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Process design of production of essential oil from *Pimenta racemosa*

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Abstract: This research presents the potential of producing essential oils of *Pimenta racemosa* to be widely applied into the medicine field, in the production of perfumes, cosmetics, among others. An experimental facility is built for extracting with steam distillation. Results demonstrated that the highest extraction levels applying the lowest steam flow are obtained from the dry and whole leaves. With these results and considering the demands of the study, a technological daily production flow of 62.4 kg is set as proposal. A procedure for designing the process where the mass and energy are considered for determining the capacity of the equipment is applied. The technical investment indicators show a net present value of 806,932.56 USD, an internal rate of return of 46% and the investment is recovered approximately in 3 years. An environmental technical analysis for proposing solutions for the deposition of residuals is done.
Keywords : *Pimenta racemosa*, essential oil, steam distillation, process design.

1. Introduction

Bay tree (*Pimenta racemosa*) has a long history of being used as a spice, in the case of its leaves and also for the production of perfumes, colognes and creams. This oil is also used to successfully repel flies.[1]It has been proven to be used as antiradical, anti-inflammatory and antibacterial.[2] These leaves present three different varieties: common or with smelling of cloves, anise-scented and citronella scented. Its composition presents around 29 individual chemical components. Each one of them has a different therapeutic effect. The eugenol, myrcene, chavicol, linalool and limonene stand out from the group.[2, 3]

The methods used for obtaining these oils are the maceration and microwave assisted extraction [4] going through ultrasonic methods [5] using fluids through supercritical conditions, especially the carbon dioxide [6, 7] and through the use of chemical solvents in the case of low volatility oils [8] as well as the steam distillation[9] with volatile solvents and steam distillation. Although exist many technologies to obtains essential oils from plants, the process design is one of the more important step. For instance, the contribution of this paper is to design a process to obtain essential oils form *Pimenta racemosa*, using the procedure for the process design developed by Perez et al (2012).[10]

2. Materials and Methods

It is evaluated the effect of three independent variables: leaves' maturation (green, dry), leaves' size (chopped, whole) and vapor flow (low, high) in the yield oil extraction, using 2^k experimental design. The objective is to obtain the influence of leaves' maturation and size in extraction process. For each set of leaves, some of them are cut into pieces of 5 cm long by 1 cm wide, which are cut with a cutter fitted for this purpose. Fig. 1 shows the experimental facilities layout.

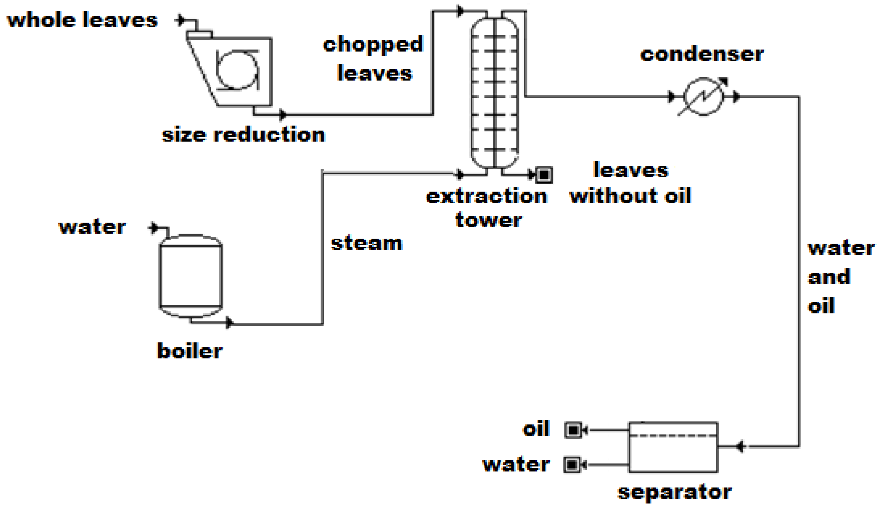

Figure 1.Experimental facility layout. Source: The authors.

To evaluate the steam flow effect, the boiler is filled with an initial volume of 3 liters of water, it is measured by the initial and final volumes of water consumption to evaluate two levels of flow experimentally measured. Energy supply to vaporize water is obtained from a hot burner where two different powers, 1000 y 800 W, are controlled. The flow levels of 5.9 mL / min in 90 minutes and 10.32 mL / min in 75 minutes for each of them are obtained The extraction tower (diameter 18cm, height 36 cm and stainless steel material) is filled with 180 g of green leaves, which are placed on a mesh located at the bottom of the column, by passing steam for a certain amount of time until oils from the extraction tower are no longer seen.

