

FORMULATION AND DEVELOPMENT OF SILICONE DERIVATIVE BASED MOISTURIZING LOTION: HARNESSING THE SYNERGISTIC EFFECTS OF DIMETHICONE (AND) CETEARYL DIMETHICONE CROSS POLYMER

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Abstract- Cosmetics are becoming more important in daily life as they are used regularly by increased number of people and very large quantities are consumed each year. If the purpose of cosmetics is examined, the most obvious is protection of the body from the element of nature, such as heat, sunlight and microbes. In modern society, the primary goals of using cosmetics are personal hygiene, improving attractiveness through the use of make-up, boosting self-confidence and, protecting the skin from UV rays, pollutants, and other environmental factors, preventing ageing, and generally assisting people in living more fulfilling lives. Moisturizing lotion plays a crucial role in maintaining the health and appearance of the skin. Some important role of moisturizing lotions are hydration by restoring skin's moisture and preventing TEWL, skin protection, skin elasticity and smoothness etc. Silicone derivatives in moisturizing lotions contribute to their efficacy by providing enhanced moisture retention, improving texture and skin feel, and facilitating the delivery of active ingredients, ultimately promoting optimal skin hydration and overall skin health. Although silicone derivatives are frequently found in personal care products, such as moisturizers, there are some common myths about their usage. Few typical misconceptions are: they clog pores and cause acne, they can suffocate the skin, they are toxic, it prevents other beneficial ingredients from working, it causes long term damage to the skin, and it can harm the environment. Misconceptions and myths about silicones in cosmetics can lead to confusion among consumers. Conducting research and disseminating accurate information helps educate consumers about the benefits and safety of silicones. This empowers consumers to make informed choices and dispels any unwarranted fears or concerns they may have regarding the use of silicones in cosmetics. The current research work has been undertaken to formulate and evaluate the properties of silicone in moisturizing lotion.

Keywords: Personal hygiene, Improving texture, Silicon, Moisturizers.

1. INTRODUCTION

A type of skincare item called a moisturizing lotion is made to hydrate and nourish the skin. The skin's moisture levels are usually restored, and the texture and appearance are enhanced. Humectants, emollients, and occlusive, among other ingredients, are used in the formulation of moisturizing lotions to aid in the retention of water in the skin. Glycerin and hyaluronic acid are examples of humectants that draw moisture from the air and bind it to the skin to keep it hydrated. Emollients, such as oils and butters, fill in the spaces between skin cells to soften and smooth the skin. On the skin's surface, occlusive like petrolatum create a barrier that stops moisture loss and shields the skin from the elements. ¹Applying moisturizing lotion to the face, body, or hands will help to reduce dryness, flakiness, and roughness of the skin. They are available in a variety of formulations, such as thin lotions for daily use, heavier creams for deeper hydration, and specific formulas for various skin types or issues. It's important to remember that moisturizing lotion is typically applied as part of a regular skincare routine after cleansing the skin. By keeping the skin moisturized, one can support a healthy skin barrier, maintain elasticity, and give your skin a smoother, younger-looking appearance.^{2,3} In a variety of personal care items, such as skincare, hair care, and makeup products, silicone derivatives are still frequently used in the cosmetics industry. Silicones are prized for their adaptable qualities, which provide advantages like better texture, improved spreadability, and increased product longevity on the skin and hair.⁴ Despite the benefits of silicone derivatives, some consumers and experts have expressed concern over potential drawbacks, which has

increased interest in substitute ingredients. Some typical issues include, they clog pores and cause acne, they can suffocate the skin, they are toxic, they interact with the activities of other ingredients of the formulation, cause long term damage to the skin, Silicones harm the environment etc. In fact it is largely untrue because it form breathable barrier on the skin surface that allows moisture retention without blocking the skin's natural functions, silicones are lightweight and have a molecular structure that allows them to create a smooth, protective layer on the skin surface, this barrier helps to prevent TEWL and protects the skin from external irritants without suffocating it. Silicones derivatives such as dimethicone or cyclopentasiloxane used in cosmetic products, have extensively been tested for safety and are considered non-toxic, they are widely used in skincare, hair care, and makeup products without causing harm to the skin or overall health. Silicones can actually enhance the performance of other ingredients in cosmetic products. They create a smooth base for makeup application, help active ingredients penetrate the skin more effectively, and improve the overall texture and feel of the products. There is no scientific evidence to support the claim that silicones causes long term damage to the skin. They are widely used and considered safe for cosmetic purposes and used for treating acne, eczema in products for baby care in the form of cream, useful for soothing diaper rash.^{5,6} Volatile low molecular wt. siloxane will evaporate into the atmosphere where they undergo indirect photolytic degradation, high molecular weight Polydimethylsiloxane fluids (PDMS), however high molecular weight silicones likely to enter the soil compartment and get decompose.⁷ The cosmetics industry has noticed a rising interest in formulating products with a focus on sustainability, environmental impact, and ingredient transparency in response to some of these worries.

Although silicon compounds have been known for a century, their use has increased dramatically since the late 1980s. Numerous personal care applications employ silicone polymers. There are numerous silicone polymers with useful properties in both aqueous and oil-based systems.^{8,9,10} With atomic number 14 and making up 28% of the earth's crust, silicon is the second most common element. Since it does not exist in its free form, elemental silicon is most frequently found in nature as silicates, which are minerals made of silicon, oxygen, and other elements like aluminum, magnesium, iron, calcium, or mica. Silicones are polymers of silicon, carbon, hydrogen, and oxygen; nitrogen or sulphur may also occasionally be added.¹¹

1.1 Nomenclature

Alfred Stock laid the groundwork for the current nomenclature of silicone chemistry in 1916. He referred to silanes as silicone hydride compounds, such as: A monosilane was SiH_4 , a disilane was Si_2H_6 , and so on. By including the appropriate prefixes, such as dichlorodisilane for $\text{Si}_2\text{H}_4\text{Cl}_2$, derivatives were given names. The compound was known as a siloxane when oxygen joined two silicon atoms, and polymeric forms were referred to as polydimethylsiloxanes. The International Union of Pure and Applied Chemistry (IUPAC) eventually adopted this system. As a result, the fundamental groups that constitute common silicone polymers are commonly given in an abbreviated notation by silicone chemists. This system predicts that silicone chemistry has four fundamental building blocks, as shown in Table 1.^{4,11}

Table no.1 Primary Building Blocks of Silicone

| Notation | Representation | Comment |
|----------|--|----------------------|
| M | $\begin{array}{c} \text{CH}_3 \\ \\ \text{CH}_3-\text{Si}-\text{O}- \\ \\ \text{CH}_3 \end{array}$ | Chain stopper |
| D | $\begin{array}{c} \text{CH}_3 \\ \\ -\text{O}-\text{Si}-\text{O}- \\ \\ \text{CH}_3 \end{array}$ | On-chain repeat unit |
| T | $\begin{array}{c} \text{CH}_3 \\ \\ \text{O}-\text{Si}-\text{O} \\ \\ \text{O} \end{array}$ | Branching unit |
| Q | $\begin{array}{c} \text{O} \\ \\ \text{O}-\text{Si}-\text{O} \\ \\ \text{O} \end{array}$ | Resinous unit |

The formation of the various silicone polymers used in personal care compositions depends primarily on four fundamental siloxane units, which were discussed in the previous section. These are the mono-, di-, tri-, and quadrafunctional siloxane that are produced when chlorosilanes are hydrolyzed (also known as alcoholized).

Silicones are produced commercially using a variety of different processes. Each manufacturing procedure involves a number of steps, beginning with the preparation of silicon metal and progressing through the formation of chlorosilanes, their conversion to silanols, and finally their conversion to organosiloxanes. The most widely utilized commercial process, referred to as the "direct process" or Rochow synthesis, which is the most popular commercial process.

1.2 Degradability of Silicones

Polydimethylsiloxane fluids (PDMS) are widely used in industrial and consumer products such as textile treatments, households and personal care products and antifoams for food processing or waste water treatment plants. These applications result in discharge to sewage treatment plants. Volatile low molecular wt. siloxane will evaporate into the atmosphere where they undergo indirect photolytic degradation, high molecular weight PDMS, however all likely to enter the soil compartment. High molecular wt. liquid PDMS can enter the environment via the water (waste water) from industrial or domestic application.

