

Development and DoE-Based Optimization of Dragon Fruit and Kiwi Fruit Herbal Syrup: Formulation, Physicochemical Characterization and Stability Evaluation

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Abstract:

Background: Herbal syrups provide a convenient oral dosage form for incorporation of plant-derived constituents, but suspended herbal extracts may present challenges related to viscosity, sedimentation and physical stability. **Objective:** The study aimed to develop and optimize a herbal syrup containing dragon fruit and kiwi fruit peel extracts using a Design of Experiments approach and to characterize the optimized formulation for physicochemical properties and accelerated stability. **Methods:** Ethanolic and aqueous peel extracts of dragon fruit and kiwi fruit were incorporated into a sucrose-based syrup containing sodium carboxymethyl cellulose (Na-CMC). A two-factor, five-level Central Composite Design consisting of 13 experimental runs was used. Na-CMC concentration (0.16-0.44%) and sucrose concentration (34.4-72.6%) were independent variables, while viscosity and sedimentation were dependent responses. Quadratic models were evaluated by analysis of variance and numerical optimization. **Results:** The viscosity model was significant ($F=29.59$, $p=0.0001$, $R^2=0.9548$) and the sedimentation model was significant ($F=14.64$, $p=0.0014$, $R^2=0.9127$). Numerical optimization identified 0.318971% Na-CMC and 66.6245% sucrose, with desirability 1.000. Predicted viscosity and sedimentation were 364.22 cP and 0.0976969%, respectively. Experimental validation gave 368.45 cP viscosity and 0.10% sedimentation. Conclusion: The CCD-based approach identified an optimized herbal syrup containing approximately 0.32% Na-CMC and 66.62% sucrose with acceptable viscosity and minimal sedimentation. The formulation was selected for accelerated stability assessment at $40\pm 2^\circ\text{C}/75\pm 5\% \text{ RH}$ for six months.

Keywords: Dragon fruit; Kiwi fruit; Herbal syrup; Central Composite Design; Design of Experiments; Na-CMC; Sucrose; Viscosity; Sedimentation; Stability.

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1. INTRODUCTION

Herbal syrups represent a link between traditional plant-based preparations and contemporary pharmaceutical formulation. Their liquid nature supports convenient administration, while incorporation of plant extracts offers a means of delivering phytochemical constituents in an acceptable oral dosage form. However, formulations containing suspended herbal material require appropriate control of viscosity, sedimentation and phase stability.

Dragon fruit (*Hylocereus polyrhizus*) has been described in the source research as a botanical source containing polyphenols, flavonoids and betacyanins, while its peel has attracted interest because of its phenolic content [2-4]. Kiwi fruit (*Actinidia chinensis* var. *deliciosa*) contains flavonoids, carotenoids, polyphenols and other constituents that support its use as a herbal source [6-9].



Figure 1. Dragon Fruit (*Hylocereus polyrhizus*)



Figure 2. Kiwi Fruit (*Actinidia chinensis* var. *deliciosa*)

The research problem identified in the supplied study was the limited application of dragon fruit and kiwi fruit extracts in an optimized herbal syrup, together with the need to establish extraction, formulation and stability conditions. The present research therefore focused on formulation development, DoE-based optimization, physicochemical characterization and stability evaluation.

2. LITERATURE REVIEW

Published work cited in the supplied research provides the scientific basis for selecting dragon fruit and kiwi fruit as herbal sources and for applying formulation optimization to a syrup dosage form. Jadhav and Jadhav ^[3] reviewed dragon fruit as an exotic fruit with pharmacognostic and phytochemical relevance, while Paško et al. ^[4] described dragon fruit as a reservoir of natural polyphenolics. Further identified polyphenols, flavonoids and betacyanins as important constituents of dragon fruit and highlighted the phenolic richness of the peel.

