SEC LAB MANUAL

CHC-143 Chemistry of Cosmetics and Perfumes

Course Objective:

- To translate certain theoretical concepts learnt earlier, into experimental knowledge by providing hands on experience of basic laboratory techniques required for Cosmeticology and perfume chemistry.
- To understand the concept of cosmetics and develop formulation skills in the preparation of various cosmetic products.

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References /Readings:

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14. Berger, Ralf Günter, Flavors and Fragrances: chemistry, bioprocessing and sustainability (ed.), 1st edition. 2007.
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GENERAL INSTRUCTIONS

- Students should know the location of the lab safety equipment and understand how to use it. In particular, know the location of the emergency exit, fire extinguisher, eyewash station, and safety shower.
- Read through the experiment before going to the lab. Make sure you understand the steps of the experiment. Jot down any questions you have so that you can ask them before starting the lab.
- The information and calculations provided during the Pre-lab exercise are intended to make the practical quicker and easier.
- Start filling out your lab notebook with information about the experiment. It's a good idea to draw out your observations table etc. in advance so all you need to do in the lab is fill it in with numbers.
- Be aware of the chemicals you will be using during the lab.
- Make certain you have all of the glassware, materials, and chemicals needed to complete the practical *before* starting any part of the procedure.
- Understand disposal procedures for the chemicals and other items used in your experiment. If you are unclear about what to do with your experiment after it has been completed, ask your batch teacher about it. Don't throw items in the trash or dump solvents down the drain or in waste disposal containers until you are certain it is acceptable to do so.
- Be prepared to take data in the lab. Bring your notebook, a pen, and a calculator.
- *Have personal safety gear, such as a lab coat and goggles, clean and ready to use before the lab.

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COSMETICS

Introduction

Cosmetic is a Greek word which means to 'adorn' (addition of something decorative to a person or a thing). It may be defined as a substance which comes in contact with various parts of the human body like skin, hair, nail, lips, teeth, and mucous membranes etc, Cosmetic substances help in improving or changing the outward show of the body and also masks the odour of the body. It protects the skin and keeps it in good condition. In general, cosmetics are external preparations which are applied on the external parts the body.

Even in earlier days, men and women used to decorate their bodies for improvement of appearance. Men used leaves of vegetables and parts of animals whereas women use to wear colored stones and flowers round their neck and wrist. Gradually, they start using colored earth and ointments on their face and body. Even bangles and necklace made of baked earth materials became very common among the people. Eye shadow were made of copper (coloured earth) ore and lamp black (coloured earth) while red colour was used for dyeing of hair. Now days, cosmetics are considered as essential components in life. They not only, attract the people towards it but also impart psychological effects. It has gained popularity in the last 3-4 decades and its use has been increased exponentially both-in males and females. The most popular cosmetics are hair dyes, powders and creams.

Examples of Cosmetics: Skin-care creams, powders, lotions, lipsticks, nail polishes, eye and face makeup, deodorants, baby products, hair colourants and sprays etc.

Uses:

1.They are used as a cleansing, moisturizing and beautifying agent.

2. They help in enhancing attractiveness of the body.

3. They help in altering the appearance of the body without affecting its functions.

4. Sunscreen products help in protecting the body from UV rays and treating sunburns.

5. Acne, wrinkles, dark circles under eyes and other skin imperfections are treated or repaired by treatment products.

6. Cosmetics help in treating skin infection.

Classification: Cosmetics are broadly categorized into four types:

1. Skin Cosmetics

2. Hair Cosmetics

3. Nail Cosmetics

4. Cosmetics for hygiene purpose

Creams are semi-solid emulsions which contain mixtures of oil and water. Their consistency varies between liquids and solids. Salve (medical ointment for soothing purpose) and unguent (soothing products) preparations in earlier days led to the development of cleansing and cold creams. With the help of additives such as emulsifying agents and newer techniques, the preparation of creams has become easy.

Experiment No: 01

Aim: To prepare and submit _A__ g of cold cream in a suitable labelled container.

Requirements: Beaker, spatula, mortar, pestle, measuring cylinder and ingredients as per table.

Theory: Cold creams are water-in-oil type of emulsion. They produce cooling sensation by the evaporation of water, after application of cream to the skin. Hence, they are known as cream. They should possess emollient action and the layer left on the skin after application should be non-occlusive.

Master Formula:

Formula	Justification	Standard Quantity	Quantity Used
White beeswax	(emollient)	20 g	
Mineral oil	(lubricant)	50 g	
Distilled water	(vehicle)	28.8 g	
Borax	(buffer)	0.7 g	
Perfume	(odour)	0.5 g	

Procedure:

1) Beeswax is melted in a container by using water bath to a temperature of about 70° C. Then mineral oil is added to the melted beeswax. This is mixture **1**.
2) In another container, water is heated to a temperature of about 70° C and borax is dissolved in it. This is mixture **2**.
3) Mixture **2** (aqueous phase) is added slowly to mixture **1** (oily phase) along with stirring.
4) Stirring is carried out until a creamy emulsion is formed.
5) Finally, perfume is added to the preparation when it attains a temperature of about 40°C.

Calculations:

1) **Calculation for final Quantiy of product**
   a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A
      = A+(10/100) x A
      = A + (0.1 x A)
      = __**Y**__ g or mL

2) **Calculation for quantity of ingredient used :**
   a) The amount of white beeswax required for 100g = 20 g

      The amount of white beeswax required for Y g = $\frac{Y \times 20}{100}$

      = ____ g

Observations:

a) Weight of empty beaker = _____ g
b) Weight of beaker + final product = _____g
c) Weight of final product = (b-a) g

Usage: designed to smooth skin and remove make up, skin treatment such as (facial mask or lip balm) **Storage:** Tightly sealed container

Label :

COLD CREAM ___ g	
Composition:	**BATCH NO.** **MFG. DATE:**
Category: Cosmetic	**MFG. LIC. NO.:** **EXPIRY DATE:**
FOR EXTERNAL USE ONLY.	
Use: **Storage:**	
MFG. BY: Batch:	Roll No.:

Result : 1) _____ of cold cream was prepared and submitted.

2) Appearance :

Experiment No: 02

Aim: To prepare and submit ___ g of Vanishing Cream in a suitable labelled container.

Requirements: Beaker, spatula, mortar, pestle, measuring cylinder and ingredients as per table.

Theory: Vanishing cream is traditionally oil in water type of emulsion. The emulsifying agent used is soap. Triethanolamine has special properties which make it very appreciable for making vanishing cream. One part of triethanolamine will combine with ≈ 2parts of stearic acid to make three parts of soap. This soap has property of being completely oil soluble which tend to the solubility of cream made of it. Further emulsification and saponification proceed very easily to completion. Borax also reacts with parts of stearic acid to form sodium soap which further stabilize the emulsion.

It is important to ensure that both phases are maintained at same temperatures during mixing. Perfume should be added relatively at low temperatures to prevent loss due to volatilization. On standing, cream develops lustre and pearliness. This is most valued characteristic of vanishing cream and is due to slow formation of minute lamellar crystal of stearic acid or storage.

Master Formula:

Ingredients	**Justification**	**Standard quantity**	**Quantity used**
Stearic acid	lubricant	14%	
Triethanolamine	Emulsifying agent	2%	
Lanolin	emollient	1%	
Borax	Reacts with stearic acid to produce a white emulsion.	4%	
Glycerin	Humectant	8%	
Propyl paraben	Preservatives	0.15%	
Purified water	vehicle	70.5%	
Perfume (rose oil/ lemon oil)	odour to the cream	6.5%	

Procedure:

1) Melt the lanolin and stearic acid in an evaporating dish on water bath.
2) Dissolve propyl paraben in glycerin and borax in required quantity of purified water, mix and then add triethanolamine.
3) Heat this mixture at around 75° C and add this to stearic acid melt at the same temperature.
4) Stir it continuously while adding and further until emulsification has taken place.

5) When temperature drops around 40° C, mix required quantity of perfume and mixture in order to obtain a smooth semi solid cream.