If the sewage system is linked to the wastewater treatment plant, nearly all of the PDMS will end up in the sewage sludge. PDMS is insoluble in water (<1ppm) and due to its high adsorption coefficient (log K_{oc} estimated to be > 4.5) it will be bind to sewage particles. It is essentially confirmed that all the PDMS added to the model activated sludge was associated with the microbial mass. This is also supported by waste water treatment plant monitoring.

PDMS was found to be highly removed during waste water treatment with PDMS, sludge concentration remaining in the effluent in most cases, below 5ug/l, the analytical detection limit. PDMS sludge can range from 290- 515mg/l and varied as a function of influent can and sludge processing method. Calculated % removal was above 94% at all the plants monitored.

The further fate of liquid PDMS retained in the waste water treatment sludge will depend on the method of disposal of the sludge.¹² There are essentially 3 methods of disposal:-

- a. Incineration
- b. Landfill and soil

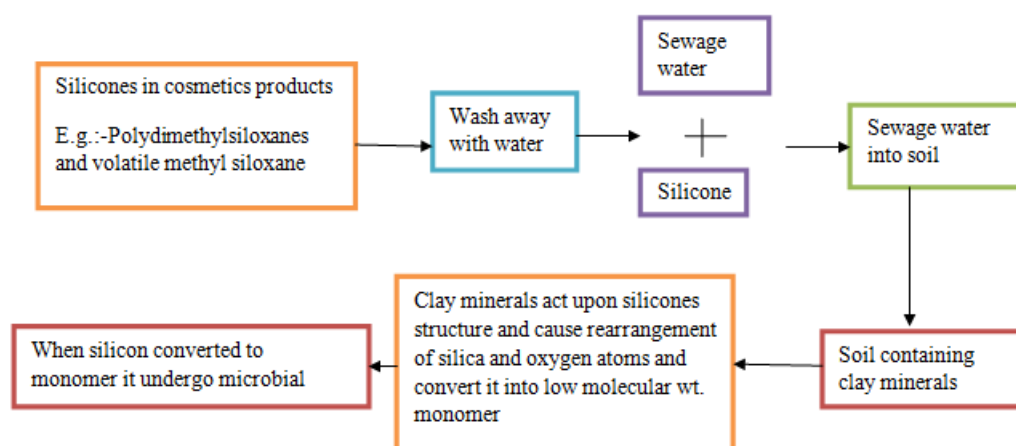


Figure no.1 Mechanism of Action of Degradability of silicone

- c. Amendment

Most of the sewage sludge at present is either incinerated or landfill the amendment of soil with activated sludge is the less widespread than in USA. Catalytic rearrangement and hydrolysis of the silicon-oxygen bonds of PDMS fluids in contact with dry soil to form low molecular weight cyclic and linear oligomers and silanols was first observed in 1979 by Bnch and Imgebrington, they found that soils were catalyzing degradation of PDMS at low soil moisture content (<5%).

Later studies, though found only silanols, this was explained by the experimental design used by Lehman et. al. in a study performed in Germany no accumulation of PDMS was found in soils which has received repeated application of sewage sludge.¹³ The degradability of silicones is clearly explained in Fig.no.1 and Fig.no.2.

1.3 Velvessil DM LC-101 PM 155993

INCI:- Dimethicone (and) Cetearyl Dimethicone Cross polymer

Description:- Velvessil DM silicone is a patented and unique multifunctional silicone co-polymer network.¹⁴ It helps deliver consumer perceivable enhanced sensory benefits in skin care and color cosmetics formulations. It spreads easily on the skin leaving an exquisite, long lasting silky feel. It is non-emulsifying silicone co-polymer network

dispersed on dimethicone and appears as a creamy, transparent gel. It contains- 17% co-polymer and exhibits shear thinning behavior, which results in cosmetic products that spreads easily during applications. Some of the physical properties of Velvisol is mentioned in table no.2

These performance properties make it an excellent candidate for anti-ageing sunscreen and moisturizer products. Velvisol DM silicone is also an effective thickener for anhydrous formulations and the oil phase of emulsion. The performance properties of Velvisol DM silicone make it an excellent material of choice for shower conditioner/in shower body lotion. In addition to providing unique sensory both during and after use, Velvisol DM silicone acts as a carrier for fragrance to enhance fragrance deposition and longevity after the wash. It can also be formulated in a wide range of hair care products such as rinse-off and leave-on conditioners. It is an excellent choice for styling products especially for heat styling applications.^{14,15} Some key features and benefits of Velvisol is given in table no.3.

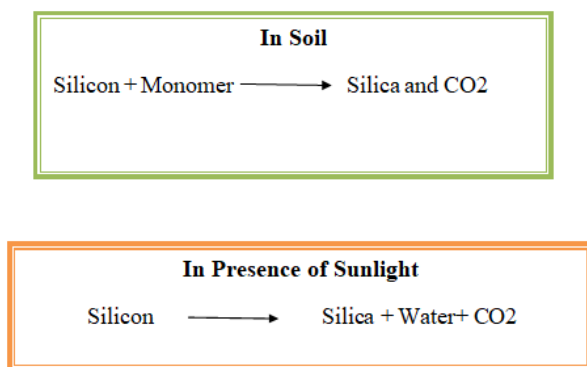


Figure no.2 Degradability of silicone in soil and in presence of sunlight

Table no.2 Physical Properties of Velvisol

| Property | Value | Unit if measure |
|-----------------|------------------------|-----------------|
| 1. Appearance | Clear/ translucent gel | - |
| 2. % Co-polymer | 16-18 | % |
| 3. Viscosity | >65,000 | cSt |
| 4. Flash point | 137 (280) | °C(°F) |
| 5. Freeze thaw | yes | n.a |

Table no.3 Key Features and Benefits

| S.no | Key features | S.no | Benefits |
|------|---|------|---|
| 1. | Long lasting silky feel | 1. | Consumer delight |
| 2. | Cold – processable | 2. | Increased manufacturing efficiency |
| 3. | Broad pH range | 3. | Ability to formulate products with low or high pH |
| 4. | Yields clear gel | 4. | Suitable for clear gel products |
| 5. | Shear thinning rheology | 5. | Excellent application properties |
| 6. | Effective thickener | 6. | Ability to formulate anhydrous products |
| 7. | No tackiness | 7. | Superior sensory feel |
| 8. | Good compatibility with personal care ingredients | 8. | Formulation flexibility. |

Potential Application

| 1. Skin care | 2. Color cosmetics | 3. Hair care | 4. Cleansing products |
|-----------------------------------|----------------------------|--|------------------------|
| a. Moisturizing cream and lotions | a. Foundation | a. Styling products:- mousse, gels, glaze, lotions | a. Shower conditioners |
| b. Anti-aging products | b. Eye shadows and blushes | b. Conditioner | b. Facial scrubs |
| c. Shaving | c. Mascaras | | c. Facial cleansers |
| | d. Make-up | | d. Shower gel |

| | | | |
|---|--|--|-------------------------|
| products d. Massage creams e. Sunscreen lotion and anhydrous sunscreen f. Skin care gels: - eye gel, after sun gel and treatment serum. | removers e. Lipstick products and lip color formulations. | | e. Liquid hand soaps |
|---|--|--|-------------------------|

Recommended Use Level

- a. Emulsion:- 4-25%
- b. Anhydrous gel:- 25-90%
- c. Cleansing products:- 1-5%
- d. Shower conditioners:- 5-40%
- e. Hair care products:- 1-5%

Many cosmetic products are flooded in the market which claims to be silicon free which has lead to the confusion among consumers. Conducting continuous research and disseminating accurate information helps educate consumers about the benefits and safety of silicones. This empowers consumers to make informed choices and dispels any unwarranted fears or concerns they may have regarding the use of silicones in cosmetics. Additionally, there is a rising demand for multifunctional cosmetic ingredients that offer not only aesthetic benefits but also provide skin care benefits. This trend is driving the development of silicone-based ingredients that offer enhanced Moisturization, UV protection, and anti-aging properties, among others. These novel silicone molecules could possess enhanced functionalities, improved biodegradability, or other desirable characteristics. Silicones, such as dimethicone and cyclopentasiloxane, are often included in moisturizing lotions for their unique texture and sensory properties. They contribute to a smooth and silky feel, allowing the lotion to spread easily on the skin. This helps in effortless application and even distribution of the product.^{16,17}

Considering all these advantages of silicone it was decided to formulate moisturizing lotion for dry skin containing different concentrations of dimethicone (and) cetareth dimethicone cross polymer incorporated into it. These formulations were evaluated by performing testing according to BIS: 6608 (2004): Skin Creams.

