

**ANTI-ACNE SYNERGISTIC HERBAL FACE WASH GEL:
FORMULATION, EVALUATION AND STABILITY STUDIES****Harsharan Pal Singh^{*1}, Neeraj Samnhotra^{1,2}, Sumeet Gullaiya³, Ishpreet Kaur⁴**¹Department of Quality Assurance, AIMIL Pharmaceuticals (I) Limited, New Delhi, India.²Formulation & Development Department, AIMIL Pharmaceuticals (I) Limited, New Delhi, India.³Amity Institute of Pharmacy, Amity University, Noida, Uttar Pradesh, India.⁴Department of Quality Assurance, Delhi Institute of Pharmaceutical Sciences & Research, New Delhi, India.**ABSTRACT**

Acne is a chronic inflammatory disorder of pilosebaceous unit, which involves increased sebum production by sebaceous glands and abnormal desquamation of hair follicles occur in response to increasing androgen levels with the onset of puberty. Obstruction of follicles causes follicular distention which is often accompanied by the proliferation of the bacteria *Propionibacterium acnes* and the activation of an inflammatory response. The Aim of this study was to formulate and evaluate the herbal face wash gel containing extracts of *Azadirachta indica* (Neem), *Curcuma longa* (Haldi), *Coriandrum sativum* (Dhaniya), *Aloe barbadensis* (Aloe Vera), *Citrus limon* (Lemon) and *Mentha* (Pudina) distillate using Carbomer Ultrez 20. The plants have been reported in the literature having good anti-microbial, anti-oxidant and anti-inflammatory activity. Prepared formulation was evaluated for various parameters like colour, appearance, consistency, pH, viscosity, stability studies and consumer acceptance test.

KEYWORDS: Acne vulgaris, Anti-acne activity, Anti-microbial, Inflammatory response, Traditional herbs.

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Correspondence*For Author****Harsharan Pal Singh**

Department of Quality
Assurance, AIMIL
Pharmaceuticals (I)
Limited, New Delhi,
India.

1. INTRODUCTION

The skin is the largest organ of the body, accounting for about 15% of the total adult body weight. It performs many vital functions, including protection against external physical, chemical, and biologic assailants, as well as prevention of excess water loss from the body and a role in thermoregulation. The skin is continuous, with the mucous membranes lining the body's surface.^[1] To keep skin healthy, clear and glossy, a balanced nutrition is required. Apart from the balanced nutrition, hormonal changes especially during the puberty in both sexes cause many changes in the body.^[2] Among various changes, Acne vulgaris is the most common. Acne vulgaris is a common skin condition with substantial cutaneous and psychologic disease burden. The pathogenesis of acne is a result of multifaceted processes within the pilosebaceous unit resulting in bacterial overgrowth and inflammation. This condition typically develops at the time of the pubertal transition when changes in the body's hormonal milieu alter pilosebaceous gland function. Initially, follicular epithelial cells differentiate abnormally and form tighter intracellular adhesions and, therefore, are shed less readily. This process leads to the development of hyperkeratotic plugs, or microcomedones, which enlarge progressively to form non-inflammatory, closed or open comedones,^[2-4] To overcome the problem of acne vulgaris and to get rid off use of synthetic drugs such as clindamycin, a synergistic anti acne face wash gel has been formulated.

2. MATERIALS AND METHODS

2.1. Collection of Herbs and Chemicals

Herbs

All the herbs were collected from plant nurseries from the region of New Delhi and dried in sunlight.

Chemicals

Ultrez 20 Carbomer (Lubrizol Advanced Materials, USA)- Gelling Agent, Glycerine-Humectant, Sodium Lauryl Ether Sulphate- Surfactant (Jangra Chemicals, Punjab), Sodium Methyl Paraben, Sodium Propyl Paraben- Preservatives and Sodium Hydroxide- Neutralizer (Thermo Fischer Scientific India Pvt. Ltd., Mumbai) and EDTA- Chelating agent (S.D. Fine-Chem Limited, Mumbai) of laboratory grade were used in the study.

