

## FORMULATION AND EVALUATION OF HERBAL ANTIDANDRUFF GEL

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### ABSTRACT

**Objective:-** The present study was aimed to design and develop antidandruff herbal gel formulation containing *Trigonella foenum graecum* (Methi ) seeds extract. *Trigonella foenum graecum* as herbal drug is selected on the basis of literature review, traditional claim and clinical evidences for antidandruff activity. **Methods:-** The seed extract of *Trigonella foenum graecum* were obtained from K. Patel phyto extraction PVT.LTD (KP/TF/017/15). Then performed systemic investigation by physicochemical evaluation of extract. Gel were prepared using carbapol ultraz 20 polymer as gelling agent. Prepared formulation were evaluated for various physicochemical properties. To

developed and validation of HPTLC method for estimation of 4 hydroxyisoleucine from extract as well as formulation. Based on in vivo dermal irritation studies using to draize scoring method on rat for antidandruff activity. **Result:-** The prepared formulation of *Trigonella foenum graecum*, gel prepared with carbapolultraz 20 showed significant reduction of dandruff on rat when compared to the control topicalbased group after 48 hr. The dermal irritation test result showed after 48 hr from removal of antidandruff methi hair gel on rat. None of erythema and none of oedema on rat. comparative HPTLC of 4 hydroxyisoleucine with placebo, extract and formulation was observed at RF 0.40. **Conclusion:-** Antidandruff methi hair gel fully standardized. Validated HPTLC method can be used for estimation of 4 hydroxyisoleucine from extract as well as formulation respectively as per ICH guidelines. This method can be used for the analysis of many marketed formulations and can be effective to avoid dandruff in hair.

**KEYWORDS:-** Antidandruff, Hair gel, Dermal Irritation, 4 hydroxyisoleucine.

## INTRODUCTION

Ayurveda is the oldest traditional alternative medicinal system of the India these systems gives the medicine of natural origin plant such as plant, shrub and herbs. The plant use as a medicine in the various forms such as leaf, powder, bhasma, etc. In the modern era of science the Ayurvedic formulations are prepared with modern technique with more accuracy, safety and effectiveness by applying modern technique such as extraction, mechanical stirring, mechanical shaking etc and different software technique. Topical drug delivery systems are gaining increased in popularity, and several drugs have been successfully delivered by this route for both local and systemic action. Gels have better potential as a vehicle to administer drug topically in comparison to the ointment because they are non-sticky, requires low energy during formulation.

**Dandruff:** It is the common problem in the majority of the population it cause hair fall and hair loss due to which the appearance of the human is affected so there is a need of antidandruff gelformulations. Drug delivery through the skin has been a promising concept for a long time because the skin is easy to access, has a large surface area with vast exposure to the circulatory and lymphatic networks and the route is non-invasive. Gel consists of a natural or synthetic polymer forming a three-dimensional matrix throughout a dispersion medium or hydrophilic liquid. After application, the liquid evaporates leaving the drug entrapped in a thin film of the gel-forming matrix physically covering the skin. The presence of a network formed by the interlocking of particles of the gelling agent gives rise to the rigidity of a gel. The nature of the particles and the type of form. that is responsible for the linkages determine the structure of the network and the property of the gel. The available anti-inflammatory drugs (steroidal and non-steroidal) present a wide range of side effects. Therefore, many studies are being directed to find anti-inflammatory agents from natural sources. Fenugreek (*Trigonella foenum-graecum*; Fabaceae) is a plant whose seeds and leaves are used in traditional medicine. Fenugreek act as powerful antioxidant mainly due to the presence of flavonoids and polyphenols. Fenugreek has been reported to possess anti-inflammatory activity mainly due to the presence of flavonoids because flavonoids act as antioxidant and potential inhibitors of cyclooxygenase, lipoxygenase, and nitric oxide synthase. Even though fenugreek has been used for the treatment of inflammation, no report exists on the development of gel formulations from an extract of fenugreek. Hence, the present study is aimed at formulation and evaluation of herbal antidandruff gel from the ethanolic extract of Fenugreek.

## MATERIALS AND METHODS

Methi extract (*Trigonella foenum-graecum*) was obtained from K. Patel Phyto Extractions PVT. LTD (KP/TF/017/15), 4 hydroxyisoleucine was obtained from natural remedies. Carbopol Ultraz 20 Polymer, Disodium EDTA, Sodium Propyl Paraben, Sodium Methyl Paraben, Propylene glycol were obtained from Research –Lab Fine Chem Industries (Mumbai). Potassium hydroxide were obtained from S.D Fine Chemical Ltd., Mumbai.