2. MATERIALS AND METHODS

2.1 Materials:- Stearic acid, Cetyl alcohol, glycerin, mineral oil, were obtained from SD fine Chemicals Ltd., IPM, Propyl paraben, CMC, EDTA, BHA, BHT, methyl paraben, and pigment were obtained from High Purity Laboratory Chemicals Pvt. Ltd., whereas TEA and distilled water were obtained from E.Merk Pvt. Ltd. and L.A.D respectively. Velvessil (Dimethicone (and) Cetearyl Dimethicone Cross Polymer) were collected from Momentive Performance Material (Nantong) Co. Ltd.

2.2 Preparation of Moisturizing Lotion

Selection of Base:- Stearic acid and TEA base have been selected for the formulation of moisturizing lotion because TEA can act as an emulsifier, helping to stabilize the oil and water phases in a lotion. It allows the ingredients to blend together and form a homogeneous product. They are compatible ingredients, and their combination can contribute to the desired texture, emulsion stability and pH control in a cream. Formulation of moisturizing lotion was done by taking 3 trials as shown in table no.4. O/W base was prepared.

| Component | Ingredients | Formulations (100%) | | |
|-------------|----------------|---------------------|-------------|-------------|
| | | F1 | F2 | F3 |
| Oil phase | Stearic acid | 1-5 g | 1-5 g | 1-5 g |
| | Cetyl alcohol | 1-2 g | 1.5-3 g | 2-4 g |
| | IPM | 1-2 ml | 1-2 ml | 1-2 ml |
| | Mineral oil | 1-1.5 ml | 1-1.5 ml | 1-1.5 ml |
| | Propyl paraben | 0.25-0.45 g | 0.25-0.45g | 0.25-0.45g |
| | BHA, BHT | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g |
| Water phase | Water | Upto 100 ml | Upto 100 ml | Upto 100 ml |
| | Glycerin | 5-10ml | 5-10ml | 5-10ml |

| | | | | |
|--|----------------|-------------|-------------|-------------|
| | CMC | 0.5-0.6 g | 0.7-0.8 g | 1-2 g |
| | EDTA | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g |
| | Methyl paraben | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g |
| | TEA | 0.4-0.5 ml | 0.4-0.5 ml | 1-2 ml |

Table no.4 Comparison of Different Base Formulations

Observation:- Formulation 3 (F3) was selected because it provide the desired consistency of lotion.

2.3 Formulation and Development of Moisturizing Lotion with Different Concentrations of Silicone

Table no. 5 Comparison of Different Formulations

| Component | Contents | Formulations (100%) | | | | | | | |
|-------------|----------------|---------------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| | | F1 | F2 | F3 | F4 | F5 | F6 | F7 | F8 |
| Oil phase | Stearic acid | 1-5g | 1-5g | 1-5g | 1-5g | 1-5g | 1-5g | 1-5g | 1-5g |
| | Cetyl alcohol | 1-2g | 1-2g | 1-2g | 1-2g | 1-2g | 1-2g | 1-2g | 1-2g |
| | IPM | 1-2ml | 1-2ml | 1-2ml | 1-2ml | 1-2ml | 1-2ml | 1-2ml | 1-2ml |
| | Mineral oil | 1-3ml | 1-3ml | 1-3ml | 1-3ml | 1-3ml | 1-3ml | 1-3ml | 1-3ml |
| | Propyl paraben | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g |
| | BHA, BHT | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g |
| | Velvesil (DM) | 1-4g | 5-6g | 7-10g | 11-12g | 13-16g | 17-18g | 19-22g | 21-24g |
| Water phase | Water | Upto 100ml | Upto 100ml | Upto 100ml | Upto 100ml | Upto 100ml | Upto 100ml | Upto 100ml | Upto 100ml |
| | Glycerin | 5-10ml | 5-10ml | 5-10ml | 5-10ml | 5-10ml | 5-10ml | 5-10ml | 5-10ml |
| | CMC | 1-2g | 1-2g | 1-2g | 1-2g | 1-2g | 1-2g | 1-2g | 1-2g |
| | EDTA | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g | 0.01-0.02 g |
| | Methyl paraben | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g | 0.25-0.45 g |
| | TEA | 1-2ml | 1-2ml | 1-2ml | 1-2ml | 1-2ml | 1-2ml | 1-2ml | 1-2ml |

Preparation of Moisturizing Lotion:

All ingredients were weighed accurately. Both phases i.e. oil phase containing Stearic acid, Cetyl alcohol, IPM, mineral oil, Propyl paraben, BHA, BHT, and different concentration of Velvesil were added into the phase and water phase contain water, glycerin, methyl paraben, glycerin, CMC and TEA were heated separately Upto 80-85°C. Oil phase were added to water phase with continue stirring with mechanical stirrer. As temperature falls below 40°C stirring was stopped to avoid excessive aeration. The moisturizing lotion base was formulated in the same manner except the silicone was not added into it.

3. EXPERIMENTATION

3.1 Preliminary Testing of Silicone: - Preliminary testing of silicon is often conducted in experimentation to assess its properties, behavior, and suitability for the intended application, understanding physical and chemical properties, it allows to characterize these properties, such as melting point, density, conductivity, and reactivity, which provides a foundation for further experimentation and helps determine how silicon may interact with other substances or materials.

1. Organoleptic Evaluation

Table no.6 Organoleptic Properties of Silicone

| | |
|------------|-----------|
| Appearance | Clear gel |
| Color | White |

| | |
|-------------------|--------------------------------|
| Odor | Odorless |
| Taste | Tasteless |
| Texture and touch | Smooth and uniform consistency |

2. Solubility

Table no. 7 Solubility of Silicone

| S.no | Solvents | Solubility Behavior |
|------|-------------|---------------------|
| 1. | Cold water | Insoluble |
| 2. | Warm water | Insoluble |
| 3. | Ethanol | Insoluble |
| 4. | Methanol | Insoluble |
| 5. | Acetone | Insoluble |
| 6. | Ether | Insoluble |
| 7. | Mineral oil | soluble |



Figure no.3 Solubility of Silicone in Different Solvents

3. **Flash Point:-** 140°C

4. **pH:-** 6.5

5. **Freeze -Thaw stability:-** The Velvesil gel was subjected to freeze thaw stability study for 7 days under -4°C and at 45°C. It was observed that the Velvesil gel was stable with no signs of change in color, consistency and no phase separation.

3.2 Evaluation of Prepared formulations

a. Rheological Characteristics

Table no. 8 Rheological Characteristics of Silicone

| S.no | Parameter | Formulation | | | | | | | | |
|------|---------------|-------------|----|----|----|----|----|----|----|----|
| | | B | F1 | F2 | F3 | F4 | F5 | F6 | F7 | F8 |
| 1. | Color | W | W | W | W | W | W | W | W | W |
| 2. | Spreadibility | G | A | A | A | E | E | E | E | E |
| 3. | Feel | - | S | S | S | S | S | S | S | S |
| 4. | Consistency | L | T | T | T | L | L | L | L | T |

B= Base, **W=** white, **G=**Good, **A=** Average, **E=**Excellent, **S=**Soft, silky, **T=**Thick, **L=**Lotion like

3.3 Analysis of Finished Moisturizing Lotion according to BIS^{18,19}

1. Test for the Thermal Stability

Apparatus:- A humidity chamber/incubator controlled at 60 to 70 % relative humidity and 45±1°C.

Clear glass bottle of around 30 ml capacity with plug and screw on cap for proper closure.

Procedure:- A 30 ml capacity of clear glass bottle with plug and screw cap was taken and with the help of spatula the cream was inserted and tapped it to the bottom. It was filled up to 2/3 capacity of bottle and plug was inserted and tightens up the cap. The filled bottle was kept inside the incubator at 45±1°C for 48hr.

