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Extraction, Purification and Equipment Designing for Eugenol

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Abstract

Eugenol is a bioactive compound which is found majorly in the dried flower buds *Szygium aromaticum* plant, which is otherwise also known as clove buds. Apart from being used as a spice in various cuisines across the world, clove buds are also used to derive the essential oil. Cloves buds are known to contain about 15-20% eugenol. The clove buds will undergo various unit operations such as crushing, grinding and blending in order to reduce their size, from which maximum oil can be obtained. Extraction method has been implemented to yield eugenol from the above source. The method utilized here is the batch distillation followed by liquid-liquid extraction of eugenol from the distillate. The eugenol that is obtained from the clove buds are used to make a wide variety of products such as soaps, toothpastes, toothpowders, fragrances, cosmetics and also plays a very important role in aromatherapy due to its analgesic, anti-inflammatory, anti-septic nature. The main purpose of this project is to find a suitable method to extract eugenol from sources widely available in the market and to make some products out of the eugenol extracted. An equipment design was also carried out as well as calculations to upscale the project if it were to be industrialized. Future scope which includes setting up of pilot plant in each household to reduce carbon footprint impact, recommending the usage of essential oils for a clean and better lifestyle, conducting plant designs, using the same methods extracting essential oils using other natural resources were also discussed.

Keywords: Eugenol; *Szygium aromaticum*; Extraction; Steam Distillation; Analgesic; anti-inflammatory; Blending; Distillate; Equipment Designing

1. Introduction

Clove oil is a naturally obtained essential oil that is present mainly in sources such as Clove buds. The major component of clove oil, Eugenol, is a natural aromatic compound which has many medicinal and also some industrial applications as well. It is known to have analgesic, antiseptic and anti-inflammatory properties and is known to be used in fragrances, cosmetics and also flavorings at times. When coupled together with zinc oxide it can be used as a dental packaging, cement or fillings, signifying its usage in dental sciences. [1][2]

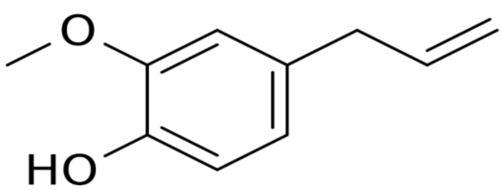

Figure 1: Chemical Structure of Eugenol

The extraction of eugenol from clove buds and other sources have undergone various amounts of research, due to the importance of eugenol as an essential oil and its uses in various sects of society such as medicinal, culinary and cosmetics industry. [3] The consumption of eugenol be it from any sources such as cloves, basil, cinnamon etc. contribute towards a protection against inflammation, hyperglycemia, increased cholesterol levels, neurological disorders, as well as microbial attacks. Due to eugenol's ability to scourge free radicals, reactive oxygen, species of nitrogen, and its hostility towards microbial DNA contribute towards its antioxidant properties. [4]

The extraction of eugenol involves many processes, some of which involve unit operations like crushing, grinding and blending in order to achieve size reduction to extract maximum eugenol from the source. Thereafter a batch distillation process is carried out in a distillation apparatus, the distillate obtained is rather a crude version of eugenol, crude being an oil in water emulsion which undergoes liquid-liquid extraction with the help of an organic solvent and then is evaporated to obtain the pure eugenol. After obtaining the eugenol, to know whether if the product that we have obtained is pure or does it contain any sort of impurities or other components in minority we can conduct various tests such as High Performance Liquid Chromatography (HPLC), Gas Chromatography (GC) coupled with mass spectrometry. [5]

1.1 History and Background

Eugenol being a naturally occurring compound, has a very abundant history has been used across various areas in this very abundant history. Eugenol was first isolated in the year 1929. The word "eugenol" was derived from the scientific name of the clove tree, *Eugenia caryophyllata* from where it had been extracted from. [2] Here are some of the examples where it has been used.

1) Ancient Uses: Cloves which are a very good source of eugenol having long history of its usage in medicinal and culinary sects of society. [6] Eugenol was held in high regards with respect to usage in ancient Egypt and China for their aromatic and medicinal properties.