Regarding dry leaves, 153 g are weighed. For each case moisture content is measured by placing leaves on a heater (glass heat exchanger double tube, length 50 cm) during 24 min at 80 ºC. Later, according to the difference between the initial and final weight, the moisture content is determined. The leaves occupy a volume between 5.8 cm diameter and 0.48 cm high in the tower. Later, the volatile oil mixture is dissolved in water forming a miscible mixture which passes through two capacitors battery, then it is filtered in a liquid separator where oil is produced. The oil yield is calculated by the initial weight of dry leaves, which goes previously through a drying process at low temperatures. For each experiment level three replicates are done and an average value is determined. Fig. 1 shows the process for the case of the chopped leaves. In the case of whole leaves study, the size reduction step is eliminated.

2.1. Procedure for the process design.

The process is based on the methodology proposed by [10, 11] which includes the basic steps to design processes in the sugar industry and its derivatives. Within these aspects are highlighted the choice of technology, the definition of the technological scheme, the estimated capacity of the plant and the financial analyzes that demonstrate the investment feasibility, among other aspects. Although the methodology has been proposed for the sugar industry, its application in an extraction process of essential oils shows its validity for the treatment of different industrials processes.

3. Results and Discussion

Fig. 2 shows the results of experimental studies. This figure shows that the best yields are obtained with a lower steam flow (5.9 mL / min) which belongs to the higher extraction time (90 minutes). For this flow, the highest yields were obtained for the whole, dry leaves. These results indicate the need to work under lower steam flows that eliminate leaves' size reduction operations. The best yield for the dry leaves sets the necessity of installing a drying system before the extraction process.

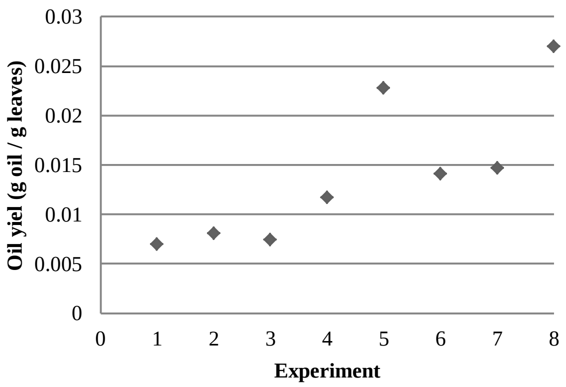

Figure 2. Oil yield. Source: The authors.

To the previous results a statistical analysis using the program Statgraphics V5.0 was done. A Pareto diagram considers the main parameters studied as well as their interactions Fig. 3 shows results.

According Pareto chart, the type of interaction between leaves and leaf size and leaf type have strong yield influence, is shown to the left of the chart. This analysis allows making the decision, once the technology is designed, that gross material can be dried or green. However, using dry leaves, which have a lower density, allows better use of the capacity of extraction column, consequently increasing the productivity of the equipment

In the case of steam flow, it significantly influences the layout inversely. An increment of steam flow reduces extracting capacity, again higher steam rates are expected to give better yields since diffusion for such oils is not important and they are easily released by breakage of the cuticular structure of the hairs and carried by the passing steam which is consistent with [12]. In the future plant design, this aspect is also of great importance since it means less spending on consumption and production of steam. In the case of interaction between the steam-sized sheets, the effect is contrary to what was stated in the previous analysis.

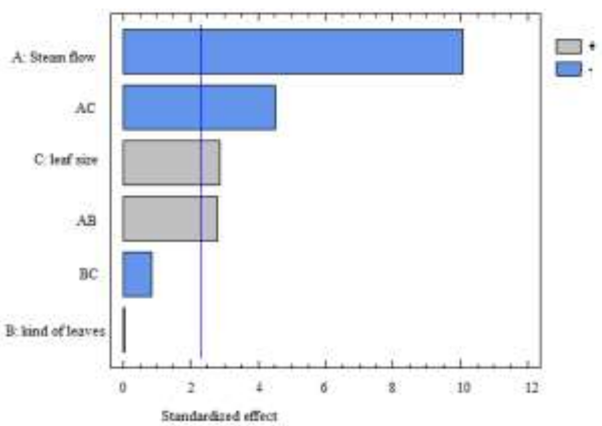

Figure 3.Pareto diagram. Source: The authors.

