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**TITULO: PREVIEW OF A CYCLE LIFE FOR STEROIDAL  
SAPOGENINS EXTRACTION FROM *AGAVE AMERICANA*  
LINNAEUS.**

Trabajo de integración curricular presentado como requisito para la  
obtención del título de Petroquímica

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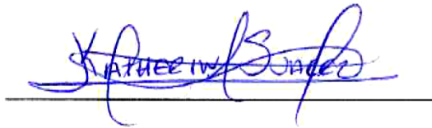
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*I dedicate this thesis to my parents and brother because it is thanks to their support to overcome obstacles that have arisen to fulfill my goals.*

*Katherin Dayana Suárez Macias*

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## RESUMEN

La planta de *Agave americana* Linnaeus también conocida como cabuya o penco azul es considerada una fuente de identidad cultural y tradición de los pueblos indígenas pertenecientes a la región interandina del Ecuador. Chaguarmishqui, fibras naturales y saponinas son los productos más populares obtenidos a partir de esta variedad de *Agave*. Las saponinas son compuestos químicos presentes en muchas especies de plantas, este es un producto que tiene una amplia aplicación en la industria farmacéutica. Con base en esto, el presente proyecto de tesis analiza el diseño de un posible proceso para extraer saponinas de una planta ecuatoriana llamada *Agave americana* Linnaeus analizando diferentes técnicas de extracción para obtener el compuesto de interés. Al definir las etapas del proceso se aplicó la metodología de Análisis del Ciclo de Vida (ACV) para conocer los impactos que tendría cada etapa en los aspectos ambientales. Más tarde, otra metodología llamada Proceso de Jerarquía Analítica (PJA) es parte del estudio para hacer una preselección de una técnica de extracción entre métodos convencionales y no convencionales que establecen varios criterios relevantes, como técnicos, operativos, ambientales y económicos. El análisis da como resultado dos posibles técnicas de extracción, un método convencional y otro no convencional, que cumple favorablemente las diferentes especificaciones de criterios. Finalmente, PJA es una herramienta útil cuando se selecciona tecnología de extracción de un grupo de alternativas para cumplir con el objetivo establecido.

**Palabras clave:** Saponinas, *Agave americana* Linnaeus, Extracción, Análisis del Ciclo de Vida, Proceso de Jerarquía Analítica, Ecuador.

## ABSTRACT

*Agave americana* Linnaeus plant, also known as *cabuya* or *penco azul*, is considered a source of cultural identity and tradition of the indigenous peoples belonging to the inter-Andean region of Ecuador. *Chaguarmishqui*, natural fiber, and saponins are the most popular products obtained from this variety of *Agave*. Saponins are chemical compounds present in many plant species, they are a product that have a wide application in the pharmaceutical industry. Based on this, this project analyzes the design of a process to extract saponins from an Ecuadorian plant called *Agave americana* Linnaeus analyzing different extraction techniques to obtain the interest compound. When defining the stages of the process, methodology Life Cycle Assessment methodology has been applied to determine the impacts that each stage would have on the environmental aspects. Subsequently, another methodology called Analytic Hierarchy Process (AHP) was part of the study to make a pre-selection of an extraction technique between conventional and non-conventional methods establishing various relevant criteria such as technical, operational, environmental, and economical. The analysis results in 2 possible extraction techniques, one conventional and one non-conventional method, which accomplishes the different favorably criteria specifications. Finally, AHP is a useful tool when selecting extraction technology from a group of alternatives to meet the stated objective.

**Keywords:** Saponins, *Agave americana* Linnaeus, Extraction, Life Cycle Assessment, Analytic Hierarchy Process, Ecuador.

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**ABBREVIATIONS**

LCA	Life Cycle Analysis Assessment
AHP	Analytic Hierarchy Process
mamsl	Meters above mean sea level
COX-2	Cyclooxygenase
PGE2	Prostaglandin E2
PPAR	Poly (ADP-ribose) Polymerase
MAPK	Mitogen-activated protein kinase
SFE	Supercritical Fluid Extraction
PLE	Pressurized Liquid Extraction
ASE	Accelerated Solvent Extraction
UAE	Ultrasound-assisted Extraction
MAE	Microwave-assisted Extraction
mg	Milligrams
CR	Consistency ratio
CI	Consistency index
RI	Random index
UV	Ultraviolet
nm	Nanometers



## CHAPTER I

### 1. INTRODUCTION

In the last years and nowadays, the industry is developing a global trend of venturing into the use and exploitation of different natural sustainably resources. In this way, techniques that do not follow a traditional procedure when obtaining products to satisfy quotidian needs in society. The present thesis topic was born following this philosophy in cooperation with the company UyamaFarms S.A (located 5 km from Mira - Carchi), analyzing a possible process to extract saponins from the *Agave americana* Linnaeus plant.

*Agave americana* L. is a plant from the Ecuadorian region, commonly known as *maguey*, *chaguarquero*, *cabuya*, and *penco azul*. This plant is relevant to indigenous groups traditions of many Ecuadorian, mainly for its use as in the elaboration of the traditional drink called *chaguarmishqui*, moreover, the UyamaFarms company redirects the practice for making *chaguarmishqui* towards the production of a drink called Equila. However, after the production process, the parts of the plant utilized to that purpose do not have an additional use, reason why the present investigation arises about the extraction of a compound called saponins to generate additional value on the leaves of the plant (also known as pencas).

The company UyamaFarms S.A is interested in obtaining saponins for their economic value throughout the world. For that reason, a necessary action to develop a strategy to extract saponins from *Agave* is to evaluate the current life cycle to obtain an equilateral and a preview of a procedure that is efficient and adapted to the company's needs. ISO 14040 mentions that the Life Cycle Assessment (LCA) is a methodology to evaluate the environmental aspects and the possible impacts of a product or service throughout its life cycle, that is, of all stages, inputs, and exits from their existence. Furthermore, inside the defined strategy, it is necessary to implement a technique to select adequate extraction methods considering the company requirements.

#### 1.1. Objectives

##### **General:**

To design a process chain with its Life Cycle Assessment focusing on the extraction technologies to obtain saponins from the blue “penco” or “maguey” (*Agave americana* Linnaeus) using the Analytic Hierarchy Process (AHP) methodology.

**Specific:**

- To identify the fundamental stages for saponins extraction using bibliographic sources.
- To choose the most convenient extraction method taking into account the economic, environmental, and energetic parameters.
- To apply the Analytic Hierarchy Process (AHP) methodology for extraction technology selection by analyzing the necessary criteria and parameters.
- To validate the proposal for saponins extraction determining its applicability in the national industry.

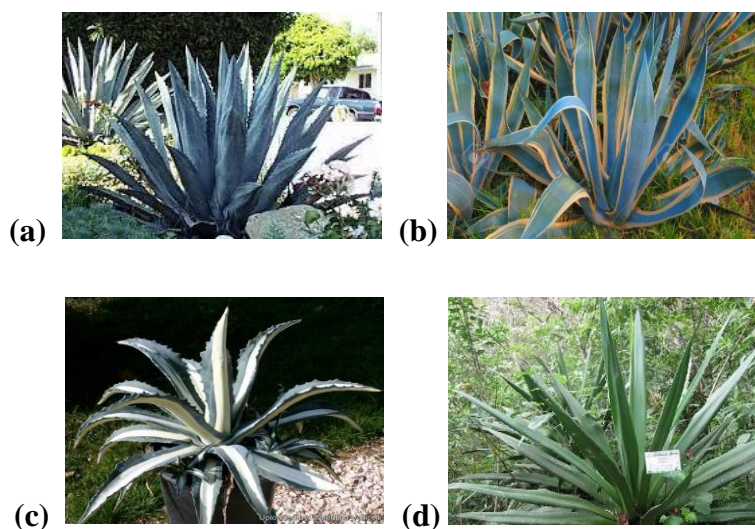
## CHAPTER II

### 2. THEORETICAL FUNDAMENTALS

#### 2.1. *Agave americana* Linnaeus at Ecuador

The genus *Agave* and related species and varieties are native from México and naturalized around America. With a distribution of approximately 340 species belonging to the *Agave* genus, this plant grows in zones of tropical, subtropical, tempered climate, and arid zones between 0 to 3500 mamsl.<sup>[1]</sup> The *Agave americana* Linnaeus is a typical plant from the Ecuadorian inter-Andean region belonging to the genus *Agave*, a gender that belongs to the *Agavaceae* subfamily and *Asparagaceae* family commonly known as *maguey*, *chaguarquero*, black or blue *penco*.

The *Agave americana* varieties found in Ecuador are Linnaeus, Marginata, and Picta (*Figure 1*). The principal difference between these varieties is the color of their leaves since the leaves of variety Marginata have yellow stripes on the edge of its leaves contrary to Picta; while that the Linnaeus *pencas* variety bluish-green color.

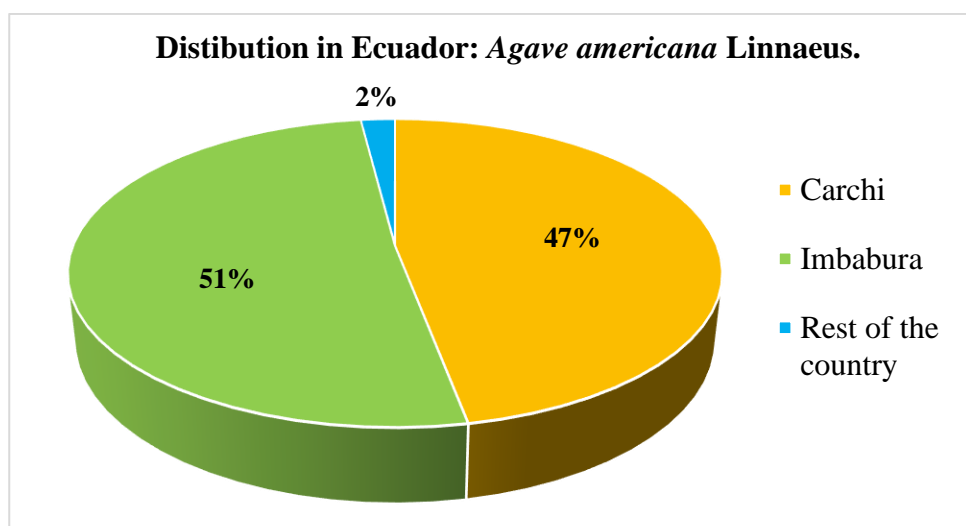


**Figure 1:** (a) *Agave americana* Linnaeus. (b) *Agave americana* Marginata. (c) *Agave americana* Picta. (d) *Furcraea andina*.

Generally, blue *penco* is a 2 meters high species, with 20 to 200 leaves per plant that measure 1-2 m long and 15-20 cm wide, with a color that varies between bluish-white, gray-white, and green. The life time is around of 7 to 10 years approximately depending on the region where its development takes place.

In addition to the *Agave*, there is the *Furcraea* genus of the *Agavaceae* subfamily with species that are usually found in the inter-Andean alley mainly in Ecuador, Perú, and Colombia [2,3,4] with the name *Furcraea andina*, which is morphologically similar to the *Agave sisalana* (another agave species found in Ecuador) which difference relies on the size of the apical spine. In Ecuador, the most common use of this plant is the fiber obtention to produce handicrafts and ropes that due to its resistance are used in the construction field.

There are 2348 hectares cultivated of the *Agave americana* Linnaeus plants in Ecuador, mostly located in Imbabura and Carchi. It can also be found in Pichincha, Tungurahua, Cotopaxi, Chimborazo, Bolívar, Azuay, Loja, Guayas and Manabí [5] (Figure 2). This plant forms a relevant part of the tradition of many indigenous groups primarily by its use as a fiber source to make representative crafts and tools to use during their working day, meals, and as a raw material in the chaguarmishqui elaboration (traditional beverages). The raw material to elaborate that drink is the *aguamiel* (mead sap) of the *Agave americana* L. [6]



**Figure 2:** *Agave americana* L distribution in Ecuador.

The *aguamiel* obtention is through the *capado* 45 days three times in the day except when it is raining because this phenomenon could damage the product. The *capado* is to make a hole at the center on the *penco* for sap accumulation. Generally, the *maguey* produces between ½ to 3 liters of *aguamiel* daily collected, applying the *capado* or *chaguado*. At Ecuador, this is an ancestral practice specifically from the Sierra region to elaborate a traditional drink named *chaguarmishqui*, [7] which is part of rituals and typical celebrations of many indigenous communities that yet keep their traditions. When the *Agave* plant is mature, it has two alternatives - *capado* or flowering - but with the same end. The first is the plant

blooms, and the second is the milking of the *penco* but both finish with plant death (See Appendix A.1).

## 2.2. Uses in Ecuador

In Ecuador, the *Agave americana* L. has an extensive list of uses depending on the parts of the *penco* (See Appendix A.2), ranging from using the plant as a fence in the delimitation of land, food source or chemical compounds. The most common products obtained from the variety *Agave americana* L. in Ecuador are the next:

- **Chaguarmishqui** is a traditional drink prepared from the *aguamiel* of the blue *penco* as aforementioned. This drink is used by the native's groups as an energy sources to carry out works that involve a great deal of physical effort.

- **Equila o miske (Ecuadorian tequila)** is an alcoholic drink elaborated from the fermentation and distillation of *chaguarmishqui*. Today, equila is a product that helps the economy of some people groups keeping the tradition of *chaguarmishqui* to elaborate that alcoholic drink. The name equila is attributed to its Ecuadorian provenance, an alcoholic drink very similar to Tequila since its raw material is a plant belonging to the *Agave* gender.

- **Sweetener** is a product made with honey (1 liter from 3 liters of mead) to replace the use of sugar for the diabetic population; low in calories, without preservatives, and 100% of natural origin. All the properties mentioned above are the reasons because this product is in constant growth in the food industry. This industry shows interest in applying it as an additive to elaborate low-calorie drinks, yogurt, nutritional supplements, confectionery, and other products.<sup>[8]</sup> Furthermore, the sweetener obtained from *Agave americana* L. is beneficial to people who suffer sickness-related directly with sugar consumption.

- **Saponins** are used by people belonging to indigenous Andean groups since after the plant has crushed, it produces a substance used as a shampoo. However, that product has the disadvantage that the *Agave americana pencas* contain a chemical compound called calcium oxalate that irritates when it is in contact with the skin.

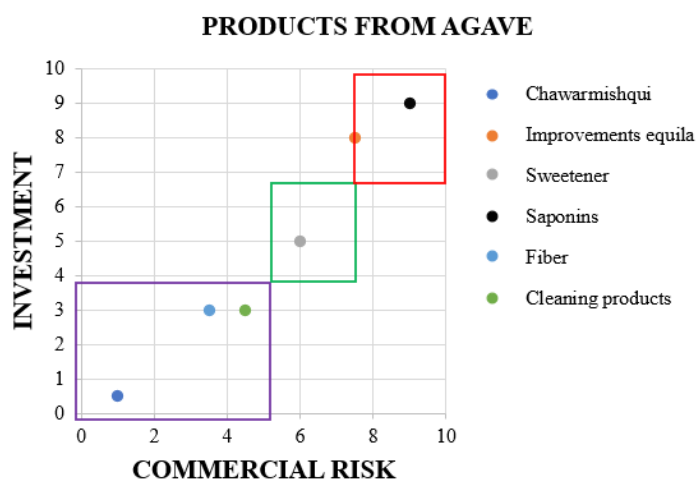
- **Fiber** from *Agave americana* L. is an adequate source for elaborating ropes, paper, filters, mattresses, rugs, and handicrafts; because of that material is very hard to manufacture clothes.

Nowadays, fiber serves to reinforce plastic materials, as it is a material that represents considerable importance in the green economy, being 100% biodegradable and a renewable resource.<sup>[9]</sup> The fiber of *Agave americana* L. is of great interest due to its cost, properties, and morphology, although the cellulosic fiber of that plant is less studied in comparison with other plants such as jute, sisal, flax, and hemp.

- **Cleaning products** obtained from *Agave americana* L., such as soaps and detergents. However, without the correct process, products cause skin irritation due to a chemical compound named calcium oxalate.

In Ecuador exist the *Agave Spirit*, a project focused on the conservation and production of *Agave americana* L., a plant considered the oldest heritage in the inter-Andean valley. This project manages four different fields, such as agave route, museum, factory, and agave trails; each of these fields had been exposed in international events and failures related to the *Agave* gender plant. *Agave Spirit* requires approximately 200 liters of juice to transform this into different products between equila, beer, honey, and others. The elaboration of each of these products implements traditional and millenary practices, especially in the harvest of the plant.

To know which product represents the best option to develop a new Life Cycle Assessment is necessary to perform an analysis relating to Investment versus Commercial Risk (*Figure 3*). The range for coordinates and abscissa is 0 to 10. Bibliographic resources were the principal tool to recover all the information related to the study of these parameters, after making the graphic representation can choose the saponins obtention from maguey.



**Figure 3:** Investment vs. Commercial Risk of products from.

The range applied to do the graphic is from 0 to 10, with a division by zones in the axis of risk: zone 1 (purple line), zone 2 (green line), and zone 3 (red line). These separations depend on the complexity to elaborate on the different products, basically in the equipment, money (Table 1), and yields of the process.

Zone 1 encompasses all the products whose production process requires a low investment (cost of manufacturing per unit of product) and does not represent a high risk of development.

Zone 2 focuses on the process of elaboration of products with a higher commercial risk, in addition to requiring higher investment than those of Zone 1; taking into account the cost of unit production of the product, continuity in the industrial field, and its demand to cover specific requirements of the population.

Finally, Zone 3 shows the products whose process is rare to find in the industry due to its high commercial and industrial risk, as well as requiring a high investment compared to the products of Zones 1 and 2.

**Table 1:** *Elaboration costs of products of Agave americana L.*

Product	Quantity per product unit	Inversion (USD)
Chaguarmishqui	250 ml	0,54 <sup>[10]</sup>
Improvements equila	750 ml	5,83 <sup>[11]</sup>
Sweetener	63 gr	3,00 <sup>[12]</sup>
Saponins	25 gr	16,00 <sup>[13]</sup>
Fiber	453,6 gr	1,20 <sup>[14]</sup>
Cleaning products (liquid soap)	130 gr	1,05 <sup>[15]</sup>

In this case, the products included in the Zone 1 represents inversions values between 0,01 to 2,99 dollars per unit, Zone 2 are products with an inversion that varies from 3 to 8,99 dollars per unit, and the prices of products in the Zone 3 are most than 9 dollars. Is necessary to focus on the equila improvements since this product is in the limit between Zone 2 and 3, representing a risk by low existing research to change or redesign the actual process in the alcoholic drink obtention from *Agave americana* (tequila and equila). On the other hand, saponins have registered in Zone 3, which implies that the investment will be quite high, as well as the risk. That is because extracting saponins from plants belonging to the *Agave*

genus is almost absent in the industrial field; therefore, carrying out said process would represent a high risk.

However, knowing the economic value of said product motivates the investigation of a possible process chain to extract saponins from the *Agave americana* Linnaeus; furthermore, with the said investigation, determine how challenging it would be to carry out the process.

### 2.3.Saponins

One of the main characteristics of the plants belonging to the *Agavaceae* family is that it contains a chemical compound called saponins, whose main application is within the medicinal field. These are compounds with a structure that consists of a nonpolar aglycone called sapogenin coupled to one or more sugar moieties, which can join to one, two, or three side chains. To obtain sapogenins from saponins, the process required is hydrolysis.

Saponins have great structural diversity because of the variability in the aglycone, sugar composition, and location.<sup>[16,17]</sup>

Sapogenins obtained from saponins have diversified pharmaceutical properties such as:

**Table 2:** *Sapogenin's pharmaceutical properties.*

Pharmaceutical properties	
Hemolytic	Antibacterial or antimicrobial
Molluscicide	Antiparasitic
Anti-inflammatory	Antitumor
Antifungal or anti-yeast	Antiviral

Saponins are secondary metabolites in which they occur in the form of glycosides. The word saponin comes from the Latin word *sapo*, which means soap.<sup>[18]</sup> A qualitative method to verify saponin's presence is observing the formation of a stable and abundant foam when it is in aqueous solution and subjected to a process of agitation.