2.2. Authentication and Evaluation of Herbs

Dried herbs were authenticated by Dr. H.B. Singh (Chief Scientist & Head (Retd), Raw materials Herbarium & Museum, NISCAIR, New Delhi), Herbs Authentication Officer, AIMIL Pharmaceuticals (I) Limited, New Delhi. Moreover, all the herbs were evaluated as per Ayurvedic Pharmacopeia of India.

2.3. Preparation of Herbal Synergistic Extract^[5,7-9]

Ethanollic Extract of aerial parts of *Azadirachta indica* (Neem), rhizomes of *Curcuma longa* (Haldi), leaves of *Coriandrum sativum* (Dhaniya), whole plant of *Aloe berbadandis* (Aloe Vera) and fruit of *Citrus limon* (Lemon) was prepared by Hot Extraction Method using water condensor.

2.4. Formulation of Polyherbal Face wash Gel

Carbomer Ultrez 20 was swelled in ethanolic extract, water and *Mentha* (Pudina) distillate along with the preservatives. After swelling of carbomer, glycerine was added. To the above mixture, Sodium lauryl ether sulphate (SLES) was introduced and gently mixed. Finally chelating agent i.e. EDTA and NaOH Solution was added to get complete gel with consistency.

2.5. Characterization and Evaluation of Gel^[6]

The prepared face wash gel was evaluated for various parameters.

2.5.1. Colour

The colour of the face wash gel was visually analyzed.

2.5.2. Odour

The formulation was evaluated for its odour by smelling it.

2.5.3. Consistency

It was determined manually.

2.5.4. Viscosity

Viscosity of the gel was determined using Brookfield viscometer. The values obtained for the sample and for water were noted down.

2.5.5. Spreadability

The spread ability of the gel was found manually by applying the gel on the skin with gentle rub.

2.5.6. Washability

The product was applied on hand and was observed under running water.

2.5.7. Foamability

Small amount of gel was taken in a beaker containing water. Initial volume was noted, beaker was shaken for 10 times and the final volume was noted. Foamability was also analysed by applying onto skin with contact with water.

2.5.8. Grittiness

The product was checked for the presence of any gritty particles by applying it on the skin.

2.6. Accelerated Stability Studies^[6]

The Face wash gel formulation was subjected to stability testing for 2 months as per ICH Guidelines at a temperature of $40^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and RH 75%. The Gel formulation was analysed for the change in appearance, pH and phytochemicals.

2.7. Consumer Acceptance test

The Product was evaluated for consumer acceptance by 30 targeted consumers who were between 15-30 years old. The Evaluation was done on the basis of four parameters: (1) Appearance; (2) Foaming; (3) Fragrance; (4) After Rinse-off feel. This test was carried out with consent of all the participants.

3. RESULTS AND DISCUSSION

3.1. Evaluation of Herbs^[10]

3.1.1 *Azadirachta indica* (Neem)



Fig 1: Neem Leaves

Nimba (leaf) consist of dried leaf of *Azadirachta indica* ; (Family: Meliaceae); a moderate sized to fairly large evergreen tree, attaining a height of 12-15m with stout trunk and spreading branches, occurring throughout the country upto an elevation of 900m.

Table 1: Evaluation and Phytochemical Screening of *Azadirachta indica* (Neem).

RESULT OF ANALYSIS			
DESCRIPTION	:	Yellowish-green pieces, taste, bitter	
MACROSCOPIC	:	Compound, alternate, rachtis 15-20cm long, 0.1cm thick, leaflets with oblique base, opposite, acute, serrate, 7-8cm long.	
PARAMETER		RESULTS	SPECIFICATIONS
FOREIGN MATTER	:	0.8% w/w	Not more than 2% w/w
TOTAL ASH	:	7.25% w/w	Not more than 10% w/w
ACID INSOLUBLE ASH	:	0.4% w/w	Not more than 1% w/w
ALCOHOL SOLUBLE EXTRACTIVE	:	15.7% w/w	Not less than 13% w/w
WATER SOLUBLE EXTRACTIVE	:	21% w/w	Not less than 19% w/w
PHYTOCHEMICAL SCREENING	:	Complies	Triterpenoids and Sterols

3.1.2. *Curcuma longa* (Turmeric)



Fig 2: Rhizomes of Turmeric

Haridra consists of the dried and cured rhizomes of *Curcuma longa* Linn (Family: Zingiberaceae); a perennial herb extensively cultivated in all parts of the country; crop is

harvested after 9-10 months when lower leaves turn yellow rhizomes carefully dug up with hand picks between October-april and cured by boiling and dried.

