Preparation and evaluation of soft beads containing vasaka extract

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Abstract

The present study focuses on the formulation and evaluation of soft rupturable beads containing Vasaka (Justicia adhatoda) extract for aromatherapy applications. Vasaka, a traditional Ayurvedic remedy known for its efficacy in treating respiratory conditions such as cough, asthma, and bronchitis, was explored for a modern drug delivery approach. Utilizing the extrusion-spheronization technique, soft beads were developed to offer a cleaner, more user-friendly alternative to conventional forms like syrups and tablets. Various batches were prepared and evaluated for physical properties, drug entrapment efficiency, particle size, and stability. Among them, Batch B4 demonstrated optimal characteristics, including a spherical shape, high entrapment efficiency (75.5%), ease of rupture, and good stability under storage conditions. UV spectroscopic analysis confirmed the presence of active constituents, and FTIR analysis ensured compatibility between the drug and excipients. The optimized formulation proved effective for aromatherapy, offering a novel and convenient delivery system for Vasaka.

Key words : Vasaka**,** Justicia adhatoda**,** Aromatherapy**,** Soft rupturable beads**,** Extrusion-spheronization**,** Drug delivery system**,** Entrapment efficiency**,** UV spectroscopy**,** FTIR analysis**,** Respiratory therapy**,** Herbal formulation

1. Introduction

"Vasaka, adulsa, Malabur nut tree" is another name for Adhatoda vasica, which is a member of the Canthaceae family. Another synonym for it is Justicia adhatoda. In Ayurvedic and Unani medicine, it is regarded as the most important medication [1, 2]. The little evergreen shrub is primarily found in Burma, Sri Lanka, and India's plains [3]. The ancient Indians once remarked, "No man suffering from phthisis needs despair as long as the Vasaka plant exists." This herb has been used for 2000 years to heal respiratory issues. The respiratory system is stimulated by this plant's leaves [4]. It is regarded as a key herb in the Ayurvedic medical system to treat conditions like bronchitis, asthma, cold symptoms, and cough [5].

Among its several therapeutic effects are those of cardiovascular protection, antitubercular, antiasthmatic, antimicrobial, antitussive, antimutagenic [6].

Fig 1.1 Adhatoda vasica (Vasaka)

Table No1.1: Botanical Classification of Adhatoda vasica [7,8]

Taxonomical Rank	Taxon
Kingdom	Plantae
Division	Angiosperm
Class	Eudicots
Order	Lamiales
Family	Acanthaceae
Genus	Justicia
Species	J. adhatoda
Common name	Adulsa (vasaka)

1.3 Soft rupturable beads:-

Microbeads are extremely small, almost spherical particles that range in diameter from 0.5 to 1000 micrometers. They function as free-flowing, solid carriers that can contain dissolved or crystalline medication particles. These microbeads are made by blending different polymers in particular proportions. These consist of binding agents like gelatin, chondroitin sulfate, and avidin, as well as cationic polymers like chitosan and anionic polymers like sodium alginate [9,10]. Because they efficiently transport active core compounds, beads—spherical particles with sizes ranging from 50 nanometers to 2 millimeters—are one of the most popular techniques for prolonged drug release [11].

1.4 Types of Beads

1. Hydrogel beads
2. Alginate beads
3. Floating beads

4. Sustained release beads
5. Mucoadhesive beads

1.5 Aromatherapy

Essential oils, which are naturally occurring molecules that plants make to defend themselves against stress, illnesses, herbivores, insects, heat, and drought, are used in aromatherapy, a subset of phytotherapy. "Aroma" means fragrance or scent, and "therapy" means treatment. These two words combine to form the phrase "aromatherapy." Inhalation is the most often used technique for employing essential oils in aromatherapy. Aromatherapy has been shown to strengthen the immunological, respiratory, and circulatory systems while also encouraging relaxation, lowering stress, elevating mood, and improving well-being, even though it cannot treat severe ailments [12].

1.6 Novel Techniques of Beads formulation

1. Ionotropic gelation method
   Ionotropic gelation methods classified into two types
   - External gelation method
   - Internal gelation method

2. Emulsion gelation method

1.7 Introduction of Pelletization [13]

The pellets are spherical or nearly spherical, free flowing granules with a limited size distribution ranging from 500 to 1500 µm for medicinal purposes. They are typically made via a pelletization method in which a powder blend containing an API and excipient particles is agglomerated into spherical granules. Pellets are typically prepared and then put into firm gelatin capsules or compacted into tablets. The spherical shape of the pellets and their low surface area to volume ratio resulted in a homogenous film coating. Pellets remove the dose dumping effect, resulting in a better plasma concentration profile and progressive absorption of the medicine compared to tablets, further reducing pharmacological side effects.

