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# **Treball Final de Grau**

Development of a range of air fresheners for homecare and preliminary design of their manufacturing process

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Perfume is a story in odor, sometimes poetry in memory.

Jean-Claude Ellena

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### SUMMARY

Every day we perceive different smells in the street, at work, on public transport, in the home... These smells can be pleasant and bring pleasant memories to mind, but sometimes they are unpleasant and we tend to try to change that sensation. Society is faced with the need to neutralise these unpleasant smells wherever they are present. Specifically, this project has focused on the space of the home, a place where people tend to spend a large part of their time. It has therefore been decided to develop a range of air fresheners for domestic use. The development of formulated products is a process in which the needs of society or the consumer are identified and transformed into commercial products.

Firstly, in order to carry out this process of developing the product range, the analysis of current trends and market needs have conceptualised the different products. With this information, it was decided to create three air fresheners, one aerosol type, one liquid plug-in type and the last one gel type. The functionalities and packaging of the product were also determined.

Next, in order to achieve the effectiveness of the air freshener range, quality factors have been established for the three types of air fresheners. The selection of ingredients and a formulation proposal for each air freshener is then carried out.

The difference between the three types of air fresheners is their mode of diffusion of the volatile active substance. This has an impact on the product development discussed above. The aerosol type air freshener will contain a propellant, Propel 38, which will assist in the atomisation of the volatile substance. As a consequence of this ingredient needs a certain container under pressure, a metal container with a capacity of 250mL has been chosen.

On the other hand, the liquid electric plug-in air freshener will be a homogeneous solution that by means of a wick and the increase of temperature, the volatile active substance can be spread in the air. This leads to a different container, a glass container with a capacity of 20mL has been chosen. The gel type air freshener will be an absorbent body in which the volatile active substance will be contained. The formation of this absorbent body will be by means of an ingredient called gelling agent. Therefore, the packaging is different again, it is now a rectangular plastic container with slots in the top and a capacity of 150g.

Finally, a preliminary design of the industrial manufacturing process for each air freshener has been proposed and equipment has been selected. In general, an agitation tank will be needed to mix the ingredients and homogenise them; and packaging, capping and packing equipment for the distribution of the product.

**Keywords**: Air fresheners, aerosol, gel, plug-in liquid, fragrance, propellant, gelling agent, process of synthesis, home care.

# RESUMEN

Día a día se perciben distintos olores por la calle, en el trabajo, en el transporte público, en las viviendas,... Esos olores pueden ser agradables y traer recuerdos placenteros a la mente, pero en ocasiones son desagradables y se tiende a intentar cambiar esa sensación. La sociedad se ve con la necesidad de neutralizar dichos olores molestos en cualquier lugar que estén presentes. En concreto, este proyecto se ha centrado en el espacio de la casa, sitio en el cual las personas suele pasar gran parte del tiempo. Por lo que se ha decido desarrollar una gama de ambientadores de uso doméstico. El desarrollo de productos formulados es un proceso en el cual, las necesidades de la sociedad o del consumidor son identificadas y transformadas en productos comerciales.

En primer lugar, para llevar a cabo este proceso de desarrollo de la gama de productos, el análisis de las tendencias actuales y las necesidades de mercado han conceptualizado los distintos productos. Con esa información se ha decidido realizar tres ambientadores uno tipo aerosol, otro tipo líquido de enchufe y el último tipo gel. Asimismo, se ha determinado las funcionalidades y envase del producto.

A continuación, con tal de conseguir la eficacia de la gama de ambientadores se han establecidos factores de calidad para los tres tipos de ambientadores. Seguidamente, se procede a la selección de ingredientes y a una propuesta de formulación para cada ambientador.

La diferencia entre los tres tipos de ambientadores es su modo de difusión de la sustancia activa volátil. Esto repercute al desarrollo del producto comentado anteriormente. El ambientador tipo aerosol contendrá un propelente, Propel 38, que va ayudar a la pulverización de la sustancia volátil. Como consecuencia de ese ingrediente necesita un determinado envase que vaya a presión, se ha escogido un recipiente de metal con capacidad de 250mL.

Por otro lado, el ambientador eléctrico líquido de enchufe será una solución homogénea que mediante una mecha y el aumento de temperatura, la sustancia activa volátil se podrá esparcir por el aire. Esto desemboco a un envase diferente, se ha escogido un recipiente de vidrio con capacidad de 20mL.

Con respecto al ambientador tipo gel será un cuerpo absorbente en el cual contendrá la sustancia activa volátil. La formación de este cuerpo absorbente será mediante un ingrediente llamado gelificante. Por esto, el envase vuelve a ser distinto, ahora es un recipiente de plástico rectangular con ranuras en la parte superior y una capacidad de 150g.

Finalmente, se ha propuesto un diseño preliminar del proceso de fabricación industrial para cada ambientador y se han seleccionado los equipos. De forma general, se necesitará un tanque con agitación para mezclar los ingredientes y homogeneizarlos; y equipos de envasado, tapado y empaquetado para la distribución del producto.

**Paraules clau**: Ambientador, aerosol, gel, líquido de enchufe, fragancias, propelente, gelificante, síntesis de proceso, cuidado del hogar.

# **1. INTRODUCTION**

Every day we perceive different fragrances, smells and aromas that are pleasant to the olfactory sense, but what is the reason for these aromas? Evidently it may be due to the scent given off by the surrounding nature. However, another factor to take into account is that the development of certain industries has triggered the manufacture of products with aromatising characteristics. This has been reflected in the growth of the global market for air fresheners. According to market research, it is expected to grow at a CAGR (Compound annual growth rate) of approximately 3,5% during the period 2018-2025 (Allied Market Research Website,2022).

First of all, it should be noted that the use of air fresheners is not a recent development in society. Historically, perfumes were not only used to provide scents for the human body, but also for bedding, rooms and even sacrificial offerings. The word perfume comes from the Latin word "per fumus" or "from smoke", which historians say means that perfumes were originally used in sacrificial halls to mask the smell of burnt offerings. Therefore, air fresheners and perfumes are not an entirely separate entity and have been around for a long time (Academia del Perfume Website, 2021).

Therefore, an air freshener can be defined as a consumer product that emits a fragrance to provide an aroma to a space, to mask an odour or both. According to the International Fragrance Association a fragrance is a chemical mixture that has an aroma but also encompasses historical, cultural, social and emotional value. For this reason, one advantage of such a product is the variety of fragrances available to choose the one you like the most and which has a personal meaning. Also, air fresheners are essential devices for our home and car, as well as for other places.

This work focuses on air fresheners for domestic use. In the home there are different places where to place an air freshener; the most important places are the bathroom, the dining room and the bedroom. There are also different types of air fresheners. They can be presented in different forms depending on the form chosen for their diffusion. One classification can be as follows:

Aerosol

- Gel
- Plug-in liquid
- Scented candle
- Diffusers
- Partially submerged batons, commonly known as Mikados
- Etc.

Due to the existence of different formats, the production of three different air fresheners covers better the demand of the people, since there are preferences according to the place where it is going to be used and the advantages that the format presents for the person.

# **2. OBJECTIVES**

As mentioned before, the market trend of air fresheners is increasing. In order to adapt to the current trend and meet the demand in this sector, the main objective of the work is the development of a range of air fresheners, as well as the preliminary design of their manufacturing process.

In order to achieve the main objective, a systematic procedure proposed by Wibowo (2002) has been followed, divided into the following steps:

- Conceptualisation of the product. The aim of this first section is to describe the product, understand people's needs through a study of market trends and establish the characteristics of the product range under study.
- Identification of quality factors. Identifying and defining the quality factors that will make it possible to define the formulated product and thus know if it satisfies the detected need. It also involves establishing the evaluation of these factors.
- Selection of ingredients. Taking into account the steps described above, the objective of this stage is the selection of ingredients to form the product in order to meet the quality factors previously identified. For this purpose, an intensive research of the products available on the market will be carried out in order to propose a product formulation.
- Preliminary design of the manufacturing process. The objective of this section is to carry
  out a preliminary design of the production including the quantity produced, the production
  structure and the selection of the necessary equipment to obtain the desired products.

# **3. CONCEPTUALIZATION**

When conceiving a new product, it is logical to think that you have to start by defining the most general concepts and then go on to detail them. In this way, success in manufacturing and market entry can be achieved.

First of all, the physical form of the desired product is to be defined. The physical form is classified as solid, semi-solid, liquid or gaseous. As there are three different types of air fresheners, each has a different physical form. The liquid plug-in type has a single phase, homogeneous liquid form, and the gel format is semi-solid, also known as soft solid. With respect to the aerosol type air freshener, no such physical form can be appropriated to it. An aerosol is a suspension of liquid particles in a gas, which is contained in a pressurised container. In general all three air fresheners are formulated products that are obtained by mixing components to obtain the desired attributes in the new product.

The conceptualization of a product can be analysed from different perspectives. Through studies of market trends and consumer needs that go hand in hand with the functionality of the product. As well as, the packaging design is necessary to define the packaging of the product.

### 3.1. MARKET TRENDS

The global air fresheners market was valued at USD 10,124.4 million in 2017, and is expected to garner USD 13,279.1 million by 2025, registering a CAGR of 3.5% over the period 2018 to 2025 (Allied Market Research Website, 2022).

