

# Preparation and Characterization of *M. × Piperita* L. Oil Based Gel Formulation

Peeyush Kumar, Sapna Mishra, and Anushree Malik, Santosh Satya

**Abstract**—The essential oil of *M. × piperita* L. was formulated into a topical gel. The prepared gel was characterized for its pH, viscosity, spreadability, consistency and extrudibility, while its stability was evaluated under different temperature conditions. The prepared *M. × piperita* oil gel was clear and transparent. The pH value of developed gel was 6.6, while its viscosity was 1200 cP. Spreadability and consistency of the *M. × piperita* oil gel was 10.7 g.cm/sec and 7 mm, respectively. The prepared gel showed good extrudibility. During the stability studies, no significant change in pH and viscosity as a function of time for gel was observed, indicating stability of prepared formulation. The gel developed in this study is expected to forward the usage of *M. × piperita* essential towards commercial application.

**Keywords**—*M. × piperita* L., formulation, gel, characterization, stability

## I. INTRODUCTION

PLANT essential oils and their components are known for their various biological activities; such as, flavouring agent for food, pharmaceuticals, antimicrobial and fungicidal [1, 2]. Even today, about 80% of the world's population relies predominantly on plants and plant extracts for health care [3]. Particularly, in recent years, essential oils and their components are gaining increasing interest due to being relatively safe for the environment as well as to the human health, their wide acceptance by consumers, and their exploitation for potential multi-purpose functional use [4].

In spite of a large body of literature, the general applicability of essential oils on commercial scale is lacking. The reasons for this could be; relatively slow action, variable efficacy, lack of persistence, inconsistent availability and lack of suitable /user friendly end product [5]. It is evident that formulation development could increase the usability, efficacy and storability of the essential oil or active ingredient. However, few studies have ventured towards development of essential oil based formulations. Manou et al. [6] prepared a cream formulation of thyme oil to be used in a topical application as a preservative against bacterial and yeast strains. Similarly, Orafidiya et al. [7] developed effective antibacterial formulation for topical usage with the essential

P.K. is Research Scholar with Centre for Rural Development & Technology, Indian Institute of Technology Delhi, New Delhi- 110 016, India (phone: +91-11-26591158; fax: +91-11-26591121; e-mail: peeyush0275@yahoo.co.in).

S.M. is Research Scholar with Centre for Rural Development & Technology, Indian Institute of Technology Delhi, New Delhi- 110 016, India (e-mail: sapz07@gmail.com).

A.M. is Associate Professor with Centre for Rural Development & Technology, Indian Institute of Technology Delhi, New Delhi- 110 016, India (e-mail: anushree\_malik@yahoo.com).

S.S. Professor with Centre for Rural Development & Technology, Indian Institute of Technology Delhi, New Delhi- 110 016, India (e-mail: ssatya@rdat.iitd.ac.in).

oil *Ocimum gratissimum*. While Ointment and cream formulations of lemongrass oil was prepared for mosquitocidal repellency [8].

Gels are semisolid systems in which the movement of dispersion medium is restricted by interlacing three dimensional network of particles or solvated macromolecules of dispersed phase [9]. Twisted matted strands in gels are tied together by vander waals forces to form crystalline and amorphous regions throughout the system. Interlacing network of the particles and consequential internal friction leads to increase in viscosity, which is responsible for the semisolid state of the system. Concentration of gel is usually between 0.5 to 2%, and gels maintain their viscosity over a wide range of temperature. The use of gel as a delivery system can increase the residence time of drugs on the skin and, consequently, enhance bioavailability [10]. Gel delivery systems have several advantages such as the ease of administration, high residence time on the skin and better drug release and diffusion [10, 11].

In the present study, gel formulation based on *M. × piperita* essential oil was attempted. The essential oil of *M. × piperita* has earlier been reported for its pesticidal, antibacterial, antifungal, and anti-cancerous properties [3, 12-14]. The gel formulation of *M. × piperita* oil was characterized for pH, viscosity, spreadability, consistency and extrudibility, while its stability was evaluated under different temperature conditions.

## II. MATERIALS AND METHODS

### A. Materials

The essential oil of *M. × piperita* was purchased from Kanta Chemicals Pvt. Ltd, Khari Bowli, New Delhi, India and stored in plastic bottles at 4 °C.

### B. Preparation

Preparation of gel formulation of *M. × piperita* oil was a two step process. Firstly (1<sup>st</sup> step), an aqueous mixture of water (67%), surfactant (20%) and co-surfactant (4%) was prepared. To this mixture 8% of active ingredient was added drop wise along with constant agitation on magnetic stirrer. Gelatin (1%) was added for viscosity enhancement.

### C. Characterization of Gel

The gel was characterized for pH, viscosity, spreadability, consistency and extrudibility through standard methods:

#### i. pH

The pH values of the gel was measured by a digital type pH meter (HI 2212 pH meter, Hannah instruments, USA) by dipping the glass electrode into the sample. The reaction was performed at room temperature (25 ± 2 °C). The measurements were performed in triplicate.