Calculations:

1) Calculation for final Quantity of product (Theoretical)

a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A

= A+(10/100) x A

= A + (0.1 x A)

= __**Y**__ g or mL

2) Calculation for quantity of ingredient used :

a) The amount of stearic acid required for 100% = 14 %

The amount of stearic acid required for Y g = $\frac{Y \times 14}{100}$

= ____ g

Observations:

a) Weight of empty beaker = _____ g
b) Weight of beaker + final product = _____g
c) Weight of final product = (b-a) g

Use of the product: It is a facial makeup cream or a foundation for facial make up (to provide an adherent base for subsequent application of face powder and other makeup cosmetics).

Storage: Wide mouth jars of glass or plastic collapsible tube made up of tin, aluminum or plastic container should be well closed or stored in cool place.

Label :

VANISHING CREAM ___g	
Composition:	**BATCH NO.** **MFG. DATE:**
Category: Cosmetic	**MFG. LIC. NO.:** **EXPIRY DATE:**
FOR EXTERNAL USE ONLY.	
Use: **Storage:**	
MFG. BY: Batch:	Roll No.:

Result : 1) _____ of Vanishing cream was prepared and submitted.

2) Appearance :

Experiment No: 03

Aim: To prepare and submit ___mL of Liquid Shampoo in a suitable labelled container.

Requirements: Beaker, mortar and pestle, glass rod, etc

Theory: A viscous cosmetic preparation with synthetic detergent used for washing hair is called shampoo. It's principle function is to clean the scalp such that it should become free from sebum and foreign substances. Shampoo also makes the hair lustrous and good looking.

Today shampoo has become an important hair cosmetic for both men and women. However the detergent and other raw materials selected for shampoo preparation should be non toxic to tile scalp, eyes etc. Apart from cleaning, shampoo may also be used for medicinal purpose (i.e., medicated shampoo).

Various types of shampoos are available and they are classified based on their consistency they are as follows: Clear liquid shampoos, Liquid cream shampoos, Cream shampoos, Gel shampoos, Powder shampoos, Aerosol shampoos (Foam type), Special shampoos.

Master Formula:

Ingredients	Justification	Standard Quantity	Quantity used
Sodium Lauryl sulfate	surfactant	20 %	
Tween 80	Co-surfactant (emulsifier)	4%	
Methyl Paraben	preservative	0.2 %	
Glycerin	Humectant	10%	
Colour		q.s	
Perfume		q.s	
Purified water		q.s	

Procedure: Prepare a paste of sodium lauryl sulphate, glycerin into which a preservative is dissolved with small quantity of hot water using a mortar and pestle. Add Tween 80 and mix

carefully to avoid frothing. Add colour and perfume. Measure and find the final volume of shampoo.

Calculations:

1) **Calculation for final Quantity of product**
   a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A
   = A+(10/100) x A
   = A + (0.1 x A)
   = __**Y**__ mL

2) **Calculation for quantity of ingredient used :**
   a) The amount of sodium lauryl sulfate required for 100% = 20 %

   The amount of sodium lauryl sulfate required for Y g = $\frac{Y \times 20}{100}$
   = ____ g

Use of the product: To cleanse the hair by removing grease and dirt

Storage: store in a cool & dry place

Label:

LIQUID SHAMPOO __mL		
Composition:	**BATCH NO.**	**MFG. DATE:**
Category: Cosmetic	**MFG. LIC. NO.:**	**EXPIRY DATE:**
FOR EXTERNAL USE ONLY.		
Use: **Storage:**		
MFG. BY:	Batch:	Roll No.:

Result : 1) _____ of liquid shampoo was prepared and submitted.

2) Appearance :

Experiment No: **04**

Aim: To prepare and submit ___ g of Talcum Powder in a suitable labelled container

Requirements: Beaker, mortar and pestle, glass rod, etc

Theory: Powders are considered as one of the important products of skin care preparations. They are used widely by both men and women for face and body care. Various types of powders are body powder, face powders, compacts medicated powders (which are used for prickly heat purposes and preventing microbial growth on the surface of the skin), deodorant powders and foot powders for treatment purposes). Powders have different physical properties when compared to the liquid preparations. They have very fine particle size, which helps in producing large surface area per unit weight. This helps in proper dispersion of powder, which covers the large surface area of the body.

Talcum Powder is one of the most popular beauty care products used by men and women including infant to keep the skin dry, to suppress the bad (sweat) odor and to feel fresh. Talcum powder is made from talc, a mineral made of the elements magnesium, silicon, and oxygen. Talc is a clay mineral composed of hydrated magnesium silicate with a chemical formula of $Mg_3Si_4O_{10}(OH)_2$.

Master Formula:

Ingredients	Justification	Standard Quantity	Quantity Used
Kaolin	Absorbent	10%	
Calcium Carbonate	Absorbent Material	10%	
Zinc Oxide	covering agent	20%	
Magnesium Carbonate	Absorbent Material	10%	
Talc	Slip character	45%	
Zinc Stearate	Adhesive Material	5%	
Perfume		1%	

Procedure: Pass all the ingredients through a 60 mesh sieve. Mix the perfume throughout with magnesium carbonate in glass mortar. Pass it through 20 mesh sieve allow it to macerate until it is homogenous, ensures complete absorption of perfume in carbonate.

Calculations:

1) **Calculation for final Quantity of product**
   a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A

= A+(10/100) x A
= A + (0.1 x A)

= __**Y**__ g

2) Calculation for quantity of ingredient used :

a) The amount of kaolin required for 100% = 10 %

The amount of kaolin required for Y g = $\frac{Y \times 10}{100}$

= ____ g

Use of the product: As beauty aid to provide smooth finish to the skin by masking blemishes and shine due to moisture, also makes skin pleasant to touch

Storage: store in a tightly sealed container

Usage: for external use only

Label:

TALCUM POWDER __g		
Composition:	**BATCH NO.**	**MFG. DATE:**
Category: Cosmetic	**MFG. LIC. NO.:**	**EXPIRY DATE:**
FOR EXTERNAL USE ONLY.		
Use: **Storage:**		
MFG. BY:	Batch:	Roll No.:

Result : 1) _____ of talcum powder was prepared and submitted.

2) Appearance :

Experiment No: 05

Aim: To prepare and submit ___g of Lather shaving cream in a suitable labelled container

Requirements: Beaker, Mortar-pestle, measuring cylinder, spatula, glass rod, evaporating dish etc.

Shaving cream or shave cream is a category of cream cosmetics used for shaving preparation. The purpose of shaving cream is to soften the hair by providing lubrication.

Different types of shaving creams include aerosol shaving cream (also known as shaving foam), latherless shaving cream (also called brushless shaving cream and non-aerosol shaving cream), and lather shaving cream or lathering shaving cream. The term shaving cream can also refer to the lather produced with a shaving brush from shaving soap or a lather shaving cream.

Shaving creams commonly consist of an emulsion of oils, soaps or surfactants, and water. In addition to soap, lather shaving creams include a humectant for softer consistency and keeping the lather moisturised. Brushless shaving creams, on the other hand, don't contain soap and so don't produce lather.

Theory: Stearic acid and oleic acid from the coconut oil react with alkali to form soap. Glycerine serves as humectants and emollient. Unsaponified coconut oil helps to impact super fatting ability to the cream and to stabilize the foam by retaining moisture. Menthol provides a cooling effect. Hydroquinone acts as antioxidant. Whereas lemon oil / peppermint oil serves as a perfume.

Master formula:

Ingredients	Justification	Standard quantity	Quantity used
Stearic acid	Fatty acid charge	32 %	
Coconut oil		6.4%	
Triethanol amine	Emulsifier & Surfactant	1%	
Glycerine	Humectant	9%	
Potassium hydroxide	Alkali for saponification	7.11%	
Sodium hydroxide		0.35%	
Menthol	Soothing and cooling effect	0.5%	
*Hydroquinone	Anti-oxidant	0.2%	
Perfume oil		q.s	
Purified water		43.44%	
Color		q.s	

Procedure:

1) Melt stearic acid, coconut oil and hydroquinone in an evaporating dish on a steam bath.
2) In another beaker place the water required and dissolve the menthol in it, followed by the given quantity of triethanolamine, potassium hydroxide, sodium hydroxide and glycerine
3) Simultaneously heat both the vessels on steam bath until alkali is dissolved and temperature is equal to stearic acid melt beaker.
4) Run the melted stearic acid into hot alkali solution with agitation (keep the dish on steam bath while mixing the contents).
5) Continue stirring hot rapidly until the soap is smooth and homogenous.
6) When cooled approximately to 45°C add perfume and color.