### Methods

#### Preparation of herbal hair gel formulation

Six different herbal hair gel formulations were prepared by simple gel formulation preparation method. The seeds of *Trigonella foenum graecum* extract were obtained from K.Patel Phyto Extractions PVT. LTD (KP/TF/017/15). The gel was prepared by using *Trigonella foenum graecum* seed extract in which carbopol ultraz 20 polymer is used as base or gelling agent and other excipients such as sodium methyl paraben as preservative, propylene glycol as humectant, Disodium EDTA as chelating agent and Potassium hydroxide for adjustment of PH. Add 0.50 ml Carbopol ultraz 20 and measured quantity of extracts was dispersed in up to 100 ml of distilled water as solvent and mixed by continuously stirring by using magnetic stirrer at 1000 rpm for 1 hour. Propylene glycol 4 ml was added in to the mixture under continuous stirring. 0.18 ml of sodium methyl paraben and 0.02ml of sodium propyl paraben and 0.10ml of disodium EDTA also add in mixture. the mixture was neutralized by drop wise addition of 1ml potassium hydroxide. Mixing was continued until a transparent gel was formed.

#### Physicochemical evaluation of extract

Standardized extract were evaluated for various physicochemical parameters such as moisture content, Ph, Water soluble extract value, Alcohol soluble extractive value, Total ash, Acid insoluble ash, Total saponin assay. Results shown in table no 1.

#### Evaluation of gel formulations

Prepared formulations were evaluated for various physicochemical parameters such as Appearance, colour, pH, viscosity, homogeneity, spreadability, washability and results are shown in table 2.

#### Physical appearance

The gel formulation was evaluated in terms of physical character like colour, odour.

**Measurement of pH**

1 gm of gel formulation was dispersed separately in 100 ml of water, and the pH of the gel formulation was determined using digital pH meter. Measurements of pH of all formulations were carried out in triplicate and the averages of three readings were noted.

**Measurement of viscosity**

The viscosity of gel was determined by using a Brookfield viscometer DVII model with a T-Bar spindle in combination with a helipath stand. 25 g of gel was filled in a 50 ml beaker. T-bar spindle (T95) was used for the measurement of viscosity of all the gels. The helipath T-bar spindle was moved up and down and viscosity was measured at 2, 4, 5 and 10 rpm.

**Homogeneity**

Formulations were tested for homogeneity by visual inspection after the formulations have been set in the container. They were tested for their appearance and presence of any aggregates and flocculate.

**Spreadability**

The weighed quantity of gel 2 g was sandwiched between two glass slides 500 g of weight was placed on the slides. The weight was placed for specific period of time for 5 min. Then weight was removed and diameter of the spread circle was measured at different points. A shorter interval indicates better spreadability. Spreadability was calculated using the following formula:

$$S = M \times L/T$$

Where, S = Spreadability

M = Weight in the pan (tied to the upper slide), L = length of glass slide

T = Time (in sec.) taken to separate the slide completely each other

**Washability**

The prepared hair gel formulation is applied on the skin and then ease and extent of washing with water is checked normally.

**Dermal irritation study of topical hair gel formulations**

This test was performed on albino rats weighing between 150-200g. The animals were given standard animal feed and had free access to water ad libitum. Dorsal hairs at the back of the rats were removed one day prior to the commencement of the study and kept individually in

cages to avoid contact with the other rats. Two groups of each were used for control (plain gel base) and standard irritant. Other group was used as a test. The 50 mg of the formulation were applied over one square centimeter area of whole and abraded skin of different animals. Aqueous solution of 0.8% formalin was used as standard irritant. The animals were observed for 48 hr for any signs of edema and erythema. Results are shown in figure no 1.

### **Chromatographic evaluation by HPTLC**

HPTLC is an important implement that can be used qualitatively as well as quantitatively for checking the purity and identification of crude drugs and also for quality control of finished products. The results of HPTLC chromatographic conditions are shown in table no 3 and figure no 2 and 3 depicts comparative HPTLC of 4 Hydroxyisoleucine with placebo, extract and formulation.

### **Method of validation by HPTLC Linearity**

The standard solution was prepared by dissolving 10 mg 4 hydroxyisoleucine in 10 ml methanol solution which gives 1000 µg/ml. The working standard of 100 µg/ml was prepared from standard solution by dilution with methanol. Different concentration of 2,5,10,15,20 µg/ml were prepared from standard solution. Linearity graph for standard 4 Hydroxyisoleucine shown in table no 4 and calibration curve shown in figure 4.

