Formulation And Evaluation of Skin Cream with Curcumin Caesia

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***Abstract*-Black turmeric (Curcuma caesia Roxb.) has emerged as a potent botanical ingredient in dermatological formulations due to its rich phytochemical profile and therapeutic properties, to study the benefits of biological active curcumin caesia, two Oil in water O/W cream formulations were prepared. One with 5 % curcumin caesia SLN which is extracted in 95 % ethanol and another without active is prepared to study physicochemical parameters, stability, microbial test and efficacy of cream formulation prepared with SLN of curcumin caesia extract. This research deliberate in detail design of formulation, process of manufacturing, test parameters and stability at various climatic conditions to stabilize cream formulation and profit desired benefits. The improvement in skin benefits like anti-ageing, antioxidant, moisturising properties can be further evaluation by in-vitro analysis**

***Keywords*: Cosmetic formulations, Solid Liquid Nanoparticle (SLN), Cosmetic Dermatology, Nanoparticles, Topical Delivery Systems, Curcumin Caesia Roxb, Black Turmeric**

Graphical Abstract:

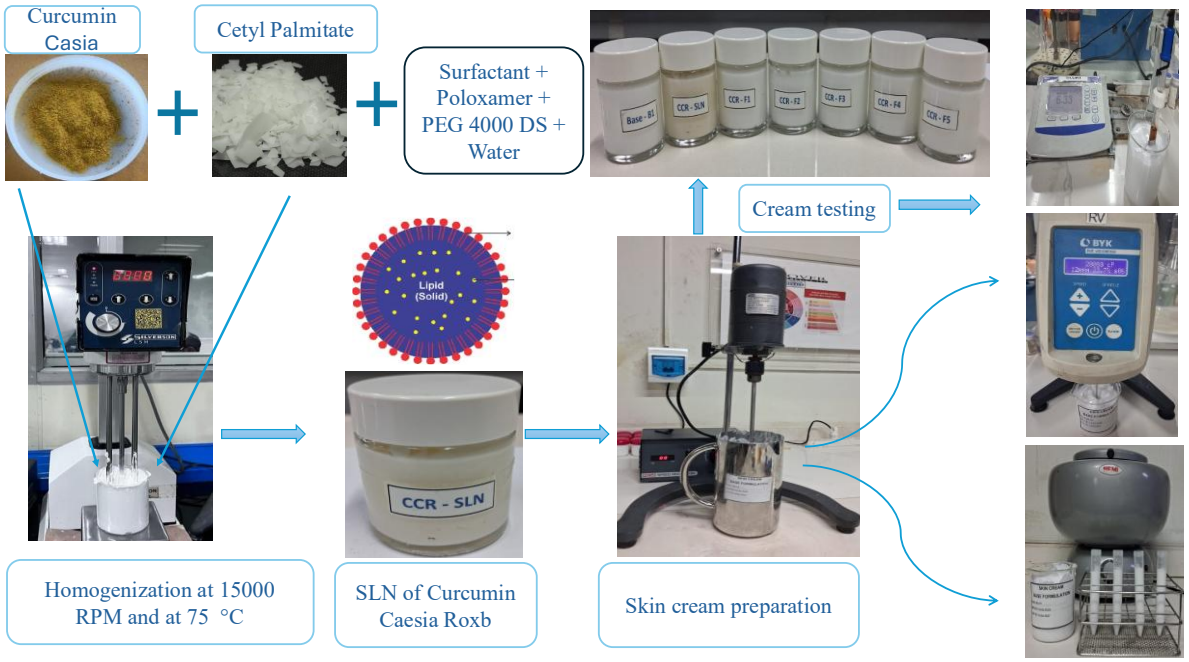

Figure 1: Graphical abstract

I. INTRODUCTION

The wish to become young is a natural behavior of every human being which is been complimented with high disposable income have given rise to many new companies in area of cosmetics. Today, there are 400 plus manufacturers of cosmeceutical products, the industry is growing at a pace of 7.5 %. Even though many of products are claiming predictable benefits, the cosmeceutical industry is unregulated, and end users shall consult skin specialists or dermatologists before using such products. In recent times, skin health and its beauty are perceived as an indicator of one's health which has resulted in an increasing demand for anti-aging products [1]. It is a necessity to identify key cosmeceutical active ingredients

which deliver exact benefits to consumers in the stipulated time frame. The usage of plant based herbal treatment is developed for effective treatment and minimal side effects. Those traditional remedies have been used by our ancestors based on their experience.