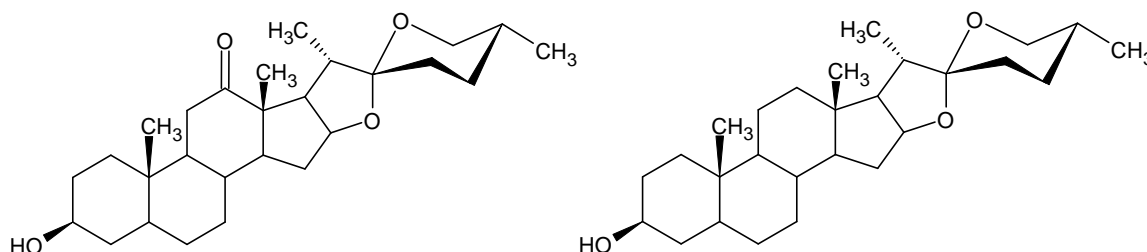
The discoveries of saponin's pharmaceutical properties have been intensified over time. Therefore, the research about obtaining saponins from plant materials, and the investigation of new extraction technologies to achieve maximum performance in terms of the amount of raw material to be processed has also increased.



The saponins commonly found in the *Agave americana* Linnaeus are hecogenin and tigogenin glycosides; however, these compounds are present in different concentrations depending on the age of the plant. Recent studies about the blue *penco* revealed that as the plant grows (seedlings, 4, 9, and 12 years), the content of hecogenin and tigogenin glycosides varies, while the content of other sapogenins remains the same. The amount of hecogenin present in the chemical composition of the *Agave americana* L. is higher if the plant is immature while in the mature plant, the presence of hecogenin is less.<sup>[19]</sup>

“Hecogenin is a steroidal sapogenin (*Figure 4*) with anti-cancer, antiproliferative, antioxidant, and anti-inflammatory properties. Hecogenin inhibits the survival of synovial cells of human rheumatoid arthritis; this effect is due to an increment in cell apoptosis”.<sup>[20]</sup> The broad field of applications that has the hecogenin generate an unsatisfied demand of 9000 tons/ year to international level.

Tigogenin is one of the steroidal sapogenins (*Figure 4*) widely used for synthesizing steroid drugs. It might have a protective effect on bone and help preventing the development of osteoporosis. “The tigogenin induces apoptosis, associated with overexpression of COX-2 (cyclooxygenase) correlated with overproduction of endogenous PGE2 (prostaglandin E2). Tigogenin inhibited PPAR (poly (ADP-ribose) Polymerase) gamma and via the p38 MAPK (mitogen-activated protein kinase) pathway”.<sup>[20]</sup>



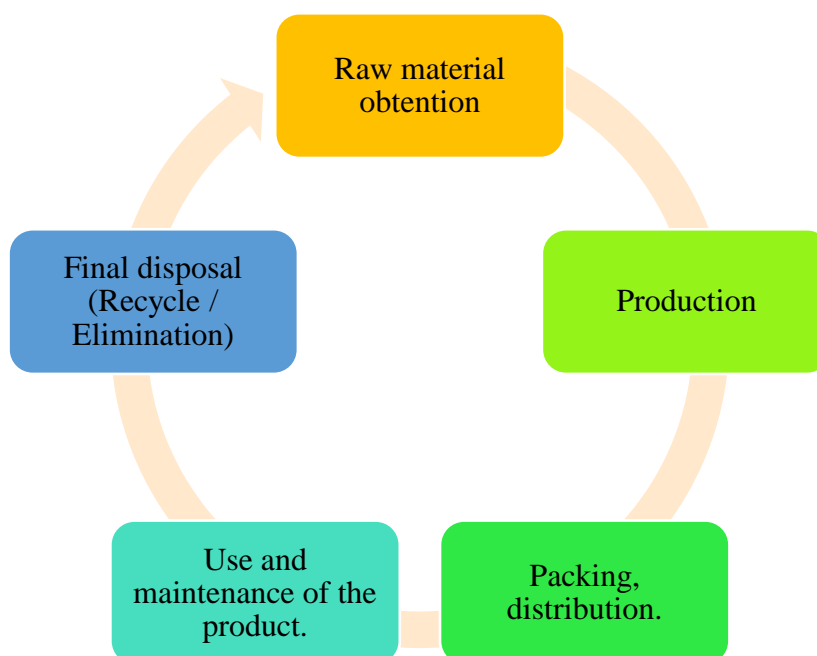
**Figure 4:** Sapogenins: hecogenin (left) and tigogenin (right).

## CHAPTER III

### 3. LIFE CYCLE ASSESSMENT FOR SAPONINS EXTRACTION

#### 3.1. General Information

Each engineering project has objectives linked to the obtention of a product, process, and service, which serve to accomplish a necessity through diverse activities. Some of these activities consist of a group of stages called Cycle Life (*Figure 5*). The ISO 14040 mentions that the Life Cycle Assessment (also known as Life Cycle Analysis) is a methodology to evaluate environmental aspects and potential impacts of a product or service throughout its life cycle, that is, of all stages, inputs, and outputs streams of its existence.<sup>[21]</sup>



*Figure 5:* General cycle life of a product.

The application of the Life Cycle Assessment (LCA) methodology is extensive since it can be part of the analysis and decision-making process focused on solving problems in the political, industrial, and environmental fields. The principal advantage coming from the application is that this tool has the characteristic that allows to obtain a qualitative and a quantitative analysis.

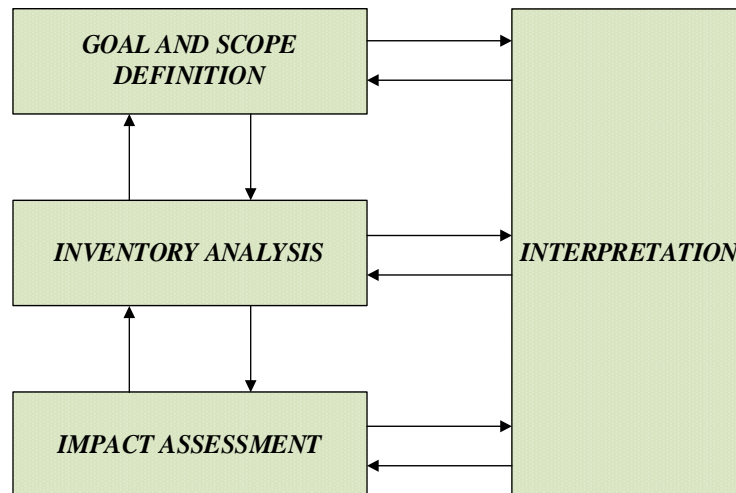
To develop a Life Cycle Assessment (LCA) it is necessary to follow a series of step (*Figure 6*) defined below:

Step 1: Goal and scope definition to know the part of a product life cycle considered in the assessment and the limits that are part of the system and the identification of the process stages. On the other hand, this stage analyses the amplitude, proof, and details of the study.

Step 2: Inventory analysis helps to identify and quantify the inputs (raw material and energy required) and outputs (emissions and waste) of the life cycle. This stage is fundamental to evaluate environmental and potential impacts.

Step 3: Impact assessment allows the classification and characterization of environmental and human health impacts.

Step 4: Interpretation is the final stage of the LCA process that consists of the union of stage 2 (inventory analysis) and stage 3 (impact assessment) to obtain conclusions and recommendations that allows to decide about the process, and develop the improvements that the project needs.



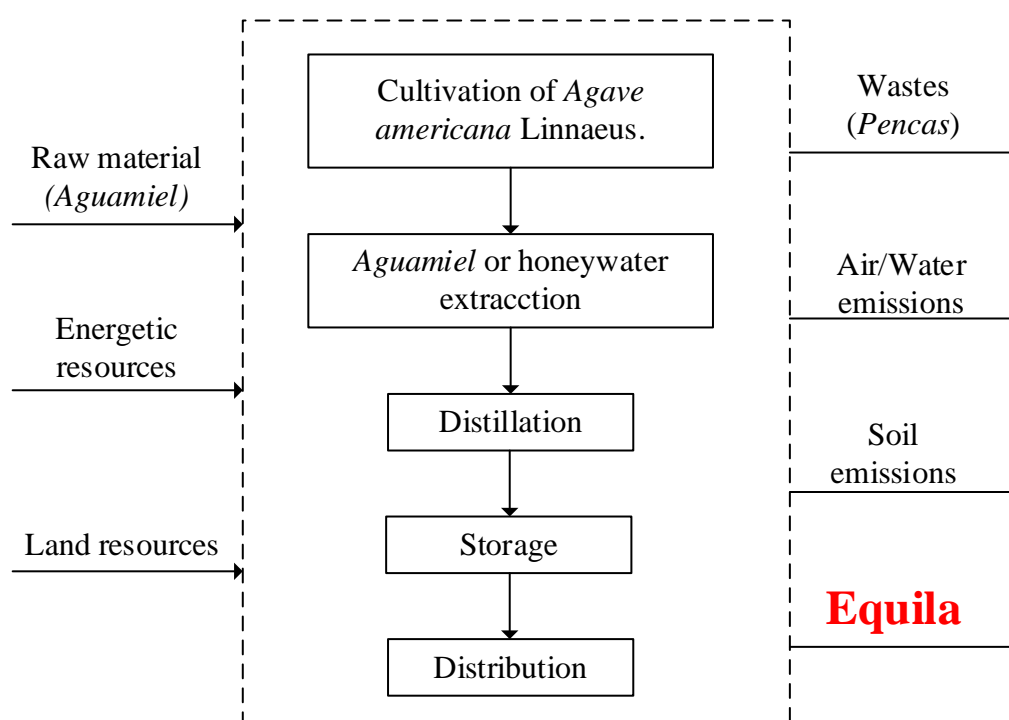
**Figure 6:** Life cycle assessment structure.

Concerning environmental impact, LCA has categories related to the use of resources, ecological consequences, and human health. Some examples of these categories are the assessed damages or effects like global warming (greenhouse gases), acidification (soil and water source), eutrophication, smog, eco-toxicological, ozone layer depletion, human toxicological pollutants, habitat destruction, desertification, land use as well as reduction of minerals and fossil fuels. <sup>[21,22]</sup>

### 3.2. LCA application

#### 3.2.1. Actual and New LCA for *Agave americana* L.

Nowadays, the company uses the *Agave americana* to extract its *aguamiel* to distillate equila or Ecuadorian tequila (*Figure 7*), an alcoholic drink elaborated from the fermentation distillation of de aguamiel obtained from the plant. The interest of the company to extract saponins from *Agave americana* Linnaeus is due to the price of this compound, for example, a gram of hecogenin in the international market costs between 6 and 124 USD while each gram of tigogenin has a value of 200 USD. <sup>[23]</sup>

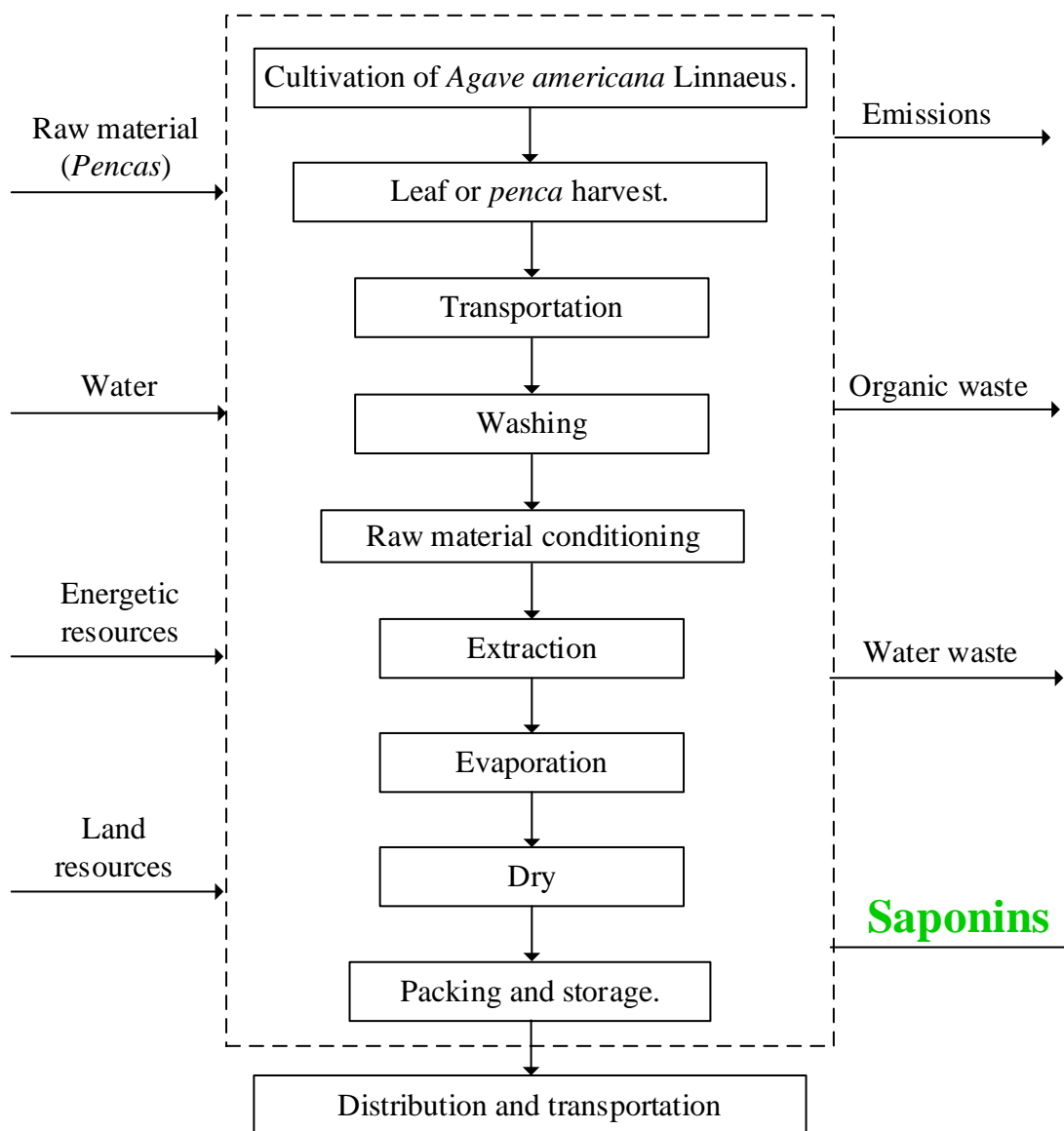


*Figure 7:* Life Cycle for equila production.

The extraction of saponins from the *pencas* (leaves) of the *Agave americana* L helps to add value to the waste after equila production. In that way, this part of the plant would have an application different than being raw material to fertilizer or as livestock food elaboration. Whereby it is necessary to develop an assessment on the current life cycle (equila production) to obtain a preview for adaptation of a new life cycle focused on the interest compound extraction process represented in *Figure 8*.

An analysis of each stage and streams of the process from the economic, environmental, and technological point of view is relevant to know the impacts that imply the saponins

extraction. The inputs of the process are principally raw material, water, solvent, energy, and land resources, while the outputs are the emissions to the air and water along with the organic waste.



**Figure 8:** Cycle life for saponins extraction.

### 3.2.1.1. Cultivation

Generally, the *Agave* plants cultivation can be wild cultivation or semi cultivation because its development needs a long time that varies between 7 to 10 years depending on the weather of the zone and cultivation treatment. To cultivate the *Agave americana* is necessary to use bulbils (basal shoots growing around the mother plant) as seed. The procedure for the collection of bulbils from the mother plant is manual, as it is a practice that requires the pups

to be cut at ground level, continuing with a transplant in pots that are adapted for plant growth then can be planted in the land. Controlled *Agave* cultivation requires different treatments compared with the traditional method because it needs control on irrigation and the use of pesticides, fungicides, and fertilizer.

**Irrigation:** The *Agave* plants do not need an excess of water due to its drought tolerance; for that reason, the watering can be done once a month.<sup>[24]</sup> An investment to acquire equipment to carry out the irrigation of the sowing would be expensive despite the few use; that is to say, it isn't necessary to invest in acquisition of an irrigation equipment to supply water to the *Agave* plant.

**Pesticides and fungicides:** A characteristic of the chemical composition of *Agave americana* L., is that these compounds work as a natural barrier to prevent the attack of insects and fungus, that is why it is unusual to find plants affected by these plagues. This plant has antifungal, molluscicide, insecticide, and antifungal properties.<sup>[25]</sup>

**Fertilization:** Generally, people use organic fertilizer to fertilize the soil before and after *Agave* planting. For chemical fertilizers application is necessary to know the land composition, plant age, and the type of chemical compound (fertilizer) used.<sup>[26]</sup> In controlled fertilization, the most common is to use nitrogen (N), phosphorus (P), and potassium (K) as fertilizers to get a large production.

#### **3.2.1.2. Leaf (*penca*) harvest**

When selecting the leaf to be the raw material for saponins extraction is relevant to consider parameters such as age, amount, and size. The saponins extraction process from *Agave* leaves should be the one of *the capado* process (*aguamiel* obtention to elaborate equila), giving the plant a different use to the traditional one (*chaguarmishqui*) since the plant dies after that. Another consideration that should be taken into account if the harvest will be mechanically or manually; although to carry out the process of extracting saponins in the UyamaFarms S.A. company, it is advisable to perform the harvest manually.

#### **3.2.1.3. Transportation**

Cargo transport must be taken into account depending on the raw material's volume to carry the *penas* from sow land to extraction facilities. For example, if the plants are far from the

installations, it is necessary to use transports such as trucks or tractors; on the contrary, with a nearby crop is possible to use wagons.

As mentioned before, the transport and energy that implies use it depends on the distance between the cultivation area and the extraction plant. In some cases, depending on the circumstance is necessary for the use of two types of energy. The energy that participates in transportation can be energy from human's physical output or a vehicle that needs fuel for its operation.

#### **3.2.1.4. Washing**

Washing be can do manually (workers wash the *pencas*) or mechanically (using a ramp and a mechanism that sprays water or ozone), generally for washing water or ozone the most used elements. Both options are accessible for UyamaFarms SA company since the company uses water to wash the avocados in the process of avocado oil extraction. They are considering using ozone to increase efficiency in eliminating wastes from the fruit.

#### **3.2.1.5. Raw material conditioning**

Extracting saponins is a process that can implement the sap obtained from the *pencas* or drying it as raw material. For that reason, depending on the type of raw material, the conditioning before of extraction of this varies. *Figure 9* shows the cut of a *penca* where it can be seen that for the most part, the stalk is solid; therefore, a different procedure is required to obtain the sap than when using the whole *penca*.



*Figure 9:* Horizontal cut of *Agave* leaf.<sup>[27]</sup>

##### **3.2.1.5.1. Raw material: sap**

There are two ways to obtain the juice or sap from blue *penco* these methods are the drying out and pressing. The drying out consists of draining the *pencas* by gravity in a closed and

sterile space to prevent the contamination of the sample. While the pressing is a procedure that implies to exert pressure on the *penca* leaves through the use of a machine that can be a screw press.

A significant advantage that exists between the two methods is the possibility of the fiber obtention. When using pressing causes the fiber destruction and it cannot be used; but if the *penca* has drained, the fiber remains intact to use it as a raw material in the elaboration of other products. It is important to note that the yield of *Agave americana* fiber is approximately 6 tons per hectare. After the juice extraction from *penca* to obtain the fiber it is necessary to apply a technique called decortication in which the *penca* should be hit to get a manageable leaf that can be scratched just until the plant fiber is extracted. Decorticated fiber is washed before drying since fiber quality depends on moisture content.<sup>[28]</sup>

#### **3.2.1.5.2. Raw material: *Agave* leaves powder**

After washing the raw material in the case of use *penca*s for extraction of saponins, it is necessary to dry and grieve them to obtain a powder to put it through an extraction process. The drying process significantly reduces the humidity of the material.