The growth of the air freshener industry is owing to the demand for air care products and increased concern for indoor air quality. Also, potential high disposable income and improving lifestyle of consumers offer lucrative opportunities for market development. Moreover, other factors expected to drive the market are rapidly growing car sales, increasing number of pets, and higher willingness of consumers to make use of high-quality air fresheners. However, factors such

as high operational cost are expected to hamper the widespread adoption and act as the major restraint for the growth of the global air fresheners market.

The global air fresheners market is segmented on the basis of product type, application, customer type, and geography.

The product type segment comprises aerosols, electric air fresheners, gels, candles and others. The aerosols segment accounted for the largest share in the product type segment. The aerosols market is driven by ease of use and availability across the globe. On the other hand, the gel air fresheners section is expected to witness substantial growth at a CAGR of 4.2% during the forecast period. This is attributed to increasing adoption of this product for use in cars.

The market based on application is segmented into residential, corporate offices, cars, and others. The residential, domestic section has dominated the application segment in the air freshener market. This is attributed to favourable demographics and increasing inclination towards sanitation across the globe. Air fresheners, such as sprays, electric air fresheners, gels, and candles are preferred for residential purposes, owing to increasing concern for indoor air quality. On the other hand, the car segment is projected to grow at a higher CAGR of 4.0%. This is attributed to growing automobile sales, and demand for aromatic products based on natural plant extracts and the launch of new car air fresheners in the form of gel cans. This will further enhance the overall market demand for the automotive air fresheners industry during the period 2021-2026 (Industry ARC Website, 2021).

Depending on the type of customer, it is bifurcated into individual customers and enterprise customers. Individual customers had the largest share in this segment. Also, attractive advertising and ease of availability also drive growth. The enterprise customer segment is expected to witness the highest CAGR of 4.3% due to increasing adoption of air care products in offices and other places of interest to maintain good environmental hygiene.

Geographically, it is analysed in North America, Europe, Asia-Pacific, and LAMEA. The key players operating in the market include Procter & Gamble Co, Reckitt Benckiser Inc, Henkel KGaA, Church & Dwight Co Inc, Car-Freshener Corporation, SC Johnson & Son Inc, Kobayashi Pharmaceutical Co. Ltd., Godrej Household Products Ltd., Farcent Enterprise Co. Ltd., and Newell Brands.

In summary, the key findings of the air fresheners market could be as follows:

- Based on product type, the aerosol section was the largest contributor to the air fresheners market and is expected to grow further.
- Based on application, the household section was the largest contributor to the market in terms of value.
- Based on customer type, the individual customer segment is expected to continue to grow at a steady CAGR.

According to Wibowo and Ka M.Ng (2002) the typical market trends to consider in consumer product development are consumer wants and needs, product safety, and legal and environmental issues.

#### 3.1.1. Consumer wants and needs

The main need of consumers who require an air freshener is that provides a pleasant smell or eliminates unwanted odors in a space. However, as time goes by, more desires that people want are being added. Increasing environmental awareness has given rise to the need for natural, eco-friendly air fresheners.

Due to all these new demands, manufacturers are focusing on developing new and innovative products by using advanced technologies. Key players are differentiating their offerings in terms of functionalities, scents, nature of ingredients, size and packaging format in order to gain a competitive advantage.

#### 3.1.2. Product safety

In order to ensure that emissions are not hazardous it will be a control on the quantity/proportion of components. Also, try to substitute hazardous chemical ingredients with natural ingredients. As well as following the IFRA (International Fragrance Association) code of practice, which contains recommendations for the use of substances used as ingredients in fragrances, grouping them into three categories: prohibited, restricted and specified according to quality and purity criteria.

#### 3.1.3. Legal and environmental issues

Consider the changes made to the regulation with regard to chemicals. Commission Regulation (EU) No 474/2014 of 8 May 2014 amending Annex XVII to Regulation (EC) No 1907/2006 of the European Parliament and of the Council on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) as regards 1,4-dichlorobenzene. As well as ensuring correct labelling in accordance with the corresponding regulations.

### 3.2. PRODUCT FUNCIONALITY

The main function of an air freshener is to impart a scent to the environment or to mask odours, with the intention of creating a pleasant interior space. For this reason, the smell is an important parameter for obtaining the final product.

Odour can be defined as a sensation, a perception and a stimulus produced in the sense of smell by the interaction of an organic substance with the olfactory receptors. This interaction depends on the volatility of the substances, since it is necessary that the molecules of the odorous substances are in a gaseous phase to be perceived by the nose (Herrera et al., 2010).

It should be noted that each person appreciates the smells differently, so when choosing a type of fragrance everyone has different preferences.

### 3.3. PRODUCT PACKAGING

In this section we will describe the characteristics of the packaging of our products.

The main purpose of product packaging is to protect and transport the contents to the point of distribution and use. In addition, it should be noted that in the air freshener sector, the packaging of the product is very relevant due to the fact that there are different ways of diffusion. Likewise, visual design has taken on greater significance in society. The aesthetics of the product must attract the consumer as well as convey a feeling related to the characteristics of the product and it is important to differentiate it from competitors. This can be done through colour, shape or logo.

As the desired products are different, they do not have the same mode of diffusion, from each other, a suitable container will be chosen for each one.

### 3.3.1. Aerosol

A non-reusable cylindrical metal container has been chosen for the convenience of the customer when holding the container and to avoid any residue, thus avoiding risk from flammable components. The lower part is arched inwards to better contain the pressure generated. In addition, it has a diffuser for controlled dosing in terms of output time, application area and amount of product, avoiding waste due to overdosing. It also provides a high shear rate so that the liquid dispersion is broken into small droplets. The different parts of the metal container, the can, summarised are as follows:

- One valve
- One nozzle
- Metal container

On the other hand, the colour or pattern plays an important role visually for the product. As we want to convey a floral feeling, we have decided on a combination of the colour violet and pink related to spring, the blossoming of flowers. Regarding the product label, it will be printed on the product container and it will show the mode of use and composition of the product, among other aspects.

With regard to the volume of the container, it has been decided that it should contain 250mL. In Figure 1, a possible packaging without the visual design is shown.

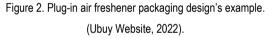


Figure 1. Selected container without visual design. (Aerosol la Revista Website, 2022).

### 3.3.2. Plug-in liquid

A rectangular glass container has been chosen. In addition, it will contain a wick immersed in the solution, a plug attached to the mouth of the container and a lid. As the container is to be plugged, it needs to be made of a material that is resistant to temperature increase and does not alter the composition of the substance inside. Also, in order not to take up space and inconvenience consumers it does not have to be a large container but can hold a capacity of 20 mL.

In this case the product label will not be printed on the container but will be placed on the distribution packaging. In Figure 2 a possible packaging of the product is shown.



#### 3.3.3.Gel

A rectangular container with slots at the top and plastic material, specifically PP (Polypropylene) plastic, has been chosen. In addition, it is important that the container can withstand the heat, as it will act as a mould for the liquid product. The label of the product will be placed in the upper part, as it has grooves. This way, at the time of use, the label is removed and the smell of the air freshener is released.

Blue has been chosen as the colour, as it is associated with stability and freshness. Also, the packaging has a capacity of 150 grams. Figure 3 shows a possible packaging of the product.





Figure 3. Gel air freshener packaging design's example (Own elaboration).

### PRODCUT PACKAGING SUMMARY

All three types of air fresheners have the same function, which is to scent a specific space. However, due to their different mode of diffusion, a suitable container is needed for each one. The following table 1 summarises the product packaging data.

Parameter	Aerosol	Plug-in	Gel	
Container	Metal can	Glass bottle	Plastic box	
	- non-reusable cylindrical metal container	- Transparent container - With a wick to diffuse the aroma	- With grooves at the top	
Characteristics	- Parts: valve, nozzle and container		- Resistant to temperature	
	- Pressurized container		increase	
Printed design etiquette	Product's name, brand's name, instructions for use, composition, manufacturing and expiration date			
Capacity	250mL	20mL	150g	

Table 1. Summary of the product packaging.
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# **4. QUALITY FACTOR**

Once the products have been conceptualized, the quality factors are determined in order to verify the requirements that must be met so that, in addition to satisfying the identified need, the product is differentiated from its competitors and is attractive and convincing to the end consumer. Depending on the form of the product or the delivery system, the quality factors may be different.

In order to quantify the quality factors it is useful to introduce the term quality index. The quality index can be defined as properties that can be measured and/or tested in order to evaluate the established quality criteria.

The article by Wibowo and Kang M.Ng (2002) shows some examples of quality criteria and quality indices that consumer products are expected to possess.

If you want to obtain three products with different shapes, not all the quality criteria will be the same. The sensory quality factor, which in addition to the analysis of the senses is related to the analysis of market acceptance of the product, is the criterion that will be applied in the same way for the range of products.

### 4.1. SENSORY QUALITY FACTOR

In this section, the factors commonly analysed with reference to sensory analysis will be described. In the UNE-EN ISO 5492:210 Standard, sensory analysis is defined as the science related to the evaluation of the organoleptic attributes of a product through the senses. This allows the analysis and interpretation of consumer opinions about a product for quality control and acceptability in the market.