Black Turmeric (*Curcumin Casia*) is known for its medicinal properties. The fresh rhizome decoction is used as antidiarrhoeic and to get relief of gastric trouble [2]. The paste of *Curcumin Casia* is applied in snake bite [3]. Herbal cosmetics have been the first choice of consumer for simple reason being is that it has less side effects and higher on performance compared to synthetic chemicals [4]. The commonly used methods to extract black turmeric are maceration, reflux, Soxhlet extraction and hydro-distillation.

Topical delivery system and Advance active delivery systems like Nanotechnology-driven delivery platforms have gained significant attention for maximizing dermal and transdermal delivery of black turmeric phytoconstituents. Liposomes, composed of phospholipid bilayers, can encapsulate both hydrophilic and lipophilic compounds, improving solubility and skin deposition. Using the tools of nanotechnology, drug delivery systems within the nanometre size regime can be developed to alter both pharmacological and therapeutic effects of drug molecules. Due to their small size, these novel nanotechnologies offer superior advantages, such as altered pharmacokinetic behaviour and improved payload, over traditional large-scale systems. In addition, the relative ease in modifying their surface chemistry permits the attachment of targeting and therapeutic molecules for specific therapeutic applications. Finally, complex nanostructures can be assembled using different building blocks with multiple functionalities ranging from targeting, detecting, imaging and therapeutic capabilities [5].

II. METHODOLOGY

Collection and authentication of Rhizomes

Fresh rhizomes of *Curcuma caesia* Roxb. were collected from the medicinal germplasm garden of the Regional Plant Resource Center (RPRC), Bhubaneswar, India. The plant material was authenticated by a qualified taxonomist, and voucher specimens were deposited for future reference [6]. Rhizomes were thoroughly washed under running tap water to remove soil and debris, followed by shade drying at ambient temperature to prevent degradation of thermolabile compounds [8] (Saurav Gaikwad, 2023).

Processing of Rhizomes

Driedrhizomes were cut into small chips and pulverized using a mechanical grinder to obtain a fine powder. The powdered material was stored in airtight containers under dry conditions to avoid moisture absorption and microbial contamination [9]

Figure 2: Rhizome of *Curcuma caesia* Roxb.

Figure 3 : Leaves of Curcuma Caesia Roxb.

Figure 4: Powder of CCR Rhizome

Figure 5 : Powder of CCR Rhizome

Preparation of Ethanol extract:

Extraction was carried out using maceration technique. Maceration is widely used for phytochemical extraction due to its simplicity and ability to preserve heat-sensitive compounds (Sonal, 2023). Approximately 50 g of rhizome powder was soaked in 200 mL of ethanol in separate beakers. The mixtures were stirred intermittently and left overnight for percolation. Filtration was performed using Whatman No. 1 filter paper, and the process was repeated thrice for exhaustive extraction. The filtrates were concentrated under reduced pressure using a rotary evaporator at 40–45°C and stored in screw-cap vials until analysis [10, 11]. The extract was dried for more than 50 % by evaporation method to yield good active levels.

Preparation of Solid Lipid nanoparticle (SLN)

This review was conducted using a systematic approach to identify, analyze, and synthesize literature on the dermatological and cosmetic applications of SLN and its formulations. A comprehensive literature search was performed using Google Scholar, supplemented by PubMed and ScienceDirect for peer-reviewed sources, using keywords like “Solid Liquid Nanoparticle, Cosmetic Dermatology, Nanocarriers, Nanoemulgel, Ethosomes, Liposomes, Organogels, Green Nanotechnology, Topical Delivery Systems, Drug delivery system” [12-17]

SLN Formulation: Table 1: Typical SLN formulation with optimized percentage of active [12-17]

Ingredient	% (W/W)	Function
Cetyl palmitate	8	Lipid
Poloxamer 407	4	Non-Ionic Surfactant
Active - Black Turmeric Extract	5	Cosmetic Active
Polysorbate 80	1	Co-surfactant
Poly Ethelene Glycol 4000 DS	0.5	Skin Moisturizing
Glycerin	2	Humectant
Water	QS to 100	Solvent

Process: Hot homogenization:

Hot homogenization is carried out at temperatures above the melting point of the lipid and is like the homogenization of an emulsion. A pre-emulsion of the drug loaded lipid melt and the aqueous emulsifier phase (same temperature) is obtained by high-shear mixing device like Silverson - type homogenizer [18].

