A Textbook of Cosmetic Formulations

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COSMETICS

1. 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 of 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 used 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
2. Lipsticks

Definition:

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.

Ideal Characteristics of Good Lipsticks:

The ideal requirements for the formation of a good lipstick may be as follows:

- It should efficiently cover lips with colour and impart a gloss which would last long.
- It should be able to maintain the intensity of colour without any alteration in the degree of its shade.
- It should be able to adhere firmly to the lips and should not provide any greasy appearance.
- It should possess good thixotropic property so as to deposit the colour with minimum pressure.
- It should show a smear proof coloring effect.
- It should possess required plasticity and be able to maintain all the properties throughout the storage period.
- It should not be gritty.
- It should be easily dried.
- The stick should possess even firmness and should maintain its strength at varying temperatures up to 55°C.
- The stick should not dry or crumble easily.
- The lipstick should possess a pleasant fragrance and a good flavour.
- Should be safe and non-irritating to the lips.
- Result in blooming or sweating of the lips.

2.1 Formulation of lipsticks

The lipstick base is made by mixing the oils and waxes in varying proportions in order to obtain a desirable viscosity and melting point.

Composition:
The raw materials involved the formulation of the lipsticks could be as follows:

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>The solid components / waxes:</td>
<td></td>
</tr>
<tr>
<td>(a) The hydrocarbon waxes</td>
<td>White bees wax</td>
</tr>
<tr>
<td>(b) The mineral waxes</td>
<td>Ozokerite wax, ceresine wax</td>
</tr>
<tr>
<td>(c) Hard waxes</td>
<td>Carnauba wax, candelilla wax, hard paraffin</td>
</tr>
<tr>
<td>(d) Micro crystalline waxes</td>
<td></td>
</tr>
<tr>
<td>The liquid components</td>
<td>Mineral oils, vegetable oils, castor oils, butylstearate, Glycol, water, silicon-fluids, IPM (isopropyl maleate)</td>
</tr>
<tr>
<td>The softening components</td>
<td>Anhydrous lanoline, lanolin cocoa butter, lecithin, petrolatum</td>
</tr>
<tr>
<td>The coloring agents</td>
<td>Carmine, dyestuff stain, pigmented stain, lakes etc.</td>
</tr>
<tr>
<td>Pearlescent pigments</td>
<td>Guanine crystals, bismuth oxychloride</td>
</tr>
<tr>
<td>Opacifying agents</td>
<td>Titanium dioxide</td>
</tr>
<tr>
<td>Perfumeries</td>
<td>Rose oil, cinnamon oil, lavender oil etc.</td>
</tr>
<tr>
<td>Miscellaneous agents :</td>
<td></td>
</tr>
<tr>
<td>(a) Preservatives</td>
<td>Parabens</td>
</tr>
<tr>
<td>(b) Antioxidants</td>
<td>BHA, BHT, tocopherol etc.</td>
</tr>
<tr>
<td>(c) Flavouring agents</td>
<td>Cinnamoniol, spearmint oil etc.</td>
</tr>
</tbody>
</table>

![Fig. 2-2. Lipsticks](image)

1. The Solid Components/waxes: the solid components are responsible for the final structure of the product by solidifying the liquid matrix. The materials required for attaining a reasonable body, hardness, melting point and shrinkage necessary for the easy release of the mould are together referred to as natural waxes.
The solid components of the formulation are mostly natural waxes which may be classified as follows:

(a) The hydrocarbon waxes: Example: White bees wax
(b) The mineral waxes: Example: Ozokerite, ceresine
(c) Hard waxes: Example: Carnauba wax, candelilla wax, hard paraffin etc.
(d) Micro crystalline waxes

(a) Hydrocarbon Waxes:

**White Bees wax:** It is a so known as the common wax and forms the oily base in the formulation of lipsticks.

Source: It is naturally obtained from honey combs of the honey bee Apis mellifera.

Melting Point: the ranges between 62 - 65°C.

Concentration: It is used in concentrations of about 3-10% of the total formulation.

Available Forms: It is available in the form of blocks, pills, slabs and cakes. The commercially available bleached form is widely used.

**Uses:**

1. It forms an important base and is extensively used for entrapping castor oil.
2. It has good plastic property and can be readily deformed when it is warmed.
3. It is used as a traditional stiffening agent for lipsticks.
4. It forms a good base in the formulation of moulded products.

**Advantages:**

1. It is compatible with vegetable minerals and animal waxes.
2. It can be moulded into required form.

**Disadvantage:** When it is used at a concentration of more than 20%, it forms a dull film on the surface of the lips. It is usually mixed along with other waxes such as Ozokerite wax, carnauba wax and candelilla wax.

(b) Mineral Waxes: They are not popular and have been replaced by the microcrystalline waxes but still used with the same names. They are:

(i) Ozokerite Wax:

Source: It is a type of amorphous hydrocarbon obtained naturally, from bituminous products.

Melting Points: It is available in various grades with melting point ranging between 56°C to 82°C.
Concentration: It is used in a concentration range of between 5 to 10%.

**Uses:**

1. It is used in order to increase the Melting point of the base.
2. It is also efficient in promoting the formulation of a fine crystalline wax gel and thus ensures the maximum retention of the Oil matrix.
3. It can be easily transformed into required shapes.

**Advantage:** It is easily available in various grades.

**Disadvantage:** It may be subjected to adulteration.

(ii) **Ceresine Wax:**

Source: It is also obtained naturally from the bituminous products like the Ozokerite wax.

Melting Point: The melting point range is between 60-75°C.

**Uses:**

1. It is used as stiffening agents to provide firmness to the finished product.
2. It is used to increase melting point of the base.

(e) **The Hard Waxes:** These waxes are mainly responsible for the shape and the hardness of the lipsticks. They include the following waxes,

(i) **Candelilla Wax:**

Source: It is obtained from Euphorbiaceae plants such as Euphorbia cerifera and Euphorbia antisiphilitica. The extraction involves the immersing of the plant in boiling water containing sulfuric acid and later skimming off the wax that rises to the surface.

Melting Point: Its melting point ranges between 65°C-75°C.

**Uses:** It is used to increase the hardness and melting point of the product either alone or in combination with carnauba wax.

(ii) **Carnauba Wax:**

Source: It is obtained as exudates from the pores of the leaves of the Brazilian wax palm tree Copernicia prunifera. The extraction involves cutting, drying and heating of the leaves.

Melting Point: Its melting point ranges between 81 to 90°C.

Available Forms: It is available in three colors yellow, gray and brown. It is available in hard forms and soft forms.

**Uses:**
1. It is used to provide rigidity to the stick.
2. It is used in modest proportion in order to ensure high melting points.
3. It helps in moulding by shrinking the stick away from the surface of the mould in order to aid easy removal.

**Disadvantage:** It is not miscible with the other waxes and remain as a separate solid phase due to its high melting point.

(iii) Hard Paraffin:

Source: It may be present as a purified blend of several solid Hydrocarbon bases that are obtained from petroleum.

Melting Point: Its melting point ranges between 55°C - 65°C.

Uses:

1. It is occasionally used in minor quantities to improve the gloss of the finished products.
2. Imparts rigidity to the product.

**Disadvantage:** It has limited solubility in the castor oil and hence doesn't dissolve and may provide a greasy look.

(d) Microcrystalline Waxes: They are the hydrocarbons containing a long carbon chain.

Melting Point: They have wide melting points ranging between 60°C to 120°C.

Uses:

They help in maintaining the crystal structure of the lipstick and hence may prevent the sweating.

**Disadvantage:** They possess low solubility in the castor oil.

2. The Liquid Components: The liquid components are mostly constituted by the oils such as mineral oil, vegetable oil, castor oil, alcohol etc. The properties of the oils should be as follows:

(i) It should possess good dissolution properties in order to dissolve all the bromo acids.

(ii) It should possess an optimum viscosity range.

(iii) It should be colourless, odourless and tasteless.

(iv) It should be non-toxic and non-irritating.

(v) It should be easily compatible and stable.

The most commonly used liquid components may be as follows:
(a) **Mineral Oils:**

(i) They consist of a blend of hydrocarbons obtained from petroleum source.

(ii) They may be available as either light mineral oils or heavy mineral oils.

(iii) They are mostly used in order to impart gloss to the product rather than their solvent property.

(iv) They are used in concentrations of less than 5% and are not rancid.

(b) **Vegetable Oils:** The vegetable oils used may be sesame oil and olive oil. The vegetable oils provide low solubility towards staining dyes and hence less commonly used.

(c) **Castor Oil:** It is obtained from the seeds of the castor plant, Ricinus communis. It forms a most valuable lipstick base. It may be used in concentration of 40 - 50% of the total formulation. It has high viscosity and good dissolving power. It possesses stability towards oxidation. It is widely compatible with other ingredients. The high viscosity may avoid smearing off of the lipsticks.

(d) **Butyl Stearates:** They are useful for the dispersion of colour though they possess less solubility. They can readily wet the colouring pigments. They are odourless and free from rancidity.

(e) **Propylene Glycol:** It is non-toxic and possesses a sweet taste. It has good wetting property towards high colouring stains. It is always used in combination with other monoesters of propylene glycol.

(f) **Water:** It is not used as a solvent but may be used in minor quantities in order to dissolve the colour.

(g) **Silicone Fluid:** It is mostly used to aid in mould release and prevent the rub-out of the wax. It is used in minor quantities.

(h) **Isopropyl Maleate (IPM):** It is used in concentration of 2.3% to increase lip gloss. It acts as a co-solvent along with mineral oil and helps in increasing lip gloss.

3. **The Softening Agents:** They are used to increase the spread ability by softening the lipstick. The most commonly used softening agents include.

(a) **Anhydrous Lanolin:** It is also known as wool fat or woolwax. It is used at low concentration of about 0.25% in order to impart gloss, softness, emolliency and protection to the lips. The melting point ranges between 36 - 42°C.

(b) **Lanolin:** It is also referred to as hydrous wool fat. It is used in minor quantities in order to improve the covering properties of the film. It contains 25-30% of water and may result in sticky and greasy products. It aids in the dispersion of colored pigments.
(c) **Lanolin Derivatives:** They include ethers, esters and lanolin oils. They are almost none drying and thus provide a non-greasy look to the film. They are also used as blending agents or plasticizers.

(d) **Cocoa Butter:** It was used in the past due to its good emollient property. The usage has been stopped due to rancidity and surface crystallization. It provides oily look on the lips and hence imparts good gloss.

(e) **Petrolatum:** It is a hydrocarbon obtained from petroleum. It is odourless and tasteless. It is added mainly to enhance the gloss.

(f) **Lecithin:** It is used in minor quantities to impart smoothness and emollient effect. It increases the ease of application.

4. **Colouring Agents:** Colour may be imparted to the lips either by staining the lip with a dye stuff colour or by covering the lips with coloring layers. The colours used in the formulation of lipsticks are of two types:

(a) **Soluble Colours:** They are dye stuff agents which are easily soluble in oil, water and alcohol.

(b) **Insoluble Colours:** They are organic or inorganic pigments which are insoluble.

**Properties of Colouring Agents:**

- They should impart good opacity to the lips by imparting good colour.
- They should be easily and uniformly miscible with the oils used.
- The colours must be certified with the F, D and C grade.
- They should possess very low content of impurities such as arsenic, lead etc.

**The commonly used colourants for lipsticks:**

(i) **Carmine:** It was extensively used in the past and is obtained as carminic acid from the cochineal insects by extracting the insects with ammonia. The carminic acid obtained is precipitated with alum and is dried and used.

(ii) **Dye Stuff Stains:** They include eosin dyes and provide a long lasting effect on the lips by retaining the color on the lip cells. They are:

(a) **Eosin Dye:** It is used to impart orange red colour to the lips.

(b) **Acid Eosin Dye:** It has orange colour and may change to intense red colour at acidic pH of 4. But they may to toxic effects such as allergic reactions or cheilitis and hence used alone with bromo acids.

(iii) **Pigmented Stains:** They form dispersion in the solvent base. They may be either organic or inorganic. They are used in combination with metallic lakes in order to improve the intensity of the colour.
(iv) **Lakes:** They are potential pigments of many of the D and C colours. They may be adsorbed on the aluminium hydroxides, barium oxides, calcium oxides etc.,

Example: Aluminium lakes, barium or calcium lakes, strontium lakes. They are used at concentrations of about 8-10%.

5. **Pearlescent Pigments:** They are used to impart nacreous or a pearl like appearance to the product when applied on the lips. The natural pearlescent pigments may be guanine crystals obtained from fish scales. Bismuth oxychloride in 70% castor oil may also provide a lustrous look.

6. **Opacifying Agent:** It is used for opacifying or whitening of lipsticks. It can also alter the basic shade of the pigment. Various shades can be obtained by, varying the proportions. Example: Titanium Dioxide.

7. **Perfumeries:** Light floral fragrances can be used in lipsticks. They include rose oil, cinnamon oil, lavender oil etc. The fruity flavours that cover fatty odour of the oily waxes may also be used. They should be tasteless, non-irritating and compatible.

8. **Miscellaneous Agents:** They include the following:

   (a) **Preservatives:** They are used to increase life period of the product by reducing the microbial growth. Though they are anhydrous preparations, preservatives such as methyl paraben and propyl paraben may be commonly used. The concentration of the preservative should not exceed 0.1%.

   (b) **Antioxidants:** The ingredients used in the formulation may be susceptible to oxidation. This may result in the degradation of the product. Thus, antioxidants are added in order to prevent oxidation of the ingredients. The commonly used antioxidants are butylated hydroxy anisole (BHA), butylated hydroxyl toluene (BHT), tocopherol, propyl gallate, butylated hydroxyl quinines etc.

   (c) **Flavouring Agents:** They are included in order to impart good flavor to the product. They may include the spearmint oil, cinnamon oil etc. Along with the flavouring agents, sodium saccharin and the ammonium glycyrrhizate may also be used in order to improve the taste.

The various formulae for the preparation of lipsticks are as follows:

<table>
<thead>
<tr>
<th>Formula 1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Castor oil (dissolving liquid)</td>
<td>54 g</td>
</tr>
<tr>
<td>Anhydrous lanoline (Emollient)</td>
<td>11 g</td>
</tr>
<tr>
<td>Candelilla wax (hardening agent)</td>
<td>9 g</td>
</tr>
<tr>
<td>Isopropyl myristate (blending agent)</td>
<td>8 g</td>
</tr>
<tr>
<td>White bees wax (stiffening agent)</td>
<td>5 g</td>
</tr>
<tr>
<td>Carnauba wax (provides rigidity)</td>
<td>3 g</td>
</tr>
<tr>
<td>Ozokerite wax (increase melting point)</td>
<td>3 g</td>
</tr>
</tbody>
</table>
### Formula 2

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Castor oil (dissolving liquid)</td>
<td>54 g</td>
</tr>
<tr>
<td>lanolin (Emollient)</td>
<td>8 g</td>
</tr>
<tr>
<td>Candelilla wax (hardening agent)</td>
<td>6 g</td>
</tr>
<tr>
<td>Carnauba wax (provides rigidity)</td>
<td>2.5 g</td>
</tr>
<tr>
<td>Ozokerite wax (increase melting point)</td>
<td>2.5 g</td>
</tr>
<tr>
<td>bees wax (stiffening agent)</td>
<td>6 g</td>
</tr>
<tr>
<td>Isopropyl myristate (blending agent)</td>
<td>4 g</td>
</tr>
<tr>
<td>Halogenated fluorescence (color)</td>
<td>3 g</td>
</tr>
<tr>
<td>Lakes (color)</td>
<td>12 g</td>
</tr>
<tr>
<td>propyl Paraben (preservative)</td>
<td>0.2 g</td>
</tr>
<tr>
<td>Rose oil (perfume)</td>
<td>0.8 g</td>
</tr>
<tr>
<td>Rose oil (perfume)</td>
<td>0.8 g</td>
</tr>
</tbody>
</table>

### Formula 3

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Castor oil (dissolving liquid)</td>
<td>27 g</td>
</tr>
<tr>
<td>bees wax (stiffening agent)</td>
<td>20 g</td>
</tr>
<tr>
<td>Ozokerite wax (increase melting point)</td>
<td>10 g</td>
</tr>
<tr>
<td>Carnauba wax (provides rigidity)</td>
<td>5.5 g</td>
</tr>
<tr>
<td>lanolin (covering agent/Emollient)</td>
<td>5 g</td>
</tr>
<tr>
<td>Paraffin(stiffening agent)</td>
<td>3 g</td>
</tr>
<tr>
<td>Isopropyl myristate (blending agent)</td>
<td>3 g</td>
</tr>
<tr>
<td>Cetyl alcohol (co-solvent)</td>
<td>2 g</td>
</tr>
<tr>
<td>Propylene glycol (humectant)</td>
<td>11 g</td>
</tr>
<tr>
<td>Propylene glycol monoricinoleate (humectant)</td>
<td>4 g</td>
</tr>
<tr>
<td>Eosin (dye)</td>
<td>2.5 g</td>
</tr>
<tr>
<td>Color</td>
<td>10 g</td>
</tr>
<tr>
<td>Rose oil (perfume)</td>
<td>q. s</td>
</tr>
<tr>
<td>Paraben (preservative)</td>
<td>q. s</td>
</tr>
<tr>
<td>Tocopherol (antioxidant)</td>
<td>q. s</td>
</tr>
</tbody>
</table>

### 2.2 Preparation of lipsticks:

Successful preparation of lipstick shades depend upon the adequate dispersion of the lake colours in the lipstick mass. It is advisable to prepare the dispersion of lake colours in castor
oil. Dispersions are generally prepared by milling about 25% concentrations of lakes in castor oil.

Method of Preparation:

- If a solvent is used for the dissolution of bromo acid, the solution is first prepared and set aside until required.
- If commercial colour pastes are not being used, then lake colours are first dispersed by mixing with suitable quantity of castor oil.
- The colour paste obtained is passed through a triple roller mill until it becomes smooth and free from agglomerates and gritty particles.
- The colour mixture is then mixed with the bromo acid mixture.
- All the ingredients of the base are identified and arranged in the increasing order of their melting points.
- This mixture is remilled until it is perfectly smooth.
- Preservatives and anti-oxidant are dissolved in remaining oil and are added to the mixture.
- Finally, the perfume is added and the mass is stirred thoroughly, but gently to avoid entrapment of air.
- Automatic ejection mould is preferred for the large scale production.
- The mould is lubricated with liquid paraffin or isopropyl myristate before pouring the mass into the mould.
- It is important to prevent settling down of the coloring mass when the moulds are chilled. Lubrication facilitates easy removal of sticks.

2.3 Evaluation of Lipsticks

The evaluation studies are important in order to determine the efficiency, stability and the consistency of the finished product. The evaluation tests for the lipsticks are as follows:

1. Melting Point Determination Test: The determination of melting point is done in order to determine the storage characteristics of the product. The inciting point of lipstick base should be between 60 to 65°C in order to avoid the sensation of friction or dryness during application. The method of determination is known as capillary tube method:

   (a) In this method, about 50 mg of lipstick is taken and is inserted into a glass capillary tube open at both ends.
   (b) The capillary tube is ice cooled for about hrs and then placed in a beaker containing hot water and a magnetic stirrer.
   (c) The temperature at which material starts moving through the capillary is said to be the melting point temperature.
   (d) Another important parameter is the droop point which determines the temperature at which the product starts oozing out the oil and becomes flattened out.
   (e) The melting point should be higher than the droop point which determines the safe handling and storage of finished product.
2. **Breaking Load Point Test:** This test is done in order to determine the strength and hardness of the lipstick. In this method, the lipstick is placed horizontal position 1 inch from the base and weights with increasing loads are attached to it. The weight at which the lipstick starts breaking, known as the breaking load point. The test shall be carried out in specific condition and at about 25 °C temperatures.

3. **Determination of thixotropic character:** This is a test for determining the uniformity in viscosity of base. The instrument used for the determination of thixotropic character is known as the penetrometer.

4. **Microbiological tests:** The test is carried out in order to determine the extent of contamination either from the raw materials or mould. The test involves the plating of known mass of sample on two different culture media for the growth of microorganism and incubating them for a specific period of time. The extent of contamination can be estimated by counting the number of colonies.