The evaluation of sensory analysis uses people, the consumers, as a mediation instrument, since the human being is a sensitive characteristic that a machine does not have. This evaluation is innate in the human being because from the moment a product is tasted, a judgment is made about it. As for example if it is pleasant or not, in addition, characteristics such as texture, taste,

smell, etc. are recognized. However, you need people who have developed their senses to be able to obtain effective results.

Therefore, those people that we will call judges/panel have to be trained. There are different types of panel; we can classify them as follows (de Vicente, 2016):

- Specialized expert judge: a person who has great experience in a certain field. Great sensitivity to perceive the differences between samples and to distinguish, also evaluate the characteristics of the product.

- Trained or expert judge: a person who is quite skilled in the detection of some sensory property, has received theoretical and practical training, and knows exactly what he/she wants to measure in a test. He/she carries out sensory tests with a certain periodicity.

- Consumer judge: person who is a regular consumer of the product to be tested, but does not carry out periodic evaluations.

The quality factors that will be evaluated as sensory will be the smell and its durability, as well as the acceptability of the product in the market.

For this, the sensory analysis will be divided into two teams, panels, to perform different tests. The first team composed of 8 air freshener expert judges, chosen according to the ISO 8586:2012 Standard, will be in charge of the smell and its durability. The second team composed of consumer judges, 40 people, will perform the acceptance tests.

#### 4.1.1.Odour analysis

The analysis of the odour of the product is related to the fragrance. It is known that the aromatic components of air fresheners include a significant amount of one or more volatile fragrance ingredients in various proportions. Typically, the perfumes incorporated in the compositions used in air fresheners are mixtures of organic compounds blended so that the combined odors of the individual components produce a pleasant or desired fragrance. Although perfumes are generally mixtures of various materials, individual compounds may also be used as a perfume ingredient. Perfume compositions usually contain several "notes", each of which has different volatility and volatility indices and are therefore subject to the chromatography process which may result in differential distribution of the notes at different times.

This test will be relevant to choose the final fragrances of the air fresheners. Different mixtures of perfumed ingredients proposed by the company in charge of scenting the air fresheners will be evaluated.

To do so, the eight panelists will carry out an evaluation using the dynamic olfactory sensory method, thus obtaining objective results.

### 4.1.1.1.DYNAMIC OLFACTOMETRY

Dynamic Olfactometry involves the measurement of an odour concentration in samples selected from the source where the emissions are produced, in accordance with the specifications of the UNE EN 13725 standard.

Dynamic olfactometry is the only internationally recognised technique for odour measurement. The group of eight people, the olfactory panel, sniffs the odour sample. The number of dilutions required for the panel to detect the odour is the odour concentration and is expressed as: European Odour Units per cubic metre (OuE/m<sup>3</sup>). Therefore, the higher the European odour unit per cubic metre, the higher the odour intensity. So that mixture will be the one chosen (Bax et al., 2020).

### 4.1.2. Market acceptance

In this section, the panel will be the regular consumers of air fresheners. The elements to be evaluated are smell and colour. The odour for aerosol format will be evaluated three times at different times to corroborate the durability/intensity of the product. To aerosol air freshener, the sampling time will be immediately after applying the air freshener, after one and a half hours and finally after three hours. In the case of the electric air freshener and the gel air freshener, it will be evaluated whether the odour is adequate and how long it lasts. All these evaluations will be done by means of an affective test.

### 4.1.2.1. AFFECTIVE TEST

In this test the panelists subjectively evaluate the product. For this reason, it is important to correctly identify the target consumer, establishing the criteria for the selection of the panel: gender, age, consumption habits, economic level, geographic location, etc. The number of participating consumers has to be representative of the population. Also, it should be noted that in order to maintain the same conditions at all times the test will be conducted in a controlled and equal facility for all panellists.

For quantification, the 9-point hedonic scale will be used, considering 1 as the lowest score and 9 as the highest (Sensory Society Website, 2021). Table 2 shows the hedonic scale.

Hedonic Scale	
Dislike extremely	1
Dislike very much	2
Dislike moderately	3
Dislike slightly	4
Neither like nor dislike	5
Like slightly	6
Like moderately	7
Like very much	8
Like extremely	9

Table 2. Nine-Point Hedonic Scale.

The panelists will be given a sample of the three types of air fresheners in this project and a question sheet for each of them that includes the evaluation of the aspects to be rated; the question sheet can be consulted in APPENDIX 1.

To evaluate the different odours, it will be necessary to do it on different days in order not to cross the odours.

In addition to the sensory quality criterion, the physicochemical quality criterion will also be defined for the different air fresheners.

### 4.2. PHYSICOCHEMICAL QUALITY FACTOR

When dealing with different modes of diffusion of air fresheners, the physicochemical quality factors will differ from each other. Therefore, this section will be divided by each desired format.

#### 4.2.1. Aerosol: ability to phase shift

The quality factor to be assessed shall be the ability to change phase upon application of the product.

The aerosol type air freshener contains the mixture in liquid form in the container, but when it is applied it turns into micro droplets, so small that they look like gas. In this way, it is possible to scent the required space. A propellant is needed for this change of the mixture.

The propellant, or propellant, is responsible for supplying the pressure necessary to expel the active ingredient from the container when the dosing valve is operated. The pressure in the container is high, between 2 and 8 atmospheres, so that the propellant is in the form of a liquefied gas and is mixed with the liquid to be vaporised. Then, when the dosing valve is actuated, since there is more pressure inside the container than outside, the liquid rises through the plastic tube to the nozzle. When the mixture of liquid and propellant exits through the nozzle, the pressure changes and is lower, causing the propellant to become gaseous, atomising so that the liquid is dispersed into the atmosphere in the form of micro-droplets.

The evaluation of this phase change shall be done by the vapour pressure. Vapour pressure can be defined as the pressure exerted by the vapour of a liquid when it is in equilibrium with the liquid. When at higher pressure than vapour pressure, the propellant will condense and when at lower pressure than vapour pressure, the propellant will evaporate and thus the active ingredient is expelled from the product.

The determination of the vapour pressure of the propellant shall follow the method used in ASTM D1267. ASTM D1267 describes that the test device must basically consist of an upper chamber and a lower chamber (the volume ratio of the upper chamber must be 3 times greater than the lower chamber) and both interconnected by a valve. Also, the upper chamber is connected to a pressure gauge that will give the pressure reading. The pressure gauge must be calibrated and with the appropriate measuring scale.

#### 4.2.2. Plug-in liquid: ability to phase shift

The quality factor to be assessed shall be the ability to change phase upon application.

This quality factor can be assessed by the vapour pressure, which is related to the boiling point. It must be taken into account in product formulations in order to meet the desired characteristics: being liquid at consumer use temperatures but gradually releasing the aroma.

The vapour pressure depends on the solvent ingredient of the desired product. The scentgiving ingredients are dissolved in a solvent, so evaporating the solvent helps to expel the active ingredient into the air. In this way, the chosen space will be impregnated with the desired scent, thus, the air freshener would be fulfilling its function. In this case, the vapour pressure is around 0.55 hPa.

The vapour pressure can be measured using the isoteniscope instrument. This is a device used to measure the vapour pressure of substances in the liquid phase.

### 4.2.3.Gel

The following two quality factors will be evaluated for the gel-type air freshener.

### 4.2.3.1. EVAPORATION OF ACTIVE INGREDIENT

The evaporation of the perfume from the air freshener cannot be immediate and sudden, in order to satisfy the consumer it has to be the opposite. Therefore, this quality factor will be evaluated to ensure that the product lasts, fulfilling its function of scenting the environment.

First of all, this type of air freshener contains an ingredient called a gelling agent, which is the reason why it takes the final form of the product, a gel. Inside the gel is the characteristic scent of the air freshener, thanks to the fact that it is absorbed the scent is released gradually.

The evaluation of the evaporation will be done by a hedonic test. This test will last for a total of four weeks, during which an evaluation sheet will be filled in by panelists specialised in the air freshening sector and with great olfactory skills. A quantitative test will also be carried out to verify the qualitatively obtained data. This test consists of weighing the mass of the gel each week by gravimetric. In this way, the gel weight loss is obtained and by making the difference between the weeks it is possible to know what has evaporated. Also, by comparing the results from week to week, it is possible to check if the amount evaporated is constant.

The stability of the gels is related to the ability of the solid structure to retain the liquid. When the liquid is released from the solid structure and flows out, the phenomenon of syneresis is said to be occurring. To avoid this phenomenon, various factors such as the type of gelling agent, temperature and pH, among others, are taken into account.

In this case, for the chosen type of gelling agent, the critical temperature starts at 60°C and the pH ranges between 7 and 9. For the evaluation of the pH, a digital measurement will be made, consisting of the use of two electrodes. One electrode will be for reference and the other for measurement. As it is a digital measurement it will be more accurate and reliable.

# 5. SELECTION OF INGREDIENTS

Following the product development proposed by Wibowo and Kang M.Hg (2002), after having conceptualised and defined the quality factors for the range, it is time to select the ingredients that meet the desired characteristics.