SLN of Curcumin Caesia Roxb Dispersion

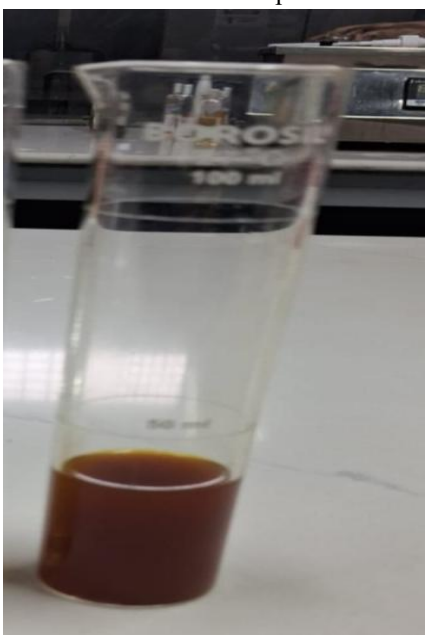

Figure 5: Ethanolic extract of CCR

Figure 6: Silverson High Shear Lab Homogenizer

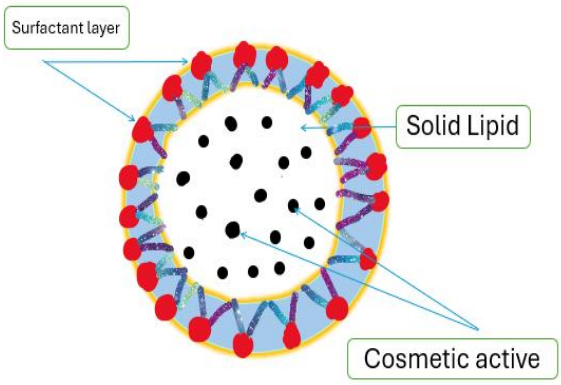

Figure 7: Structure of SLN

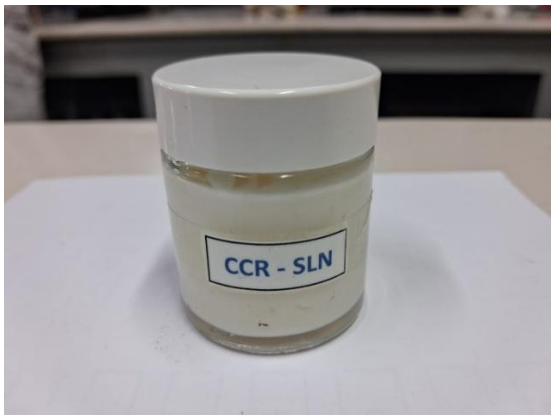

Figure 8: SLN of CCR – Lab made sample

Skin Cream Base Formulation:

Skin cream preparation formulae have been made with varied percentage of SLN Black turmeric active mentioned below along with base formulation

Table 2: CCR SLN formulation with various percentage of CCR SLN

Skin Cream: Formulations							
Ingredients	Base Formulation in %	Formulation with Black Turmeric SLN Active					Function
Code	B1	F1	F2	F3	F4	F5	
Black Turmeric SLN Extract	0.00	0.50	1.00	2.50	4.00	5.00	Cosmetic Active
Carbopol-940	0.25	0.25	0.25	0.25	0.25	0.25	Rheology modifier
Propylene Glycol	2.00	2.00	2.00	2.00	2.00	2.00	moisturizer