Table no. 9 Test for the Thermal Stability

| S.no | Formulation | Thermal stability at 45±1°C |
|------|----------------|-----------------------------|
| 1. | Formulation F1 | No oil separation |
| 2. | Formulation F2 | No oil separation |
| 3. | Formulation F3 | No oil separation |
| 4. | Formulation F4 | No oil separation |
| 5. | Formulation F5 | No oil separation |
| 6. | Formulation F6 | No oil separation |
| 7. | Formulation F7 | No oil separation |
| 8. | Formulation F8 | No oil separation |

2. Determination of pH

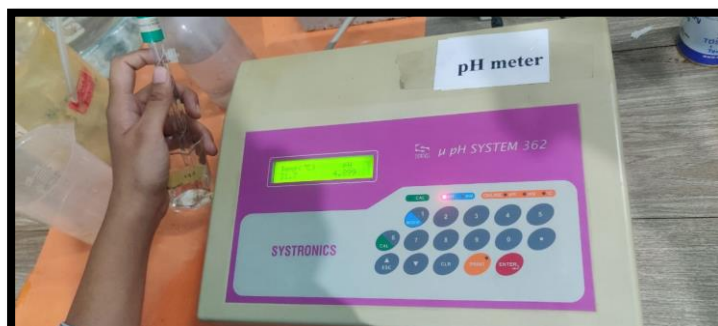
Apparatus: - pH meter, preferably equipped with a glass electrode.

Procedure:-

For oil-in-water emulsion :- 5±0.01g of the cream was weighed accurately in 100ml beaker. 45ml of water was added and cream was dispersed into it. Then the pH of the emulsion at 27°C was determined using the pH meter as shown in Fig no.4. The results are shown in table no. 10 and graph no.1.

Table no.10 pH of the formulations

| S.no | Formulation | Day 1 | Day 7 |
|------|----------------|-------|-------|
| 1. | Formulation F1 | 7.07 | 7.07 |
| 2. | Formulation F2 | 7.69 | 7.69 |
| 3. | Formulation F3 | 7.31 | 7.31 |
| 4. | Formulation F4 | 1.88 | 6.88 |
| 5. | Formulation F5 | 6.87 | 6.87 |
| 6. | Formulation F6 | 6.88 | 6.88 |
| 7. | Formulation F7 | 7.01 | 7.01 |
| 8. | Formulation F8 | 6.87 | 6.87 |

**Figure no.4 pH meter**

3. Determination of Total Fatty Substance Content

Principle: - The emulsion was broken with dilute mineral acid and the fatty matter was extracted with petroleum ether. It was weighed after removal of the solvent.

Reagents: - Dilute hydrochloric acid (1:1), petroleum ether, methyl orange indicator solution, sodium sulphate.

Procedure: - 2g of sample was weighed accurately in conical flask. 25ml of dilute hydrochloric acid was added. Reflux condenser was fitted in the flask and contents were boiled until the solution was perfectly clear. The contents of the flask were poured in 300 ml separating funnel and cooled at room temperature. The flask was rinsed with 50ml of petroleum ether in portions of 10 ml. Petroleum ether rinsing were poured into separating funnel and shaken well and left until the layers separate. The aqueous phase was separated out and shaken it with 50ml portions of petroleum ether twice. All the ether extracts were combined and washed with water until free of acid (when tested with methyl orange indicator solution). The petroleum ether extracts were filtered through a filter paper containing sodium sulphate into a conical flask which have been previously dried at a temperature of 90±2°C and then weighed. The sodium sulphate was washed with petroleum ether on the filter paper and combined the washing with filtrate. The petroleum ether was distilled off and material remaining in the flask was dried at a temperature 90±2°C of to constant mass. The results are shown in table no. 11 and graph no.2.

Total fatty sub. = $100 \frac{M_1}{M_2}$
 % by mass

Where

M_1 - Mass in g of the residue

M_2 - Mass in g of the material taken for use.

Table no.11 Percentage of Total Fatty Matter in Formulations.

| S.no | Formulation | TFM,% by mass |
|------|----------------|---------------|
| 1. | Formulation F1 | 0.1g |
| 2. | Formulation F2 | 0.2g |
| 3. | Formulation F3 | 0.2g |
| 4. | Formulation F4 | 0.2g |
| 5. | Formulation F5 | 0.2g |
| 6. | Formulation F6 | 0.2g |
| 7. | Formulation F7 | 0.2g |
| 8. | Formulation F8 | 0.3g |

4. Determination of Residue

Procedure:- 5g of material was weighed accurately in clean, dry weighing bottle and dried to constant mass at $105 \pm 1^\circ\text{C}$. Cool in desiccators and weighed. The results are shown in table no. 12 and graph no.3.

Residue % by mass = $100 \frac{M_1}{M_2}$

Where,

M_1 = mass in g of the residue, and

M_2 = mass in g of the material taken for test.

Table no.12 Percentage of residue in Formulations

| S.no | Formulation | Residue, % by mass at $105 \pm 1^\circ\text{C}$ |
|------|----------------|---|
| 1. | Formulation F1 | 7.3g |
| 2. | Formulation F2 | 5.7g |
| 3. | Formulation F3 | 4.6g |
| 4. | Formulation F4 | 5.8g |
| 5. | Formulation F5 | 5.7g |
| 6. | Formulation F6 | 6.3g |
| 7. | Formulation F7 | 6.2g |
| 8. | Formulation F8 | 6.2g |

5. Test for Heavy Metals

Outline of the method: - The color produced with hydrogen sulphide solution was matched against that obtained with standard lead solution.

Apparatus: - Nessler cylinders- 50 ml capacity

Reagents: - Dilute hydrochloric acid (5N), dilute acetic acid (1N), dilute ammonium hydroxide (5N), hydrogen sulphide solution, and standard lead solution

Procedure: - 2.000g of material was weighed accurately in a crucible and heated on a hot plate and then in a muffle furnace to ignite it at 600°C to constant mass. 3ml dilute hydrochloric acid was added, warmed (waited till no more dissolution occurs) and volume was made up to 100 ml. The solution was filtered. 25ml of the filtrate was transferred into Nessler's cylinder. In the second nessler's cylinder, 2ml of dilute acetic acid was added, 1.0ml of standard lead solution and volume was made up with water to 25 ml.

10 ml of hydrogen sulphide solution was added to each Nessler cylinder and made up the volume with water to 50ml. Mixed and allowed to stand for 10 min. compared the color produced in two nessler's cylinders. Blank determination without samples was recommended to avoid errors arising out of reagents.

6. Subjective Evaluation

For Subjective evaluation 10 subjects were selected on the basis of their skin type (normal skin, dry skin, oily skin). The formulations from F1-F8 were applied to 10 subjects of age group 22-23 the subjects were asked not to apply any skin care product 2 days prior to performing test. The hands of subjects were washed with soap and dried on the day of testing. The product was applied on their forearm and after that they were instructed to fill the questionnaire form

according to their preferences on the basis of following parameters on the scale of 1 to 10. Where 1 was the minimum/poor effect and 10 was the maximum/excellent effect. The results are shown in table no.14 and graph no. 4,5.

Poor = 1-3, Average = 4-6, Above average = 7-9, Excellent = 10

The parameters were

1. Feel of the product
2. Dragness of the product
3. Silkiness of the product
4. Greasiness of the product
5. Easy of spreadibility of the product

7. Evaluation by Corneometer

The Corneometer CM 825 is the most widely used instrument to determine the hydration level of the skin surface, mainly stratum corneum

Principle: - The measurement of the skin moisture was based on the internationally recognized corneometer- method, a capacitance method. This measurement was based on the completely different dielectric constant of water (81) and other substances (mostly <7). The measuring capacitor shows changes of the capacitance according to the moisture content of the samples. A glass lamina separates the metallic track (gold) in the probe head from the skin in order to prevent current conduction in the sample. An electric field between the tracks with alternating attraction develops. One track builds up a surplus of electrons (minus charge) the other a lack of electrons (plus charge). The scatter field penetrates the very first layer of the skin during the measurement and the capacitance was determined.

Unlike the impedance measurement no galvanic relation between the device and the measuring object or polarization effects exist.

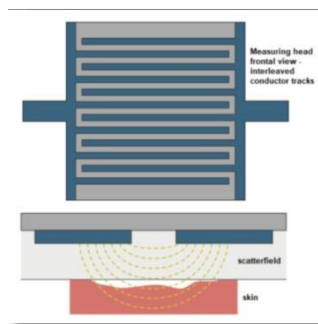


Figure no. 5 Working Principle of Corneometer

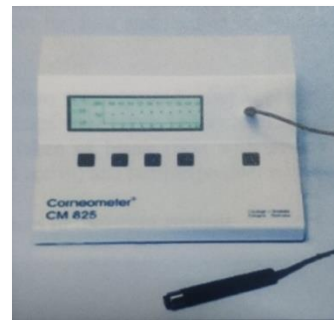


Figure no.6 Corneometer

Preparation of the Measurement

The corneometer CM 825 was a highly sensitive measurement instrument. For exact and reproducible measurement values it is important to follow the following instructions.