An opposite effect in the case of leaf's size is observed, as the extraction of the largest leaves favors efficiency. It is because while the smaller size of particles, the smaller are the holes, which difficult the steam rate across of them. This result indicates the proposed technology leaves should not be crushed, saving energy in the process.

In general, results show that variables which influence the oil extraction yield are the steam flow and the leaf's size. This can be observed in Table 1, which follows. Under these operating conditions; the highest yield is 0.02543 mL of oil / g dry leaves when working with the flow of 5.9 mL / min, so we can see that leaves are dry and whole.

Table 1.Best variant extraction results.

Factor	**low**	**high**	**Optimous**
Steam flow	low	high	low
Leaf type	dry	green	dry
Leaf size	chopped	whole	whole

3.1. Process Design

Step 1.Product.

The essential oil as is known is obtained from the *Pimenta racemosa* o malagueta or the bay-rum. Its extracts include: cloves, anise (anis) and the citronella. This is an 18m high tree. The leaves was collect in the province of Pinar del Rio (San Ubaldo, 22° 4' 59.880" N; 84° 1' 0.120" W), Cuba.[13] In the essential oil elements there are 17 components determined by gas chromatography and mass spectrometry, which represent 99% of its composition. The lighter than water are: eugenol (60.4%), myrcene (11.7%), chavicol (6.0%), limonene (5.4%) and linalool (4.4%), in contrast 13 components which are heavier than water are in the essential oil, which represent 98.3%. They are composed by eugenol (82.9%) and chavicol (9.3%).[3]

According determinations by gas chromatography and mass spectrometry, in the elements essential oil, the presence of 17 components representing 99.9% of its composition within the lighter than water.

Step 2.Technology Selection.

Although there are several technologies reported in the literature, the most widely used for the essential oil extraction is steam distillation.[12]

Step 3.Flowsheet Technological scheme definition.

Fig. 4 shows the technological scheme proposed. The process starts in a conveyor of *Pimentaracemosa* leaves, which are introduced into the extraction tower depending on the desired production and the designed capacity. Water is stored in a cistern or tank. It goes through a steam generator until it reaches the temperatures of 300 °C and 350 °C which was intended to operate at the base of the tower. In order to condense the water steam and the extracted essential oil obtained a condenser is installed. The condensed mixture goes through an oil separator where the final product is stored in a tank. Water is intended to be incorporated into the process again.