Sodium Methyl Paraben	0.15	0.15	0.15	0.15	0.15	0.15	Preservative
Disodium EDTA	0.10	0.10	0.10	0.10	0.10	0.10	Chelating agent
Glycerin	2.00	2.00	2.00	2.00	2.00	2.00	Humectant
Cetosteryl alcohol	7.50	7.50	7.50	7.50	7.50	7.00	Emulsion stabilizer
Stearic Acid	1.00	1.00	1.00	1.00	1.00	1.00	Lipid
Mineral Oil	4.00	4.00	4.00	4.00	4.00	4.00	Emollient / Oil solvent
Emulgade 165	2.00	2.00	2.00	2.00	2.00	2.00	Emulsifier
Iso Propyl Myristate	2.00	2.00	2.00	2.00	2.00	2.00	Emollient
Dimethicone 350	1.00	1.00	1.00	1.00	1.00	1.00	Emollient
Propyl Paraben	0.10	0.10	0.10	0.10	0.10	0.10	Preservative
Sodium Hydroxide	0.20	0.20	0.20	0.20	0.20	0.20	Neutralizer
Phenoxyethanol	0.10	0.10	0.10	0.10	0.10	0.10	Preservative
Perfume	0.50	0.50	0.50	0.50	0.50	0.50	Fragrance
Water - QS to 100 %	QS	QS	QS	QS	QS	QS	Solvent

The cream is oil in water (O/W) emulsion where oil is dispersed in water whereas water is in continuous phase and oil is dispersed in droplets. Cream preparation formulations process in two phases [19-22].

1) Water phase consists of water, glycerine, propylene glycol, Disodium EDTA, Carbopol-940, Sodium Methyl Paraben which is taken into a main mixer and heated up to 78 to 80 °C.

2) Oil phase - in another mixer oil phase is prepared with Cetosteryl alcohol, Stearic Acid, Mineral Oil, Iso Propyl Myristate, Dimethicone 350, Emulgade 165, Propyl Paraben and heated up to 78 to 80 °C.

3) At temperature of 78 to 80 °C, oil phase is added slowly to water phase under vigorous stirring at 1500 rpm for about 30 to 40 mins and homogenized entire batch at 2800 rpm for 10 to 15 mins once emulsification is done then cooling is started

4) Below 50 °C, at slow stirring with anchor stirrer between 30 to 40 rpm Sodium Hydroxide is added and below 45 °C, SLN of Black Turmeric Extract, Phenoxyethanol & Perfume is added slowly at about 15 to 20 rpm until homogeneous cream is form

Figure 9: Lab preparation of CCR Formulation

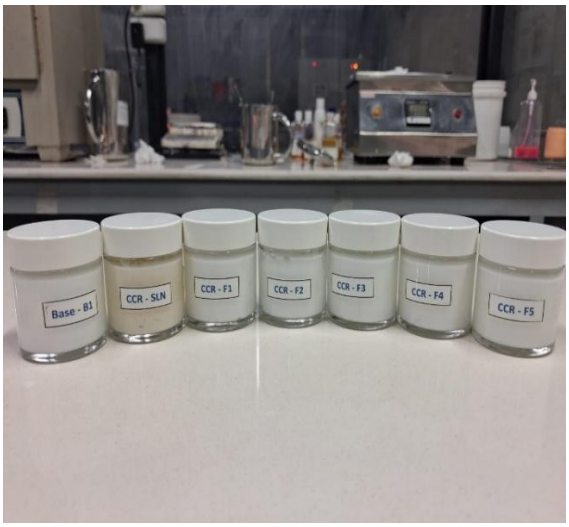

Figure 10: Skin Cream trial with various % of CCR

Evaluation of Skin Cream preparations with SLN of Black turmeric extract

Sensorial test: A sensorial test is carried out by trained panel of professionals to assess how skin cream feels, smells, looks, appears and behaves during and after application feel [23].

Appearance: The lab made formulation of skin cream with SLN active samples visually checked and found to be uniform, homogeneous, viscous, firm nevertheless soft feel by touch, easy to pick up from jar with figure and white colored cream easily spreadable on skin and glides easily during rub off [23].

Odour Test: Take both standard sample and test sample on smelling strip pre-coded with so panel shall not know the codes Give the coded samples to Panel members and evaluate the sample and record the responses [24].

Homogeneity Test: A small amount of cream as a drop is applied on the glass slide and other glass slides are placed on each other to spread the cream across slide, the cream shall look homogenous and do not appear as a coarser particle [25].

Skin Application Method: Apply about 1 gram of cream on the volar surface of the forearm and with the help of figure give 2 to 3 circular motion and capture the response in terms of easy of spreading, drag, coverage uniformity and residue on surface [23].

