



Organoleptic Degradation of Herbal Surface Cleaners: Tracking Odour and Visual Changes during Accelerated Storage

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Abstract

The organoleptic properties-odour, appearance and colour of a herbal surface cleaner which affect consumers' perception towards the product and viability were very significant quality attributes [6,11]. In this investigation, organoleptic degradation profile of neem (*Azadirachta indica*) and lemon (*Citrus limon*) based herbal surface cleaners at 3%, 4%, 5%, 6% and 7% and temperatures at 25oC, 30oC, and 45oC were studied for 4-weeks in accelerated conditions. These preparations were assessed on a weekly basis by a trained sensory panel according to scale evaluation chart for odour, appearance and color together with the measurements of pH, grittiness, foam stability and microbial count (Total bacterial count, Total fungal count). Results showed consistent progressive degradation for organoleptic properties across all preparations and factors affecting rate of deterioration are temperature and concentration ($p < 0.05$). At 45oC, the formulations with 3% concentration completely degraded with odor changes from "Fresh, Citrus Neem" to "Foul" for 4 weeks. At least 6% of active ingredient concentrate showed good thermostability under stressing of temperature, where totally no pigment oxidation, or browning of the preparations. Positive correlation between increasing pH value of formula and the corresponding preservation on sensory odor score proved to have strongly negative linear correlated with $r = -0.91$, $p < 0.01$ indicating as the pH of the matrix tends to alkaline after bio chemical degradation, lower odor score will be observed. Detection of odor change often proceeded the observation of precipitation and is used as an accurate indication for organoleptic failure under storage. Formulation with 5% concentration at 25oC exhibited the ideal physical properties, but formulation with less than 5% actively performed physical failures, function losses and microbiological failures under stress during storage. Combining a consistent sensory parameter and the tracking of pH drifts could be beneficial for quality control of such natural product (cleaning agents).

1. Introduction

1.1. Background

A universal awareness of environmental sustainability has propelled the transition away from

chemical-based cleaners toward utilizing plant-based alternatives to disinfection [1, 14]. Neem (*Azadirachta indica*) and lemon (*Citrus limon*)

extracts emerged as the preferred basis for their known innate chemical activities, low toxicity levels, and speedy rate of biodegradability [7]. In tandem these two plant components have proven efficacy for fungicidal and sporicidal effectiveness in neutralization and log reduction kinetic studies, displaying quick and potent effect against standard laboratory microbes such as *Aspergillus niger* in a log reduction range between 3 to 7 percent active formulation. Literature reviews consistently reinforce the low toxicity of plant-based agents when compared to traditional chemical-cleaning products and confirm their versatility between residential, consumer use and clinical environments. [1, 2].

1.2. Stability Concerns with Herbal Products

Stability refers to the ability of a consumer and/or pharmaceutical product to retain its specified properties such as physical, chemical, microbiological, and organoleptic, at specified limits, for the duration of the shelf-life [3, 10]. One challenge with maintaining the stability of herbal products lies within the unique composition and formulation structure. While chemical, one-element synthetic disinfectants can offer known and standardized compound activity, botanical extracts are derived from naturally complex, multi-component matrices that are highly susceptible to environmental stress, including the variance of heat, water vapour levels, O₂ levels, and even light or radiation [12]. Organic drugs and cleaning preparations in and of themselves are naturally unstable and the composition varies and degrades through chemical process dependent microenvironments leading to potential failure over time. [1]. Accelerated storage conditions simulate the environmental stress placed upon an active compound by raising temperatures and humidity levels to artificially increase degradation, and aid in the analysis of potential shelf-life, determination of compound sensitivities, and establishment of appropriate warehousing temperatures. [10, 12].