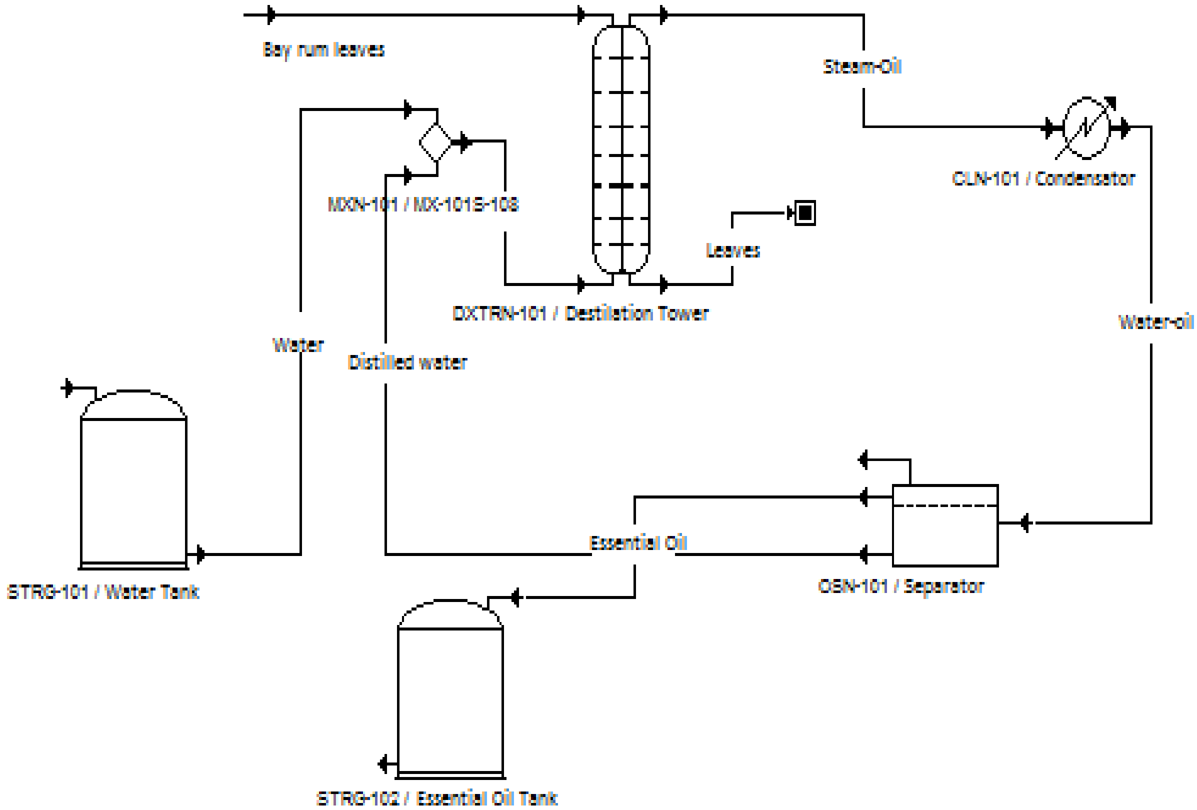

Figure 4.Flowsheet. Source: The authors.

Step 4.Determination of plant capacity.

The estimated production capacity is set according to product demand. The main consumers are studied in the country, such as pharmaceuticals and SUCHEL S.A. among others, giving approximate values of 62.4 kg/d.

Step 5.Carrying out a macro-location study.

This stage is proposed to carry out a further study to assess the main sources of raw materials in the country. The mains plantations are reported in Pinar del Rio, Cuba and eastern place.

Step 6. Mass and energy balances.

After determining the capacity of the plant, we carry out a mass and energy balances study, which establishes the main consumption of raw materials and materials needed in the process and current intermediate expenditure. At this stage the major environmental concerns, as well as the daily production and consumption rates to produce 1 kg oil are presented on Table 2.

The Table 2 shows the raw materials needed for daily oil production of 62.4 kg. It suggests guaranteeing a stable raw supply to meet the established demand. Part of the water needed to produce steam can be recovered by the process itself, once it was separated from oil, which results in considerable resource saving. Regarding to solid waste, it was considered only the deposition of leaves used into the process but no longer contain a reasonable oil level to be reused in the extraction oil process again. Gas emissions are determined by the fuel consumption for producing steam.

Table 2.Consumption Indicators and technology emissions.

Environmental indicator	Items	Unit	Quantity
Raw material consumption (leaves)		kg/kg product	70.93
Renewable raw material consumption (leaves)		%	100
Water consumption in the process		kg/kg product	26.85
Consumption of energy	Fuel	kg/kg product	7.23
	Electricity	kW/d	43.37
Consumption of renewable energy		%	0
Liquids discharge		kg/kg product	26.85
Solids discharge		kg/kg product	67.13
Gasses discharge SO_2 y CO_2		kg/kg product	11.5
Oil production		kg/d	62.4

Step 7.Raw materials availability.

Similarly to step 5, a raw materials availability study must be done to ensure the daily production of 62.4 kg/d.

Step 8.Environmental compatibility.

The essential oil's production plant's production process emits liquids', solid's and gases 'waste (see table 2).

The production process of the essential oils plant generates liquid, solid, and gases residuals. (see Table 2). The emitted liquid waste does not generate any compound which can affect environment; consequently water can be used in both; for the steam vapor regeneration and for watering the *Pimentaracemosa* plantations. Solid residuals generated after the distillation process can be used not only as organic fertilizer but also for steam generation required by the process, saving the fuels emission. Gases emitted throughout the process are CO_2 which produces about 11.5 m^3 SO_2 and CO_2 / kg it was determined considering the consumed fuel. This residual is the result of using non-renewable energy sources, such as fossil fuel, consequently this study comes through with the use of renewable energy and solid waste from the plant.

Step 9. Determination of equipment capacity and implementation cost.

Taking into account the mass and energy balances and the technological scheme, equipment capacity is determined. Indeed all technological equipment needed is calculated, in addition, the main design variable for each one determined. In this way the investment costs can be calculated [14], updated to 2015 was considered. The purchase equipment cost is $ 124 800.00. Table 3 shows results.

Table 3. Equipment acquisition cost results.

Equipment	Quantity	Variable of design	Cost	Current cost (USD)
Distilation tower	1	Diameter(m)	1.236	110 500
		High (m)	5	
Water tank	1	Volume (m^3)	0.0379	5 900
Condenser	1	Lenght (m)	2.032	7 200
		High (m)	0.889	
Separator	1	Diameter (m)	0.305	1 200
Total	4	-		124 800

Step 10.Determination of equipment availability.