5. **Test for rancidity:** the oxidation of oil such as castor oil and many other ingredients may result in bad odour and taste and also result in a sticky product. The test for rancidity can be done by using hydrogen peroxide and determining its peroxide number.

6. **Test for the Application Force:** This is a test to determine the force to be applied during application. In this method, two lipsticks are cut to obtain flat surfaces which are placed one above other. A smooth paper is placed between them which is attached to a dynamometer to determine force required to pull the paper indicates the force application.

7. **Storage Stability:** This test is done in order to determine the stability of product during storage.

8. **Stability to Oxidation:** The oxidation characteristics of the finished product are determined in order to check the stability of the product to oxidation. The extent of oxidation can be determined by peroxide number of product after exposure or substance to oxygen for a specific period of time.

9. **Determination of Surface Characteristics:** the study of surface property of the product is carried out in order to check the formation crystal on the surface or the contamination by microorganism or formation of wrinkles and the exudation of liquid.

10. **Determination of Colour dispersion:** the test is done in order to determine the uniform dispersion of color particle. The size of the particle is determined by the microscopic studies and it should not be more than 50µ.

3. **Shampoos**

   **Definition**
A viscous cosmetic preparation with synthetic detergent used for washing hair is called shampoo. Its 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. In olden days detergent soap were used for washing hairs, but nowadays it has been replaced by shampoo. 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 the scalp, eyes etc. Apart from cleaning, shampoo may also be used for medicinal purpose (i.e., medicated shampoo). After preparation each and every shampoo must be evaluated.

Properties:

- It should have optimum viscosity such that it facilitates ease during application.
- It should have good spreading properties.
- It should produce sufficient lather after application.
- It should be able to remove waste material such as debris, soil, sebum, dead cells, salts (due to sweat) etc., from the scalp.
- It should not form any kind of film on scalp.
- It should rinse out completely after washing.
- It should produce lather with both hot and cold water.
- It should facilitate ease of combing after shampooing.
- After drying, the hair should not give rough appearance.
- It should provide lustre to the hair.
- It should produce good odour both before and after shampooing.
- It should not produce any kind of irritation or itching to the scalp.
- It should not support any microbial growth.
- It should be stable and have a half life of about 2 to 3 years.
- It should be economical.

3.1 Types of shampoo

Various types of shampoos are available and they are classified based on their consistency. They are as follows:
1. Clear liquid shampoos
2. Liquid cream shampoos
3. Cream shampoos
4. Gel shampoos
5. Powder shampoos
6. Aerosol shampoos (Foam type)
7. Special shampoos

**1. Clear Liquid shampoo:** These are clear liquid preparations that are most widely used. They are usually made by using detergent of low cloud point. Alkanolamides can also be used in these preparations. Some of these shampoos may be transparent.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triethanolamine lauryl sulphate (surfactant)</td>
<td>50 g</td>
</tr>
<tr>
<td>Lauricisopropanolamine (foam booster)</td>
<td>2 g</td>
</tr>
<tr>
<td>Perfume, color, preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Water</td>
<td>62 g</td>
</tr>
</tbody>
</table>

**2. Liquid Cream Shampoos:** These are called as lotion shampoos which are modification of clear liquid shampoos. Addition of opacifier such as glycercylmonostearate, glycol stearate etc., to the clear liquid shampoo yields liquid cream shampoo. Solubilising agents such as magnesium stearate is also used to dissolve the added opacifier.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triethanolamine lauryl sulphate (surfactant)</td>
<td>35 g</td>
</tr>
<tr>
<td>Glycerylmonostearate (opacifier)</td>
<td>2 g</td>
</tr>
<tr>
<td>Magnesium stearate (stabilizer)</td>
<td>1 g</td>
</tr>
<tr>
<td>Perfume, color, preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Water</td>
<td>62 g</td>
</tr>
</tbody>
</table>

**3. Cream Shampoos:** These shampoos have a paste like consistency and are packed in a collapsible tube. They find great use in hair salons. They are also available in jars with wide mouth. The paste consistency is developed by addition of alkyl sulphates, also Cetyl alcohol is added, which serves as a builder.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium lauryl sulphate (surfactant)</td>
<td>38 g</td>
</tr>
<tr>
<td>Cetyl alcohol (builder)</td>
<td>7 g</td>
</tr>
<tr>
<td>Perfume, color, preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Water</td>
<td>55 g</td>
</tr>
</tbody>
</table>
4. Gel Shampoo: These are transparent and thick usually made by incorporating a gelling agent, (e.g., cellulose). There is great use in hair salons and beauty parlors. The principle ingredient is detergent which can be used either alone or in combination with soap. By altering the proportion of detergent, gel of required consistency can be obtained. Addition of methyl cellulose to clear liquid shampoo and its subsequent thickening also gives rise to gel shampoo.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alkyl dimethyl benzyl ammonium chloride</td>
<td>15 g</td>
</tr>
<tr>
<td>Triethanolamine lauryl sulphate (surfactant)</td>
<td>28 g</td>
</tr>
<tr>
<td>Coconut diethanolamide</td>
<td>7 g</td>
</tr>
<tr>
<td>Hydroxyl propyl methyl cellulose</td>
<td>1 g</td>
</tr>
<tr>
<td>Perfume, color, preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Water</td>
<td>49 g</td>
</tr>
</tbody>
</table>

5. Powder Shampoos: As name suggests, it is available in the form of dry powder, initially it was prepared from dry soaps, but nowadays dry synthetic detergents are used for their preparation. Powder shampoo is prepared where addition of water or other solvent reduces the activity of the components, especially in case of medicated shampoo. Nowadays, these shampoos are not used due to the difficulty experienced in their application.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium lauryl sulphate (surfactant)</td>
<td>20 g</td>
</tr>
<tr>
<td>Sarcoside</td>
<td>5 g</td>
</tr>
<tr>
<td>Sodium bicarbonate</td>
<td>10 g</td>
</tr>
<tr>
<td>Sodium sulphate</td>
<td>65 g</td>
</tr>
<tr>
<td>Perfume</td>
<td>q. s</td>
</tr>
</tbody>
</table>

Another formulation called dry shampoo is also a type of powder shampoo. Initially they are applied on to the head and then removed by the brush. It doesn't involve the use of water. They are usually preferred, when the hair are greasy. This formulation usually includes adsorbents.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Starch (adsorbent)</td>
<td>15 g</td>
</tr>
<tr>
<td>Talc (adsorbent)</td>
<td>45 g</td>
</tr>
<tr>
<td>Kieselgur (adsorbent)</td>
<td>40 g</td>
</tr>
<tr>
<td>Perfume</td>
<td>q. s</td>
</tr>
</tbody>
</table>

6. Aerosol Shampoos (Foam Type): They are called aerosol shampoos because they are packed in aerosol containers. Their formulation, preparation and packing is complicated as an additional propellant is included. The propellant added must be compatible and should not
reduce the activity of shampooing ingredients. The container opening is provided with a valve. Shampoo comes out as foam when the valve is pressed. Hence also called as foam type shampoo.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triethanolamine lauryl sulphate (surfactant)</td>
<td>60 g</td>
</tr>
<tr>
<td>Coconut diethanolamide</td>
<td>2 g</td>
</tr>
<tr>
<td>Propellant</td>
<td>10 g</td>
</tr>
<tr>
<td>Perfume, color, preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Water</td>
<td>28 g</td>
</tr>
</tbody>
</table>

7. Special Shampoos: These are the shampoos which are meant for special purpose. They are

(a) Medicated Shampoo: These shampoos contain medicinal agents. These agents treat the disorders of the scalp or hair. Examples of medicated shampoos are: Anti-lice shampoo, Anti-dandruff shampoo, Anti-baldness shampoo etc.

The medicinal agent added should not irritate the sebaceous glands. It should not sensitize the scalp. The degree of itching and scaling should also be reduced. Among all, anti dandruff type of medicated shampoo is most widely used. Formula for which is given below:

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triethanolamine lauryl sulphate (surfactant)</td>
<td>60 g</td>
</tr>
<tr>
<td>Thymol (anti dandruff)</td>
<td>0.1 g</td>
</tr>
<tr>
<td>Camphor (counter irritant)</td>
<td>0.1 g</td>
</tr>
<tr>
<td>Perfume, color, preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Water</td>
<td>38.8 g</td>
</tr>
</tbody>
</table>

(b) Conditioner Shampoos: These shampoos serve for hair conditioning. Initially they clean the hair (and scalp) and keep them in smooth and lustrous condition. They also prevent sticking of hairs. Conditioner shampoo nowadays is widely used by both men and women. Most of the conditioners are made from Quaternary ammonium compounds. These compounds have the property of reducing electric charges between the hair, as a result hair become lustrous easily manageable. These compounds can also exhibit a bactericidal effect.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stearyl dimethyl benzyl ammonium chloride</td>
<td>5.5g</td>
</tr>
<tr>
<td>Ethylene glycol monostearate</td>
<td>2 g</td>
</tr>
<tr>
<td>Cetyl alcohol</td>
<td>2.5 g</td>
</tr>
<tr>
<td>Perfume, preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Water</td>
<td>90 g</td>
</tr>
</tbody>
</table>
3.2 Formulation of shampoo

Formula of Shampoo contains the following ingredients:

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Examples</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Surfactants</td>
<td></td>
</tr>
<tr>
<td>(a) Anionic Surfactants</td>
<td>Alkyl sulphates, alkyl ether sulphate</td>
</tr>
<tr>
<td>(b) Non-ionic Surfactants</td>
<td>Alkanolamides</td>
</tr>
<tr>
<td>(c) Cationic Surfactants</td>
<td>Alkyl amines, alkyl imidazolines</td>
</tr>
<tr>
<td>(d) Amphoteric surfactant</td>
<td>Acyl amino acids</td>
</tr>
<tr>
<td>2. Foam booster</td>
<td>Monoethanolamides, lauramides DEA, cocamide DEA</td>
</tr>
<tr>
<td>3. Germicide and anti-dandruff agent</td>
<td>Banzalkonium chloride, cetrimide, selenium sulphide, cadmium sulphide</td>
</tr>
<tr>
<td>4. Conditioning agent</td>
<td>Lanolin, egg, amino acids</td>
</tr>
<tr>
<td>5. Pearlescent agent</td>
<td>4-methyl-7-diethylamino coumarin, 4-methyl-5,7-dihydrocoumarin</td>
</tr>
<tr>
<td>6. Sequestrants</td>
<td>EDTA, citric acid, triphosphosphate</td>
</tr>
<tr>
<td>7. Thickeners</td>
<td>Alginates, polyvinyl alcohol, methyl cellulose</td>
</tr>
<tr>
<td>8. Perfuming agent</td>
<td>Herbal fruits or floral fragrance</td>
</tr>
<tr>
<td>9. Preservatives</td>
<td>p-hydroxyl benzoic acid phenyl mercuric nitrate</td>
</tr>
<tr>
<td>10. Colour</td>
<td>FD and C dye</td>
</tr>
</tbody>
</table>

1. **Surfactants**: The main use of surfactant is to cleanse and to produce foam. They are generally categorized into four types. They are: (a) Anionic Surfactants (b) Non-ionic Surfactants (c) Cationic Surfactants (d) Amphoteric Surfactants

(a) **Anionic Surfactants**: These surfactants have good foaming property, hence they are used as principle surfactant. They are considered as main ingredient of shampoo formulation. Examples of Anionic Surfactants:

(i) **Alkyl Sulphates**: When fatty acids are subjected to catalytic reduction, it results in formation of long chain sulphated derivatives called as Alkyl sulphates. (Example: Lauryl sulphate, Myristyl sulphate). A combination of above two compounds is most widely used because they give foam. Sulphates with lauryl chain are widely used when compared to octyl or decyl chain. Previously, sodium lauryl sulphate was used but now triethanolamine lauryl sulphate is widely used.

(ii) **Alkyl polyethylene Glycol Sulphates**: These anionic surfactants exhibit good cleaning as well good foaming property. They are alkyl ether sulphate which forms water soluble sodium salt. Solubility of this salt is greater than sodium lauryl sulphate, hence also serves as a solvent for non-polar ingredients. Because of low cost, they are widely used by small manufacturers.
(iii) Alkyl Benzene Sulphonates: These surfactants are most widely used in the preparation of washing powder but not in cosmetics (i.e. shampoo). Because they cause excessive cleaning, this may lead to damage of scalp and hair. They may also lead to hair fall and skin irritation. Although they have deleterious effects, they are used for cleaning of greasy hair.

(iv) \( \alpha \)-olefin Sulphate: It is an alkyl sulphonate obtained by sulfonation of linear olefin. It produces an excellent foam and the property of foaming is unaffected by sebum and hard water. It produces mild detergent effect without harming the scalp. It is stable at both acid and basic pH and widely used to prepare low pH shampoo. It has low cloud point hence also used to prepare clear liquid shampoo. Apart from the above, other anionic surfactants such as sulphosuccinates, Acyl lactylases etc, are also used.

(b) Non-ionic Surfactant: These are considered as secondary surfactants. They are not used to produce foam but used as foam boosters, viscosity inducers, emulsion stabilizers and opacifiers. This is because they have less foaming power. Even though they have good cleaning property, they are not used as principle surfactant. Examples of Non-ionic Surfactants:

(i) Poly Alkoxylated Derivatives: These are ethoxylated alcohols and phenols, block polymers, sorbitol ester (polyethoxylated) and polyglyceryl ethers. These derivatives are obtained when hydrogen (labile) containing hydrophobic compound is subjected to poly-addition reaction with either ethylene or propylene oxide. They are stable at wide range of pH. They have stabilizing, emulsifying, pearlescent and foaming properties. They are available at low cost and cause irritation to eye mucosa. However, they are used as mild detergents and impart a good rinsing property. They can also be used in high concentration.

(ii) Fatty Acid alkanolamides: These include monoalkanolamides and diethanolamides etc.. Monoalkanolamides are made from long chain fatty acids (i.e., \( C_{14} \)- \( C_{16} \)). They are insoluble in water due to their Waxy nature. Hence, they are added directly to detergent solution and dissolved by gentle warming. The detergent solution is made by using principle surfactant to which various ethanolamides are added to serve as:

- Solubilising Agent: Example: Lauric Monoethanolamide.
- Viscosity Inducing Agent: Example: Lauric Monoethanolamide
- Pearlescent and Thickening Agent: Example: Stearic Ethanolamide
- Softening and Hair Conditioning Agent: Example: Oleic Ethanolamide.
- Foam Boosters.

However the ratio of detergent solution to the monoethanolamide must be 100:15 and above this ratio may be harmful to scalp and hair.

Whereas diethanolamides are available as low melting point solids or even as simple liquids. They are used as powerful solublizing agents. They solubilize the shampoo ingredients rapidly and more efficiently compared to monoethanolamides. The shampoos containing high soap content and free ethanolamides (Example: Kritchevsky condensation products) must be used with precautions.
(iii) **Amine Oxides:** Amine oxides are obtained by the oxidation of tertiary aliphatic amine with hydrogen peroxide. These compounds possess strong polar linkage between nitrogen and oxygen hence they are also called as polar non-ionic surfactants. They constitutes major group of synthetic surfactants. They are water soluble and compatible with various surfactants. They are added as secondary surfactants because of their conditioning, dam boosting and anti-static property. Coconut and dodecyl dimethylamine oxides are most commonly used for this purpose.

(c) **Cationic Surfactants:** Surfactants that contains positive charge are called as cationic surfactants. They are used as both principle and secondary surfactants. These surfactants are used in low concentrations because they are toxic to eye. Hence, they are considered as secondary surfactants. Apart from the above toxic effect, they also have good foaming and partly cleaning properties. Hence, they are also used as principle surfactants in conditioner shampoos. Examples Cationic Surfactants:

(i) **Alkylamines:** They constitute a major group of cationic surfactants. They are used in combination with hydrophilic surfactants in order to provide conditioning and anti-static property to the shampoo. However they precipitate when combined with anionic surfactants. Usually they are used in the form of water soluble salts.

(ii) **Ethoxylated amines:** These are nitrogen containing surfactants which are obtained by ethoxylation of long, chain alkylamine. They are waxy in nature with low melting point. Because of their waxy nature; they are also used as viscosity inducer. However their main function is emulsification and hair conditioning. Sometimes, they are also used as foam boosters. Due to their emulsifying property, complete dispersion of various ingredients is achieved.

(iii) **Alkyl-Betains:** These classes of cationic surfactants are obtained from N dimethylglycine. They are readily compatible with majority of surfactants and have following properties.

- Enhance the efficiency of Foam. Example: Foam Booster.
- Contain Conditioning and Anti-static Property.
- Have viscosity inducing property.
- Possess good stability.
- Non-irritant to skin and eye.

Based on the above properties, Alkyl Betains are considered as secondary surfactant. They are also used as principle surfactant in baby shampoo and are often used combination with ionic surfactants. A part from the above, various other cationic surfactants like imidazolines and morphollrx derivatives, tetra alkyl ammonium salts are also used.

(d) **Amphoteric Surfactants:** The surfactants which possess both cationic and anionic charges with respect to acidic and basic media are called as amphoteric solvents. They form zwitterions when the pH of media is neutral. These agents produces a mild action and show
compatibility with surfactants. They possess excellent hair conditioning property and hence used as secondary surfactants. Examples Amphoteric Surfactants:

(i) Dialkyl Ethylene Diamines: These surfactants are soluble in water and compatible with surfactants. They are used as detergents and to a lesser extent as emulsifier. They are usually prepared as aqueous solution or paste into which remaining shampoo ingredients are added. These agents are combined with anionic surfactants in order to minimize the irritation caused by them. These agents neither enhances nor inhibits the foaming property of the principle surfactant. They are most widely used an anti-irritating agent when anionic compounds are used as principle surfactant. (Anionic surfactants are irritant to eye). These agents also possess conditioner and anti-static property as a result of which the hair becomes smooth and soft. However the pH of the shampoo prepared by using these surfactants must be neutral (i.e., in between 6.5 to 7.5).

(ii) N-alkyl Amino Acids: The important compounds of this class are derived from amino acids and asparagine. A compound called N- alkyl-b iminoproperonate is derived from b- amino acid and it exhibits good foaming property, possesses slightly alkaline pH by changing the pH to acidic range the manageability of hair is improved. Whereas, The derivatives of asparagine are well compatible with both anionic and cationic surfactants. It also posses the properties like foaming, cleaning and conditioning. Depending upon the pH, these compounds change their nature i.e., they become zwitterions at pH 6 and at neutral pH, they become amine. Solubility of N-alkyl amino acids is greater than they are in the form of sodium salts, whereas the solubility decreases with zwitter ionic form. The foaming property of these agents decreases with decrease in pH. This is because at low pH they become cationated (i.e., cationic form). These agents are highly stable and sometimes also employed as emulsifiers.

2. Foam Boosters: The surfactants used in the preparation also serves as foaming agents. They, form rich lather i.e., foam which is stabilized or strengthened by using a substance called foam boosters. The substances like fatty acid alkanolamides, amine oxides are used. They make the foam dense and it to remain for long duration. Usually they are added in quantity of about 2 to 5%. Fatty acids and fatty alcohols when added in a range of 0.25 to 0.50% concentrations, they also act as foam boosters.

3. Germicide and Anti-dandruff Agents: Germicides are the agents which prevent the growth of micro-organism on the scalp whereas anti-dandruff agents are used to eliminate dandruff from the scalp.

Examples of Germicides are:

- Quaternary ammonium compounds: Example: Benzalkonium Chloride, Cetrimide etc.

Examples of Anti-dandruff Agents are:

- Selenium Sulphide
- Cadmium Sulphide
4. **Conditioning Agents:** These agents improve the condition of hair. These agents have the property of reducing electric charges the hair, as a result, hair become lustrous and hence easily manageable. These agents also exhibit a bactericidal effect. They make the hair silky and shiny. Most commonly used conditioning agents are lanolin, oils, herbal extracts, egg, amino acids etc. Among the above; amino acid gives an efficient conditioning effect.