Mix it well. Fill into collapsible tube.

Calculations:

1) **Calculation for final Quantity of product**
   a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A
   = A+(10/100) x A
   = A + (0.1 x A)
   = **Y** g

2) **Calculation for quantity of ingredient used :**
   a) The amount of stearic acid required for 100% = 32 %

   The amount of stearic acid required for Y g = $\frac{Y \times 32}{100}$

   = ____ g

Observations:

d) Weight of empty beaker = _____ g
e) Weight of beaker + final product = _____g
f) Weight of final product = (b-a) g

Use of product: Cream cosmetics used for shaving preparation.

Storage: It should be stored in a cool and closed containers or collapsible tube.

Label:

LATHER SHAVING CREAM __g		
Composition:	**BATCH NO.**	**MFG. DATE:**
Category:	**MFG. LIC. NO.:**	**EXPIRY DATE:**
FOR EXTERNAL USE ONLY.		
Use: **Storage:**		
MFG. BY:	Batch:	Roll No.:

Result : 1) _____ of lather shaving cream was prepared and submitted.

2) Appearance :

Experiment No: 06

Aim: To device a suitable preparation for tooth enamels -toothpaste

Requirements: Calcium carbonate, preservatives, sweetener, mortar & pestle etc.

Master Formula:

Ingredients	Justification	Standard Quantity	Quantity used
Calcium carbonate	abrasive	28 g	
Glycerin	humectant	0.5 g	
Sodium lauryl sulphate	Surfactant	11 g	
Sodium saccharin	sweetener	0.75 g	
Para hydroxyl benzoic acid	preservative	0.05 g	
menthol	Flavouring/Cooling effect	0.75 g	
water		q.s.	
colour		q.s.	

Procedure:

1) All the other ingredients are weighed and taken in the beaker.
2) Calcium carbonate, sodium lauryl sulfate, glycerine and saccharin sodium are mixed in water.
3) p-hydroxy benzoic acid, menthol are added to the above mixture.
4) This solution is added dropwise in mortar containing herbal ingredients, and triturate well until a paste consistency is formed.

Calculations:

3) Calculation for final Quantity of product

a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A

= A+(10/100) x A

= A + (0.1 x A)

= __**Y**__ g

4) Calculation for quantity of ingredient used :

a) The amount of calcium carbonate required for 100% = 28 g

The amount of calcium carbonate required for Y g = $\frac{Y \times 28}{100}$

= ____ g

Observations:

g) Weight of empty beaker = _____ g
h) Weight of beaker + final product = _____g
i) Weight of final product = (b-a) g

Use of product: As dentrifice in conjunction with tooth brush.

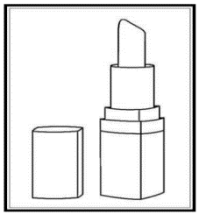
Storage: It should be stored in a cool place. closed containers or collapsible tube.

Label:

TOOTHPASTE __g		
Composition:	**BATCH NO.**	**MFG. DATE:**
Category:	**MFG. LIC. NO.:**	**EXPIRY DATE:**
FOR EXTERNAL USE ONLY.		
Use: **Storage:**		
MFG. BY:	Batch:	Roll No.:

Result : 1) _____ of toothpaste was prepared and submitted. 2) Appearance :

Experiment No: 07



Aim: To prepare and submit ___ g of Lipstick in a suitable labelled container

Requirements: beakers, burners, beeswax, spatula, etc.

Theory:

Lipstick may be basically defined as dispersion of the colouring matter in a base consisting of a suitable blend of oils, fats and waxes with suitable perfumes and flavours moulded in the form of sticks to impart attractive gloss and colour, when applied on lips. Lipsticks provide moist appearance to the lips accentuating them and disguising their defects.

Ingredients	Justification	Standard quantity	Quantity Used
Castor oil	(dissolving liquid)	54.0 g	
Anhydrous lanoline	(Emollient)	11.0 g	
Candelilla wax	(hardening agent)	9.0 g	
Isopropyl myristate	(blending agent)	8.0 g	
White bees wax	(stiffening agent)	5.0 g	
Carnauba wax	(provides rigidity)	3.0 g	
Ozokerite wax	(increase melting point)	3.0 g	
Eosin	(dye)	2.0 g	
Rose flavour	(perfume)	q.s	
Tocopherol	(antioxidant)	q.s	
Paraben	(preservative)	q.s	

Procedure :

1) Melt White beeswax, Anhydrous lanoline, Candelilla wax, Carnauba wax Ozokerite wax to 75°C-85°C.
2) Simultaneously heat castor oil with tocopherol and paraben to temperature slightly above waxy medium. Heating has to be carried out using water bath(∼90°C).
3) Add both the phases ensuring that they are maintained at the same temperature and stir continuously until semisolid emulsion is formed (the mass is stirred thoroughly, but gently to avoid entrapment of air.).
4) At around 40°C add perfume and further stir to uniformly distribute the perfume in the emulsion.
5) The mould is lubricated with liquid paraffin or isopropyl myristate before pouring the mass into the mould.

Calculations:

1) **Calculation for final Quantity of product**
   a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A
      = A+(10/100) x A
      = A + (0.1 x A)
      = __**Y**__ g

2) **Calculation for quantity of ingredient used :**
   a) The amount of castor oil required for 100g = 54 g

      The amount of castor oil required for Y g = $\frac{Y \times 54}{100}$

      = ____ g

Observations:

a) Weight of empty beaker = _____ g
b) Weight of beaker + final product = _____g
c) Weight of final product = (b-a) g

Use of product: As lip accentuating agents.

Storage: It should be stored in a cool place.

Label: For external use only.

LIPSTICK __g	
Composition:	**BATCH NO.** **MFG. DATE:**
Category:	**MFG. LIC. NO.:** **EXPIRY DATE:**
FOR EXTERNAL USE ONLY.	
Use: **Storage:**	
MFG. BY: Batch:	Roll No.:

Result : 1) _____ of Lipstick was prepared and submitted.

2) Appearance :

Hair Dyes

A variety of hair colours are observed between the people living in east and the people living in west. The agents that are responsible for variety of hair colours are only two which are Pheomelanins and eumelanins. Pheomelanins impart different shades of red and yellow whereas, eumelanins impart different shades of dark brown and black.

Classification of hair colourants

1. Temporary hair colourants.
2. Semi-permanent hair colourants/Direct
3. Oxidative dyeing systems: It includes:

(a) Semi-permanent hair colourants.
(b) Permanent hair colourants.

4. Gradual hair colourants.
5. Natural dyes.

Experiment No: 08

Aim: To prepare and submit ___ g of Hair dye in a suitable labelled container

Requirements: Stearic acid, Triethanolamine, Beeswax, Carnauba wax, beakers, glass rod.

Theory: Temporary Hair Colorants, they are leave-in preparations. The hair is not rinsed after the application of the colorant. The colorant is easily removed with one wash using a shampoo because they are absorbed in to the cuticle and cannot enter into the cortex of the hair. Basically temporary hair colorants consist of dye stuffs and acid. The different dye stuffs are acid dyes, basic dyes, metalized dyes and disperse dyes. Chemically the dye stuffs are azo dyes, anthraquinone dyes, benzoquinoneimine dyes, triphenyl methane dyes, phenazanic dyes and xanthenic dyes. The hair colourants are available in different formulations like powders, crayons, liquids and shampoos.