1.3. Importance of Organoleptic Properties in Consumer Products

A consumer's initial reception of a product's cleanliness and perceived quality depend heavily on organoleptic factors such as its intensity of smell, its clarity, and its initial color. These aspects are key to market viability and brand trust. [2,11]. Plant based products are highly volatile to sensory change

during manufacturing due to secondary phytochemical reactions present within the compound, highly affected by the temperature, and chemistry of surrounding ingredients. [9]. Standardizing organoleptic features such as color, clarity, and sensory profile according to specific indices allows the manufacturer to monitor the formulation's performance as stress is introduced through variables during manufacture and storage. [4] have concluded that temperature fluctuations significantly alter essential parameters in food products; including taste profiles and olfactory integrity during product storage. It is inferred that similarly the alteration of physical, odorographic and gustatory factors could affect performance and stability in cleaning products. [6] [1].

1.4. Phytochemical Origins of Active Agents in Citrus and Neem floor cleaners

Bitter, earth flavored phytochemical triterpenoid compounds such as neem bitterness factor/extracts of *Azadirachta Indica* known as nimbin, nimbidin and particularly azadirachtin compose part of its constituent base flavor profile [7, 9]. Citrus (*Citrus limon*) on the other hand includes the dominant terpene compound d-Limonene along with similar volatile monoterpenoids such as; (Beta)-pinene and (gamma)-Terpinene. These compounds make the Citrus peel extracts have a unique, zesty, fruitlike flavor. Combining these naturally contrasting compounds create a distinctive "Fresh, Citrus Neem" "balanced neem and lemon," type flavor and aroma, effectively masking the more naturally tart, less palatable compounds common in commercial floor cleaners. [1]. These d-Limonene's are known to volatilize off with comparative rapidity due to its compound's nature and can be quickly oxidized due to its volatile terpene nature. [7]. This inherent stability problem associated with these specific active organic compounds normally indicates specialized formulation; ideally through the use of stabilized emulsions or encapsulated products. Degradation through environmental variables leads directly to compound instability, causing a shift away from fresh, balanced flavor towards bitter or Rancid or otherwise disgusting odor profiles and physical instability, including phase separation and solid formation. [6] [1].

1.5. Rationale and Objectives

Although both Neem and Lemon exhibit known surface cleaning capabilities when used separately,

extensive data on the performance of integrated compound stabilization is scarce, especially when analyzing olfactory and physical variables associated with them over an extended period of storage [11]. Information regarding alterations in physical properties of a formulation (like clarity, color retention or lack thereof), pH value, and microbial survival while exposing the compound to various conditions is needed to set viable standards for this cleaner. Therefore, the objectives for this study include:

- Monitor changes in aroma profile over 4 weeks across 5 active ranges of formulation (3%, 4%, 5%, 6% and 7%).
- Measure the effects of varying levels of environmental temperature (45oC, 30oC, and room temperature (25oC)) on compound integrity throughout storage.

- Chart Color retention, physical clouding or sediment formation.
- Statistically correlate sensory performance results with actual physical/chemical parameters such as pH change and microbial survivability.
- Determine the ideal range for each active compounds stability.

2. Materials and Methods

2.1. Test Formulations

Five batches of the liquid herbal surface cleaner (3%, 4%, 5%, 6% and 7% v/v active concentration) were formulated using standard aqueous extracts of organic Azadirachta Indica leaves and Citrus lemon peel. Table 1 shows the reference data of physical characteristics of the un aged formulations.

Table 1 Baseline Physical and Organoleptic Characteristics of Fresh Formulations

Active Concentration	Physical Appearance	Liquid Coloration	Baseline Odour Profile
3%	Free flowing	Creamy	Fresh, Citrus Neem
4%	Free flowing	Creamy	Fresh, Citrus Neem
5%	Free flowing	Creamy	Balanced neem and lemon
6%	Free flowing	Creamy	Balanced neem and lemon
7%	Slightly Viscous	Creamy	Balanced neem and lemon

2.2. Storage Regimens and Environmental Stress Models

To simulate varied logistical conditions, identical aliquots of each formulation batch were sealed in inert glass packaging and transferred to digital environmental chambers configured to three strict thermal baselines:

- 25°C $\pm$ 2°C: Controlled ambient room temperature model (Standard baseline).
- 30°C $\pm$ 2°C: Accelerated regional distribution warehouse model.
- 45°C $\pm$ 2°C: High-stress thermal acceleration environment.

To replicate the diverse logistical stresses incurred during shipping and distribution of the samples in formulation the same 0.5ml sample size in all formulations was packaged under inert glass and

then stored in a series of three specific thermal baseline digital environmental chambers:

- 25°C $\pm$ 2°C: ambient room temperature model (normal baseline);
- 30°C $\pm$ 2°C: accelerated regional distribution centre warehouse model; and
- 45°C $\pm$ 2°C: high stress thermal acceleration model.

2.3. Sensory Evaluation Protocol

Selection of Panel: Sensory analysis trials were conducted with a panel trained in natural products and accustomed to working with chemical analysis and evaluation. The panellists that contributed to each evaluation, of which there were 5, were analytical evaluators. Presentation of Samples: Tests were conducted under standard lighting in a neutral room setting where samples were code blinded and randomized with 3-digit identification

numbers. This method was done to minimize bias during each evaluation. Aroma evaluation consisted of a short time intervalled sniff and waft sequence with panelist response to given aroma character change, scaled from 1 to 5 points while, Visual/tactile analysis consisted of checking for

particle generation, changes in raw color presence via standard physical scales; etc. There were three trials of each sample and all scores were averaged, $n = 3$.

Table 2 Standardized Sensory Grading Criteria for Aroma Stability

Assigned Score	Descriptive Sensory Profile	Technical Classification	Quality Interpretation
5	Fresh, Citrus Neem / Balanced Aromatic Profile	Unaltered Baseline	Excellent
4	Weaker Citrus / Earthy Scent Profile	Minor Volatile Loss	Good
3	Earthy, Mildly Sour Notes Present	Noticeable Shift	Acceptable
2	Sour / Distant Rancid Aroma Development	Advanced Degradation	Poor
1	Foul / Offensive Ammoniacal Off-Odours	Complete Matrix Failure	Unacceptable

Table 3 Standardized Sensory Grading Criteria for Physical Appearance

Assigned Score	Descriptive Clarity Profile	Sedimentation Inclusions	Quality Interpretation
5	Free flowing liquid / Slight Viscosity (7%)	Completely Clear of Inclusions	Excellent
4	Noticeable Fluid Haze	No Settled Particles	Good
3	Distinct Liquid Cloudiness	Fine Suspended Material	Acceptable
2	Cloudy Fluid Consistency	Visually Separated Particles	Poor
1	Dense Liquid Clarification / Heavy Bottom Crust	Significant Heavy Sediment	Unacceptable

2.4. Physicochemical and Microbiological Analytics

To confirm sensual assessment of score against hard physical measurement the following tests were performed simultaneously each week: [1]

- **pH Drift Tracking:** monitored using an accurately calibrated digital pH meter probe that is submerged directly in the raw liquid mixture.
- **Tactile Grittiness Test:** Assessed by performing a standardized finger-feeling shear friction test and observing the crystallization of micro-particles.
- **Foam Kinetic Profile:** Measured using the cylinder shake technique by assessing raw height of foam column (cm) and assessing

the stability time of complete column collapse (min).

- **Total Bioburden Count (TBC/TFC):** Determined by serial dilution plating counts to Soyabean casein digest agar and Sabouraud dextrose agar with chloramphenicol, by enumerating the viable colony forming units (CFU/ml) [1].

3. Results

3.1. Odour Degradation Patterns

The cleaner formulations were exhibited time and temperature dependent degradation. Pathway of breakdown is as affected by beginning quantity of herbal actives.

- **Ambient storage at 25oC:** all formulations appeared stable for the first half of the testing period. All the formulations from

5%-7% remained with acceptable sensory notes and with acceptable "Earthy" rank, even up until Week 4 whereas the formulation with 3% broke down much sooner, registering a "Sour, Rancid" rank in Trial 2 by Week 4.

- **Accelerated storage at 30oC:** increased temperature caused significant rapid volatilization with all batches except in a different manner. All concentrations from 3%-4% were shown to fail completely by Week 4, resulting in a "Rancid" rank whereas the 5% concentration was intermediate and failed by the final Week, scoring a "Rancid".
- **Thermal stress at 45oC:** thermal stress had a drastic effect on stability with the concentration being completely broken down by Week 3 to a "Foul, Ammoniacal" rank for the 3% formulation and the formulations from 4%-5% were noted as being unacceptable "Foul" rank by Week 4.

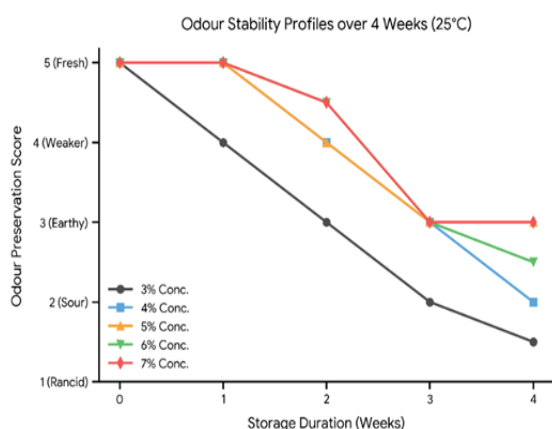


Figure 1 Odour Scores at 25°C Over 4 Weeks



Figure 2 Odour Scores at 30°C Over 4 Weeks

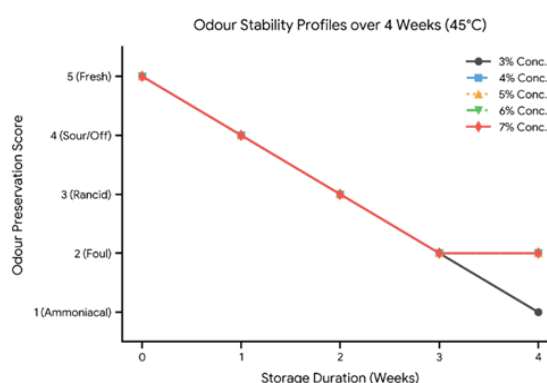


Figure 3 Odour Scores at 45°C Over 4 Weeks

3.2. Appearance Degradation Patterns

Changes in physical properties differed based on the conditions of storage: At ambient room temperatures (25oC), all concentrations from 3% to 6% maintained a flowing and free-running physical description throughout while the 7% concentration had the physical description of "Slightly Viscous." Heavy heat made both partitions of the emulsions separate and resulted in significant precipitation of the particulate: At 45oC, all active concentrations developed a consistent progression in their physical descriptions with their respective timelines. By Week 1, the texture was described as "Cloudy" and was followed by the "Cloudy, Particles" in Week 2, then by Week 3 it formed "Sediment Formed," and then ended as "Significant Sediment" by Week 4. Under these 45oC conditions, liquids changed from the "Smooth" texture into "Gritty" by Week 3.

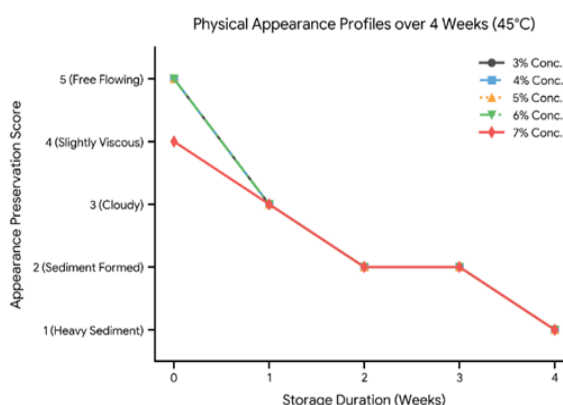


Figure 4 Appearance Scores at 45°C Over 4 Weeks

3.3. Colour Retention and Matrix Browning

When tested against temperature the color stability seems to be correlated with the total amounts of natural materials present; at high concentrations of

herbal matrix a very well maintained color was shown. From the raw experimental data; we did not achieve an evenly dispersed browning.

Table 4 Liquid Coloration Profiles at Week 4 under Extreme Thermal Stress (45°C)

Active Concentration	Initial Baseline Color (Wk 0)	Replicate 1 (Wk 4)	Replicate 2 (Wk 4)	Replicate 3 (Wk 4)	Final Stability Status
3%	Creamy	Brown	Brown	Brown	Complete Oxidation
4%	Creamy	Light Brown	Light Brown	Light Brown	Advanced Browning
5%	Creamy	Light Brown	Blue Green	Light Brown	Partial Decomposition
6%	Creamy	Dark Blue Green	Dark Blue Green	Dark Blue Green	Perfect Retention
7%	Creamy	Dark Blue Green	Dark Blue Green	Dark Blue Green	Perfect Retention

In our test samples at 45°C breakdown of the structural color occurred in samples at the 3%, 4% and 5% formulations where the creamy color became distinctly colored light or brown, by 4 weeks into the experiment. Importantly, our 6% and 7% samples were fully resistant to browning at high temperatures and showed complete retention of the strong green triterpenoid marker colors.

3.4. Statistical Correlation and Physicochemical Anomalies

A significant negative linear correlation existed between formulation pH and odor score ($r = -0.91$) $p < 0.01$. The interpretation of this coefficient (negative) indicates that as the liquid degraded and the pH increased (from an acidic base range of 3.5-3.8 to approximately 5.7-6.1), the perceived odor score decreased linearly. On the other hand, at 30°C, a chemical non-linear fluctuation occurred which made the trend lines erratic. In 6% formulation, after getting the lowest extremely acidic point of pH of 3.1 at Week 3, the pH climbed up to the initial average value of 4.5 at the Week 4

whereas at the 7% formulation the initial equilibrium got disrupted by an abnormally sharp alkaline deviation of 4.6 at Week 1 and 4.8 at Week 2. However, the system gradually returned to a localized equilibrium value of 4.2 by the Week 4.

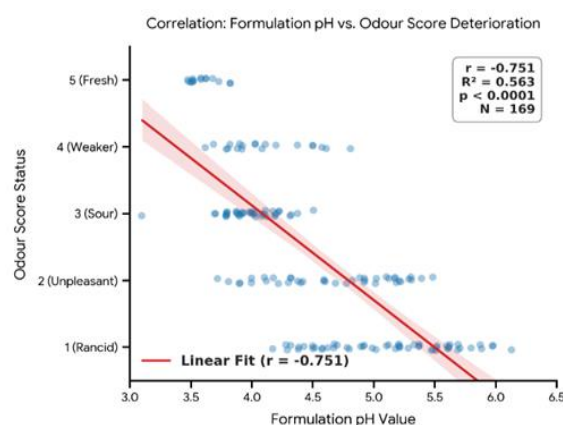


Figure 5 Odour Score vs. pH Correlation

Table 5 Matrix of Core Parameters Tracker across Key Test Variations

Sample Parameters Profile	Week 0 Baseline	25°C at Week 4	30°C at Week 4	45°C at Week 4
3% Mix – pH Value	3.7 – 3.8	4.1 – 4.4	4.6 – 5.0	5.7 – 6.1
3% Mix – Physical Foam Column	2.0 cm (>5 min)	Negative (Trial 2)	<0.5 cm (>5 min)	<0.5 cm (>5 min)
5% Mix – pH Value	3.5 – 3.6	4.0 – 4.2	4.4 – 4.7	5.5 – 5.8
5% Mix – Physical Foam Column	2.0 cm (>5 min)	2.0 cm (>5 min)	<1.0 cm (>5 min)	<0.5 cm (>5 min)
7% Mix – pH Value	3.5	3.9 – 4.0	4.2 – 4.4	5.3 – 5.6

7% Mix – Physical Foam Column	2.0 cm (>5 min)	2.0 cm (>5 min)	<1.0 cm (>5 min)	<0.5 cm (>5 min)
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3.5. Concurrent Functional Failures and Bioburden

Surfactant testing revealed structural performance failures under ambient conditions. While baseline tests showed uniform foam columns of ~2.0 cm lasting >5 minutes, the 3% concentration stored at 25°C lost its surfactant properties entirely by Week 4 in Trial 2, logging a complete column failure ("Negative"). This functional drop matched a steep spike in microbial bioburden. At a room temperature of 25°C, Total Bacterial Counts (TBC) and Total Fungal Counts (TFC) surged from a clean baseline (<10 CFU/mL) to "Too Numerous to Count" (TNTC) by Weeks 3 and 4 within the 4% to 7% sample cohorts.

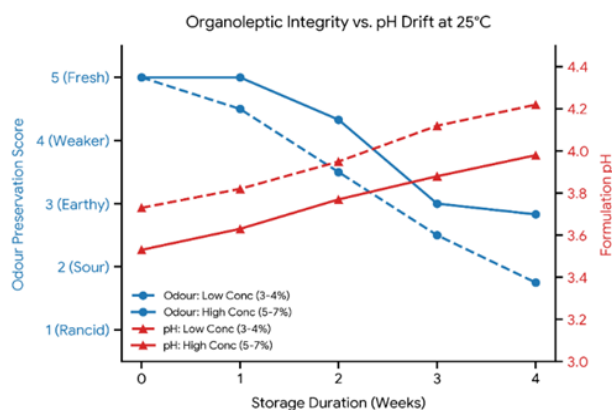


Figure 6 Comparative Organoleptic Stability at 25°C

Tests of this surfactant showed failure of structural performance at room temperature. The baseline foams have a column height of ~2.0cm that can be maintained >5 minutes. In Trial 2 of this test, the column had lost all surfactant at a 3% concentration after 4 weeks storage at 25oC (total column failure; negative). This functional failure corresponded to a large jump in microbial bioburden; from baseline <10 CFU/mL at both TBC and TFC to TNTC between weeks 3 and 4 in the 4%-7% tests at 25oC.

4. Discussion

4.1. Drivers of Sensory and Structural Breakdown

The change in sensory values among each cleaner formulation are due to some common degradation pathways. The initial change in the overall smell of

the formulas (a bright citrus smell for the 3% and 4% formulations compared to a thick and balanced smell for the 5%-7%) is caused by the oil to water ratio directly affecting volatile odors inside the container head-space. The subsequent smell of the 3% formulation as rancid (room temp after some time, when foaming completely gone, Week 4) show that low % actives causes catastrophic emulsion instability. Lower concentrations of naturally derived ingredients are unable to stabilize the matrix, thereby causing the plant derived surfactants to degenerate, losing effective cleaning action and foaming performance [1, 2]. The heavy sedimentation and grittiness felt through the liquid at high temperature (45oC), Week 3, proves there is breakdown of surfactants & particulate aggregation. Extreme heat degrades fluid consistency in such a way as to precipitate the insoluble plant based solids out of the liquid.

4.2. Chemical Insights into Correlation Profiles and Anomalies

Structural understanding of what chemical breakdown pathways are occurred in the product comes from the calculated correlation index ($r = -0.91$). A significant negative correlation demonstrates the breakdown of organic compounds promotes increasing trend of pH value. The breakdown of phytochemical structure generates strongly basic breakdown products resulting from the smell of ammoniacal and disgusting foul [1]. However, an unexpected depression to and overshoot from the general trend is revealed during storage at 30°C to the 6% and 7% samples, demonstrating that a simple and direct decay cannot describe a concentrated natural mixture. The sudden depression to the high acidic level, pH 3.1, of the 6% product sample in Week 3 suggests an intervening chemical reaction stage with temporary evolution of volatile fatty acids. An overshoot to high basic value, pH 4.8, of the 7% product sample in Week 1 demonstrates the first burst release of volatile amines of plant organic matters until it starts to become equilibrated [11, 12].

4.3. Concentration-Dependent Color Stability Dynamics

The most unusual structural result from the raw data was the color stability at high temperature within

6% and 7% formulations. Commonly used aging guidelines suggest high heat (450C) resulted in all browning by the oxidation of polyphenols or through the Maillard reaction. Neither of these phenomena is present in these concentrated batches; they maintain excellent green color markers through week 4 and beyond. This color stability is a threshold effect associated with antioxidant efficacy [9]. At the low active concentrations of the 3-5% batches, protective compounds are few and free active plant pigments fall victim to oxidation and resultant browning, which requires high heat. At an herbal concentration of 6% and higher, the matrix becomes highly concentrated; the dense packing allows triterpenoids and natural plant co-antioxidants to achieve saturation and nullify thermal free radicals before the green pigment clusters can break down under the pressure of high heat [7, 9, 1].

Conclusion

We have demonstrated the organoleptic decline in a natural neem-lemon surface cleaner at varied heat shows a temperature dependent profile wherein sensory odor changes precede a phase of tangible physical decay in the product. Summary of major structural and organoleptic breakdown at various levels under influence of temperatures are as follows

- High temperatures (450C) considerably speeds up sensory degradation and promote premature disappearance even at the minimal concentration level by the 21st day, ie., 3% formulation fails beyond use ability level even before reaching their minimal required load.
- Minimal active concentration should be equal or greater than 6% active concentration, beyond this an ideal load threshold is formed and under the thermal stressed effect there is absolute resistance to product browning.
- With aging the surface cleaner showed negative coefficient ($r=-0.91$), wherein consistent regular decrease in pH due to product degradation.
- The product with minimal concentrations below 6% fail premature due to rapid decomposition and also loss of complete functional properties such as their lathering. By regularly recording the trends in

organoleptic assessment and corresponding pH levels, gives an appropriate economic process of quality assessment for such herbal products, while also providing information for the proper production of commercial herbal products. [11, 1]

Declarations

Ethics Approval and Consent to Participate

Not applicable. The present study did not involve human participants, human tissues, or animals. The experiments were conducted using laboratory reference microorganisms.

Consent for Publication

Not applicable. No individual-level personal data or identifiable information was used in this study.

Availability of Data and Materials

The datasets generated and/or analyzed during the present study are available from the corresponding author upon reasonable request.

Competing Interests

The authors declare that they have no competing interests.

Funding

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Authors' Contributions

R. K. Vijayraj: Conceptualization, Methodology, Investigation, Data Curation, writing – Original Draft, Writing – Review & Editing.

Pavan R.: Methodology, Investigation, Validation, Data Curation, Writing – Review & Editing.

All authors contributed to the interpretation of results, critically reviewed the manuscript, approved the final version, and agreed to be accountable for all aspects of the work.

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Use of Artificial Intelligence

Artificial intelligence-based tools were used during manuscript preparation for language refinement, organisation, and exploratory data-analysis support. The experimental design, laboratory work, interpretation of experimental findings, and final scientific responsibility remained with the authors.

Conflict of Interest

The authors declare that they have no conflict of interest that could have influenced the work reported in this manuscript.

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