2) Traditional Medicine: Eugenol has been used in traditional medical sciences such as ayurveda and chinese medicine, where cloves and its oil, eugenol has been used to relieve various other ailments. They have

been used for their properties which have been discussed earlier such as analgesic, anti-inflammatory, anti-microbial due to its digestive properties towards microbial cell walls. [7]

3) Western Medicine: In the early 19th and 20th centuries, eugenol was used extensively in western medicine as an anesthetic and antiseptic in dental procedures like tooth extractions and oral surgery. [8]

4) Flavoring Agents: Eugenol's aroma and spicy flavor has made it a widely used ingredient in the culinary world. It is used in limited quantities in the production foods and bevarages such as bakeries, confectionaries and spice-flavored drinks. [9]

5) Industrial Applications: Looking beyond the medicinal and traditional uses, eugenol is also used in today's time to manufacture soaps, cosmetics, perfumes and insecticides as well. [10]

1.2 Need for Project

The need for this project is as follows:

Educational Purposes: This project can serve as an educational tool to better teach students about the distillation processes to extract the eugenol from clove and the liquid-liquid extraction process on how to separate the distillate that is obtained which is an oil in water emulsion, as well as give an overview on how unit operations are carried out in order to study about size reduction of any material given using crushing, grinding and blending, and also provides an opportunity to learn about the natural sources and what benefits they may contain and how we can put it to good use.

Research Purposes: Eugenol is a natural compound which has wide variety of uses such as industrial, medicinal, this project can help build a base for research on the properties of eugenol and its potential benefits.

Health Benefits: Eugenol being a natural compound have many health benefits such as anti-inflammatory, analgesic, anti-septic properties, this can be a huge benefit to the health industry and also to the customer base.

Economic Benefits: Eugenol is used in many industries such as fragrances, cosmetics, flavorings and also medicinal. It can be beneficial to the economic aspect if we study the process of extraction, cost and how the industry can be scaled up.

1.3 Aims and Objectives

The aims and objectives of this project is as follows:

1) To extract eugenol from using clove buds as starting material via batch distillation.

2) Collect results of all processes

3) Performance evaluation and studies are conducted by calculating the yield which is done by dividing the mass of eugenol obtained by the initial mass of the clove buds used for extraction. Same can be carried out by assuming volume basis.

4) Equipment design is carried out considering factors such as the desired production scale, processing conditions, and the properties of the materials being processed. Based on these considerations, design the appropriate distillation equipment and ensure that it can handle the required amount of cloves and solvents.

5) To conduct scale up calculations in order to determine what the equipment design should be when industrial production is considered.

2. Problem Definition

The problem that needs to be defined here is the extraction of eugenol from a naturally available resource that is, clove buds by implementing a suitable extraction method. Challenges to this experiment include the selection of right methods, time, temperature, safety precautions, all of which must be maintained while achieving the main goal, that is extracting pure eugenol.

3. Methodology

3.1 Crushing, Grinding and Blending:

The first part of the process is to perform the unit operations, where the clove buds are reduced to a fine powder by means of using crushing, grinding and the blending processes. The crushing and the grinding can be performed by making use of the mortar and the pestle, but in order to reduce the size further, that is to obtain a fine powder, a kitchen blender can be used. The fine powder obtained is then mixed with water to form a solution and this will undergo distillation process. [11]

3.2 Distillation Process:

Distillation is a process which is used to separate two or more substances which have separate boiling points, the solution or the feed is converted into steam which then enters into the condenser, where they are cooled back to liquid form with the help of water which is being circulated in the condenser apparatus, thereafter which the distillate is collected and then extraction or the purification process is carried out. The distillation process that is being carried out here is steam distillation and the process lasts around 2-4 hours before distillate is obtained properly. Then the distillate is sent for liquid-liquid extraction since the distillate is an oil in water emulsion and separation must be done in order to extract eugenol.

3.3 Liquid-Liquid Extraction and Evaporation:

The distillate obtained as mentioned is an emulsion of oil in water which must be separated and suitable method through which this can be done is liquid-liquid extraction. The distillate is mixed with ethyl acetate which is an organic solvent, the separation takes place in a separatory funnel. After some time, the oil and water layers will tend to separate. The aqueous layer will undergo extraction process till it cannot be separated and the organic layer consists of the oil and ethyl acetate, which is then mixed with calcium chloride, a drying agent to remove any traces of water, and henceforth this organic layer undergoes evaporation process, wherein the ethyl acetate will completely evaporate and the left over is pure eugenol. [12] [13]

3.4 Process Flow Diagram:

Process flow diagram is generally used to depict the processes taking place in a pictorial form.

