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Design and evaluation of vitamin E hair creams

Plackett Burman Screening design with seven variables at two levels

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Abstract : Hair styling and hair grooming is an age old phenomena, forms an integral part of the consumer's need for both men and women. Hair creams improve the dull and dry hairs. Hair creams of oil in water emulsions type are particularly beneficial because when the external aqueous phase is absorbed/evaporated, the emulsion breaks and the remaining oil forms a protective film on the hair shaft provides both protection and nourishment. The vitamin E loaded hair creams were formulated using Plackett Burman screening design, by preparing dummy bases first and finally incorporating vitamin E later. The prepared creams were evaluated for different critical quality attributes. All formulations were found to be white and semisolid in nature. The glossy nature was found to be shiny to very shiny and none of the formulations was dull. Washability was found to be satisfactory and none of the formulations fall in non-washable criteria. The spreadability of all the formulations was in the range of 4.0 to 5.5 cm. Among all, F7 formulation was found to be the best based on high CQA score of 10.7. The pH of the creams was in acceptable range from 5 to 9 and hence all the formulations may be non-irritant to scalp. The viscosity data showed the decrease in the viscosity with increase in RPM, hence hair cream F7 is shear thinning Non-Newtonian system. The *in vitro* free radical scavenging activity of 400 mg hair cream F7 was found to 2.44%. The vitamin E content of the best formulation (F7) was found to be 1.98 gm/100 gm of cream which is 99% of theoretical label claim. In conclusion, the vitamin E loaded hair creams is a better alternative to daily vitamin E dietary supplements.

***IndexTerms* –Hair creams, Vitamin E, CQA scores , Viscosity, pH, drug content, free radical scavenging activity.**

I. INTRODUCTION

In humans, hair has various functions such as protection against external factors, sebum, apocrine sweat and pheromones production and thermoregulation. The hair also plays important roles for the individual's social and sexual interaction [1, 2]. Hair is important for life influences, appearance and self-confidence. The role of hair is important in both men and women for its functions such as protection against UV rays, temperature regulation and sweat evaporation [3]. Hair styling and hair grooming forms an integral part of the consumer's need. Those products which will satisfy all the desirable properties and allow them to enjoy the benefit of the product all the daylong are better option.

Hair creams fulfils all the requirements and improve dull and dry hairs. Creams made by oil in water emulsions are particularly beneficial because when the external aqueous phase is absorbed, the emulsion breaks and the remaining oil forms a protective film on the hair shaft [4]. The hair cream gives a natural-looking shine to the hair. They are also called as styling creams, as they eliminate frizz, smoothen the tamed hair without making them heavy and also hold it in place, so it does not fall out. The hair cream should not only fulfill the styling needs, but should also ensure the hair health. Hair creams are not solid like wax or sticky like gel, but they are in the form that are suitable for all hair types. They are especially useful for curly, long and wavy hairs that get tangled easily. By applying the cream, it strengthens the curls and wavy hairs without weighing them down. Hair creams can be used before and after washing the hairs [5].

Although water in oil emulsions has greasy film, they do not wash-off like oil-in-water emulsion creams. The later are easily removed from hands without soap or shampoo [6]. Hair creams bind to keratin to protect, rejuvenate, thicken, polish and moisturize the hair, forms transparent sheet that reflects light and gives shine to the hair. The emulsion breaks down readily on application to hairs the moisture in this case is absorbed and the oil or fat forms of protective film on the hair shaft greatly minimizing in the damage [7].

Hair loss affects millions worldwide. A healthier scalp leads to healthier hair. Vitamin E combats harmful oxidative stress that can damage hair follicles and lead to hair loss. Its role in improving scalp health by reducing inflammation results in an environment conducive to hair growth. Extra blood flow to the scalp delivers nutrients and oxygen to hair follicles, adding another layer of support for hair growth. Vitamin E may regulate the scalp's oil levels may help in achieving balance, providing necessary hydration and protection without excess oil. Vitamin E may restore its shine and may add life to dull hair. Vitamin E's antioxidant properties shield hair from harmful free radicals that can weaken strands. By doing this, vitamin E may help maintains our hair's structure, leaving it less prone to breaking, stronger, and more resilient [8]. By fighting oxidative stress and

inflammation, Vitamin E may help keep scalp skin healthy. This helps to avoid common scalp issues like dryness, irritation, or flaking which may lead to hair loss or other hair problems.