For kiwi fruit, Lee et al^[6]. investigated antioxidant and glycation-inhibitory activities of gold kiwifruit. Brevik et al^[7]. evaluated the effects of golden kiwifruit supplementation on biomarkers of oxidative damage and antioxidant protection, whereas Hunter et al. ^[8] discussed gold kiwifruit in relation to immune support. Khutare and Deshmukh^[9] reviewed medicinal applications of kiwi fruit. These reports, as summarized in the source document, supported the selection of kiwi fruit as a phytochemical-rich component of the proposed herbal syrup.

The source document also cited several studies demonstrating the feasibility of incorporating botanical extracts into syrup formulations. Nerkar and co-workers reported formulation and evaluation of herbal syrups containing turmeric ^[11], ginger ^[12], Indian mulberry (Noni) ^[13] and Arjuna ^[14], and subsequently reported in-vitro anticancer screening of a Vinca extract syrup ^[15]. Collectively, these studies supported syrup as a practical vehicle for botanical extracts and provided a formulation background for the present work.

Response-surface methodology and Central Composite Design have also been used in formulation and process optimization. Rezvanian et al. ^[16] applied a response-surface methodological approach to syrup production, Noorulla et al. ^[17] reported optimization of a herbal syrup using Central Composite Design, and Benali et al. ^[18] used Central Composite Design in the development of a date fruit syrup-based natural jelly. These studies support selecting CCD to simultaneously evaluate the effects of Na-CMC and sucrose on viscosity and sedimentation.

Despite the available evidence for the phytochemical and biological potential of dragon fruit and kiwi fruit and the availability of reports on herbal syrup formulation, the supplied research document identified a specific gap: limited application of these two fruits together in an optimized herbal syrup, insufficient optimization of extraction and formulation variables, and limited information on the stability of such a formulation ^[9,10]. Accordingly, the present work integrated dragon fruit and kiwi fruit peel extracts into a 5.0% w/v herbal-extract load and used a two-factor Central Composite Design to optimize Na-CMC and sucrose with viscosity and sedimentation as the critical physical responses.

Table 1. Summary of Published Literature Relevant to Dragon Fruit, Kiwi Fruit, Herbal Syrup Formulation, and DoE-Based Optimization

Reference	Study / material	Main focus reported in source	Relevance to present study
Jadhav & Jadhav, 2023 [3]	Dragon fruit	Comprehensive review of dragon fruit	Supports pharmacognostic and phytochemical background
Paško et al., 2021 [4]	Dragon fruit	Natural polyphenolics and chemopreventive properties	Supports use of dragon fruit as a polyphenol-rich herbal source
Lee et al., 2011 [6]	Gold kiwifruit	Antioxidant and glycation-inhibitory activities	Supports antioxidant relevance of kiwi fruit
Brevik et al., 2011 [7]	Golden kiwifruit	Biomarkers of oxidative damage and antioxidant protection	Supports antioxidant background
Hunter et al., 2011 [8]	Gold kiwifruit	Immune-support-related review	Supports medicinal/nutritional relevance of kiwi fruit
Khutare & Deshmukh, 2023 [9]	Kiwi fruit	Review of medicinal applications	Supports selection of kiwi as a herbal component
Nerkar & Nagarkar, 2023 [11]	Turmeric herbal syrup	Formulation and evaluation	Supports feasibility of botanical syrup formulation
Nerkar & Ghadge, 2023 [12]	Ginger herbal syrup	Formulation and evaluation	Supports herbal syrup development approach
Nerkar & Gade, 2023 [13]	Noni herbal syrup	Formulation and evaluation	Supports incorporation of fruit-derived extract into syrup
Nerkar et al., 2023 [14]	Arjuna herbal syrup	Formulation and evaluation	Supports use of syrup as herbal delivery vehicle
Rezvanian et al., 2025 [16]	Sweet potato starch syrup	Response surface methodological approach	Supports response-surface optimization
Noorulla et al., 2024 [17]	Phaseolus vulgaris herbal syrup	CCD-based formulation optimization	Directly supports Central Composite Design for herbal syrup
Benali et al., 2015 [18]	Date fruit syrup-based jelly	Central Composite Design	Supports CCD for fruit-based formulation optimization
González-González et	Drug stability	ICH versus accelerated predictive stability studies	Supports accelerated stability framework

al., 2022 [23]