Master Formula:

Ingredients	Justification	Standard quantity (for 100g)	Quantity Used
Stearic acid	(anionic surfactant)	15g	
Triethanolamine	(surfactant)	7.0 g	
Beeswax	(wax)	50 g	
Carnauba wax	(wax)	13 g	
Ozokerite	(wax)	7.0 g	

Glyceryl mono stearate	(surfactant)	12.0 g	
Tragacanth	(gum)	2.0 g	
color		q.s	

Procedure:

1) Take Triethanolamine, glyceryl monostearate and tragacanth in a beaker and heat to 70°C.
2) Add stearic acid to the above mixture and heat to 75°C.
3) Melt Beeswax and carnauba wax separately at 70-80°C.
4) Add the molten waxes to the above mixture and stirr well.
5) Color is added and the mixture is stirred well. This mixture is then poured into the moulds.

Calculations:

1) **Calculation for final Quantity of product**
   a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A
   = A+(10/100) x A
   = A + (0.1 x A)
   = **Y** g
2) **Calculation for quantity of ingredient used :**
   a) The amount of stearic acid required for 100g = 15 g

   The amount of stearic acid required for Y g = $\frac{Y \times 15}{100}$

   = ____ g

Observations:

a) Weight of empty beaker = _____ g
b) Weight of beaker + final product = _____g
c) Weight of final product = (b-a) g

Use of product:

Storage:.

Label:

HAIR DYE __g	
Composition:	**BATCH NO.** **MFG. DATE:**
Category:	**MFG. LIC. NO.:** **EXPIRY DATE:**
FOR EXTERNAL USE ONLY.	
Use: **Storage:**	
MFG. BY: Batch:	Roll No.:

Result : 1) _____ of hair dye was prepared and submitted.

2) Appearance :

Experiment No: 09

Aim: To prepare and submit ___g of Henna hair dye in a suitable labelled container

Requirements: Henna powder etc.

Theory: Natural dyes: Since, antiquity, plant materials are looked upon as beneficial sources for various ailments and other purposes. The leaves are used as colourants: The leaves of henna are powdered and sold. The paste is formed by mixing the henna powder in hot water. The paste is directly applied on hair and a warm towel is wrapped around the head to enhance the colouring effect. lt gives reddish colour to the hair. Henna is non-toxic and non-sensitizing. The active constituent of henna is lawsone, which is chemically 2-hydroxy-l4 - napthaquinone. It is responsible for imparting the color. Indigo leaves or synthetic indigo is added to henna to alter the colour. Apart from this, pyrogallic acid and metallic salts like copper sulphate are added. An increased level of pyrogallic acid added to henna, gives darker shades.

Master Formula:

Ingredients	Justification	Standard quantity (for 100g)	Quantity Used
Powdered henna	(color)	89 g	
Pyrogallic acid *	(color)	6.0 g	
Copper sulphate	(color)	5.0 g	
coffee	(colour)	17.8	

Procedure: The paste is formed by mixing the henna powder, copper sulphate coffee in hot water.

II -Evaluation of henna hair dye

The following Physico-chemical parameters were assessed for determining the quality of prepared formulation against marketed herbal Soap.

1) Organoleptic parameters: The color, odour, texture of the prepared dye is observed with naked eye keeping it on white background.

2) pH: The pH of the prepared hair dye is checked with pH paper followed by measuring by the digital pH meter.

3) Patch test: spot the limited quantity of watery arrangement of hair dye behind the ear (more common) on or internal elbow in a space 1sq.cm and passing on it to dry. The indication of disturbance or feeing of non-health is noted, if any. Irritancy, redness, and enlarging to be noted for standard stretch as long as 24 hours if any.

4) Moisture content: A method commonly used for moisture contant determination is the loss on drying method or LOD. The crude henna (~2.0g) first weighed along with a clean evaporating dish and heated at 105°C to constant weight, weigh again after heating and calculate the total loss of weight.
The following equation is used to calculate the sample's moisture content.
%Weight = A-B/B × 100
A = weight of wet sample (g), B = weight of dry sample (g).

Calculations:

1) **Calculation for final Quantity of product**
   a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A
   = A+(10/100) x A
   = A + (0.1 x A)
   = **Y** g

2) **Calculation for quantity of ingredient used :**
   a) The amount of powdered henna required for 100g = 89 g

   The amount of powdered henna required for Y g = $\frac{Y \times 89}{100}$

   = ____ g

3) **Moisture content** %Weight = (A-B)/B × 100

= _____ %

Observations: I.

a) Weight of empty beaker = _____ g
b) Weight of beaker + final product = _____g
c) Weight of final product = (b-a) g

II. Evaluation: Organoleptic & Physico-chemical parameters of formulation

Parameter	Observations-Prepared henna dye
colour	
odour	
texture	
pH	
Patch test (if yes, mention type: redness; swelling etc.)	
Moisture content (%)	

Use of product:

Storage:.

Label:

HENNA HAIR DYE __g		
Composition:	**BATCH NO.**	**MFG. DATE:**
Category:	**MFG. LIC. NO.:**	**EXPIRY DATE:**
FOR EXTERNAL USE ONLY.		
Use: **Storage:**		
MFG. BY:	Batch:	Roll No.:

Result : 1) _____ of henna hair dye was prepared and submitted.

2) Appearance :

Experiment No: 10

Aim: To prepare and submit _____ g of herbal soap

Requirements: distilled water (or cooled infusion), NaOH, Coconut oil, Olive/Sunflower/sweet almond/castor oil, organic herbs, flower petals, organic essential oils

Theory: Soaps are sodium or potassium salts of long chain fatty acids. When triglycerides in fat/oil react with aqueous NaOH or KOH, they are converted into soap and glycerol. This is called alkaline hydrolysis of esters. Since this reaction leads to the formation of soap, it is called the **Saponification** process. A pharmaceutical or medication that contains antibacterial and antifungal ingredients is known as an "herbal soap preparation." It's made up of plant parts including leaves, stems, roots, and fruits, and it's used to treat damage, disease, and keep people healthy

Master Formula:

Ingredients	Standard Quantity	Quantity used
NaoH	27.28g	NaoH
Distilled water/ cooled infusion (cinnamon)	67ml	
Coconut oil	52ml	
Olive oil	88.5ml	
Castor oil	11ml	
Sunflower/ sweet almond oil	25.87ml	
Herbs/flowers/essential oils	q.s	

Procedure: I -Preparation

1) In a vessel (porcelain/ stainless steel/glass) add the required distilled water or infusion (prepared by previously boiling the herb with water).
2) Add the NaoH and stir well till it completely dissolves and cool it for around 30 mins or till it reaches 36-47 °C and keep it aside.
3) Melt coconut oil over low heat and combine with the other oils.
4) Cool these combined oils till they reach 35- 45 °C. Pour the cooled alkali solution into the warm oils & mix thoroughly till soap reaches trace & pour this into the mould add the flowers etc. and texture the soap with a spoon.
5) Cover soap lightly with wax paper and allow it to set for one or two days.

II -Evaluation of herbal soap

The following Physico-chemical parameters were assessed for determining the quality of prepared formulation against marketed herbal Soap.

1) Organoleptic parameters: The color and clarity of the prepared soap is observed with naked eye keeping it on white background. The odour of the soap is checked.

2) pH: The pH of the prepared soap is measured by the digital pH meter. The prepared formulation(2.5g) is dissolved in 50 ml distilled water(without agitation to avoid foaming) and kept for 2 h. pH measurement of the solution is done using a previously calibrated pH meter.

3) Moisture content: Weigh 2.0g of soap sample and note this as the "wet weight of the sample." this wet sample is dried in a hot air oven to a constant weight at a temperature not to exceed 115 °C. After cooling, the sample is weighed once more to determine its "dry weight."
The following equation is used to calculate the sample's moisture content.
%Weight = A-B/B × 100
A = weight of wet sample (g), B = weight of dry sample (g).