Hence the present study concentrates on Vitamin E loaded oil water emulsion creams with the excipient selection and composition is designed in such a way that the prepared creams are spreadable, provides gloss and easily washable.

II. EXPERIMENTAL METHODOLOGY:

2.1. FORMULATION DEVELOPMENT:

Formulation of Hair Creams

The emulsifier and other oil-soluble ingredients were dissolved in the oil phase with aid of warming (Phase A). Preservatives and other water-soluble ingredients were dissolved in the aqueous phase (Phase B). The aqueous phase was added to the oil phase with continuous stirring. The prepared dummy bases were mixed with vitamin E to get drug loaded creams. Total 8 different cream based formulations were prepared as shown in Table-5.

2.2. EVALUATION METHODS

2.2.1 Appearance:

The creams were subjected to physical parameters such as description which were judged by naked eye.

2.2.2 MEASUREMENT OF CRITICAL QUALITY ATTRIBUTES:

2.2.2.1 Glossy Nature

Took 1 gm of cream on the glass slide and spread it on glass slide using spreader and observed the slide under sunlight after 1 min. The ranking was given based on the glossy nature as given in table 1

Table 1: Standard scores of glossy nature and washability of cream

Name of Critical quality attribute	Scoring of attributes		
	1	2	3
Gloss	Dull	Shiny	Very shiny
Washability	Not washable	Washable	Easily washable

2.2.2.2 Washability:

Took 1 gm of cream on the glass slide and spread it on glass slide using spreader. The glass slide was placed under running water at distance of 5 cm for 60 seconds with a constant flow rate of 100 ml per minute from tap diameter of 0.5 cm. The scores were given as mentioned in table 1, based on removability of cream from surface in 60 sec i.e nature of cream to get completely washed off from the glass slide.

2.2.2.3 Spreadability

1 gm of cream sample was placed between 2 glass slides of 10x10 cm with thickness of 3mm. Then the cream was allowed spread uniformly. The average of minimum and maximum diameter of cream spread was measured by using ruler. All the values were noted in centimeter scale taken as spreadability score.

2.2.3 MEASUREMENT OF PERFORMANCE CHARACTERISTICS:

2.2.3.1 pH Determination

5 gm of hair cream was dispersed in 45ml of distilled water in 100 ml beaker and the pH was measured using a pH meter [1]. The standard pH should be in the range of 5 to 9.

2.2.3.2 Measurement of Viscosity

Viscosity of formulated creams was determined by using Brookfield viscometer at different RPM using spindle no. LV-64. The graph was plotted between RPM and Viscosity in cps.

2.2.3.3 Drug content estimation

Standard Preparation: The primary stock solution of Vitamin E (5 mg/ml) was prepared in methanol. From the primary solution, pipetted out 1 ml and diluted to 100 ml with 0.1N HCl to get a concentration of 50 µg/ml. This solution was further diluted with 0.1N HCl to get concentrations of 10-50 µg/ml. The absorption scan was taken from 200 nm to 400 nm to find the wave length maxima by using U.V spectrophotometer (Schimadzu). The absorbance of all the dilutions was measured at wave length maxima. The standard graph was plotted by taking concentration (µg/ml) on x-axis and absorbance on Y-axis.

Test preparation: Weighed 1 gm of cream and added 5 ml of methanol and shaken for about 5 min, later added 0.1 N HCl agitated for 5 min. Finally, volume adjusted to 10 ml with 0.1 N HCl. The above solution was further diluted suitably with 0.1 N HCL. The absorbance of this solution was taken with 0.1 N HCl as a blank.

2.2.3.4 Nitric oxide free radical scavenging activity

Principle: Nitrous Oxide (NO) is generated in biological tissue by specific nitric oxide synthesis, which metabolizes arginine to citrulline with a formation of NO via a five electron oxidative reaction. The compound sodium nitroprusside is known to decompose in aqueous solution at physiological pH (7.2) producing NO. Under aerobic condition NO reacts with the oxygen to produce stable products (nitrate and nitrite), the quantity of which can be determined using Griess reagent.

Preparation of reagents:

Phosphate buffer solution pH 7.4: 250 ml of 0.2 M potassium dihydrogen phosphate was added to 394.4 ml of 0.1 M sodium hydroxide and the volume made up to 1000ml with the distilled water.

Potassium dihydrogen phosphate (0.2M) solution: 2.72 grams of potassium dihydrogen solution was dissolved in distilled water and made up the volume to 1000 ml.