3. AIM AND OBJECTIVES

The aim was to develop and optimize a herbal syrup containing dragon fruit and kiwi fruit extracts. The formulation-focused objectives were to prepare a suitable syrup, optimize the levels of Na-CMC and sucrose, evaluate physicochemical parameters including viscosity, pH and sedimentation, validate the optimized composition, and subject the selected formulation to accelerated stability testing.

4. MATERIALS AND METHODS**Formulation Composition**

The formulation contained ethanolic and aqueous peel extracts of dragon fruit and kiwi fruit. Sucrose was used as the syrup vehicle and sweetener, Na-CMC as the suspending and viscosity-modifying agent, and methylparaben and propylparaben as preservatives. The total herbal extract load was 5.0% w/v.

Table 2. Composition of Dragon Fruit and Kiwi Fruit Herbal Syrup

Ingredient	Quantity per 100 mL	Role
Dragon Fruit Peel (Ethanolic Extract)	1.5 g	Active ingredient
Kiwi Fruit Peel (Ethanolic Extract)	1.5 g	Active ingredient
Dragon Fruit Peel (Aqueous Extract)	1.0 g	Active ingredient
Kiwi Fruit Peel (Aqueous Extract)	1.0 g	Active ingredient
Sodium CMC (Na-CMC)	0.16-0.44 g	Suspending agent
Sucrose	34.4-72.6 g	Vehicle/sweetener
Methylparaben	0.1 g	Preservative
Propylparaben	0.02 g	Preservative
Purified Water	q.s. to 100 mL	Solvent

Preparation of Herbal Syrup

The syrup base and formulation were prepared as described in the supplied research document. Na-CMC was hydrated in a portion of purified water, the herbal extracts were incorporated, and the sucrose syrup base was combined with the extract-polymer system. Methylparaben and propylparaben were incorporated as preservatives, the volume was adjusted to 100 mL with purified water, and the formulation was mixed to obtain a homogeneous syrup.

Design of Experiments

Optimization was carried out using a two-factor, five-level Central Composite Design (CCD). The study type was Response Surface, subtype Randomized, with a quadratic design model, 13 runs and no blocks. The design included factorial points, axial points and five center-point replicates.

Table 3. Central Composite Design Parameters for Optimization of Herbal Syrup

Parameter	Specification
Study type	Response Surface
Subtype	Randomized
Design type	Central Composite
Runs	13
Design model	Quadratic
Blocks	No blocks
Factor A	Na-CMC: 0.16-0.44%
Factor B	Sucrose: 34.4-72.6%
Response 1	Viscosity (cP)
Response 2	Sedimentation (%)

Table 4. Numerical Optimization Criteria for Dragon Fruit and Kiwi Fruit Herbal Syrup

Variable/Response	Goal	Lower limit	Upper limit
Na-CMC (%)	In range	0.16	0.44
Sucrose (%)	In range	34.4	72.6
Viscosity (cP)	Target range	200	480
Sedimentation (%)	Minimize	0	1

Physicochemical Evaluation

Viscosity was determined using a Labman rotational viscometer with spindle 2 at 60 RPM. Three readings were taken for each batch and the average was used for DoE analysis. The pH was determined using a digital pH meter, with a target range of 4.5-6.5.

For accelerated sedimentation testing, a 10 mL aliquot of thoroughly homogenized syrup was transferred to a graduated high-clarity conical centrifuge tube and the initial height (H_o) was recorded. Samples were centrifuged at 3000 RPM for 15 minutes. The height of the separated or settled phase (H_s) was measured and sedimentation was calculated as: $\text{Sedimentation (\%)} = (H_s/H_o) \times 100$. Formulations showing 0-1% sedimentation were considered to meet the research goal.