Calculations: 1) Calculation for final Quantity of product

a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A
= A+(10/100) x A
= A + (0.1 x A)

= **Y** g

2) Calculation for quantity of ingredient used :

a) The amount of NaOH required for 100g = 27.28 g

The amount of NaOH required for Y g = $\frac{Y \times 27.28}{100}$

= ____ g

3) **Moisture content** %Weight = (A-B)/B × 100

= ____ %

Observations: I.

a) Weight of empty beaker = _____ g
b) Weight of beaker + final product = _____g
c) Weight of final product = (b-a) g

II. Evaluation: Physico-chemical parameters of formulation

Parameter	Observations-Prepared herbal soap
colour	
odour	

clarity	
pH	
Moisture content (%)	
Free alkali content (%)	

- **Use of product**: As a skin cleansing alternative.
- **Storage**: It should be stored in a cool and dry place

Label:

HERBAL SOAP__g	
Composition:	**BATCH NO.** **MFG. DATE:**
Category:	**MFG. LIC. NO.:** **EXPIRY DATE:**
FOR EXTERNAL USE ONLY.	
Use: **Storage:**	
MFG. BY: Batch:	Roll No.:

Result : 1) _____ of herbal soap was prepared and submitted.

2) Appearance :

Face pack is the smooth powder which is used for facial application. These preparations are applied on the face in the form of liquid or pastes and allowed to dry and set to form film giving tightening, strengthening and cleansing effect to the skin.

They are usually left on the skin for ten to twenty five minutes to allow all the water to evaporate, the resulting film thus contracts and hardens and can easily be removed.

The warmth and tightening effect produced by application of face pack produces the stimulating sensation of a rejuvenated face, while the colloidal and adsorption clays used in these preparations remove the dirt and grease from the skin of the face.

When the applied face pack is eventually removed skin debris and deposited dirt gets removed with it. Face packs are basically additives delivering some additional benefits. Different types of herbal face packs are used for different types of skin. Herbal face packs are helps to reduce wrinkles, pimples, acne and dark circles.

These packs are available in various types and forms and broadly classified into the following categories:

1. Plastic masks: Wax based, latex based, or vinyl based

2. Hydrocolloid masks: Gel masks (ready to use)

3. Argillaceous masks: Clay based or earth based (ready to use or dry powder)

Experiment No: 11

Aim: To prepare and submit ___g of papaya face pack in a suitable labelled container

Requirements: beakers, mortar & pestle, measuring cylinder, watch glass etc.

Theory: Papaya has often been called the angel fruit. This sweet fruit with butter-like consistency has numerous health and beauty benefits. Papayas have gained popularity as a natural home treatment, and for their use in skin and hair products. It has also been shown that papayas have high content of vitamins A, B, and C, and its proteolytic enzymes, such as papain and chymopapain also have antibacterial, antifungal, and antiviral properties. While as a common practice, the black jelly-like seeds of papaya are thrown away, researches have surprised by proving various benefits of papaya seeds for skin.

Master Formula:

Ingredients	Justification	Quantity for 100g	Quantity Used
Rice Flour	exfoliant	8.0 g	
Gram Flour	Anti-acne agent	8.0 g	
Vitamin C	enhances skin glow	4 capsules (1 capsule≈ 500 mg)	
Honey	Moisturizer	10 mL	
Coconut Oil	emollient	10 mL	
milk		40 mL	
Papaya seeds (optional)		8.0 g	
Papaya paste/powder		3.0 g	
Rose extract		q.s.	

Procedure: I- Preparation

Collect all the ingredients mentioned in the above list (except the coconut oil) and triturate this mixture to make a paste like a rough sand. Add the coconut oil to the mixture at the time of application.

II. Evaluation:

1) Organoleptic: Morphological evaluation of the herbal face pack by its color, odor, appearance, texture etc. The external characters of the formulation are examined and noted in a tabular column.

2) Irritancy test: Mark an area (1sq.cm) on the left-hand dorsal surface. Definite quantities of prepared face packs are applied to the specified area and time is noted. Irritancy, is checked if any for regular intervals up to 24 hrs and reported.

3) pH : dissolve a definite quantity in 25 mL of distilled water let it stand for 20 minutes after thorough mixing , filter the solution and test the filtrate with a previously calibrated digital pH meter.

Calculations:

1) **Calculation for final Quantity of product**
   a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A
   = A+(10/100) x A
   = A + (0.1 x A)
   = **Y** g

2) **Calculation for quantity of ingredient used :**
   a) The amount of rice flour required for 100g = 8.0 g

   The amount of rice flour required for Y g = $\frac{Y \times 8.0}{100}$

   = ____ g

Observations:

a) Weight of empty beaker = _____ g
b) Weight of beaker + final product = _____g
c) Weight of final product = (b-a) g

Evaluation: Organoleptic & Physico-chemical parameters of formulation

Parameter	Observations-Prepared herbal face pack
colour	
odour	
Appearance	
texture	
pH	
Irritancy (if yes, mention type: redness; swelling etc.)	

Use of product: as a face exfoliating product and provides a soothing, calming and cooling effect on the skin.

Storage: It should be stored in a cool and dry place in a closed container

Label:

HERBAL FACE PACK__g		
Composition:	**BATCH NO.**	**MFG. DATE:**
Category:	**MFG. LIC. NO.:**	**EXPIRY DATE:**
FOR EXTERNAL USE ONLY.		
Use: **Storage:**		
MFG. BY:	Batch:	Roll No.:

Result : 1) _____ of herbal face pack was prepared and submitted.

2) Appearance :

Experiment No: 12

Aim: To prepare and submit ___ g of turmeric face pack in a suitable labelled container

Requirements: beakers, mortar & pestle, measuring cylinder, watch glass etc.

Theory: Turmeric is mainly used to rejuvenate the skin. It delays the signs of aging like wrinkles and also possesses other properties like antibacterial, antiseptic and anti-inflammatory. It is best source of blood purifier. It is effective in treatment of acne due to its antiseptic and antibacterial properties that fight pimples and breakouts to provide a youthful glow to your skin. It also reduces the oil secretion by the sebaceous glands.

Prepared face pack

Master Formula:

Ingredients	Justification	Quantity for 100g	Quantity used
Multani Mitti (Bentonite clay)	Versatile clay	12.5g	
Turmeric powder		10g	
Aloe Vera	moisturizer	5g	
Sandal wood	anti-tanning and anti-aging property.	12.5g	
Orange peel	radial scavenging	5g	
Neem	anti-acne	2g	
Nutmeg	Reduces scars	3 g	
Distilled water	Vehicle	q.s	

Procedure: Collect all the ingredients mentioned in the above list and triturate this mixture to obtain the face pack of desired consistency using distilled water.

Evaluation:

1)Irritancy test: Mark an area (1sq.cm) on the left-hand dorsal surface. Definite quantities of prepared face packs are applied to the specified area and time is noted. Irritancy, is checked if any for regular intervals up to 24 hrs and reported.

2) pH : dissolve a definite quantity in 25 mL of distilled water let it stand for 20 minutes after thorough mixing , filter the solution and test the filtrate with a previously calibrated digital pH meter.

Calculations:

3) **Calculation for final Quantity of product**

a) Ingredients are taken 10% in excess to compensate for the losses during preparation = A + 10% of A

= A+(10/100) x A

= A + (0.1 x A)

= **Y** g

4) Calculation for quantity of ingredient used :

a) The amount of bentonite clay required for 100g = 12.5 g

The amount of bentonite clay required for Y g = $\frac{Y \times 12.5}{100}$

= ____ g

Observations:

d) Weight of empty beaker = _____ g

e) Weight of beaker + final product = _____g

f) Weight of final product = (b-a) g

Evaluation: Physico-chemical parameters of formulation

Parameter	Prepared herbal face pack
colour	
odour	
Appearance	
texture	
pH	
Irritancy (if yes, mention type: redness; swelling etc.)	

Use of product: as a face exfoliating product and provides a soothing, calming and cooling effect on the skin.

Storage: It should be stored in a cool and dry place in a closed container

Label:

HERBAL FACE PACK__g	
Composition:	**BATCH NO.** **MFG. DATE:**
Category:	**MFG. LIC. NO.:** **EXPIRY DATE:**
FOR EXTERNAL USE ONLY.	
Use: **Storage:**	
MFG. BY: Batch:	Roll No.:

Result : 1) _____ of herbal face pack was prepared and submitted.