In general, the products have three types of ingredients:

- Active ingredients: Ingredients that are intended to fulfil the stated purpose of the product, i.e. components of the formulation related to the main function of the product.

- Excipients: They are the vehicle for the active ingredients to be applied.

 Additives: These are additional ingredients that can be added to the formula to provide properties such as preservation, aroma, colour, thickness, among others. They are not considered as the main function of the product.

This section has been carried out by researching available databases, such as CPID (Consumer Product Information Database) or Prospector, as well as patents to determine the ingredients and their proportions.

As people's environmental awareness has grown, efforts will be made to select ingredients of natural origin or ingredients with less effect on the environment and human health.

### 5.1. AEROSOL

The aerosol is a dispersed system in which the dispersed phase is liquid and the continuous phase is gas. The objective of this aerosol application is to have the liquid product uniformly distributed, and for this purpose the aerosol must be made up of small droplets, with a low droplet concentration. Therefore, its preparation is "in situ" for immediate application due to its low stability.

There are water-based aerosol air freshener concentrates that can be formulated to offer an alternative to a totally solvent-based formulation. In this project it has been decided to design one of this type. For this reason, the formulation contains the diluent component which can be defined as liquids that dilute the concentrations of other liquids. This ingredient has a large percentage of approximately 90%.

In order to satisfy the consumer's need, the air freshener should provide a good scent for the respective room or mask unpleasant odours. For this purpose, fragrances are to be used, in the consulted formulations the percentage varies between 0.25% and 1%.

Another important component that is reflected in the formulations consulted is the propellant. As mentioned above, the propellant is the propellant for the fragrances to be expelled into the air. A classification of propellants can be as follows:

- Compressed gases are those that cannot easily be converted to a liquid state. Such as nitrogen, carbon dioxide and nitrous oxide.
- Liquefied gases are those that can be brought to their liquid state by subjecting them to moderate pressures, which facilitates and reduces the cost of handling, storage and transportation. For example, hydrocarbons, chemical synthesis gases (DME: dimethyl ether) and halogenated hydrocarbons.

Inside the container there is a very high pressure, so the propellant is liquid. Liquefied gases are the most commonly used due to their characteristics. It is odourless so as not to change the aroma of the product, commercially available and economically viable, wide vapour pressure range according to product and packaging requirements, and soluble in concentrate are some of these characteristics. Thus, the propellant has been chosen to be a hydrocarbon mixture of liquefied gases and its percentage is between 40%-60% of the total weight of the product.

The solvent compound can also be said to be a substance that allows the dispersion of another substance within it. It is the one that allows the fragrances to integrate well, the percentage found is between 2% and 5%.

Finally, an additive called emulsifier can be found which is useful to keep water and oils mixed. Its percentage is about 2% of the total weight of the product.

It should be noted that when the propellant and the liquid mixture are to be packaged, their percentages are 60% and 40% respectively.

### 5.2. PLUG-IN LIQUID

The final product is a homogeneous liquid mixture of different compounds. The solution is basically composed of fragrances and solvent.

Fragrances again play a major role in the composition, this is due to the characteristic aroma they give off. The percentage of this component of the total weight of the product is around 6%.

On the other hand, the solvent, in order to help the aromatic substances to dissolve, has a proportion between 30%-60% of the total weight. The diluent is also present with a percentage of about 20%.

Finally, there may be additive components such as emulsifiers with a percentage of approximately 6%. Also, there may be colouring compounds, these are used as a lure for consumers. However, they may not be necessary as the other compounds may have their own colour. The percentage ranges from 0.5% to 0.7% if a particular colour is desired.

## 5.3. GEL

Gel is a solid continuous phase dispersed system. According to the IUPAC (International Union of Pure and Applied Chemistry) definition, a gel is a non-fluid colloidal or polymeric network filled by a fluid phase. According to this definition, the network may be formed by colloidal particles or by the cross-linking of polymeric chains.

It is composed of fragrance oil, a percentage of about 6%. Also, there is a solvent, with percentage ranging between 20% and 90%, and a gelling agent. This compound is the most characteristic because it is the one that gives the gel form, it is found in a percentage between 1% and 10%.

Also, there may be additives such as fixatives, preservatives and colouring agents with a percentage of around 2%. They may or may not be incorporated into the formulation.

## 5.4. FORMULATION

Once the different ingredients for each air freshener had been selected, a formulation proposal was drawn up.

The reference formulas have been used as a starting point to know the different ingredients of the product, as well as the percentages of each one.

#### 5.4.1. Aerosol formulation

Therefore, in a summarised form for the aerosol type air freshener, on the one hand we have the propellant with 60% (CPID and Prospector Website, 2022) and we have chosen from the Repsol propellant range number 38; and on the other hand the liquid mixture. This liquid mixture, which will be 40% in the can, has the following ingredients:

- Triethylene glycol and Isopropyl alcohol as solvent with 2.5% and 4% respectively
- As you want an air freshener with a spring and floral feel the fragrances chosen are: Terpineol, benzyl acetate, Citrus Aurantium Dulcis Oil (Orange oil), eucalyptus oil and menthol.
- Span 80, sorbitan oleate, as an emulsifier with 2 % as an emulsifier
- Water as diluent with 87%

Table 3 shows the ingredients, their function in the formulation and the percentage of mass selected.

Ingredient	Weight %	Function
Water	88	Diluent
Isopropyl alcohol	4	Solvent
Triethylene glycol	2.5	Solvent
Span 80	2	Emulsifier
Terpineol	0.5	Fragrance
Benzyl acetate	1	Fragrance
Citrus Aurantium Dulcis Oil	1	Fragrance
Eucalyptus oil	0.5	Fragrance
Menthol	0.5	Fragrance

Table 3. Specifications of the ingredients of the aerosol air freshener.

Repsol has a range of products called REPSOL PROPEL which are liquefied and purified gases of mineral origin (petroleum) that are composed of selected mixtures of hydrocarbons (C3-C4), suitably treated and deodorised to guarantee compliance with the regulations and requirements of our customers in any type of application and final finish. Typical 1,3-butadiene content is less than 0.1% (Repsol Website, 2022).

On the other hand, Span 80 MBAL is a biodegradable surfactant based on a natural fatty acid (oleic acid) and the sugar alcohol sorbitol, and has been manufactured using the RSPO mass

balance system. This sorbitan ester is highly effective in the formation of O/W emulsions. Span<sup>™</sup> 80 (sorbitan oleate) provides an effective water-in-oil emulsion, another serious alternative emulsifier Span<sup>™</sup> 20, sorbitan monolaurate, (Ulprospector Website, 2022).

## 5.4.2. Plug-in liquid formulation

With regard to the liquid plug-in air freshener, the following has been proposed:

- Dipropylene glycol methyl ether solvent with percentage 70%.
- Fragrances with 6%.
- Water as diluent with 18%.
- Etocas 29, castor oil, as emulsifier with 6%.

Etocas 29 is a non-ionic surfactant of vegetable origin. It is suitable for a wide variety of domestic, industrial and institutional (I&I) applications, providing emulsifying and solubilising properties. They provide effective solubilisation of fragrance and help control fragrance release in aqueous systems (Ulprospector Website, 2022).

Table 4 shows the ingredients, their function in the formulation and the percentage of mass selected.

Ingredient	Weight %	Function
Dipropylene glycol methyl ether	70	Solvent
Water	18	Diluent
Etocas 29	6	Emulsifier
Benzyl acetate	1.5	Fragrance
Terpineol	1	Fragrance
Phenethyl Isobutyrate	1	Fragrance
Linalool	0.5	Fragrance
Methylionone	1	Fragrance
Dispentene	1	Fragrance

Table 4. Specifications of the ingredients of the plug-in liquid air freshener.

#### 5.4.3. Gel formulation

Finally, for the gel-type air freshener it has been proposed:

- Water as solvent with 87%.
- Fragrance 5% Fragrance
- Polypropylene glycol as a fixative 4.5%
- Genugel RVL (Carrageenan) as gelling agent 3%.
- Dibutyl lauroyl glutamide (GP-1 from Ajinomoto Co, Tokyo, Japan )with 0,5%

Table 5 shows the ingredients, their function in the formulation and the percentage of mass selected.

Ingredient	Weight %	Function
Polypropylene glycol	4.5	Fixative
Water	87	Diluent
Genugel RVL (Carrageenan)	3	Gelling agent
Dibutyl lauroyl glutamide	0.5	Co-gelling agent
Linalool	0.5	Fragrance
Methylionone	1	Fragrance
Dispentene	1	Fragrance
Benzyl acetate	1.5	Fragrance
Eucalyptus oil	1	Fragrance

Table 5. Specifications of the ingredients of the gel air freshener.

There are three types of carrageenan: kappa, iota and lambda. Kappa and iota type carrageenan form a gel structure, with the exception of lambda. During the cooling process, strong hydrogen bonding occurs, which allows the appearance of double helix chains. However, they are not strong enough for a gel texture but in the presence of cations they are linearly ordered to give way to a three-dimensional helical formation. These ions can be calcium or potassium (Mancilla, 2012).

