

FORMULATION & EVALUATION OF SHAMPOO

Introduction

Definition: A shampoo is a preparation of a surfactant (i.e. surface active material) in a suitable form – liquid, solid or powder – which when used under the specified conditions will remove surface grease, dirt, and skin debris from the hair shaft and scalp without adversely affecting the user.

Introduction

Requirements of a Shampoo:

- 1.It should effectively and completely remove dust or soil, excessive sebum or other fatty substances and loose corneal cells from the hair.
- 2.It should produce a good amount of foam to satisfy the psychological requirements of the user.
- 3.It should be easily removed on rinsing with water.
- 4.It should leave the hair non-dry, soft, lustrous with good manageability and minimum fly away.
- 5.It should impart a pleasant fragrance to the hair.
- 6.It should not cause any side-effects / irritation to skin or eye.
- 7.It should not make the hand rough and chapped.

Types of Shampoo

Shampoos are of the following types:

- Powder Shampoo
- Liquid Shampoo
- Lotion Shampoo
- Cream Shampoo
- Jelly Shampoo
- Aerosol Shampoo
- Specialized Shampoo
 - Conditioning Shampoo
 - Anti- dandruff Shampoo
 - Baby Shampoo

PRODUCT INGREDIENTS

- **Surfactants are the main component of shampoo.** Mainly anionic surfactants are used.
- The raw materials used in the manufacture of shampoos are:
 1. Principal surfactants: Provide detergency and foam.
 2. Secondary surfactants: Improve detergency, foam and hair condition.
 3. Other additives.

CLEANSING ACTION OF SHAMPOO

A surfactant consists of two part- one hydrophilic (water loving) while the other is hydrophobic in nature.

Surfactants

- **Anionic surfactants** are mostly used (good foaming properties). The hydrophilic portion carries a negative charge which results in superior foaming, cleaning and end result.
- **Non-ionic surfactants** have good cleansing properties but do not have sufficient foaming power.
- **Cationic surfactants** are toxic and are hence not used. However, they may be used in low concentration in hair conditioners.
- **Ampholytics**, being expensive, are generally not used. However, they are mainly used as secondary surfactants and good hair conditioners.

ANIONIC SURFACTANTS

CLASS	EXAMPLE	COMMENT
Alkyl benzene sulfonates	Sodium dodecyl benzene sulfonate	Tend to yield an “airy” or low density foam and often are drying to the hair
Primary alkyl sulfates	Lauric acid, stearic acid and their salts	Good lathering effect in hard water, free from rancidity, easy to wash.
Secondary alcohol sulfates	Sodium sec-lauryl sulfate	Low cost, dispersing and emulsifying action, dissapointing as detergets and shampoo components
Alkyl benzene polyoxyethylene sulfonates	Triton X200	Stable in acid or alkaline solution, excellent emulsifier, detergent and wetting agent; extremely stable at pH of skin
Sulfated monoglycerides	Lauric monoglyceride ammonium sulfate	Stable in hard water
Alkyl ether sulfates	Derivatives of lauryl alcohol ether with PEG	Good cleansers, act as solvents for non polar additives
Sarcosines	Lauroyl and cocoyl sarcosines	Excellent foaming and conditioning action
Sulfosuccinates	Aerosol OT	Less irritating to skin and eye (baby shampoo)
Maypon	Protalbinic and lysalbinic acid derivatives (maypon 4C)	Hydrolysatation product of proteins with fatty acid chlorides in presence of alkali

NON-IONIC SURFACTANTS

CLASS	EXAMPLE	COMMENTS
Fatty acid alkanolamides (should not be used > 15%)	Lauric monoethanolamide	Improves solubility of SLS
	Stearic ethanolamide	Pearlescent thickener
	Oleic ethanolamides	Hair conditioning agents
Polyalkoxylated derivatives	Ethoxylated fatty alcohols	Stable in wide range of pH; stabilizing emulsifying and opacifying properties
	Block polymers (pluronic)	Good rinsability, can be used in high %
	Sorbitol esters (TWEENS)	Solubilizers and emulsifiers, used in baby shampoos
Amine oxides	Coconut and dodecyl dimethyl amine oxides	Foam booster and anti-static agents

AMPHOTERIC SURFACTANTS

N-alkyl aminoacids	β – aminoacid derivatives	Foaming agents
	Asparagine derivatives	Compatible with both anionic and cationic surfactants
Betains	Amido betains	High foaming properties, mild.
Alkyl imidazoline	MIRANOL™	Baby shampoos