#### **3.2.1.6. Filtration**

In the case of extraction from the sap, this stage helps to obtain a clean juice eliminating impurities as residual fiber that remains after the juice extraction; the product is commonly called clear juice. But, if the raw material is the *penca*s powder, the filtration acts as a barrier to separate the solid from the product after extraction.

#### **3.2.1.7. Saponins extraction**

The extraction definition is the obtention or removal of soluble material from an insoluble matrix. This procedure is a fundamental stage for the saponins extraction process. At present exist extraction procedures done by the application of conventional or non-conventional techniques. Conventional techniques work based on the solubility of plant material in the solvent, and often the volume of the solution that works as a solvent used to extract the solute desired is so high. For that reason, these methods are the least friendly to the environment, because the pollution caused by the type of solvents and its concentration can result in significant damage. The non-conventional techniques are considerate of less dangerous,



safer chemical products, more energetic efficiency, and pollution prevention due to the minimal use of solvent.

#### **3.2.1.8. Evaporation**

The principal function of the evaporation phase is the removal of the solvents used during the extraction phase. In this course of extraction, it is possible to obtain a concentrated solution of the compound of interest to undergo a drying process. The recommendable equipment to use in this stage of the saponins extraction process is the evaporator.

#### **3.2.1.9. Drying**

The drying helps to obtain saponin's powder, which is a more handling form of the material for its packaging and distribution. Furthermore, an adequate dried on the sample provide a better product conservation and helps to obtain a product almost without enzymatic activity and microbial growth facilitating its storage.

#### **3.2.1.10. Packing, storage, and distribution**

These are the last three stages in the process of the interest compound extraction. Packing depends on the customer and the amount required by them. At the same time, storage and distribution are activities that depend on the company responsible for the extraction process of sapogenins or saponins.

### **3.2.2. Extraction Process**

To extract saponins from the leaves of the *Agave americana* L. plant it is recommendable to use its sap or the solids part as a raw material; for this reason, an analysis focused on the two options is relevant to know the best raw material for saponins extraction. To get it requires a mass balance taking into account the next general considerations:

- From all hectares for *Agave americana* Linnaeus production in the Carchi and Imbabura provinces, only 2,5% are near to the extraction plant, and 10% of the selected area is in the maturity (ready to *aguamiel* production) age. The final area selected as raw material is equal to 5,75 hectares.
- The selected crop density is 2000 plants per hectare.

- The *penas* would weight 200,2 kg considering an average of 70 leaves per plant.<sup>[29]</sup>  
The humidity of the *penas* is 67%, while the difference is the solids part.<sup>[30]</sup>

Figure 10 and 17 represents the flowchart of the process to extract saponins from the sap or leaves of *Agave americana* L., from the raw material reception until the final product obtention; the graphic contains the inputs and outputs of each stage considering the function of each one of these described in Table 3 and Table 4.

### 3.2.2.1. Saponins extraction from *Agave* sap.

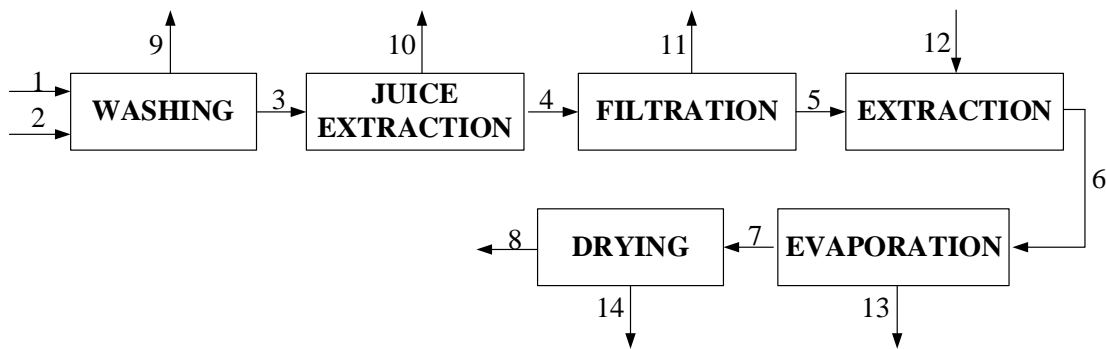
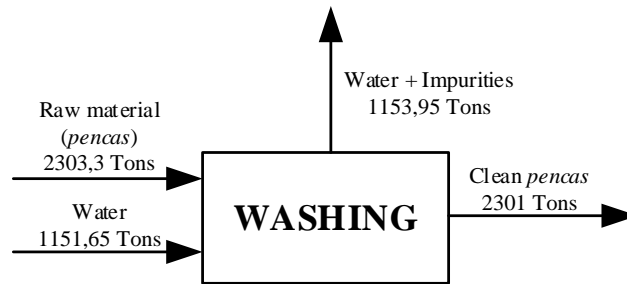


Figure 10: Flowchart for saponins extraction from the sap.

Table 3: Streams list for Fig. 10.

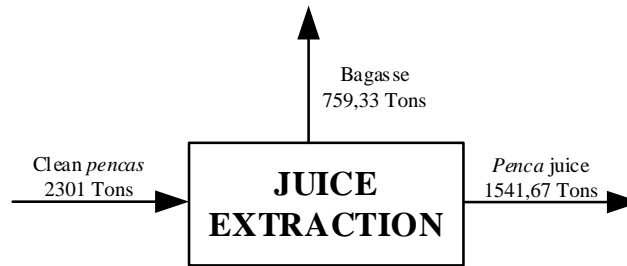
STREAMS DESCRIPTION			
1	Raw material input	8	Dried saponin powder
2	Water	9	Inert water
3	Clean <i>Agave penas</i>	10	Bagasse
4	<i>Penca</i> juice or sap	11	Residual bagasse
5	Clear <i>penca</i> juice	12	Ethanol (96%)
6	Dilute saponin solution	13	Steam
7	Concentrated saponin solution	14	Humidity

Then to know the process stage with the streams that are part of this it is necessary to elaborate a mass balance for these stages considering some specifications indicated in each phase. The relation *penas*: water for washing (Figure 11) is equal to 2:1, while the percentage considered for impurities and dust present in the *penas* is 0,1% over its mass.



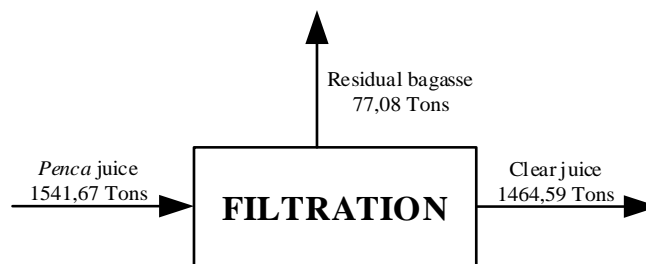
**Figure 11:** Flowchart of mass balance for the washing stage.

The density of *aguamiel* is  $1,0226 \text{ g/cm}^3$ ,<sup>[31]</sup> and the analysis register as the bagasse the solids of the plant, 33% in the juice extraction phase (*Figure 12*).



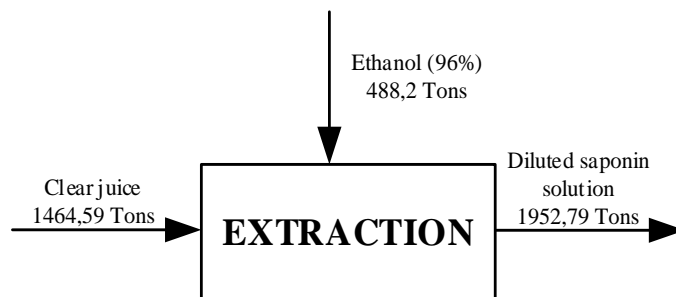
**Figure 12:** Flowchart of mass balance for the juice extraction stage.

In the next phase, the filter works to eliminate the residues that remain in the raw material after the juice extraction phase; for mass balance (*Figure 13*), the assigned percentage to the residues is 5%.



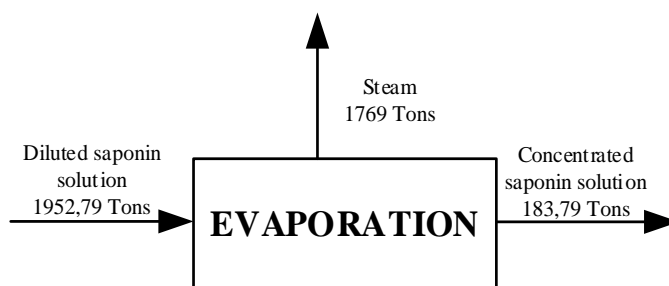
**Figure 13:** Flowchart of mass balance for the filtration stage using the sap.

For the extraction (*Figure 14*), the mass relation between *aguamiel* – solvent is 3:1. The ethanol with a purity of 96% is the solvent used to carry out the saponins or sapogenins extraction from *Agave americana* Linnaeus.



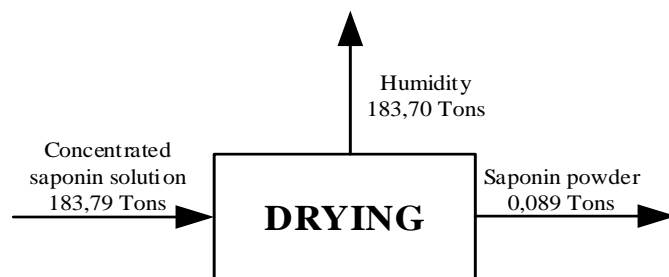
**Figure 14:** Flowchart of mass balance for the extraction stage using the sap.

*Figure 15* shows the mass balance for the evaporation, it represents a fundamental phase of the process since this helps to increase the concentration of the interest compound. In this case, the alcohol vaporization helps to obtain a product that passes from a chemical concentration of saponins equal to 8% until 85% (commercial requirement).



**Figure 15:** Flowchart of mass balance for the evaporation stage using the sap.

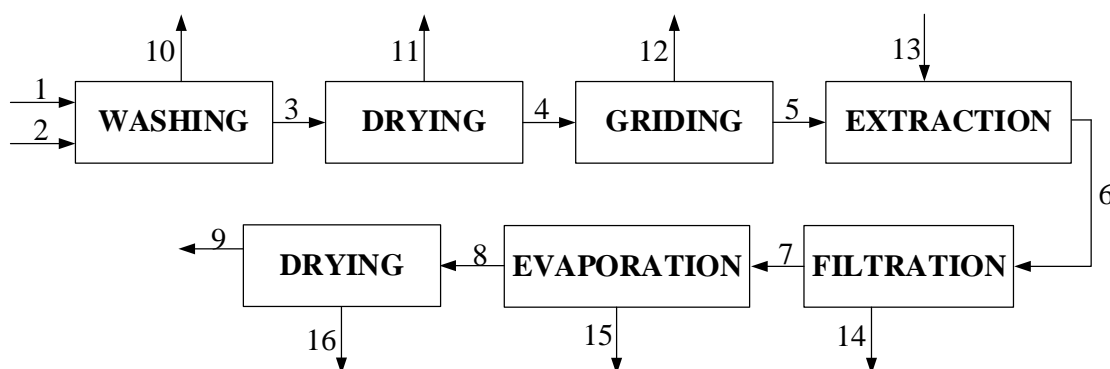
The calculus for the mass of saponins powder (*Figure 16*) obtained from the *Agave americana* L. was performed considering a registered percentage <sup>[19]</sup> equal to 0,00605%, considering that the harvest of the *pencas* is from a mature *Agave*.



**Figure 16:** Flowchart of mass balance for dry stage using the sap.

### 3.2.2.2. Saponins extraction from *Agave* leaves.

Next, it is relevant to know the mass of saponins in the extraction process that uses the solids part of the leaves of the plant as raw material (*Figure 17*), and thus compare this result with that obtained in the case of using the mead (sap) of the plant. The streams that are part of the process have been identifying in Table 4.

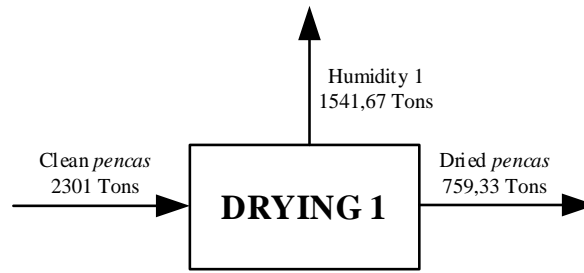


**Figure 17:** Process to extract saponins from the leaves.

**Table 4:** Streams list of Fig. 17.

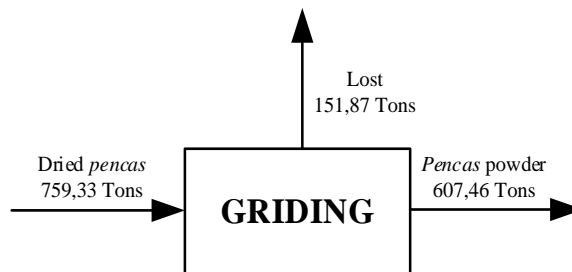
STREAMS DESCRIPTION			
1	Raw material ( <i>Agave pencas</i> ) input	9	Saponin powder
2	Water	10	Inert water
3	Clean <i>Agave pencas</i>	11	Humidity 1
4	Dried <i>pencas</i>	12	Lost
5	<i>Penca</i> powder	13	Solvent (Ethanol 96%)
6	Solvent + Powder	14	Solids
7	Diluted saponin solution	15	Steam
8	Concentrated saponin solution	16	Humidity 2

For extraction of saponins from leaves (solids) of the *Agave americana*, the mass balance for the washing phase (*Figure 18*) is the same because of the compound of interest for the extraction will come from the sap. After washing the *pencas*, they go through drying and milling to obtain a powder that would undergo the extraction phase.



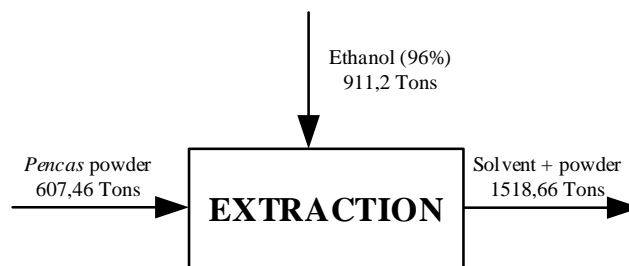
**Figure 18:** Flowchart of mass balance for the first dry stage.

However, in the gridding or milling phase (*Figure 19*) is considered a material loss percentage equivalent to 20% on the mass that enters the machine in charge of the process.



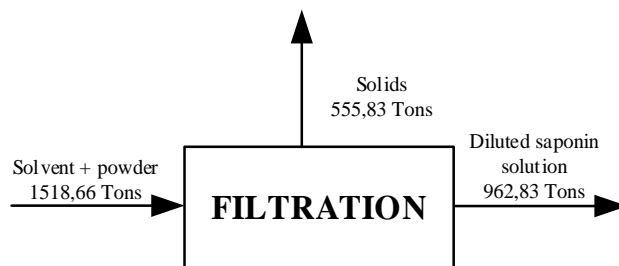
**Figure 19:** Flowchart of mass balance for the gridding stage.

This phase (*Figure 20*) requires ethanol at 96% purity and the extraction that uses the *aguamiel* (sap) as raw material. Additionally, the relation solvent is equal to 3:2.

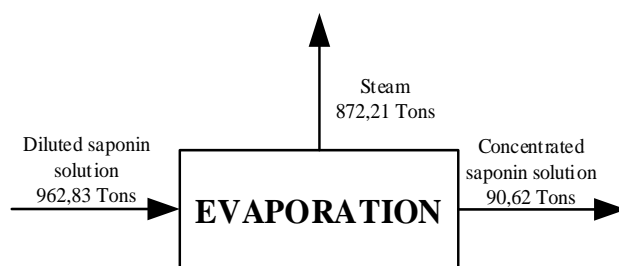


**Figure 20:** Flowchart of mass balance for the extraction stage.

For the phase represented in *Figure 21*, the specifications considered are that 10% of the mass (powder) represents the soluble solids in ethanol. This mass is not part of the filtered solids however, 1% of the solvent is part of the filtered solids (cake).

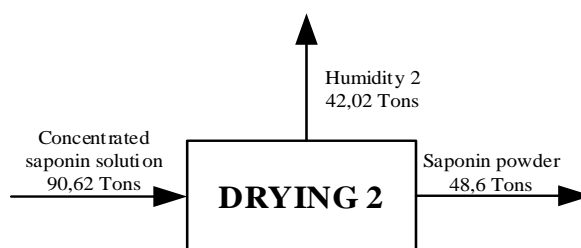


**Figure 21:** Flowchart of mass balance for the filtration stage.



**Figure 22:** Flowchart of mass balance for the evaporation stage.

Then to have a concentrated saponin solution after evaporation (*Figure 22*) is necessary to dry the material to obtain the saponin powder (*Figure 23*). The saponins of the dry raw material represent 8%.<sup>[32]</sup>



**Figure 23:** Flowchart of mass balance for the second dry stage.

Knowing the information related to the quantities of possible saponins to extract from the plant, either using the sap or the leaves of the *Agave americana*, it is necessary later to know the economic benefit that the extraction of the product of interest implies in both procedures (Table 5). For that reason, the saponins price set for the analysis is 20 USD per kilogram of product. However, the price of saponins can vary in the market, 25 grams of saponins can cost around 30 dollars (USD), depending on the concentration of the product.

The established economic value considers that the purity and saponin concentration of the final product to obtain once each of both designed processes are applied is unknown.

**Table 5:** Total economic value of saponins.

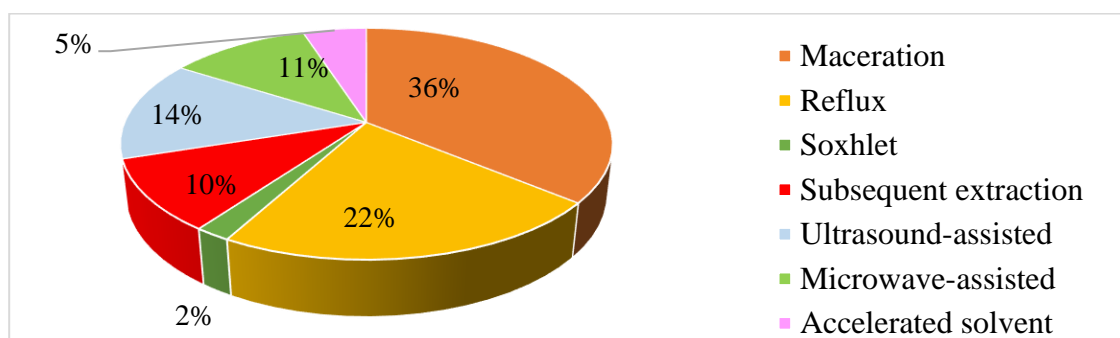
	Mass (kg)	Total (USD)
<b>Saponins from sap</b>	89	1780,00
<b>Saponins from leaves</b>	40600	812000,00

### 3.2.3. Extraction methods

#### 3.2.3.1. Solvent

This convectional technique to extract saponins can use water or alcohol. In the past, to obtain saponins was made by using aqueous-alcoholic solutions followed of the evaporation of the more volatile solvent, and then, the obtention of saponins was carried out by liquid-liquid extraction.

Maceration, Soxhlet, and Reflux are the methods most used among solvent extraction. Currently, maceration is the most common process to extract saponins for researching (Figure 24), although this technique uses a high amount of solvent; the widely used of this technique is because of the equipment and utensils are of low complexity.