Preparation of the Room

- For all measurements of skin parameters it is important to keep constant ambient conditions. Temperature and relative humidity should be constant. This is vital for the comparison of long-term measurements. The optimum room condition was 18- 20°C and 40-60% relative humidity.
- Do not measure under direct lamp light or direct sunlight. Heat radiation might cause measurement inaccuracies. In case of a series tests, always measure at the same time of the day and with the same light conditions.

Preparation of Volunteer

- Allow your test persons to rest for at least 10-20 min, so that their blood circulation can regain a normal level after possible physical exercise. The skin area to be measured should not be covered with clothes during the acclimatization time. Emotional stress may also lead to increased transpiration. Make sure the acclimatization room is clean.
- If possible measure on hairless skin area. Should you wish to measure on hairy skin area, it is recommended to shave the respective area some time (1-2 days) before the measurements or out it very short with a pair of scissors.²⁰

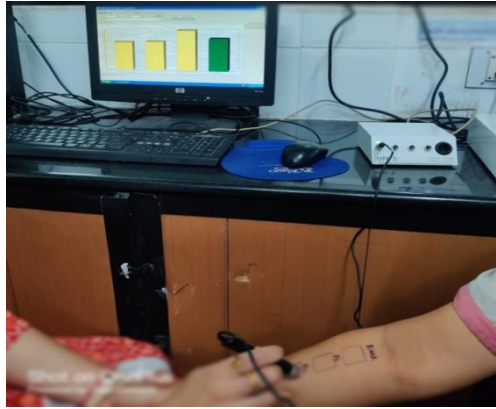


Figure no. 7 Determination of Skin Moisture by Corneometer

The Interpretation of the results

The following values are valid for healthy skin and normal room conditions (20°C and 40-60% air humidity) and will help to determine the skin type:

In arbitrary units between 0 and 130. The results are shown in table no.15 and graph no.6,7,8,9,10..

Table no.13 Interpretation of Corneometer Results

| | Inner Forearm |
|--------------------------|---------------|
| Very Dry | <30 |
| Dry | 30-45 |
| Sufficiently Moisturized | >45 |

4. RESULT

4.1 Preliminary Testing of Silicone

From the experiment and procedures, following results were obtained.

- 1. Organoleptic Evaluation:-** Silicone (Velvesil DM LC-101 PM 155993) was subjected to Organoleptic evaluation from table no. 9 it was observed that silicone was in the form of clear gel, odorless, tasteless, was smooth in texture and uniform in consistency.
- 2. Solubility :-** Silicon (Velvesil DM LC-101 PM 155993) was tested for its solubility in different solvents and from table no. 10 was found to be insoluble in cold water, warm water, ethanol, methanol, acetone, ether, but was found to be soluble in mineral oil.
- 3. Flash Point:-** Melting point of silicon (Velvesil DM LC-101 PM 155993) was found to be 140°C.
- 4. pH:-** The pH of silicon (Velvesil DM LC-101 PM 155993) was found to 6.5.
- 5. Freeze Thaw Stability:-** The silicone (Velvesil DM LC-101 PM 155993) was subjected to cyclic testing under -4°C and 45°C for 7 days and from table no. 11 it was found to be stable over the time period with no phase separation, color change and no consistency change

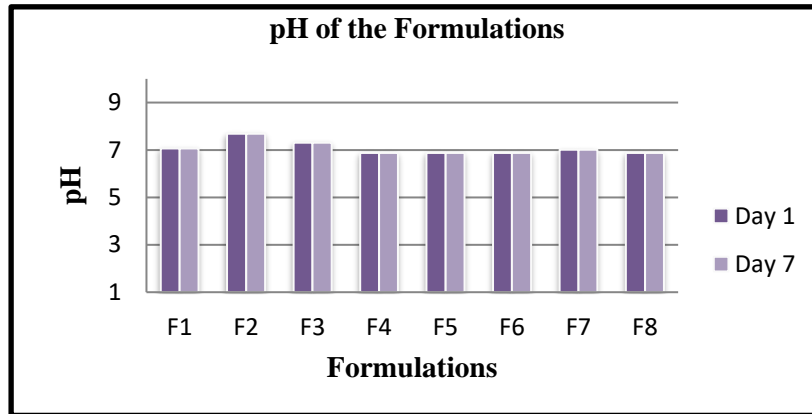
4.2 Experimentation of Moisturizing Lotion

a. Rheological Characteristics:- Moisturizing lotion was formulated with different concentrations of silicone and was evaluated for its rheological properties. It was found from table no.14 that all the formulation from F1-F8 were white in color including base, spreadability of base was found to be good whereas spreability of the F1,F2,F3 was found to be average as compared to F4,F5,F6,F7,F8 which was found to have excellent spreability, the feel of the base was found to be quit sticky as compared to F1-F8 which was found to be soft, silky smooth and on the basis of consistency of the formulated creams base, F4,F5,F6,F7 was found to have lotion like consistency whereas F1,F2,F3 and F8 was found to have thick cream like consistency. Due to this reason former formulations have been selected.

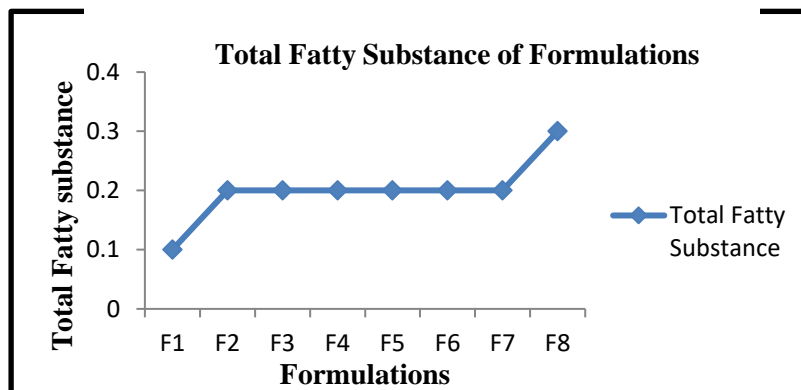
4.3 Evaluation of Moisturizing Lotion according to BIS^{17,18}

| S.no | Characteristics | Requirements | Lab values |
|------|-------------------|------------------|---|
| 1. | Thermal stability | To pass the test | All the formulations were found to be stable with no sign of separation |
| 2. | pH | 4.0-9.0 | All the formulation were found to have pH |

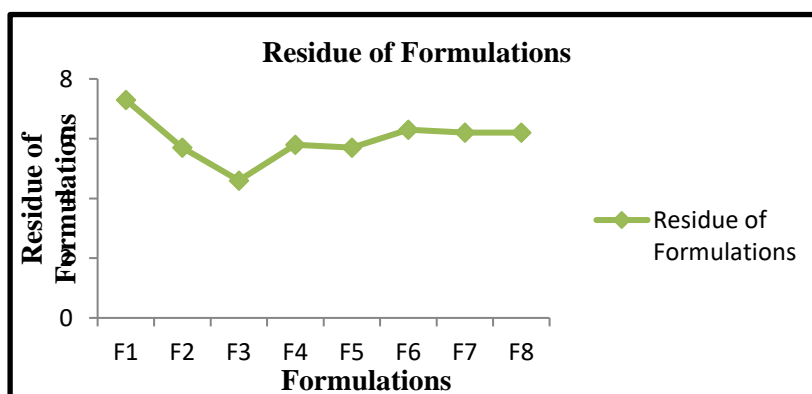
| | | | |
|----|--|-----|--|
| | | | within the range (refer graph no.1) |
| 3. | Total Fatty Substance Content, %by Mass, Min | 5.0 | All the formulation were found to have total fatty substance content, %by mass within the range |
| 4. | Total Residue, percent by mass, min | 10 | All the formulation were found to have Total Residue, percent by mass, within the range (refer graph no.3) |
| 5. | Heavy Metals (as Pb), parts per million, Max | 20 | The color produced by formulation was less as compared to the standard lead solution. |