The methodology proposed by [15, 16] was used to determine the plant reliability and the optimum number of redundant equipment. This technical indicator is called "optimal equipment number," which is derived from objective function defined as the minimization of operating costs plus the cost of investment subject to the constraint of system reliability. The Parallel diagram is used where reliability is considered the set of computers that define a stage as an equipment module is used. In this case, the optimal number of teams calculated matches the equipment number set in the design.

Step 11.Automatic process control.

The automatic control system of the steam generator must be foreseen, if the equipment installed is not being used as well as the extraction column. However, the low process complexity eases to dispense it. The expenses originated at this stage are not taken into account in this analysis.

Step 12.Carrying out the economic analysis.

The investment cost is based the equipment acquisition (step10). This one represents the percentage of investment and for each different item a percentage within the costs is assigned. The methodology set out by Peters and Timmerhaus (1991) is applied to do it.

Table 4. Investment cost.

No.	Components	Cost (USD)
1	Equipment	124 800
2	Facilities	48 627
3	Instrumentation	0
4	Pipeling	38 688
5	Electrycity	12 480
6	Installations	36 192
7	Service facilities	68 640
8	Engineering-Supervision	39 936
9	Infrastructure	12 480
10	Infrastructure cost	22 464
Total		404 307.00

Similarly, the production costs are determined, but in this case they are based on the raw materials acquisition cost, this is determined according to the plant capacity and the cost of *Pimenta racemosa's* leaves established in the market. Table 5 shows production cost results.

Table 5. Production cost.

No.	Components	Annual Cost ($)
Variable cost		
1	Raw material	218 139.00
2	Labor direct force	3 635.65
	Auxiliar facilities	
3	-water	15 078.45
	-fuel	80 712.90
Total variable cost		317 566.00
Fixed cost		
4	Amortization	24 960.00
5	Maintenance	5 154.24
6	Operation'supply	2 577.12
7	Labs	36.35
8	General cost.	181 782.50
9	Administrative cost.	36 356.50
Total fixed cost		250 866.71
Total		568 432.71

The operating cost of the process is $ 568 432.71, which is similar to the plant in the Caribbean Islands.

Step 13.Optimization.

This stage has not been taken into account in this research, and will be addressed in further studies.

Step 14.Economically feasible alternative.

Based on the oil's international market price the incomes are determined. In order to determine the Net Present Value (NPV) and the Internal Rate of Return (IRR), this research is being done considering a life-long term of 10 years. The estimated amount is $ 806 932.56 and 46%, respectively, with the Payback period (PB) of 3 years. Fig. 5 shows NPV and the PB in 10 years.

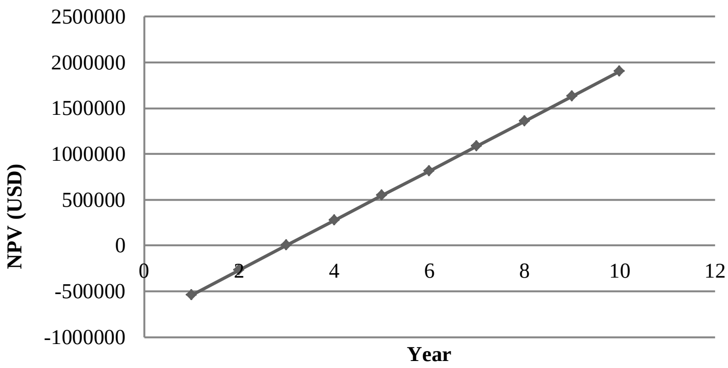

Figure 5.Fluctuation of economic indicators NPV y PB.

In addition to this study case, it should be taken into account that it is a high value product. This is due to the high global demand, the solid waste can be used in agronomy (organic fertilizer), the equipment is available and the technology installed allows people to reuse it with lower investment cost. The qualified labor force and the raw materials availability would stabilize the supply.

4 Conclusions

This research demonstrates that when using whole dry leaves as well as a lower steam flow the best oil yield is obtained. This aspect is very important because it eliminates the application of the grinding process which should be done before. As a result there is an economic impact for both the investment and operation. Regarding the dry leaves, drying methods should be studied to best fit operating conditions.

The dynamic investment indicators, NPV, IRR and the PB show favorable results demonstrating the technical and economic feasibility of this technological proposal.

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