Sodium hydroxide solution(0.1M): 0.4 gms of sodium hydroxide was dissolved in distilled water and volume was made up to 1000 ml

Procedure: Nitric oxide(NO) generated from sodium nitroprusside will be measured by Griess reagent. Sodium nitro prusside (5 Mm) in phosphate buffer saline was mixed with 400 mg dummy cream and 400 mg vitamin E loaded cream separately and incubated at 25^0C for 150 minutes (Test Preparations). The samples were allowed to react with the Griess reagent (1% sulphanilamide and 0.1% naphthyl ethylene diamine dihydrochloride in 2% H_3P0_4). The absorbance was measured at 546 nm. A similar procedure was repeated with solvent water instead of cream, which served as the control. All the tests were performed in triplicate. The percentage of scavenging activity was measured using the formula.

% Inhibition of free radicals: $\frac{control-test}{control} * 100$

III. RESULTS AND DISCUSSION:

3.1. Formulation development:

Different excipients were selected for the preparation of oil in water emulsion creams to meet purpose. The list of excpients in both phases along with their purpose was shown in table-2.

Table 2: The list materials used in formulations with their function

Name of material	Function
Oil phase	
Stearic acid	Texture enhancer
Cetyl alcohol	Emulsion Stabilizer
Mineral oil	Oil phase
Bees wax	Emulsifying base
BHT	Antioxidant
Propyl paraben	Preservative
Span 60	Surfactant
Water phase	
Triethanolamine	Balance pH
Propylene glycol	Moisturizer
Glycerin	Humectant
Methyl paraben	Preservative
Drug	
Vitamin E, gm	Antioxidant

The formulations were prepared by using Plackett Burman screening design with total number of variables restricted to seven at two levels. Hence the total number of experiments with this screening design was eight. The concentration of excipient to be screened was taken as independent variables and critical quality attributes of hair creams were taken as dependent variables. The list of independent variables with their levels was shown in Table-3

Table 3: List of independent variables with their levels

Name of Independent variable (concentration of excipients)	Levels of variable	
	- (low)	+ (High)
X1= Con. of stearic acid, %	1	5
X2= Con. of cetyl alcohol, %	1	3
X3= Con. of paraffin oil, %	2	7
X4= Con. of bees wax, %	0	1
X5= Con. of triethanolamine, %	0.5	1
X6= Con. of propylene glycol, %	0	2
X7= Con. of glycerine, %	1	5

SigmaTech soft ware was used for the design of experimentation and subsequent analysis of impact of each independent variable on quality of formulations. The design of experimentation (DOE) was depicted in Table-4.

Table 4: Plackett Burman design of experimentation,

Name of excipient	Excipient concentration, %							
	F1	F2	F3	F4	F5	F6	F7	F8
Stearic acid	5	5	5	1	5	1	1	1
Cetyl alcohol	3	3	1	3	1	1	3	1
Paraffin oil	7	2	7	2	2	7	7	2
Bees wax	0	1	0	0	1	1	1	0
Triethanolamine	1	0.5	0.5	1	1	1	0.5	0.5
Propylene glycol	0	0	2	2	2	0	2	0
Glycerine	1	5	5	5	1	5	1	1

The experimental runs were taken based on DOE suggested by SigmaTech software and the concentration of remaining excipents was maintained as constant. The composition of hair cream formulation was reported table-5.

Table 5: Composition of hair creams, gm

Ingredients	F1	F2	F3	F4	F5	F6	F7	F8
Oil phase								
Stearic acid	5	5	5	1	5	1	1	1
Cetyl alcohol	3	3	1	3	1	1	3	1
Mineral oil	7	2	7	2	2	7	7	2
Bees wax	-	1	-	-	1	1	1	-
Span 60	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
Propyl paraben	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
BHT	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Water phase								

Triethanolamine	1	0.5	0.5	1	1	1	0.5	0.5
Propyleneglycol	-	-	2	2	2	-	2	-
Glycerin	1	5	5	5	1	5	1	1
Methyl paraben	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Water, Quantity sufficient to 100	q.s	q.s	q.s	q.s	q.s	q.s	q.s	qs
Drug loading								
Vitamin E	2	2	2	2	2	2	2	2

3.2 EVALUATION OF HAIR CREAMS

3.2.1 VITAMIN-E STANDARD PLOT:

The calibration of vitamin E was performed by measuring the absorbance of vitamin E solutions in 0.1N HCl using UV spectrophotometer at 298nm. Standard curve was plotted between concentration and absorbance as in Fig.1.