### **Precision**

#### **Intra-day precision**

Repeatability of sample application and measurement of peak area was carried out using the three replicates of same spot. Repeatability is also termed as intra day precision.

#### **Inter-mediate precision**

Interday precision was evaluated by analysis of replicate applications of standard solution of same concentration on three different day.

### **Recovery study**

Recovery study was performed by spiking 80,100,120% of standard drug externally to the preanalyzed sample. The experiment was conducted in triplicated and applied onto the plate in duplicate and run. This was conducted to check the recovery of drugs at different levels of formulation results shown in table no 5.

### Robustness

The robustness of method can be performed by allowing variation in sample application time to scanning. Robustness parameters, %RSD results shown in table no 6

## RESULT

### 1. Physicochemical parameters of extract

**Table 1: Results of physicochemical parameters of extract.**

| Parameters                       | Specification | Result |
|----------------------------------|---------------|--------|
| Moisture content                 | Nmt 8%        | 4.93%  |
| Ph                               | 3.0-6.0       | 5.35%  |
| Water soluble extractive Value   | Nlt 80.0%     | 81.68% |
| Alcohol soluble extractive Value | Nlt 70.0%     | 76.59% |
| Total ash                        | Nmt 10.0%     | 7.50%  |
| Acid insoluble ash               | Nmt 5.0%      | 1.24%  |
| Total saponin assay              | Nlt 40.0%     | 50.89% |

### 2. Evaluation of antidandruff methi hair gel formulation

**Table 2: Results of evaluation of antidandruff methi hair gel formulation.**

| Parameter           | Specification                                                     | Observation     |
|---------------------|-------------------------------------------------------------------|-----------------|
| Physical Appearance | Light yellow to yellow coloured Gel with characteristic fragrance | Complies        |
| Ph                  | 4.5 – 5.5                                                         | 4.928           |
| Viscosity           | Nlt 20000                                                         | 24,000          |
| Homogeneity         | Homogeneous                                                       | Complies        |
| Spreadability       | Satisfactory                                                      | Satisfactory    |
| Washability         | Washable                                                          | Easily washable |

### 3. Dermal irritation study



**Apply Antidandruff Methi Hair Gel**



**After Removal of Antidandruff Methi Hair Gel (48 Hr)**

**Erythema scale:** 0- none; 1-slight; 2- welldefined; 3-moderate; and 4- scar formation

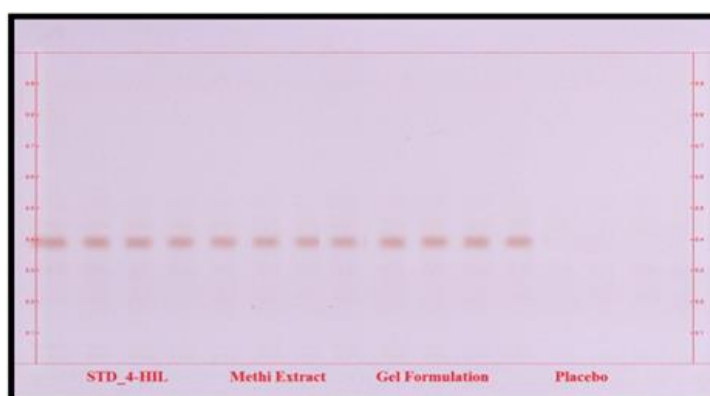
**Oedema scale:** 0- none; 1- slight; 2- welldefined; 3- moderate; and 4- severe.

**Figure 1: Photographs of dermal irritation test for antidandruff methi hair gel.**

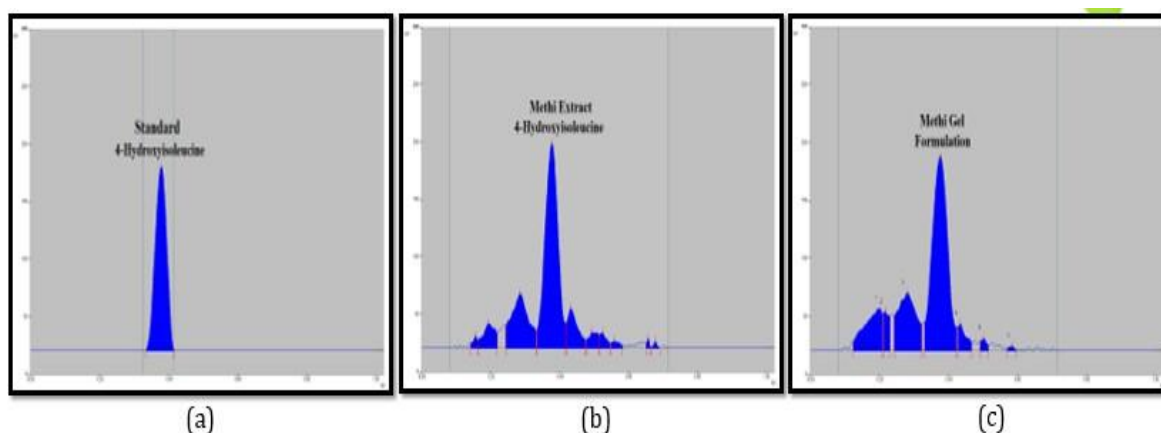
#### 4. HPTLC Chromatographic Conditions