2) Appearance :

Extraction of essential oils as perfumery and identification of compound

Essential oils or etheric oils mean volatile oils and are obtained from plants by steam distillation method Essential oils are used for medicinal and pharmaceutical purposes, food and food ingredients, herbal tea, cosmetics, perfumery, aromatherapy, pest and disease control, advantages of distillation methods are extracting pure and refine essential oils by evaporating the volatile essence from the plant. Essential oils can be extracted from all plants or different parts of the plant like bark, leaves, roots, wood, seeds or fruits, flowers, burgeons, branches

Water Distillation: Water distillation is used to extract of essential oils from raw or dried plants by diffusion mechanism (Fig. below). The plants are soaked in the container, which has water for preventing overheating and charring of the plants, and then heating water with plants till the steam comes out. The oil comes out and it goes to the condenser where the oil and water are collected in separation flasks. The oil collected in the top layer of hydrosol can be isolated. In this method, the extraction temperature always is below 100°C at the surface of the plants to avoid the evaporation of water and oil together . Heating systems in the extraction of essential oils using water distillation are direct fire, steam jacket, closed steam jacket, closed or open steam coil.

Advantages of water distillation :

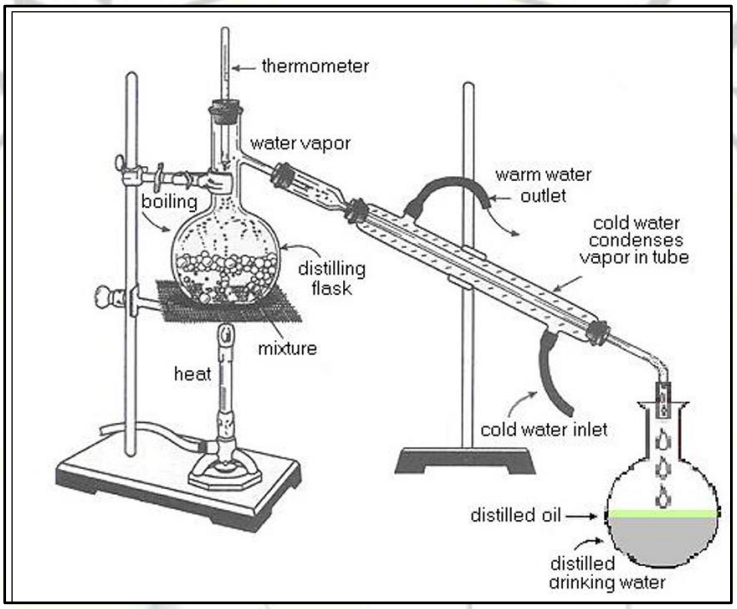
- It is widely used in the world.
- Water distillation method is inexpensive and easy to construct. It is proper for field operation.
- Boiling water causes motion of plant into distilling flask, which leads to improvement heat transfer.
- There is a direct contact between plant and boiling water.

Disadvantages of water distillation:

- Complete extraction is not possible.

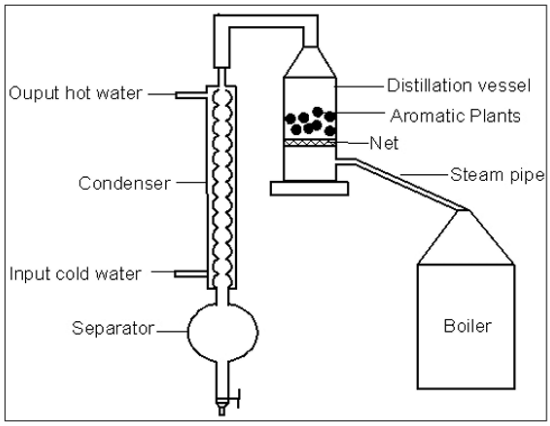
 Oil ingredients such as esters are sensitive to hydrolysis while other compounds like acyclic monoterpene hydrocarbons and aldehydes are susceptive to polymerization (water pH is mostly reduced during distillation result in readily hydrolytic reactions).

 Oxygenated ingredients like phenols have a tendency to liquefy in the distilled water, as a result water distillation is not able to removal them completely.



(Assembly for water distillation)

Steam Distillation: Steam distillation (SD) is a widespread method for isolating essential oils commercially. About 80 to 90% of the essential oils are produced by steam distillation method. This method is used for extracting essential oils from fresh plant materials that have a high boiling point such as roots and seeds. Also, the essential oil in the peppermint, spearmint, oil roses and chamomile are extracted by using steam distillation method. In this method, the plant material is placed on the perforated grid, then steam is released from steam boiler to the extraction vat and passing through the plant material, and as a result the essential oil is separated from plant material by the diffusion process, and comes out with steam to the condenser, and then to the separation unit. Flow chart of the essential oil extraction by using steam distillation is shown below



(Assembly for steam distillation)

Advantages of steam distillation:

- It has high energy efficient.
- Cheapest way for extracting essential oils when on a small scale.
- Essential oils produced by steam distillation have a high quality.
- The control on the distillation rate is better.
- Working pressure can be changed according to the working conditions.
- No decomposition of in oil compounds due to steam.

Disadvantages of steam distillation

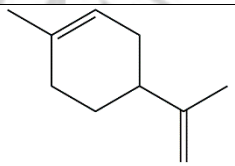
- Set up cost of a large scale of essential oil extractor by this method is high.

Experiment No: 13

Aim: Extraction of volatile oil, d-limonene from orange/lemon peel.**(Water distillation)**

Requirements: Hickman head, round bottom flask, condenser, conical flask, beaker, separating funnel, pasture pipette, orange/lemon peel.

Theory:

Limonene is a chiral molecule with two optical isomers (enantiomers). The major biological form d-limonene, the (R)-enantiomer, is used in food manufacture and medicines. It is also used as a fragrance in cleaning products, a botanical insecticide, and due to its flammability, a potential biofuel. The (S)-enantiomer, l-limonene, is also used as a fragrance but has a piney, turpentine odour.		 D-limonene (1-methyl-4-prop-1-en-2-yl-cyclohexene) is an unsaturated hydrocarbon, classed as a terpene. At room temperature it is a colourless oily liquid with the smell of oranges. Its molecular formula is C10H16 and its boiling point is 176 °C.

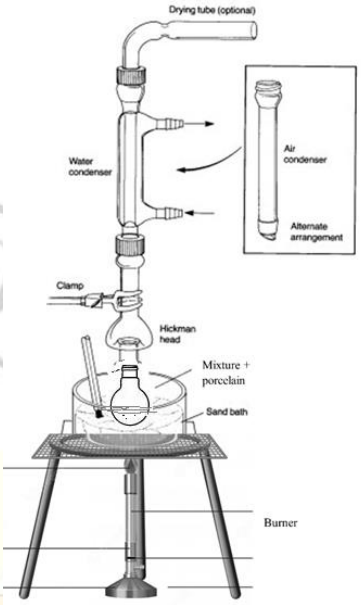
Procedure: 1.0g of crushed peels of orange/lemon and 10ml of water are taken in a round bottom flask and soaked for 10 min. The Hickman head apparatus is assembled. The orange/lemon peel water suspension is heated using a sand bath by maintaining the temperature of the sand bath at approx. 130^0C. care has to be taken that the suspension is not heated vigorously, which could result in foaming, thus could contaminate the distillate with crushed peels. The distillate from Hickman head is periodically transferred into a conical flask. Some more water is added and distillation is continued until about 8.0-10.0 mL of distillate is obtained.

The obtained distillate is extracted using Dichloromethane in a separating funnel. It is shaken well and the layers are allowed to separate. Dichloromethane layer is transferred to a clean conical flask. The extraction is repeated two more times using Dichloromethane.

Action of bromine water: Measure out approximately 1.0 mL of bromine water into each of three test tubes. Add a few drops of the limonene oil to one test tube, a few drops of cyclohexane to another, and a few drops of cyclohexene to the third. shake the test tubes well. If the bromine water is decolourised the molecule contains double bonds.*potassium permanganate can be substituted for the bromine water for use. However, students need to know the action of bromine water.