ADDITIVES

- **Conditioning agents:** Lanolin, mineral oil, herbal extracts, egg derivatives.
- **Foam builders:** Lauroyl monoethanolamide, sarcosinates
- **Viscosity modifiers :**
 - Electrolytes – NH_4Cl , NaCl
 - Natural gums – Gum Karaya, tragacanth, alginates
 - Cellulose derivatives – Hydroxy ethyl cellulose, methyl cellulose
 - Carboxy vinyl polymers – Carbopol 934
 - Others – PVP, phosphate esters.
- **Sequestering agents :** EDTA
- **Opacifying agents :** Alkanolamides of higher fatty acids, propylene glycol, Mg, Ca and Zn salts of stearic acid, spermaceti, etc.
- **Clarifying agents :**
 - Solubilizing alcohols – ethanol, isopropanol
 - Phosphates –
 - Non-ionic solubilizers – polyethoxyated alcohols and esters.

ADDITIVES

- **Perfumes** : Herbal, fruity or floral fragrances.
- **Preservatives** : Methyl and propyl paraben, formaldehyde (*most effective*).
- **Anti-dandruff agents**: The shampoos contain small amount of these actives, which are in contact with the scalp for only a short time. In order to be effective the active ingredient must work in the oil-water environment of the scalp and must be readily substantive to the scalp for continuing activity.
Ex: Selenium sulfide, zinc pyrithone, salicylic acid.

FORMULATIONS

POWDER SHAMPOO

Henna powder	5%
Soap powder	50%
Sodium carbonate	22.5%
Potassium carbonate	7.5%
Borax	15%
Perfume	q.S

LOTION SHAMPOO

TLS	35%
Glyceryl monostearate	2%
Magnesium stearate	1%
Water	Upto 100%
Color	q.s
Perfume, preservatives	q.s

LIQUID SHAMPOO

SLS	40%
NaCl (to desired viscosity)	2-4%
Water	Upto 100%
Perfume, color, preservatives	q.s

FORMULATIONS

CREAM SHAMPOO

SLS	38%
Cetyl alcohol	7%
Water	Upto 100%
Color, perfume	q.S
Preservative	q.s

AEROSOL SHAMPOO

TLS	60%
Coconut diethanolamide	2%
Water	Upto 90%
Propellent	10%
Color, perfume, preservative	q,.s

JELLY SHAMPOOS

Alkyl dimethyl benzalkonium chloride	15%
TLS (40%)	28%
Coconut ditethanolamide	7%
HPMC	1%
Water	Upto 100%
Color, perfume, preservative	q.s

FORMULATIONS

CONDITIONING SHAMPOOS

Steryl dimethyl benzyl ammonium chloride	5.5%
Ethylene glycol monostearate	2%
Cetyl alcohol	2.5%
Water	Upto 100%
Color, perfume, preservative	q.s

BABY SHAMPOO

Magnesium lauryl sulfate (27.5%)	11%
Cocamidopropyl betaine (30%)	5%
Polysorbate 20	1%
PEG 600	3.5%
Perfume	q.S
Preservative	q.S
Citric acid	To pH 6
Color	q.S
Water (deionised); Aqua (INCI)	To 100%

FORMULATIONS

ANTI-DANDRUFF SHAMPOO

Thymol	0.05%
Menthol	0.1%
Camphor	0.1%
TLS	55%
Water	upto 100
Color, perfume, preservative	q.s

ANTI-DANDRUFF SHAMPOO

Selenium sulfide	2.5%
Bentonite	5%
SLS paste	35%
Water	upto 100
Color, perfume, preservative	q.s

FORMULATION

HERBAL SHAMPOO

Natural essential oil blend	0.5%
Cyamopsis tetragonoloba (Guar Gum)	1%
Camellia sinensis (Green Tea) extract	2%
Glycerin	1%
Hydrolysed wheat protein	2.5%
Salvia officinalis (Sage) leaf extract	1.5%
Salvia officinalis (Sage)	1.5%
Glyceryl oleate	1%
Polysorbate 20	0.5%
Potassium sorbate	5%
Aloe barbadensis (Aloe vera) extract	0.5%
Arctium minus (Burdock) root extract	0.5%
Disodium coco-glucoside sulfosuccinate	0.5%
Preservatives	q.s.
Water	Upto 100%

Method of Preparation

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

1. Initially the detergent is converted into a solution form.
2. To about half of the detergent solution, the total amount of secondary surfactant i.e., alkanolamide is added and dissolved by stirring or heating gently.
3. To the remaining half of the detergent solution, suitable amount of perfume is added and dissolved. The perfume solution is then added to the alkanolamide solution.
4. Colours and preservatives are dissolved separately in sufficient volumes of water and then added to the main solution.
5. The whole solution is mixed well by gentle stirring.
6. Final volume of the preparation is usually adjusted by the addition of clear sterile water. This gives clear liquid shampoo.
7. Preparations containing lauryl alcohol, ether sulphate require viscosity adjustment which is done by adding controlled amounts of an electrolyte solution. Usually, a solution of sodium chloride is added with constant stirring.