1.8 Pelletization By Extrusion And Spheronization

The most often used process for creating pellets is extrusion-spheronization. Reynolds and Conine and Hadley were the first to disclose this procedure, which consists of four steps: (i) the wet mass is prepared (granulation); (ii) the wet mass is shaped into cylinders (extrusion); (iii) the extrudate is broken up and the particles are rounded into spheres (spheronization); (iv) and the pellets are dried [14].

EXTRUSION SPHERONIZATION MAINLY INVOLVES FOUR STEPS [15]

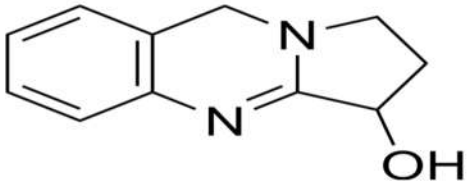
1. Granulation
2. Extrusion
3. Spheronization
4. Drying of pellets

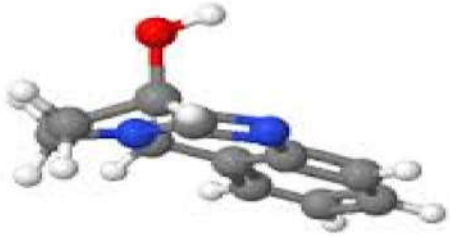
2. DRUG PROFILE

2.1 Vasaka (Justicia Adhatoda)[16]

Fig.8 Vasaka Leaves

Synonyms :- Adhatoda vasica
Biological source:- It is dried or fresh leaves of Adhatoda vasica or Malabar nut
Family :- Acanthaceae
Chemical Constituent:- Vasaka's main chemical components include a number of alkaloids, the most important of which are vasicine and vasicinone, which are found in vasicine leaves.
Major components:- Vasicine





Chemical Name :- (3S)-1, 2, 3, 9-Tetrahydropyrrolo [2, 1-b] quinazolin-3-ol

Table No. 2.1: Physical Properties

Chemical formula	$C_{11}H_{12}N_2O$
Molar weight	188.230 g·mol^{-1}
Melting point	210 °C (410 °F; 483 K)
Boiling point	393.8°C at 760 mmHg
Colour	Dark green
Taste	Bitter
Odour	Slight characteristic, and unpleasant
Density	1.1124

Solubility:- Soluble in acetone , alcohol, methanol, chloroform and also slightly soluble in water especially for decoctions and aqueous extracts.

2.2 MEDICINAL USES OF VASAKA

1. Respiratory Health
2. Anti-Inflammatory & Analgesic
3. Antimicrobial Activity
4. Fever And Malaria

Table No. 2.2: Available Marketed Dosage Forms

Sr. No.	Brand Name	Manufacturer	Formulations
1	Himalaya	Himalaya Wellness Company	Syrup (Oral)
2	Bixa Botanical	SA Herbal Bioactives LLP	Powder (Oral)
3	Arogyam	Arogyam Ayurveda	Tablet (Oral)

5. Methods and materials

5.1 Extraction Of Vasaka[17]

Vasaka leaf extract was prepared by using Maceration method

5.2 Identification Tests For Vasaka Extract [18]

Table no. 5.1: Chemical test for vasaka

Sr.no.	Chemical test	Observation	Inference
1	**Test For Alkaloids: Mayer's Test**	Whitish yellow or cream coloured precipitate	Present
2	**Test for terpenoids: Salkowski test**	Reddish brown ring	Present
3	**Test for flavonoids: Alkaline reagent test**	Yellow colour	Present
4	**Test for Saponin : Froth test**	Formation of froth	Present

5.3 UV Spectrum Studies (λMax) Of Vasaka Extract

UV spectrophotometry has been used to validate drug structures in identification investigations. The medication was dissolved in chloroform, and the solution was scanned between 200 and 400nm with a UV spectrophotometer [LABMAN scientific instrument].