### ii. Viscosity

Viscosity of the *M. × piperita* gel was measured using a Brookfield DV-III programmable cone and plate rheometer (Brookfield, USA) fitted with a CP-42 cone spindle. The jacketed sample cup was connected to a circulating water bath operating at 25 °C and a shear rate of 60.0 s<sup>-1</sup>. A sample volume of 1 mL was used. The measurements were performed in triplicate.

### iii. Spreadability

Spreadability is a term expressed to denote the extent of area to which the cell readily spreads on application to the affected area. The spreadability of gel was evaluated through extensometer set up, which consists of two glasses [15]. The gel sample (0.25 gm) was placed on lower immovable glass plate. The upper glass plate (movable) was placed on the top of the sample. Force was generated by adding known weight (50 gm) on the upper glass plate. The experiment was repeated thrice at constant temperature and exerted weight and the mean values of spread surface area on the lower plate was calculated by using the formula.

$$S = \frac{M \times L}{T}$$

Where M= wt. tied to upper slide, L = length of glass slides, T= time taken to separate the slides

### iv. Consistency

The measurement of consistency of the prepared gels was done by dropping a cone attached to a holding rod from a fix distance of 10 cm in such way that it should fall on the centre of the glass cup filled with the gel. The penetration by the cone was measured from the surface of the gel to the tip of the cone inside the gel [16]. The distance traveled by cone was noted down after 10sec.

### v. Extrudibility

Extrudibility reflects the capacity of the gel, to get ejected in uniform and desired quantity when the tube is squeezed. For the analysis of gel extrudibility, 20g of gel was filled into collapsible tubes and pressed firmly at the crimped end to extrude the gel until the pressure was dissipated [17].

### C. Stability of Gel

The formulated *M. × piperita* oil gel was filled in the collapsible tubes and stored at 5 different temperature condition; 20±2 °C, 25±2 °C, 30±2 °C, 35 ± 2 °C and 40 ± 2 °C, for a period of three months and studied for appearance, pH, viscosity, consistency, spreadability and extrudability [18].

## III. RESULTS

Gels are defined as a substantially dilute cross-linked system. This crosslinks within the fluid are responsible for gel structure (hardness) and contribute stickiness. In this way gels are a dispersion of molecules of a liquid within a solid in which the solid is the continuous phase and the liquid is the discontinuous phase. By weight, gels are mostly liquid, yet they behave like solids due to a three-dimensional cross-linked network within the liquid. Gels are typically formed from a liquid phase that has been thickened with other components, where continuous liquid phase allows free diffusion of molecules through the polymers scaffold [15].

### A. Characterization of Gel

The prepared gel was clear and transparent. Table I represents all the properties of gel. The pH value of developed gel was 6.6. For a gel, the range of pH between 5-7 is considered good, as below 5 the solubility of active ingredient decreases [17]. Viscosity of the prepared gel was 1200 cP. Viscosity of gel was reported to be significantly affected by crosslinking of the inherent components, as well as concentrations and molecular weight of the active ingredient.

TABLE I  
PHYSICOCHEMICAL PARAMETERS OF THE *M. × PIPERITA* OIL GEL

Properties	Values
Spreadability	10.7 g.cm/sec
pH	6.6
Extrudibility	Good
Consistency	7 mm

The values of spreadability indicate that the gel prepared in the present study is easily spreadable by small amount of shear. Spreadability of the *M. × piperita* oil gel was 10.7 g.cm/sec, which was more than that reported by Shivhare et al. [15] for polyacrylamide gel containing diclofenac sodium (6.5 g.cm/sec). Bhanu et al [17] reported spreadability between 10.03-22.89 g.cm/sec for diclofenac Sodium emulgel. Spreadability is a term expressed to denote the extent of area to which the gel readily spreads on application to skin or the affected area. The value of spreadability also indicates the therapeutic efficiency of the formulation [17].

The consistency reflects the capacity of the gel, to get ejected in uniform and desired quantity when the tube is squeezed. Consistency is inversely proportional to the distance traveled by falling cone. In the present study, consistency of the *M. × piperita* oil gel in terms of distance traveled by cone was 7 mm. The consistency of diclofenac sodium gel was reported to be slightly better at 5 mm [15] while the consistency of aceclofenac sodium gel was found to be at 6 mm [19]. *M. × piperita* oil gel showed good extrudibility. The extrusion of gel has significance during application.

TABLE II  
STABILITY STUDY OF *M. × PIPERITA* OIL GEL

Months	Appearance	pH	Consistency (mm)	Extrudibility	Viscosity (cP)	Spreadability (g.cm/sec)
0	Clear	6.6	7	Good	1200	10.7
1	Clear	6.6	7	Good	1202	10.7
2	Clear	6.6	7.5	Good	1215	10.6
3	Clear	6.7	8	Good	1250	10.4

#### D. Stability of Gel

The *M. × piperita* oil gel was subjected to stability study as per ICH guidelines for the period of three months and the stability evaluation data is mentioned in Table II. During the stability studies, no significant change in pH and viscosity as a function of time was observed, indicating stability of prepared formulation. Also, values for consistency and spreadability showed slight change with storage. Extrudibility of the gel remained good during the storage period.