The Genugel RVL ingredient is a mixture of carrageenan, calcium acetate and potassium chloride used in the application of air freshening gels. It is dispersible in cold water and soluble in hot water, a hot solution has a high viscosity, forms a firm gel when the solution is cooled, and the gels exhibit little or no syneresis (Ulprospector Website, 2022).

Carrageenan gels are commonly used for the diffusion of volatile substances, but their performance with respect to the diffusion of the volatile active substance is not always ideal as a result of their tight matrix. According to WO 2013/030153, mixing the carrageenan component with protein components would help the release of the volatile active substance to be more regular and prolonged. In this case, the component dibutyl lauroyl glutamide has been used.

# 6. PRELIMINARY DESIGN OF THE MANUFACTURING PROCESS

Finally, once the products studied, the quality factors and the selection of ingredients and formulation have been defined, it is time to design the production process for the three formats of air fresheners. To do this, first the annual production will be determined, followed by the relevant manufacturing process for each air freshener and the selection of the necessary equipment. Finally, the annual air freshener production plan will be drawn up.

It should be noted that the air fresheners will be produced in a plant where the company manufactures all types of air fresheners with a wide range of fragrances.

#### 6.1. ANNUAL PRODUCTION AND BATCH SIZES

In order to decide on the manufacturing system, it is necessary to know the sales forecast of the product and, therefore, the annual production to be carried out.

According to Statista, a portal specialising in market and consumer data, in 2020 around 26.196 million people in Spain will use air fresheners, of any kind. In the same year, the INE (National Statistics Institute) recorded that the population in Spain is made up of 47.355.685 people, including women and men, so around 55% use air fresheners on a regular basis. If the product is aimed at people between 25 and 65 years of age, taking into account the INE data and applying the above percentage, around 16.126.423 people habitually use air fresheners. Furthermore, applying 5% to simulate the choice between the product studied and other products on the market, the demand for air fresheners of any type is 806.321 units over the population of Spain.

However, this work addresses the production of aerosol air fresheners, plug-in liquid and gel which will not have the same demand. For aerosol air fresheners, according to Statista approximately 4,5 million people use them regularly, i.e. 17% of the total use of air fresheners. This translates into a demand of 137.075 units over the population of Spain. In the case of liquid

air freshener, approximately 3 million people use it, i.e. 11% of the population. Therefore, its demand would be 88.695 units. Finally, the air freshener gel is used by approximately 835.000 people, i.e. a very small percentage compared to the other two. Its demand would be very small, so a demand of 2000 units has been proposed.

This estimated demand has been rounded to an annual production of 137.000 units of aerosol air freshener, 88.000 units of liquid plug-in air freshener and 2.000 units of gel air freshener, considering that the estimated population purchases each of the products once a year.

The capacity of the aerosol and liquid air freshener containers is 250 mL and 20 mL, so the annual volume production can be estimated at 34.250 L/year for the aerosol air freshener and 1.760 L/year for the liquid air freshener. With regard to the air freshener gel, the container contains 150 grams of product, so the annual mass production is 300 kg/year.

The manufacturing system for the three types of air fresheners will be batch production. This system is generally used in multi-product situations, as well as in small volume production. It is also more economically favourable, allows for some versatility in changing product operating conditions and facilitates the shutdown and cleaning of equipment (Cunill et al., 2010).

It has been decided to use 500L batches to produce the aerosol air freshener and the liquid plug-in air freshener. On the other hand, it has been decided to make batches of 100kg for gel air freshener. In order to estimate the manufacturing time of the products, the estimated campaign time and the number of campaigns to be carried out will be proposed.

It should be noted that production will be adapted to sales during the first six months that each product is on the market.

### 6.2. AEROSOL AIR FRESHENER

As there are three different products, the manufacturing process will not be completely the same between them. For this reason, they will be explained separately for clarity. Also, the different compounds will be supplied by an external company.

#### 6.2.1. Manufacturing process

In this process, two parts of the aerosol air freshener are distinguished, the liquid concentrate and the propellant. In this case, as mentioned above, a mixture of isobutane, n-butane and propane gases is used. To begin with, the liquid concentrate is prepared. First, the triethylene glycol is added to the water and stirred to unify the two ingredients, triethylene glycol is soluble in water, which makes mixing easier. At the same time, sorbitan oleate, isopropyl alcohol and fragrances are mixed. The time for these two pre-mixes at room temperature is 30 minutes.

The mixture of sorbitan oleate, isopropyl alcohol and fragrances is then added to the main water phase and stirred with a propeller agitator. This mixture shall be homogenised for 30 minutes at room temperature.

In this way, the liquid concentrate is obtained and stored in large steel drums and then pumped automatically to the filling line.

Once the liquid concentrate is ready, it is passed to the filling and packaging line.

The empty cans, opened at the top, are fed onto the conveyor belt. The first stop is to fill the can with a controlled amount of the liquid concentrate. Next, the valve is placed on the can so that it can be filled with propellant and then sprayed. The next step is to add the propellant to the liquid solution already inside. In this way, the product can be used as an aerosol. The gas filling section is separated from the rest of the production plant due to the sensitivity of the process.

Once the cans have been filled with both liquid and liquefied gas, they are checked to ensure that they have been filled to the correct levels. As a precaution, the can does not have to be completely full. Any can that is above or below the accepted margin of error is automatically removed from the production line.

After the fill level check, the cans are subjected to a final leak test. This is a water bath test, in which the cans are completely immersed in hot water. If bubbles appear above any of the cans, it is known that the can is not completely leak tight and must be removed for further testing.

Finally, the cans are ready for the finishing touches, which are the fitting of a spray nozzle to each can and the attachment of a safety cap to ensure maximum freshness and quality of the product.

Once these finishing touches have been applied, the can is fitted with the lid and placed in boxes for distribution to customers.

The packaging stage of this process is very relevant because it is where the propellant is incorporated. In addition, as mentioned above, this stage goes through several sections in order to obtain the final product. Therefore, the packaging time, including filling and packaging for

commercialisation, is approximately 6 hours. Taking into account that the volume of a batch is 500mL, it will be possible to pack 2000 cans of 250mL capacity.

In Figure 4 shows the diagram of the aerosol manufacturing process.

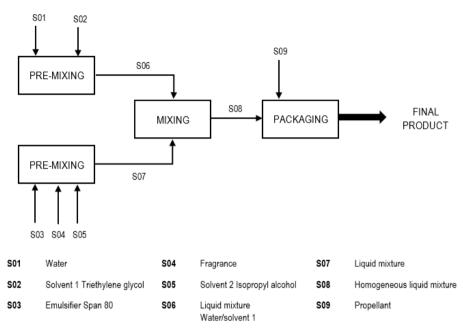


Figure 4. Process diagram for aerosol air freshener.

### 6.2.2. Selection of equipment

#### Mixed vessel

When the batch size and the operations to be performed on the equipment are defined, the characteristics of the mixing vessel are described.

The tank shall have a cylindrical shape in a vertical position. In addition to the shape, according to Martínez (2020), the type of bottom of the tank is important; it can be hemispherical, flat or conical. It will also have a top lid that can be opened and closed to facilitate the cleaning of the equipment.

As mentioned above, each batch will produce 500L of product. Batch process plants are usually oversized (Notes project subject, 2021), therefore, the capacity of the tank will be 600L, oversized by a factor of 1,2.

According to a heuristic article by Anaya et al. (2006) it is preferable to maintain a liquid level that is approximately equal to the tank diameter. Therefore, the ratio of liquid height to tank diameter will be said to be 1.

The diameter (D) and height (H) of the tank have been calculated assuming a cylindrical shape with the above assumptions.

D=0,9 m

H=1,1 m

## AGITATION MODULE

The agitation system is important for homogenising the mixture and depends on the size of the tank, the type of flow and the type of impeller, among others.

For the design of the agitation of the tank that will manufacture aerosol air fresheners, the impeller has been chosen in the form of marine propellers, which stand out for their fluidity in axial direction and are more recommendable for low viscous fluids, through the information in the book Perry Chemical Engineers.

### TANK ON THE MARKET

### LIQUIDMIX MIXING EQUIPMENT, INOXPA

As can be seen in Figure 5 this equipment has been selected for the process of aerosol air freshener. It is designed for low viscosity mixtures and has a vertical agitator with a three-bladed propeller. Likewise, the introduction of products is carried out through at the top bottom, through the manhole or through the tank bottom valve if the equipment works under vacuum. In addition, rapid agitation is used for homogenising liquids or dissolving easily soluble solids.



Figure 5. Liquidmix from INOXPA.

## Packaging

For the packaging of the product, automatic equipment is required to fill each container with the precise amount of product. In this case a quantity of 250mL is set. In addition, this packaging system must also close the container so that it is ready to be marketed. It should be noted that this stage of the process is very relevant in the aerosol manufacturing process as it is where the propellant is incorporated.

After a search on the market, the equipment of Kinetic (Hubei) Energy Equipment Engineering Co. has been selected for the aerosol air freshener. The selected equipment is Aerosol Can Filling Machine model number CJXH-2800A, which fills and closes the cans in a simple way.

# 6.3. PLUG-IN LIQUID AIR FRESHENER

### 6.3.1. Manufacturing process

This process is similar to the process followed for the aerosol liquid concentrate in terms of the equipment used.