Table 2: Evaluation and Phytochemical Screening of *Curcuma longa* (Turmeric)

RESULT OF ANALYSIS			
DESCRIPTION	:	Ovate, oblong, yellowish to yellowish-brown	
MACROSCOPIC	:	Rhizome ovate, oblong or cylindrical, about half as broad as long, latter 2-5cm long and about 1-1.8cm thick, externally yellowish to yellowish brown with root scars.	
PARAMETER		RESULTS	SPECIFICATIONS
FOREIGN MATTER	:	0.85% w/w	Not more than 2% w/w
TOTAL ASH	:	6.2% w/w	Not more than 9% w/w
ACID INSOLUBLE ASH	:	0.6% w/w	Not more than 1% w/w
ALCOHOL SOLUBLE EXTRACTIVE	:	9.7% w/w	Not less than 8% w/w
WATER SOLUBLE EXTRACTIVE	:	14% w/w	Not less than 12% w/w
PHYTOCHEMICAL SCREENING	:	Complies	Polyphenolic curcuminoids ,Volatile oil

3.1.3. *Coriandrum sativum* L. (Coriander)



Fig 3: Leaves of Coriander

Dhanyaka consists of dried leaves of *Coriandrum sativum* Linn (Family: Umbelliferae) ; a slender, glabrous, branched, annual herb, cultivated all over India, 30-90 cm high; giving characteristic aroma when rubbed; crop matures in 2-3 months after sowing.

Table 3: Evaluation and Phytochemical Screening of *Coriandrum sativum* L. (Coriander)

RESULT OF ANALYSIS			
DESCRIPTION	:	Small chopped leaves and twigs	
MACROSCOPIC	:	Dried leaves, 30-90 cm high; giving characteristic aroma	
PARAMETER		RESULTS	SPECIFICATIONS
FOREIGN MATTER	:	0.42% w/w	Not more than 1% w/w
TOTAL ASH	:	1.36% w/w	Not more than 3% w/w
ACID INSOLUBLE ASH	:	0.3% w/w	Not more than 0.5% w/w
ALCOHOL SOLUBLE EXTRACTIVE	:	6.80% w/w	Not less than 4% w/w
WATER SOLUBLE EXTRACTIVE	:	6.96% w/w	Not less than 5% w/w
PHYTOCHEMICAL SCREENING	:	Complies	Volatile oil

3.1.4. *Aloe berbadensis* Linn (*Aloe Vera*)**Fig 4: Leaves of Aloe Vera**

Aloe vera is a stemless or very short-stemmed succulent plant growing to 60–100 cm (24–39 in) tall, spreading by offsets. The leaves are thick and fleshy, green to grey-green, with some varieties showing white flecks on their upper and lower stem surfaces. The margin of the leaf is serrated and has small white teeth. The flowers are produced in summer on a spike

up to 90 cm (35 in) tall, each flower being pendulous, with a yellow tubular corolla 2–3 cm (0.8–1.2 in) long.

Table 4: Evaluation and Phytochemical Screening of *Aloe berbadensis* Linn (*Aloe Vera*)

RESULT OF ANALYSIS			
DESCRIPTION	:	Leaves are large with a wide base and an apex.	
MACROSCOPIC	:	Leaves are thick and fleshy, green to grey-green, spike up to 90 cm (35 in) tall, each flower being pendulous, with a yellow tubular corolla 2–3 cm (0.8–1.2 in) long.	
PARAMETER		RESULTS	SPECIFICATIONS
FOREIGN MATTER	:	2.75% w/w	Not more than 4% w/w
TOTAL ASH	:	3.4% w/w	Not more than 5% w/w
ACID INSOLUBLE ASH	:	0.4% w/w	Not more than 1% w/w
ALCOHOL SOLUBLE EXTRACTIVE	:	7% w/w	Not less than 5% w/w
WATER SOLUBLE EXTRACTIVE	:	13% w/w	Not less than 10% w/w

3.1.5. *Mentha viridis* (Mint)



Fig 5: Leaves of Mint

Pudinah consists of the aerial part of *Mentha viridis* Linn (Family: Lamiaceae) a perennial, creeping aromatic herb of 30 to 90cm high, widely cultivated throughout the plains of India for culinary and medicinal purposes.