Accelerated Stability Study

The optimized formulation was transferred into three separate 30 mL amber-colored glass bottles sealed with airtight HDPE screw caps. Samples were stored in a calibrated stability chamber at $40^\circ\text{C} \pm 2^\circ\text{C}$ and $75\% \text{ RH} \pm 5\% \text{ RH}$ for six months. Evaluations were scheduled at baseline, 1 month, 3 months and 6 months.

At each interval, organoleptic characteristics, pH, viscosity, sedimentation, phase separation/non-redispersible caking and qualitative presence of flavonoids and phenols were specified for evaluation. The formulation was considered stable if viscosity variation remained within $\pm 10\%$, pH remained within 4.5-6.5 and sedimentation remained below 1% [23]. The supplied document did not contain a month-wise numerical stability-result table; therefore, no unreported stability values were inserted into this manuscript.

5. RESULTS AND DISCUSSION

Table 5. Central Composite Design Experimental Runs and Observed Responses

Run	Na-CMC (%)	Sucrose (%)	Viscosity (cP)	Sedimentation (%)
1	0.300000	34.4081	150	2.2
2	0.300000	53.5000	282	0.5
3	0.300000	53.5000	288	0.6
4	0.300000	53.5000	290	0.4
5	0.300000	53.5000	285	0.5
6	0.200000	67.0000	210	1.2
7	0.441421	53.5000	455	0.1
8	0.400000	67.0000	410	0.2
9	0.300000	72.5919	380	0.4
10	0.158579	53.5000	95	3.8
11	0.300000	53.5000	285	0.5
12	0.400000	40.0000	240	0.8
13	0.200000	40.0000	120	2.5

Viscosity ranged from 95 to 455 cP and sedimentation from 0.1 to 3.8%. Increasing Na-CMC increased viscosity and reduced sedimentation. High sucrose levels also contributed to the total solid content and body of the syrup. Runs with low Na-CMC or low sucrose showed higher sedimentation, whereas the center-point formulations at 0.3% Na-CMC and 53.5% sucrose produced viscosities of 282-290 cP and sedimentation of 0.4-0.6%, demonstrating reproducibility around the central design region.

Table 6. Analysis of Variance (ANOVA) for the Quadratic Model of Viscosity

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	1.329E+05	5	26589.21	29.59	0.0001
A-Na-CMC	85929.35	1	85929.35	95.61	<0.0001
B-Sucrose	42817.49	1	42817.49	47.64	0.0002
AB	1600.00	1	1600.00	1.78	0.2239
A ²	960.43	1	960.43	1.07	0.3356
B ²	1951.74	1	1951.74	2.17	0.1841
Residual	6291.16	7	898.74	--	--
Lack of Fit	6253.16	3	2084.39	219.41	<0.0001
Pure Error	38.00	4	9.50	--	--
Cor Total	1.392E+05	12	--	--	--

The quadratic viscosity model was significant ($F=29.59$, $p=0.0001$). Na-CMC and sucrose were significant linear terms. The fit statistics were $R^2=0.9548$, adjusted $R^2=0.9225$,

predicted $R^2=0.6802$, $C.V.=11.17\%$ and adequate precision=17.3615. The lack of fit was significant ($F=219.41$, $p<0.0001$), which was acknowledged as a limitation of the model.

Final equation in terms of actual factors: Viscosity = $-445.87605 + 948.80351(\text{Na-CMC}) + 10.80873(\text{Sucrose}) + 14.81481(\text{Na-CMC} \times \text{Sucrose}) - 1175.00000(\text{Na-CMC}^2) - 0.091907(\text{Sucrose}^2)$.