5. **Pearlescent Agent:** these agents are usually added as adjuvants to the conditioning agents. They improve the conditioning property. Addition of these agents also imparts brightness to hair. They make the preparation transparent or opaque; hence they are also called as opacifying agents. The commonly used pearlescent agents are alkanolamides and coumarins like 4-methyl-7-diethyl amino coumarin, 4-methyl-5, 7-dihydrocoumarin etc. Also alcohols and phosphates improve transparent solubilization.

6. **Sequestrants:** These are complex forming agents. They form complex with metal ions like calcium and magnesium. Surfactant are liable to form complex with the metals present in water i.e., calcium and magnesium. Hence addition of Sequestrants prevents complex formation between metal and surfactant. The Sequestrant itself forms complex with the metal ions. Thus, it prevents the formation of film on the scalp i.e., the film formed by surfactant and metal ions. The commonly used Sequestrants are EDTA, citric acid etc.,

7. **Thickening Agents:** These agents are usually added to make the preparation thick i.e. viscous. Such viscous preparation facilitates ease of handling. Also, they prevent wastage during application. Already the addition of various agents, such as surfactants, foam boosters etc make the preparation viscous even though thickening agent is added. Substances like methyl cellulose, alginates polyvinyl alcohol, polyethylene glycol etc are commonly used to adjust the viscosity of a shampoo.

8. **Perfumes:** Addition of these agents imparts good fragrance to the shampoo. It also neutralizes the undesirable odour of other ingredients of formulation especially surfactants. Nowadays it has become an important factor for consumer satisfaction. The selected perfumes must be such that they should retain good smell for fixed period of time even after shampooing. The added perfumes should not affect the solubility and stability of the preparation. They are usually obtained from natural sources such as flowers, fruits, herbs etc.

9. **Preservatives:** These agents have the ability to prevent the growth of micro-organisms. They are usually added to maintain the stability of the preparation for a desired period of time. Shampoo is a wet preparation that provides a media for various micro organisms hence addition of preservative is essential. Preservative used should not cause any irritation to the scalp. Para-hydroxybenzoic acid and phenyl mercuric nitrate are commonly used preservatives.

10. **Colour:** Addition of colour gives pleasant appearance to the preparation. Various FD & C dyes are used for colouring the preparation. The added colour must be water soluble and it should not impart any colour to hair or scalp.
3.3 Preparation of shampoo

Simple procedure is involved in the preparation of shampoo. Initially only one method available for the preparation of shampoo, but later the basic method was modified in order to obtain different type of shampoo like cream, gel, aerosol etc.

**General Method for preparation of shampoo:**

Liquid shampoo is usually prepared by this method which involves the following steps:

- Initially the detergent is converted into a solution form or a detergent solution ma ho directly obtained from the manufacturer.
- Take about half of the detergent solution into a separate container. To it, add the total amount of secondary surfactant i.e., alkanolamide.
- Dissolve the alkanolamide along with stirring. Sometimes, gentle heat is also applied.
- To the remaining half of the detergent solution add suitable amount of perfuming agent and dissolve it.
- The perfume solution is then added to the alkanolamide solution.
- Colour and preservatives are dissolved separately in sufficient volume of water and then added to the main solution.
- The whole, solution is mixed well by gentle stirring. Excessive stirring may lead to bubble formation.
- Final volume of the preparation is usually adjusted by the addition of clear sterile waste. This gives clear liquid shampoo.
- However, When the preparation contains lauryl alcohol ether sulphate. It is required to adjust the viscosity of the shampoo.
- Viscosity adjustment is done by using an electrolyte solution. Usually, a solution of sodium chloride is added subsequently with constant stirring. Care must be taken to it event the excess addition of sodium chloride.

**Methods of Preparation:** The methods of preparation of various types of shampoos are modification of the above mentioned general method of preparation of shampoos.

(a) **Preparation of Cream Shampoo:** Certain formulae of cream shampoo may include glycol stearate or waxes. Usually, glycol stearate is used as an opacifier and preparation method for such formulae is similar as discussed above. But when wax is included in the formula, the process involves the following steps.

- Initially, a solution of detergent and water are heated to about 80°C.
- The wax is heated separately in a container at 80°C which facilitates the melting of wax.
- Both the solutions are kept at 80°C and mixed uniform mixing by constant and gentle stirring.
- The solution is allowed to cool to about 40- 45°C. After which the remaining ingredients, such as additives, colours, perfume and preservatives are added. The stirring is continued.
Finally, under warm conditions, the mixture is transferred into a suitable container and packed.

(b) Preparation of Gel Shampoo: The method involved in the preparation of gel shampoo is similar to that of clear liquid shampoo. After preparation, the liquid shampoo is usually treated with a suitable thickening or gelling agent such as hydroxy propyl methyl cellulose, this gives a gel like consistency. Addition of appropriate amount of anionic and amphoteric surfactants also leads to the formation of gels.

(c) Preparation of Aerosol Shampoo: This type of shampoo is initially prepared by using (earlier discussed) general method. The prepared shampoo is then incorporated with a suitable propellant. The whole mixture is packed in an aerosol container. The propellant creates a pressure within the container due to which spraying action is achieved and the product (shampoo) is sprayed in the form of foam. Here packing plays an important role and the propellant used should not react with the shampoo.

(d) Preparation of Powder Shampoo: Powder shampoo is prepared by simple blending. Here, all the ingredients are taken in a state. They are powdered to suitable degree of fineness. The powdered ingredients are blended by using a suitable blender. Two separate solutions of perfume and colour are prepared by using alcohol or water as solvents. The prepared solutions are then sprayed onto the blended mixture. The wet mixture is dried and packed. Otherwise, the ingredients are internally soaked into the solutions of colour and perfume. Wet mass is dried and then subjected to blending.

3.4 Evaluation of shampoo

According to the regulatory authorities each and every batch of shampoos must be evaluated prior to marketing. Evaluation is a measure of activity and safety. It also notifies the toxicity, if nowadays most of the shampoos are prepared, from synthetic detergents, hence evaluation becomes an essential factor. However, there is also a need to evaluate herbal shampoo, since it may contain natural ingredient which are liable to contamination.

Shampoos are evaluated for the following aspects.

(I) Evaluation of Safety

(II) Evaluation of Antimicrobial Property.

(1) Evaluation of Safety: Safety is an important aspect which must first and foremost parameter of evaluation. As stated earlier the shampoos are made from synthetic detergents, which are liable to irritate skin, scalp and eye. Hence, it becomes essential to evaluate the safety of a shampoo. Over all, the shampoo must be non-toxic and non-irritative. The safety is usually evaluated it, terms of toxicity i.e., if the preparation is found to be non toxic then it is regarded as safe and vice-versa. However, the toxicity is determined by using “Draize test” which suggests two separate methods for testing skin and eye toxicity respectively. The methods are as follows:
(a) **Skin Toxicity Test:** The steps involved in this test are as follows:

- A set of six albino rabbits are selected. They should weigh about 2 kgs.
- On the skin of each rabbit, a round patch is made by removing hair.
- Dilute preparation (8-10%) of shampoo is usually applied onto the patches of a rabbits.
- The shampoo is allowed to react for a period of 3-4 hours. After that it is removed completely from the skin.
- After efficient washing, the skin is examined for any adverse reactions such as erythema, edema etc.
- Based on the results obtained the shampoo is considered as either safe or toxic.

Usually, there might be chances of adverse reactions because the shampoo was kept in contact for 4 hours. But usual practice of shampooing in human being is for 10-15 minutes. Alternatively, the skin test is also performed on human being.

(b) **Eye Toxicity Test:** The steps involved in this test are as follows:

- A set of six adult albino rabbits are selected. They must weigh about 2 kgs.
- One eye of each rabbit is considered as test eye and another as control eye.
- To each of the six test eyes of six rabbits, the product (shampoo) is applied.
- Washing is done after 20 seconds with 200 ml of tap water.
- The eyes are rewashed after 5 minutes and then after 24 hours.
- The control eye are also washed on first day and then after 24 hours.
- The test eyes are observed at 1, 24, 48 and 72 hours respectively. They are also examined on 7th and 14th day.
- The product is said to be toxic, if there is a development of iris and corneal lesions which remains for more than 7 days.

(II) **Evaluation of Antimicrobial Activity:** Shampoos are liquid or viscous preparations, they are liable to microbial growth. Hence, preservative is usually added to prevent mid growth. The added preservative should have following properties.

- It should be non toxic.
- It should be compatible with other ingredients.
- It should be effective at low concentration,
- It should be effective against wide variety of microorganism.

However, all the above points are considered prior to the selection of preservative. Evaluation of preservative usually involves the study of antimicrobial activity is generally done by using a method called as "Challenge Study". According to this study, the product is said to be preserved when it does not support microbial growth even after repeated attacks of various micro-organisms.
Procedure (Challenge Study):

1. Initially an appropriate strain of microorganism is selected and is considered as test organism. Usually the species of *Pseudomonas* are selected i.e., *P. aeruginosa*, *P. Putida* etc.
2. A culture of any one of the above test organisms is prepared.
3. The product (shampoo) is then inoculated repeatedly in the culture medium and the studies are carried out for a period of 10 to 12 weeks.
4. The inoculums usually contain 5 lakhs to 1 crore microorganisms/gm of product.
5. Along with the test, control samples are also prepared and reserved for reference.
6. Usually two types of control samples are prepared i.e., one sample with preservative and another without preservative.
7. The test comes to a conclusion only when it has been proven that the product has not supported the microbial growth.
4. Powders

Definition

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.

Characteristics:

- It should possess good covering power in order to hide blemishes present on the skin.
- Adhesion property should be good, so that it should not blow-off easily from the skin.
- It should remain on the skin for longer period of time to avoid re-powdering.
- It should be able to impart matt or peach like appearance to the skin.
- It should remove the shine present on the skin as well as around the nose.
- It should possess good absorbent property.
- It should be able to produce slip property to the skin for easy spreading by puff without producing any blotches (irregular marks).
- It should be able to produce transparency effect.

4.1 Formulation of powders

Ingredients used in the formulation of powders are properly studied before selection. Their character, role and quality are taken into consideration, as they have effect on the finished product. The ingredients used should be of good quality.

Properties:

- They should not be hard in nature. If the materials are present in crystal form then they should not contain any sharp edges as it may damage the skin.
- They should have less solubility in water and mixtures of fat.
- They should be non-toxic in nature.
- They should be chemically stable, in order to prevent interaction with each other.
- They should not cause irritation to the skin.
Ingredients used in the formulation are classified based on their functions. They are as follows:

1. **Covering Materials**: These materials should be able to cover small imperfections, enlarged pores and minor blemishes of the skin. The covering power of powder is high, when its surface area is more. This can be achieved if the particles are in finely divided form. Medium in which these covering materials are dispersed plays an important role for imparting efficiency. Dry skin offers better covering power compared to moist skin. Examples:

   (a) **Titanium Dioxide**: It is considered as the best covering agent who is widely used in the formulation of face powders. It is inert in nature. It has 1.6 times more covering power on dry skin and 2.5 times more covering power on moist and greasy skin compared to zinc oxide; however it has less sunscreen property.

   (b) **Zinc Oxide**: It is also a good covering agent with good sunscreen property because zinc oxide has protective effect against ultraviolet rays. It consists of fine particles, which impart better covering power. But if the particle size is below 0.25 gm, then the covering power is reduced. And in case of moist and oily environment, of zinc oxide covering power is less i.e., 37% compared to dry powders. Others materials which have less covering power are kaolin, zinc stearate, magnesium stearate and rice starch. They are used in combination to obtain products of different covering ability.

2. **Adhesive Materials**: Adhesive materials are essential as they are helpful in imparting adhesion i.e., it cling the powder materials not only to the surface of the skin but also to the powder puff. The adhesion of powder to the puff is necessary to take the powder out of the container in case of compact powders. Example:

   **Magnesium and Zinc Stearate**: Magnesium stearate is more preferred in the formulation of face powders in 3-10% and it has more adhesive property compared to zinc stearate. Whereas zinc stearate is used in the formulation of talcum powders.
Good quality of magnesium and zinc stearate is used because they provide excellent colour texture with minimum odour and also helps in provide velvety softness to the final product. These materials are water proof in nature. This helps in maintaining the complexion impact even in damp weather.

Other materials are lithium stearate, calcium stearate, talc, cetylalcohol (1-2%), stearyl alcohol, glyceryl monostearate, petrolatum, lanolin, and magnesium as well as calcium salts of myristic acid.

3. Slip Materials: Slip character helps in easy application and spreading of the powder on the skin, which in turn provide smoothness to the skin. Example:

(a) Talc: it is a purified hydrated magnesium silicate.

Formula: \( \text{H}_2\text{Mg}_3(\text{SiO}_3)_4 \) or \( \text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2 \)

It is widely used in the formulation of face powders. It helps in imparting slip character along with softness. It is neutral and cannot absorb water.

(b) Aluminium hydrosilicate:

- It is considered as the basic material for the formulation of powders.
- It is smooth, fatty and non-toxic in nature.
- It is prepared by treating acid and then washing with water. Finally drying is carried out.
- It also produces cooling effect.
- It also absorbs fatty secretions and water (in small quantity).

Other materials are zinc stearate, magnesium stearate, zinc undecanate and magnesium undecanate.

4. Absorbent Materials: these materials should be able to eliminate shine from the skin surface by absorbing the secretion of the skin i.e., sebum and perspiration (sweat).

Examples:

(a) Colloidal Kaolin:

- It is fine, white colour powder, which is soft in nature.
- It is non-toxic and inert in nature.
- It does not cause irritation to the skin.
- It absorbs aqueous and fatty substances which show that it has good absorbing capacity.
- It has good covering power along with less slip property.

(b) Bentonite or Aluminium Silicate:

- It is fine, whitish-grey powder.
• It has good swelling power i.e., can swells up to 12 times of its own volume.
• It is not widely used in cosmetics.

(c) **Magnesium Carbonate:**

• It has good absorbing capacity for water and fatty substances.
• It is less alkaline in nature.
• Covering power and adhesive property are more, which all the perfume oil to be added first in magnesium carbonate and then mixed with other materials.

(d) **Calcium Carbonate:** It is fine, white coloured powder, which is soft in nature. It has properties similar to that of magnesium carbonate. It is less preferred, as it undergoes alkaline reaction with skin. Other materials are rice, wheat, corn, potato etc., which have both absorbing and swelling properties. These materials impart sticky character and are non-toxic in nature.

5. **Peach-like Finish Materials:** These materials help in imparting peach-like finish appearance, which provide bloom to the skin. Examples:

(a) **Rice Starch:** Other starches are also used like maize starch. They are used after drying (i.e., few hours) in order to get better effect.

(b) **Silica:** it is finely divided material which imparts fluffy appearance to the skin.

(c) **Powdered Silk:** This material is obtained from silk proteins. These proteins are subjected to the process of partial hydrolysis which produces hydrolysate. This hydrolysate is grounded to obtain fine powder. They are used in 30% quantity.

6. **Materials imparting Frosted-look:** This material is capable of producing translucent lustre and shiny look to the skin. It also imparts pearlescence.

Example: Guanine (It is not widely used because it is expensive), bismuth oxychloride, mica, aluminium, bronze.

7. **Colouring Materials:** These materials are mainly used in the formulation of face powders and compacts in order to impart colour.

Examples:

(a) **Iron Oxide:** It is an inorganic pigment, which is used for imparting yellow, red and brown colour.

(b) **Ultramarine:** This material is used to impart green and blue colour.

(c) **Organic Lakes and Pigments:** These materials are capable of producing better brilliance the skin. They should not bleed (i.e., loss of colour) in oil and water solvents.
8. **Perfumes**: Flowery fragrance or synthetic odour are used in the formulation of powders. Perfumes should be compatible with the other ingredients in the formulation. Otherwise, the perfume character will change.

### 4.2 Classification of powders

Three different types of powder products are used in cosmetics, which are as follows:

1. Face powders
2. Compacts

![Baby Powder](image)

Fig. 4–2. Baby Powder

### 1. Face Powders:

These powders have the ability to complement the skin colour by providing velvety finish to it.

**Properties:**

- It should impart smooth finish to the skin.
- It should mask minor imperfections (which are visible) on the skin.
- It should eliminate shine present on the skin due to moisture or grease.
- It should have long lasting property to avoid frequent application.
- It should be resistant to the secretions of the skin i.e., sebaceous and perspiration.
- It should serve as a vehicle to perfumes so that the particles of perfumes may spread easily.

A single substance is unable to impart all the characters i.e., covering power, slip character, absorbent capacity, adhesive property. Hence, mixture of substances is used in the formulation of powders to impart desired properties to the skin.

Depending on the type of skin to be powdered, the face powders are classified into 3 types.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Talc (slip character)</td>
<td>63 g</td>
</tr>
<tr>
<td>Kaolin (covering materials)</td>
<td>20 g</td>
</tr>
<tr>
<td>Calcium carbonate (absorbent)</td>
<td>5 g</td>
</tr>
<tr>
<td>Zinc oxide (covering materials)</td>
<td>5 g</td>
</tr>
<tr>
<td>Zinc stearate (slip character)</td>
<td>5 g</td>
</tr>
<tr>
<td>Magnesium carbonate (absorbent)</td>
<td>1 g</td>
</tr>
<tr>
<td>Color</td>
<td>0.5 g</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>0.5 g</td>
</tr>
</tbody>
</table>
They are light type, medium type and heavy type.

(i) **Light Type:** These types of powders are applied on dry skin. They have low covering power since the dry skin does not secrete any oils. Large amount of talc will be present in the formulation of powders.

**Method:** It is a dry mixing method.

- Perfume is added to some part of calcium carbonate, which is absorbent and mixed thoroughly. This preparation is kept aside for some time. This is mixture A.
- Color is added to some part of talc and mixed thoroughly. This is mixture B.
- Then kaolin, zinc oxide, zinc stearate, magnesium carbonate and remaining part of calcium carbonate and talc are added to mixture B and mixed properly.
- Mixture A is added to the above mixture and mixing is carried out.
- Finally, the preparation is sieved by using either a silk mesh or nylon cloth.

(ii) **Medium type:** Type of powders is applied on the normal or moderate oily skins. These skins are shiny in nature due to the sebaceous secretions or perspiration (sweat). They have good covering power compared to light type. They contain less quantity of talc along with slightly more quantity of zinc oxide (The less quantity of talc is balanced by zinc oxide).

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Talc (slip character)</td>
<td>39.7 g</td>
</tr>
<tr>
<td>Kaolin (covering materials)</td>
<td>39.5 g</td>
</tr>
<tr>
<td>Calcium carbonate (absorbent)</td>
<td>5 g</td>
</tr>
<tr>
<td>zinc oxide (covering materials)</td>
<td>7 g</td>
</tr>
<tr>
<td>zinc stearate (slip character)</td>
<td>7 g</td>
</tr>
<tr>
<td>Magnesium carbonate (absorbent)</td>
<td>1 g</td>
</tr>
<tr>
<td>Color</td>
<td>0.2 g</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>0.6 g</td>
</tr>
</tbody>
</table>

Method The method is same as that of light type of face powders.

(iii) **Heavy type:** These types of powders are applied on extremely oily skins, which have more shine due to secretions. They have high covering power, in order to cover the shine of the skin. They contain less quantity of talc and more quantity of zinc oxide.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>magnesium stearate (covering materials)</td>
<td>5 g</td>
</tr>
<tr>
<td>Kaolin (covering materials)</td>
<td>20 g</td>
</tr>
<tr>
<td>zinc oxide (covering materials)</td>
<td>15 g</td>
</tr>
<tr>
<td>Calcium carbonate (absorbent)</td>
<td>39 g</td>
</tr>
<tr>
<td>Talc (slip character)</td>
<td>20 g</td>
</tr>
</tbody>
</table>
2. **Compacts**: loose powder or dry powders are compressed in the form of cake along with binder by compaction process; in order to form compact Powders. Compact are applied on the face with help of powder puff. The pressure used in compaction process is an important factor in the formulation of compacts. As low pressure may form cake which break easily during use and high pressure form very hard cake which will not adhere to the puff easily. The average particle in compact powders is looser compared tri loose powders, due to compaction.