First, dipropylene glycol methyl ether, fragrance and Etocas 29 are mixed to form a solution. The time for this pre-mixe at room temperature is 30 minutes. Subsequently, the solution obtained

is added to water and stirred to mix all the ingredients. The time for this mix at room temperature is 15 minutes.

Once the desired solution is in place, it is stored in large steel drums and then automatically pumped to the filling line.

The empty containers are fed onto the conveyor belt. The first section is the filling of the solution into the container, checking the quantity. Then, the wick, immersed in the liquid, is placed in the container, with a stopper attached to the mouth of the container.

Finally, it is capped and goes to the packaging line for distribution.

In Figure 6 shows the diagram of the aerosol manufacturing process.

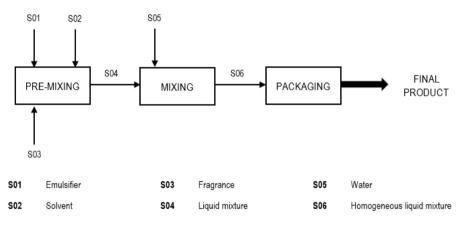


Figure 6. Process diagram for plug-in liquid air freshener.

### 6.3.2. Selection of equipment

#### Mixed vessel

In this manufacturing process, the same mixing equipment has been selected as mentioned for the aerosol air freshener process. This is because the mixture is at room temperature and is not very viscous. Furthermore, the capacity of the batch to be produced is the same, 500L.

### Packaging

For the packaging of the product, automatic equipment is required to fill each container with the precise amount of product. In this case, a quantity of 20mL is set. In addition, this packaging system must also close the container so that it is ready to be marketed.

After a search on the market, the Equitek equipment has been selected. The three independent machines belong to the same range, so they are compatible with each other and allow the process to be efficient.

For the liquid air freshener plug-in packaging machine, the equipment for level packaging of liquid products has been selected (Equitek Website, 2021).

For the capping machine, a model has been chosen that screws the cap onto the liquid air freshener container, it is the ERS SERIES. This model requires the intervention of an operator who places the cap on the bottle and the equipment caps them.

Finally, for product labelling, the SERIE ESZ model has been selected. This equipment allows self-adhesive labels to be placed on the container, which can be cylindrical, oval or flat, thus allowing the equipment to be used for other processes.

### 6.4. GEL AIR FRESHENER

#### 6.4.1. Manufacturing process

The gel air freshener production process can be simplified to melting and mixing the ingredients and moulding the product. However, there are many things to consider in order to design the most efficient process with a better product as a result.

The process starts by introducing the carrageenan gelling agent, Genugel RVL, into the tank with distilled water. It is then heated to a temperature of 60°C to 70°C, by means of a jacket covering the tank with circulating water vapour, and under agitation until the Genugel RVL is dissolved. In parallel, the dibutyl lauroyl glutamide and the fixative are mixed at 60°C to 70°C under stirring to form a uniform mixture. Then, the two mixtures are brought together and stirred further to obtain a homogeneous fluid gel.

Once the mixture is homogeneous, the temperature is lowered by circulating water from the tower through the jacket before adding the fragrance because it is a heat-sensitive ingredient. Fragrance is a volatile substance and exposure to high temperatures will cause it to evaporate. The fragrance will be added when the temperature is between 55°C and 60°C. Stirring is continued until the solution is homogeneous.

Subsequently, when the fluid gel is obtained, it is poured into a suitable container and allowed to cool. The cooling can be done at room temperature or a cold air tunnel can be used. It is poured

into the container before it cools down to obtain a solid gel with the desired shape, shape and decorative design.

Finally, once it is cold and in its mould, it continues its packaging and labelling process.

In Figure 7 shows the diagram of the aerosol manufacturing process.

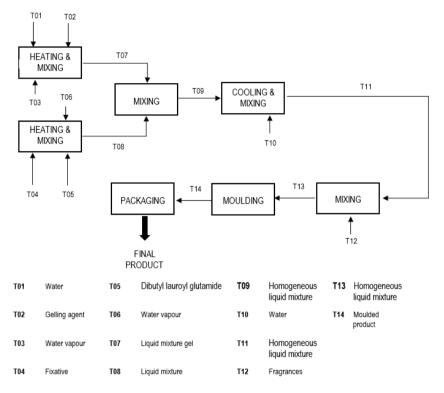


Figure 7. Process diagram for gel air freshener.

#### 6.4.2. Selection of equipment

The production process of the air freshener gel is carried out using two pieces of equipment, the first one a stirred tank with heating and cooling system, which melts and mixes the ingredients of the process, and the second one a packaging, capping and labelling machine. The selection of this equipment will be made on the basis of efficiency versus cost.

The two pieces of equipment and their accessories are described below, and the equipment is sought after on the market.

#### Mixed vessel

Once the batch size and the operations to be carried out in the equipment have been defined, the characteristics of the mixing vessel (the dimensions and shape of the tank), its capacity, the heat supply and extraction, the agitation system and finally the tank discharge are described.

The tank shall be cylindrical in shape with a conical bottom placed in a vertical position. In addition, it shall have a top lid that can be opened and closed to facilitate cleaning of the equipment Furthermore, this tank shall require a heating system reaching a temperature of 70 degrees.

As mentioned above, each batch will produce 100kg of product. Taking into account that water has the highest percentage of the ingredients, a density of 1kg/L is considered and therefore the batches will be 100L each. Batch process plants are usually oversized (Notes project subject, 2021), therefore, the capacity of the tank will be 130L, oversized by a factor of 1,3.

According to a heuristic article by Anaya et al. (2006) it is preferable to maintain a liquid level that is approximately equal to the tank diameter. Therefore, the ratio of liquid height to tank diameter will be said to be 1.

The diameter (D) and height (H) of the tank have been calculated assuming a cylindrical shape with the above assumptions.

D=0,5 m

H=0,7 m

On the other hand, the agitated tank can be made of different materials; in general they are made of steel, stainless steel or glass (Cunill et al., 2010). Currently, the most commonly used is stainless steel AISI 316.

#### AGITATION MODULE

In order to homogenise the contents of the tank, the agitation module has been designed. When choosing the agitation system, the power that the motor can reach and the type of agitator must be taken into account. There are also three types of flow, depending on the flow characteristics, the size of the tank and the type of impeller. This flow can be radial, axial or tangential depending on the flow movement. The three main types of impellers are blades, turbines or propellers.

In this case the agitation of the tank that will manufacture gel air fresheners, the anchor impeller, recommended for viscous mixtures and has a tendency to create a tangential flow, has been chosen from the Perry Chemical Engineers book.

#### HEAT SUPPLY AND EXTRACTION

During the course of the manufacturing process of the air freshener gel, the temperature of the tank must be modified by providing or extracting heat through an intermediate fluid, this exchange can be through a coil or a jacket. The jacket will be the option of choice as it does not interfere with the product inside the tank and facilitates cleaning (Martínez, 2020). However, its shape will be half serpentine to improve transmission (Cunill et al., 2010). Its surface will be the entire lateral area with a height equal to that of the liquid.

As mentioned above, during the process heat has to be supplied up to a temperature of between 60°C and 70°C and then extracted up to a temperature of between 55°C and 60°C. The fluids chosen for heat exchange are water at 25°C from a cooling tower and water vapour at a pressure greater than 0 barg and temperature greater than 100°C to heat the fluid.

Following this, an approximation of the time needed to heat and cool the tank has been made in order to roughly estimate the duration of the process. The minimum fluid mass flow required for a proper transfer has been considered.

The starting point is the macroscopic energy balances in the tank to be heated or cooled with the fluids that will circulate in the jacket.

$$\frac{d(H-H^*)}{dt} = \sum_{m} (\widehat{H} - \widehat{H^*})_m w_m - \sum_{i} \Delta \widehat{H}^* R_i + \dot{Q} + \dot{W} + \frac{d(PV)}{dt}$$
(1)

Considering a closed, unreacted system, and neglecting the last term, the balance would be as follows:

$$\frac{dH}{dt} = \dot{Q} \tag{2}$$

$$mC_p dT = \dot{Q} dt \tag{3}$$

Where m is the mass of the fluid to be heated or cooled,  $C_p$  is the heat capacity of the fluid and Q is the heat flux required for the temperature change.

With respect to the external fluid, in the case of water, the enthalpy balance is as follows:

$$w_c C_{pc} \Delta T_c = \dot{Q} \tag{4}$$

When the external fluid is vapour it remains:

$$w_c \lambda_c = \dot{Q} \tag{5}$$

Where  $w_c$  the flow rate of water or water vapour is,  $C_{pc}$  is the heat capacity of the water, and  $\Delta T$  is the temperature difference between the inlet and outlet water temperatures, and  $\lambda_c$  is the latent heat of the water vapour.

Finally, the design equation of the interchanger system has been used:

$$\dot{Q} = UA[(T_C - T)]_{ml} \text{ or } UA(T_C - T)$$
(6)

Where U is the overall transfer coefficient, A represents the exchange area, and in the case of water the logarithmic median between the temperatures of the fluid and those of the tank, and in the case of steam, its temperature and those of the tank.