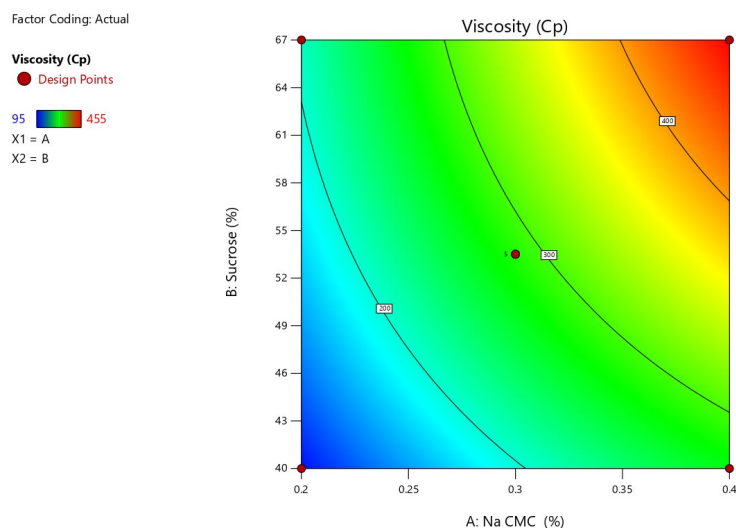


Figure 3. Contour Plot Showing the Effect of Na-CMC and Sucrose Concentration on Viscosity

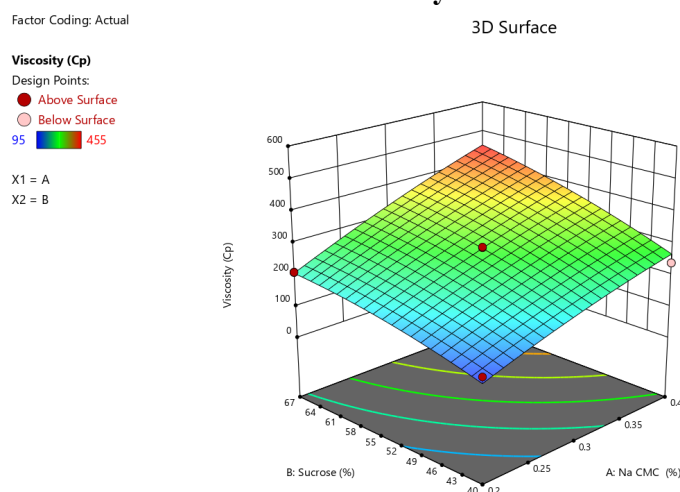


Figure 4. Three-Dimensional Response Surface Plot Showing the Effect of Na-CMC and Sucrose Concentration on Viscosity

Table 7. Analysis of Variance (ANOVA) for the Quadratic Model of Sedimentation

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	13.37	5	2.67	14.64	0.0014
A-Na-CMC	7.87	1	7.87	43.05	0.0003
B-Sucrose	2.47	1	2.47	13.52	0.0079

AB	0.1225	1	0.1225	0.6705	0.4399
A ²	2.61	1	2.61	14.29	0.0069
B ²	0.5750	1	0.5750	3.15	0.1193
Residual	1.28	7	0.1827	--	--
Lack of Fit	1.26	3	0.4196	83.92	0.0005
Pure Error	0.0200	4	0.0050	--	--
Cor Total	14.65	12	--	--	--

The sedimentation model was significant ($F=14.64$, $p=0.0014$). Na-CMC, sucrose and the quadratic term A^2 were significant. The fit statistics were $R^2=0.9127$, adjusted $R^2=0.8504$, predicted $R^2=0.6869$, C.V.=40.56% and adequate precision=10.6742. The lack of fit was significant ($F=83.92$, $p=0.0005$).

Final equation in terms of actual factors: Sedimentation = $17.78520 - 53.60092(\text{Na-CMC}) - 0.248845(\text{Sucrose}) + 0.129630(\text{Na-CMC} \times \text{Sucrose}) + 61.25000(\text{Na-CMC}^2) + 0.001578(\text{Sucrose}^2)$.