**Table 3: Optimum HPTLC Chromatographic Conditions.**

| Parameters          | Specifications                                      |
|---------------------|-----------------------------------------------------|
| Stationary phase    | Aluminium plate precoated with silica gel (Merck)   |
| Diluent             | Water: Methanol (50 : 50)                           |
| Mobile phase        | n-Butanol: Glacial acetic acid: Water [7:2:1 (v/v)] |
| Mode of application | Band                                                |
| Sample volume       | 10 µl                                               |
| Sonication time     | 30 minutes                                          |
| Saturation time     | 30 minutes                                          |
| Spraying reagent    | Ninhydrin Reagent                                   |
| Scanning wavelength | 550 nm                                              |



**Figure 2: Comparative HPTLC Photograph of 4-Hydroxyisoleucine with Placebo, Extract and Formulation.**



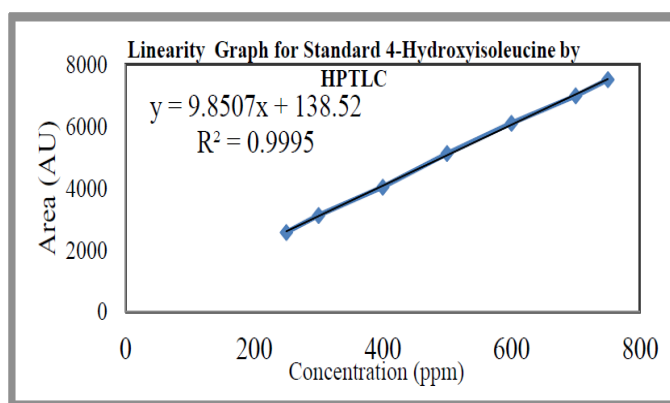
**Figure 3: HPTLC Densitogram (a)Standard 4-Hydroxyisoleucine Peak; (b) Methi ExtractPeak; (c) Methi Gel Formulation Peak.**

## Results of validation parameters

### 1. Linearity

Table 4 and Figure 4: Linearity Graph for Standard 4-Hydroxyisoleucine by HPTLC.

| Conc. of 4-Hydroxyisoleucine (ppm)       | Avg. Peak area of 4-Hydroxyisoleucine |
|------------------------------------------|---------------------------------------|
| 250                                      | 2568                                  |
| 300                                      | 3120                                  |
| 400                                      | 4036                                  |
| 475                                      | 5120                                  |
| 600                                      | 6102                                  |
| 700                                      | 6989                                  |
| 750                                      | 7512                                  |
| (Correlation coefficient) r <sup>2</sup> | 0.9995                                |



### 2. Precision and % Recovery of Extract and Formulation

Table 5: Precision and % Recovery of Extract and Formulation

| Parameters                  |      | Extract | Formulation |
|-----------------------------|------|---------|-------------|
| Intra-day Precision (% RSD) |      | 1.31    | 1.19        |
| Inter-day Precision (% RSD) |      | 1.51    | 1.05        |
| % Recovery                  | 80%  | 99.13   | 99.25       |
|                             | 100% | 99.09   | 99.52       |
|                             | 120% | 98.46   | 99.51       |

### 3. Robustness Parameter For HPTLC

Table 6: Robustness Parameter For HPTLC.

| Robustness parameter studied |      | % RSD |
|------------------------------|------|-------|
| Composition of mobile phase  | +0.2 | 0.41  |
|                              | -0.2 | 0.70  |
| Volume of mobile phase       | +5   | 1.51  |
|                              | -5   | 0.87  |
| Saturation Time              | +5   | 1.19  |
|                              | -5   | 1.62  |

## CONCLUSION

Antidandruff Methi Hair Gel - fully standardized. Analytical method are very robust, simple and accurate.

Validated HPTLC method can be used for estimation of 4-Hydroxyisoleucine from extractas well as formulation respectively as per ICH guidelines.

This method can be used for the analysis of many marketed formulations.

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