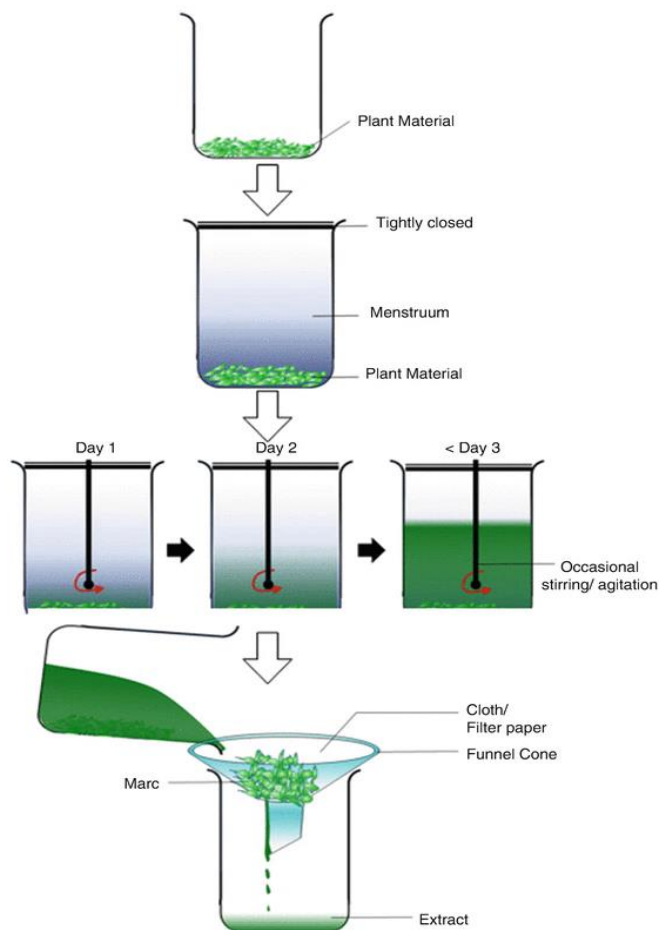


**Figure 24:** Techniques for extraction of saponins from plants.<sup>[33]</sup>

Generally, the solvents used are water, ethanol, and methanol, although the use of methanol in saponins extraction affects its chemical structure; because it may result in the formation of methyl derivates not found originally in the plant composition. Thus, for obtaining the real structure of saponins, cold extraction with ethanol-water solutions is recommended.<sup>[34]</sup> On the other hand, ethanol is an environment friendly solvent.



**Maceration** is a simple method that consists of a solid-liquid extraction (*Figure 25*) that needs a long time, a high amount of solvent (generally ethanol and methanol), and the efficiency of extraction is low.

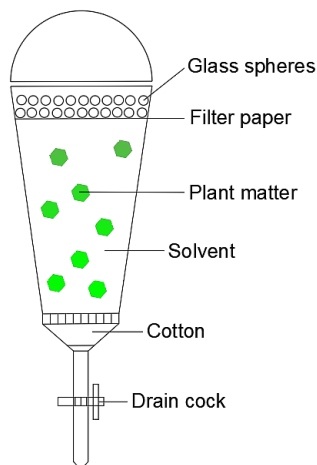


**Figure 25:** Maceration extraction.<sup>[35]</sup>

In this conventional method, heat is transfer through convection and conduction, and the choice of solvents will determine the type of compounds extracted from the samples.<sup>[36]</sup> The maceration consists of soaking the plant material (powder) in the solvent at room temperature during the required time until get a high yield. This technique results favored in terms of large or industrial scale adaptation.

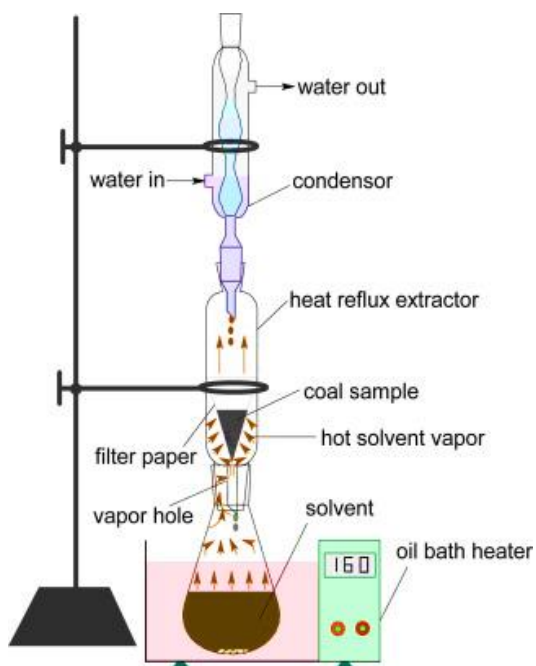
**Percolation** is a technique that operates at a moderate rate until complete the extraction.<sup>[37]</sup> This technique is a continuous process that often replaces the solvent used. The yield of percolation for saponins extraction is similar to reflux (See Appendix B) while comparing it with maceration which has less yield.

The percolation works as the maceration, but this technique uses a piece of equipment called percolator (*Figure 26*). This equipment has a cover with two openings, one for the entry of the plant material, and the other for the solvent. It has glass waits which press material preventing it from floating at the end of the percolator; there is a layer of cotton which works as a filter preventing an obstruction in the stopcock when collecting the percolated product.



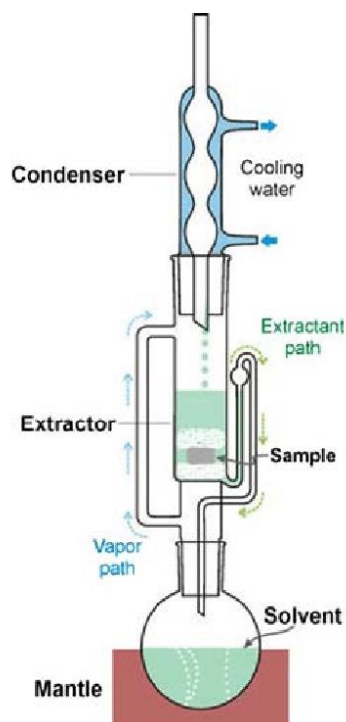
**Figure 26:** Scheme of a percolator.

**Reflux and Soxhlet extraction** work with a similar principle. Both methods involve a distillation process heating a solution until it boils and then returning the condensed vapors to the original flask.<sup>[38]</sup>



**Figure 27:** Scheme Reflux extraction.<sup>[39]</sup>

However, to compare Reflux (*Figure 27*) and Soxhlet (*Figure 28*) the required extraction time with the rest of solvent extraction techniques, it is less than the maceration and percolation time. Although this parameter is a disadvantage for Reflux and Soxhlet extraction since its time consuming where it required at least one hour for an extraction.<sup>[33]</sup>



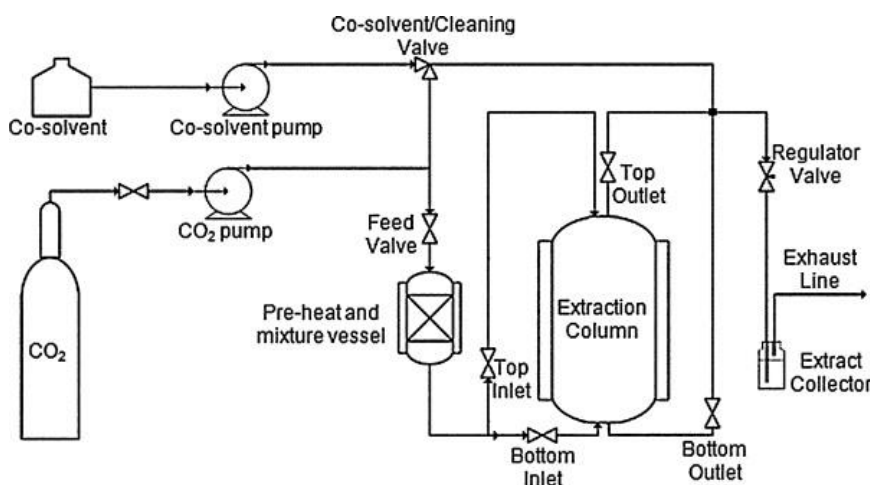
*Figure 28:* Scheme of Soxhlet extraction.<sup>[40]</sup>

### 3.2.3.2. Supercritical Fluid Extraction (SFE)

SFE is a non-conventional technique environmentally friendly; since it replaces the use of excess of solvents by supercritical fluids. The SF is a substance with the same behavior as a gas (diffusivity) or liquid (solubility). That carbon dioxide ( $\text{CO}_2$ ) is the most used in the extraction process by supercritical solvent (SC –  $\text{CO}_2$ ) being a compound with the following advantages: <sup>[41]</sup>

- Selectivity
- Low supercritical temperature ( $31^\circ\text{C}$ )
- Inertness
- Non-toxicity
- Low investment
- Lower trace of organic compound

For extraction of saponins, it can be use water and ethanol additional to  $SC - CO_2$ , causing an increase in the efficiency of the procedure. The reason why this method is less popular is due to the high equipment (*Figure 29*) and energy costs.<sup>[42]</sup>



**Figure 29:** Scheme of Supercritical fluid extraction.<sup>[43]</sup>

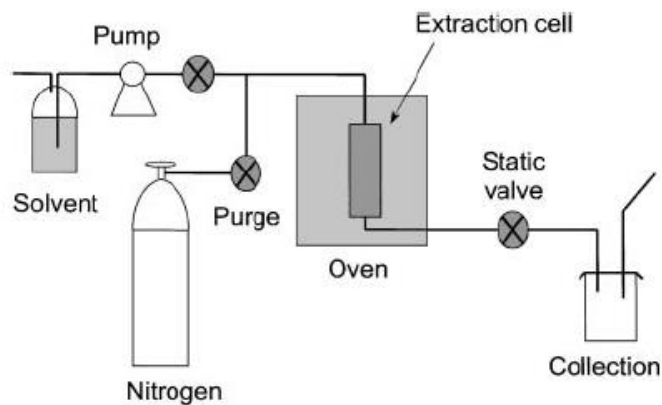
### 3.2.3.3. Pressurized Liquid Extraction (PLE)

This technique consists in the application of high pressure to increase extraction efficiency. A clear difference between SFE and PLE extraction is the solvent conditions since the solvent used in SFE works at its critical condition (temperature and pressure) while the PLE solvent is in its boiling point temperature and pressure that keeps the liquid state of the solvent. PLE represents an alternative with many advantages than traditional extraction techniques (Soxhlet, percolation, maceration, and Reflux) focused on extraction time, solvent consumption, extraction yields, and reproducibility. This green technology has applied in the saponins extraction using methanol as solvent.<sup>[44]</sup> A form of PLE is the technique named Accelerated Solvent Extraction (ASE).

#### 3.2.3.3.1. Accelerated Solvent Extraction (ASE)

This green technique (non-convective) is an efficient form of liquid solvent extraction that uses a small amount of solvent (cyclohexane, methanol, and water) to get a high extraction yield in comparison with maceration and Soxhlet evaporation.<sup>[36]</sup> Concerning the yield of extraction, to get certain extraction yield the sonication (described below) time or reflux time required is 30 min and 1 hour, respectively. The needed extraction time of ASE is 7 min getting the same yield results of the extracted components.<sup>[45]</sup>

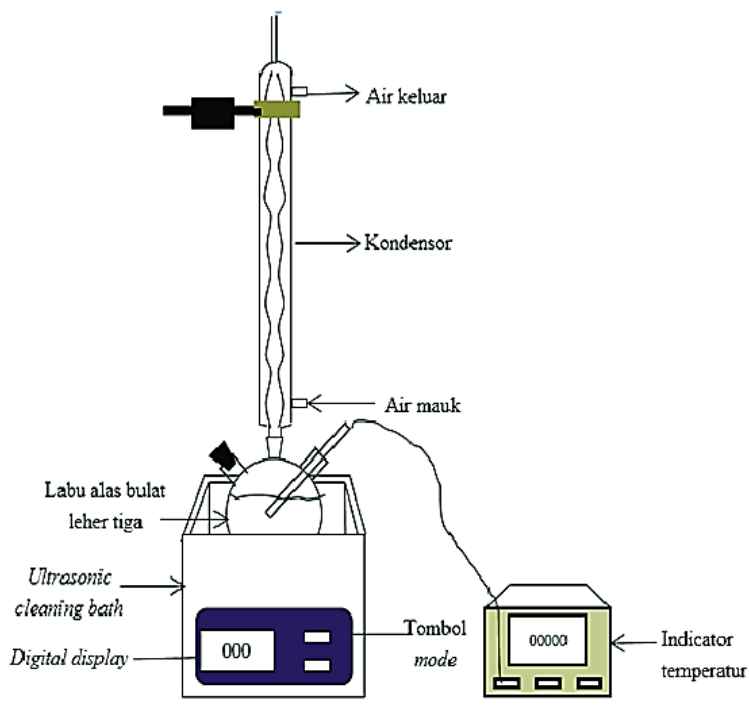
A clear advantage of ASE is the control of temperature and pressure of the process (*Figure 30*) favoring the solubility and mass transfer during the extraction process of the compound of interest.



*Figure 30:* Accelerated solvent extraction (ASE) system.<sup>[46]</sup>

### 3.2.3.5. Ultrasound-Assisted Extraction (UAE)

UAE or sonication (*Figure 31*) is a method that applies the mechanic effect of cavitation, which disrupts the wall cell plants by the bubbles collapse to extract the compound of interest from matrices.<sup>[33]</sup>

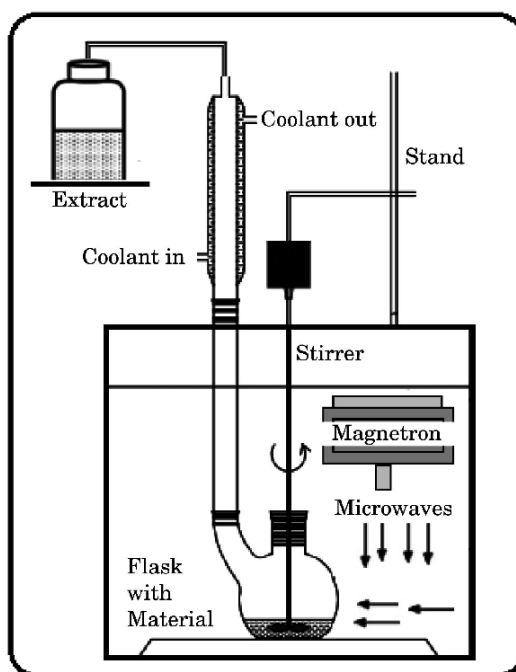


*Figure 31:* Ultrasonic assisted extraction (UAE) system.<sup>[47]</sup>

The main advantages of this technique are the significant reduction of extraction time, temperature, energy, and solvent consumption. Between UAE and solvent extraction, the first has a better efficiency by the benefits of time-saving and high yields comparing with solvent extraction and low energy consumption. Furthermore, this technology is easy to apply in small and large scale, less waste production, and low equipment investment.<sup>[36]</sup> However, its application in saponin extraction is less common than maceration or Reflux.

### 3.2.3.6. Microwave-Assisted Extraction (MAE)

MAE is a non-conventional technique of extraction that has many advantages found in previous literature such as: less solvent used amount, short time extraction in comparison with solvent extraction, higher extraction rate, less energy consumption, fewer wastes, and products with lower cost.<sup>[49]</sup> The less time to extraction is due to the big difference in heating performance between conventional heating and the heating used by the microwave technique. To carry out the typical heating it is necessary to have a finite time; for that, the heat circulates through a vessel to the solution while MAE heats it directly (*Figure 32*).

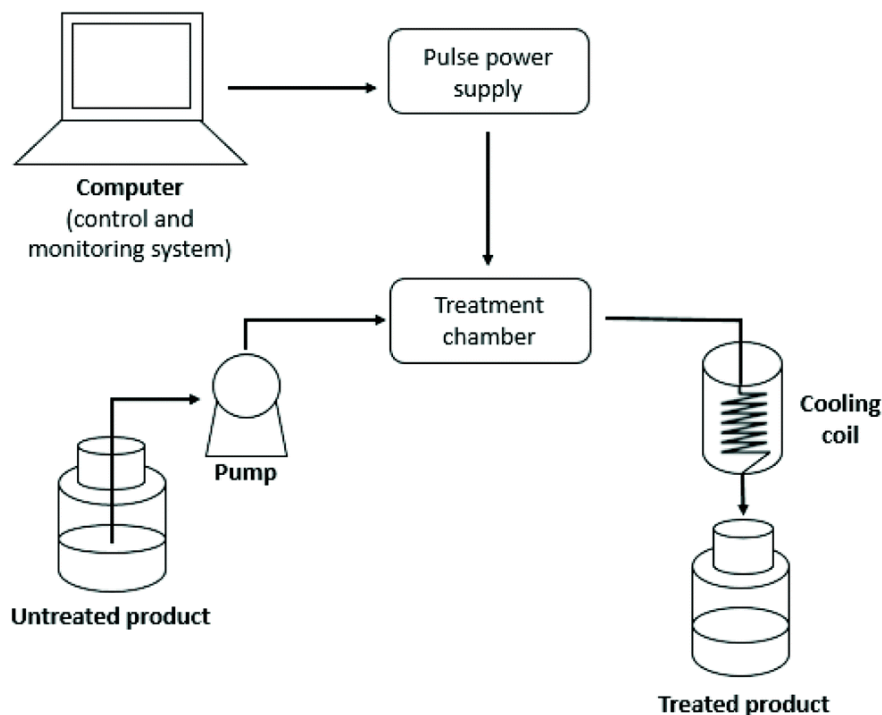


*Figure 32:* Microwave-assisted extraction (MAE) system.<sup>[48]</sup>

On the other hand, MAE uses less solvent volume because it enables quick dissolution, acidic digestion, drying, and extraction of organic compounds from complex matrices.<sup>[50]</sup>

MAE is a good option and ally in the extraction of phytochemical compounds such as flavonoids, saponins, and anthocyanidins.<sup>[51]</sup>

### 3.2.3.7. Pulsed Electric Field (PEF) extraction



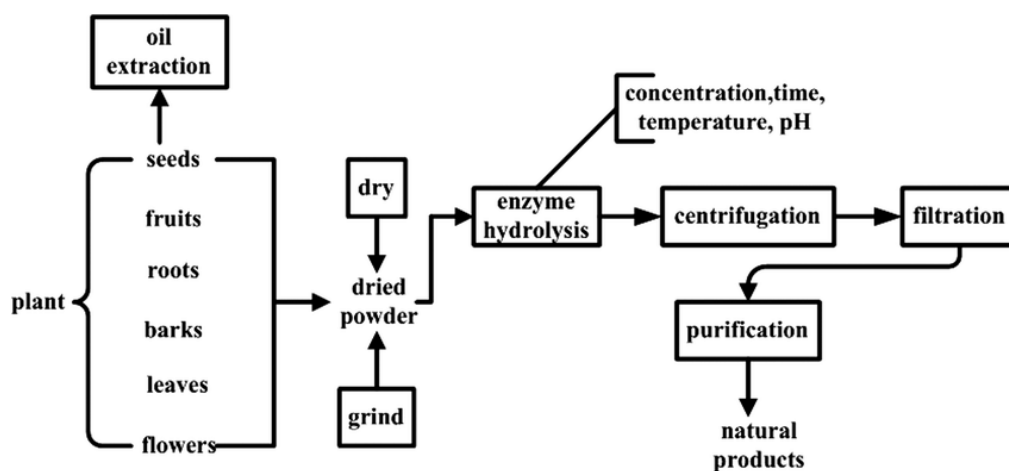
**Figure 33:** Pulsed Electric Field Extraction (PFE).<sup>[52]</sup>

This green technique (*Figure 33*) implies the application of electric pulses to destroy the cell membrane structure to increase the extraction yield; because with the membrane rupture, it is possible the extraction through diffusion since the phenomenon of electroporation helps increase the permeability of the cell.<sup>[53]</sup>

The PEF is a non-thermal technique that does not degrade the thermolabile compounds present in the plants or resources used as raw material to extract determined compounds. Moreover, it leads to an improved extraction yield, decreased extraction time, facilitation of the purified extract, and the reduction of energy cost and environmental impacts.<sup>[54]</sup>

### 3.2.3.8. Enzyme Assisted Extraction (EAE)

The application of EAE works in the extraction process (*Figure 34*), where the challenge is the extraction of compounds using solvents; this technique is an eco-friendly extraction technology that shows a high yield extraction.