5.4 Calibration Curve of Marketed Vasaka

The standard stock solution of vasaka included 1000µg/ml concentration, and aliquots were made with varied concentrations such as 10µg/ml, 20µg/ml, 30µg/ml, 40µg/ml, and 50µg/ml using chloroform as a solvent. The absorbance was then measured in a UV single beam spectrophotometer at 268.3 nm using chloroform as a blank. [Labman Scientific Instrument]

5.5 Calibration Curve of Extracted Vasaka

The standard stock solution of vasaka included 1000µg/ml concentration, and aliquots were made with varied concentrations such as 10µg/ml, 20µg/ml, 30µg/ml, 40µg/ml, and 50µg/ml using chloroform as a solvent. The absorbance was then measured in a UV single beam spectrophotometer at 261.1 nm using chloroform as a blank. [Labman Scientific Instrument][19]

5.6 Formulation And Preparation of Soft Rupturable Beads

5.6.1 Formulation (F_3): Method Of Preparation Of Soft Beads Without Drug

Table No. 5.4: Composition Of Soft Beads Without Drug

Composition	F1	F2	F3
Agar (%)	0.5	1	1
Gelatine (%)	0.25	1.5	2
Sodium alginate (%)	0.5	1.5	1
MCC (%)	0.25	2.5	1.5
PEG-400 (%)	0.25	1	3
Water (%)	25	50	50
Calcium chloride (%)	0.5	1	1

5.6.2 Formulation of Soft Rupturable Beads Containing Vasaka Extract By Using Extrusion-Spheronization

Table No. 5.5: Preparation Containing Vasaka Extract [Preparation 1]

Sr. No	Contents	Quantity
1	Vasaka Extract (%)	20
2	Petroleum Jelly (%)	3.7
3	Bees Wax (%)	0.74
4	Cocoa Butter (%)	6.66
5	Surfactant (%)	0.74
/6	Water (%)	Q.S

Table No. 5.6: Composition Of Base For Preparation Of Different Batches Containing Soft Rupturable Beads [Preparation 2]

Components	B1	B2	B3	B4	B5	B6
Magnesium carbonate (%)	-	-	-	0.65	0.70	0.75
MCC (%)	0.72	0.69	0.60	-	-	-
Vasaka base (%)	2	4	2	6	4	6
Zinc stearate (%)	0.4	0.5	1	0.6	0.5	0.5
Zinc oxide (%)	1	3	4	4.5	5	5
Talc (%)	9.60	9.25	13.35	14.45	16.25	16.25
Binder HPMC (%)	5.5	3	5	2	4	2.5

6. Result And Discussion

6.1 Identification Test Of Vasaka

Test	Result
Mayer's test	Pass
Salkowski test	Pass
Alkaline reagent test	Pass
Froth test	Pass

Fig 6.1 Mayer's Test

Fig 6.2 Salkowski Test

Fig 6.3 Alkaline Reagent Test

Fig 6.4 Froth Test

6.2 Physical properties of vasaka

Table No 6.1 Physical Properties

Sr.No	Description	Properties
1	Colour:	Dark Green
2	State/Form:	Shrub Or Small Tree
3	Odour:	Characteristic, Slightly Pungent
4	Taste:	Bitter
5	Solubility:	Solubility In Acetone, Alcohol, Chloroform.
6	Boiling Point:	393.8°C At 760 Mmhg
7	Density	1.1124

6.3 UV Analysis Of Vasaka

UV-Visible spectroscopy revealed a maximum absorption at 268.3 nm for marketed vasaka and 261.6 nm for the extract, confirming the presence of vasaka (Figures 7.5 and 7.6).

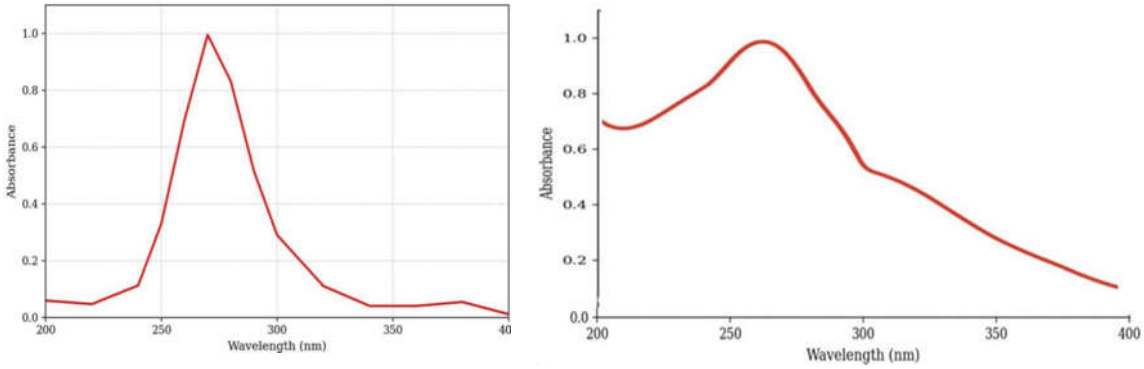


Fig 6.5 UV Analysis of Marketed Vasaka Product And Vasaka Extract

6.4 Standard Calibration Curve of Vasaka

6.4.1 Calibration Curve of Marketed Vasaka and Extracted Vasaka

The λmax of vasaka was 268 nm. A standard calibration curve was prepared for concentrations ranging from 10–60 µg/ml at this wavelength. Absorbance vs. concentration was plotted, and linear regression analysis gave an r^2 value of 0.997and 0.991.