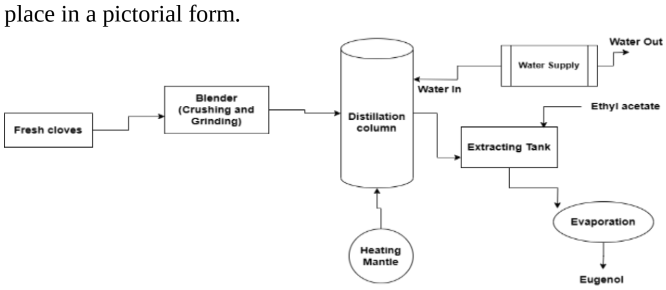

Figure 2. Process flow diagram for manufacture of eugenol

As per Figure 2, this process flow diagram depicts all the steps ranging from collecting clove buds to crushing, grinding and blending the cloves, to being fed in the distillation column mixed with water with the help of water supply and undergoing boiling with the help of the heating mantle, distillate going to the extracting tank, thereafter ethyl acetate is fed to it to undergo liquid-liquid extraction to separate the oil and water layers, thereafter will undergo evaporation process to evaporate the ethyl acetate to finally yield eugenol.

3.5 Experimental Procedure

1) **Size Reduction process**: Cloves are procured from a local supermarket or grocery store and then will undergo a process of crushing, grinding and blending in order to obtain finest form of clove powder.

2) **Distillation Process:** The clove powder is to be weighed and then put into a round bottom flask and then water is added accordingly to create a solution. Then the solution is added into a round bottom flask of the apparatus and then a thermometer is fixed over this round bottom flask to control the temperature. Water supply is then circulated through the condenser apparatus and the round bottom flask is then subjected to heat using a heating mantle. Temperature is to be under control at around 100°C and is done for around 1-2 hrs. Distillate is then collected and then further liquid-liquid extraction is done.

3) **Liquid-Liquid Extraction Process:** After the distillate is obtained it will undergo liquid-liquid extraction in order to separate the water and oil layers. This will be undergone until there is no more separation to take place. The extraction is performed using a solvent like ethyl acetate to separate the layers. [14] Collect the oil layer and water layer into separate beakers. Water layers must undergo subsequent process until the layers cannot be separated. After obtaining the oil layer, evaporation process must be done in order to obtain the final product.[15]

4) **Evaporation Process:** The oil layer that is finally obtained is then subjected to evaporation process, but before that some amount of calcium chloride is added, which is a drying agent to remove any water content that may be present. After which a filtration process is done in order to remove the calcium chloride and the filtrate (liquid) which we obtain is an ethyl acetate-oil mixture which must be evaporate to finally obtain the oil. Oil obtained will then be measured to determine how much ml of oil has been obtained and then will undergo confirmatory test to conclude the experimental process.

5) **Confirmatory Test for Eugenol:** After the eugenol is obtained, a confirmatory test was performed to confirm the presence of eugenol.

1) Ferric Chloride test: Few drops of ferric chloride were added into a test tube followed by few drops of eugenol as well. Color change of red (ferric chloride) to green, indicates the presence of eugenol. [16]

4. Results and Discussions

Table 1. Amount of Eugenol Obtained in ml

Amount of Clove Powder taken (g)	Amount of Oil obtained (ml)
20	3
40	6
60	9

Table 1. depicts the amount of the eugenol that is obtained after the evaporation process in terms of milliliters (ml) corresponding to the weight of clove powders taken as feed in grams (g).

4.1 Yield Calculations

For mass basis yield calculations:

Using theoretical content value of eugenol which is 15-20% by weight, we can obtain the theoretical mass.

$$\rho = \left(\frac{m}{v}\right) \quad (1);$$

$$m = \rho \ x \ v \quad (2);$$

$$Yield\ \% \left(\frac{experimental\ mass}{theoretical\ mass}\right) \text{ x } 100 \quad (3)$$

Using Equations 1,2,3 and theoretical weight content of eugenol we can obtain the yield percentage for mass basis.

Table 2. Percentage yield of eugenol obtained (mass basis)

Amount of Clove Powder taken (g)	%Yield of Eugenol Obtained (mass basis)
20	88.33
40	88.34
60	88.35

Table 2. Depicts the yield percentage of eugenol obtained finally after using equation 3 with the help of equations 1 and 2 and also the theoretical value of eugenol content. The yield percentage obtained here for each of the clove powders taken shows us that yield obtained is good and there is nearly no wastage.

For volume basis yield calculations:

Using theoretical content value of eugenol which is 15-20% by weight, we can obtain the theoretical mass.

$$v = \left(\frac{m}{\rho}\right) \quad (4)$$

$$\left(\frac{\text{experimental volume}}{\text{theoretical volume}}\right) \text{ x } 100 \quad (5)$$

Using equations 1, 4, 5 and the theoretical weight content of eugenol we can obtain the yield percentage of eugenol for volume basis.

Table 3. Percentage yield of eugenol obtained (volume basis)

Amount of Clove Powder taken (g)	%Yield of Eugenol Obtained (volume basis)
20	88.49
40	88.36
60	88.44

Table 3. Depicts the yield percentage of eugenol obtained finally after using equation 5 with the help of equations 1 and 4 and theoretical value of eugenol content. The yield percentage obtained here for each of the clove powders taken shows us that yield obtained is good and there is nearly no wastage.

4.2 Batch Distillation Calculations

$F.\ X_F = D.\ X_D + W.\ X_W$ (6)
Since there is no wastage during the process;
$\therefore W.\ X_W = 0$
Hence,
$F.\ x_F = D.\ X_D$ (7)
F= (Volume of clove powder + water mixture) in ml
X_F = Mass fraction of eugenol in the feed (theoretical value) = 0.18
D= Volume of distillate collected in ml
X_D= Mass fraction of eugenol in the distillate

Using equations 6 and 7 we can calculate the fraction of distillate obtained X_D, for all the feed material taken accordingly.

Table 4. Fraction of distillate and distillate obtained

Amount of Clove Powder taken (g)	Distillate collected (ml)	Fraction of the distillate, X_D
20	125	0.315
40	135	0.317
60	145	0.318

Table 4. depicts the amount of distillate that is collected in milliliters (ml) and the fraction of the distillate obtained corresponding to the weight of clove powder that is taken as feed material in grams (g).

4.3 Extraction Calculations

Since this is a liquid-liquid extraction operation the raffinate phase will be equal to zero, and also the solvent fraction will be equal to zero as there is no reuse of the solvent in the stages (i.e. a fresh solvent is used during each stage of the extraction operation. Hence y_s=0).
We have;

$$F+S_1= R_1+E_1 \quad (8)$$

$$F.\ X_F+S_1.\ y_s= R_1.x_1+E_1.y_1 \quad (8^*)$$

Where
F= amount of distillate obtained in ml
S_1 or S = the amount of solvent added for the extraction process in ml
R_1 = amount of raffinate obtained, which in the case of liquid-liquid extraction=0
x_1 = fraction of raffinate obtained, which in the case of liquid-liquid extraction=0
E_1 = amount of extract obtained in ml
y_1= fraction of the extract

4.3.1 Extraction calculation for 20g of clove powder feed taken

Extraction calculations can be performed using equations 8 and 8* keeping the required parameters in mind.

Table 5. Fraction of extract obtained for 20g of clove powder

Stage Number	Fraction of the Extract(y_1)	Extractant Amount (ml) ($F+S_1=E_1$)
1	0.175	225
2	0.168	215
3	0.161	205
4	0.153	195
5	0.144	185
6	0.135	175
7	0.124	165
8	0.111	155
9	0.097	145
10	0.081	135
11	0.063	125
12	0.041	115
13	0.028	110

Table 5. Depicts the number of stages that has taken place during the extraction operation for distillate obtained using 20g of clove powder as feed and y_1 will depict the fraction of the layer separated at each stage and extractant amount in terms of milliliters (ml) will depict the mixture of distillate and solvent used for the extraction at each stage and $E_1.y_1$ at the end of 13^{th} stage will show the final amount of eugenol extracted, which is depicted in table 1.