**Ingredients:** The composition of compact powder is similar to face powder but binding agents e incorporated, in order to increase adhesion property.

<table>
<thead>
<tr>
<th>Binders</th>
<th>Examples</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Dry binders (requires increased pressure for Compaction)</td>
<td>zinc stearate, magnesium stearate</td>
</tr>
<tr>
<td>2. Oil binder</td>
<td>mineral oil, isopropyl myristate, lanolin derivatives</td>
</tr>
<tr>
<td>3. Water-soluble binders</td>
<td>tragacanth, karaya, Arabic</td>
</tr>
<tr>
<td>(a) Aqueous solution gums</td>
<td>polyvinyl pyrrolidine (PVP), methyl cellulose</td>
</tr>
<tr>
<td>(b) Aqueous solution of synthetic or Semi synthetic gums</td>
<td>Carboxy methyl cellulose (CMC)</td>
</tr>
<tr>
<td>(c) Preservatives are added along with gum to avoid microbial growth</td>
<td>Mineral oil, fatty esters, derivatives of lanolin. (These materials are used in combination with water)</td>
</tr>
<tr>
<td>4. Water-repellant binder. Wetting agent is also used for uniform distribution of moisture</td>
<td>Triethanolamine stearate, non-ionic emulsifiers, glycerol Monostearate.</td>
</tr>
</tbody>
</table>

**Preparation of Compacts:** Compact powders are prepared by three methods. They are:

(a) **Wet Method**

(b) **Dry Method**

(c) **Damp Method,**

(a) **Wet Method:** The basic material (i.e., powder), colour and binders are formulated in the form of paste with the help of water. Then the pastes are pressed into moulds and slowly the products are dried by air. This method is not widely used, as there is a possibility of producing cracks and other faults in the preparation.
(b) **Dry Method:** In this method, the basic materials and binders are compressed in special presses with the help of pressure. This method is carried out under controlled conditions. Examples:

- Mixture of ammonia, stearic acid and starch.
- Mixture of stearic acid and starch
- Mixture of sodium stearate, lanolin and cetyl alcohol or
- Mixture of triethanolamine stearate, lanolin and cetyl alcohol.

(c) **Damp Method:** This method is most widely used for commercial purposes. The base powder, colour and perfume are properly mixed to form a mixture. Then liquid binder i.e., aqueous mucilage or mucin rich emulsion (oil-in-water type) are added to the mixture. Then it is properly blended until the desired plasticity of the product is obtained. Screening of mixture is carried out followed by compression by machine. Finally, the product is dried at elevated temperature.

<table>
<thead>
<tr>
<th>Formula 1 (Without Binder)</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Talc (slip character)</td>
<td>69 g</td>
</tr>
<tr>
<td>Kaolin (covering materials)</td>
<td>18 g</td>
</tr>
<tr>
<td>Titanium dioxide (covering materials)</td>
<td>8 g</td>
</tr>
<tr>
<td>zinc stearate (slip character)</td>
<td>5 g</td>
</tr>
<tr>
<td>Color</td>
<td>q. s</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>q. s</td>
</tr>
<tr>
<td>Binder (with Binder)</td>
<td>q. s</td>
</tr>
<tr>
<td>gum Arabic (water-soluble Binder)</td>
<td>1 g</td>
</tr>
<tr>
<td>Glycerol (emulsion Binder)</td>
<td>5 g</td>
</tr>
<tr>
<td>Water (vehicle)</td>
<td>94 g</td>
</tr>
<tr>
<td>Preservatives</td>
<td>q. s</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formula 2 (Without Binder)</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Talc (slip character)</td>
<td>79 g</td>
</tr>
<tr>
<td>Calcium carbonate (absorbent)</td>
<td>9 g</td>
</tr>
<tr>
<td>zinc oxide (covering materials)</td>
<td>7 g</td>
</tr>
<tr>
<td>c (slip character)</td>
<td>5 g</td>
</tr>
<tr>
<td>Color</td>
<td>q. s</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>q. s</td>
</tr>
<tr>
<td>Binder (with Binder)</td>
<td>q. s</td>
</tr>
<tr>
<td>Gum tragacanth (water-soluble Binder)</td>
<td>2 g</td>
</tr>
<tr>
<td>Glycerol monostearate (emulsion Binder)</td>
<td>6 g</td>
</tr>
<tr>
<td>Mineral oil (oil binder)</td>
<td>4 g</td>
</tr>
<tr>
<td>Sorbitol</td>
<td>5 g</td>
</tr>
<tr>
<td>Water (vehicle)</td>
<td>83 g</td>
</tr>
<tr>
<td>Preservatives</td>
<td>q. s</td>
</tr>
</tbody>
</table>
Method: The method is same as that of face powder but here binders are incorporated in the formulation.

3. Body powder/ talcum powder/ dust powders:

These powders are most widely used preparation for multiple purposes. They contain covering materials, adhesives, absorbency material, antiseptic and perfumes. The main function of body powder is absorption of perspiration (sweat). Due to the presence of fat film in the body powder, they adhere to the surface of the skin.

Fig. 4–3. Talcum Powder

Properties:

- They should provide good slip character to the skin.
- They should provide cooling and lubrication effect to the skin.
- They should be able to prevent irritation of the skin.

Ingredients: Ingredients which are used in the formulation of body powders are as follows:

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Metallic compound</td>
<td>zinc stearate, aluminium stearate, magnesium carbonate(light), precipitate calcium carbonate (chalk)</td>
</tr>
<tr>
<td>2. Antiseptic materials</td>
<td>Boric acid, chlorohexidine diacetate, bithional</td>
</tr>
<tr>
<td>They are incorporated in the formulation in order to prevent the growth of microorganism which are responsible for the development of perspiration (sweat) and odour.</td>
<td></td>
</tr>
<tr>
<td>3. Adsorbent material</td>
<td>Kaolin, magnesium carbonate, precipitate chalk, starch</td>
</tr>
<tr>
<td>4. Slip character</td>
<td>Talc, zinc stearate</td>
</tr>
<tr>
<td>5. Adhesive materials</td>
<td>Kaolin, zinc oxide, magnesium stearate</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Talc (slip character)</td>
<td>75 g</td>
</tr>
<tr>
<td>Colloidal kaolin (adhesive material)</td>
<td>10 g</td>
</tr>
<tr>
<td>Colloidal silica (binding agent)</td>
<td>5 g</td>
</tr>
<tr>
<td>Magnesium carbonate (absorbent)</td>
<td>5 g</td>
</tr>
<tr>
<td>Aluminium stearate</td>
<td>4 g</td>
</tr>
<tr>
<td>Boric acid (antiseptic)</td>
<td>0.3 g</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>0.7 g</td>
</tr>
</tbody>
</table>
Method:

- Initially perfume is mixed with magnesium carbonate (absorbent) properly. This mixture is kept aside for some time. This is mixture A.
- Talc, colloidal kaolin, colloidal silica, aluminium stearate and boric acid are mixed together. This is mixture B.
- Mixture A is added to mixture B and then mixing is carried out properly.
- Then the preparation is passed through a sieve.
- Finally, the product is packed in a suitable container.

Formulation:

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Talc (slip character)</td>
<td>70 g</td>
</tr>
<tr>
<td>Calcium carbonate (absorbent)</td>
<td>25 g</td>
</tr>
<tr>
<td>Zinc stearate (slip character)</td>
<td>4 g</td>
</tr>
<tr>
<td>Boric acid (antiseptic)</td>
<td>0.3 g</td>
</tr>
<tr>
<td>Perfume oil (odour)</td>
<td>0.7 g</td>
</tr>
</tbody>
</table>

Method:

- Perfume oil is mixed with calcium carbonate (absorbent) properly. This mixture is kept aside for some time. This is mixture A.
- Talc, zinc stearate and boric acid are mixed together. This is mixture B.
- Mixture A is added to mixture B and then mixing is carried out properly.
- Then the preparation is passed through a sieve.
- Finally, the product is packed in a suitable container.

4.3 Evaluation of powders

Evaluation is carried out in order to know the quality of the finished product. General tests include determination of contents in the formulation along with the stability test. This is carried out to know whether the product remains stable for prolonged period of time (i.e.1 shelf life). Other tests are also carried out. They are:

1. Shade Test
2. Colour Dispersion Test
3. Pay-off Test
4. Pressure Test
5. Breakage Test
6. Flow Property Test
7. Particle Size Determination
8. Abrasive Character

9. Moisture Content

1. Shade Test: In this test, the variations of colour shade is determined and controlled. It is carried out by spreading the powder sample on a white paper and appearance is observed which is compared with the standard one. Another method involves, applying powder sample and standard one with the help of puff on the skin and then comparing it. The puff used to perform this test is also used for the final product. Evaluation of colour is carried out by using artificial light.

2. Colour Dispersion Test: in this test, a sample of powder is spread on a white paper and with the help of magnifying glass, segregation or bleeding of the colour is observed. the colour should be properly distributed in the powder base of the formulation.

3. Pay-off Test: This test is carried out to check the adhesive property of powders with the puff. This test is mainly carried out on compact powders.

4. Pressure Test: For compaction purpose in compact powders, pressure required. Uniform pressure should be applied to avoid formation of air pockets, which will lead to either breaking or cracking of compact powders. This is because low pressure will make the compact powder soft, whereas high pressure will lead to formation of hard cake.

With the help of penetrometer, uniformity of hardness of the cake is checked. This is done by taking the reading at different points on compact powder and then comparing them.

5. Breakage Test: In this test, compact powders are allowed to fall on a wooden surface from a height of about 8-10 inches. This is carried out several times and then checking is done to see whether any breakage has occurred on compact powder. If the compact powder remains unbroken, then it shows the resistance to travel and normal handling by the users.

6. Flow Property Test: This test is carried out mainly on body powders to determine their flow property (from the container upon usage). This intern helps in easy application of powder to skin. In this method, angle of repose of powder is measured by allowing the powder product to fall on a plate through a funnel. Then the height and the radius of heap formed is measured, and even the time taken for the powder to fall is noted.

7. Particle Size Determination: With the help of microscope, sieve analysis or by utilizing other techniques and instrument, particle size of powder product is determined.

8. Abrasive Character: Abrasive character of powder can be determined by, rubbing, the powder on a smooth surface of the skin. Then with the help of a microscope, the effects of powder are studied.

9. Moisture Content: Moisture content present in the powder can be determined by using following formula.
Moisture Content % = \frac{\text{Weight of water in sample} \times 5}{\text{Weight of dry sample}}

This is usually carried out by using various suitable analytical methods. These methods are also suitable for determining limits for colour.
5. Nail Lacquers

Definition:

Nail lacquers or nail paints may be defined as viscous or semi-liquid preparations that are intended for the decoration of the nails of the fingers and toes. Nail lacquers form the most commonly used the most popular type of manicure preparations.

Nail polishes are quite distinct from those of nail lacquers and are regarded as a type of manicure preparations that produce a gloss by means of huffing action. The action is mainly by causing abrasion on the surface of the nail and secondly by drawing more blood into the capillaries of the nail.

Ideal Properties of Nail Lacquer:

The ideal properties of a nail lacquer should be as follows:

- It should be safe for the skin and nails and should not lead to any harmful effects.
- It should be easy to apply and easy to remove.
- It shall maintain its properties even during long storage. Hence, an efficient nail lacquer should possess consistent stability.
- The most important property is that it should form a uniform and satisfactory film on the nails.
- It should have good wetting and flow properties and should be viscous in nature in order to form an appropriate film.
- The distribution of the colour should be uniform which can be achieved by using finely divided pigments that are uniformly ground and evenly wetted by the solvent.
- It should provide a good shine on the nail on application.
- It should possess sufficient adhesive property so that it may uniformly adhere to the nail without slipping.
- It should possess the required flexibility so that it may not become brittle and crack upon application.
- The surface should be sufficiently hard in order to prevent the effect of impact and scratch.
- The drying time of the film should not be too rapid or too slow; say about - minutes without forming any bloom.
- It should be able to preserve all these properties at least for a week after its application.
5.1 Formulation of nail lacquers

The formation of an efficient nail polish may be based on the selection of a proper and an essential ingredient. The ingredients involved in the formation of a good variety of nail polish could be as follows:

<table>
<thead>
<tr>
<th>S.N.</th>
<th>Ingredients</th>
<th>Example</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Film forming agents</td>
<td>Nitro cellulose, ethyl cellulose, vinyl polymers</td>
</tr>
<tr>
<td>2</td>
<td>Resinous substances</td>
<td>Aryl sulphonamide-formaldehyde</td>
</tr>
<tr>
<td>3</td>
<td>Dissolving solvent</td>
<td>Ether, ethyl acetate, amyl acetate, butyl acetate</td>
</tr>
<tr>
<td>4</td>
<td>Dissolving solvent/co-solvent</td>
<td>Ethyl alcohol, butyl alcohol</td>
</tr>
<tr>
<td>5</td>
<td>Plasticizing agents</td>
<td>d-butyl phthalate, n-butyl stearate</td>
</tr>
<tr>
<td>6</td>
<td>Coloring agent</td>
<td>5% titanium dioxide (TiO2)</td>
</tr>
<tr>
<td>7</td>
<td>Nacreous/pearly pigments</td>
<td>Guanine crystal</td>
</tr>
<tr>
<td>8</td>
<td>Miscellaneous substances</td>
<td>(1) suspending agent (2) perfumeries</td>
</tr>
</tbody>
</table>

1. **Film Forming Agents:** The selection of a film forming agent is an important step in the formation of a relevant type of nail lacquer. The most commonly used film forming substance is nitrocellulose, due to the following properties.

   - The films formed using nitrocellulose pigment stay flexible for a sufficient period of time.
   - It has good adherent property and hence does not allow any chipping and peeling.
   - Its solvent retaining capacity is very low.
The films formed by nitrocellulose are impermeable to water and air and hence fungal infections can be eliminated.

It imparts relevant transparency to the nail enamel.

When compared to other film forming agents it is quite hard, tough and has good abrasion resistance ability.

Other Film Forming Substances: Several grades of nitrocellulose are available with varying viscosities but only low viscosity grades are used for the preparation of nail polishes. The degree of polymerization determines the viscosity which is necessary in order to prepare a nail polish of required consistency.

Two types of grades of nitrocellulose are readily used for nail polish preparation. They may be 'RS' and 'SS' type. The most common used type is 'RS' grade with viscosity range 0.25 to 0.5 cps. When nitrocellulose is used alone it produces a poor gloss and hence in order to avoid this resins are added.

2. Resinous Substances: Resins enhance the glossy nature of the nail polish and also impart adhesive property.

- Natural resins such as benzoin, shellac, damar, sandarac and ester gums were used initially but have been replaced by synthetic resins as they provide good gloss, better adhesion and also increased water resistance capacity.
- The most commonly used synthetic resin is sulphonamides-formaldehyde resin. It is a polymer made by mixing equimolar proportions of formaldehyde and para toluene sulphonamide.
- The two commercial types of aryl-sulphonamide-formaldehyde forms are santolite MHP and santolite MS 80 percent.
- The santolite MHP forms a harder film and the santolite MS 80% provides good gloss, flexibility and now property. But the resin combination is known to cause certain allergic reactions and hence it has now been replaced by other synthetic resins such as polystyrene, polyvinyl polyacrylic ester.
- Acrylic esters are compatible with nitrocellulose and they provide excellent gloss, adhesion, durability, good flexibility etc.

3. Solvent System: The solvents normally used for preparation of nail polishes may be volatile organic liquids that can dissolve all the ingredients and make a homogenous and uniform preparation. The solvent should be volatile enough in order to leave a continuous, impermeable and hard film but the evaporation should not be too rapid. The selection of a solvent plays an important role in order to provide a balanced rate of evaporation.

Generally a mixture of solvents is preferred rather than a single solvent. The solvents used for the formation of nail polishes are of the following types.

(a) Low Boiling Solvents: They include the solvents having boiling point below 100°C. They take more time to evaporate. Examples for low boiling solvents with their respective boiling points are as follows:
(b) **Medium Boiling Solvents**: these are solvents that have a boiling point ranging between 100°C to 150°C. The example of medium boiling solvents with their boiling point are as follows:

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Boiling point</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amyl formate</td>
<td>110°C</td>
</tr>
<tr>
<td>Butyl alcohol</td>
<td>113°C</td>
</tr>
<tr>
<td>Diethyl carbonate</td>
<td>126°C</td>
</tr>
<tr>
<td>Ethylene glycol Monoethyl ether</td>
<td>135°C</td>
</tr>
<tr>
<td>Ethyl lactate</td>
<td>135°C</td>
</tr>
<tr>
<td>Butyl propionate</td>
<td>145°C</td>
</tr>
</tbody>
</table>

(c) **High Boiling Solvents**: Liquids with boiling points more than 150°C are regarded as high boiling solvents. Examples with their boiling points are as follows:

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Boiling point</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclohexanone</td>
<td>154°C</td>
</tr>
<tr>
<td>Methyl Cyclohexanone</td>
<td>160°C</td>
</tr>
<tr>
<td>Diacetone alcohol</td>
<td>164°C</td>
</tr>
<tr>
<td>Methyl hexalin</td>
<td>165°C</td>
</tr>
<tr>
<td>Ethyl hexalin</td>
<td>185°C</td>
</tr>
<tr>
<td>Butyl lactate</td>
<td>185°C</td>
</tr>
<tr>
<td>Cyclohexanone pthalate</td>
<td>190°C</td>
</tr>
</tbody>
</table>

Generally, a combination of two or more solvents is preferred over a single solvent. The solvent system used in the preparation of nail polish influences the ease of its application. It also influences its drying rate and hardening ability and other characteristic properties of the film such as gloss shine etc. The solvent combinations should not have either too high or too low evaporation rates. Solvents that evaporate very quickly may cause intense cooling. This may cause precipitation of moisture from surrounding atmosphere making the film dull with
unattractive finish blushing. The phenomenon of blushing and blooming can be prevented during the preparation of a nail polish by selecting a suitable solvent.

The viscosity of a nail lacquer is also influenced by the boiling point of the solvent. Lower the boiling point of the solvent, lower will be the viscosity of the resultant nail lacquer and hence better flow property. The rate of evaporation of solvents depends on many factors such as specific heat, latent heat of evaporation, molecular weight, degree of association etc. The solvent, with high boiling points generally provide at brighter film than low boiling point solvent.

4. Diluting Solvents/ Co-solvents: They are not the actual solvents for the dissolution of nitrocellulose but are the co-solvents which increase the strength of the normal solvents. The various reasons for the addition of diluents are to:

- Maintain the viscosity of the lacquer to form a stable film.
- Increase the solubility of the incorporated resins, thus working as a co-solvent.
- Abate the effect of freshly applied polish or a recently applied lacquer.
- Reduce overall cost of the product since the solvents used might be costly.
- The most commonly used diluents are alcohols such as ethyl alcohol, butyl alcohol etc., they may also be used in combination with their esters. Example: Ethyl alcohol with ethyl acetate etc.,

The quantity of diluent used may also influence the formation of a good film. The limit for use of diluent may be expressed in terms of tolerance ratio or dilution ratio. The dilution may be defined as the maximum ratio of the diluent to the solvent (diluents/solvent) that can be tolerated by nitrocellulose solution without causing precipitation of nitrocellulose pigment.

Thus, selection of a proper combination of diluent and the solvent system is necessary. The combination should be such that the diluent should have a faster evaporation rate than the solvent system which would prevent the precipitation of nitrocellulose due to reduction in diluent solvent ratio. Thus, a clear, smooth and continuous film may be formed rather than a rough and cloudy film. The other examples of diluents beside alcohol are benzene, Xylene, toluene etc.

5. Plasticizing Agents: The plasticizing agent constitutes an important part in the formulation of nail polish preparation. Plasticizers are used for the following purposes.

- In order to improve the flexibility of the nail lacquer and minimize its tendency to shrink in order to form a uniform film.
- The nitrocellulose fibres alone make a dull and brittle film, but the addition of a plasticizer increases the gloss and adhesive property. Example: Castor Oil.