After application feel Emollience: The cream glides nicely on the skin when applied it gets absorbed easily into skin, leaving no residue and feels smooth and silky after application

Skin irritation test: A patch test is conducted on 10 members of panel. The cream is applied on forearms and assessed after 24 hours and checked for any reaction, irritation, erythema or edema [25].

Spreadability test: Weigh 0.5 grams of cream samples, place on the middle part of petri dish, place another dish on the top. Apply standard weight of about 500 gm and wait for 1 minute and measure diameter of spread circle [25, 31, 32].

pH Test

Weigh accurately 5+0.01 g of the cream in a 100 ml beaker. Add 45 ml of water and disperse the cream in it. Determine the pH of the suspension at 25°C using the pH meter [25].

Determination of Viscosity by Brookfield viscometer: Take a 250 ml beaker and place about 90 % with cream, Place beaker under viscometer, select spindle No. 4 & 12 RPM. Place spindle into beaker up till the groove mark of spindle. Set speed to 12 RPM, start viscometer and measure the reading on the digital screen in cps after 2 minutes when reading is stable [25].

Determination of specific gravity: Weigh empty jar with lid, then fill water and weigh again. Fill the jar with cream and note weight & calculate by below formula [26].

$$\text{Specific Gravity} = \frac{\text{Weight of sample}}{\text{Weight of equal volume of water}}$$

Moisture & Volatile content: [27].

Set the oven at 105°C. Weigh around 2 to 5 grams of sample in a clean and dry Petridis and Record reading as a 'A'. Keep a sample in hot air oven for minimum 3hrs. Carefully remove the sample from hot air oven using pair of tongues and keep it in desiccators, wait to cool to Room temperature. Then weigh the sample and record as 'B'

$$\text{Calculation: \% MIV} = 100 - \left[\frac{(A-B) \times 100}{A}\right]$$

Whereas - A – Weight of sample + Petridis in gm

B – Weight of sample after drying in gm

Stability preparation

Thermal Stability (Ref. IS 6608:2004 - Bureau of Indian standard)

With the help of spatula, insert the cream into clear glass bottles of around 30 ml capacity with plug and screw on cap for proper closure and tap it to settle to the bottom. Fill up to two thirds of bottle and insert plug and tighten the cap. Keep the filled bottle erect inside the incubator at 45 + 1 °C for 48 h. The sample shall be taken to have passed the test, if on removal from the incubator shows no oil separation or any other phase separation [28,].

Emulsion stability centrifuge test

The cream preparation samples are placed in glass test tubes designed for centrifuge test by Remi equipment. Keep the samples in the centrifuge slot diagonally opposite each other. Put on the centrifuge button on and set at 4000 rpm and run for 30 min put off centrifuge. Take out test tubes and furthermore observations are made and check for separation of cream [33].

Stability at various environmental conditions
SLN samples and related formulations with actives are stable at 45 °C and 75% relative humidity, compared with those kept at room temperature, in accordance with ICH guidelines for stability testing. The average particle size of nanoparticles tends to increase during storage, possibly due to particle aggregation under varying conditions. However, this effect is not observed in formulations kept at 45 °C and 75% RH [34].

Determination of Total fatty matter
Weigh accurately about 2 g of the material into a conical flask, add 25 ml of diluted hydrochloric acid, fit a reflux condenser into the flask, and boil the contents until the solution is perfectly clear. Pour the contents of the flask into a 300 ml separating funnel and allow it to cool to room temperature. Rinse the conical flask with 50 ml of petroleum ether in portions of 10 ml. Pour the petroleum ether rinsing into the separating funnel, shake the separating funnel well and leave until the layers separate. Separate out the aqueous phase and shake it out with 50 ml portions of petroleum twice. Combine all the ether extracts and wash them with water until free of acid (when tested with methyl orange indicator solution). Filter the petroleum extracts through a filter paper containing sodium sulphate into a conical flask which has been previously dried at a temperature of 90 + 2°C and then weighed. Wash the sodium sulphate on the filter with petroleum ether and combine the washing with filtrate. Distil off the petroleum ether and dry the material remaining in the flask at a temperature of 90 +/- 2°C to constant mass [35].

CALCULATION
Total fatty substance, percentage by mass = 100 X M1/M2
Where, Ml = mass in g of the residue, and M2 = mass in g of the material taken for the test.

