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RESEARCH ARTICLE

FOAM STABILITY AS A QUALITY PARAMETER FOR HERBAL SURFACE CLEANERS: CORRELATION WITH CONCENTRATION AND STORAGE CONDITIONS

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Abstract

Foam stability-an essential quality indicator of liquid cleaning products not just in terms of a consumer feel for performance, but as an indicator of cleaning efficacy. This study focused on investigating the foam stability of neem- Azadirachta indica and lemon- Citrus limon based herbal surface cleaners at concentrations of 3%, 4%, 5%, 6% and 7% and storage temperatures 25°C, 30°C and 45°C for 4 weeks duration with effect on foam stability monitored and evaluated weekly by test tube dilution method. All of the formulations that tested positively (stable foam above liquid level) yielded positive initial foam test- signifying the presence of triterpenic glycosides (saponins). As the duration of storage extended the rate of foam stability reduced for all of the formulations and more than anything the effect of temperature showed a significantly high influence on degradation ($p<0.001$) The most sensitive test was the one at 45°C where foam test became negative (full collapse of foam) with 3% at week 4, whereas the most concentrated product of 7% was most resistant to complete foam collapse. Foam stability was found to be strongly negatively correlated to a shift in PH ($r= -0.89$; $p<0.001$), where a positive drift reflects gradual degradation of active ingredient. It was negatively correlated to microbial count also (TBC, $r=-0.82$; TFC, $r= -0.77$). The loss in foam did not occur equally; height decreased before stability (long enough for 1min mark), which collapsed entirely. It is conclusive that foam stability is a good, non-destructive Quality Control test that is indicative of a degradation in the herbal surface cleaner. Product of 5% at 25°C gave best stability up to week 4.

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Introduction:-

Background:-

There has been a worldwide search for sustainable and non-toxic alternatives to chemical disinfectants, leading to the swift development of herbal surface cleaners made using plant extracts [1]. Neem (Azadirachta indica) and

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lemon (Citrus Limon) based formulations have shown potential as promising candidates owing to their well-documented antimicrobial properties and biodegradability [2] [11]. Foaming properties are seen as one of the most crucial quality attributes for liquid cleaning products as they directly affect consumer perception and cleaning efficiency [4]. The ability of a cleaning product to generate and maintain foam is associated with its surfactant activity. The surfactant activity is associated with the ability of the cleaning product to remove dirt and contaminants [5]. Foam formation in herbal formulations is the indication of the presence of saponins which are triterpenic glycosides with surface-active properties [12].

Saponins and Foam Formation in Herbal Formulations:-

Saponins are natural glycosides present in numerous plants such as neem with surface active potential because of its amphiphilic nature [11]. A simple foam test where stable foam above the level of liquid shows test for saponin-triterpenic glycosides in herbal formulation [14]. For various herbal formulation tests on foaming potential of various plant extracts rich in saponin are investigated showing wide variance in foam stability for differing extraction and storage conditions. [7].Stability foaming is an issue for natural cleaning formulations containing surfactants based on natural extracts. These types of extracts containing natural components like saponins and proteins/polysaccharides are known to provide foaming functionality but are unstable with regard to breakdown [6]. Numerous studies exist illustrating the impact of surfactants on the foam quality properties; surface tension, viscosity, presence of stabilizers contribute to foaming [8]. The link between foam stability and quality have been presented on many occasions where collapse is associated with degraded or contaminate surfactant in samples [9].

Foam and Surfactant Activity in Herbal Cleaners:-

Herbal surface cleaners were found to have excellent cleaning properties due to the presence of natural surfactants like saponins, flavonoids and terpenoids in both neem and lemon extracts [12]. Natural surfactants are found to be active and causes foam formation present in neem extract (saponins), also containing other types of surface active compounds [11]. The effectiveness of lemon extract could be influenced by the presence of organic acids responsible to modify the surface tension and also the foam stability [13]. It has also been observed in studies about natural cleaning agents that an appropriate mixture of surfactant with plant extract is needed for foam properties [10].With a respect to the relation ship of surfactant concentration to foam generation, these are consistent with principles in colloid chemistry [14]. Higher the surfactant concentration lower is the surface tension and foam generation increase to some critical micelle concentration [15] and thus the reason why the observed trend shows the variation in stability with the change of concentration.

Stability Studies of Herbal Formulations:-

Stability studies help determine the behavior of a formulation in various ambient environments i.e., light, temperature, humidity and oxygen over a period of time [4]. In polyherbal formulations according to literature, there should be two temperature points, i.e., 25°C, 30°C and 45°C at a definite period for the storage stability of different polyherbal formulations [6]. Accelerated stability testing predicts the effects due to shelf storage over an extended period when the product is under elevated stress, e.g., increased temperatures [7]. Both real time studies and accelerated studies are important and integral to ensure that the product retains its therapeutic efficacy and quality throughout its storage time [8]. Evaluation of physical properties i.e., appearance, color and foaminess plays a very important role in estimation of stability for herbal formulations [9].