Graph no.1 Determination of pH



Graph no.2 Determination of Total Fatty Substance



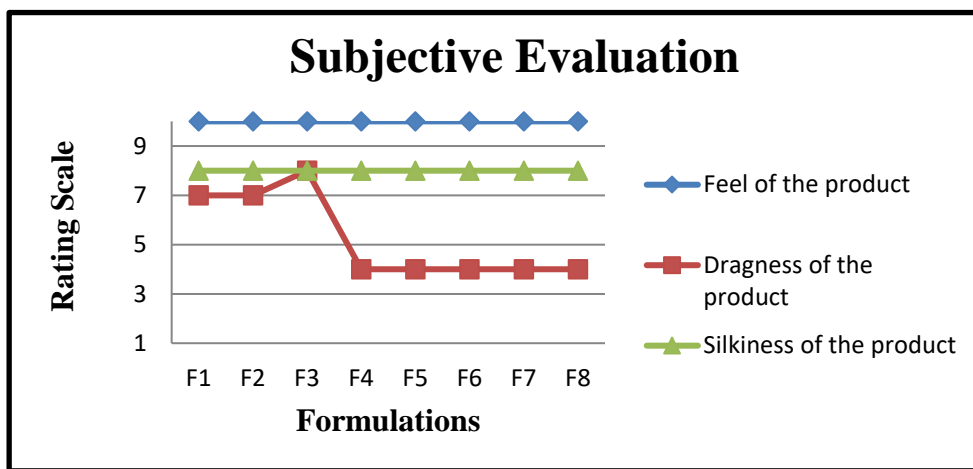
Graph no.3 Determination of Residue

6. Subjective Evaluation

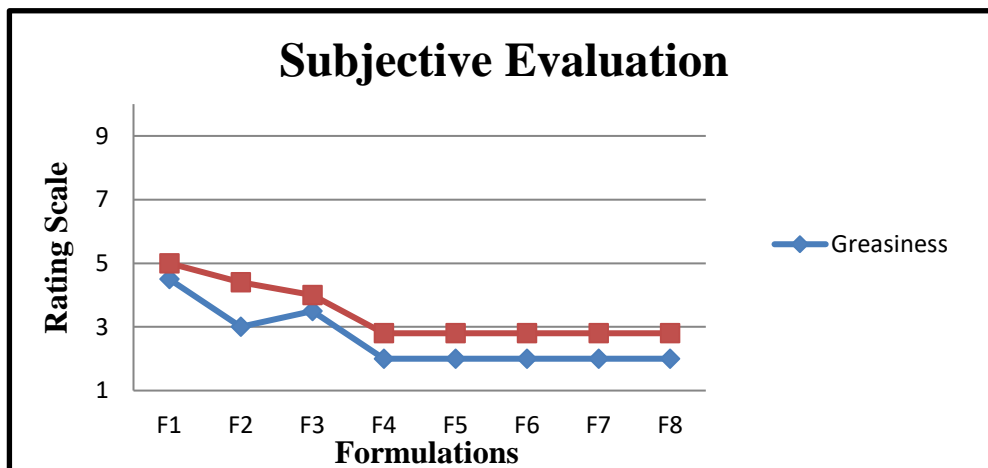
Table no.14 Subjective Evaluation of Formulations

| Formulations | Rating | | | | |
|--------------|--------|----------|-----------|------------|-------------------|
| | Feel | Dragness | Silkiness | Greasiness | Ease of spreading |
| F1 | 10 | 7 | 8 | 4.5 | 5 |
| F2 | 10 | 7 | 8 | 3 | 4.5 |
| F3 | 10 | 8 | 8 | 3.5 | 4 |
| F4 | 10 | 4 | 8 | 2 | 2.8 |
| F5 | 10 | 4 | 8 | 2 | 2.8 |
| F6 | 10 | 4 | 8 | 2 | 2.8 |
| F7 | 10 | 4 | 8 | 2 | 2.8 |
| F8 | 10 | 4 | 8 | 2 | 2.8 |

Poor = 1-3, Average = 4-6, Above average= 7-9, Excellent = 10



Graph no. 4 Determination of Feel, Dragness, and Silkiness of product



Graph no. 5 Determination of Greasiness, Ease of spreadability of Product

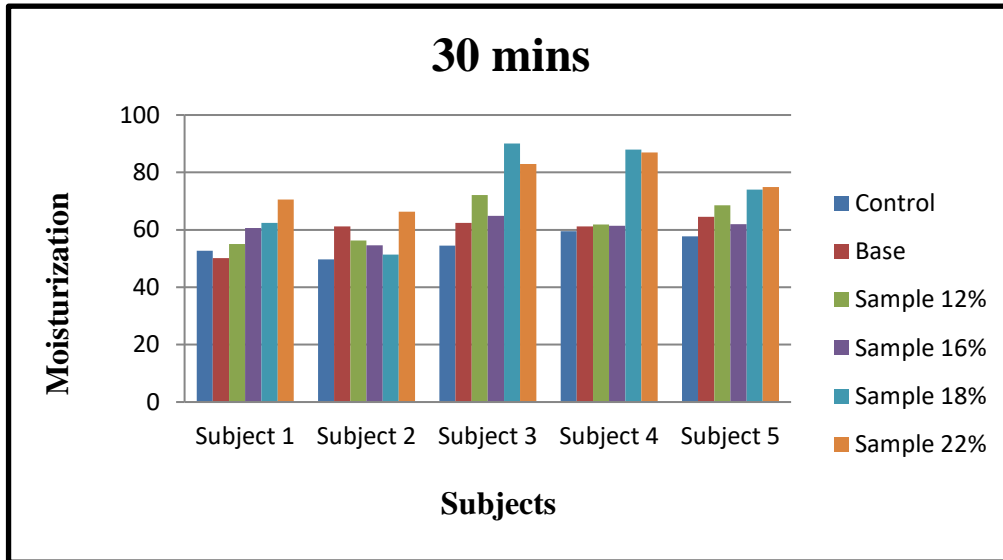
7. Evaluation by Corneometer

Moisture content valued of 5 subject obtained by corneometer is mentioned in the following table:-
 Room temperature:- 18-20°C

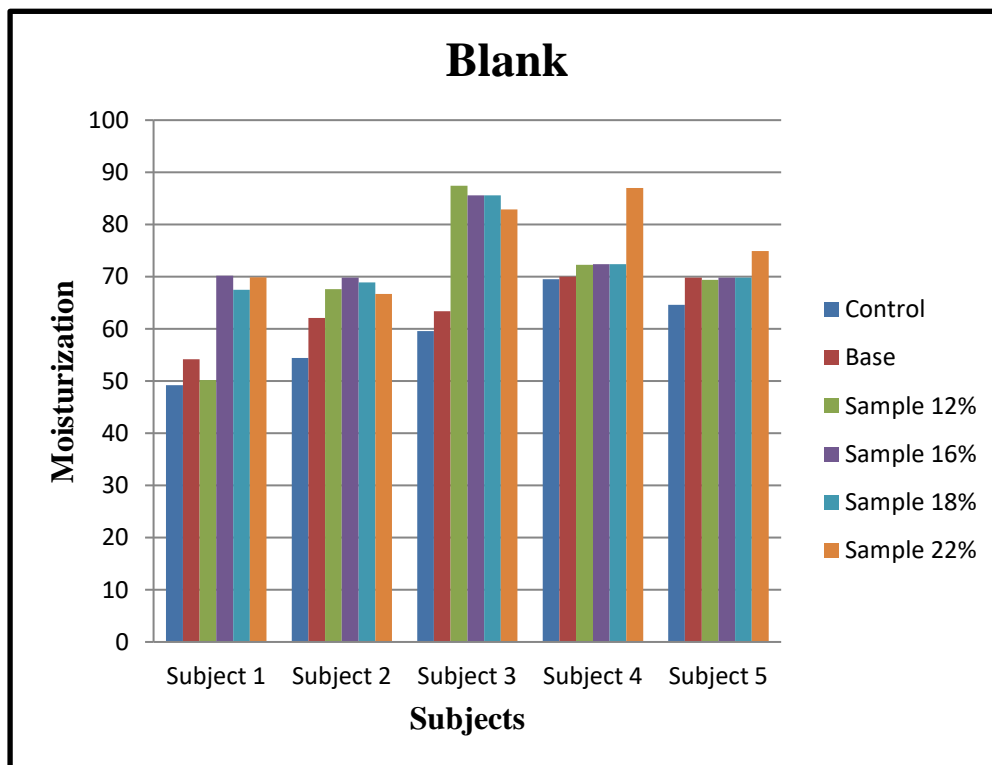
Table no.15 Moisture Content Obtained by Corneometer