Factor Coding: Actual

Sedimentation (%)

● Design Points

0.1 3.8

X1 = A

X2 = B

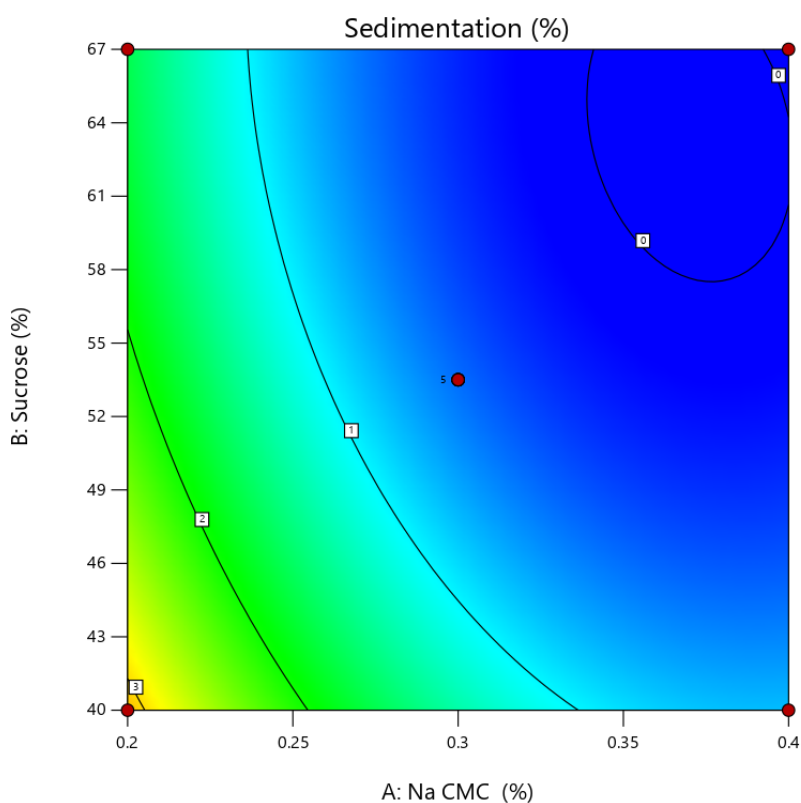


Figure 5. Contour Plot Showing the Effect of Na-CMC and Sucrose Concentration on Sedimentation

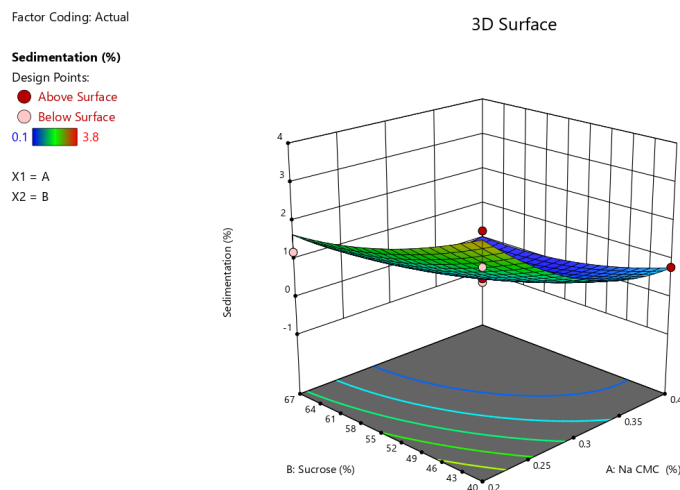


Figure 6. Three-Dimensional Response Surface Plot Showing the Effect of Na-CCMC and Sucrose Concentration on Sedimentation

Numerical Optimization

Numerical optimization identified Na-CCMC at 0.318971% (approximately 0.32%) and sucrose at 66.6245% (approximately 66.6%) as the optimized factor levels. The predicted viscosity was 364.22 cP and the predicted sedimentation was 0.0976969% (approximately 0.1%). The reported desirability was 1.000.