MICROBIOLOGICAL TEST
Microbiological test has been carrying out by with ref. to IS 11548: 2007 Std. by plate and pour method using laminar air flow [36].

III. RESULTS & DISCUSSION

Phytochemical Screening of Black turmeric extract
Qualitative analysis confirmed the presence of tannins, terpenoids, flavonoids, phenols, phytosterols, and saponins. These compounds are associated with antioxidants, antimicrobial, and anti-inflammatory activities [6]. Similar profiles have been reported in other studies on Curcuma caesia and related species, indicating consistency in phytochemical composition [37, 38].
Instrument Details: GC-MS analysis was performed using an Agilent Technologies 5977A-MSD system equipped with a DB-5MS capillary column (30 m × 0.25 mm, 0.25 µm film thickness). Data acquisition and interpretation were carried out using Mass Hunter software. There were 15 major compounds some of the prominent compounds are listed below

Table 3: Phyto chemical analysis of CCR

Compound	Methanol	Ethanol	Hydro Methanol	Oils
Elemene	1	0.95	0.98	0.7
Cineole	1.25	1.1	1.2	0.8
Borneol	1.85	1.7	1.8	1.2
Curcumin	2.1	2.0	2.05	1.5
Eicosapentaenoic Acid	3.45	3.2	3.3	2.5
Megastigma 3,7(E),9 triene	5.12	4.9	5.0	3.1
Retinal	10.72	9.5	10.0	6.8
ar-Turmerone	10.38	9.8	10.0	8.5
α-Santalol	46.9	42.5	44.0	30.2

Figure 11: Agilent Technologies 5977A-MSD GC-MS system used for phytochemical profiling.

Skin cream base Physico-chemical & Microbial tests

Tabel No 4 – Skin cream formulations Evaluation Results of Cream formulations

Test Parameter	Evaluation Results of Cream formulations					
Code	B1	F1	F2	F3	F4	F5
Black turmeric SLN	0.00	0.50	1.00	2.50	4.00	5.00
Homogeneity	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous
Odour	Pleasant	Pleasant	Pleasant	Pleasant	Pleasant	Pleasant
Spreadability in cm	2.80	2.86	9.95	3.05	3.08	3.10
Moisture volatile content % w/w	80.27	79.76	79.27	78.17	77.88	76.45
pH of 10 % Solution	6.55	6.51	6.5	6.45	6.4	6.38
Viscosity @ 25 °C in cps	30,500	30,800	30,800	31,200	31,400	31,500
Specific Gravity @ 25 °C in cps	0.9875	0.9846	0.9866	0.9887	0.9857	0.9862
Total Fatty Matter % w/w	18.50	19.10	19.22	19.45	19.88	20.54
Thermal Stability at 45 °C for 48 hours	No separation	No separation	No separation	No separation	No separation	No separation
Centrifugal Stability @ 4000 RPM	No separation	No separation	No separation	No separation	No separation	No separation
Microbial test cfu/gm						
Total Viable Count	10 cfu/gm	Nil	Nil	Nil	Nil	Nil