Functionally plasticizers may be divided into two types:

(a) Solvent Plasticizers

(b) Non-solvent Plasticizers
(a) **Solvent Plasticizers:** Solvent plasticizers, besides imparting flexibility to the nail polish, may also act as solvents for the dissolution of nitrocellulose. Many of them are the high molecular weight esters that have low volatility and relatively high boiling point. Example: butyl acetyl ricinoleate.

(b) **Non-solvent plasticizer:** These are not compatible with nitrocellulose and hence can’t be used alone. They cannot act as solvents but only act as plasticizer.

- The ideal properties for the choice of a good plasticizer could be as follows:
  - It should be compatible with other ingredients of the preparation.
  - It should be able to impart flexibility and enhance the glossiness and adhesive property of the nail polish.
  - It should not evaporate quickly.
  - It should not affect the stability of the preparation.
  - It should not affect colour of the product.
  - It should be non-irritating and non-toxic to the skin.
  - It should be odourless and colourless.
  - It should not cause any change in viscosity of the preparation.

The most commonly used plasticizers are dibutyl phthalate, n-butyl stearate, butyl glycolate, tributyl phosphate, resorcinol diacetate, castor triethyl citrate, dibutyl tartrate, dibutoxy ethyl phthalate, butyl acetyl ricinoleate.

Dibutyl phthalate and glycolate plasticizers are considered to be the best as they provide better hardness, feel and adhesion to the nail. They generally contribute 5% of the total mixture or as 25% to 30% in combination with the film forming agents.

The use of acetylated monoglycosides along with other plasticizers may increase the stability and flexibility of the product thus improving the long lasting ability.

6. **Colouring Substances:** The colouring substances also form an important component of the nail lacquer as they are required to impart a desirable shade.

- It should also be able to opacify the nail lacquer so that the most delicate shade may be able to cover the nail.
- More than 10 basic colours are required to produce large variety of shades used in polishes.
- All the colours must conform to the terms and conditions of the Drugs and Cosmetics Act.
- The coloured substances are available as colouring agents and are incorporated with the pigments and lakes.
- They are mostly available in the form of dispersion. The usual concentration is between 3 to 5%. Examples: Lithopone or 5% titanium dioxide is incorporated along with lakes to produce pastel shades. Iron oxides are used to produce brown or tan shades. The dinitrobenzene pigments are used to produce brilliant brown colours.
7. **Nacreous/ Pearlescent Substances**: They are used in order to produce an iridescent or a nacreous appearance. They have high refractive index and hence produce a glow when light falls on them. The can be obtained either from nun-al or synthetic origin.

(a) **Natural Pigment**: The example for the substance from natural origin is guanine crystals. Chemically they are 2-amino, 6-oxypurine crystals. They are obtained from the skin and the scales of fish. They are marketed mostly in the form of suspensions or pastes.

(b) **Synthetic Pigments**: Synthetic pigments can be obtained from the coating of bismuth oxychloride or titanium dioxide or the flakes or platelets of mica. They are less expensive than natural pigments.

8. **Miscellaneous Agent**:

(a) **Suspending Agents**: Suspending agents such as Bentone - 27 and Bentone-34 are most commonly used in nail polishes in order to prevent settling of the pearlescent pigments, thus avoiding sedimentation. The concentration of these substances varies between 0.5 to 2%.

(b) **Perfumes**: Perfumes are basically used to cover the odour of other ingredients and to provide a pleasant smell. Mostly synthetic perfumes are used in an optimum quantity of about 1% concentration. The formulae for the preparation of nail lacquer are as follows:

<table>
<thead>
<tr>
<th>Formula-1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrocellulose (film former)</td>
<td>7 g</td>
</tr>
<tr>
<td>Dibutyl phthalate (plasticizer)</td>
<td>5 g</td>
</tr>
<tr>
<td>Polyvinyl acetate (resin)</td>
<td>8 g</td>
</tr>
<tr>
<td>Methylene chloride</td>
<td>29.4 g</td>
</tr>
<tr>
<td>Ethylene glycol monomethyl ether (solvent)</td>
<td>28 g</td>
</tr>
<tr>
<td>Diethyl glycol monomethyl ether (solvent)</td>
<td>2 g</td>
</tr>
<tr>
<td>Ethyl alcohol (diluents)</td>
<td>14 g</td>
</tr>
<tr>
<td>Perfume oil</td>
<td>6 g</td>
</tr>
<tr>
<td>Color</td>
<td>0.6 g</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formula-2</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrocellulose (film former)</td>
<td>10 g</td>
</tr>
<tr>
<td>Sentolite MHP (resin)</td>
<td>10 g</td>
</tr>
<tr>
<td>Dibutyl phthalate (plasticizer)</td>
<td>3 g</td>
</tr>
<tr>
<td>Ethyl alcohol (diluents)</td>
<td>6 g</td>
</tr>
<tr>
<td>Ethyl acetate (solvent)</td>
<td>20 g</td>
</tr>
<tr>
<td>Butyl acetate (solvent)</td>
<td>15 g</td>
</tr>
<tr>
<td>Toluene (solvent)</td>
<td>36 g</td>
</tr>
</tbody>
</table>
### Formula-3

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nitrocellulose (film former)</td>
<td>4 g</td>
</tr>
<tr>
<td>Dibutyl phthalate (plasticizer)</td>
<td>4 g</td>
</tr>
<tr>
<td>Polypropyl methacrylate (resin)</td>
<td>18.6 g</td>
</tr>
<tr>
<td>Ethyl alcohol (diluents)</td>
<td>25.6 g</td>
</tr>
<tr>
<td>Butyl acetate (solvent)</td>
<td>23.9 g</td>
</tr>
<tr>
<td>Toluene (solvent)</td>
<td>23.4 g</td>
</tr>
<tr>
<td>Color</td>
<td>0.5 g</td>
</tr>
</tbody>
</table>

#### 5.2 The preparation of nail lacquers

- The preparation of nail polishes can be carried out as follows:
  - The film forming substance i.e., nitrocellulose is dissolved in a suitable solvent.
  - Resins plasticizers can be added directly or after dissolving them in small amounts of solvent.
  - The finely divided pigments are added by forming dispersion of the pigment as they form aggregates. The dispersion can be formed by milling the pigments in a ball mill or a triple roller mill.
  - The dispersion of the pigments, nitrocellulose and plasticizer are ground together in a solvent in order to form a plastic mass.
  - The final mixing of the ingredients for the manufacture of nail polishes is carried out in stainless steel tanks with a stirrer.
  - Initially, the tank is charged with the diluent and nitrocellulose (suitably wetted the diluent) is added to it.
  - The plasticizer and the resin are added next and the mixing process continues.
  - The mixing process is carried out till sufficiently uniform solution is formed. The clear lacquer is then subjected to filtration and centrifugation in order to remove any particles.

#### 5.3 Evaluation of Nail Lacquers

The various methods required for the evaluation of nail polishes are as follows.

1. Test for Non-volatile Content: The test is done in order to check the quantity of the non-volatile content in the preparation. The method is known as dish method and involves a simple process described below:
   - The sample is spread on a flat plate as a circle 8 cms in diameter.
   - The quantity is weighed and kept in an oven at a temperature o 105 for 1 hr.
   - The quantity of substance remaining on the plate is weighed and this constitutes the non-volatile content.

2. Rate of Drying: The test is done in order to check the rate of evaporation of the preparation. It involves a simple process in which the film is applied with an applicator on to a completely non-porous surface. It is kept at 25°C and 50% RH and the time required to dry
is noted by touching it with finger. When no matter is adhered to the finger tip, then the product is said to be completely dried.

3. **Colour of the Product:** The colour of the product is tested by comparing it with, a standard colour. This can be done by applying the standard colour on one nail and the prepared product on the adjacent nail. From this comparison, the contrast in the colours can then be easily noted.

4. **Test for Smoothness of the Film:** The smoothness is the most important characteristic of the film. The surface property can be studied by the microscopic analysis. The film should not contain any foreign matter or particles of the coating material. It should also be free from the orange peel effect when seen under microscope.

5. **Estimation of Gloss:** The gloss of the product can be determined by the use of an instrument that works on the principle of reflection of light.

6. **Test for Hardness of the Film:** The test is done in order to measure the extent of hardness of the substance.

   - It is done by spreading the film on a glass plate and then drying it for 48 hrs at 25°C.
   - It is then further dried at 70°C for 2hrs.
   - It is then cooled at 25°C for 48 hrs.
   - The hardness is then checked by applying mechanical force externally.

7. **Test for Adhesive property:** This is done in order to measure the extent of adhesion of the film with adhering material. This is done by the following method.

   - The film is spread on metal surface and allowed to settle for some time.
   - The adhesion character is then determined by measuring the mechanical force applied externally to remove the film.

8. **Test for Resistance to Abrasion:** This is done by applying mechanical abrasive forces externally on the film surface. The surface characteristic of the film before and after the application of abrasive force are then studied.

9. **Test for Resistance to Water Permeability:** This is a measure of resistance of the film towards absorption of water. This is done as follows.

   - A continuous film is spread on the surface of a metal plate.
   - The plate is then immersed in water.
   - The weight of the film before and after the immersion into water is noted.
   - An increase in the weight is calculated. The lesser the increase in weight, the greater is the water resistance.

10. **Test Application Property:** it is a measure of ease of application of the product. It is carried out more reliably by applying on nails. The degree of evenness and smoothness of brushing and the presence of any air bubbles are checked out.
11. Test for Viscosity: it is the most important parameter that determines the evenness of application.

- The viscosity can be measured by using Brookfield’s viscometer.
- It can be easily carried out by checking the flow of product from the applicator and comparing it with standard product.

12. Test for Stability: it is the measure of long lasting ability of the product. It can be done by using the acceleration stability test.
6. Creams

Definition:

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.

6.1 Classification

Creams are classified according to their functions. They are:

1. Cleansing and Cold Creams.
2. Foundation and Vanishing Creams.
5. All-purpose Creams is Cleansing and Cold Creams

1. Cleansing and Cold Creams:

Cleansing Creams: They are used for the purpose of removing makeup, surface grime (layer of dirt on skin) and secretions of skin from the face and throat respectively.

Properties:

- They are easy to apply.
- They spread easily on the skin.
- They are pleasant in appearance.
- They cause less irritation to the skin.
- They should melt or liquefy when applied on to the skin.
- They should produce flushing action on skin and its pore openings.
- They should form an emollient film on the skin after application.
- They should not make skin dry which happens in case, when the skin is washed with water and soap.
- They should remove chemicals of facial makeup effectively. They dissolve the greasy binding materials which hold the pigment and finally remove them.
• They should remove solidified oil, sebum, sebum plaques and surface oil layer from the skin.
• They also help in softening, lubricating and protecting skin apart from cleansing purposes.

They are applied on face and throat with the help of finger tips. Then the fingers are rotated upwards on the skin for spreading purpose. Tissue paper or cotton wool used to remove the residue of the cream. The layer which is left on the skin should be non-occlusive and emollient in order to prevent drying. Cleansing creams are of two types. They are:

(i) Bees wax-borax type / Emulsified type. (ii) Liquefying type.

(i) Bees Wax-borax Type / Emulsified Type: It is considered as an important formulation in cleansing creams. This type of preparation liquefies when 'applied to the skin, which helps in easy spreading. It is white, lustrous and good consistency.

It is an oil-in water type of emulsion, in which high percentage of mineral oil is present. This mineral oil helps in imparting cleansing property. Phase inversion takes place due to evaporation of water after the creams are rubbed on the skin. The phase inversion (i.e., water in-oil type) helps in imparting the cleansing action.

<table>
<thead>
<tr>
<th>Formula-1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral oil (lubricant)</td>
<td>28 g</td>
</tr>
<tr>
<td>Isopropyl myristate (lubricant and emollient)</td>
<td>14 g</td>
</tr>
<tr>
<td>Acetoglyceride (luster)</td>
<td>2.5 g</td>
</tr>
<tr>
<td>Petroleum jelly (lubricant)</td>
<td>7.5 g</td>
</tr>
<tr>
<td>Beeswax (emollient)</td>
<td>15 g</td>
</tr>
<tr>
<td>Borax (buffer)</td>
<td>1 g</td>
</tr>
<tr>
<td>Water (vehicle)</td>
<td>32 g</td>
</tr>
<tr>
<td>Preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>q. s</td>
</tr>
</tbody>
</table>

Mineral oil, isopropyl myristate, acetoglyceride, petroleum jelly and bees wax heated to a temperature of about 75°C in a separate glass container (ingredients having least melting point are melted first and then high melting point ingredients are melted). This is mixture A.

In other glass container borax and water are heated to same temperature i.e., 75°C, preservatives are dissolved in water before heating. This is mixture B.

Mixture B is added to the mixture A slowly, along with continuous stirring. Stirring carried out until a thick stable emulsion is formed.

Perfume is added to the preparation when it attains a temperature of 35°C and stirring is carried out.

Then the preparation is passed through a triple roller mill for milling purpose.

Preparation is transferred and stored in a suitable container.
(ii) Liquefying Type: This type of creams consist of a mixture of oil and water which are translucent in nature. They are translucent in nature. They are anhydrous creams with thixotropic character i.e., they liquefy when applied on skin.

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Uses</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Paraffin wax</td>
<td>Responsible for thixotropic character.</td>
</tr>
<tr>
<td>2. Mineral oil and wax (proportion should be proper)</td>
<td>Phase separation, sweating and granular appearance is avoided.</td>
</tr>
<tr>
<td>3. Amorphous ozokerite and petrolatum</td>
<td>To avoid formation of crusty surface.</td>
</tr>
<tr>
<td>4. Lanolin, cetyl, alcohol, spermaceti and cocoa butter</td>
<td>They impart emollient property</td>
</tr>
<tr>
<td>5. Zinc oxide, titanium dioxide, magnesium stearate, zinc stearate or hydrous lanolin (used in 2% concentration)</td>
<td>To impart opaque appearance</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral oil (lubricant)</td>
<td>80 g</td>
</tr>
<tr>
<td>Petrolatum (protective agent)</td>
<td>15 g</td>
</tr>
<tr>
<td>Ozokerite wax (humectants)</td>
<td>5 g</td>
</tr>
<tr>
<td>Preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>q. s</td>
</tr>
</tbody>
</table>

Method:
- Mineral oil, petrolatum and ozokerite wax are heated together to a temperature of about 65°C (First ozokerite wax is melted followed by petrolatum and mineral oil).
- The above mixture is cooled along with continuous stirring.
- Preservative and perfume are added to the mixture after it attains a temperature of 40°C.
- Then the preparation is transferred and stored in a syllabic container.

Cold Creams: These types of 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.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>White beeswax (emollient)</td>
<td>20 g</td>
</tr>
<tr>
<td>Mineral oil (lubricant)</td>
<td>50 g</td>
</tr>
<tr>
<td>Distilled water (vehicle)</td>
<td>28.8 g</td>
</tr>
<tr>
<td>Borax (buffer)</td>
<td>0.7 g</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>0.5 g</td>
</tr>
</tbody>
</table>
Method:

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

2. Vanishing and Foundation Creams:

These creams are also referred to as 'Day Creams' as they are applied during day times. These creams provide emollient as well as protective action to the skin against environmental conditions by forming a semi-occlusive residual-film. This film is neither greasy nor oily.

(a) Vanishing Creams: They are oil in water type of emulsion. When applied on the surface of skin, they spread as thin oil less film which is not visible to the naked eye. Hence, they are called as vanishing creams. They are used to hold powder on the skin as well as to improve adhesion.

Properties:

- It should have high melting point.
- It should be pure white in colour.
- It should possess very little odour.
- It should have less number of iodine.

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Uses</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Main ingredient</td>
<td>It governs the consistency of the cream and imparts pearlescent property to the cream by forming crystals.</td>
</tr>
<tr>
<td>Example: stearic acid</td>
<td></td>
</tr>
<tr>
<td>2. Humectants</td>
<td></td>
</tr>
<tr>
<td>Example: glycerin, sorbitol, Propylene glycol</td>
<td></td>
</tr>
<tr>
<td>3. Alkalies</td>
<td></td>
</tr>
<tr>
<td>Example: (a) Potassium hydroxide</td>
<td>It imparts fine texture and consistency without providing harshness</td>
</tr>
<tr>
<td>(b) Sodium hydroxide</td>
<td>It is used in combination with potassium hydroxide because it forms hard cream, when used alone.</td>
</tr>
</tbody>
</table>
(c) Carbonates i.e., potassium and sodium carbonate

They are widely used, because they liberate carbon dioxide due to this, creams become spongy.

(d) Ammonia

It is effective, but difficult to handle because of odour and volatility. It is also make cream yellow in color with age. It is used in combination with potassium hydroxide to produce a white emulsion.

(e) Borax

4. Emulsifying agent.
Example: triethanolamine soap, Amino glycol soap or Glyceryl monostearate

It provides stability to the cream. If hard water is used, it leads to the formation of soaps of lime and magnesium, which causes inversion of emulsion and hence stability is reduced.

5. Purified water (i.e., distilled and deionized)

They prevent deterioration caused by bacteria or fungi.

6. Preservatives
Example: methyl paraben and propyl paraben

7. Perfume i.e., perfume solvent or perfume is dissolved in alcohol. They should be added when the cream attains a temperature of about 40°C. Example: geranium, sandal wood, lavender oil, terpineol etc.

It provides odour to the cream and also has aesthetic value.

<table>
<thead>
<tr>
<th>Formula-1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stearic acid (lubricant)</td>
<td>24 g</td>
</tr>
<tr>
<td>Potassium hydroxide (softening agent)</td>
<td>1 g</td>
</tr>
<tr>
<td>Water (vehicle)</td>
<td>64 g</td>
</tr>
<tr>
<td>Glycerin (humectants)</td>
<td>10.5 g</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>0.5 g</td>
</tr>
</tbody>
</table>

**Method:**

- Stearic acid is melted in a container by using water bath.
- Potassium hydroxide is dissolved in water and then glycerin is added. This mixture is heated to a temperature of about 75°C. This is aqueous phase.
- Slowly aqueous phase is added to melted stearic acid along with continuous stirring.
- Perfume is added to the preparation when it attains a temperature of 40°C.

**Note:** During cooling, care should be taken, as the cream passes, through two transformations i.e., softening and hardening. Then cream attains its desired form. Even
formation of crust on the top surface of cream should be avoided by stirring to prevent lump formation.

(b) **Foundation Creams**: They provide emollient base or foundation to the skin. They are applied before applying face powder or other preparations of make-up.

**Properties:**
- Adhesion of powder to the skin is improved by these creams, as they possess good holding capacity.
- They should be easily spread on the skin.
- They should be non-greasy in nature.
- They should be capable of leaving a non-occlusive film on the skin after application.

**Ingredients**: Ingredients are similar to that of vanishing creams. Except some of the ingredients which are as follows:

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Uses</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Humectant and lanolin</td>
<td>They cause retention of powder on the skin</td>
</tr>
<tr>
<td>2. Mineral oil</td>
<td>It improves powder adhesion to the skin</td>
</tr>
<tr>
<td>3. Isopropyl myristate, butyl stearate and ester</td>
<td>They also improve adhesion power due to their low surface tension property</td>
</tr>
<tr>
<td>4. Pigments like titanium dioxide, talc, calamine</td>
<td>They impart color</td>
</tr>
</tbody>
</table>

They are of two types:

(i) Pigmented Foundation Creams: They are colored creams.

(ii) Unpigmented Foundation creams: These creams do not contain pigments in the formulation.

<table>
<thead>
<tr>
<th>Formula-2</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lanolin (emollient)</td>
<td>2 g</td>
</tr>
<tr>
<td>Cetyl alcohol</td>
<td>0.50 g</td>
</tr>
<tr>
<td>Stearic acid (lubricant)</td>
<td>10 g</td>
</tr>
<tr>
<td>Potassium hydroxide (softening agent)</td>
<td>0.40 g</td>
</tr>
<tr>
<td>Propylene glycol (humectants)</td>
<td>8 g</td>
</tr>
<tr>
<td>Water (vehicle)</td>
<td>79.10 g</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>q. s</td>
</tr>
<tr>
<td>Preservatives</td>
<td>q. s</td>
</tr>
</tbody>
</table>
Method:

Lanolin, cetyl alcohol, stearic acid and potassium hydroxide are heated to a temperature of about 75°C in one container. This is oily phase.

