen
Additives		Xanthan gum, PEA, phenoxyethanol,	Xanthan gum, vanilla fragrance, DMDM hydantoin
Emulsifiers		Glyceryl monostearate, ceteareth-20	

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