#### IV. CONCLUSION

The essential oil of *M. × piperita* presents an eco-friendly approach towards insect control as well as its various applications as antibacterial, antifungal, and anti-cancerous substance. The gel developed from *M. × piperita* oil in the present study met all the standard requirements for its characterization as a stable gel. Further, its stability under thermal stress was also established. The gel developed in this study is expected to steer the usage of *M. × piperita* essential towards commercial application. However, further study could be done to estimate the economically viability of the developed product, routing the research towards ensuring the product availability for mass usability.

#### ACKNOWLEDGMENT

The authors acknowledge Mr. Jai Kumar (Formulation Division, IPFT, Gurgaon) and Mr. Sabal Singh & Mr. Satendar Singh (IIT Delhi, India) for their help in experimental work.

#### REFERENCES

[1] Baris O, Güllüce M, Shahin F, Özer H, Kilic H, Özkan H, Sökmen M, Özber T (2006). Biological activities of the essential oil and methanol extract of *Achillea biebersteinii* (Asteraceae) Turk J Biol 30: 65-73.

[2] Kumar P, Mishra S, Malik A, Satya S (2011). Repellent, larvicidal and pupicidal properties of essential oils and their formulations against the housefly, *Musca domestica*. Med Vet Entomol

[3] Werka JS, Boehme AK, Setzer WN (2007). Biological activities of essential oils from Monteverde, Costa Rica. Nat Prod Commun 2: 1215-1219.

[4] Ormancey X, Sisalli S, Coutiere P (2001). Formulation of essential oils in functional perfumery. Parfums Cosmetiques Actualites 157: 30-40.

[5] Bagwe RP, Kanicky JR, Palla BJ, Pantajali PK, Shah DO (2001). Improved drug delivery using microemulsions: rationale, recent progress and new horizons. Crit Rev Ther Drug 18: 77-140.

[6] Manou I, Bouillard L, Devleschouwer MJ, Barel AG (1998). Evaluation of the preservative properties of *Thymus vulgaris* essential oil in topically applied formulations under a challenge test. J Appl Microbiol 84: 368-376.

[7] Orafidiya LO, Oyedele AO, Shittu AO, Elujoba AA(2001). The formulation of an effective topical antibacterial product containing *Ocimum gratissimum* leaf essential oil. Int J Pharm 224: 177-83.

[8] Oyedele, AO, Gbolade AA, Sosan MB, Adewoyin FB, Soyelu OL, Orafidiya OO (2002). Formulation of an effective mosquito-repellent topical product from lemongrass oil. Phytomedicine 9: 259-262.

[9] Quinones D, Ghaly ES (2008). Formulation and characterization of nystatin gel. P R Health Sci J 27: 61-67.

[10] Kumar S, Himmelsten K (1995) Modification of in situ gelling behavior of carbopol solution by Hydroxypropyl methyl cellulose. J Pharm Sci 84:344-8.

[11] Rama rao P, Diwan PV (1998) Formulation and In vivo Evaluation of Polymeric Film Dikiazim Hydrochloride and Indomethacin for Transdermal Administration. Drug Dev. Ind Pharm 24:327-338.

[12] Lee B, Choi W, Lee S, Park B (2001). Fumigant toxicity of essential oils and their constituent compounds towards the rice weevil, *S. oryzae* (L.). Crop Prot 20: 317-320.

[13] Bakkali F, Averbeck S, Averbeck D, Idaomar M (2008). Biological effects of essential oils. Rev Food Chem Toxicol 46: 446-475.

[14] Kumar P, Mishra S, Malik A, Satya S (2012). Efficacy of *Mentha × piperita* and *Mentha citrata* essential oils against housefly, *Musca domestica* L. Ind Crop Prod 39 (2012) 106–112

[15] Shivhare UD, Jain KB, Mathur V B, Bhusari K P, Roy A (2009). Formulation development and evaluation of diclofenac sodium gel using water soluble polyacrylamide polymer. Dig J Nanomater Bios 4: 285-290.

[16] William L (2000) Remington: The Science and Practice of Pharmacy. 20th edition. Mack Publishing Company. Easton, PA.

[17] Bhanu PV, Shanmugam V, Lakshmi P K (2011) Development And Optimization Of Novel Diclofenac Emulgel for Topical Drug Delivery . Internat J Compre Pharmacy 9 (10): 1-4.

[18] ICH Harmonized Tripartite Guidelines (2003) Stability Testing of New Drug Substances and Products. ICH Committee 8.

[19] Kumar R, Patil MB, Patil SR, Paschapur MS (2009) Evaluation of *Anacardium occidentale* gum as gelling agent in Aceclofenac Gel. Int J ChemTech Res Coden (USA): Ijprif. 1 (3): 695-704.