In another container, water and propylene glycol are heated to same temperature i.e., 75°C. Preservatives should be dissolved in water before heating is carried out. This is aqueous phase.

Then slowly aqueous phase is added to oily phase along with continuous stirring until the preparation becomes cold. 4. Perfume is added to the preparation when the above mixture reaches a temperature of 35°C.

Finally the preparation is passed through a triple roller mill for milling purpose, (milling is carried out to obtain a good product).

Foundation Make-up: Foundation make-up cream helps in overcoming the trouble associated with foundation creams i.e., application of foundation cream is a two-step process where it acts as a base to hold the powder makeup. These two step can be avoided by using foundation make-up. These are available various forms especially the liquid foundation make-up is popular because it easy to apply compared to lose powders and it also provide smooth appearance to the skin.

Surfactants present in the foundation make-up may allow the pigments or colours to penetrate into hair follicles and fissures present in the epidermis of the skin. Hence, should be completely removed after application.

<table>
<thead>
<tr>
<th>Formula-3</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lanette wax</td>
<td>8 g</td>
</tr>
<tr>
<td>Stearic acid (lubricant)</td>
<td>8 g</td>
</tr>
<tr>
<td>Water (vehicle)</td>
<td>64 g</td>
</tr>
<tr>
<td>Glycerin (humectants)</td>
<td>10 g</td>
</tr>
<tr>
<td>Powder (base)</td>
<td>10 g</td>
</tr>
<tr>
<td>Color</td>
<td>q. s</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>q. s</td>
</tr>
<tr>
<td>preservatives</td>
<td>q. s</td>
</tr>
</tbody>
</table>

Method:

Lanette wax, stearic acid and water are heated to a temperature of about 85-900 C in a separate container. Preservative should be dissolved in water before heating of mixture. This is mixture A.

Colour and perfume are added to powder base and mixed. Then this mixture is dispersed in glycerin. This is mixture B.

Mixture B is added to mixture A and then it is mixed thoroughly.
3. Night and Massage Creams:

(a) **Night Creams:** The preparations which are applied during night time and removed in the morning are called night creams.

(b) **Massage Creams:** The preparations which are gently applied and rubbed on the skin through massage technique are called massage creams. Skin becomes dry due to the following reason:

- When stratum corneum is exposed to low humidity, excessive loss of water takes place which attributes to dryness of skin.
- When the lower layer of epidermis does not hydrate properly.
- When the skin is in contact with soap or solutions of detergent for long time.

**Reason:** The hygroscopic substances present in the stratum corneum of the skin are responsible for water binding capacity. These hygroscopic substances are protected by fatty materials which are not easily removed by water alone. But with the use of solvent and water or detergent solutions, these substances are removed and makes the skin dry. In order to make the dry skin smooth, water is incorporated into the horny layer. This can be achieved by:

- Increasing the process of diffusion of the living cells of epidermis.
- Water is incorporated into the horny layer of the epidermis from outside i.e., by using creams, lotions etc.,
- Surface of the skin is occluded in order to prevent evaporation of water. Creams i.e., night and massage creams act in the same way in order to make the dry skin smooth. Hence, these creams are also known as emollient creams.

**Properties:**

- These creams are formulated with fatty substances which help in easy spreading on the skin.
- These creams help in providing occlusive layer to the skin, which reduce the rate of water loss from the transepidermal layer. The occlusive layer is also responsible for providing moisturizing effect on the skin.

**Ingredients:** Ingredients are either water soluble or fat soluble.

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Uses</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Water soluble ingredients Example: Propylene glycol, Glycerol, sorbitol.</td>
<td>They reduce evaporation of water in case of oil-in-water type of emulsion. The activity of retaining water in external Phase is known as emollient activity, which in turn provides water to stratum corneum.</td>
</tr>
</tbody>
</table>
2. Fat soluble ingredients
Example: mineral oil, petroleum jelly, Paraffin, ceresin, dimethyl polysiloxanes, Methyl phenyl polysiloxanes etc.

<table>
<thead>
<tr>
<th>Formula -1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral oil (lubricant )</td>
<td>38 g</td>
</tr>
<tr>
<td>Petroleum jelly (lubricant )</td>
<td>8 g</td>
</tr>
<tr>
<td>White beeswax (emollient )</td>
<td>15 g</td>
</tr>
<tr>
<td>Paraffin wax (base and lubricant )</td>
<td>1 g</td>
</tr>
<tr>
<td>Lanolin (emollient )</td>
<td>2 g</td>
</tr>
<tr>
<td>Borax (buffer)</td>
<td>1 g</td>
</tr>
<tr>
<td>Water (vehicle)</td>
<td>35 g</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>q. s</td>
</tr>
<tr>
<td>Preservatives</td>
<td>q. s</td>
</tr>
<tr>
<td>Antioxidant (to prevent oxidation )</td>
<td>q. s</td>
</tr>
</tbody>
</table>

**Method:**
- Mineral oil, petroleum jelly, white beeswax, Paraffin wax and lanolin are heated to a temperature of about 75°C in a one container. This is mixture A.
- Borax, water and antioxidant are heated in another separate container to the same temperature i.e. 75°C. Preservative is dissolved in water before heating the mixture. This is mixture B.
- Slowly mixture B is added to mixture A along with continuous stirring.
- Perfume is added after the preparation has attained a temperature of about 35°C.

**4. Hand and Body Creams:** Due to exposure of skin to water, soaps and detergents many times a day, removal of lipids and other secretions from the skin occurs. Cold and dry winds are responsible for chapping of the skin. Chapping occurs due to loss of moisture from the skin, which is also associated with cracking.

Water is sufficient enough to treat the dryness of the skin, but evaporation of water takes place rapidly, which again, makes the skin dry and no emollient effect is produced.

In case, if hands are immersed in water for longer time then abnormal hydration takes place. This hydration will lead to swelling of cells in stratum corneum, which ultimately results in rupturing of cells.

Hence, hand and body creams are formulated with suitable emollient, which not only make water available but also regulates the water take-up by the cells of stratum corneum.

**Properties:**
They are easy to apply.
They help in softening or imparting an emollient effect to hands.
They should not leave behind a sticky film after their application.
They should not interfere with perspiration of the skin as it may reduce bioavailability.
Perfume and colour should be added in the cream preparation for pleasant smell and appearance.

Ingredients:

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Uses</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Humectants</td>
<td>To prevent evaporation of water from the skin.</td>
</tr>
<tr>
<td>Example: propylene glycol, glycerin and Sorbitol.</td>
<td></td>
</tr>
<tr>
<td>2. (a) natural gums</td>
<td>They form occlusive film on the skin, which in turn prevent evaporation of water.</td>
</tr>
<tr>
<td>Example: karaya, acacia, tragacanth, Agar-agar.</td>
<td></td>
</tr>
<tr>
<td>(b) synthetic substances</td>
<td></td>
</tr>
<tr>
<td>Example: carboxy celluloses, polyvinyl alcohol</td>
<td>They are used to impart an emollient property.</td>
</tr>
<tr>
<td>3. Emollients</td>
<td>They help to increase the porosity of the skin.</td>
</tr>
<tr>
<td>Example: mineral oil, waxes and lanolin or its derivatives, sterol, phospholipids, fatty acid, fatty acid ester, fatty alcohols etc.</td>
<td>They help in preventing scaling of the surface of the skin.</td>
</tr>
<tr>
<td>4. Healing ingredients</td>
<td>They prevent the growth of microorganisms.</td>
</tr>
<tr>
<td>Example: allantoin, urea, uric acid</td>
<td>They are used to impart aesthetic value to creams.</td>
</tr>
<tr>
<td>5. Alkyl ester of poly unsaturated (C18) fatty acids, Linoleic acid and linolenic acid</td>
<td></td>
</tr>
<tr>
<td>6. Preservatives like methyl paraben, propyl paraben and butyl para hydroxybenzoate.</td>
<td></td>
</tr>
<tr>
<td>7. Perfumes like phenyl ethyl alcohols, pine, geranium, Bourbon, lavender, lilac type, light floral type etc.</td>
<td></td>
</tr>
</tbody>
</table>

Method:
- Isopropyl myristate, mineral oil, emulsifying wax and lanolin are heated in a container. This is mixture A.
- Glycerin, triethanolamine and water are heated in a separate container. Preservative is dissolved in water before heating the mixture. This is mixture B.
- Mixture B is added to mixture A along with continuous stirring until cream is formed.
- Perfume is added to the preparation when it reaches a temperature of 35°C.
- Finally, the preparation is passed through a triple roller mill for milling, which provides good texture.
5. All-purpose creams/sports creams: These creams are used by sport persons and also by people who do outdoor activities. Hence, they are called as sport creams.

- They are oily in nature but non-greasy type.
- They provide protective film to the skin.
- They make the rough surfaces of the skin smooth.
- When it is applied in more quantity, it act as
  (a) Nourishing agent
  (b) Protective cream in order to protect the skin from sunburn.
  (c) Night cream.
  (d) Cleansing cream
- When it is applied in less quantity, it act as
  (a) Hand creams
  (b) (b) Foundation creams

**Ingredients:** The various ingredients used in the formulation are as follows:

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Uses</th>
</tr>
</thead>
</table>
| 1. Wool alcohol  
It contains 28% of cholesterol which is obtained by saponification of wool of the sheep. | It helps in absorption of water. |
| 2. Antioxidants like butylated hydroxyanisole. | It prevents oxidation. |
| 3. Macrornamalline wax | It helps in easy spreading of the cream on the skin. |
| 4. Mineral oil, paraffin | They form a protective layer on the skin. |
| 5. Magnesium sulphate, The ions of magnesium are present in aqueous phase. | It helps to increase the stability of the cream. |
| 6. Preservatives like methyl paraben and propyl paraben. | They inhibit the growth of microorganism. |
**Method:**

1. Wool alcohol, hard paraffin, soft paraffin, liquid paraffin and antioxidant are melted.
2. Stirring is carried out until the preparation is cooled.
3. Perfume is added to the preparation, when it reaches a temperature of 35°C. Hydrous ointment can be prepared by using the same base ingredients but with the incorporation of equal amount of water.

### 6.2 Evaluation of Creams

Due to the use of number of additives, it is necessary to evaluate the effectiveness of the skin products. Evaluation is carried out by two methods. They are:

1. In-vitro methods
2. In-vivo methods.

#### 1. In-vitro Methods:

Tests are carried out to know the performance of the products. These tests also help in evaluating, new product concepts. Various instruments have been developed by the investigators to know the effect of temperature and humidity on the skin. Since, the softness of skin is directly related to the water content present in it. The effects of temperature and humidity on skin are studied by observing the changes in the mechanical properties of the stratum corneum. The instruments help in evaluating moisturizing capacity of the products and screening of raw materials used in the formulation.

Various techniques or instruments involved in in-vitro method are:

(a) Tensile strength tester

(b) Hargen's Gas Bearing Electro dynamometer (GBE)

(c) Occlusive potential of ingredients.

(d) Gravimetric analytical method.

(e) Thermal analytical methods.

(f) Electrical methods.

<table>
<thead>
<tr>
<th>Formula-1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wool alcohol (emollient)</td>
<td>6 g</td>
</tr>
<tr>
<td>Hard paraffin (soothing agent)</td>
<td>24 g</td>
</tr>
<tr>
<td>White soft paraffin (emollient)</td>
<td>10 g</td>
</tr>
<tr>
<td>Liquid paraffin (emollient)</td>
<td>60 g</td>
</tr>
<tr>
<td>Perfume (odour)</td>
<td>q. s</td>
</tr>
<tr>
<td>Antioxidant</td>
<td>q. s</td>
</tr>
</tbody>
</table>
(a) Tensile Strength Tester: This method is useful for determining the tensile property of the excised stratum corneum of the skin. It provides information on the water content present in stratum corneum and also acts as a screening device for moisturizing ingredients. The stress or strain characteristics of stratum corneum obtained from various sources can be studied by using this instrument (i.e., tensile strength tester), and it also helps in knowing the effects on stratum corneum passed through various treatments.

(b) Hargen's Gas Bearing Electro Dynamometer (CBE): This instrument is helpful in determining and monitoring the viscoelastic behavior of the skin. It also helps in determining the effects on the skin by passing it through various treatments. It is used both as in-vitro and in-vivo test.

Disadvantages: The instrument lacks sensitivity sometimes.

(c) Occlusive Potential of Ingredients: The occlusive potential of raw materials or ingredients used in the formulation of skin cream, are determined by knowing the water diffusion rate. Membranes used in this method can be stratum corneum of neonatal rat or artificial membrane.

(d) Gravimetric Analytical Method: This method is helpful in establishing relationship between water content present in stratum corneum and relative humidity. This is done by suspending hits of callus (undifferentiated mass of cells) in different dilutions of sulfuric acid. Then the weight of the sample (i.e., callus) is determined by using sensitive electro balance. This weight of the sample is taken after it reaches an equilibrium state (i.e., one week). After this the water content is determined by subtracting dry weight of the tissue and weight of the sample which has attained equilibrium state, (i.e., equilibrium value).

Water Content (Stratum Corneum) = Dry Weight of the Tissue - Equilibrium value.

This method is also useful in determining sorption and desorption phenomena which takes place in test stratum corneum after passing through various treatments.

Advantages:

• It is a simple method.
• It is inexpensive method.

Disadvantages:

• It is a time consuming method.
• It requires lot of labour efforts.

(e) Thermal Analytical Methods: Various thermal analytical methods like Differential Scanning Calorimetry (DSC), Thermo-Mechanical Analysis (TMA) and Thermo-Gravimetric Analysis (TGA) are used. They are used in order to provide information about the effect of temperature which causes changes in the stratum corneum. These methods also provide
information on physical properties and components of stratum corneum, but are not popular in determining the moisturizing efficacy.

(f) Electrical Methods: Various electrical properties such as capacitance, impedance and dielectric constant are measured by electrical methods which provide information about the variations in the water content present in the stratum corneum of the skin. One such method is four-point micro electrode method. This method helps in measuring the resistivity (resistance power) of the excised stratum corneum. It also helps in measuring electrolyte levels and water binding capacity of stratum corneum. This method is considered to be more sensitive and reliable than another electrical method except for measuring moisturizing efficacy.

**Advantages of In-vitro Method:**
- It provides data which is less variable.
- Environment can be easily controlled by this method.
- Large number of products are easily and rapidly evaluated or assessed.

**Disadvantage of In-vitro Method:** simulated and artificial environment which is not close to the real condition.

2. In vivo Methods: In-vivo methods are helpful in providing information on hydration or moisturization process of the skin. Various methods are:

   (a) Transpirometry
   (b) Scanning electron microscopy (SEM)
   (c) Optical microscopy and macro photography.
   (d) Skit friction
   (e) Sensitivity tests.

(a) **Transpirometry:** This method helps in measuring Trans Epidermal Water Loss (TEWL) of the skin which helps in providing information on moisturizing potential. In this method, skin surface of the fore arm is used, in this surface, a collection chamber is attached through which nitrogen or stream of air of known relative humidity is introduced. The water vapours leave the surface of the skin and enter into the collection chamber. Then the gas present in the chamber carries water vapour to suitable detection devices like dew point, hygrometry, thermal conductivity or gas chromatography. This method is useful in detecting three sources of water i.e., eccrine sweat, transepidermal water loss and stratum corneum water and also detects the water supplied by cosmetic products.

Note: Detection of eccrine sweat is troublesome due to its volume and sporadic nature. The excessive loss of eccrine sweat can be prevented by either conditioning the test in a cold temperature i.e., 20° C or by giving anticholinergic which help in avoiding excessive sweating.

(b) **Scanning Electron Microscopy (SEM):** Skin replicas are used in this method to know the effects of topical preparations on the skin conditions i.e., dry and rough skin (good substrate). Polyethylene beads are melted on the surface in order to get impression of skin on
the silicon rubber. This rubber is then metalized to prevent charging and observed under the microscope. This method provides surface architectures which bears no resemblance to artificiality and hence effects are easily determined.

(c) **Optical Microscopy and Macro photography**: with the help of low magnification photography, stereomicroscopic tests, biopsies of skin surfaces and microphotographs, the changes in the dry rough skin are observed before and after application of moisturizers. They also provide information on moisturizing potential preparations.

(d) **Skin Friction**: Damp (slightly wet) skin has high friction surface compared to wet and dry skin. Investigation of friction surface shows the relation between the effect of hydration on stratum corneum and process of moisturization. Frictional properties are also related to elastic nature of skin and helps in evaluating the performance of the product.

(e) **Sensitivity tests**: these tests are performed in order to measure the irritancy, sensitization potential and phototoxicity of the skin.

(i) **21 Day (or 3 Weeks) Cumulative Irritancy Patch test**: In this test, the test material is applied daily on the same site i.e., fore arms of 24 subjects under the occlusive tapes. Then score are recorded daily. This test is carried out for 21 days or until irritation produced on the fore arm. This irritation is noted as maximum score. The score ranges from 0-4, where '0' score indicates no visible reaction on typical erythema (redness of the skin due to dilation and congestion) of capillaries) and '4' score indicates erythema with edema and vesicular erosion (erosion of vesicles). This test can also be carried out with fewer subjects and less application of test material.

(ii) **Draize-shelanski repeat-insult Patch Test**: This test is carried out on 100 individuals to measure the extent of sensitization and irritation caused by the product to the skin. The test material is repeatedly applied on the same site under occlusion for 10 alternate days. After a gap of 7 days, test material is again applied to a new site only for 24 hours. The scores are recorded after the removal of occlusive tape. Then the score is again recorded after 24 hours. The score ranges from 0-4, where '0' score indicates no visible reaction on erythema and '4' score indicates erythema with edema and vesicular erosion.

(iii) **Kligman “Maximization” Test**: This test is used to measure sensitizing potential of the product, when it comes in contact with the skin. The test material is applied on the site by using an occlusive tape for a period of 48 hours. Then the site is treated with sodium lauryl sulfate solution on each exposure under occlusion. After a gap of 10 days, the test material is again applied on a new site under occlusion for a period of 48 hours, which is then treated with solution of sodium lauryl sulfate.

**Advantages:**

- The test consumes less time.
- The test materials are applied on fewer subjects only.
• Sodium lauryl sulfate solution is used as it helps in detecting weaker allergens easily and rapidly.

Sensitivity tests are also suitable for detecting weak irritants and contact sensitizers. If the tests give positive results then the product should not be immediately discarded or considered unsafe. The actual risk arises if the product is used for longer time or the product concentration is more or on the condition of the skin.

Example: Benzoyl peroxide is a potent sensitizer which is used in Draizeshelanski and Kligman maximization test. But, it still produces low sensitization in case of patients suffering from acne.
7. Toothpastes

Introduction

Dentifrices such as toothpastes, tooth powders and tooth gels are meant for the cleaning the surface of the teeth by removing the food debris and plaque adhered to surface of the teeth which is the main cause for tooth problems.

The general requirements for a dentifrice are as follows:

- It should be capable of cleaning the teeth adequately by removing food debris, plaque and stains efficiently.
- It should leave a pleasant, cool and refreshing sensation in the mouth.
- It should be harmless, non-toxic and should not cause irritation in the mouth or any ulcers in the buccal cavity.
- It should be able to maintain its flow properties all through its commercial period of storage.
- It should be easy to pack and easy to use.
- The abrasive character of the dentifrice should be under the limits of the standards and should not be harsh on the enamel and the dentine.
- It should confirm to the standards of the EC cosmetic directive which states that it is not liable to cause damage to human health when used under normal conditions.
- The assessment of any claims shall be certified based on properly conducted clinical trials.
- Most of all it should be economical to purchase in order to encourage regular and frequent use by common people.

The dentifrice with all the above mentioned qualities is said to be an efficient dentifrice.

7.1 Formulation

Toothpastes are the most popular form of dentifrices. They include the following ingredients which determine the quality and efficiency of toothpastes.

1. Polishing Agents / Abrasive Agents: The abrasives or the polishing agents are used to polish the teeth and remove food debris adhered to the surface of the teeth. They are used in concentration of about 20 - 50% of the total formulation.