Yeast & Mold	Nil	Nil	Nil	Nil	Nil	Nil
Pathogen	Nil	Nil	Nil	Nil	Nil	Nil

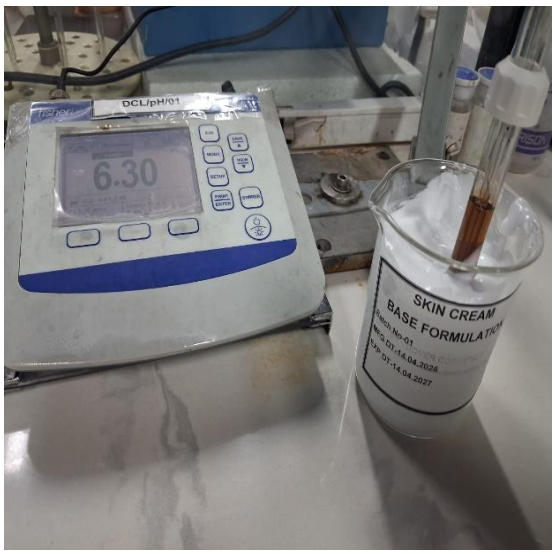

Figure 12: pH test of cream

Figure 13: Centrifuge test – emulsion stability test

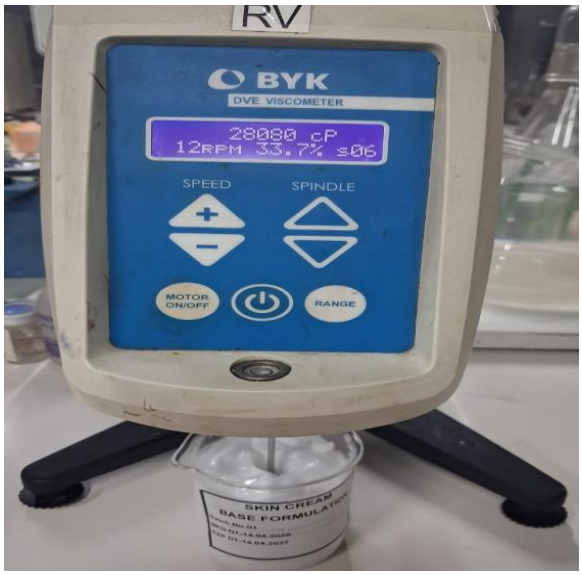

Figure 14: Brookfield viscometer viscosity test

Figure 15: Laminar Airflow: 0.45micron Filter

Figure 16: Thermal stability test

Figure 17: Stability test chambers

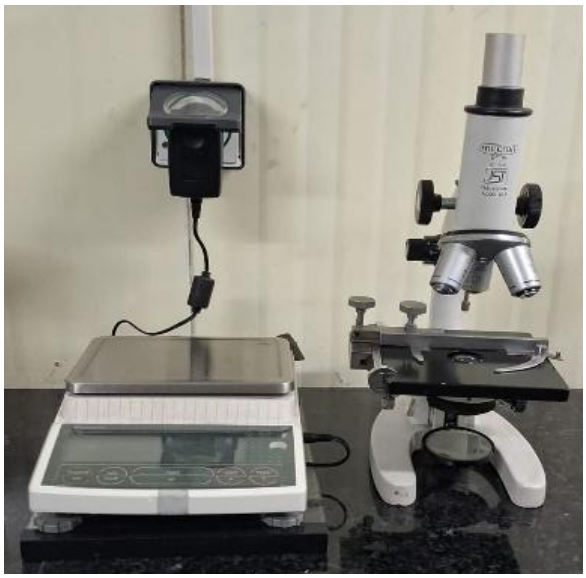

Figure 18: Electronic microscope for micro test

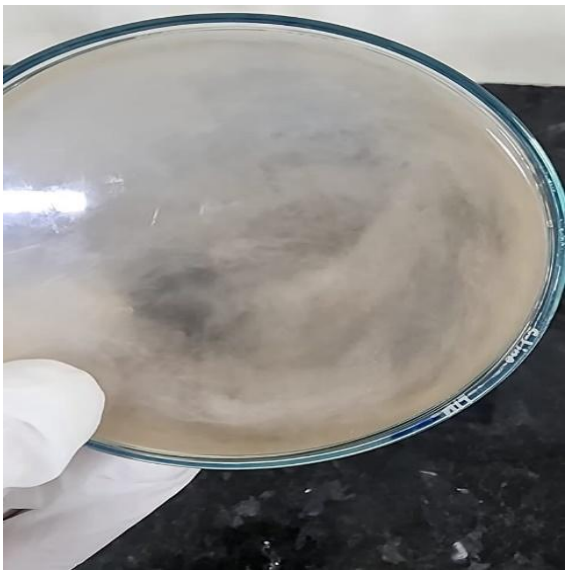

Figure 19: SLN of CCR cream result – nil count

IV. CONCLUSION

The skin cream formulation with SLN curcumin caesia extract is studied for its various properties like appearance, homogeneity, texture and smoothness while application demonstrates that the extraction, formulation and processes are robust to verify benefits of curcumin caesia. It is clear from Physico-chemical, microbial and stability test parameters that the formulation is stable during application, handling and usage of cream at various environmental conditions and efficient to deliver benefits like antiaging, antioxidant, moisturizing properties.

ABBREVIATIONS

CCR	–	Curcumin Caesia Roxb
SLN	–	Solid lipid Nano Particle
TFM	–	Total Fatty Matter
RPM	–	Rotations Per Minute
QS	–	Quantity sufficient

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