**Figure 34:** Process for enzyme-assisted extraction method from the natural products.<sup>[55]</sup>

The hydrolytic action of the enzymes that work on the compounds increases the yield of extraction, facilitating the release of the natural products from the cell.<sup>[41]</sup> But the application of this technique is expensive due to the cost of the enzymes used.



**Table 6:** *Extraction methods for natural products.*

Method	Solvent properties	Volume of organic solvent	Temperature	Pressure	Time	Investment	Yield
Maceration	Water, aqueous and non-aqueous solvents	Large	Room temperature	Atmospheric	Long (weeks)	Low	Low
Percolation	Water, aqueous and non-aqueous solvents	Large	Room temperature or under of that.	Atmospheric	Long (weeks)	Low	Medium
Soxhlet extraction	Water, aqueous and non-aqueous solvents	Moderate	Under heat	Atmospheric	Moderate (weeks)	Moderate	Medium
Reflux extraction	Water, aqueous and non-aqueous solvents	Moderate	Under heat	Atmospheric	Moderate (days)	Moderate	Medium
Supercritical fluid extraction	Supercritical fluid (S – CO <sub>2</sub> sometimes with a modifier)	None or minimal	Near room temperature	High	Long (hours)	High	Low
Accelerated Solvent Extraction	Water, aqueous and non-aqueous solvents	Small	Under heat	High	Short (hours)	High	Medium-High
Ultrasound-assisted extraction	Water, aqueous and non-aqueous solvents	Moderate	Room temperature or under heat	Atmospheric	Short (minutes)	High	High
Microwave-assisted extraction	Water, aqueous and non-aqueous solvents	Moderate	Room temperature	Atmospheric	Short (minutes)	Moderate	High

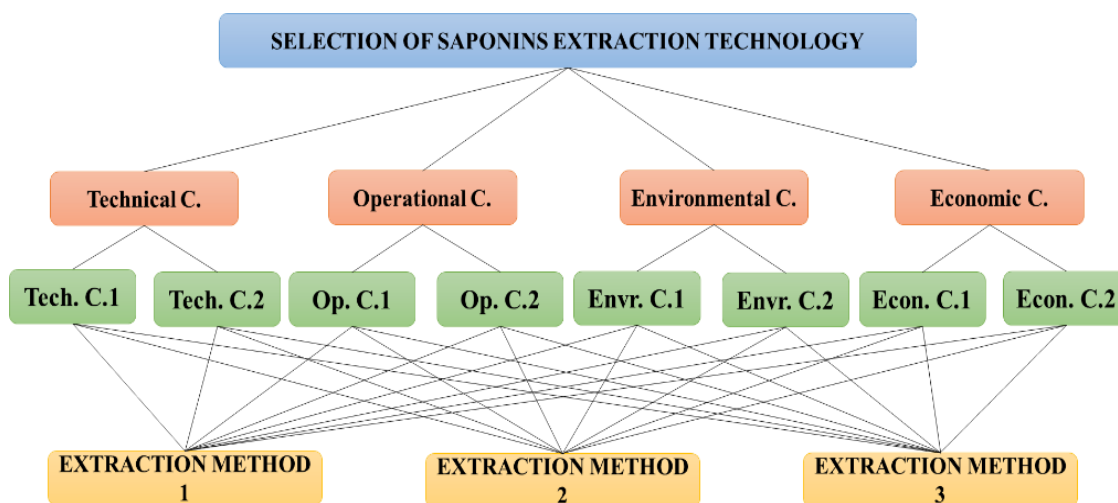
## CHAPTER IV

### 4. METHODOLOGICAL PROPOSAL TO SELECT AN EXTRACTION TECHNOLOGY

#### 4.1. Theoretical fundamentals

For the selection of an extraction method among those mentioned above (Chapter III) which is suitable for the extraction of saponins, it is necessary to apply a technique called Analytical Hierarchy Process (AHP). This technique was developed by the mathematician Tomas L. Saaty during the 1970s to solve the strategic arms reduction treaty between the United States and the USSR.

The AHP is a flexible system of discrete methodology for multicriteria decision analysis, that is, the study of a finite number of alternatives or choice options to be able to achieve an objective or goal;<sup>[56]</sup> Fig. 35 is the schematic representation of the final goal, criteria, and alternatives for selecting a technology for saponins extraction. One significant advantage of this technique is its reproducibility, that means that the AHP procedure is replicable in the scientific, political, and environmental field at the moment to choose an alternative to reach a goal.



*Figure 35:* Hierarchy of extraction technology selection.

The strategy for preparing this proposal must take into account some considerations such as:

- Analyze extraction techniques that are consistent in the extraction of saponins from natural resources with bibliographic information available to the public.
- Determine some technical, operational, economic, and environmental criteria.

#### 4.2. AHP methodology

After criteria and sub-criteria selection continue the weight assignation to each one of them depending on its importance following the methodology of Analytical Hierarchy Process (AHP), the values used in the weight assignation are the reflected ones in the table elaborated by Saaty called “Saaty scale” (Table 7). The Saaty scale considers the intermediate values and the reciprocal of each one of them.

**Table 7:** Saaty scale.

Value	Definition	Description
1	Equal importance	Two activities contribute equally to the objective.
3	Moderate importance	Experience and judgment favor one action over another.
5	Great importance	Experience and judgment strongly favor one action over another.
7	Very great importance	An activity that is energetically favored and its mastery demonstrated in practice.
9	Extreme importance	The greater importance of one activity over others is irrefutable.
2, 4, 6, 8	Intermediate values between two adjacent sentences	When commitment is necessary.
Reciprocal	Values placed to preserve consistency.	
To express reciprocity has uses the inverses of these values.		

**Source:** Own elaboration based on Saaty (1990:15)

The process to apply the AHP technique is the next one:

1. Elaboration of a pairwise comparison matrix called A of nxn dimensions each criterion taking into account the next considerations:

- Each element of the matrix has represented as  $a_{ij}$  where i represents the rows and j the columns.
- The elements  $a_{ij}$  where  $i=j$  is equal to 1.

$$A = \begin{bmatrix} 1 & a_{12} & \cdots & a_{1n} \\ a_{21} & 1 & \cdots & \vdots \\ \vdots & \cdots & 1 & \vdots \\ a_{n1} & \cdots & \cdots & 1 \end{bmatrix}$$

- The product between  $a_{ij} * a_{ji} = 1$  that is to say  $a_{ji} = 1/a_{ij}$ .

$$A = \begin{bmatrix} 1 & a_{12} & \cdots & a_{ij} \\ 1/a_{12} & 1 & \cdots & \vdots \\ \vdots & \cdots & 1 & \vdots \\ a_{ij} & \cdots & \cdots & 1 \end{bmatrix}$$

2. Calculate the priorities of each criterion and alternative compared.
  - For values assignation of the Saaty scale it is necessary to take into account the importance that represents a criterion of the column over the row criteria, considering that 1 represents equal importance and the number 9 an extreme importance.
  - Calculate the sum of each one column of the pairwise comparison matrix.
  - Divide each value in the matrix by the resulting sum of each column corresponding value, generating the normalized pairwise comparison matrix (N).
3. Calculate the average of the normalized vector dividing the sum of each row from the normalized matrix (N) by the number of parameters analyzed in the pairwise comparison matrix (n).
4. Check the consistency of the pairwise comparison matrix to know its reliability.
  - To calculate a priority vector applying the multiplicity theory that consists of the product between the pairwise comparison matrix and the average vector.
  - Calculate a new vector dividing the priority vector obtained in the last step by using the average vector for each technology analyzed.
  - Calculate the largest eigenvalue ( $\lambda_{max}$ ), calculating the average of the value of each technology in the new vector.
  - Find the consistency index using the next equation:

$$CI = \frac{\lambda_{max} - n}{n - 1} \text{ Equation 1}$$

n refers to the number of elements studied in the pairwise comparison matrix.

- Calculate the consistency ratio (CR) dividing the consistency index (CI) by the random consistency index (RI):

$$CR = \frac{CI}{RI} \text{ Equation 2}$$

Taking account that RI corresponds to the consistency index of a random pairwise comparison matrix. The values of RI are registered in Table 8 or calculated using equation 3.

**Table 8:** *Random consistency index (R.I).*

<b>n</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>	<b>10</b>
<b>R.I.</b>	0	0	0,58	0,90	1,12	1,24	1,32	1,41	1,45	1,49

$$RI = \frac{1,98(n - 2)}{n} \text{ Equation 3}$$

Once calculated the correspondent value of consistency ratio (CR) it is necessary to identify if the value is higher or lower than 0,1 (10%). If the value is lower than 0,1 it means that the pairwise comparison matrix is consistent, whereby the values considered on the criteria for the matrix analyses are acceptable. However, if the value of CR is higher than 0,1 it represents an inconsistency in the pairwise comparison matrix by which it is a necessary to repeat the procedure with a new judgment on the values assignment.

To calculate the consistency ratio of the different matrices is necessary to use the table of random consistency index (Table 8), taking into account the variables number analyzed in the study; or applying *Equation 3*, where *n* is the number of variables.

### 4.3. Methodology to define the extraction process

#### 4.3.1. Selection criteria

The present document analyzes many methods of extraction, very necessary to choose one of them, taking into account the specific criteria such as energy, cost, accessibility to the technology and solvent, efficiency, emissions, and solvent quantity. Each of these parameters are part of four different general criteria: technical, operational, economic, and environmental.

**4.3.1.1. Technical criteria (TC):** Refers to the aspects related to the functioning of the extraction technologies.

*Technology maturity:* This criterion takes into account the constancy the application of the method for extraction of chemical compounds from natural products.

*Yield:* The yield consists of how many mg of saponins and sapogenins are obtained from 100 mg of raw material. This parameter can vary depending on the extraction method.

In this case, for saponins extraction yield of the extraction methods is considered the bibliographic resources related to it; however, for determining precise information is necessary to subject the final product to analytical characterization techniques (See Appendix C).

*Extraction (Operation) conditions:* Temperature and pressure required to carry out an extraction process.

*Extraction time:* Time necessary to extract the maximum sapogenin or saponin quantity from natural resources or vegetal material.

*The volume of organic solvent:* The most common solvents used for the extraction of saponins or sapogenins compounds are ethanol and methanol. For that reason, the Analytic Hierarchy Process technique considers the volume of solvent to carry out the extraction methods.

**4.3.1.2. Operational criteria (OC):** refers to the operational aspects of the extraction technologies.

*Solvent accessibility:* The company's accessibility to acquire the solvent depends on the extraction method, either for the convenience of acquisition or for being a product or by-product from activities carried out by the company.

*Complexity:* The complexity of the extraction methods depends on the independence of their operation and maintenance, besides to assign the value on this sub-criterion, it takes into account the experience of the company implementing each extraction method.

*Technical support:* Technical support refers to the technical assistance provided by the suppliers for each extraction method (equipment).

**4.3.1.3. Environmental criteria (EnC):** are those impacts that can be generated because of the extraction technologies to the environment (water, air, and soil).

*Solvent Emission:* Environmental damage due to emissions caused by extraction technologies, these emissions can change depending on the type and amount of solvent used in the extraction.

*Organic waste management:* Refers to the need of treating *Agave* residues after the extraction of sapogenins or saponins.

*Wastewater management:* It refers to the required treatment for wastewater after the extraction process.

**4.3.1.4. Economic criteria (EC):** referring to the economic impact due to the extraction technology.

*Investment cost:* Refers to the value in the acquisition of the equipment to use in the extraction process.

*Operation cost:* It is associated with costs of maintenance in extraction equipment.

*Maintenance cost:* It refers to the economic values related to the operating costs of the equipment for each extraction methods.

To assign the weights or percentage to each criterion and its respective sub-criterion to analyze the present research, the methodology to follow is the Analytic Hierarchy Process (AHP). That consists of the design of pairwise comparison matrices, starting with the selection of the range of valorization (Table 9) with its respective reciprocal and intermediate values (2, 4) used to determine the relative weight for design the technology selection matrix from Saaty scale.

The tool used to apply the mathematical calculus is “Excel 2019” to obtain the final percentage for each one criterion, sub-criterion, and extraction technologies.

**Table 9:** *Selected Saaty scale.*

Value	Definition
1	Equal importance
3	Moderate importance
5	Great importance

The first pairwise comparison matrix compares the priority of the qualitative or quantitative criteria (TC, OC, EnC, and EC), defining their relative importance taking into consideration the goal and bibliographic resource related to the extraction methods. The same principles are an essential factor to elaborate on the matrices for sub-criterion and extraction technologies.

The technology’s matrix analyzes nine conventional extraction methods for saponins obtaining. Those technologies are maceration, percolation, Soxhlet, reflux, supercritical fluid extraction, accelerated solvent extraction, ultrasound, and microwave extraction.

Methods such as enzyme assisted and pulse electric field extraction are not part of the analysis due to their low technology maturity, hence the reason these methods do not have representative information focused on the saponins extraction.



## CHAPTER V

### 5. RESULTS AND DISCUSSION

#### 5.1. Criteria and sub-criteria weight

The selection of extraction technology from the final development of AHP methodology is the one with the highest weight in each criterion and sub-criterion. It is necessary to emphasize that the considered criteria to assign the numerical values on the pairwise comparison matrix were from the engineering point of view since weight assignment is considered from the cost and yield perspective.

Table 10 represents the final percentage of the criteria analyzed in the matrix (Technical, Operational, Environmental, and Economic), in this table economic criterion is the most representative with a higher percentage compared with the rest. However, that value do not diminish the importance of the others; the reason by which the economic criterion has that percentage is due to the point of view implemented.

**Table 10:** *Global criteria weight.*

Criteria	Percentage (%)
Technical criterion	17
Operational criterion	19
Environment criterion	14
Economic criterion	50

After the analyzes on the criteria it is necessary to carry on new analyzes focused on the sub-criteria relating them with the extraction techniques generating a valorization of each one of the extraction methods. Table 11 shows the weights assignment of parameters (sub-criteria) that are part of technical criterion. The yield and technology maturity have a higher value because of their relevance to select an adequate extraction method. But between the extraction time and the volume of the organic solvent parameters, there is a minimal difference in the final weight, this reflects that both sub-criteria are of similar importance. Therefore, temperature and pressure have equal percentages (lower than the rest) because although these sub-criteria are relevant when choosing the technology, they should not be the pillars to make the final decision.

**Table 11:** *Technical criteria weight.*

Technical sub-criteria	Percentage (%)
Technology maturity	22
Yield	35
Temperature	7
Pressure	7
Extraction time	13
The volume of organic solvent	15

Table 12 reflects the values of the weights for each of the operational sub-criteria. These values show that the accessibility to the solvent is the parameter with more importance due to the current productive activity developed by the company. The operational complexity is the parameter with a lower percentage than accessibility to the solvent since all the extraction technologies are relatively new for the application in the company's production process. Technical support is the parameter with the lowest value compared to the previous ones, although the selection process represents a fundamental parameter when carrying out that technology in the company.

**Table 12:** Operational criteria weight.

Operational sub-criteria	Percentage (%)
Accessibility to the solvent	41
Complexity	33
Technical support	26

Concerning the results obtained for the parameters belonging to the environmental criteria (Table 13), the treatment on the organic residues produced by the process represents the highest value, since the plant matter is in contact with the solvent directly whereby it requires a better treatment to use it as a by-product for example compost. The parameter of the solvent emissions produced has a lower value than the waste management. Least, the treatment required on the water used in the extraction process is the parameter with the lowest weight because the dominant participation of this resource is in washing (removal of impurities), so its treatment is not highly arduous.

**Table 13:** *Environmental criteria weight.*

Environmental sub-criteria	Percentage (%)
Solvent emission	39
Organic waste management	44
Management of water	17

Finally, in the assignment of weights to the economic sub-criteria, among the resulting percentages, it can be seen that the parameter with the most significant proportion is the investment required in the acquisition of extraction equipment. The importance of this parameter lies in the fact that it is essential to know how economically risky it would be to apply the selected technique from an engineering point of view. Unlike investment, the operation and maintenance costs shown in Table 14 have a lower percentage, but operation costs are parameters with a weight value pretty near the obtained value for investment since it is necessary to know how expensive it is to start an extraction technology.

For values assignment in the pairwise comparison matrix, a range of high, medium, and low is considered, taking into account that for the low cost, it is less than 100,000 dollars and high greater than 200,000 dollars.

**Table 14:** *Economic criteria weight.*

Economic sub-criteria	Percentage (%)
Investment cost	44
Operation cost	39
Maintenance cost	17

## 5.2. Extraction methods weight

Figures 36 to 42 are the graphical representations of different sub-criteria on the extraction methods weight (See Appendix G) classified in two groups conventional (maceration, percolation, Soxhlet, and Reflux) and non-conventional (supercritical fluid, accelerated solvent, ultrasound-assisted, and microwave-assisted extraction) methods.

The bars of the final weights of conventional methods are represented with green color, while for non-conventional, the weights are the yellow bars. The comparison for choosing

the extraction technology takes into account the several considerations of each of the sub-criterion on which the study focuses

### 5.2.1. Technology maturity

Figure 36 reflects each extraction method's technological maturity, evincing that the maceration is the most constant method on the extraction field. But microwave and ultrasound-assisted extractions are most applicable to extract saponins or sapogenins concerning non-conventional methods.

Although the maceration is the method most used in the interest compound extraction, it is not recommendable by the high tendency of sample contamination; nonetheless, this method is a good option as a pre-treatment on the material to apply before extraction.

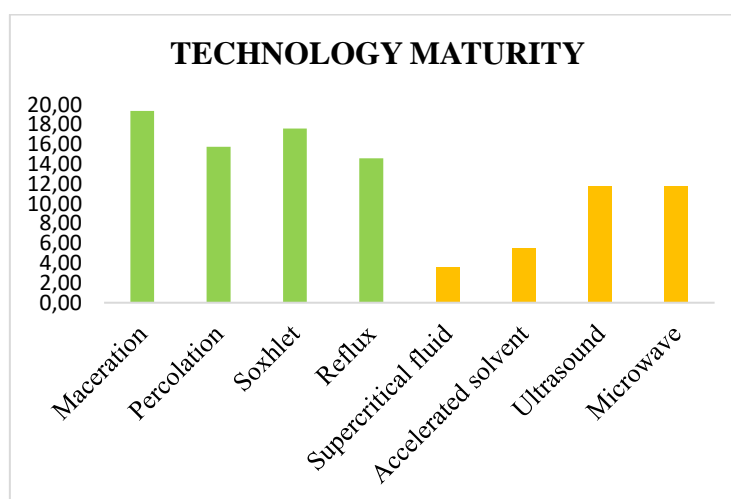
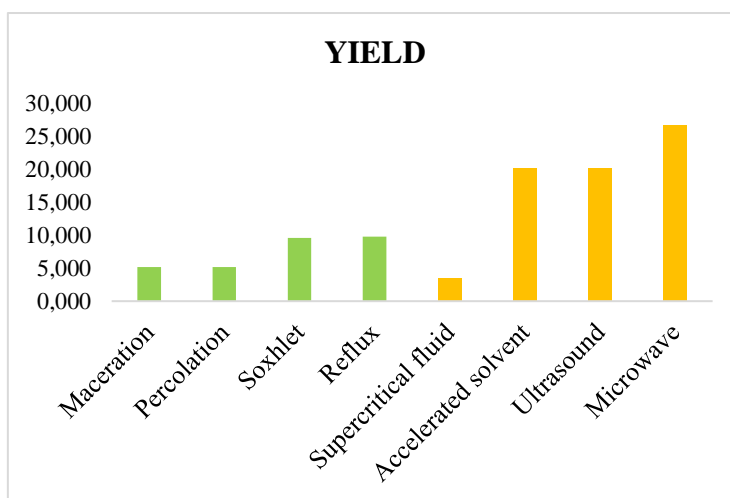


Figure 36: Graphical for technology maturity weight.