Observations:

g) Weight of empty beaker = _____ g
h) Weight of beaker + oil = _____g
i) Weight of oil = (b-a) g



Assembly for water distillation of d-limonene

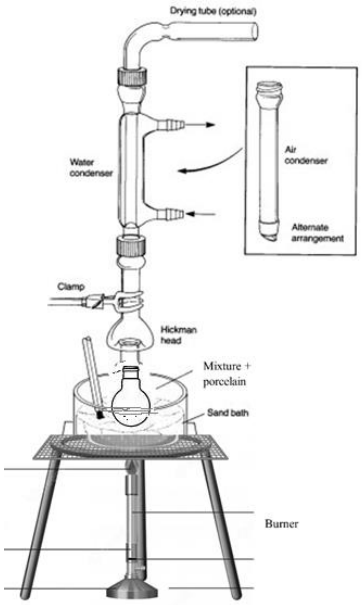
Result:

Experiment No: 14

Aim: Extraction of volatile oil, citronella oil from lemon grass**. (water distillation)**

Apparatus: Hickman head, round bottom flask, condenser, conical flask, beaker, separating funnel, pasture pipette, lemon grass.

Theory: The essential oil of the lemongrass plant known as citronella oil is characterized by a strong lemon fragrance due to the citral compound in the oil . Commercial citronella oil comprises as Ceylon citronella and Java type citronella. In industry, citronella oil is one of the ingredients used to produce ionone, vitamin A, and carotene . The aroma of the oil is widely used in soaps, detergents, and perfumes. In addition, citronella oil contains several bioactive compounds that are medicinal , antioxidants, anti-microbial, and anti-fungal . Citronella oil also functions as an insect repellent because of its high efficacy and low toxicity. Although citronella oil is widely used in the pharmaceutical, cosmetic, and food industries, its selling price is still low, so it is needed to increase its economic value by isolating its active components such as citronellal, citronellol, and geraniol.



Assembly for water distillation of citronella oil

Procedure: 1.0g of lemon grass (prefer dried) and 10ml of water are taken in a round bottom flask and soaked for 10 min. The hickman head apparatus is assembled as shown. The lemon grass water suspension is heated using a sand bath by maintaining the temperature of the sand bath at approx. 130^0C. care has to be taken that the suspension is not heated vigorously, which could result in foaming.

The distillate from hickman head is periodically transferred into a conical flask. Some more water is added and distillation is continued until about 10 ml of distillate is obtained.

The obtained distillate is extracted using Dichloromethane in a separating funnel. It is shaken well and the layers are allowed to separate. Dichloromethane layer is transferred to a clean conical flask. The extraction is repeated two more times using Dichloromethane.

Observations:

a) Weight of empty beaker = _____ g
b) Weight of beaker + oil = _____g
c) Weight of oil = (b-a) g

Result:

Experiment No: 15

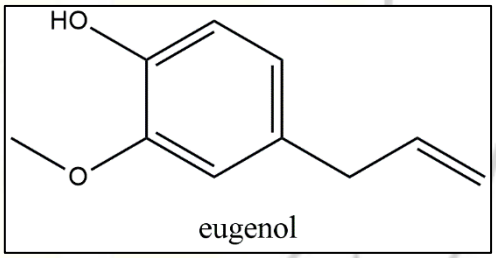
Aim: Extraction of volatile oil from cloves. (steam distillation)

Requirements: Steam distillation assembly, round bottom flask, condenser, conical flask, beaker, separating funnel, pasture pipette, cloves.

Theory:

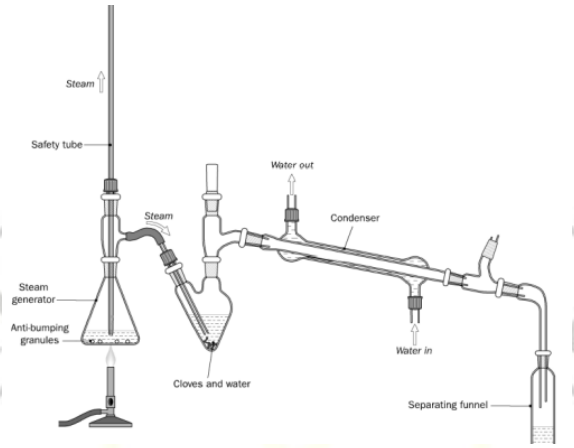
Eugenol is a phenolic component that can be obtained from a wide range of plant sources including clove oil, nutmeg oil, cinnamon extract and many other plants. It owns strong health promoting functions that make it a versatile natural ingredient. Eugenol was firstly extracted from the leaves and buds of *Eugenia caryophyllata* commonly named as clove.

Eugenol is used as a flavor or aroma ingredient in teas, meats, cakes, perfumes, cosmetics, flavorings, and essential oils. It is also used as a local antiseptic and anaesthetic. Eugenol can be combined with zinc oxide to form zinc oxide eugenol which has restorative and prosthodontic applications in dentistry.



eugenol

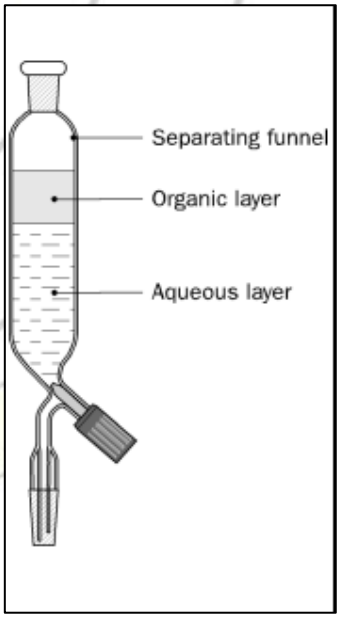
(2-methoxy-4-(prop-2-en-1-yl) phenol) is a colourless to pale yellow liquid, one of the major compounds found in Clove oil, it has a pungent taste with molecular formula $C_{10}H_{12}O_2$



(Assembly for steam distillation of clove oil)

Procedure: Set up the steam distillation assembly as figure above. Weigh 2.0g of cloves and place in the round bottom flask. Place an adequate supply of water in the steam generator, connect it to the rest of the apparatus and set it to boil. make sure that you use anti-bumping granules in the steam generator (porcelain pieces). If the level of the boiling water in the steam generator falls too low, the system will not work smoothly. Remove the heat, carefully loosen the safety valve, and top up the steam generator with hot water. Reconnect everything and resume heating. Collect the distillate. It should have a pale milky appearance. After 20 to 30 minutes disconnect the steam generator to avoid the possibility of suck-back problems and turn off the heat under it. Note the smell of the distillate.

Separation of clove oil from water: Disconnect the round bottom flask from the rest of the apparatus. Add about 8 mL of cyclohexane to the distillate in the separating funnel. Stopper the separating funnel and shake the mixture. Release any pressure build-up carefully after each shake by inverting the dropping funnel while holding the stopper and slowly opening and shutting the tap. Run the lower aqueous layer off. Collect the top layer - this contains the clove oil and the cyclohexane. Dry the organic layer by shaking with anhydrous sodium sulfate in a conical flask. If possible, allow to stand overnight, before removing the solid by filtration or decanting. Separate the more volatile cyclohexane from the clove oil by placing the mixture in a small beaker (whose mass is known) on a waterbath in a fume cupboard. The cyclohexane evaporates, leaving the clove oil behind. (Note that if the cyclohexane is to be distilled off it will boil at 81^0C,low yield of clove oil expected). Note the smell of the clove oil. Do not allow the clove oil to come in contact with your skin.



Prelab/ Post-Lab exercise:

1. Explain why the clove oil could not be distilled directly from the cloves.
2. Explain the principles by which steam distillation works
3. Suggest a reason why the clove oil is much more soluble in nonpolar solvents (than in water).

Result:

Experiment No: 16

Aim: Extraction of volatile oil from cinnamon. (steam distillation)

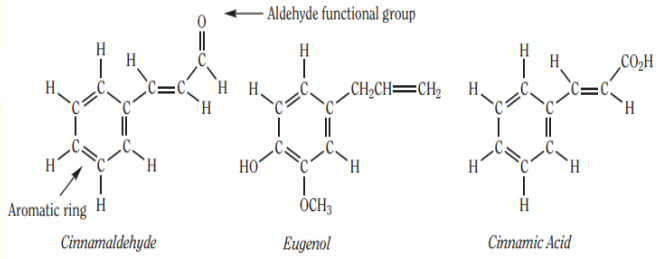
Apparatus: steam distillation assembly, round bottom flask, condenser, conical flask, beaker, separating funnel, pasture pipette, cinnamon.