4.3.2 Extraction calculation for 40g of clove powder taken

Extraction calculations can be performed using equations 8 and 8* keeping the required parameters in mind.

Table 6. Fraction of extract obtained for 40g of clove powder

Stage Number	Fraction of the Extract(y_1)	Extractant Amount (ml) ($F+S_1=E_1$)
1	0.150	285
2	0.137	265
3	0.122	245
4	0.105	225
5	0.085	205
6	0.060	185
7	0.035	175

Table 6. Depicts the number of stages that has taken place during the extraction operation for distillate obtained using 40g of clove powder as feed and y_1 will depict the fraction of the layer separated at each

stage and extractant amount in terms of milliliters (ml) will depict the mixture of distillate and solvent used for the extraction at each stage and $E_1.y_1$ at the end of 7th stage will show the final amount of eugenol extracted, which is depicted in table 1.

4.3.3 Extraction calculation for 60g of clove powder taken

Extraction calculations can be performed using equations 8 and 8* keeping the required parameters in mind.

Table 7. Fraction of extract obtained for 60g of clove powder

Stage Number	Fraction of the Extract(y_1)	Extractant Amount (ml) ($F+S_1=E_1$)
1	0.156	295
2	0.144	275
3	0.130	255
4	0.115	235
5	0.096	215
6	0.060	185
7	0.050	178

Table 7. Depicts the number of stages that has taken place during the extraction operation for distillate obtained using 60g of clove powder as feed and y_1 will depict the fraction of the layer separated at each stage and extractant amount in terms of milliliters (ml) will depict the mixture of distillate and solvent used for the extraction at each stage and $E_1.y_1$ at the end of 7th stage will show the final amount of eugenol extracted, which is depicted in table 1.

5. Equipment Designing

5.1 Distillation Column

For a distillation column the operating temperatures would be around 100°C considering boiling of water and clove powder mixture hence borosilicate material would be the best option, as it can withstand higher temperatures. The Reaction vessel chosen depends upon the volume of the reaction mixture. An additional 25% must be allotted to decide on reacting vessel. Hence the final volume of the reaction mixture is calculated using equation 9:

$V_{final(reaction\ mix)} = V_{calculated} + 0.25\ V_{calculated}$ – (9)

Condenser that is chosen for this operation will depend on the V_{final} and is specific. The operation that takes place here is batch operation.

5.2 Extraction tank

This design will depend mainly upon the L/D ratio. The diameter of the tank can be calculated using equation 10:

D= (0.5-0.75) * L – (10)

The thickness of the tank will depend upon the operating pressure which is atmospheric pressure and the operating temperature will be room temperature. The operation taking place here will be a batch operation and the best suited material for the extracting tank will be Stainless steel.

5.3 Equipment Design to manufacture 1 Liter of Eugenol

5.3.1 Distillation Column Volume

Using equation 9 we can calculate the V_{final} of the distillation column required to scale upto 1 litre of eugenol to be generated using the parameters in the given formula.

$V_{final} = V_{(calculated)} + 0.25(V_{(calculated)})$; from – (9)

V_{final}= 136.78 liters

5.3.2 Extraction Tank Volume

Using equation 11 we can calculate the tank volume need for extraction in order to obtain 1 liters of eugenol as end product keeping in mind the parameters in given formula.

Tank Volume= Amount of Distillate+ Solvent Amount – (11)

Tank Volume= 112.5 liters

6. Conclusion

To conclude, this experiment serves as a reminder that with very little machinery involved, one can extract essential oils at even the household level. Essential oils and its uses are increasing due to some part of the people shifting towards more of a “slow living or vegan” life style, moving away from the usage of animal products and using more of nature or green based products. This extracting technique not only can be used on clove buds to extract eugenol, but may as well be used on other natural sources to expand the horizons of essential oil study.

7. Future Scope

To conduct a proper plant design for the manufacture of eugenol and make some products out of the eugenol that has been obtained and using the same extraction methods to study other natural sources and extract essential oils from them. To take proper initiatives to set up pilot plants or lab scale plants at the household level to promote the use of essential oils such as eugenol and others to promote green living and also at the same time by setting up lab scale or pilot plants, we will reduce our dependency on industrial machinery thereby reducing the impacts of carbon footprint on our ecosystem.

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