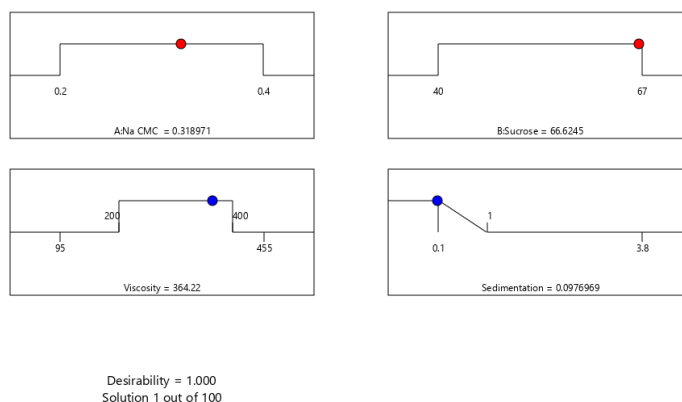


Figure 7. Numerical Optimization and Desirability Plot for the Optimized Herbal Syrup Formulation

Table 8. Optimized Master Formula of Dragon Fruit and Kiwi Fruit Herbal Syrup

Sr. No.	Ingredient	Quantity	Role
1	Dragon Fruit Peel (Ethanollic Extract)	1.5 g	Active ingredient
2	Kiwi Fruit Peel (Ethanollic Extract)	1.5 g	Active ingredient
3	Dragon Fruit Peel (Aqueous Extract)	1.0 g	Active ingredient
4	Kiwi Fruit Peel (Aqueous Extract)	1.0 g	Active ingredient

5	Sodium CMC (Na-CMC)	0.32 g	Optimized suspending agent
6	Sucrose	66.62 g	Optimized vehicle/sweetener
7	Methylparaben	0.1 g	Preservative
8	Propylparaben	0.02 g	Preservative
9	Purified Water	q.s. to 100 mL	Solvent

Table 9. Comparison of Predicted and Observed Responses of the Optimized Herbal Syrup

Response	Predicted Value	Final Observed Value	Prediction Error (%)
Viscosity (cP)	364.22 cP	368.45 cP	-1.15%
Sedimentation (%)	0.097%	0.10%	-3.00%

The final observed viscosity of 368.45 cP showed only a minor deviation from the predicted value of 364.22 cP. The observed sedimentation of 0.10% was within the research target of less than 1%. These observations supported selection of the optimized batch for subsequent evaluation.

Accelerated Stability Evaluation

The source research specified accelerated stability testing for the optimized formulation at $40^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and $75\% \text{ RH} \pm 5\% \text{ RH}$ for six months, with evaluations at 0, 1, 3 and 6 months. The predefined stability criteria were viscosity variation within $\pm 10\%$, pH 4.5-6.5 and sedimentation below 1%. The conclusion of the source document states that accelerated stability studies confirmed physical and chemical integrity under the selected stress conditions.

6. CONCLUSION

The study developed a 5.0% w/v peel-extract herbal syrup containing ethanolic and aqueous extracts of dragon fruit and kiwi fruit. A two-factor Central Composite Design successfully characterized the effects of Na-CMC and sucrose on viscosity and sedimentation. Numerical optimization identified approximately 0.32% Na-CMC and 66.62% sucrose as the optimized formulation composition with a desirability of 1.000. Experimental validation produced a viscosity of 368.45 cP and sedimentation of 0.10%, in close agreement with predicted responses. The optimized formulation therefore achieved the intended balance between pourability and suspension stability. Significant lack of fit for both quadratic models should be considered when interpreting the wider design space, although validation at the selected optimum showed close prediction-to-observation agreement.

7. LIMITATIONS

The study reported significant lack of fit for the quadratic models of viscosity and sedimentation. In addition, the document specified the accelerated stability protocol and summarized stability in the conclusion.

8. ACKNOWLEDGEMENT

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9. CONFLICT OF INTEREST

The authors declare no conflicts of interest related to the publication of this article.

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