As interpreted from the above equations, the driving force is the temperature difference between the tank and the external flow. It is assumed that when saturated water vapour flows through the jacket, its temperature is constant.

To find the heating time, equations 3 and 6 have been equated, resulting in the following equation:

$$mC_p dT = UA(T_c - T)dt \tag{7}$$

Where the water vapour temperature is constant, the tank temperature varies between 25°C and 70°C and a global heat transfer coefficient has been assumed, the equation has been obtained by integrating.

$$mC_p \int_{25}^{70} \frac{dT}{T_c - T} = UA \int_0^t dt$$
(8)

A similar process has been followed to find the cooling time, with the difference that the fluid in the jacket does not circulate at a constant temperature. The inlet temperature of the water in the tower was taken to be 25°C and the outlet temperature was assumed to be 30°C. To simplify the calculations, the outlet temperature has been considered constant, although it is known that as the driving force decreases.

$$mC_p \int_{70}^{55} \frac{ln \frac{T-25}{T-30}}{(T-25) - (T-30)} = UA \int_0^t dt$$
(9)

The resulting times are shown in Table 6, and the intermediate calculations can be found in APPENDIX 2.

System	Initial temperature [°C]	Final temperature [°C]	Total time [min]
Heating	25	70	10.1
Cooling	70	55	6.7

Table 6. Time needed to heat up and cool down the tank.

#### DOWNLOAD

The tank will be discharged from the bottom and the driving force will be gravity. An automatic valve will empty the tank for the storage drums connected to the filling line.

#### TANK ON THE MARKET

After defining the specifications that the tank must have for the gel air freshener process, the market was searched for the options that best matched the characteristics of the designed tank.

#### VISCOMIX MIXING EQUIPMENT, INOXPA

INOXPA's ViscoMix was the tank selected to meet all the design specifications of the air freshener gel process equipment, which is designed to produce medium-high viscosity mixtures. This equipment features anchor type agitator and high shear mixers, whose speed can be controlled from the electrical control panel along with the mixing temperature. Also, the unit is equipped with a jacket and two feed nozzles at the top to feed the tank. The selected equipment can be seen in figure 8.



Figure 5. Viscomix from INOXPA.

#### Packaging

For the packaging of the product, automatic equipment is required to fill each container with the precise amount of product. In this case, a quantity of 150 g is set. In addition, this packaging system must also close the container so that it is ready to be marketed.

After a search on the market, the Equitek equipment has been selected. The three independent machines belong to the same range, so they are compatible with each other and allow the process to be efficient.

For the air freshener gel packaging machine, the DVS volumetric packaging system has been chosen, which is designed for viscous formulations in the personal care and home care sectors (Equitek Website, 2021).

The capping machine for the gel air freshener has been selected with a belt pressure capping system. It consists of a variable speed belt that applies pressure to the caps placed on the bottles. It is coupled to a conveyor, so that by means of this pressure the caps are closed on the container (Equitek Website, 2021).

Finally, for product labelling, the SERIE ESZ model has been selected. This equipment allows self-adhesive labels to be placed on the container, which can be cylindrical, oval or flat, thus allowing the equipment to be used for other processes.

## 6.5. PRODUCTION PLANNING

Once the process operations and the equipment used have been defined, the annual manufacturing campaigns for each air freshener are proposed. The time required for mixing, filling and packaging for each type of air freshener must be taken into account.

Table 7 shows the process time for the aerosol air freshener mixing and packaging equipment. The total process time is 7 hours, with the packaging stage being the longest stage of the process. Knowing the total process time, it is proposed to produce two batches per day, from Monday to Saturday.

Action	Time [min]
Initial feeding	5
Heating and mixing	30
Mixing	30
Packaging	360
Total time	425 min ≈ 7 h

Table 7. Timing of the aerosol process.

The process time for the liquid plug-in air freshener is approximately 5 hours. Table 8 shows the times for each part of the process, again with the packaging stage being the longest. Taking into account the total time, it is proposed to produce two batches per day, from Monday to Saturday.

Table 8. Timing of the plug-in process.

Action	Time [min]
Initial feeding	5
Pre-mixing	30
Mixing	15
Packaging	250
Total time	300 min ≈ 5 h

As for the air freshener gel, table 9 shows the times for the different parts of the process. A factor of 1.3 has been applied to the heating time to ensure the desired temperature and a

constant mixing time has also been added to homogenise. The total process time is 10 hours, the packaging stage takes the longest because it has to be cooled. For this reason, it is proposed to produce one batch per day, from Monday to Saturday.

10
13
30
7
5
15
500
500 min ≈ 10 h

Table 9. Timing of the gel process.

Knowing the total process time of each air freshener, the annual production will be scaled to schedule the 96 batches throughout the year. To do this, the production will be carried out in three campaigns, the first one will take place over 4 weeks and will produce the aerosol air freshener. The second will last 2.5 weeks and will produce the liquid plug-in air freshener. Finally, the third campaign will take place over 3 weeks and will produce the air freshener gel. The choice of the type of air freshener for each campaign was based on consumer preferences.

On the other hand, the plant will have a layout as different types of air fresheners are manufactured. The differentiation between them will be the way the air freshener is diffused.

# 7. CONCLUSIONS

Recovering the main objective of this project identified in the first pages of the paper, the development of a range of formulated air fresheners and the preliminary design of the manufacturing process has been achieved. Although the nature of this work is purely bibliographical, various conclusions, facts and decisions made related to product development can be summarised as follows.

In the context of wanting to mask or scent a space, the need for the design of a range of home air fresheners has been identified. This range is composed of three air fresheners with different ways of diffusion of the aromatic substance. An aerosol air freshener where the active substance is sprayed into the environment with the help of a propellant. A liquid plug-in air freshener, where the active substance is dispersed by means of a wick and an increase in temperature. A gel air freshener that, thanks to a gelling agent, achieves an absorbent body that releases the active substance found inside by evaporation.

With the target and the product described, the quality factors of the air freshener range have been established. A differentiation has been made between sensory and physicochemical factors, and the factors have been selected with the objective that the product meets the olfactory expectations and ensures that it meets the consumer's need. The sensory factors that have been selected to evaluate the product are an odour analysis and market acceptance. Two panels have been designed to evaluate the sensory factors, an expert panel on the one hand and a consumer panel on the other hand. For the evaluation of the physicochemical factors, it is also necessary to differentiate between types of air fresheners. For the aerosol air freshener the factor is the phase change capacity which will be evaluated by the vapour pressure of the propellant. For the liquid air freshener, the phase change capacity is also evaluated by the vapour pressure of the solvent.

For the air freshener gel, the evaporation factors of the active ingredient and the stability are evaluated.

The ingredients have been selected to ensure compliance with the chosen quality factors. As there are three different products, the ingredients are not the same in all three products. The one ingredient that is found in all of them is the fragrance/perfume.

A first formulation has been proposed, designed on the basis of a database search of products on the market. It is considered that the proposed formulation should be experimentally tested before arriving at the final formulation for production.

The manufacturing process will be carried out in batches. The annual production will be organised with a total of 96 batches of air fresheners. For this purpose, three campaigns will be carried out, the first campaign will last 4 weeks and will produce aerosol air fresheners. A total of 48 batches of aerosol air fresheners will be produced. In order to carry out the manufacturing process, a 600L mixing vessel with a marine propeller impeller has been designed. The equipment selected is of the INOXPA brand, specifically Liquidmix. Likewise, the filling and packaging equipment will be connected to the mixing vessel to obtain the final product. The process takes a total of 7 hours.

The second campaign will last 2.5 weeks and will produce 30 batches of liquid plug-in air freshener. The manufacturing process will also be done in a mixing vessel which will then be connected to the packaging equipment. The process takes a total of 5 hours.

Finally, the third campaign will last 3 weeks and will produce 18 batches of gel-type air freshener. For the manufacturing process of the air freshener gel, a mixing vessel with a capacity of 130L with a jacket through which tower water and water vapour will circulate as cooling and heating flow has been chosen; and an anchor impeller has been selected. The equipment selected is of the INOXPA brand, specifically Viscomix. Afterwards, a moulding and wrapping equipment will be connected to the mixing vessel to obtain the final product. The process takes a total of 10 hours.

# **REFERENCES AND NOTES**

1. Academia del perfume. https://www.academiadelperfume.com/ (accessed Sep 24, 2021).

2. Allison, G. and O'leary, N. (2013) WO 2013/030153. Carrageenan gel air freshener.

3. Allied Market Research. Air Freshener Market. https://www.alliedmarketresearch.com/airfreshener-market (accessed Nov 29, 2021)

4. Anaya, A; Alarid, J; Gallegos, G; Leon, M. A; Sierra, J.P. (2016). Heuristic rules for process equipment, Chemical Engineering, Volume 113, number 10, Pg. 44-47.

5. Anderson, J. et al. (2007) WO 2007/052016A2 Aerosol, composition and method.

6. Bax, C; Sironi, S; Capelli, L. (2020). How can odors be measured? An overview of methods and their aplications, Atmosphere, volume 11, number 92, Pg. 3-4.

7. Caserta, A., Garcia Fabrega, R. and Moreno Perez, D. (2004) WO 2004/110559 A1. Adjustable non-electric liquid air freshener device.