They should possess the following characteristics:
(a) They should not produce any gritty sensation in the mouth.
(b) They should possess good abrasive properties.
(c) They should not lead to any incompatibilities and should be compatible with the other ingredients.
(d) They should be harmless to the enamel and the abrasive property should be under limits.
(e) They should provide a good shine to the enamel.

Fig. 7–2. Toothpaste

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Examples</th>
</tr>
</thead>
</table>
| Agents responsible for cleansing Action | (a) precipitated calcium carbonate  
(b) phosphates of calcium  
(c) dental graded silica / polymers of silica (SiO₂)ₙ  
(d) trihydrated alumina  
(a) sodium lauryl sulphate (ROSO₃Na)  
(b) sodium lauryl sarcosinate |
| Agents responsible for the formation Of toothpastes | (a) Sorbitol 70  
(b) Glycerin  
(c) Propylene glycol  
(a) Sodium carboxy methyl cellulose (SCMC)  
(b) Cellulose ethers |
| Agents responsible for improving Palatability | (a) Sodium saccharin  
(b) Chloroform  
(c) Cinnamon bark  
(d) Spearmint oil etc. |
| Miscellaneous agents |  |

The most commonly used Abrasive agents are as follow:

(a) Precipitated Calcium Carbonate (CaCO₃): It is also known as precipitated chalk and is available in a number of grades. The crystalline form of the precipitated chalk may be available as:

(i) Calcite: Contains rhombohedral crystals.

(ii) Aragonite: Contains orthorhombic crystals.
Advantages:

- It is of very low cost.
- It is available in different grades in white or off-white colours.
- The lighter grades are very stable and do not get hardened on storage.

Disadvantages:

- The abrasivity is not consistent within the lots of same grade of powder due to the presence of impurities.
- It is incompatible with sodium fluoride which is used as anticaries agent.

(b) Phosphates of Calcium: A large variety of insoluble calcium phosphates are used as abrasive agents. They may be as follows:

Dicalcium Phosphate (DCP) Dihydrate \([\text{CaHPO}_4\cdot2\text{H}_2\text{O}]\): It is a commonly used abrasive agent among the phosphate of calcium. Its properties and the advantages and disadvantages are as follows.

Advantages:

- It provides good flavour stability.
- Toothpastes made with Dicalcium phosphate are better than toothpastes made with chalk.
- They do not make use of additional whitening agents.
- The hardening of the paste during preparation is accelerated in the presence of fluoride ions.
- It has less abrasive effect on dentine.

Disadvantages:

- It is incompatible with sodium fluoride.
- The only source of fluoride is sodium monofluoro phosphate since it consists of free calcium ions that react with other fluoride sources leading to incompatibility.
- The DCP Dihydrate is unstable in its natural form and may convert into anhydrous form which may result in hardening of the paste.

The other commonly used phosphates of calcium are tricalcium phosphate, calcium pyrophosphate etc., The insoluble sodium metaphosphate, dibasic ammonium phosphate are also used as abrasive agents.

(c) Dental grade silica / Polymers of Silica \((\text{SiO}_2)_n\): They are polymer of silica that are commonly used as abrasive agents in the formulation of toothpaste `gels in large quantities. They are available in two forms as:

- Abrasive Form of Silica.
- Thickening Form of Silica.
Abrasive Silica: They are also referred to as xerogels. They possess good abrasive property and are used in low concentration. They have least effect on the consistency of the finished product.

Thickening silica: They are referred to as aerogels. The particles are small in size and possess a greater surface area. They have the ability to swell and provide a thickening effect to the pastes.

Advantages:
- The silicas are mostly used as abrasives in gels.
- They are inert and easily compatible with other ingredients.
- They provide good gloss to the dentine due to their high refractive index.
- They can be used in low concentration.

Disadvantage: The abrasive property is not consistent within the different grades.

Fig. 7–2. Toothpaste

(d) Trihydrated Alumina (Al₂O₃·3H₂O): It may be available in two forms: As suspension or as crystalline powder.

Advantages:
- It is less costly.
- It possesses stability with fluorides.
- It is easily available and is stable during storage.
- It is compatible with other ingredients.
- It possesses a good abrasive property.

Disadvantage: It has poor thickening property.

2. Foaming Agents / Surfactants: They are also known as wetting agents. The mechanism of cleansing action is by reducing the surface tension at the interface of the adhered material and enamel of the teeth.

They aid in abrasive action by wetting the surface of the teeth. They help in the diffusion of into narrow spaces, thus enhancing the cleansing action. The properties of the surfactants are as follows:

- It should be compatible with other ingredients of the formulation.
- It should possess good surface active property.
- It should be non-toxic and non-irritant to the oral mucosa of the buccal cavity.
• It should be tasteless.

The most commonly used surfactants are:

(a) Sodium Lauryl Sulphate \((\text{ROSO}_3\text{Na})\): It is used in concentrations of 0.5 to 2% in order to provide necessary foaming action.

**Advantages:**

• It is available in a large variety of graded forms.
• The recrystallized grades have good surfactant property.
• They are more compatible with other ingredients of the formulation.
• They have a neutral pH range.

**Disadvantages:**

• The nature of the foaming agent may be altered by the presence of any free alcohol content.
• The different grades are very expensive.

(b) Sodium Lauryl Sarcosinate: It is one of the most preferred detergents for oral products.

**Advantages:**

• It shows anti-enzymatic activity besides acting as a surface active agent.
• It is easily soluble in aqueous solvents and hence most preferred for the formulation of oral products.
• It is consistently stable with a neutral pH range.

**Disadvantage:** It may alter the taste of the final formulation when used in high concentrations.

3. Humectants: Humectants are used in order to prevent the rapid drying of dentifrices. They prevent excessive moisture loss from the product. They may additionally impart plasticity to the final product. The concentration of the humectant used in the formulation may vary from 20% to 40%.

The most commonly used humectants in the formulation of dentifrices are as follows:

(a) Sorbitol 70: It consists of 70% w/v concentration of the sorbitol solution. It comprises the largest pan the humectant phase.

**Advantages:**

• It has high viscosity and can produce firm toothpastes with good plasticity.
• It imparts cool sensation in the mouth and may also enhance the sweetening property.
• It possesses good compatibility with other ingredients; it is less expensive than glycerin.
(b) **Glycerine:** It can be used at concentrations ranging between 5 to 10%.

**Advantages:**

- It provides a good gloss and good shine to the product.
- It is safe, stable and compatible with other ingredients.
- It is easily available both from natural and synthetic sources.

**Disadvantages:**

- It is very expensive.
- It provides a warm sensation in the mouth.

(c) **Propylene Glycol:** It is less commonly used and has been replaced by sorbitol.

**Advantage:** It has good solvent properly and can also be used as a co-solvent.

**Disadvantage:** It has very low viscosity and may also impart a bitter taste to the product.

4. **Ceiling/Binding Agents:** The binding agents are used in order to hold the solid and the liquid components together to form a smooth paste and maintain its property, particularly during storage. They prevent bleeding from the paste and also add up to the body and viscosity of the final formulation.

The commonly used binding agents are cellulose derivatives such as Carboxy Methyl Cellulose (CMC), Sodium Carboxy Methyl Cellulose (SCMC), Hydroxyethyl cellulose, Cellulose ethers etc.

(a) **Sodium CMC:** It is a commonly used cellulose derivative and used in concentrations between 0.9 to 2.0%. It is sensitive to pH value outside 5.5 to 9.5. The properties with its advantages and disadvantages are as follows:

**Advantages:**

- It provides stability to the gels.
- It resists change in the efficiency of the formulation even in the presence of divalent calcium ions and other electrolytes.

**Disadvantage:** It may react with cationic substitutes of antibacterial agents due to its anionic nature. Hence it cannot be used in such formulations.

(b) **Ethers of Cellulose:** Methyl cellulose and hydroxyethylcellulose are the most commonly used cellulose ethers.

**Advantages:**

- They are stable over a wide range of pH changes.
- They are not affected by the metallic ions.
- They can be used in the toothpastes containing cationic antibacterials.
• The properties can be adjusted as required by varying the degree of substitution of the components.

Disadvantages:

• The toothpastes made with cellulose ethers are more viscous at and stiff and disperse slower than those made with SCMC.
• They cannot be used with glycerine as they are incompatible with it.

The other naturally available gelling agents may be Gum karaya, Gum tragacanth, Iris moss (Chondrus), Gum Arabica etc.

(c) Water: Water is used in the deionized form in the formulation of toothpastes. It can be used either as a solvent for the soluble ingredient of the formulation or as a supporting media for the binding agents. Binding agents swell after imbibing water. It is used in concentrations of more than 10% in the formulation of clear gels.

5. Sweetening Agents: These are added in order to improve the sweetening properties and cover the bitter taste of the other ingredients like surfactants, binders etc. They help in promoting the acceptance of the product when administered orally.

The most commonly used sweetening agents are Saccharin sodium, Chloroform, Aspartame, Cyclamates and Potassium acesulfame.

(a) Saccharin Sodium: It is the most widely used sweetening agent. It is used at concentrations of about 0.05 0.3 1 %. The concentration may vary depending upon the amount of humectant (glycerine) used.

Advantages:

• It is of low cost.
• It is widely distributed and easily available.
• It is compatible with all other ingredients.
• It provides good sweetening property.

(b) Chloroform:

Advantages:

• It masks the taste of precipitated chalk and prevents dry feeling in the mouth.
• It provides a fresh and sharp sweetness.
• It also has antibacterial property besides the sweetening property.

Disadvantages:

• It is expensive.
• It is incompatible with certain ingredients.
6. **Flavouring Agents:** Flavouring agents may comprise the most proprietary and most crucial part of the formulation essential to meet the consumer preferences. They are generally a mixture of edible volatile oils consisting of spearmint and peppermint oil as major components. The other components included may be thymol, anethol, eucalyptol, aniseed oil, oil of winter green etc. Flavouring agents are used in the concentration range of about 0.5 to 1.5% and constitute the most costly part of the formula; they may interact with other components of the formulation which may result in incompatible.

7. **Colouring Agents:** They are used in concentration of less than 0.01% as permitted by the EEC Cosmetics Directive. They can be used generally in combination with a portion of a white creamy base. They are mainly in order to influence consumer preferences and increase the purchase intent.

8. **Whitening Agents:** Whitening agents such as Titanium dioxide (TiO$_2$) shall be preferentially added in order to provide additional whiteness and brilliance to the paste.

9. **Preservatives:** Preservatives are used in the formulation in order to maintain the properties of the product throughout the storage period and to improve the shelf-life of the product. Generally, a mixture of 5% methyl paraben and 0.02% propyl paraben is the most effective and commonly used combination preservatives. Sodium benzoate is not preferred due to its incompatibility with some of the therapeutic agents.

10. **Therapeutic Agents:** Therapeutic agents are included in toothpastes in order to provide additional beneficial effects besides normal cleansing properties.

**Examples:**

(a) **Anticaries Agents:**
Example: Fluoride derivatives like NaF, Na$_2$FPO$_3$, etc,
(b) **Antiplaque Agents:**
Example: Chlorohexidine, Triclosan etc.,
(c) **Antitartar Agents that prevent the Colouring of Teeth:**
Example: Zn salts, Pyrophosphate ions, Tetra sodium pyrophosphate, Disodium dihydrophosphate.
(d) **Sensitive Dentine Agents:**
Example: Strontium chloride, Strontium acetate, Formaldehyde etc.,
(e) **Optical Brightness:**
Example: Substituted coumarins in long chain alkylamines.
(f) **Bleaching Agents:**
Example: H$_2$O$_2$, Sodium peroxide.
(g) **pH Regulators:**
Example: Zirconium silicate.
Toothpaste formula:

<table>
<thead>
<tr>
<th>Formula-1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calcium carbonate (adhesive agent)</td>
<td>28 g</td>
</tr>
<tr>
<td>sodium lauryl sulphate (surfactant)</td>
<td>0.5 g</td>
</tr>
<tr>
<td>Glycerin (humectants)</td>
<td>11 g</td>
</tr>
<tr>
<td>Gum tragacanth (binding agent)</td>
<td>0.75 g</td>
</tr>
<tr>
<td>Water (liquid phase)</td>
<td>9.7 g</td>
</tr>
<tr>
<td>Saccharin sodium (sweetening agent)</td>
<td>0.05 g</td>
</tr>
<tr>
<td>Flavor (flavoring agent)</td>
<td>q. s</td>
</tr>
<tr>
<td>Preservative (for storage)</td>
<td>q. s</td>
</tr>
</tbody>
</table>

7.2 Preparation of toothpaste:

The preparation of toothpastes may be carried out by using two methods which are as follows:

- Dry gum method.
- Wet gum method.

1. Dry Gum Method:

   - In this method, all the solid components of the formulation like abrasive agent, binding agent etc., except the surfactants are mixed together in a dry mixer. The mixer may be an agitation mixer which consists of slow rotating blades.
   - The liquid components such as the humectants and water are gradually added to the dry mix.
   - The mixing process is carried out till a smooth paste is formed.
   - The remaining ingredients like the surfactants and the flavouring agents are added to the homogenous paste under vacuum.

2. Wet Gum Method:

   - In this method, all the liquid components are mixed together to form a liquid phase.
   - The binding agent is then mixed with the liquid phase with uniform stirring in order form mucilage.
   - The solid ingredients excluding the surfactants are then gradually added to the mucilage with uniform mixing in an agitation mixer, in order to form a homogenous paste.
   - The remaining ingredients i.e., the surfactants, the flavoring agents, coloring agents are added under vacuum to the homogenous paste.

Based on the principle involved in the above methods, some acceptable techniques have been proposed for the manufacture of toothpaste which is as follow:
1. **Cold compression technique:** The preparation of toothpaste using this technique can be carried out as follows.

- Initially, the humectants such as sorbitol (70% w/v) or glycerine are taken in the bowl of the mixer.
- The binding agent is then sprinkled over the humectant under agitation for uniform dispersion.
- The liquid components such as water, sweetener and the preservatives are mixed to form a separate liquid phase and any therapeutic additives if necessary are also added to the liquid phase.
- This liquid phase is then added to the humectant-binder mixture in the bowl and mixing is carried out for 5 minutes in order to remove the air from the thick gelatinous liquid phase.
- The vacuum is stopped and the abrasive agents are added with constant mixing until they are completely dissolved.
- The vacuum is reapplied and mixing is continued for at least 30 minutes.
- The surfactants and the flavouring agents are dispersed separately in 5% humectant.
- This mixture is added to the vacuum at the end and 5 minutes of additional mixing is carried out.
- Finally, it leads to the formation of an air free smooth paste.

2. **Multiple Liquid Phase technique:** This method is suitable for formulations that make use of carboxy methyl cellulose (CMC) and magnesium aluminium silicate hinder combination. The preparation can be carried out as follows:

- Initially, hot water is taken in a mixer bowl and magnesium aluminium silicate is added to it.
- The humectants, the flavouring agent, the binding agent and the preservatives are mixed separately to form a separate liquid phase.
- This solution is then added to the mixer and the final volume is made using the humectants.
- Vacuum is introduced into bowl in order to remove the air from the liquid mixture.
- The vacuum is removed and the abrasive agents are added and the vacuum is again introduced in the mixed for 30 minutes.
- Finally, the surfactants are added with constant inking for 5 minutes. The method is also suitable for the preparation of clear-gel dentifrices.

3. **Hot Liquid Phase Technique:** The method preparation using this technique is as follows:

- In this method, the abrasive agent, binding agent and preservatives are mixed separately in a dry mixer.
- The humectant, sweeteners and water are mixed separately and this liquid phase is heated.
- The hot solution is then slowly added to dry powder with constant mixing.
- The resultant mass is then mixed under vacuum for 30 minutes.
Finally, the solutions of the flavouring agent and the surfactant are added and vacuum, mixing is carried out for 5 more minutes.

A clear and homogeneous paste is formed by this method.

7.3 Evaluation of toothpaste

The quality control studies and evaluation tests are necessary in order to check the purity, consistency and efficiency of the product. The specific evaluation tests for dentifrices are as follows:

1. Tests for Abrasive Character: The cleansing action of dentifrices mainly depends on their abrasive property. The abrasion should not lead to any damage to the enamel and hence the test for checking the abrasive property has been done on the extracted teeth. The teeth are brushed by mechanical means with paste or powder and the effect of dentifrices on the teeth is studied by comparing the results before and after brushing.

2. Determination of Particle Size: Particle size determination is important as the cleansing nature and abrasive property of the dentifrice mainly depends on the particle size. The particle size can be determined by using microscopical techniques or by involving the method of sieving.

3. Test for Cleansing Property: This test is done in order to determine the cleaning ability of the dentifrice. The tooth cleansers such as powders and pastes are brushed onto a polyester film and the change in reflectance character of the lacquer coating is measured. The in-vivo method involves brushing of the teeth with dentifrices for 2 weeks and determination of the condition of the teeth before and after brushing and comparing them by means of photographs.

4. Determination of Consistency of the Product: This test is done in order to determine the consistency of the product for the maintenance of its flow property all throughout its storage period. The consistency of the product mainly depends on the ‘theological properties such as particle size, viscosity etc.

5. Determination of pH of the Product: A 10% solution of the paste in water is made and the pH of the dispersion is measured using a pH meter. The pH should be in the range of 6.8 to 7.4 in order to maintain the consistency of the product.

6. Determination of Foaming Character: This test for the foaming character is applicable only to foaming tooth powders and pastes. In this test, specific amount of the product is mixed with a known amount of water. The solution is then shaken sometimes in order to produce foam. The foam produced is then collected and studies on its nature, washability and stability are carried out.

7. Determination of the Volatile Matter and the Moisture Content: This test is done in order to determine the amount of volatile matter and moisture content in the product. In this method, a specified amount of the product is taken and is kept for drying till a constant weight is obtained. The weight of the product before and after drying is measured and the
loss in weight is calculated which determines the percentage of moisture content and volatile matter.

8. Determination of the Test for the Special ingredient: The use of therapeutic ingredients may lead to certain incompatibilities and hence specific tests are done in order to determine the effect of the specific ingredients such as antiseptics, enzymes etc.

9. Limit Test for Heavy Metals: The test is done in order to check the presence of any heavy metals such as arsenic and lead which may lead to toxicity. The occurrence of these metals can be avoided by carrying out the limit tests for heavy metal, for raw materials, which may reduce usage of these materials.
8. Hair Dyes

Introduction:

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. A variety of hair colours are observed due to the following parameters:

- The combination of Pheomelanins and eumelanins.
- The quantity of the pigment present.
- The size of the granules of the pigments.
- The distribution of granules of the pigments.

Definition: Hair colourants are the cosmetic preparations which are used by men and women either to change the natural hair colour or to mask grey hair. The properties of typical hair colourants are:

- The formulation of the hair colourant should be stable.
- They should colour the hair evenly.
- They should not lead to loss of the natural shine of hair.
- The shaft of the hair must not be damaged.
- The natural moisture of the hair must not be lost.
- Must possess properties like non-irritant and non-sensitizing.
- Must be non-toxic in nature. Must impart stable color to the hair.
- The colored hair must be unaffected by air, water, sunlight, sweat, friction, shampoos, lotions, gels, oils etc.

8.1 Classification of hair colourants

The major classification is listed as follows:

1. Temporary hair colourants.
2. Semi-permanent hair colourants/Direct dye
3. Oxidative dyeing systems: It includes:
   (a) Semi-permanent hair colourants.
   (b) Permanent hair colourants.
4. Gradual hair colourants.
5. Natural dyes.
1. **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 into the cuticle and cannot enter into the cortex of the hair. They are rarely called as water rinses.

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.

(a) **Powder Formulations:** They are mostly used in theoretical make up and masquerades. The powder consists of dye stuff and acid like citric acid or tartaric acid. They are available in sachets.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Certified color</td>
<td>5 g</td>
</tr>
<tr>
<td>Tartaric acid (buffer)</td>
<td>95 g</td>
</tr>
</tbody>
</table>

Application Technique: The powder is dissolved in 250 ml of water and this solution is applied on wet hair after shampooing.

(b) **Crayon Formulations:** These temporary hair colorants are applied between the applications of permanent hair colorants. They color the new growing hair. They are available in many shades of colors. The composition of crayon is soap, waxes, dyes or pigments.