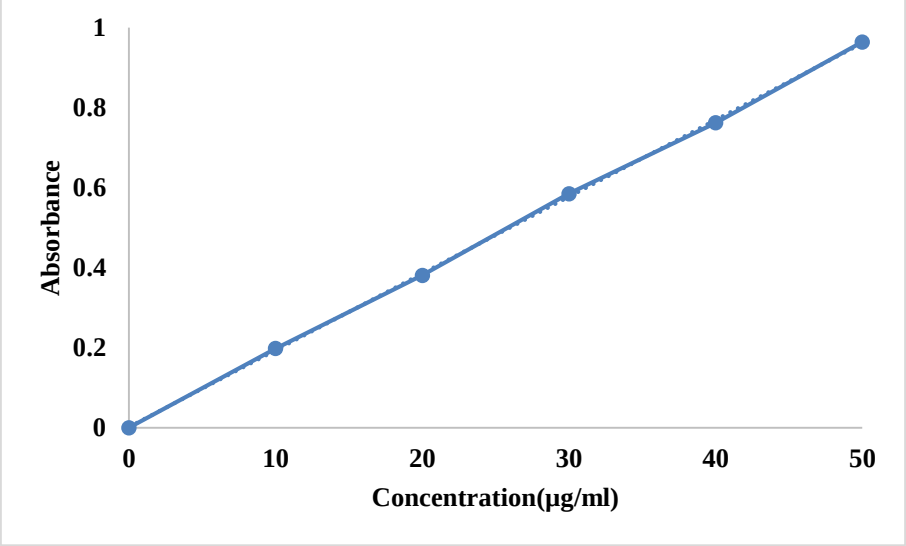

Figure 1: Standard plot of vitamin E at 298 nm

The graph obeyed Beers Lombard law in concentration range of 10-50 µg/ml with R^2 value of 0.9998 at wave length maxima of vitamin E found to be 298 nm. The obtained regression equation is y = 0.0192x + 0.002.

3.2.2 APPEARANCE:

All formulations were in semi-solid nature with snow white shining appearance.

3.2.3 CRITICAL QUALITY ATTRIBUTE (CQA) DATA

The performance characteristics were evaluated and critical quality attribute data was collected based on the standard score criteria as per Table-1. The CQA data of gloss, washability and spreadablity was presented in table 6.

Table 6: CQA score data of hair cream of different formulations F1-F8

FORMULAION CODE	GLOSSY NATURE	WASHABILITY	SPREDABILIY, cm	TOTAL CQA SCORE
F1	2	2	4.2	8.2
F2	2	3	4.3	9.3
F3	2	2	4.4	8.4
F4	2	2	4.5	8.5
F5	2	2	5	9
F6	2	2	4.5	8.5
F7	**3**	**3**	**4.7**	**10.7**
F8	3	2	5.2	10.2

The data presented in table 6 showed that all formulation prepared were showing satisfactory scores in range of 8.2 to 10.7. Among all formulations, F7 formulation was found to be the best formulation with CQA score of 10.7.

3.2.3.1 Gloss nature:

The glossy nature data was used to screen the excipient's role on gloss induced by formulation. It was indicated that all excipients were against the gloss after application except paraffin oil and bees wax as per the coefficient data shown in Fig. 2.

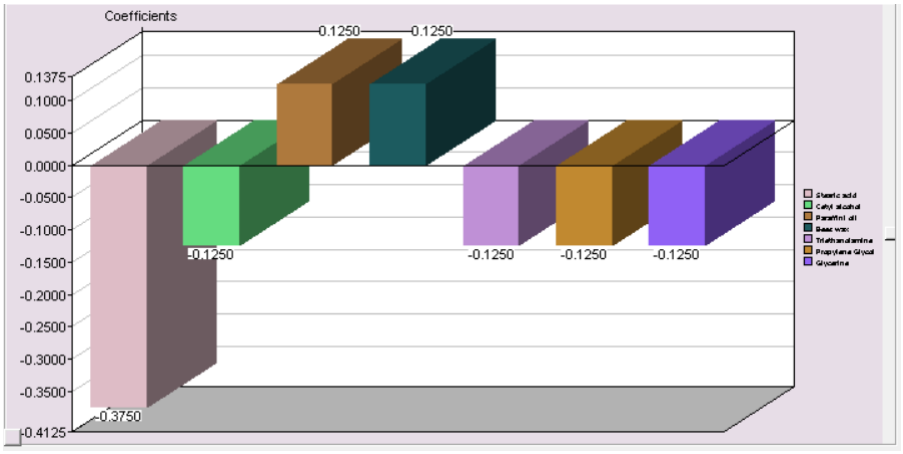

Figure 2: Comparison of Coefficient data of gloss

3.2.3.2 Spreadability:

There was a positive impact on spreadability shown by bees wax and propylene glycol. But their impact was less than other excipient's negative role on spreadability. The same comparative coefficient data was represented as bar graph in Fig. 3.