8. Consumer Product Information Database. Wha's in products? https://www.whatsinproducts.com/pages/index/1 (accessed Dec 01, 2021)

9. Cunill F., Iborra M., Tejero J. Reactores Químicos Apuntes, 2010. Universidad de Barcelona.

10. De Vicente, P. (2016). Estudio sensorial para conocer al consumidor, Aenor, Pg. 14-19.

11. Deplan. Olfatometría dinámica según norma UNE-EN 13725. https://www.tratamientodeolores.com/es/paginas/olfatometria-medicion-de-olor (accessed Oct 23, 2021).

12. Envasado Xiomara SL. Una empresa líder en su sector de fabricación de aerosoles. https://envasadoxiomara.com/empresa/ (accessed Jan 05, 2022).

13. Equitek. Empresa destinada al diseño y fabricación de maquinaria para empaque. https://equitek.com.mx/ (accessed Jan 6, 2022).

14. Herrera, Y; Mendoza, R; García, O; Cruz, S; Muñoz, O. (2010). El facinante mundo de los olores, La ciencia y el hombre, Volume 13, number 1.

15. Industry ARC. Car Air Freshener Market - Forecast(2022 - 2027). https://www.industryarc.com/Research/Car-Air-Freshener-Market-Research-504024 (accessed Nov 29, 2021).

16. INOXPA. Empresa a la fabricación y comercialización de componentes de acero inoxidable y equipos para el tratamiento de fluidos, gestión de procesos y servicios en las industrias alimentaria, cosmética y farmacéutica. https://www.inoxpa.es/home (accessed Jan 05, 2022).

17. Martin, J. and Mark E., W. (1999) US005976503A. Disposable plug-in air freshener with heat activated cartridge.

18. Martínez Aguilella, P. (2020) Diseño de un tanque mezclador con sistema de calentamiento para la producción de protectores solares. Universitat Jaume I.

19. Noticias Jurídcas. Reglamento (UE) n.º 474/2014 de la Comisión, de 8 de mayo de 2014, que modifica el anexo XVII del Reglamento (CE) n.º 1907/2006 del Parlamento Europeo y del Consejo, relativo al registro, la evaluación, la autorización y la restricción de las sustancias y preparados químicos (REACH), por lo que respecta al 1,4-diclorobenceno https://noticias.juridicas.com/base\_datos/Admin/529037-reglamento-ue-n-o-474-2014-de-la-comision-de-8-de-mayo-de-2014-que-modifica.html (accessed Nov 23, 2021).

20. Regulation (EC) Nº 1223/2009 (2009).

21. Repsol (2013) "Propelentes Repsol."

22. Perry, R. (1997). Perry's Chemical Engineers' Handbook, McGraw Hill, Tomo I, Seccion 19, Pg 3 - 28.

23. Sintez. Engineering and production group. Aerosol production line. https://ipgsintez.com/packaged\_solutions/aerosol\_production\_line/ (accessed Jan 05,2022).

24. Society of sensory professionals. The 9-point Hedonic Scale. https://www.sensorysociety.org/knowledge/sspwiki/Pages/The%209point%20Hedonic%20Scale.aspx (accessed Nov 12, 2021).

25. Smith Scott, E. (2012) WO 2012/158731 A2 Method of manufacturing an aerosol dispenser.

26. Statista. Business Data Platform. https://www.statista.com/statistics/440972/air-fresheners-scented-candles-and-fabric-fresheners-usage-by-type-in-spain/ (accessed Dec 21, 2021).

27. Steinemann, A. (2017) "Ten questions concerning air fresheners and indoor built environments," *Building and Environment*, 111, pp. 279–284. doi:10.1016/j.buildenv.2016.11.009.

28. Traas, P.C. and Roehl, E.-L. (1988) United States Patent (19) Traas et al. (54) PROCESS FOR THE PREPARATION OF AIR-FRESHENER GELS.

29. UL Prospector. Global independent safety science company. https://www.ulprospector.com/en/eu (accessed Dec 01, 2021)

30. Wibowo, C. (2002). Product-Centered processing: Manufacture of chemical-base consumer products, Process systems engineering, AIChE Journal, volume 47, number 12, Pg. 2746-2765.

# ACRONYMS

CAGR: Compound annual growth rate IFRA: International Fragrance Association PP: Polypropylene CPID: Consumer Product Information Database IUPAC: International Union of Pure and Applied Chemistry INE: National Statistics Institute

# **APPENDICES**

# APPENDIX 1: SHEETS TO BE FILLED OUT FOR SENSORIAL ANALYSIS

		Aerosol air freshener Affective test
Answer the following ques	stions marking with a cros	SS.
Immediately after applying:	How did you like the smell o	of the product?
Dislike extremely Dislike very much Dislike moderately	Dislike slightly Neither like nor dislike Like slightly	Like moderately Like very much Like extremely
After an hour and a half of ap Dislike extremely Dislike very much Dislike moderately	pplication: How did you like Dislike slightly Neither like nor dislike Like slightly	the smell of the product?  Like moderately Like very much Like extremely
After three hours of applicat	ion: How did you like the sn	nell of the product?
Dislike extremely	Dislike slightly Neither like nor	Like moderately
Dislike very much Dislike moderately	dislike Like slightly	Like very much Like extremely

Answer the following gu	estions marking with a cr	Plug-in liquid air freshener Gel air freshener Affective test
Answer the following qu	estions marking with a cr	055.
After an hour and a half of	application: How did you lik	e the smell of the product?
Dislike extremely	Dislike slightly	Like moderately
Dislike very much	Neither like nor dislike	Like very much
Dislike moderately	Like slightly	Like extremely
After three weeks of applic Dislike extremely Dislike very much Dislike moderately	cation: How did you like the Dislike slightly Neither like nor dislike Like slightly	smell of the product?
After one month of applica	tion: How much of the good	d smell do you still smell?
Dislike extremely	Dislike slightly	Like moderately
Dislike very much	Neither like nor dislike	Like very much
Dislike moderately	Like slightly	Like extremely

# APPENDIX 2: COMPLEMENTARY INFORMATION OF THE MANUFACTURING PROCESS

CALCULATIONS OF THE TIME REQUIRED TO HEATING AND COOLING THE TANK. GEL AIR FRESHENER

Calculation of the time required to increase the temperature from 25°C to 70°C	
Diameter (D)	0.5 m
Radius (r)	0.25 m
Liquid height (h)	0.5 m
Total height (H)	0.7 m
Transfer area (lateral and where liquid is)	1.1 m <sup>2</sup>
U assumed	200 W/m² K
Water vapour temperature	115°C
Latent vapour to water heat	2216.03 kJ/kg
Initial internal fluid temperature	25 ℃
Final internal fluid temperature	70 °C
Mass (m)	90 kg
Liquid specific heat (C <sub>p</sub> )	2146.3 J/kg K
Integral solution	Ln(2)
Time -	608.6 s
- Time	10.1 min

Calculation of the time required to decrease the temperature from 70°C to 55°C	
Diameter (D)	0.5 m
Radius (r)	0.25 m
Liquid height (h)	0.5 m
Total height (H)	0.7 m
Transfer area (lateral and where liquid is)	1.1 m <sup>2</sup>
U assumed	200 W/m² K
Initial water temperature	25 °C
Final water temperature	30 °C
Water specific heat (Cp)	4180 J/kg K
Initial internal fluid temperature	70 °C
Final internal fluid temperature	55 °C
Mass (m)	95 kg
Liquid specific heat (C <sub>P</sub> )	2146.3 J/kg K
Integral solution	-0.43612
Time —	404.2 s
Time —	6.7 min

ROUGH ESTIMATION OF THE HEAT FLOW AND STEAM FLOW TO BE SUPPLIED BY THE HEATING JACKET

The Viscomix equipment, INOXPA ®, has a heating jacket because in the proposed manufacturing process it is desired to raise the temperature from 25° C to 70°C by means of water vapour. Below are the calculations that have been carried out to obtain an estimated value.

The heat to be supplied can be calculated as the mass of liquid to be heated multiplied by the heat capacity of the mixture, multiplied by the temperature change of the mixture, as given in equation (1). The values of the parameters used are those given in the table of *Calculation of the time required to increase the temperature from 25°C to 70°C*.

$$Q = m \cdot C_p \cdot (T_{final} - T_{initial}) \tag{1}$$

As estimated above, the heating time for the mixture is 10.1 min, so that the heat flow to be supplied at that time can be obtained. This heat flow will be supplied by a stream of water vapour at 115 °C, which is assumed to be constant so that the required water vapour can be estimated as indicated in equation (2).

$$Q_{flow} = m_c \cdot \lambda_c \tag{2}$$

By isolating the mass of water vapour required per minute, equation (3) is obtained.

$$m_c = \frac{Q_{flow}}{\lambda_c} \tag{3}$$

The following table shows the heat flow to be supplied by the heating jacket and the water vapour flow.

	Heating and mixing
Q [kJ]	8692,5
Q <sub>flow</sub> [kJ/min]	860,6
Q <sub>water vapour</sub> [kg/min]	0,388

Table 2.1. Heat flow supplied and water vapour flow.