| Subjects | | Time (minutes) | | | |
|----------|------------|-----------------|--------|--------|--------|
| | | Blank 150min | 30 min | 60 min | 90 min |
| Sub 1 | Control | 49.2 | 52.7 | 48.1 | 57.1 |
| | Base | 57.1 | | | |
| | Sample 12% | 54.2 | 50.2 | 54.6 | 54.2 |
| | Sample 16% | 54.2 | | | |
| | Sample 18% | 50.2 | 55 | 52.4 | 55.2 |
| | Sample 22% | 55.2 | | | |
| | | 70.2 | 60.6 | 61.2 | 60.6 |
| | | 60.6 | | | |
| | | 67.5 | 62.4 | 61.9 | 63 |
| | | 63 | | | |
| Sub 2 | Control | 54.4 | 49.7 | 48.2 | 54.6 |
| | Base | 54.6 | | | |
| | Sample 12% | 62.1 | 61.2 | 62.4 | 62.2 |
| | Sample 16% | 62.2 | | | |
| | Sample 18% | 67.6 | 56.3 | 58.7 | 58.9 |
| | Sample 22% | 58.9 | | | |
| | | 69.8 | 54.6 | 54.8 | 54.8 |
| | | 54.8 | | | |
| | | 68.9 | 51.4 | 51.5 | 51.6 |
| | | 51.6 | | | |
| Sub 3 | Control | 59.6 | 54.5 | 54.7 | 54.6 |
| | Base | 54.6 | | | |
| | Sample 12% | 63.4 | 62.4 | 63.2 | 63.2 |
| | Sample 16% | 63.2 | | | |
| | Sample 18% | 87.4 | 72.1 | 71.5 | 72.3 |
| | Sample 22% | 72.3 | | | |
| | | 85.6 | 64.9 | 65 | 65.2 |
| | | 65.2 | | | |
| | | 90.1 | 72.1 | 72.4 | 73 |
| | | 82.9 | 66.6 | 65.8 | 66.6 |
| Sub 4 | Control | 69.5 | 59.5 | 60.5 | 60 |
| | Base | 60 | | | |
| | Sample 12% | 70 | 61.2 | 69.9 | 69.7 |
| | Sample 16% | 69.7 | | | |
| | Sample 18% | 72.3 | 61.9 | 69.8 | 69.8 |
| | Sample 22% | 69.8 | | | |
| | | 72.4 | 61.4 | 61.7 | 61.7 |
| | | 61.7 | | | |
| | | 88 | 68.9 | 70.4 | 70.3 |
| | | 70.3 | | | |
| Sub 5 | Control | 64.6 | 57.7 | 57.5 | 57.2 |
| | Base | 57.2 | | | |
| | Sample 12% | 69.8 | 64.5 | 65.2 | 65 |
| | Sample 16% | 69.4 | 68.5 | 68.7 | 68.6 |

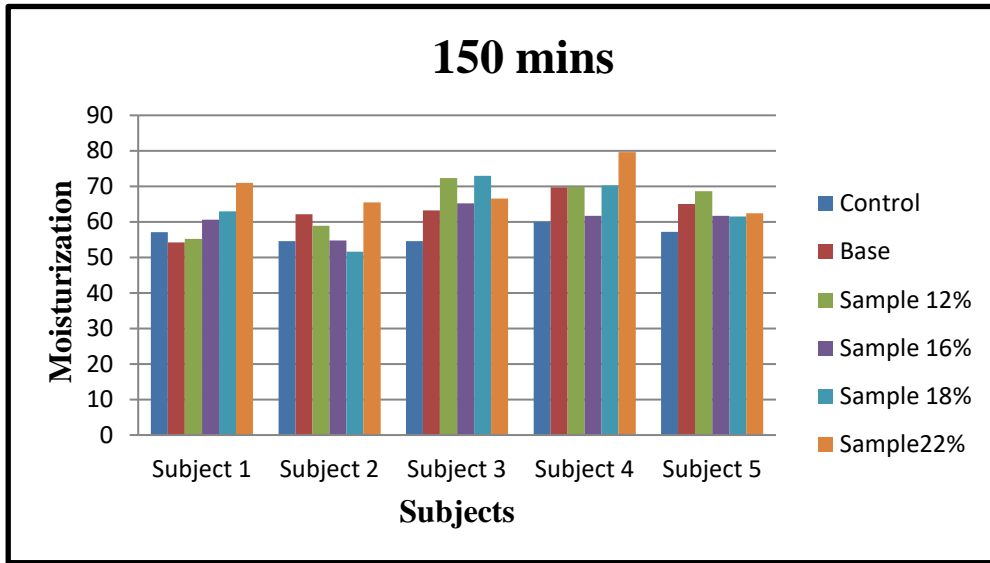
| | | | | | |
|--|------------|------|------|------|------|
| | Sample 18% | 68.6 | 62 | 61.9 | 61.7 |
| | Sample 22% | 69.8 | | | |
| | | 61.7 | 62.3 | 61.7 | 61.5 |
| | | 74 | | | |
| | | 61.5 | | | |
| | | 74.9 | 63.3 | 62.1 | 62.4 |
| | | 62.4 | | | |



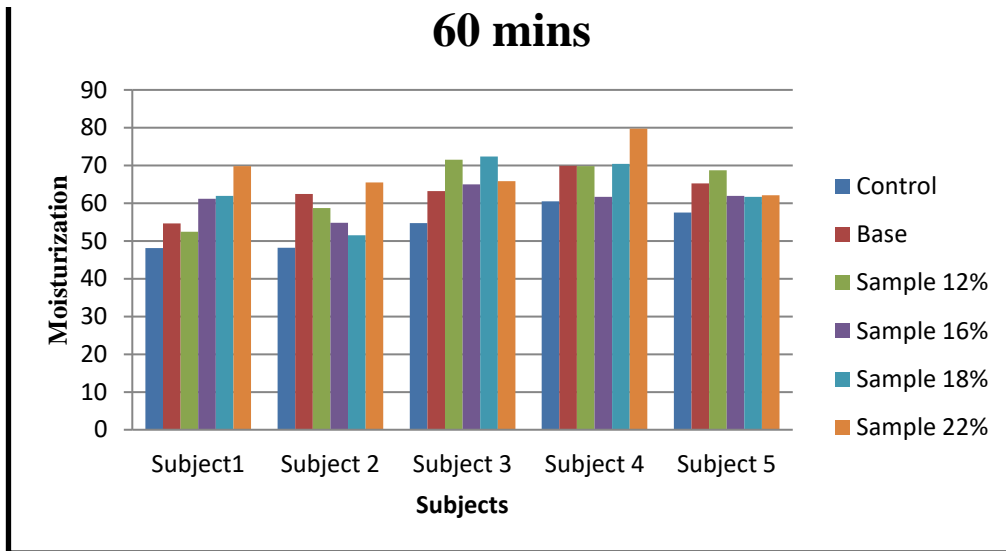
Graph no.6 Determination of Moisture Content of subjects at Blank Time



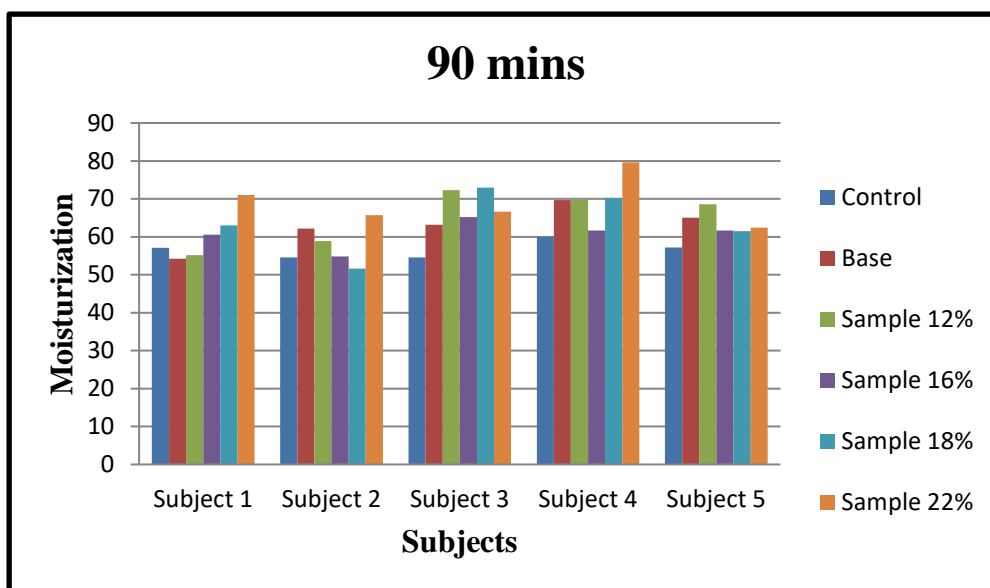
Graph no. 7 Determination of Moisture Content of subjects After 30 mins