Packaging

- Shampoos are usually packed in plastic containers. Some medicated shampoos are also packed in glass containers.
- The containers used are either coloured or transparent. The product is usually packed in an air-tight container, which prevents the product from microbial attack and also from drying.
- The size of the container varies depending upon the quantity to be packed. When the quantity of shampoo is less i.e., 5 ml or below it is packed in sachets.
- The packed product must be labelled with brand name, ingredients, quantity, expiry date etc., and directions for use.

Evaluation of Shampoos

- ✓ Foam and foam stability
- ✓ Detergency and cleaning action
 - Effect of water hardness
 - Surface Tension and wetting
 - Surfactant content and analysis
- ✓ Rinsing
- ✓ Conditioning action
 - Softness
 - Luster
 - Lubricity
 - Body, texture and set retention
 - Irritation and toxicity
 - Dandruff control
- ✓ Microbiological assay
- ✓ Eye irritancy test

■ Product characteristics

- ✓ Fragrance
- ✓ Colour
- ✓ Consistency
- ✓ Package

Additional Notes From GPAT point of View

1. Foam and foam stability:

- The Ross-Miles foam column test is accepted. 200 ml of surfactant solution is dropped into a glass column containing 50ml of the same solution. The height of the foam generated is measured immediately and again after a specified time interval, and is considered proportional to the volume.
- Barnett and Powers developed a latherometer to measure the effect of variables such as water hardness, type of soil and quantity of soil on foam speed, volume and stability.
- Fredell and Read titrated actual standard oiled heads of hair with additive increments of shampoo until a persistent lather end point appeared.

2. Detergency and cleaning action:

- Cleansing power is evaluated by the method of Barnet and Powers
- 5gm sample of soiled human hair is placed at 35°C in 200 cc of water containing of 1 gm of shampoo.
- The flask is shaken 50 times a minute for 4 minutes. Then washed once again with sufficient amount of water, then after filter the hair dried and weighed.
- The amount of soil is removed under these condition is calculated.

3. Wetting Action:

- Canvas disk sinking test: A mount veron cotton duck # 6 canvas disc 1 inch in diameter, is floated on the surface of a solution, and the time required for it to sink is measured accurately.

4. Rinsing:

- Skilled beauticians are employed to make comparisons on the performance of several shampoos.

5. Conditioning Action:

- Conditioning action is a difficult property to assess. This is because it is basically dependent on subjective appraisal.
- No method has been published for measuring conditioning action.
- The degree of conditioning given to hair is ultimately judged by shampoo user who is making the evaluation on the basis of past experience and present expectations.

6. Microbiological assay:

- **PREPARATION OF PRE-INOCULUM** Take the loopful culture of staphylococcus aureus (ATCC6532) aseptically and transfer to sterilized and cooled 100 ml SCDM (broth).
- Mix well. Incubate the broth at 37°C for 24 hrs.
- **PREPARATION OF MEDIA** Soya bean casein digests medium, soya bean casein digest agar and nutrient agar.
- **PREPARATION OF POUR PLATES** Sterilized SCD agar (100 ml) is cooled to 40°C and mixed with 5 ml of 24 hrs old pre inoculated culture.
- This is immediately poured in plates (340 ml each) and allows to set.
- **MAKING THE WELLS ON AGAR PLATES** The wells are dig on agar plates with sterilised well digger aseptically.
- Take 100µml of each sample, add to well aseptically. Incubate the plates at 37°C for 24 hrs to 48 hrs.
- Observe the effectiveness of sample on culture growing on the agar plate and we can see the effectiveness of sample in the form of zone of inhibition around each well containing different sample.

7. Evaluation of eye irritancy:

- The test calls for dropping 0.1 ml of liquid shampoo in the conjunctiva sac of one eye of the rabbit , the other eye serving as control.
- In the case of the first three animals, the treated eye remains unwashed. Since washing the eye may or may not alleviate symptoms of injury.
- The six remaining animals are divided into two equal groups.
- In the first of these groups eyes instilled with the substances are washed with 20 ml of lukewarm water two seconds after treatment and in the second group after instillation.
- Readings are then made at 24, 48 and 72 hr and again four and seven days after treatment.
- If the lesions have not cleared up in seven days the test material is considered as severe irritant.

8. Viscosity:

- Viscosity of the liquid shampoo is determined using a Brookefield viscometer
100 mL of the shampoo is taken in a beaker and the spindle is dipped in it for about 5 min and then the reading is taken.

Thank You