<table>
<thead>
<tr>
<th>Formula-1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stearic acid (anionic surfactant)</td>
<td>15 g</td>
</tr>
<tr>
<td>Triethanolamine (surfactant)</td>
<td>7 g</td>
</tr>
<tr>
<td>Beeswax (wax)</td>
<td>50 g</td>
</tr>
<tr>
<td>Carnauba wax (wax)</td>
<td>13 g</td>
</tr>
<tr>
<td>Ozokerite (wax)</td>
<td>7 g</td>
</tr>
<tr>
<td>Glyceryl mono stearate (surfactant)</td>
<td>6 g</td>
</tr>
<tr>
<td>Tragacanth (gum)</td>
<td>2 g</td>
</tr>
<tr>
<td>Color</td>
<td>q. s</td>
</tr>
</tbody>
</table>

**Method:**

- Triethanolamine, glyceryl monostearate and tragacanth are heated to 70°C.
- Stearic acid is incorporated in the above mixture and the mixture is heated to 75°C.
- Beeswax and carnauba wax are melted separately at 70–80°C.
- The molten waxes are added to the above mixture and stirred well.
- Color is added and the mixture is stirred well.
- This mixture is then poured into the moulds.
Text book of Cosmetic Formulations

<table>
<thead>
<tr>
<th>Formula-2</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium stearate (thickener)</td>
<td>18 g</td>
</tr>
<tr>
<td>Gum Arabic (gum)</td>
<td>25 g</td>
</tr>
<tr>
<td>Glycerin (surfactant)</td>
<td>15 g</td>
</tr>
<tr>
<td>Color</td>
<td>17 g</td>
</tr>
<tr>
<td>Water (solvent)</td>
<td>25 g</td>
</tr>
</tbody>
</table>

**Method:**
- A mixture of water and glycerin is prepared and divided into two parts.
- Gum Arabic is added to one portion.
- Sodium stearate is added to the other portion and it is dissolved by warming.
- Both the portions are mixed and colour is added.
- This mixture is milled to form a paste.
- The paste is introduced into moulds and allowed to dry with the help of heat.

Application Technique: It may be applied in one of the two ways.

1. The crayons are rubbed over the hair, (or)
2. It is applied using a brush.

(c) **Colour Shampoos:** They develop a temporary tinge of colour. The base used in the preparation consists sulphonated oils, anionic or nonionic surfactants. They are available in only few colour shades.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonium lauryl alcohol sulphate (surfactant)</td>
<td>30 g</td>
</tr>
<tr>
<td>Coco diethanolamide (pearlescent stabilizer)</td>
<td>2 g</td>
</tr>
<tr>
<td>Water (solvent)</td>
<td>To make up to 100 g</td>
</tr>
</tbody>
</table>

**Water Rineses:**

1. The water rinses are acidic in nature, thus
   - (a) Prevents the degradation of hair by alkali.
   - (b) Gives pastel shades to bleached hair.
   - (c) Auburn (reddish brown), blue, blonde, pink colours may be obtained.

2. The water used in water rinses must be deionized or distilled water, otherwise. The colours of the colourants get changed by the metal ions present in water.

3. EDTA, sequestering agent is included in water rinses.
4. A compatibility is observed between dye and acid which is responsible for imparting particular colour. That is why appropriate acid is used with a particular dye.

5. Solutions of basic dyes like Methylene blue, gentian violet and rhodamine gives pastel shades.

6. Bleaching mixture is added to solution of dye to minimize deep red and yellow colours but to obtain white or platinum blonde colours.

7. The dye stuff when added to a detergent base shampoo, exerts similar action as that of water rinses. It is prepared in the following manner.

(a) Dye is mixed with water to form a solution.

(b) The above solution is added to shampoo detergent base like triethanolamine lauryl sulfate.

(c) The pH of the above mixture is adjusted to 5.

Formula for water rinses or rinse solution is given below:

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid dyestuff (color)</td>
<td>6 g</td>
</tr>
<tr>
<td>Alcohol (antiseptic)</td>
<td>10 g</td>
</tr>
<tr>
<td>30% acetic acid (buffer)</td>
<td>10 g</td>
</tr>
<tr>
<td>Water (solvent)</td>
<td>74 g</td>
</tr>
</tbody>
</table>

2. Semi-permanent Hair Colourants / Direct Dyes: These colourants have a long lasting, colour retaining ability when compared to colour shampoos. The colour produced is stronger as well. Dark colours are obtained with the colourants though they do not contain H_2O_2. This offers an advantage that the melanin of the hair doesn't get bleached but is only masked with the colourant. The colour obtained on the grey hair is different than the black (pigmented) hair because of which the hairs are highlighted. The colourants are easily applied. This colour is not lost with one wash, but is gradually lost in 5 - 8 washes with shampoo. Fragrance may be added in the composition of the colourant.

**Ingredient:** The semi-permanent hair colourants are composed of the following constituents.

(a) Dye
(b) Water
(c) Organic solvent like alcohol, derivatives of glycol.
(d) Fatty acid, fatty acid amide.
(e) Thickener.
(f) Surfactant
(g) Perfume

(h) Aliphatic primary amines which work as co-solvent and buffer.

Example: 2 - amino, 2-methylpropanol.

(a) Dyes: The action of the dye or dyes is observed on hair or white wool before proceeding for the colour preparation. The following factors are of great concern dining the use of the dyes.

- Aqueous solution of the dye.
- The pH effect on the dye.
- The composition of the base added.
- The effect of solvents added.

The dyes which impart different shades belong to the following categories:

(i) O-nitro anilines. (Gives yellow and orange shades)
(ii) Aminonitrophenols and their ethers (gives yellow and orange shades)
(iii) Azo dyes (Gives yellow and orange shades)
(iv) Nitrodiphenylamines (Gives ‘orange to red shades).
(v) Nitrophenylenediamines (Gives colour in the range red to violet).
(vi) Anthraquinone (Gives violet to blue shades).

The semi-permanent hair colourants diffuse into and out of the hair which lead to off-shade fading. Therefore, colourants are selected which have a wide range of molecular sizes. This help in,

- Even colouring of the hair.
- The properties of the dye like permeability and substantivity for porous tips of hair and undamaged root ends are compensated.

Demerits of Semi-permanent Hair Colourants: The hair ends get damaged which is referred as warm wearing. Large sized amino-containing molecules like Disperse Blue 1 and Disperse Violet 1 are used to prevent warm wearing of the ends. These molecules are easily washed off.

<table>
<thead>
<tr>
<th>Formula-1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quaternary ammonium compound (color)</td>
<td>10-12 g</td>
</tr>
<tr>
<td>Anionic surfactant (surfactant )</td>
<td>8-10 g</td>
</tr>
<tr>
<td>Acid (buffer)</td>
<td>6-8 g</td>
</tr>
<tr>
<td>Alkanolamide (surfactant )</td>
<td>4-6 g</td>
</tr>
</tbody>
</table>
Dye stuff (color) | 1-2 g  
---|---
Water (solvent) | To make 100 g

Method:

- A mixture of alkanolamide and anionic surfactant is prepared.
- The dye is added to the above mixture* and is dissolved.
- The acid and quaternary ammonium compounds are dissolved in water.
- This aqueous solution is added to the solution of dye with stirring.
- Now the viscosity of the dye is adjusted by adding hydrophilic colloids like methylcellulose, natural gum etc.
- The viscosity of the colourant is increased by the addition of non-ionic thickener in its composition .The addition of amphoteric surfactant in the colourant accompanied by basic dyes.

<table>
<thead>
<tr>
<th>Formula-2</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Amphoteric surfactant (surfactant )</td>
<td>10 g</td>
</tr>
<tr>
<td>Lauric isopropanolamide (surfactant)</td>
<td>1 g</td>
</tr>
<tr>
<td>Non- ionic surfactant (surfactant )</td>
<td>5 g</td>
</tr>
<tr>
<td>Oleyl alcohol</td>
<td>1 g</td>
</tr>
<tr>
<td>Non- ionic thickener(thickener)</td>
<td>2 g</td>
</tr>
<tr>
<td>Dye (color)</td>
<td>2 g</td>
</tr>
<tr>
<td>Perfume</td>
<td>q. s</td>
</tr>
<tr>
<td>Water (solvent)</td>
<td>To make 100 g</td>
</tr>
</tbody>
</table>

3. Oxidative Dyeing Systems: These dyes are also called as ‘para dyes’. At the time of application, these dyes are colourless but turn to a particular colour after undergoing chemical reactions on the hair. The chemical reactions include the following reactions in alkaline pH, which are oxidation and coupling and condensation.

**Ingredients:** The ingredients of these dyes which render the above reactions are bases, couplers and oxidizing agent.

(a) **Bases:** They are primary intermediates. Chemically they are aromatic compounds.

(b) **Couplers:** They are aromatic in nature, and are referred as modifiers. They are the derivatives of benzene which show - NH2 and - OH substitutions at meta position. Oxidation of couplers with hydrogen peroxide is difficult to achieve. Example: 2, 4-diaminoanisole, Resorcinol, m-chloro resorcinol, m-phenylene-diamine.
(c) **Oxidizing Agents:** Commonly used oxidizing agent is hydrogen peroxide. Formulation of Oxidative Dyeing Systems: The following factors are of great concern during the preparation of oxidation dyes.

1. Formulation bases

2. Dye components: It includes oxidation base and coupling agent.

3. Alkali.

4. Oxidizing agents

5. Antioxidant.

1. **Formulation Bases:** They are used as vehicles for dyes (amino dyes) and modifiers. The vehicle is one which uniformly distributes the colourant mixture on the hair. Example: In amino dyes, a mixture of water (48-7945%), ethyl alcohol (20-50%) glycerine (0.5 - 2%) is used because the amino dye has low aqueous solubility.

If the preparation is an emulsion i.e., cream or lotion (rather than a solution) the distribution of the preparation on hair is more even. The formulation bases may be of the following kinds such as emulsion type, bleach-dye combination products, and powder preparations. The emulsion type preparations are of two types. They are foaming and non-foaming types.

(a) **Foaming-type Creams:** They are emulsified using surfactants like monoethanolamine lauryl sulfate and ethylene glycol monostearate.

<table>
<thead>
<tr>
<th>Formula</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monoethanolamine lauryl sulphate (surfactant)</td>
<td>10 g</td>
</tr>
<tr>
<td>Ethylene glycol monostearate (surfactant)</td>
<td>1 g</td>
</tr>
<tr>
<td>Preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Water (solvent)</td>
<td>To make 100 g</td>
</tr>
</tbody>
</table>

(b) **Non-Foaming-type Creams:** They are emulsified by using mineral oil, cetyl alcohol and non-ionic emulsifier.

<table>
<thead>
<tr>
<th>Formula-1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral oil (emulsifying agent +emollient)</td>
<td>1.5 g</td>
</tr>
<tr>
<td>Cetyl alcohol (emulsifying agent +emollient)</td>
<td>5 g</td>
</tr>
<tr>
<td>Non-ionic emulsifier (emulsifying agent)</td>
<td>3-5 g</td>
</tr>
<tr>
<td>Preservative</td>
<td>q. s</td>
</tr>
<tr>
<td>Water (solvent)</td>
<td>To make 100 g</td>
</tr>
</tbody>
</table>

Other additives like hydrous lanolin, lecithin, sequestering agent may he added to improve the formulation as a whole.
Bleach-dye Combination Products: They are used to bleach as well as colour the hair. Increased levels of ammonium hydroxide are used along with proportionate amounts of hydrogen peroxide.

Powder Preparation: It contains oxidizing agent such as sodium peroxide and alkali ammonium hydroxide. This powder preparation is made into a paste using water and is then applied.

2. Dye Components:

(a) Oxidation bases: By using varying concentrations of p-phenylene diamine or p-toluene diamine, a number of shades can be achieved.

<table>
<thead>
<tr>
<th>Percent of oxidation base</th>
<th>Shade obtained</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.3</td>
<td>Light brown</td>
</tr>
<tr>
<td>0.45</td>
<td>Medium brown</td>
</tr>
<tr>
<td>0.5</td>
<td>Brown</td>
</tr>
<tr>
<td>0.9</td>
<td>black</td>
</tr>
</tbody>
</table>

(b) Coupling Agents: Instead of coupling agents, direct colouring agent can also be used, coupling agents modify the shade and stabilize it. The time required to develop color with different modifiers.

3. Alkali: The oxidation dyes work best in alkaline medium. Therefore, alkali is incorporated in their composition. The best alkali is ammonium hydroxide. It leaves no evidence of its presence on the hair. It is used in a concentration of 1 - 2% in the final preparation. Because of its odour, it is completely or partially replaced with ammonium carbonate, monoethanolamine, guanidine or arginine derivatives, diethanolamine, triethanolamine, alkanolamide etc.,
4. **Oxidizing Agent:** On exposure to air, dyes such as amino dyes turn black. However, oxidizing agent is added in its composition to achieve the desired colour. Examples are ferric chloride, potassium permanganate, potassium dichromate, hydrogen peroxide etc. Hydrogen peroxide is popularly used. It is used in a concentration of 5 - 6% solution which generates 20 volumes of oxygen. \( \text{H}_2\text{O}_2 \) is responsible to develop colour on the hair. It is sold in a package containing two containers. One container contains dye and the other contains the developer.

5. **Antioxidant:** During the manufacturing of dyes, especially amino dyes, an atmosphere of nitrogen is maintained to prevent the darkening of the dye. Since dyes (amino dye) are darkened on exposure to air. Instead of maintaining nitrogen atmosphere, chemical antioxidant like sodium sulfite is included in the preparation.

The total amount of base and the coupling agent used gives the amount of sodium sulfite to be used in the preparation. If darker shades are desired, then the amount of sodium sulfite is increased. The oxidative dyeing system consists of the semi-permanent hair colourants and the permanent hair colourants.

(a) **Semi-permanent Hair Colourants:** The semi-permanent and permanent hair colourants are the two classes of oxidation dyes or oxidative dyeing systems. They differ in the extent of giving light colour shades to the hair. The common constituents of both the classes are alkalizing agents, oxidants, dyes, solvents and surfactants.

(I). **Alkalizing Agents:** The alkalizing agents are added.

- To increase the pH of the formulation to an optimal level.
- To generate active oxidizers from hydrogen peroxide.
- To swell the hair fibres for absorption of dye.

Examples of alkalizing agents include ammonia, Monoethanolamine.

The rate of bleaching of hair is based on the following factors and the rate of bleaching is directly proportional to the following factors.

- pH.
- Concentration of hydrogen peroxide.
- Amine added.

The rate of bleaching of different amines and ammonia is shown.

Tertiary amine < secondary amine < primary amine < ammonia.

It means ammonia is a strong alkalizing agent, which is used-widely.

Instead of ammonia, high level of monoethanolamine is used alone or monoethanolamine and ammonia are used in combination. The semi permanent products employ monoethanolamine alone, where a little bleaching is required, whereas hindered primary, secondary or tertiary amines are employed, when no bleaching is required.
(II). Oxidant: Oxidant is added in the composition of the colourants to generate active species (like p-phenylene diamine, benzoquinone monoamine) for coupling. Oxidants are used to bleach melanin present in the hair. Light colour shades are obtained when the grey and pigmented hair are coloured evenly by using semi permanent colourants.

(III). Dye: Dyes are used to impart the desired colour shade to the hair.

(IV). Solvents: The constituents of the colourants which are not soluble in water, are dissolved by using solvents, so that a homogenous system is obtained.

(V). Surfactant: It reduces the surface tension between the different ingredients, to make a homogeneous preparation.

(b) Permanent Hair Colourants: The colour produced by these colourants last longer when compared to semi-permanent colourants. Actually it is the precursor of dye which when applied undergoes chemical changes to form the colour rather than the dye itself.

They are available in light colour shades to dark colour shades. It is the growth of hair more than fading of colour, which arises the need to re-dye. This results in stripped appearance of the hair.

The oxidation dyes may cause allergic reactions in some individuals. According to the rules of drugs and cosmetics, the preparation must contain the caution in English, local and other regional languages on both the inner and outer labels.

Caution: “The preparation may cause irritation of skin in some individuals; therefore, it is advised to go for patch testing before using it on hair. The eyelashes and eyebrows are not dyed because it may cause blindness.”

Example: Metallic hair colourant / colour restorers.

The instructions to proceed with the patch testing are written in English, local and other regional languages.

The individuals are advised to go for testing before using it on hair. The test is carried out either behind the ear or on inner surface of forearm. The area is cleansed with soap water or alcohol. The dye is prepared according to the instruction given on its leaflet and applied on tile cleaned area. It is kept under observation for 24 hrs. After that, it is washed with water. The area is observed for any irritation or inflammation, there are any signs of them, then the individual is hypersensitive to the dye, and if there are no signs, then the individual is not hypersensitive to the dye. The patch test is required before each application oldie dye.

They are compounds of metals like cadmium, copper, cobalt, lead and silver. These metals are present in their salt forms. They are also called as progressive hair colourants since they colour the grey hair gradually. The colour is achieved by the deposition of the metallic salt on the hair shaft.
<table>
<thead>
<tr>
<th>Formula-1</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lead acetate (color)</td>
<td>5.5 g</td>
</tr>
<tr>
<td>Sodium thiosulphate (reducing agent)</td>
<td>11 g</td>
</tr>
<tr>
<td>Glycerin (humectants)</td>
<td>8.5 g</td>
</tr>
<tr>
<td>Ethyl alcohol (antiseptic)</td>
<td>10 g</td>
</tr>
<tr>
<td>Perfume</td>
<td>q.s</td>
</tr>
<tr>
<td>Water (solvent)</td>
<td>To make 100 g</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Formula-2</th>
<th>Quantity for 100 g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lead acetate (color)</td>
<td>12 g</td>
</tr>
<tr>
<td>Precipitated sulfur</td>
<td>24 g</td>
</tr>
<tr>
<td>Propylene glycol</td>
<td>12 g</td>
</tr>
<tr>
<td>Ethyl alcohol (antiseptic)</td>
<td>10 g</td>
</tr>
<tr>
<td>Perfume</td>
<td>q.s</td>
</tr>
<tr>
<td>Water (solvent)</td>
<td>To make 100 g</td>
</tr>
</tbody>
</table>

Metallic dyes also include silver dyes, they were used before the organic chemical dyes. A number of shades can be obtained by, varying the concentration of silver in the preparation. Silver dyes were left behind with the popularity of synthetic organic dyes. One of the example is pyrogallol. Skin irritations and harmful effects upon internal administration were reported which led to the discontinuation of pyrogallol.

4. **Gradual colourant:** it includes heavy metals in its composition. The hair is gradually coloured with several application of the colourant. The heavy metals used are lead or bismuth in their salt forms. The salts of the heavy metals are mad into solutions and are used in the preparations. The preparation is applied many times because the colour develops gradually.

**Demerit:** since, the preparation includes heavy metals, it offer negative effects on the health. Therefore the use of these colourants is declined.

5. **Natural dyes:** Since, antiquity, plant materials are looked upon as beneficial sources for various ailments and other purposes. The leaves are used as colourants:

(a) **Henna:** 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. It 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-14-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.
(a) Camomile: The flowers of camomile are used to obtain the colour. The flowers which contain the active principle are powdered. Its paste is made by mixing the powder with hot water and applied on the hair. A warm towel is wrapped over the head to enhance the colouring effect. The colour achieved is due to the navy blue volatile oil obtained in the process. Either 2 parts of kaolin or 1 1 part of fuller’s earth is added to camomile powder to form a cohesive composition. Henna is mixed with camomile in varying proportions, to modify the colours.

8.2 Evaluation of hair colourant:

The following tests are carried out to evaluate hair colourants.

1. The sensitization test

2. The toxic effect test

1. The Sensitization Test: The test is carried out on animal skin. The colourants applied on the skin and is kept under observation for 24 hrs. If no reaction occurs, then the colourant is said to be non-sensitizing or non-irritant. Histopathological study is carried out as per requirements.

2. The Toxic Effect Test: Toxic effects are studied in animals to know about the long term effects of the preparations.
9. References