### 5.2.2. Yield of extraction

Extraction yield is one of the parameters with crucial importance at the time to choose the adequate extraction method. Between all the extraction methods (Figure 37), highest yield corresponds to the non-conventional methods except for SFE.

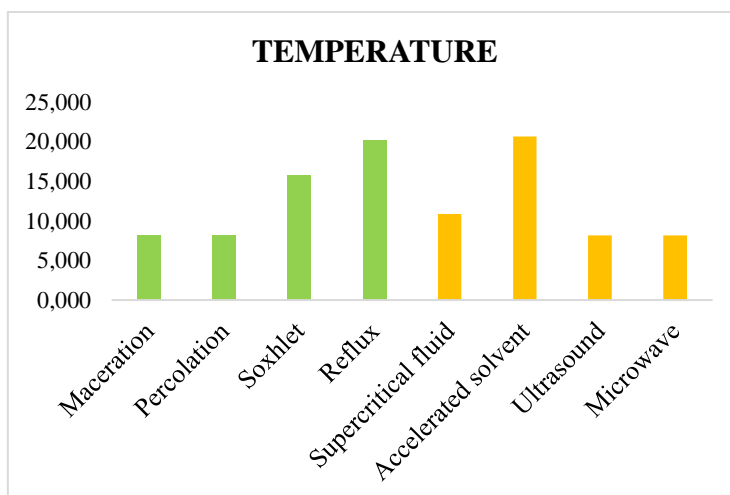
Extraction methods such as Soxhlet and Reflux are the most representative of this parameter for conventional methods, but that advantage has a contradiction since these techniques require a supply of energy to obtain a better yield, which would cause an increment on the energy costs that are part of the extraction process.



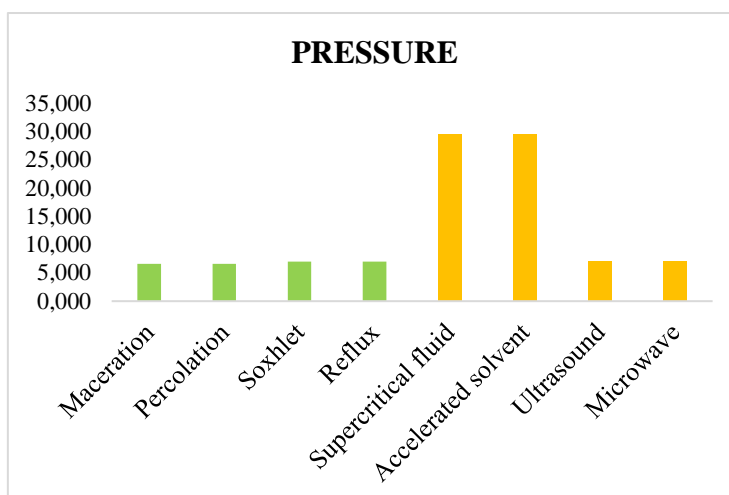
**Figure 37:** Graphical representation of yield weight.

### 5.2.3. Temperature and pressure

The consideration to take into account when selecting the extraction technique for temperature and pressure is one that requires supplying less heat and pressure (*Figures 38 and 39*). Concerning the conventional methods, maceration and percolation need less temperature than reflux and Soxhlet. Nevertheless, the non-conventional technique called microwave-assisted and ultrasound-assisted extraction have a small percentage weight indicating that this requires a lower temperature than the other.



**Figure 38:** Temperature weight.

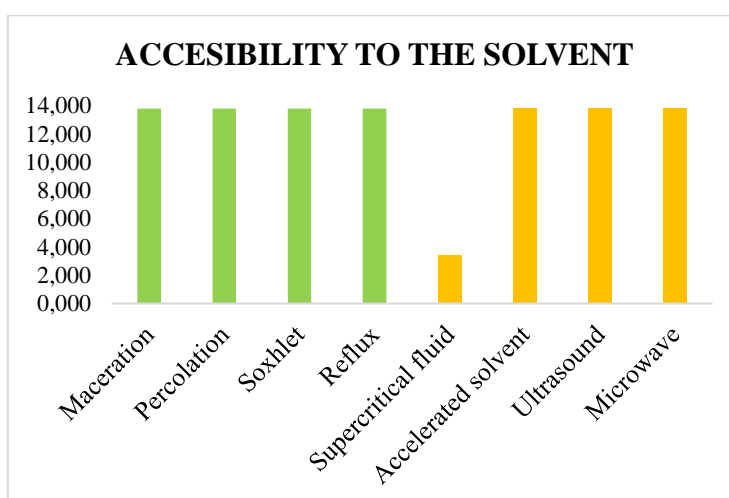


**Figure 39:** Pressure weight.

The extraction methods that do not need additional pressure are all conventional techniques, ultrasound-assisted, and microwave-assisted extraction; this is because these techniques work at atmospheric pressure.

#### 5.2.4. Extraction time

The final time necessary to achieve the maximum interest compound is less for Reflux and Soxhlet in conventional methods (*Figure 40*). While ultrasound and microwave-assisted are the technologies with the requirement of minor time that the other, it should be emphasized that although the supercritical fluid extraction requires a little time to extract some active compounds in the case of saponins or sapogenins needs much time.



**Figure 40:** Graphical representation of extraction time weight.

### 5.2.5. Accessibility to the solvent and volume of organic solvent

Figure 41 shows the final weight percentage on parameters associated with the solvent used to extract saponins; the first parameter is the accessibility to the dissolvent demanded extraction. Almost all the extraction techniques require ethanol as a solvent, by which just the supercritical fluid is the technique with less percentage; that is why, the company's actual productive activity is related to ethanol production.

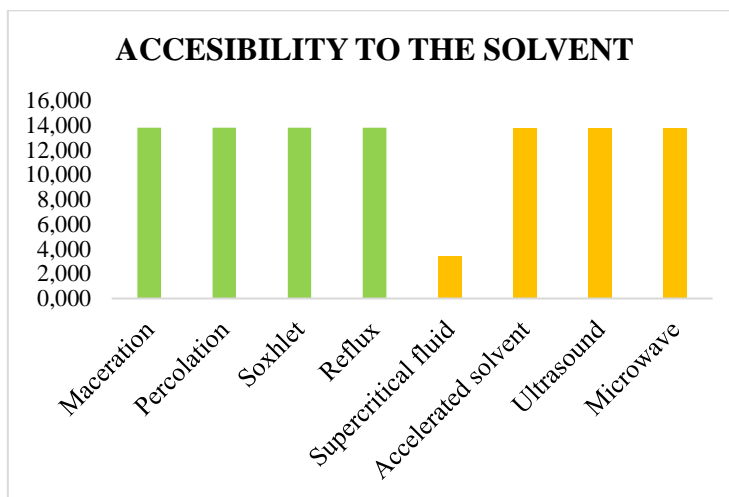


Figure 41: Accessibility to the solvent weight.

Another parameter embodied in Fig.42 is the volume of organic dissolvent, showing that the methods that require a lower amount of organic dissolvent are reflux, Soxhlet, supercritical fluid, and accelerated solvent. The last three techniques are directly associated with their dependency on the pressure.

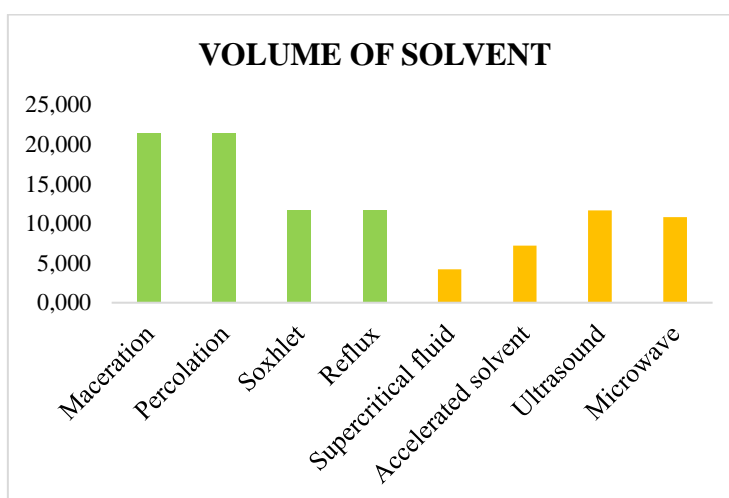


Figure 42: Volume of organic solvent weight.

### 5.2.6. Complexity

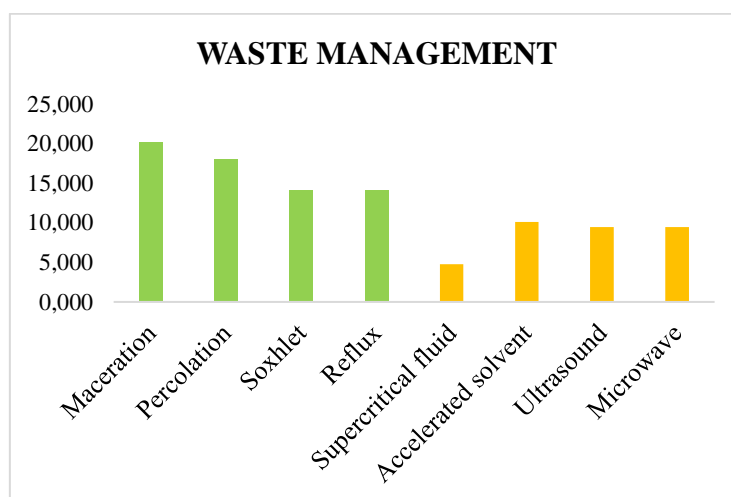
The operational complexity in the proposed extraction techniques considers that the conventional methods (maceration, percolation, Soxhlet, and reflux) result from less operational complexity as they have been widely applied in the field of saponin extraction. On the contrary, the non-conventional techniques (SFE, PLE, ASE, UAE, and MAE) have been categorized as methods of greater complexity due to their limited use in extraction processes.

To apply a saponin extraction process at the company of interest one had considered the same operational complexity in all of the extraction techniques since these are new for the company.

### 5.2.7. Technical support

The analysis focused on technical support is unnecessary since the technical support had offered by the technology providers, by which the study does not consider the application of AHP methodology for that parameter. However, in this case, it has been considered that the providers would give competent (excellent) technical support for the technologies.

### 5.2.8. Waste management



*Figure 43:* Waste management weight.

Concerning wastes produced during the extraction process, for its management they are divided into organic waste and wastewater. Both waste management takes into account

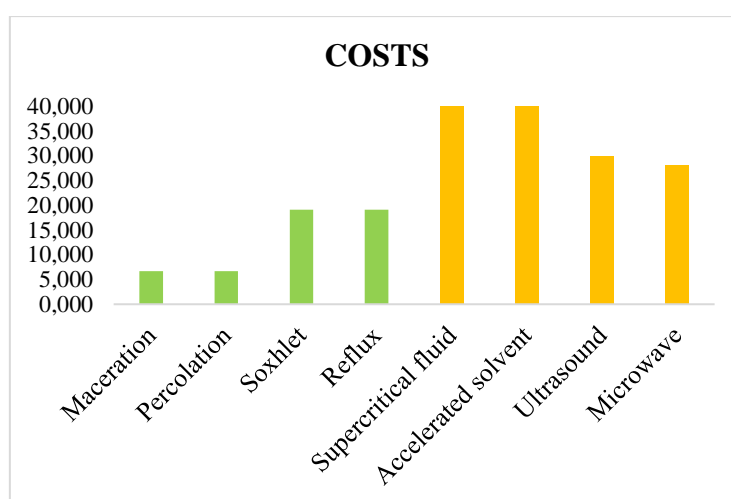


the amount (volume) of solvents and chemical products that are part of the process and the purpose that the residual material would have, whether it will be the recycling or its elimination.

Fig. 43 is a graphical representation of the organic residues as a whole; in the figure, one can see that applying conventional techniques implies handling waste, as opposed to the non-conventional methods by not using a high amount of solvent during extraction requires less waste management. For wastewater management is considered the treatment over the residues after pencas washing, whereby the complexity of this depends on the impurities (insects, land residues, and others) present in the pencas.

### 5.2.9. Costs (Investment, operation, and maintenance)

Fig. 44 is the representation concerning the cost the analyses encompass the investment, operation, and maintenance costs. The technologies with less cost than the rests are maceration and percolation, while between the non-conventional methods, the microwave-assisted extraction is the technique that requires less cost.



*Figure 44:* Graphical representation of costs weight.

## 5.3. Technique extraction selection

### 5.3.1. Preliminary selection

Continuously knowing the weight of the criteria, sub-criteria, and technologies, and by using that information is possible to deduce in a superficial form the adequate method to

extract saponins from *Agave americana* L. It is necessary take into account that the selected method should accomplish the next specifications:

- A technique common in the compound's extraction field (high technology maturity)
- The technology that allow to obtain a high yield for interest compound extraction.
- Extraction method requires a minor supply of heat and pressure to carry out.
- The required solvent should be accessible and in small quantity for the extraction.
- Moderate or low operational complexity of the extraction system.
- The waste produced along the extraction process needs the application of minimal treatment to give it other use.
- Minimal inversion to cover the inversion and costs involved in the operation of the technology.

The most appropriate extraction methods are maceration, reflux, ultrasound-assisted, and microwave-assisted extraction. Reflux extraction has the disadvantage that requires the modification of its temperature to reach a good yield, considering the conditions mentioned above.

### **5.3.2. Criteria valorization**

However, to know which extraction method is the most adequate, it is crucial to implement a mathematical analysis on the selected extraction methods; therefore, a valorization is designed for each of the parameters analyzed in the pairwise comparison matrix. For the elaboration of table 15, the values taken into account are 1,2, and 3 being the number 3 of more importance.

**Table 15:** *Criteria and sub-criteria valorization.*

Parameters	Valorization scale		
	1	2	3
<b>Technical criteria</b>			
Technology maturity	Low	Medium	High
Yield	Low	Medium	High
Temperature	Under heat	Medium	Room temper.
Pressure	High		Atmospheric
Extraction time	Long	Moderate	Short
Volume of organic solvent	Large	Moderate	Small
<b>Operational criteria</b>			
Accessibility to the solvent	No		Yes
Complexity	High	Medium	Low
Technical support	No		Yes
<b>Environmental criteria</b>			
Solvent emissions	High		Low
Organic waste management	High		Low
Management of water	High		Low
<b>Economic criteria</b>			
Investment costs	Greater than \$200000	\$100000 - \$200000	Less than \$100000
Operational costs	Greater than \$10000	\$5000 - \$10000	Less than \$5000
Maintenance costs	Greater than \$10000	\$5000 - \$10000	Less than \$5000

After evaluating the different parameters, it is necessary to design a matrix to evaluate these parameters using the information shown in the table of modified extraction methods for natural products (Table 6) and the weights obtained for each of the criteria and sub-criteria. The procedure to know the final score of the extraction methods consists of the following steps:

- To calculate a product between the weight of each sub-criterion with its respective value, adding in table 15.
- To calculate the score of each criterion (technical, operational, environmental, and economic) by multiplying the value of the sum of the products previously calculated that corresponds to the sub-criteria and the weight obtained through the AHP methodology.

- The final score for each extraction method is the total sum of the criteria's scores.

Table 16 is the result of the mathematical analysis on the preselected extraction technologies; these objects indicate that the most adequate methods are maceration (conventional technique) and microwave-assisted extraction (non-conventional technique), but to take a final decision it is necessary a study focused on the point of view of the company.

**Table 16:** *Evaluation to select the saponin extraction method.*

Parameters	Weight	Maceration		Reflux		Ultrasound-assisted extraction		Microwave-assisted extraction	
		Score	Total	Score	Total	Score	Total	Score	Total
<b>Technical criteria</b>	<b>17</b>	<b>1,7</b>	<b>30</b>	<b>2,2</b>	<b>39</b>	<b>2,6</b>	<b>45</b>	<b>2,6</b>	<b>46</b>
Technology maturity	22	3		3		2		2	
Yield	35	1		2		3		3	
Temperature	7	3		1		2		3	
Pressure	7	3		3		3		3	
Extraction time	13	1		2		3		3	
Volume of organic solvent	15	1		2		2		2	
<b>Operational criteria</b>	<b>19</b>	<b>3</b>	<b>57</b>	<b>2,7</b>	<b>51</b>	<b>2,7</b>	<b>51</b>	<b>2,7</b>	<b>51</b>
Accessibility to the solvent	41	3		3		3		3	
Complexity	33	3		2		2		2	
Technical support	26	3		3		3		3	
<b>Environmental criteria</b>	<b>14</b>	<b>1,3</b>	<b>18</b>	<b>1,8</b>	<b>24</b>	<b>2,2</b>	<b>30</b>	<b>2,6</b>	<b>35</b>
Solvent emissions	39	1		1		2		3	
Organic waste management	44	1		2		2		2	
Management of water	17	3		3		3		3	
<b>Economic criteria</b>	<b>50</b>	<b>2,7</b>	<b>132</b>	<b>2,2</b>	<b>110</b>	<b>1,6</b>	<b>77</b>	<b>2,0</b>	<b>99</b>
Investment costs	44	3		2		1		2	
Operational costs	39	3		3		2		2	
Maintenance costs	17	1		1		2		2	
<b>TOTAL</b>			<b>238</b>		<b>225</b>		<b>203</b>		<b>231</b>

## CHAPTER VI

### 6. CONCLUSIONS AND RECOMMENDATIONS

#### 6.1. Conclusions

With the present investigation, it has been possible to determine that the most favorable raw material for saponins extraction are the leaves of the *Agave americana* Linnaeus plant than using the sap.

Research on a process to extract saponins from the *Agave* would give additional economic value in the plant since the saponins are a product that had a wide application field, especially in the pharmaceutical industries. Therefore, it would be profitable to develop the process in the national industry.

From bibliographic information, it was possible to determine and define the stages of the saponin extraction process, being the following: washing, drying, gridding, extraction, filtration, evaporation, and drying.

Once the stages of the extraction process were identified, the most common extraction methods in saponin extraction were subsequently determined to apply the hierarchical analysis process (AHP) methodology to them.

To apply the AHP methodology it has been specified four main criteria: the technical, operational, environmental, and economic criterion. These criteria encompass several sub-criteria that are essential when deciding on extraction techniques.

After applying the AHP, it has been concluded that the more advantageous extraction techniques are maceration and microwave-assisted extraction, as they are the more representative, fulfilling the criteria and sub-criteria defined throughout the investigation.

#### 6.2. Recommendations

It is recommended to expand the research from an operational, environmental point of view, and considering the application of the large-scale extraction technique. Thus, beginning with the engineering design considering technical and economic calculations, dimensioning, capacity, materials, and others.

To consider an implementation of a pre-treatment on the raw material before carrying out the extraction to increase the number of saponins to be obtained from the *Agave americana* Linnaeus.

To investigate more deeply the feasibility of extracting saponins using the plants' roots to be able to take advantage of this part of it.

It is recommended to verify the results obtained using the AHP methodology through the use of a software designed to achieve this objective. Furthermore, it is advisable to submit this procedure to a sensitivity analysis.