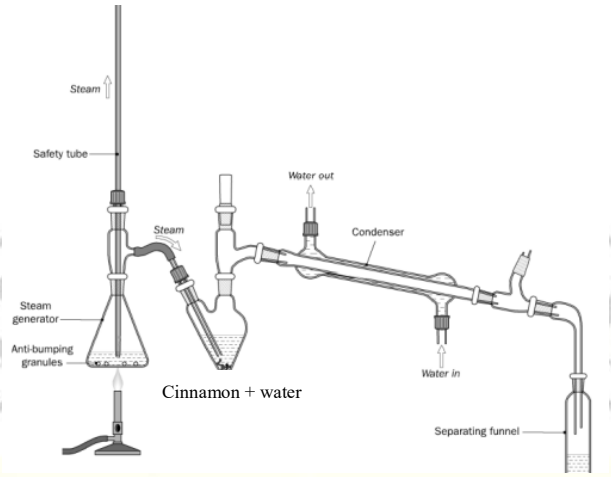
Theory:

Cinnamon is obtained from the inner bark of Cinnamomum zeylanicum, a small evergreen that is native to Sri Lanka and India. Oil of cinnamon is obtained from cinnamon bark by steam distillation. The major componant of oil of cinnamon is cinnamaldehyde (70–80%).

Cinnamaldehyde is classified as an aromatic aldehyde—it contains an aldehyde functional group and has a benzene (aromatic) ring. The compound has natural antimicrobial properties but is highly irritating to the skin. Minor components of oil of cinnamon include eugenol (10–20%) and cinnamic acid (5–10%).

Components of cinnamon oil

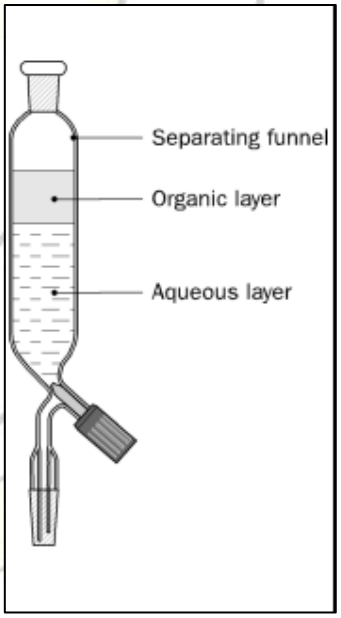




(Assembly for steam distillation of cinnamon oil)

Procedure: Set up the steam distillation assembly as figure above. . Weigh 2.0g of cinnamon sticks and place in the round bottom flask. Place an adequate supply of water in the steam generator, connect it to the rest of the apparatus and set it to boil. make sure that you use anti-bumping granules in the steam generator (porcelain pieces). If the level of the boiling water in the steam generator falls too low, the system will not work smoothly. Remove the heat, carefully loosen the safety valve, and top up the steam generator with hot water.

Reconnect everything and resume heating. Collect the distillate. It should have a pale milky appearance. The cloudiness of the distillate is due to the presence of an insoluble suspension of **Cinnamaldehyde**. Hence, the level of cloudiness is a good indicator of the amount of Cinnamaldehyde present in the distillate. 100mL of the distillate collected is transferred into an Erlenmeyer flask. To collect further amount of distillate, 100mL of water is added to the distilling flask. The next batch of distillate obtained is more clear indicating low amount of Cinnamaldehyde. The distillates are combined and transferred to a separatory funnel.



Separation of cinnamon oil from water: Disconnect the round bottom flask from the rest of the apparatus. Add about 20 mL of cyclohexane to the distillate(~50mL) in the separating funnel.(carry out separation in parts) Stopper the separating funnel and shake the mixture. Release any pressure build-up carefully after each shake by inverting the dropping funnel while holding the stopper and slowly opening and shutting the tap. Run the lower aqueous layer off. Collect the top layer - this contains the cinnamon oil and the cyclohexane. Dry the organic layer by shaking with anhydrous sodium sulfate in a conical flask. If possible, allow to stand overnight, before removing the solid by filtration or decanting.

Separate the more volatile cyclohexane from the cinnamon oil by placing the mixture in a small beaker (whose mass is known) on a waterbath in a fume cupboard. The cyclohexane evaporates, leaving the cinnamon oil behind. (Note that if the cyclohexane is to be distilled off it will boil at 81^0C.

Identification of Cinnamaldehyde:
a) Tollens test
b) Schiff's test
c) Solubility test:
Cinnamaldehyde is popularly known to be insoluble in water and soluble in oils and alcohols.

Conclusion:

Safety Precautions**:** Oil of cinnamon and cinnamaldehyde are severe skin irritants. Hexane is a flammable liquid and a dangerous fire risk—do not use around flames. Avoid contact of all chemicals with eyes and skin. Wear chemical splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Please review current Safety Data Sheets for additional safety, handling, and disposal information. Wash hands thoroughly with soap and water before leaving the laboratory.

Experiment No: 17

Aim: Extraction of rose oil from rose. (water distillation)

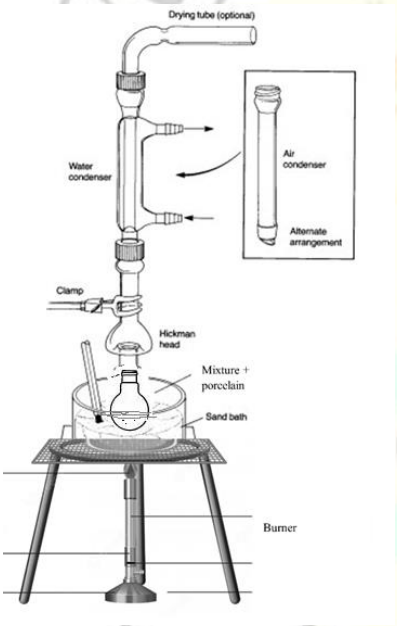
Requirements: round bottom flask, condenser, conical flask, beaker, separating funnel, pasture pipette, rose petals.

Theory: Rose oil (rose otto, attar of rose, attar of roses, or rose essence) is the essential oil extracted from the petals of various types of rose. Rose '*ottos'* are extracted through steam/water distillation, while rose *'absolutes'* are obtained through solvent extraction, the absolute being used more commonly in perfumery.

The principal constituents of rose oil are citronellol (34–55%), geraniol, (around 14%) and nerol (around 7%). Other main constituents are paraffin (around 16%), polyethyl alcohol (around 2.8%), farnesol (around 1.2%), rose oxide (a terpenoid ether, around 0.5%), eugenol (also around 1.2%).

Procedure: Set up the water distillation assembly as shown. Place 0.5g of rose and 2.0ml of water with few drops of ethanol in a round bottom flask and soak for 10 min. The rose petal water suspension is heated using a sand bath by maintaining the temperature of the sand bath at approx. 130^0C. care has to be taken that the suspension is not heated vigorously, which could result in foaming. The distillate is periodically transferred into a conical flask. Some more water is added and distillation is continued until about adequate amount of distillate is obtained.

Separation of rose oil from water: Disconnect the round bottom flask from the rest of the apparatus. Add about 10 mL(adjust according to amount of distillate) of cyclohexane to the distillate in the separating funnel. (carry out separation in parts) Stopper the separating funnel and shake the mixture. Release any pressure build-up carefully after each shake by inverting the dropping funnel while holding the stopper and slowly opening and shutting the tap. Run the lower aqueous layer off. Collect the top layer - this contains the rose oil and the cyclohexane. Dry the organic layer by shaking with anhydrous sodium sulfate in a conical flask. remove the solid by filtration or decanting. Separate the more volatile cyclohexane from the rose oil by placing the mixture in a small beaker (whose mass is known) on a waterbath in a fume cupboard. The cyclohexane evaporates, leaving the rose oil behind. (Note that if the cyclohexane is to be distilled off it will boil at 81^0C.



Identification : physical identification includes colour and odour

(Assembly for water distillation of rose oil)

Result: