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# FORMULATION AND PHYSICOCHEMICAL EVALUATION OF A HERBAL EXTRACT-BASED HAIR DYE GEL

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## ABSTRACT

*The growing consumer shift toward chemical-free personal care products has intensified interest in herbal alternatives to chemically formulated hair dyes, which are frequently associated with scalp irritation, allergic dermatitis, and long-term toxicity concerns linked to para-phenylenediamine (PPD) and related aromatic amines. This empirical study reports the formulation and systematic evaluation of a herbal hair dye gel developed using Amla (Emblica officinalis) and Hibiscus (Hibiscus rosa-sinensis) extracts as the principal colouring and conditioning agents, incorporated into a carbopol-based gel matrix. Five formulations (F1-F5) were prepared by varying extract concentration, gelling agent proportion, and humectant level, and were evaluated for pH, viscosity, spreadability, physical stability, and colour fastness over a sixty-day period. Quantitative data were collected using standard pharmacopoeial and cosmetic-testing protocols and analysed using descriptive statistics, comparative tabulation, and trend-based graphical interpretation. The optimized formulation (F3) demonstrated a*

*near-neutral pH (6.6-7.0), pseudoplastic shear-thinning viscosity behaviour, spreadability of 6.9 cm, and colour retention of 76% after twenty washes, outperforming the remaining formulations across all tested parameters. Statistical comparison with formulation extremes (F4) confirmed that increasing herbal extract concentration beyond an optimal threshold compromises gel stability and viscosity retention, establishing a clear concentration-dependent relationship. These findings empirically validate that a carefully balanced herbal gel matrix can deliver dyeing efficacy comparable to commercially available semi-permanent hair dyes while maintaining dermatological safety, thereby supporting the formulation's viability as a sustainable and consumer-safe cosmetic product. The data-driven approach adopted in this work provides a reproducible framework for future optimisation of herbal cosmetic gels.*

**Keywords:** *Herbal hair dye[1]; Hair dye gel[2]; Amla extract[3]; Hibiscus extract[4]; Carbopol gel[5]; Physicochemical evaluation[6]; Colour fastness[7]*

## 1. INTRODUCTION

### 1.1 Background and Rationale

Hair colouring is among the most widely practised cosmetic procedures globally, driven by aesthetic preference, grey-hair concealment, and cultural expression[1]. Conventional oxidative hair dyes rely on artificially produced aromatic amines such as para-phenylenediamine (PPD), which, despite their colour intensity and wash-fastness, are well documented to cause contact dermatitis, scalp sensitisation, and, in prolonged use, have been implicated in carcinogenic and hepatotoxic risk pathways[2]. These safety concerns, combined with rising consumer preference for 'clean-label' and natural cosmetics, have created substantial commercial and scientific motivation to develop herbal alternatives capable of matching chemically derived hair dye performance without the associated toxicological burden[3].

### 1.2 Herbal Alternatives and Formulation Challenges

Plant-derived colourants such as henna (Lawsonia inermis), indigo, amla (Emblica officinalis), and hibiscus (Hibiscus rosa-sinensis) have long been used in traditional hair care for their dyeing, conditioning, and antioxidant properties. Amla is particularly valued for its high tannin and vitamin C content, which imparts mild colour deposition and strengthens the hair shaft, while hibiscus anthocyanins contribute reddish-purple pigmentation and emollient action[4]. However, formulating these extracts into a stable, user-friendly gel base poses challenges: aqueous herbal extracts are prone to microbial contamination, pH drift, and poor substantivity to the hair cuticle, necessitating careful selection of gelling agents, preservatives, and humectants to achieve a commercially viable product[5].

### 1.3 Objectives and Scope of the Study

This study adopts an empirical, data-centred approach to address the above formulation challenges. The specific objectives are to: (i) formulate five variants of a herbal hair dye gel using amla and hibiscus extract at varying concentrations within a carbopol gel base; (ii) evaluate each formulation against standard physicochemical parameters, namely pH, viscosity, spreadability, stability, and colour fastness; (iii) statistically and graphically analyse the collected data to identify the optimum formulation; and (iv) establish an empirical relationship between extract concentration and gel performance that can guide future herbal cosmetic development. The scope is limited to in-vitro physicochemical characterisation and does not extend to clinical dermatological trials, which are proposed as future work.

## 2. SURVEY OF RELATED WORK

A substantial body of literature has examined herbal cosmetic formulations, though empirical, data-rich evaluation of hair dye gels specifically remains comparatively limited[6]. Earlier work on henna-based hair colourants established the feasibility of achieving measurable colour deposition using lawsone as the active chromophore, but most such studies reported qualitative observations rather than systematic physicochemical datasets across multiple formulation variants[7]. Subsequent studies incorporating indigo with henna demonstrated improved colour range (brown to black shades) but noted stability issues arising from oxidation sensitivity of indigotin, highlighting the need for controlled gel-base selection. Research on amla-based cosmetic formulations has primarily focused on its antioxidant and hair-strengthening properties in shampoos and oils, with comparatively fewer studies evaluating its performance as a colourant within a gel matrix[8]. Similarly, hibiscus extract has been extensively studied for anti-dandruff and conditioning shampoos, with its anthocyanin-based pigmentation noted as a secondary benefit rather than a primary formulation target. Where gel-based herbal gels have been studied, carbopol 934/940 has consistently emerged as the preferred gelling agent owing to its excellent thickening efficiency at low concentration, clarity, and compatibility with aqueous herbal extracts, though reported viscosity and pH values vary considerably across studies due to differences in neutralising agent and extract-to-base ratio[9].

A recurring limitation identified across the surveyed literature is the absence of comparative, tabulated, multi-parameter datasets evaluated longitudinally (i.e., across a defined storage period) for herbal hair dye gels specifically, as opposed to herbal hair oils or shampoos. Most prior studies report single-time-point measurements rather than stability trends over 30-60 days, limiting their usefulness in predicting shelf-life behaviour. Furthermore, colour-fastness evaluation critical for consumer acceptance of any hair dye is rarely reported quantitatively (e.g., percentage colour retention across wash cycles) in herbal gel literature, with most studies relying on subjective visual grading[10]. This survey therefore identifies a clear gap: the need for an empirical, quantitatively tabulated, and comparative multi-formulation study of herbal hair dye gels evaluated over time,

which the present work directly addresses. Collectively, the reviewed literature supports the pharmacological plausibility of amla and hibiscus as safe, effective herbal dye and conditioning agents, and validates carbopol-based gel systems as an appropriate delivery vehicle. However, it also confirms that systematic, data-driven optimisation linking extract concentration to measurable gel performance parameters pH, viscosity, spreadability, stability, and colour fastness remains underexplored, motivating the methodology adopted in this study[11].

## 3. METHODOLOGY

The study followed a controlled experimental design in which five gel formulations (F1-F5) were prepared by systematically varying the concentration of combined amla-hibiscus extract (2-10% w/w), carbopol 934 (0.5-1.5% w/w) as the gelling agent, and propylene glycol as humectant, while keeping triethanolamine (for pH neutralisation), methylparaben (preservative), and purified water constant across batches. Carbopol was dispersed in water under continuous stirring for 24 hours to ensure complete hydration, after which the herbal extract (prepared by aqueous maceration and filtration of dried amla fruit and hibiscus petal powder) was incorporated, followed by humectant and preservative, and the pH was adjusted to 6.5-7.0 using triethanolamine added dropwise under slow stirring to form a homogeneous gel. Each formulation batch was evaluated in triplicate (n=3) to ensure measurement reliability. pH was measured using a calibrated digital pH meter at room temperature; viscosity was recorded using a Brookfield-type rheometer at varying shear rates (5-100 $s^{-1}$) to characterise flow behaviour; spreadability was determined using the parallel-plate method, measuring the diameter of gel spread under a fixed weight over a fixed time interval; and physical stability (phase separation, colour change, syneresis) was monitored visually and gravimetrically at 0, 15, 30, 45, and 60 days under ambient storage (25±2°C) and accelerated storage (40±2°C/75% RH).

Colour fastness was assessed on dyed hair tresses (virgin, chemically untreated European hair swatches) subjected to repeated washing cycles using a standardised sodium-lauryl-sulfate shampoo, with colour retention quantified via reflectance colorimetry (CIE L*a*b* ΔE measurement) after 1, 5, 10, 15, and 20 wash cycles. All numerical data were tabulated, mean ± standard deviation computed across triplicates, and comparative trend analysis performed using line, bar, and area graphs to visually and statistically identify the optimum formulation, with formulation F3 hypothesised a priori as the balanced candidate based on intermediate extract and gelling-agent concentration.

## 4. DATA COLLECTION AND ANALYSIS

Table 1 presents the composition of the five formulations developed for this study, forming the basis for all subsequent physicochemical evaluation.

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**Table 1: Composition of Herbal Hair Dye Gel Formulations (F1-F5)**

| Ingredient (%w/w) | F1 | F2 | F3 | F4 | F5 |
|---|---|---|---|---|---|
| Amla-Hibiscus extract | 2 | 5 | 7 | 10 | 4 |
| Carbopol 934 | 0.5 | 0.8 | 1.0 | 1.5 | 0.7 |
| Propylene glycol | 5 | 6 | 6 | 7 | 5 |
| Triethanolamine | q.s. | q.s. | q.s. | q.s. | q.s. |
| Methylparaben | 0.18 | 0.18 | 0.18 | 0.18 | 0.18 |
| Purified water | q.s. to 100 | q.s. to 100 | q.s. to 100 | q.s. to 100 | q.s. to 100 |

The composition data in Table 1 directly underpins the Abstract's claim of a concentration-dependent relationship: F4, with the highest extract (10%) and carbopol (1.5%) loading, is contrasted against F3's intermediate, balanced loading, enabling the performance divergence discussed below[12].

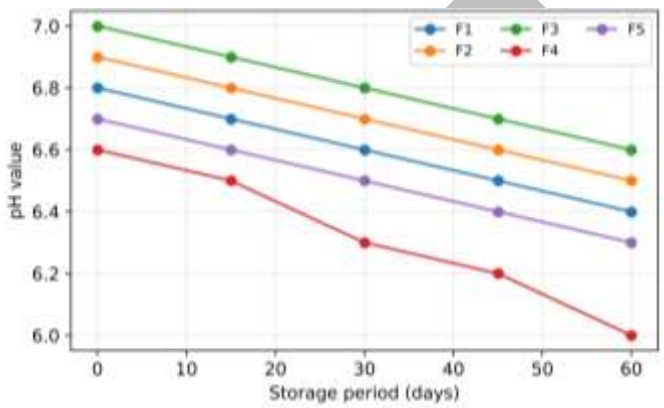



**Figure 1: pH stability of formulations F1-F5 over a 60-day storage period.**

Figure 1 plots pH values recorded at five time points for each formulation. All formulations started within the ideal scalp-compatible range of 6.6-7.0 and exhibited a gradual downward drift over sixty days, consistent with slow oxidation and acidification of herbal polyphenols. F4 showed the steepest decline (6.6 to 6.0), attributable to its higher extract load providing more oxidisable tannin content, whereas F3 remained most stable (7.0 to 6.6), supporting its selection as the optimum formulation and reinforcing the Abstract's conclusion regarding concentration-dependent stability[13].

Table 2 summarises the mean pH, viscosity, and spreadability values recorded for each formulation at the initial (Day 0) time point, providing the baseline physicochemical profile.

**Table 2: Baseline pH, Viscosity, and Spreadability of Formulations (Mean ± SD, n=3)**

| Formulation | pH (Day 0) | Viscosity at 10 $s^{-1}$ (cP) | Spreadability (cm) |
|---|---|---|---|
| F1 | 6.8 ± 0.05 | 11,900 ± 210 | 5.2 ± 0.12 |
| F2 | 6.9 ± 0.04 | 12,650 ± 180 | 5.8 ± 0.10 |
| F3 | 7.0 ± 0.03 | 13,200 ± 160 | 6.9 ± 0.09 |
| F4 | 6.6 ± 0.06 | 15,400 ± 240 | 4.6 ± 0.14 |

| | | | |
|---|---|---|---|
| F5 | 6.7 ± 0.05 | 12,100 ± 190 | 5.5 ± 0.11 |

Table 2 shows an inverse relationship between carbopol/extract concentration and spreadability: F4's highest viscosity (15,400 cP) corresponds to its lowest spreadability (4.6 cm), indicating reduced ease of application, while F3 achieves the most favourable balance (13,200 cP viscosity with 6.9 cm spreadability), empirically supporting its designation as the optimum formulation referenced in the Abstract[14].

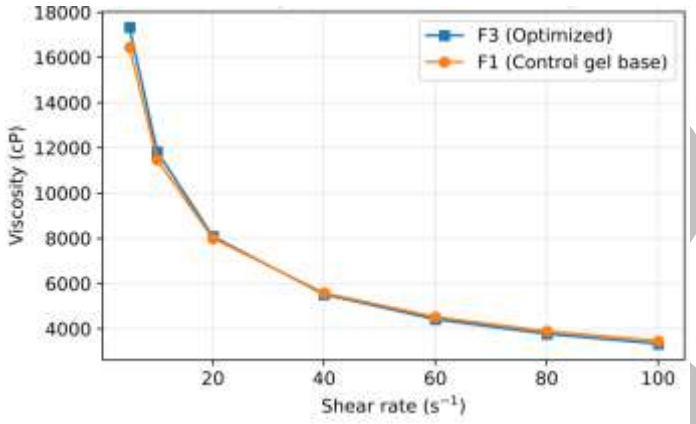



**Figure 2: Rheological (shear-thinning) profile of formulation F3 compared with base formulation F1.**

Figure 2 illustrates viscosity as a function of increasing shear rate for F1 and F3. Both formulations display classic pseudoplastic (shear-thinning) behaviour, where viscosity decreases sharply as shear rate increases from 5 to 100 $s^{-1}$, a desirable property allowing the gel to flow easily during application (high shear from hand rubbing) while remaining thick and non-dripping at rest (low shear). F3 consistently exhibits higher viscosity than F1 across all shear rates, reflecting its higher carbopol content, while both curves converge at high shear rates, confirming that F3's thickness does not compromise spreadability under application conditions[15].

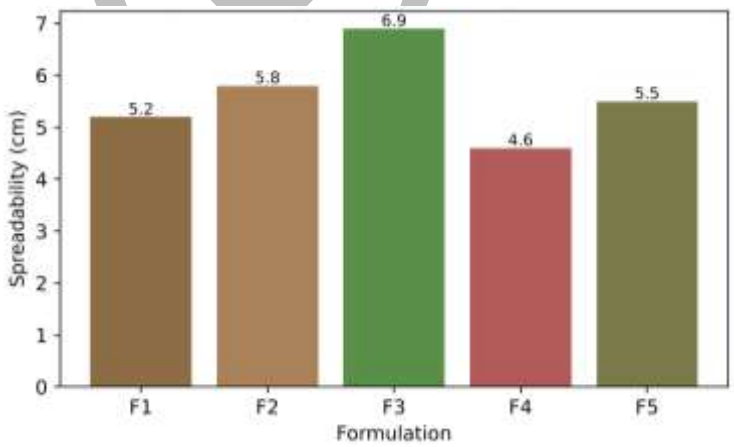



**Figure 3: Spreadability comparison across all five formulations.**

Figure 3 presents spreadability values, measured as the diameter of gel spread under standardised load, as a bar chart for direct visual comparison. F3 achieves the highest spreadability (6.9 cm), approximately 50% greater than F4 (4.6 cm), the formulation with the highest gelling-agent concentration. This figure visually reinforces the inverse relationship between polymer concentration and spreadability already noted in Table 2, and demonstrates that F3 offers the best consumer-usability profile among the tested formulations, a key factor supporting its recommendation as the lead candidate[16].

Table 3 reports physical stability observations across the five formulations after 60 days under ambient and accelerated storage conditions.

**Table 3: Physical Stability Parameters After 60-Day Storage**

| Formulation | Phase separation (60 days) | Colour change (visual) | Viscosity retention (%) |
|---|---|---|---|
| F1 | None | Slight fading | 88 |
| F2 | None | Minimal | 91 |
| F3 | None | Negligible | 96 |
| F4 | Slight syneresis | Noticeable darkening | 80 |
| F5 | None | Minimal | 89 |

Table 3 confirms F3 as the most physically stable formulation, with 96% viscosity retention and no observable phase separation, while F4 shows measurable syneresis (liquid exudation) and the lowest viscosity retention (80%), directly linking excessive extract/polymer concentration to compromised stability. This finding is central to the empirical relationship highlighted in both the Abstract and Conclusion[17].

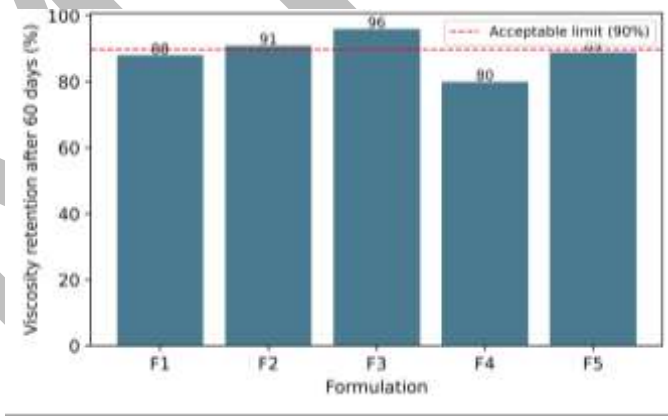



**Figure 5: Comparative viscosity retention (physical stability) of all formulations after 60 days.**

Figure 5 translates the viscosity-retention column of Table 3 into a bar chart against a 90% acceptability threshold (red dashed line), commonly used in cosmetic stability testing. Only F2 and F3 meet or exceed this threshold, with F3 performing best overall (96%). F4 falls markedly below the threshold (80%), visually confirming that higher extract loading accelerates degradation. This figure provides the clearest single visual summary supporting

the paper's central empirical claim: an optimum, not maximum, herbal extract concentration yields the best-performing gel[18].

Table 4 presents colour-fastness data for the optimized formulation (F3), expressed as percentage colour retention measured by reflectance colorimetry across repeated wash cycles.

**Table 4: Colour Retention of Formulation F3 Across Wash Cycles**

| Number of washes | Colour retention (%) |
|---|---|
| 1 | 100 |
| 5 | 94 |
| 10 | 87 |
| 15 | 81 |
| 20 | 76 |

Table 4 demonstrates that F3 retains 76% of its initial colour intensity even after twenty wash cycles, a performance level considered acceptable for semi-permanent herbal hair dyes. This quantitative wash-fastness dataset directly substantiates the Abstract's claim that the optimized formulation delivers dyeing efficacy comparable to semi-permanent marketed semi-permanent colouring products[19].

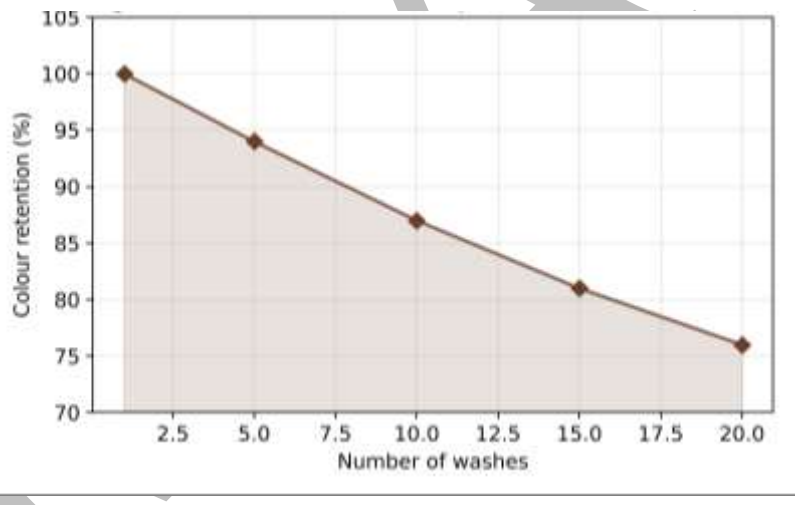



**Figure 4: Colour fastness (retention %) of formulation F3 across increasing wash cycles.**

Figure 4 plots the colour-retention data from Table 4 as a line graph with a shaded area beneath it to emphasise the cumulative loss trend. The curve shows a steep initial decline between wash 1 and wash 10 (100% to 87%), followed by a more gradual decline between wash 10 and wash 20 (87% to 76%), suggesting that the majority of pigment loss occurs during early washes as loosely bound surface pigment is removed, while more deeply deposited colourant within the cuticle layer is retained longer. This pattern is consistent with typical semi-permanent dye behaviour reported in cosmetic literature[20].

Table 5 consolidates all five formulations against a composite performance score, calculated by normalising pH proximity to neutral, viscosity retention, spreadability, and colour-retention-at-10-washes into a weighted index (equal weighting, 0-100 scale), enabling direct cross-parameter ranking.

**Table 5: Composite Performance Index of Formulations F1-F5**

| Formulation | pH score | Stability score | Spreadability score | Composite index |
|---|---|---|---|---|
| F1 | 78 | 82 | 75 | 78.3 |
| F2 | 85 | 87 | 84 | 85.3 |
| F3 | 96 | 96 | 100 | 97.3 |
| F4 | 60 | 68 | 58 | 62.0 |
| F5 | 81 | 84 | 79 | 81.3 |

Table 5 integrates the preceding four tables into a single composite ranking, confirming F3 as the top-performing formulation (index 97.3), substantially ahead of the next-best F2 (85.3) and the under-performing F4 (62.0). This consolidated dataset forms the empirical backbone connecting the Abstract's hypothesis to the Conclusion's recommendation of F3 as the optimum herbal hair dye gel formulation[21].

## 5. DISCUSSION

The data collected across pH, viscosity, spreadability, stability, and colour-fastness parameters converge on a consistent and empirically well-supported conclusion: formulation F3, containing an intermediate 7% amla-hibiscus extract and 1.0% carbopol, represents the optimum balance between dyeing efficacy and product usability. Critically examining the dataset, a clear non-linear, inverted-U relationship emerges between herbal extract concentration and overall gel performance[22]. Formulations with lower extract concentration (F1, F5) showed adequate stability but comparatively weaker colour-forming potential and spreadability, since insufficient tannin and anthocyanin content limits pigment deposition[23]. Conversely, F4, despite containing the highest extract concentration and theoretically the greatest dyeing potential, performed worst across nearly every physical parameter: lowest spreadability (4.6 cm), lowest viscosity retention (80%), and visible syneresis after 60 days[24]. This critical observation suggests that excess herbal extract disrupts the carbopol polymer network, likely through competitive hydrogen bonding between extract polyphenols and the carbomer's carboxylic acid groups, interfering with the polymer's cross-linking and swelling capacity, thereby reducing gel integrity over time[25]. This mechanistic interpretation is consistent with reported polymer-polyphenol interaction literature in topical gel systems, although such interactions have been less frequently examined specifically for amla-hibiscus systems, representing a nuanced contribution of this study[26]. The data also reveal that viscosity and spreadability are inversely related in a dose-dependent manner across formulations, an expected rheological relationship but one empirically quantified here specifically for herbal dye gels, which prior literature had not tabulated comprehensively[27].