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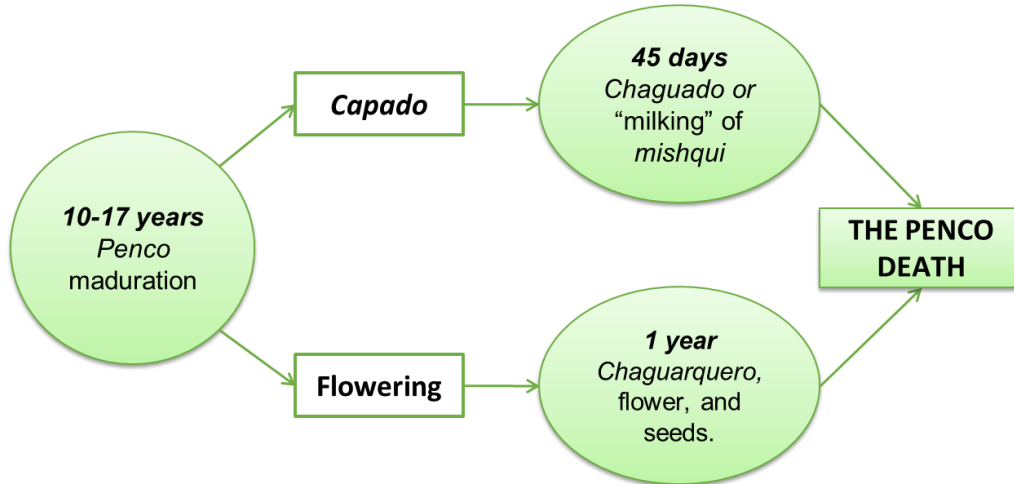
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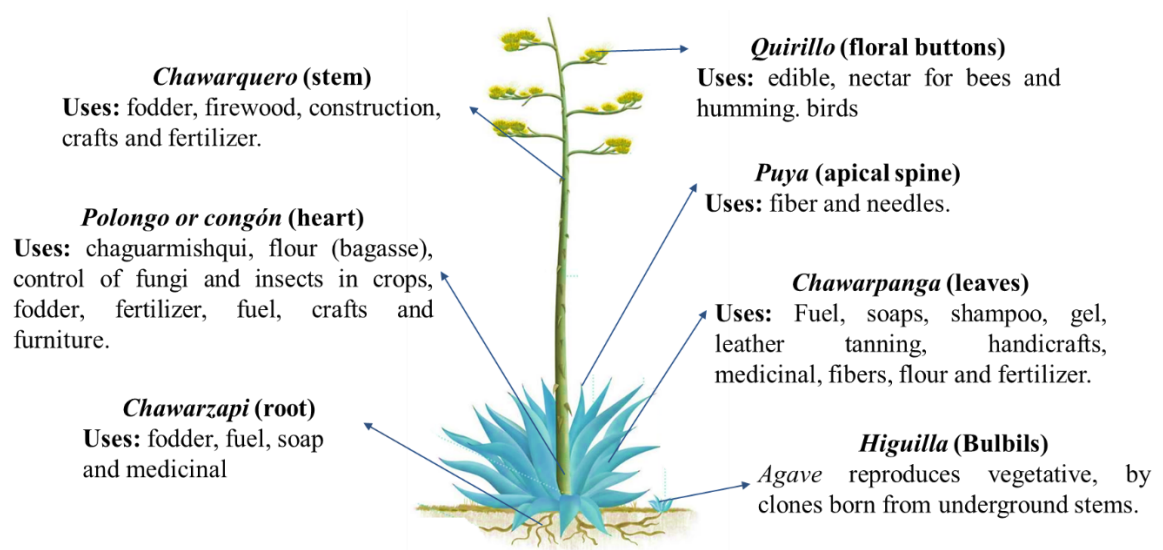
ANNEXES

**Appendix A:** Figures related with *Agave americana* L.

**Appendix A.1:** Life cycle of agave.



**Appendix A.2:** Uses of parts of the “maguey”.



**Appendix B:** Yield of saponins.

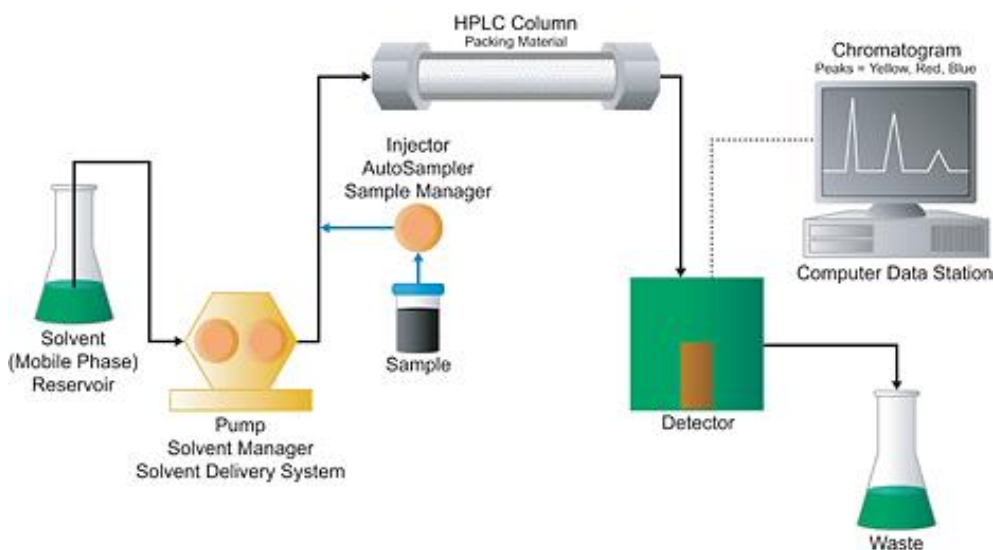
Extraction method	Yield of saponins (%)
Maceration	2,5±0,1% <sup>[57]</sup>
Percolation	Information not available.
Reflux	6,7 mg/100 mg (DM) <sup>[58]</sup>
Soxhlet	7,7 mg/100 mg (DM) <sup>[58]</sup>
Supercritical Fluid extraction.	0,975% <sup>[59]</sup>
Accelerated Solvent Extraction	Information not available.
Ultrasound-assisted extraction	7,6 mg/100 mg (DM) <sup>[58]</sup>
Microwave-assisted extraction	7,4 mg/100 mg (DM) <sup>[58]</sup>

**Appendix C: Analytic technique for saponins determination.**

The techniques most recommended to determine the quantity of saponins are:

**Appendix C.1: High Performance Liquid Chromatography (HPLC)**

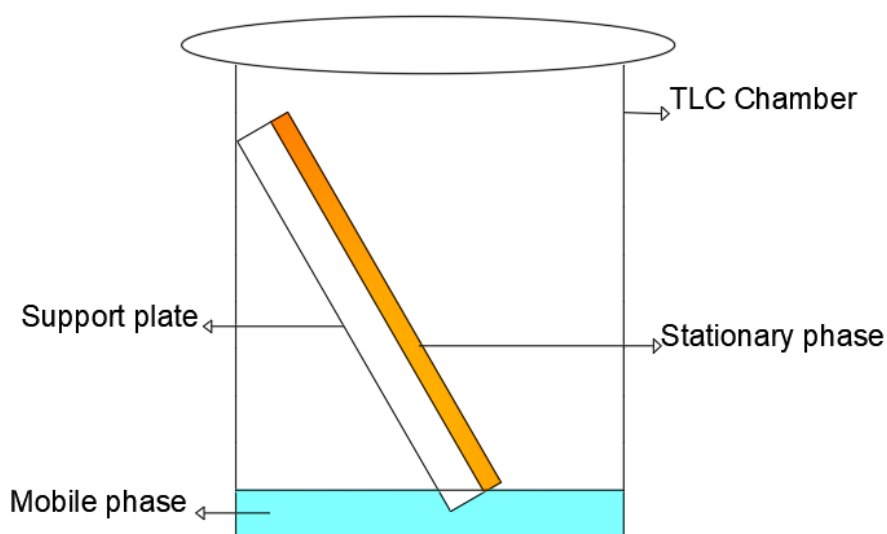
This versatile technique is used for separation, purification, and identification of active compounds present in the plant material.<sup>[60]</sup> HPLC equipment (*Figure C.1*) consists of a solvent reservoir (mobile phase), a pump, an injector, a packed column where is in contact with the sample and solvent, and a detector. For saponins determination using HPLC is implemented the UV (Ultraviolet) detector since the saponin quantity depends on the ultraviolet light absorbed by the compound; generally, the ultraviolet light wavelength to identify saponins selected varies between 200nm to 210nm.<sup>[61]</sup> The principal disadvantage of HPLC is the expensive cost of the equipment.



**Figure C.1:** High Performance Liquid Chromatography (HPLC) system.

### Appendix C.2: Thin Layer Chromatography (TLC)

TLC is a technology used to separate and identify compounds from a mixture. The TLC system consists of a stationary phase (layer of an adsorbent material on a support plate) and a mobile phase (solvent); the mobile phase moves through the stationary phase due to capillary forces.[62] Usually, the support is a plate of plastic, aluminum, or glass, while the adsorbent material is silica gel, cellulose, and alumina. An advantage of the TLC is the inexpensive equipment in comparison with HPLC.



*Figure C.2:* Thin Layer Chromatography (TLC) system.



**Appendix D: Methodology to assign the weight on technical, operational, environmental, and economic criteria of each one extraction method.**

**Table D.1:** Pairwise comparison matrix with the total sum for each one criterion.

	Technical Criteria	Operational Criteria	Environmental Criteria	Economic Criteria
Technical Criteria	1,00	1,00	2,00	0,20
Operational Criteria	1,00	1,00	2,00	0,33
Environmental Criteria	0,50	0,50	1,00	0,50
Economic Criteria	5,00	3,00	2,00	1,00
TOTAL	7,50	5,50	7,00	2,03

**Table D.2:** Normalized pairwise comparison matrix of the general criteria.

	Technical Criteria	Operational Criteria	Environmental Criteria	Economic Criteria
Technical Criteria	0,13	0,18	0,29	0,10
Operational Criteria	0,13	0,18	0,29	0,16
Environmental Criteria	0,07	0,09	0,14	0,25
Economic Criteria	0,67	0,55	0,29	0,49
TOTAL	1,00	1,00	1,00	1,00

**Table D.3:** Average obtained from the normalized pairwise comparison matrix.

Criterion	Average
Technical Criteria	0,1748
Operational Criteria	0,1912
Environmental Criteria	0,1366
Economic Criteria	0,4974

**Table D.4:** Consistency analyze on general criteria.

Four variables are part of this analyses whereby the random consistency index is 0,9; while resultant consistency ratio is  $0,0977 \leq 0,1$  concluding that the values assigned to the general criteria in the pairwise comparison matrix are consistent.

Criterion	Priority Vector	New Vector	$\lambda_{\text{máx}}$	CI	CR
Technical Criteria	0,7386	4,2256	4,2640	0,0880	0,0978
Operational Criteria	0,8050	4,2101			
Environmental Criteria	0,5683	4,1608			
Economic Criteria	2,2182	4,4595			

**Appendix E: Methodological strategy to assign the weight to technical, operational, environmental, and economic sub-criteria.****Table E.1:** Pairwise comparison matrix of technical parameters with the total.

	<b>Technology maturity</b>	<b>Yield</b>	<b>Temperature</b>	<b>Pressure</b>	<b>Extraction time</b>	<b>Volume of organic solvent</b>
<b>Technology maturity</b>	1,00	0,50	3,00	3,00	2,00	2,00
<b>Yield</b>	2,00	1,00	4,00	4,00	3,00	3,00
<b>Temperature</b>	0,33	0,25	1,00	1,00	0,50	0,50
<b>Pressure</b>	0,33	0,25	1,00	1,00	0,50	0,50
<b>Extraction time</b>	0,50	0,33	2,00	2,00	1,00	1,00
<b>Volume of organic solvent</b>	0,50	0,33	2,00	2,00	2,00	1,00
<b>TOTAL</b>	4,67	2,67	13,00	13,00	9,00	8,00

**Table E.2:** Normalized pairwise comparison matrix of technical sub-criteria.

	<b>Technology maturity</b>	<b>Yield</b>	<b>Temperature</b>	<b>Pressure</b>	<b>Extraction time</b>	<b>Volume of organic solvent</b>
<b>Technology maturity</b>	0,21	0,19	0,23	0,23	0,22	0,25
<b>Yield</b>	0,43	0,38	0,31	0,31	0,33	0,38
<b>Temperature</b>	0,07	0,09	0,08	0,08	0,06	0,06
<b>Pressure</b>	0,07	0,09	0,08	0,08	0,06	0,06
<b>Extraction time</b>	0,11	0,13	0,15	0,15	0,11	0,13
<b>Volume of organic solvent</b>	0,11	0,13	0,15	0,15	0,22	0,13
<b>TOTAL</b>	1,00	1,00	1,00	1,00	1,00	1,00

**Table E.3:** Average vector, priority vector, and consistency ratio.

In this case the variable used are six being the random index value equal to 1,24. The analyses is consistent since the consistency ratio obtained is less than or equal ( $\leq$ ) to 0,1.

	Average	Priority Vector	New Vector	$\lambda_{\text{máx}}$	CI	CR
Technology maturity	0,2226	1,3913	6,2504	6,2023	0,0405	0,0326
Yield	0,3545	2,2140	6,2446			
Temperature	0,0728	0,4471	6,1377			
Pressure	0,0728	0,4471	6,1377			
Extraction time	0,1293	0,7980	6,1708			
Volume of organic solvent	0,1478	0,9274	6,2726			

**Table E.4:** Pairwise comparison matrix of operational sub-criteria with its respective total.

	Accessibility to the solvent	Complexity	Technical support
Accessibility to the solvent	1,00	1,00	2,00
Complexity	1,00	1,00	1,00
Technical support	0,50	1,00	1,00
TOTAL	2,50	3,00	4,00

**Table E.5:** Normalized pairwise comparison matrix for environmental sub-criteria.

	Accessibility to the solvent	Complexity	Technical support
Accessibility to the solvent	0,40	0,33	0,50
Complexity	0,40	0,33	0,25
Technical support	0,20	0,33	0,25
TOTAL	1,00	1,00	1,00

**Table E.6:** Average vector, priority vector, and consistency ratio of operational parameters.

The random index to calculate the consistency ratio to be three variables is 0,58; while the consistency ratio is  $0,015 \leq 0,1$ .

	Average	Priority Vector	New Vector	$\lambda_{\text{máx}}$	CI	CR
Accessibility to the solvent	0,4111	1,2611	3,0676	3,0537	0,0268	0,0463
Complexity	0,3278	1,0000	3,0508			
Technical support	0,2611	0,7944	3,0426			

**Table E.7:** Pairwise comparison matrix with the total sum for each one column.

	Solvent emission	Organic waste management	Management of water
Solvent emission	1,00	1,00	2,00
Organic waste management	1,00	1,00	3,00
Management of water	0,50	0,33	1,00
TOTAL	2,50	2,33	6,00

**Table E.8:** Normalized pairwise comparison matrix for environmental parameters.

	Solvent emission	Organic waste management	Management of water
Solvent emission	0,40	0,43	0,33
Organic waste management	0,40	0,43	0,50
Management of water	0,20	0,14	0,17
TOTAL	1,00	1,00	1,00

**Table E.9:** Average and priority vector with the consistency ratio.

The random index to analyze three variables is 0,58; while the consistency ratio is  $0,016 \leq 0,1$ .

	Average	Priority Vector	New Vector	$\lambda_{\text{máx}}$	CI	CR
Solvent emission	0,3873	1,1698	3,0205	3,018	0,0092	0,0158
Organic waste management	0,4429	1,3397	3,0251			
Management of water	0,1698	0,5111	3,0093			

**Table E.10:** Pairwise comparison matrix of economic sub-criteria with its respective total.

	Investment cost	Operation cost	Maintenance cost
Investment cost	1,00	1,00	3,00
Operation cost	1,00	1,00	2,00
Maintenance cost	0,33	0,50	1,00
TOTAL	2,33	2,50	6,00

**Table E.11:** Normalized pairwise comparison matrix of economic criteria.

	Investment cost	Operation cost	Maintenance cost
Investment cost	0,43	0,40	0,50
Operation cost	0,43	0,40	0,33
Maintenance cost	0,14	0,20	0,17
TOTAL	1,00	1,00	1,00

**Table E.12:** Average vector, priority vector, and consistency ratio of economic parameters.

The random index to analyze three variables is 0,58; while the consistency ratio is  $0,015 \leq 0,1$ .

	Average	Priority Vector	New Vector	$\lambda_{\max}$	CI	CR
Investment cost	0,4429	1,3397	3,0251	3,0183	0,0092	0,0158
Operation cost	0,3873	1,1698	3,0205			
Maintenance cost	0,1698	0,5111	3,0093			

**Appendix F: Methodology to develop the assignation of weights for extraction methods depending the criteria.****Appendix F.1: Analytic Hierarchy Process for technology maturity weight assignation.**

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	1,00	1,00	2,00	2,00	5,00	5,00	1,00	1,00
<b>Percolation</b>	1,00	1,00	1,00	1,00	5,00	5,00	1,00	1,00
<b>Soxhlet</b>	0,50	1,00	1,00	1,00	5,00	5,00	2,00	2,00
<b>Reflux</b>	0,50	1,00	1,00	1,00	5,00	5,00	1,00	1,00
<b>Supercritical fluid</b>	0,20	0,20	0,20	0,20	1,00	1,00	0,33	0,33
<b>Accelerated solvent</b>	0,20	0,20	0,20	0,20	1,00	1,00	1,00	1,00
<b>Ultrasound-assisted</b>	1,00	1,00	0,50	1,00	3,00	1,00	1,00	1,00
<b>Microwave-assisted</b>	1,00	1,00	0,50	1,00	3,00	1,00	1,00	1,00
<b>TOTAL</b>	5,40	6,40	6,40	7,40	28,00	24,00	8,33	8,33

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	0,19	0,16	0,31	0,27	0,18	0,21	0,12	0,12
<b>Percolation</b>	0,19	0,16	0,16	0,14	0,18	0,21	0,12	0,12
<b>Soxhlet</b>	0,09	0,16	0,16	0,14	0,18	0,21	0,24	0,24
<b>Reflux</b>	0,09	0,16	0,16	0,14	0,18	0,21	0,12	0,12
<b>Supercritical fluid</b>	0,04	0,03	0,03	0,03	0,04	0,04	0,04	0,04
<b>Accelerated solvent</b>	0,04	0,03	0,03	0,03	0,04	0,04	0,12	0,12
<b>Ultrasound-assisted</b>	0,19	0,16	0,08	0,14	0,11	0,04	0,12	0,12
<b>Microwave-assisted</b>	0,19	0,16	0,08	0,14	0,11	0,04	0,12	0,12
<b>TOTAL</b>	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

The random index to analyze eight variables is 1,4.