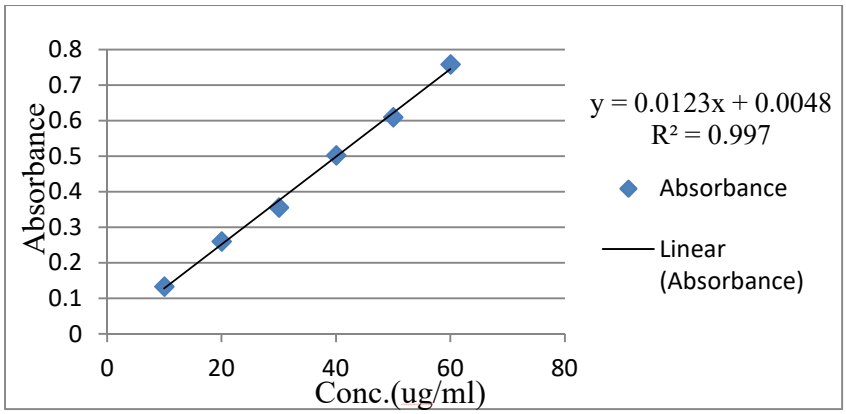


Fig 6.6 Calibration Curve of Marketed Vasaka

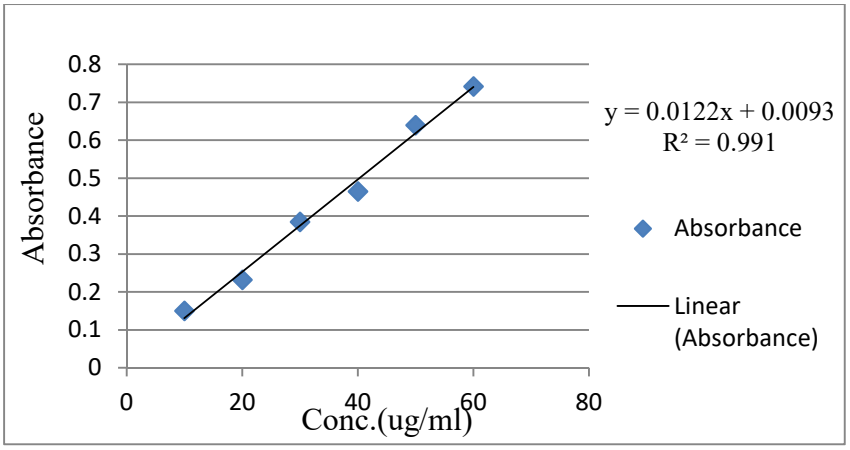


Fig 6.7 Calibration Curve of Extracted Vasaka

6.5 Compatibility Study of Vasaka

Fourier Transform Infrared Absorbtion Spectroscopy (FTIR)