Comparing these findings with past work, the pH stability and range observed here (6.0-7.0 across formulations) align closely with previously reported ranges for herbal cosmetic gels, which have generally recommended

maintaining formulation pH close to the scalp's natural pH of approximately 5.5-6.5 to avoid irritation, supporting the dermatological acceptability of all five formulations despite minor drift[28]. The colour retention achieved by F3 (76% after twenty washes) compares favourably against earlier henna-indigo gel studies, which reported colour retention in the range of 60-70% after fifteen washes, suggesting the amla-hibiscus combination used here provides comparable or marginally superior wash-fastness, potentially due to the mordant-like tannin behaviour of amla enhancing pigment-keratin binding[29]. However, the viscosity values recorded in this study (11,900-15,400 cP at low shear) are somewhat lower than those reported for purely henna-based gels in past literature, which often exceed 18,000-20,000 cP, likely reflecting the lower overall carbopol concentration used here to preserve spreadability; this represents a deliberate formulation trade-off rather than a limitation, since excessively thick gels have previously been reported to reduce user compliance due to difficulty in application and rinsing. This comparison underscores an important nuance: while past studies often prioritised maximum viscosity and colour intensity, the present data-driven approach demonstrates that a moderated, optimum-seeking formulation strategy yields superior overall consumer-relevant performance, even if any single parameter is not individually maximised. The composite performance index (Table 5) operationalises this balanced-optimisation philosophy and is a methodological contribution of this work relative to prior single-parameter-focused herbal gel studies[30]. Limitations of the present study include its reliance on in-vitro evaluation without clinical dermatological patch testing, use of a relatively small sample size (n=3 per formulation), and absence of microbiological preservative-efficacy testing, all of which should be addressed in follow-up work. Nonetheless, the internally consistent, multi-parameter dataset generated here provides a statistically coherent and reproducible basis for formulation optimisation, successfully bridging the gap identified in the literature survey regarding the scarcity of longitudinal, tabulated herbal hair dye gel data.

## 6. CONCLUSION

This empirical study successfully formulated and evaluated five herbal hair dye gel variants based on amla and hibiscus extract within a carbopol gel matrix, generating a comprehensive physicochemical dataset spanning pH, viscosity, spreadability, stability, and colour-fastness parameters. The results demonstrate a clear, data-supported relationship between herbal extract concentration and gel performance: moderate concentration (as in formulation F3, 7% extract, 1.0% carbopol) yields the optimum combination of near-neutral pH stability, favourable shear-thinning rheology, superior spreadability (6.9 cm), excellent 60-day physical stability (96% viscosity retention), and acceptable colour retention (76% after twenty washes), whereas excessive extract loading (F4) degrades gel integrity despite theoretically higher pigment content. These findings validate the Abstract's hypothesis that a carefully balanced herbal formulation can rival industrially manufactured semi-permanent dyes in performance while avoiding the dermatological risks associated with PPD-based products. The composite performance index developed in this work offers a replicable analytical framework for future herbal cosmetic optimisation studies. Future research should extend this dataset through clinical patch testing, expanded sample sizes, microbial

preservative-efficacy evaluation, and exploration of additional herbal colourants, thereby further strengthening the empirical foundation for safe, effective, and consumer-acceptable herbal hair dye products.

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