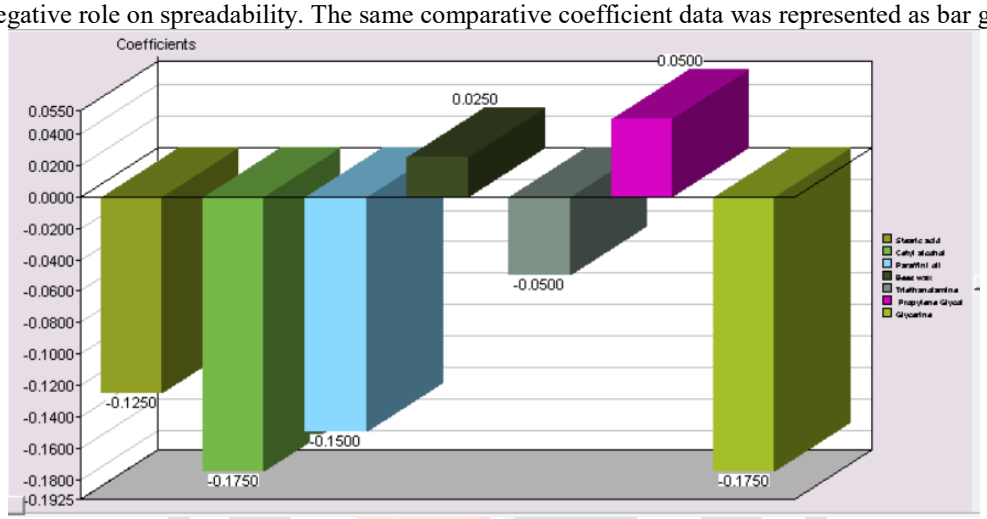

Figure 3: Comparative coefficient data of spreadability

3.2.3.3 Washability:

The rinsing ability of cetyl alcohol and beeswax was more among all excipients in combination screened. The excipient like stearic acid, paraffin oil, propylene glycol and glycerine shown nil contribution at concentrations used in the experimental design. The washability coefficient data was shown in Fig.4.

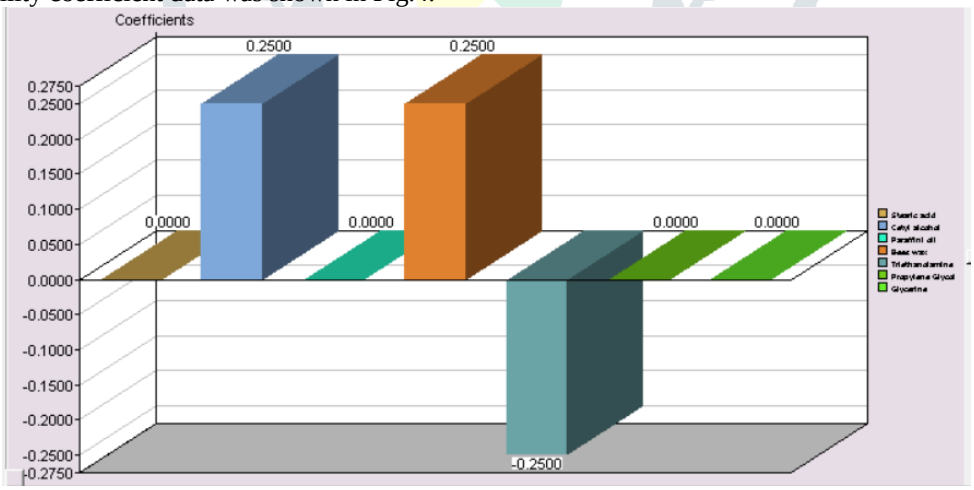

Figure 4: Comparative coefficient data of washability

Even though all components did not impact on all quality parameters equally, the excipients screened in combination had shown satisfactory results. As the oil in water creams are less greasy and leave hair damp after application because water is a continuous phase. The spreadable and rinsable texture allows a thin and even layer of oil to be distributed evenly. A continuous film of residual oils and wax form, which adds shine. Hair creams are non-greasy and non-sticky when applied to the hairs and provide nourishment, whereas, hair oils alone containing vitamin E can provide nourishment, but also makes hair greasy and sticky.