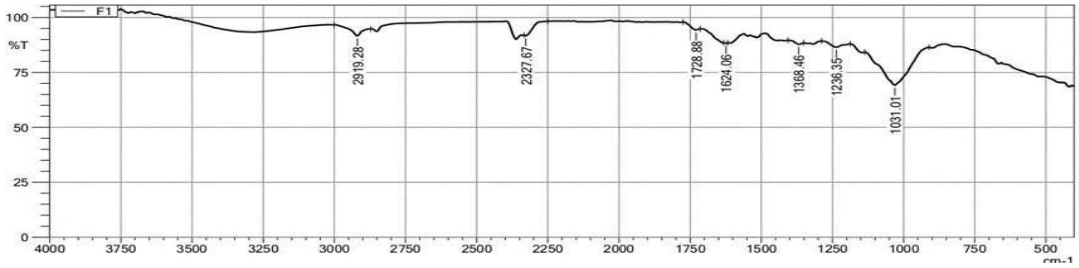


Fig 6.8 FTIR of Pure Drug- Vasaka

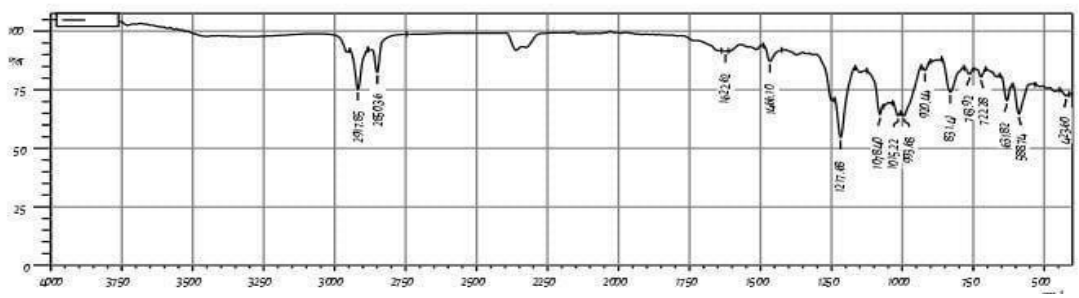


Fig 6.9 FTIR of Physical Mixture of Excipients With Pure Drug

Table No. 6.2: Fourier Transform Infrared Spectroscopy Peak Assignment Table

Frequency (cm^{-1}) (F1)	Frequency (cm^{-1}) (F2)	Probable Assignment
2919.28	2917.85	C-H stretching (Allkanes)
2327.67	-	C≡N or CO2 (possible artifact)
1728.88	-	C=O Streching (Carbonyl)
1624.06	1622.62	N-H Bending or Aromatic C=C

1368.46	-	C-H bending (Alkanes)
1236.35	-	C-N Streching (Amines)

6.6 Formulation of Soft Rupturable Beads Containing Vasaka Extract By Using Extrusion-Spheronization

It was found that batch out of all 6 batches, batch no. B4 in terms showed the best results of quality, being able to rupture easily and having a spherical shape. Therefore, batch no. B4 were chosen as the final optimized batches for further testing.

Fig 6.10 Soft Rupturable Beads By Using Extrusion- Spheronization [B4]

6.7 Evaluation of Prepared Soft Rupturable Beads

1. Physical Appearance

The formulated soft beads were uniform in shape and composition, surface texture was smooth and there were significant odour present of Vasaka.

2. Physical Parameters

Table No. 6.3 Physical Parameters of Soft Rupturable Beads

Sr. No	Batches	B1	B2	B3	B4	B5	B6
1	Angle of repose	25.5±0.28	17.7±0.36	19.2±1.05	18.4±1.25	21.3±0.26	20.7±0.8
2	Hausner's ratio	1.26±0.02	1.15±0.03	1.10±0.01	1.13 ± 0.3	1.05±0.05	1.17±0.5
3	Bulk density	1.5±0.025	1.3±0.025	1.25±0.025	1.25±0.22	1.06±0.03	1.23±0.3

4	Tap density	1.5±0.024	1.38±0.021	1.23±0.025	1.22±0.45	1.42±0.05	1.12±0.2

The angle of repose (18.4–25.5°) indicated good to excellent flow, while bulk density (1.25–1.3) reflected adequate material packing.

Hausner's ratio (1.13–1.26) indicated passable to excellent flow properties.

3. Sieve Analysis For Determining Particle Size

Bead particle size was analysed using the sieve shaker method. After sieving, the weight of beads on each sieve was measured, and their percentage of the total weight was calculated. Results are shown in the Table.

Table No.6.4: Particle Size Analysis of Beads Formulation

Formulation	Percent fraction of beads retained on mesh				
Sieve Size	**#10**	**#16**	**#44**	**#60**	**#100**
B1	3.24 ± 0.5	9.23 ± 0.19	1.05 ± 0.31	0.82± 0.07	0.1 ± 0.03
B2	2.45± 0.25	9.36 ± 0.29	0.63 ± 0.23	0.45±0.24	0.05±0.02
B3	3.87±0.55	8.87 ± 0.53	1.24 ± 0.46	0.52± 0.24	0.03 ± 0.02
B4	2.35±0.25	9.56 ± 0.29	0.53 ± 0.23	0.34±0.24	0.07 ± 0.02
B5	3.25 ± 0.25	9.26 ± 0.54	0.83 ± 0.23	0.54 ± 0.34	0.07 ± 0.02
B6	2.54± 0.12	9.34 ± 0.14	1.53 ± 0.25	0.62± 0.25	0.05 ± 0.03