Table 5: Evaluation and Phytochemical Screening of *Mentha viridis* (Mint)

RESULT OF ANALYSIS			
DESCRIPTION	:	Small chopped leaves and twigs	
MACROSCOPIC	:	Small chopped twigs; leaves opposite, decussate, shortly petiolate, petioles 2mm long; mature leaves 2.5 to 3.5 cm long and 1.5 to 2.0cm broad.	
PARAMETER		RESULTS	SPECIFICATIONS
FOREIGN MATTER	:	0.7% w/w	Not more than 2% w/w
TOTAL ASH	:	8% w/w	Not more than 14% w/w
ACID INSOLUBLE ASH	:	2.5% w/w	Not more than 4% w/w
ALCOHOL SOLUBLE EXTRACTIVE	:	4% w/w	Not less than 2% w/w
WATER SOLUBLE EXTRACTIVE	:	9.2% w/w	Not less than 7% w/w
PHYTOCHEMICAL SCREENING	:	Complies	Essential oils

3.2. Formulation of Face wash Gel

Anti-acne face wash gel was fabricated using extract of herbs. The composition of gel developed in the laboratory is given in Table 6.

Table 6: Composition of Anti-acne face wash gel of polyherbal extract.

Ingredients	Concentration
Synergistic Herbal Extract	3.0%
De-mineralized Water	q.s. to 100
Mint distillate	10.0%
Carbomer Ultrez 20	1.0%
Glycerine	4.0%
Sodium Lauryl Ether Sulphate (SLES)	15%
NaOH (18% Solution)	2.4%
Sodium Methyl Paraben	0.1%
Sodium Propyl Paraben	0.1%

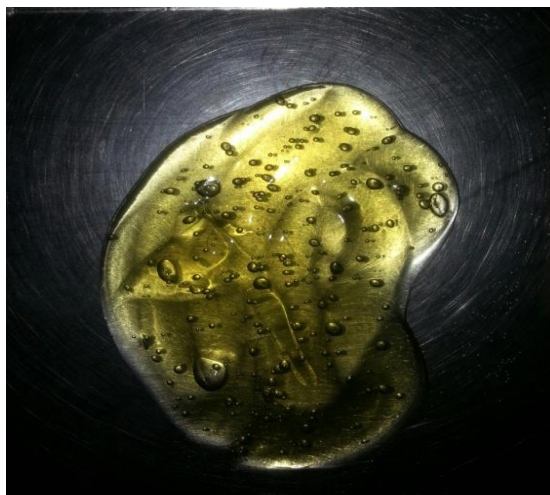


Fig 6: Anti-acne Face Wash Gel Formulation

3.3. Evaluation of Gel

3.3.1. Determination of Gel Viscosity

Table 7: Viscosity of Face wash Gel

Formulation	Viscosity (cp)
Anti-acne Face Wash Gel	1748

3.3.2. Determination of pH, Physical properties

Table 8: Physical properties & pH of Face wash Gel

Physical parameters	Inference
Colour	Slightly yellowish
Odour	Sweet Lemon
Appearance	Translucent
Feel on application	Smooth & slippery
pH	6.15

3.3.3. Spreadability, Consistency, Washability, Foamability and Grittiness of Formulation.

Table 9: Spreadability, Consistency, Washability, Foamability and Grittiness of Developed face wash gel

Formulation	Spreadability (g.cm/sec)	Consistency (60 sec)	Washability	Foamability	Grittiness
Anti-acne Face wash Gel	7.0	8 mm	Easily Washable	Foam volume 92ml at 5 minutes	No gritty particles
Marketed Face wash Gel	7.5	11 mm	Easily Washable	Foam volume 85ml at 5 minutes	No gritty particles

The values of Spreadability indicate that the face wash gel is easily spreadable by small amount of shear. Spreadability of marketed face wash gel was 7.5 g.cm/sec.