Extraction Methods	Average	Priority Vector	New Vector	$\lambda_{\text{máx}}$	IC	RC
<b>Maceration</b>	0,1939	1,6857	8,6943	8,5179	0,0739	0,0528
<b>Percolation</b>	0,1575	1,3639	8,6619			
<b>Soxhlet</b>	0,1759	1,5029	8,5443			
<b>Reflux</b>	0,1459	1,2670	8,6845			
<b>Supercritical fluid</b>	0,0355	0,3042	8,5718			
<b>Accelerated solvent</b>	0,0555	0,4615	8,3162			
<b>Ultrasound-Assisted</b>	0,1179	0,9830	8,3352			
<b>Microwave-Assisted</b>	0,1179	0,9830	8,3352			

**Appendix F.2:** Analytic Hierarchy Process for yield weight assignation.

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	1,00	1,00	0,33	0,50	2,00	0,25	0,25	0,25
<b>Percolation</b>	1,00	1,00	0,33	0,50	2,00	0,25	0,25	0,25
<b>Soxhlet</b>	3,00	3,00	1,00	0,50	3,00	0,33	0,33	0,33
<b>Reflux</b>	2,00	2,00	2,00	1,00	3,00	0,33	0,33	0,33
<b>Supercritical fluid</b>	0,50	0,50	0,33	0,33	1,00	0,20	0,20	0,20
<b>Accelerated solvent</b>	4,00	4,00	3,00	3,00	5,00	1,00	1,00	0,50
<b>Ultrasound-assisted</b>	4,00	4,00	3,00	3,00	5,00	1,00	1,00	0,50
<b>Microwave-assisted</b>	4,00	4,00	3,00	3,00	5,00	2,00	2,00	1,00
<b>TOTAL</b>	19,50	19,50	13,00	11,83	26,00	5,37	5,37	3,37

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	0,05	0,05	0,03	0,04	0,08	0,05	0,05	0,07
<b>Percolation</b>	0,05	0,05	0,03	0,04	0,08	0,05	0,05	0,07
<b>Soxhlet</b>	0,15	0,15	0,08	0,04	0,12	0,06	0,06	0,10
<b>Reflux</b>	0,10	0,10	0,15	0,08	0,12	0,06	0,06	0,10
<b>Supercritical fluid</b>	0,03	0,03	0,03	0,03	0,04	0,04	0,04	0,06
<b>Accelerated solvent</b>	0,21	0,21	0,23	0,25	0,19	0,19	0,19	0,15
<b>Ultrasound-assisted</b>	0,21	0,21	0,23	0,25	0,19	0,19	0,19	0,15
<b>Microwave-assisted</b>	0,21	0,21	0,23	0,25	0,19	0,37	0,37	0,30
<b>TOTAL</b>	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

Extraction Methods	Average	Priority Vector	New Vector	$\lambda_{\text{máx}}$	IC	RC
<b>Maceration</b>	0,0519	0,4209	8,1174	8,3004	0,0429	0,0307
<b>Percolation</b>	0,0519	0,4209	8,1174			
<b>Soxhlet</b>	0,0957	0,7825	8,1773			
<b>Reflux</b>	0,0978	0,8233	8,4216			
<b>Supercritical fluid</b>	0,0347	0,2847	8,2064			
<b>Accelerated solvent</b>	0,2010	1,7037	8,4758			
<b>Ultrasound-Assisted</b>	0,2010	1,7037	8,4758			
<b>Microwave-Assisted</b>	0,2662	2,2388	8,4115			



**Appendix F.3:** Analytic Hierarchy Process for temperature weight assignation.

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	1,00	1,00	0,50	0,50	1,00	0,33	1,00	1,00
<b>Percolation</b>	1,00	1,00	0,50	0,50	1,00	0,33	1,00	1,00
<b>Soxhlet</b>	2,00	2,00	1,00	1,00	2,00	0,33	2,00	2,00
<b>Reflux</b>	2,00	2,00	1,00	1,00	2,00	3,00	2,00	2,00
<b>Supercritical fluid</b>	1,00	1,00	0,50	0,50	1,00	2,00	1,00	1,00
<b>Accelerated solvent</b>	3,00	3,00	3,00	0,33	0,50	1,00	3,00	3,00
<b>Ultrasound-assisted</b>	1,00	1,00	0,50	0,50	1,00	0,33	1,00	1,00
<b>Microwave-assisted</b>	1,00	1,00	0,50	0,50	1,00	0,33	1,00	1,00
<b>TOTAL</b>	12,00	12,00	7,50	4,83	9,50	7,67	12,00	12,00

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	0,08	0,08	0,07	0,10	0,11	0,04	0,08	0,08
<b>Percolation</b>	0,08	0,08	0,07	0,10	0,11	0,04	0,08	0,08
<b>Soxhlet</b>	0,17	0,17	0,13	0,21	0,21	0,04	0,17	0,17
<b>Reflux</b>	0,17	0,17	0,13	0,21	0,21	0,39	0,17	0,17
<b>Supercritical fluid</b>	0,08	0,08	0,07	0,10	0,11	0,26	0,08	0,08
<b>Accelerated solvent</b>	0,25	0,25	0,40	0,07	0,05	0,13	0,25	0,25
<b>Ultrasound-assisted</b>	0,08	0,08	0,07	0,10	0,11	0,04	0,08	0,08
<b>Microwave-assisted</b>	0,08	0,08	0,07	0,10	0,11	0,04	0,08	0,08
<b>TOTAL</b>	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

Extraction Methods	Average	Priority Vector	New Vector	$\lambda_{\max}$	IC	RC
<b>Maceration</b>	0,0815	0,6830	8,3777	8,6242	0,0892	0,0637
<b>Percolation</b>	0,0815	0,6830	8,3777			
<b>Soxhlet</b>	0,1576	1,2971	8,2298			
<b>Reflux</b>	0,2011	1,8478	9,1889			
<b>Supercritical fluid</b>	0,1087	1,0272	9,4496			
<b>Accelerated solvent</b>	0,2065	1,7790	8,6149			
<b>Ultrasound-Assisted</b>	0,0815	0,6830	8,3777			
<b>Microwave-Assisted</b>	0,0815	0,6830	8,3777			

**Appendix F.4:** Analytic Hierarchy Process for pressure weight assignation.

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	1,00	1,00	1,00	1,00	0,20	0,20	1,00	1,00
<b>Percolation</b>	1,00	1,00	1,00	1,00	0,20	0,20	1,00	1,00
<b>Soxhlet</b>	1,00	1,00	1,00	1,00	0,25	0,25	1,00	1,00
<b>Reflux</b>	1,00	1,00	1,00	1,00	0,25	0,25	1,00	1,00
<b>Supercritical fluid</b>	5,00	5,00	4,00	4,00	1,00	1,00	4,00	4,00
<b>Accelerated solvent</b>	5,00	5,00	4,00	4,00	1,00	1,00	4,00	4,00
<b>Ultrasound-assisted</b>	1,00	1,00	1,00	1,00	0,25	0,25	1,00	1,00
<b>Microwave-assisted</b>	1,00	1,00	1,00	1,00	0,25	0,25	1,00	1,00
<b>TOTAL</b>	16,00	16,00	14,00	14,00	3,40	3,40	14,00	14,00

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	0,06	0,06	0,07	0,07	0,06	0,06	0,07	0,07
<b>Percolation</b>	0,06	0,06	0,07	0,07	0,06	0,06	0,07	0,07
<b>Soxhlet</b>	0,06	0,06	0,07	0,07	0,07	0,07	0,07	0,07
<b>Reflux</b>	0,06	0,06	0,07	0,07	0,07	0,07	0,07	0,07
<b>Supercritical fluid</b>	0,31	0,31	0,29	0,29	0,29	0,29	0,29	0,29
<b>Accelerated solvent</b>	0,31	0,31	0,29	0,29	0,29	0,29	0,29	0,29
<b>Ultrasound-assisted</b>	0,06	0,06	0,07	0,07	0,07	0,07	0,07	0,07
<b>Microwave-assisted</b>	0,06	0,06	0,07	0,07	0,07	0,07	0,07	0,07
<b>TOTAL</b>	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

Extraction Methods	Average	Priority Vector	New Vector	$\lambda_{\text{máx}}$	IC	RC
<b>Maceration</b>	0,0660	0,5288	8,0064	8,0125	0,0018	0,0013
<b>Percolation</b>	0,0660	0,5288	8,0064			
<b>Soxhlet</b>	0,0697	0,5582	8,0066			
<b>Reflux</b>	0,0697	0,5582	8,0066			
<b>Supercritical fluid</b>	0,2945	2,3650	8,0303			
<b>Accelerated solvent</b>	0,2945	2,3650	8,0303			
<b>Ultrasound-Assisted</b>	0,0697	0,5582	8,0066			
<b>Microwave-Assisted</b>	0,0697	0,5582	8,0066			

**Appendix F.5:** Analytic Hierarchy Process for extraction time weight assignation.

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	1,00	1,00	3,00	3,00	0,25	4,00	4,00	4,00
<b>Percolation</b>	1,00	1,00	3,00	3,00	0,25	4,00	4,00	4,00
<b>Soxhlet</b>	0,33	0,33	1,00	1,00	0,33	3,00	3,00	3,00
<b>Reflux</b>	0,33	0,33	1,00	1,00	0,33	3,00	3,00	3,00
<b>Supercritical fluid</b>	4,00	4,00	3,00	3,00	1,00	4,00	4,00	4,00
<b>Accelerated solvent</b>	0,25	0,25	0,33	0,33	0,25	1,00	1,00	1,00
<b>Ultrasound-assisted</b>	0,25	0,25	0,33	0,33	0,25	1,00	1,00	2,00
<b>Microwave-assisted</b>	0,25	0,25	0,33	0,33	0,25	1,00	0,50	1,00
<b>TOTAL</b>	7,42	7,42	12,00	12,00	2,92	21,00	20,50	22,00

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	0,13	0,13	0,25	0,25	0,09	0,19	0,20	0,18
<b>Percolation</b>	0,13	0,13	0,25	0,25	0,09	0,19	0,20	0,18
<b>Soxhlet</b>	0,04	0,04	0,08	0,08	0,11	0,14	0,15	0,14
<b>Reflux</b>	0,04	0,04	0,08	0,08	0,11	0,14	0,15	0,14
<b>Supercritical fluid</b>	0,54	0,54	0,25	0,25	0,34	0,19	0,20	0,18
<b>Accelerated solvent</b>	0,03	0,03	0,03	0,03	0,09	0,05	0,05	0,05
<b>Ultrasound-assisted</b>	0,03	0,03	0,03	0,03	0,09	0,05	0,05	0,09
<b>Microwave-assisted</b>	0,03	0,03	0,03	0,03	0,09	0,05	0,02	0,05
<b>TOTAL</b>	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

Extraction Methods	Average	Priority Vector	New Vector	$\lambda_{\text{máx}}$	IC	RC
<b>Maceration</b>	0,1778	1,5671	8,8115	8,5507	0,0787	0,0562
<b>Percolation</b>	0,1778	1,5671	8,8115			
<b>Soxhlet</b>	0,0995	0,8236	8,2735			
<b>Reflux</b>	0,0995	0,8236	8,2735			
<b>Supercritical fluid</b>	0,3111	2,8676	9,2170			
<b>Accelerated solvent</b>	0,0438	0,3672	8,3792			
<b>Ultrasound-Assisted</b>	0,0495	0,4079	8,2410			
<b>Microwave-Assisted</b>	0,0408	0,3424	8,3987			

**Appendix F.6:** Analytic Hierarchy Process for volume of organic solvent weight assignment.

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	1,00	1,00	2,00	2,00	4,00	3,00	2,00	2,00
<b>Percolation</b>	1,00	1,00	2,00	2,00	4,00	3,00	2,00	2,00
<b>Soxhlet</b>	0,50	0,50	1,00	1,00	3,00	2,00	1,00	1,00
<b>Reflux</b>	0,50	0,50	1,00	1,00	3,00	2,00	1,00	1,00
<b>Supercritical fluid</b>	0,25	0,25	0,33	0,33	1,00	0,50	0,33	0,33
<b>Accelerated solvent</b>	0,33	0,33	0,50	0,50	2,00	1,00	0,50	1,00
<b>Ultrasound-assisted</b>	0,50	0,50	1,00	1,00	3,00	2,00	1,00	1,00
<b>Microwave-assisted</b>	0,50	0,50	1,00	1,00	3,00	1,00	1,00	1,00
<b>TOTAL</b>	4,58	4,58	8,83	8,83	23,00	14,50	8,83	9,33

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	0,22	0,22	0,23	0,23	0,17	0,21	0,23	0,21
<b>Percolation</b>	0,22	0,22	0,23	0,23	0,17	0,21	0,23	0,21
<b>Soxhlet</b>	0,11	0,11	0,11	0,11	0,13	0,14	0,11	0,11
<b>Reflux</b>	0,11	0,11	0,11	0,11	0,13	0,14	0,11	0,11
<b>Supercritical fluid</b>	0,05	0,05	0,04	0,04	0,04	0,03	0,04	0,04
<b>Accelerated solvent</b>	0,07	0,07	0,06	0,06	0,09	0,07	0,06	0,11
<b>Ultrasound-assisted</b>	0,11	0,11	0,11	0,11	0,13	0,14	0,11	0,11
<b>Microwave-assisted</b>	0,11	0,11	0,11	0,11	0,13	0,07	0,11	0,11
<b>TOTAL</b>	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

Extraction methods	Average	Priority Vector	New Vector	$\lambda_{\text{máx}}$	IC	RC
<b>Maceration</b>	0,2138	1,7286	8,0837	8,0684	0,0098	0,0070
<b>Percolation</b>	0,2138	1,7286	8,0837			
<b>Soxhlet</b>	0,1167	0,9424	8,0783			
<b>Reflux</b>	0,1167	0,9424	8,0783			
<b>Supercritical fluid</b>	0,0420	0,3377	8,0421			
<b>Accelerated solvent</b>	0,0723	0,5819	8,0491			
<b>Ultrasound-assisted</b>	0,1167	0,9424	8,0783			
<b>Microwave-assisted</b>	0,1080	0,8702	8,0538			

**Appendix F.7:** Analytic Hierarchy Process for waste management weight assignation.

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	1,00	1,00	1,00	1,00	3,00	4,00	3,00	3,00
<b>Percolation</b>	1,00	1,00	1,00	1,00	3,00	4,00	2,00	2,00
<b>Soxhlet</b>	1,00	1,00	1,00	1,00	3,00	2,00	1,00	1,00
<b>Reflux</b>	1,00	1,00	1,00	1,00	3,00	2,00	1,00	1,00
<b>Supercritical fluid</b>	0,33	0,33	0,33	0,33	1,00	0,33	0,50	0,50
<b>Accelerated solvent</b>	0,25	0,25	0,50	0,50	3,00	1,00	2,00	2,00
<b>Ultrasound-assisted</b>	0,33	0,50	1,00	1,00	2,00	0,50	1,00	1,00
<b>Microwave-assisted</b>	0,33	0,50	1,00	1,00	2,00	0,50	1,00	1,00
<b>TOTAL</b>	5,25	5,58	6,83	6,83	20,00	14,33	11,50	11,50

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	0,19	0,18	0,15	0,15	0,15	0,28	0,26	0,26
<b>Percolation</b>	0,19	0,18	0,15	0,15	0,15	0,28	0,17	0,17
<b>Soxhlet</b>	0,19	0,18	0,15	0,15	0,15	0,14	0,09	0,09
<b>Reflux</b>	0,19	0,18	0,15	0,15	0,15	0,14	0,09	0,09
<b>Supercritical fluid</b>	0,06	0,06	0,05	0,05	0,05	0,02	0,04	0,04
<b>Accelerated solvent</b>	0,05	0,04	0,07	0,07	0,15	0,07	0,17	0,17
<b>Ultrasound-assisted</b>	0,06	0,09	0,15	0,15	0,10	0,03	0,09	0,09
<b>Microwave-assisted</b>	0,06	0,09	0,15	0,15	0,10	0,03	0,09	0,09
<b>TOTAL</b>	1,00	1,00	1,00	1,00	1,00	1,00	1,00	1,00

Extraction methods	Average	Priority Vector	New Vector	$\lambda_{\text{máx}}$	IC	RC
<b>Maceration</b>	0,2016	1,7749	8,8025	8,4797	0,0685	0,0489
<b>Percolation</b>	0,1799	1,5863	8,8177			
<b>Soxhlet</b>	0,1407	1,1960	8,4998			
<b>Reflux</b>	0,1407	1,1960	8,4998			
<b>Supercritical fluid</b>	0,0476	0,3965	8,3266			
<b>Accelerated solvent</b>	0,1008	0,8570	8,5028			
<b>Ultrasound</b>	0,0943	0,7729	8,1944			
<b>Microwave</b>	0,0943	0,7729	8,1944			

**Appendix F.8:** Analytic Hierarchy Process for costs weight assignation.

Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	1,00	1,00	0,33	0,33	0,20	0,20	0,25	0,25
<b>Percolation</b>	1,00	1,00	0,33	0,33	0,20	0,20	0,25	0,25
<b>Soxhlet</b>	3,00	3,00	1,00	1,00	0,33	0,33	0,50	0,50
<b>Reflux</b>	3,00	3,00	1,00	1,00	0,33	0,33	0,50	0,50
<b>Supercritical fluid</b>	5,00	5,00	3,00	3,00	1,00	1,00	2,00	2,00
<b>Accelerated solvent</b>	5,00	5,00	3,00	3,00	1,00	1,00	2,00	2,00
<b>Ultrasound-assisted</b>	4,00	4,00	2,00	2,00	0,50	0,50	1,00	2,00
<b>Microwave-assisted</b>	4,00	4,00	2,00	2,00	0,50	0,50	0,50	1,00
<b>TOTAL</b>	26,00	26,00	12,67	12,67	4,07	4,07	7,00	8,50



Extraction methods	Maceration	Percolation	Soxhlet	Reflux	Supercritical fluid	Accelerated solvent	Ultrasound-assisted	Microwave-assisted
<b>Maceration</b>	0,19	0,18	0,05	0,05	0,01	0,01	0,02	0,02
<b>Percolation</b>	0,19	0,18	0,05	0,05	0,01	0,01	0,02	0,02
<b>Soxhlet</b>	0,57	0,54	0,15	0,15	0,02	0,02	0,04	0,04
<b>Reflux</b>	0,57	0,54	0,15	0,15	0,02	0,02	0,04	0,04
<b>Supercritical fluid</b>	0,95	0,90	0,44	0,44	0,05	0,07	0,17	0,17
<b>Accelerated solvent</b>	0,95	0,90	0,44	0,44	0,05	0,07	0,17	0,17
<b>Ultrasound-assisted</b>	0,76	0,72	0,29	0,29	0,02	0,03	0,09	0,17
<b>Microwave-assisted</b>	0,76	0,72	0,29	0,29	0,02	0,03	0,04	0,09
<b>TOTAL</b>	4,95	4,66	1,85	1,85	0,20	0,28	0,61	0,74

Extraction methods	Average	Priority Vector	New Vector	$\lambda_{\max}$	IC	RC
<b>Maceration</b>	0,0668	0,5656	8,4652	8,2490	0,0356	0,0254
<b>Percolation</b>	0,0668	0,5656	8,4652			
<b>Soxhlet</b>	0,1910	1,3390	7,0086			
<b>Reflux</b>	0,1910	1,3390	7,0086			
<b>Supercritical fluid</b>	0,3992	3,7727	9,4506			
<b>Accelerated solvent</b>	0,3993	3,7738	9,4509			
<b>Ultrasound</b>	0,2981	2,5596	8,5876			
<b>Microwave</b>	0,2818	2,1288	7,5556			

**Appendix G:** *Results of the weight for each sub-criterion.*

	Technology maturity	Yield	Temperature	Pressure	Extraction time	Vol. of organic solvent	Accessibility to the solvent	Waste management	Costs
<b>Maceration</b>	19,4	5,2	8,2	6,6	17,8	21,4	13,8	20,2	6,7
<b>Percolation</b>	15,7	5,2	8,2	6,6	17,8	21,4	13,8	18,0	6,7
<b>Soxhlet</b>	17,6	9,6	15,8	7,0	10,0	11,7	13,8	14,1	19,1
<b>Reflux</b>	14,6	9,8	20,1	7,0	10,0	11,7	13,8	14,1	19,1
<b>Supercritical fluid</b>	3,5	3,5	10,9	29,5	31,1	4,2	3,4	4,8	39,9
<b>Accelerated solvent</b>	5,5	20,1	20,7	29,5	4,4	7,2	13,8	10,1	39,9
<b>Ultrasound-assisted</b>	11,8	20,1	8,2	7,0	4,9	11,7	13,8	9,4	29,8
<b>Microwave-assisted</b>	11,8	26,6	8,2	7,0	4,1	10,8	13,8	9,4	28,2