3.2.4 pH:

The data of pH showed that all the formulations are in the acceptable range of 5-9. This could be possible with usage of very low concentration of tri-ethanolamine in the range 0.5-1%. The extreme pH below 5 and above 9 considered to be irritating to scalp and damages hair. The pH data of all formulations were mentioned in table 7.

Table 7: pH data formulations F1 to F8

Formulation code	pH
F1	7.85
F2	7.49
F3	7.83
F4	8.02
F5	7.16
F6	8.11
F7	7.80
F8	7.94

3.2.5 VISCOSITY:

As CQA score data of formulation F7 was high, viscosity was determined using Brookfield viscometer at different RPM using spindle LV64 to find the nature of cream upon varying stress values. The data of viscosity of formulation F7 was reported in table 8.

Table 8: Values of viscosity at different RPMs for formulation, F7

S. No	RPM	VISCOSITY, cps
1.	100	1800
2	60	2400
3	50	3000
4	30	4060
5	20	5000
6	10	8500
7	6	11000
8	5	14000
9	4	16000
10	3	20000

The above data showed that, as RPM increases the viscosity decreases, hence this formulation F7 behaving as shear thinning system without any plug flow. This might be the reason for spreadability of cream. The same data was picturized in Fig.5 with curvilinear relationship.

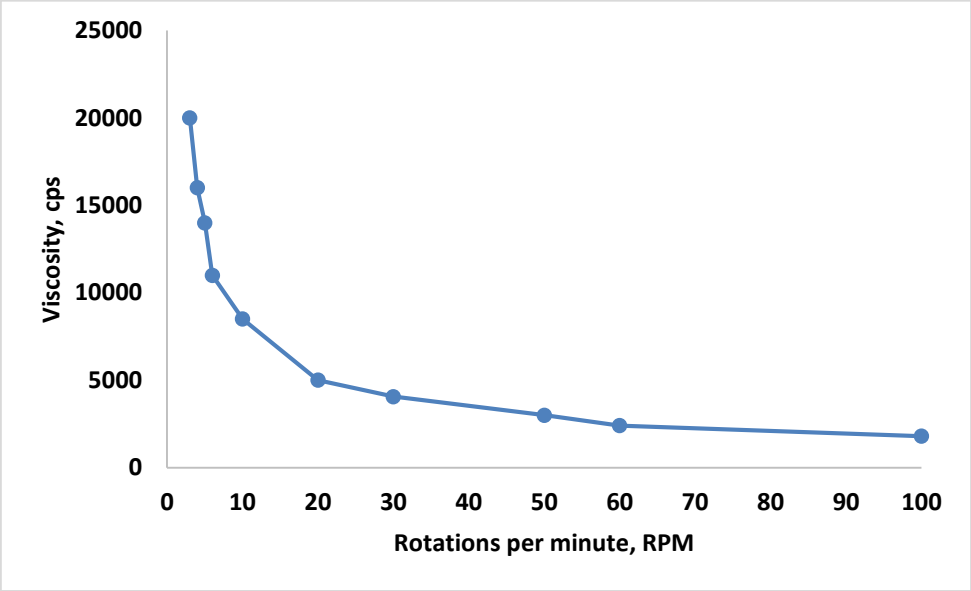

Figure 5: Rheogram of hair cream F7

3.2.6 DRUG CONTENT

The drug content of the formulation (F7) was studied to find the amount of drug in cream. The vitamin E content in cream was found to be 1.98 gm/100gm of cream. This is 99% of theoretical content of labeled claim (2% Vitamin E hair cream).

3.2.7 FREE RADICAL SCAVENGING ACTIVITY:

The free radical scavenging activity is generally estimated for pure drugs. But to find the effect of cream base on Vitamin E functionality, this test was performed. For this purpose, 400 mg of formulation F7 was used and free radical inhibition was calculated. The free radical inhibition activity of dummy cream was zero and cream formulation F7 was fund to be 2.44 %.

Conclusion: Vitamin E is a powerful antioxidant with potential benefits for hair health. It may avoid hair loss, improve scalp circulation, and add shine to hair. Incorporating vitamin E into hair care routine can be done through dietary supplements, but topical application via cream mode is the best option for daily usage.

IV. ACKNOWLEDGMENT

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