Graph no. 8 Determination of Moisture Content of subjects After 60 mins



Graph no.9 Determination of Moisture Content of subjects After 90 mins



Graph no.10 Determination of Moisture Content of subjects After 150 mins

5. Discussion and Conclusion

Human skin is exposed to a variety of factors in daily life that have a negative impact on dermal integrity and cause dry skin and wrinkles. Moisturizing lotions are cosmetic products designed to hydrate and nourish the skin, helping to restore and maintain its moisture balance the use of emollients, moisturizing creams and lotions is the most typical defense and prevention measure against dry skin. Silicones, such as dimethicone and cyclopentasiloxane, are often included in moisturizing lotions for their unique texture and sensory properties. They contribute to a smooth and silky feel, allowing the lotion to spread easily on the skin. This helps in effortless application and even distribution of the product. Current study involves formulation of moisturizing lotion for dry skin containing 8 different concentrations of silicone incorporated into it. These formulations were evaluated for their rheological characteristics and their stability was evaluated by performing testing according to BIS: 6608 (2004): Skin Creams. The stability and compatibility of silicone with product was carried out for 7 days.

From the results of rheological characteristics it may be concluded that F1, F2, F3 had average spreadability, smooth, silky texture but F1 was found to be having cream like consistency as compared to F2, F3 whereas on the other hand F4, F5, F6, F7, F8 were found to have excellent spreadability, smooth, silky texture and have lotion like consistency except F8 which possessed cream like consistency. Due to this reason F1, F8 were not selected.

From the results of thermal stability it may be concluded that all the formulations were found to be stable at $45 \pm 1^\circ\text{C}$ without any signs of physical changes such as phase separation, creaming, flocculation, and chemical separation such as odor change, color change, staining etc was not observed in any of the formulations.

From the results of pH it may be concluded that the pH of all the formulation taken on the day after formulation was found to be within the pH range of 4-9 and when taken after 14 days the pH of the formulations does not change and remained the same. Hence all the samples have passed the test.

The total fatty substances content, percent by mass, min, required should be 5 and from the results of TFM it may be concluded that all the formulations were found to have values within the range. Hence all the formulations have passed the test.

The total residue, percent by mass, required should be 10 and from the results of residue it may be concluded that all the formulations were found to have values within the range and all the formulations have passed the test.

In heavy metals test the color developed in the sample solution is less than that of standard solution. Hence, from the results it may be concluded that all the formulations have passed the test and does not produce any color.

In subjective evaluation the formulations were applied on 10 subject and from the result it may be concluded that feel and silkiness of all the formulations was found to be excellent whereas the F1,F2,F3 were rejected due to poor spreadability, dragness as compared to F4,F5,F6,F7,F8, and F8 were rejected due to its cream like consistency. F4,F5,F6,F7 were selected for their feel, silkiness, low Dragness, low greasiness and ease of spreadability and further subjected for corneometer evaluation. From the results of corneometer it may be concluded that all the sample provide sufficient moisturization to the skin of subjects i.e. greater than 45 (upto 2.5hr.) but sample 22% provide

higher moisturization as compared to other formulations in the skin of subjects. From the results of subjective evaluation and results of corneometer it can be concluded that but sample 22% was found to excellent as compared to other formulations in terms of its Moisturization, feel, consistency, texture, spreadability, dragness and greasiness.

Consumers today have a lot of myth about silicones use in cosmetic products but from the current research work done and from subjective evaluation it can be concluded that silicones are non-toxic, easily washable with water and surfactant ,safe to use, non-irritant, non-comedogenic, non- greasy, non-sticky and can actually enhance the performance of other ingredients in cosmetic products, they undergo indirect photolytic degradation and are not harmful for the environment hence they are biodegradable.

These advanced silicone formulations are likely to gain popularity in the future as consumers seek products that offer multiple benefits. Moreover, technological advancements and scientific research may lead to the development of new types of silicones specifically designed for cosmetic applications. These novel silicone molecules could possess enhanced functionalities, improved biodegradability, or other desirable characteristics. Continuous innovation in the field of silicone chemistry leads to new possibilities in cosmetic formulations.

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REFERENCES:

1. Mezail Mawazi. S, Ann.j, Othman. N, Khan.J, Othman Alolayan. S, S.Al Thagfan.S, Kaleemullah.M, A Review of Moisturizers,History, Preparartion, Characterization and applications, MDPI, 2022:Vol9(3):61
2. Purnamawati. S,Indrastuti .N, Danarti. R, Saefudin. T, The Role of Moisturizers in Addressing Various Kinds of Dermatitis: A Review, Clin. Med. Res.,2017:Vol 15, 75–87.
3. Lodén. M, Prevention or promotion of dryness and eczema by moisturizers? *Expert Rev. Dermatol.*, 2008: Vol 3, 667–676.
4. O'Lenick. A & O'Lenick. K, Silicone Polymers in Skin Care, MRS Bulletin.,2007Vol 32(10):167.
5. Kraft. JN, Lynde. CB, Lynde. CW. Moisturizers: An essential component in eczema management. (“Probiy Medical Research Inc - Charles Lynde”). *Skin Ther Lett.* 2009:Vol 5(2):5–7.
6. Shin. HT, Diaper dermatitis that does not quit, *Dermatol Ther*, 2005: Vol 18(2):124–35.
7. Kumar Singh. V, Silicone in Cosmetics, Pharamatutor Pharmacy Infopedia, 2016.
8. Andrade.FF, Santos. ODH, Oliveira .WP, Rocha-Filho.PA, Influence of PEG-12 Dimethicone addition on stability and formation of emulsions containing liquid crystal. *Int J Cosmet Sci*, 2007:Vol 29(3):211–8.
9. Akiyama. E, Kashimoto. A, Hotta. H, Kitsuki. T, Mechanism of oil-in-water emulsification using a water-soluble amphiphilic polymer and lipophilic surfactant. (“Energies | Free Full-Text | Study on the Influencing Factors of the ...”), *J Colloid Interface Sci*, 2006: Vol 300(1):141–8
10. Suitthimeathegorn. O, Jaitely. V, Florence AT. Novel anhydrous emulsions: Formulation as controlled release vehicles, *Int J Pharm*, 2005:Vol 298(2):367–71.
11. James V. Gruber, Principles of Polymer Science and Technology in Cosmetics and Personal Care, edited by E. Desmond Goddard, Silicones in cosmetics: 289-338.
12. Alvarez-Munoz.D, Liorca.M, Blasco .J, Barcelo.D, Current Knowledge and Future Issues, edited by Julián Blasco, Peter M. Chapman, Olivia Campana, Miriam Hampel, Marine Ecotoxicology,Academic Press, Contaminants in the Marine Environment:,2016:1-34
13. Sayyed. A, Kulkarni. R, Silicone Chemical in Cosmetics Applications to the Environment, Health and Sustainability, *Research Gate*, 2022:Vol.30:18-24.
14. Momentive, Momentive Performance Material (Nantong) Co.Ltd. Certificate of Analysis:1.
15. Glombitza. B, Muller-Goymann .CC.,Investigation of interactions between silicones and stratum corneum lipids. *Int J Cosmet Sci.* 2001: Vol 23:25–34.
16. Disapio. A, Fridd. P, Silicones: use of substantive properties on skin and hair. (“The Truth About Silicone - Southern Marin Dermatology”). *Int J Cosmet Sci.* 1988: Vol 10:75–89.
17. Bureau of Indian Standards IS: 6608 (2004): Skin Creams: 1-7.
18. Indian Pharmacopoeia 1996 Volume-II, published by the Controller of Publications Delhi, 813, 765.
19. User Manual, Courage Kazaka, Analytical Method of Corneometer CM 825:1-7.