The consistency reflects the capacity of gel, to get ejected in uniform and desired quantity when tube is squeezed. Consistency in terms of distance travelled by cone was between 7-8 mm of developed formulation as compared to 11 mm of marketed one. Consistency is inversely proportional to the distance travelled by falling cone. Hence, the consistency of face wash formulation was better as compared to that of marketed wash face gel. Developed anti-acne face wash gel showed foam volume of 92ml at 5 minutes and No gritty particles were observed in the formulation.

3.4. Accelerated Stability studies

On storage of anti-acne face wash gel sample at 40°C and 75% RH, the appearance of the formulation was found to be clear with no significant variation in pH. The result of accelerated stability studies are presented in Table 10.

Table 10: Accelerated stability study results of developed Anti-acne Face wash Gel

Days	Appearance	pH	Phytochemicals
0	Slightly yellowish	6.15	Present
10	Slightly yellowish	6.15	Present
20	Slightly yellowish	6.17	Present
30	Slightly yellowish	6.17	Present
45	Slightly yellowish	6.19	Present
60	Slightly yellowish	6.20	Present

Based on the results of physicochemical evaluations (Viscosity, Spreadability, consistency, pH and accelerated studies) of the gel formulations, it was observed that the formulation was appropriate and it was further analysed for Consumer Acceptance Test.

3.5. Consumer Acceptance Test

Parameters evaluated by 30 consumers were noted and proportional representation is given below.

- 85% of the users were satisfied with the Appearance of the Face Wash Gel.
- 77% of the consumers were satisfied with the light foaming formed by the Face Wash.
- 90% users liked the natural fragrance i.e. Lemon characteristic Odour of Face Wash.
- The After rinse-off feel by using the face wash gel was liked by 80% of the targeted consumers.

- No skin irritation case was found during and after the use of the Face Wash Gel.
- 4% users were not satisfied with the Face Wash Gel.

CONCLUSION

Herbal anti-acne face wash gel was developed and characterized along with the stability studies. The optimal formula was 65% de-mineralised water, 1% Carbopol Ultrez-20, 2.4% NaOH (18%), 4% Glycerin, 10% Mint distillate, 15% SLES, 0.1% Sodium propyl paraben, 0.1% Sodium methyl paraben and 3% herb extract (30.0% Neem, 20.0% Turmeric, 10.0% Coriander, 20.0% Fresh Lemon, 20.0% Aloe Vera juice) , which gave high effectiveness. In consumer test, the product was accepted by 83% of targeted consumers.

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REFERENCES

1. Kanitakis J. Anatomy, histology and immunohistochemistry of normal human skin. *European Journal of Dermatology.*, 2002; 12(4): 390–401.
2. Brown SK, Shalita A. Acne vulgaris. *Lancet*, 1998; 351(9119): 1871–1876.
3. Uhlenhake E, Yentzer BA, Feldman SR. Acne vulgaris and depression: a retrospective examination. *J Cosmet Dermatol.*, 2010; 9: 59–63.
4. Burkhart CG, Burkhart CN, Lehmann PF. Acne: a review of immunologic and microbiologic factors. *J Post grad Med.*, 1999; 75: 328–331.
5. Ahmad I, Mehmood Z., Screening of some Indian medicinal plants for their antimicrobial properties. *Journal of Ethnopharmacology.*, 1998; 62(2): 183-193.
6. Vogel H.G.; *Drug Discovery and evaluation, Pharmacological assays*, Second edition, Springer publications., 2002; pp-1336.
7. Chopra R.N., Nayer S.L., Chopra I.C., *Glossary of Indian Medicinal Plants*, 3rd edn. Council of Scientific and Industrial Research, 1992, New Delhi, p. 7–246.
8. Caius J., *The Medicinal and Poisonous Plants of India*. Scientific Publishers, India; 2003, p. 527.
9. Aburijai T., F.M.Natsheh., *Plants used in cosmetics*. *Phytother.Res.*, 2003; 17: 987-1000.

10. Ayurvedic Pharmacopoeia of India; Part I & Part II, 2007; Govt. Of India; Published by IPC, Ghaziabad.
11. Kanlayavattanakul M. and Lourith N., Therapeutic agents and herbs in topical application for acne treatment, International Journal of Cosmetic Science., 2011; 33: 289–297.