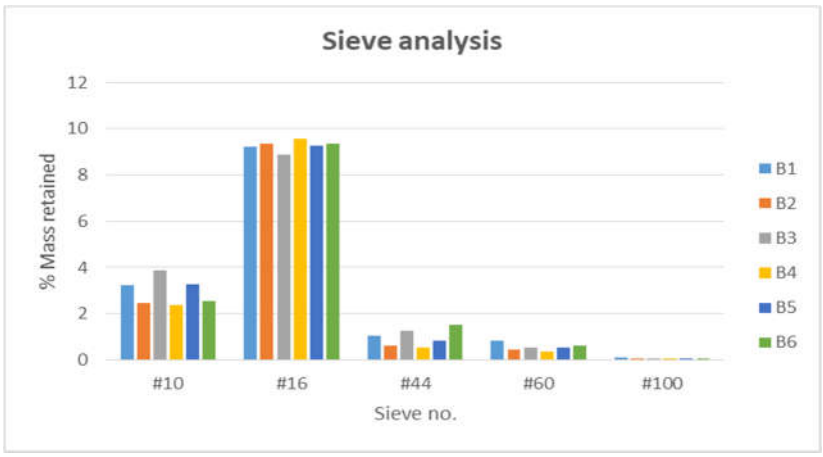


Fig 7.11 Sieve Analysis

Particle size analysis of formulations B1 to B6 showed that ~75% of beads were retained on sieve #16 and ~22% on sieve #10, indicating a bead size range of 1100–1190 µm.

4. Drug Entrapment Efficiency

Formulation B4 met the requirements by fully disintegrating without residue when rubbed on fabric and remaining stable during storage, as shown in Table 6.5.

Table No.6.5: Characterization of Rupturable Beads

Batch	B1	B2	B3	B4	B5	B6
Entrapped efficiency (%)	60.5±0.50	68.5± 0.20	71.5± 0.35	75.5± 0.20	68.9± 0.45	72.5 ±0.50
Feel of foreign matter	Extreme feeling	Extreme feeling	Slight feeling	No feeling	Slight feeling	Slight feeling
Escape of beads	Average	Average	good	Very good	good	good

Batch B4 showed excellent encapsulation efficiency with no foreign particle sensation, making it the optimized formulation.

5. Percentage Drug Entrapped Efficiency Of Optimized Formulation (B4) And Marketed Preparation Himalaya (Adulsa Syrup)

The optimized batch B4 was compared with Himalaya Adulsa syrup (2% Vasaka) for drug entrapment efficiency. Results are shown in the table below.

Table No.6.6: % Percentage Drug Entrapped Efficiency

Assay	Result
% Drug entrapment efficiency (Prepared beads)	93.2%
% Drug entrapment efficiency (Marketed formulation)	98.7%

6. Stability Studies:

Table 6.7: Stability Studies

Evaluation	15 days(40±2^{0}C)	30 days(40±2^{0}C)
%Entrapment efficiency	76.5±0.28	66.35±0.23

Vasaka beads (B4) remained stable at 40 ± 2 °C, with no significant changes in drug content or entrapment efficiency, confirming suitable storage conditions.

7. Summary And Conclusion

7.1 Summary

This project aimed to develop an improved delivery method for Vasaka (Justicia adhatoda), a traditional Ayurvedic remedy for respiratory issues like cough, asthma, and bronchitis. Typically taken as syrups or tablets, Vasaka can be messy or hard to swallow. To address this, soft, round, easily rupturable beads were formulated for use in aromatherapy.

Various formulations and preparation methods, including extrusion-spheronization, were tested. Batch B4 emerged as the best, offering a smooth, round shape, easy rupture upon rubbing, good storage stability, and high drug entrapment efficiency. The beads also showed favorable size and flow properties.

7.2 Conclusion

Overall, this bead-based approach provides a cleaner, more convenient, and modern way to use Vasaka in herbal treatments.

This project aimed to create soft beads using Vasaka extract, a traditional remedy for respiratory issues like cough, asthma, and bronchitis. Unlike traditional syrups, powders, or tablets, these beads are designed for aromatherapy and release the medicine when gently rubbed, offering a cleaner and more convenient option.

Various methods were tested, and the extrusion-spheronization technique proved most effective. Batch B4 was the best, producing smooth, round beads that broke easily on rubbing, had high drug entrapment efficiency, and showed good storage stability.

Overall, Vasaka beads offer a modern, user-friendly alternative to conventional Ayurvedic medicine forms.

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