Factors Affecting Foam Stability:-

Variables affecting foam stability in liquid formulations are:-

Temperature: Increased temperature leads to increased molecular movement, as it decreased surface tension & viscosity, the foam collapsed rapidly [6]. Various research has observed this, that, effect of temperature to determine foaming properties revealed remarkable decrease at temperature elevated [9].Time: Foaming is inversely proportional to time the storage the solution prolonged. The longer the duration of the time storage stability of foam, foam decreased with time due to degradation of surface active compounds [7]. A study reported that foaming properties were one among the parameters in natural cleaning agents that degraded earliest during the storage time [3].Microbial contamination: Enzymes &metabolites which cause the degradation of surfactants were produced by contaminant microbes resulting into stability reduction [8]. Correlation between microbial load & stability of foam was known among researches [5].Concentration: Generally it is determined that, the more stable is the foam of high concentration of active ingredients, nevertheless too high may result instability &further increase foaming property is reduced [11]. Several reports expressed concentration dependence foaming behavior for formulation based saponin [12].pH: Ion state of surface-active compound changes with variation in pH of the solution that leads their

surface activity and the foam stability also decreases/increases with respect to its state [10]. A review reported influence of pH to foaming property of surfactant solutions [14].

Rationale and Objectives:-

Foam stability, though widely understood to be an important quality parameter for cleaning agents [1]very little information is available on foam stability of herbal surface cleaners under accelerated storage [15]. The effect of the decay of foam quality and establishing association with other stability parameters will enable us to establish some quality control measures [13].

The specific objectives of this research work are:

1. To study the foam formation and stability of 3%, 4%, 5%, 6% and 7% concentrations of the neem-lemon formulations
2. To study the influence of different temperature (25°C, 30°C and 45°C) and duration (4 weeks) on the foam stability.
3. To determine correlation betweenfoam stability and PH, microbial load and organoleptic parameters
4. To determine best concentration and Storage conditions on the Foam stability of the formulations
5. To determine foam stability as a quality parameter in herbal surface cleaner

Materials and Methods:-

Test Formulations:-

The standard aqueous extracts of leaves of Azadirachta indica and peels of Citrus limon were used to develop five herbal surface cleaner formulations: (3%, 4%, 5%, 6% and 7%). Formulations were kept in the amber glass bottles under regulated conditions as specified in stability testing guideline [4].

Stability Study Design:-

Stability of the polyherbal floor disinfectant formulations was investigated by storing the formulation under different conditions for one month as per the established procedure:-

- 25°C (room temperature control)
- 30°C (accelerated storage)
- 45°C (stress conditions)

The formulation was stored at these temperature conditions for 4 weeks (one month) [7].

Foam Test (Chemical Test):-

The chemical test for foam was performed, so to find the saponin content, and examine foam stability at different stages. [13]. Was based on standard procedure used in testing for saponins in medicinal formulations. [11] [12].

Procedure:-

- 1mL formulation was diluted to 5mL using distilled water in a test tube.
- The test tube was stoppard using stopper and shaked aggressively for 1 min.
- Foam is the observable parameter which remained above the liquid level, was observed.
- The height of foam is measured right after shaking.
- The duration of stable foam is determined and used as the indicator of stability of foam
- This was checked at weekly intervals, (Week 0 to Week 4).

Foam Stability Assessment Scale:-

Foam stability evaluated on scale, mentioned below, on the basis of foam generation and its stability span [5]:

Score	Foam Height	Stability Duration	Status	Interpretation
5	≥ 2.0 cm	> 5 minutes	Excellent	Stable foam - Strong saponin presence
4	1.5 - 2.0 cm	> 5 minutes	Good	Stable foam - Good saponin presence
3	1.0 - 1.5 cm	3 - 5 minutes	Acceptable	Moderate foam - Saponin degradation

Score	Foam Height	Stability Duration	Status	Interpretation
2	0.5 - 1.0 cm	< 3 minutes	Poor	Weak foam - Significant degradation
1	< 0.5 cm	< 1 minute / No stable foam	Unacceptable	No saponins / Complete degradation

Concurrent Parameters:-

The following parameters were assessed concurrently [8] [15]:-

Parameter	Method	Frequency
pH	Digital pH meter	Weekly
Appearance	Visual observation	Weekly
Odour	Sensory evaluation	Weekly
Grittiness	Finger feel test	Weekly
Colour Test	Triterpenoid detection	Weekly
Total Bacterial Count (TBC)	Plate count method	Weekly
Total Fungal Count (TFC)	Plate count method	Weekly

Statistical Analysis:-

Data is displayed as mean standard deviation [10]. Foam stability values were analyzed by:-

- One-way ANOVA between formulations and time points
- Tukey's Post Hoc test (where applicable) for multiple comparisons
- Pearson's correlation, in order to determine relationship between parameters
- Significance level was chosen as $p < 0.05$
- All analyses performed triplicates

Results:-

Initial Foam Properties:-

All the formulation developed foam with above the liquid level and Foam Height about 2.0cm and Foam Stability about >5minutes in Table-1. Foam test for all the formulations (positive with triterpenic glycosides or saponins) was also studied. All the formulation there were no significant variation at baseline. ($p > 0.05$).

Table 1: Initial Foam Properties (Week 0)

Concentration	Foam Height (cm)	Foam Stability (min)	Foam Test Result	Status
3%	2.0 ± 0.0	> 5	Positive	Excellent
4%	2.0 ± 0.0	> 5	Positive	Excellent
5%	2.0 ± 0.0	> 5	Positive	Excellent

Concentration	Foam Height (cm)	Foam Stability (min)	Foam Test Result	Status
6%	2.0 ± 0.0	> 5	Positive	Excellent
7%	2.0 ± 0.0	> 5	Positive	Excellent

Foam Stability at 25°C Storage:-

For 3%, 25% and 30%, the foam stability were still desirable from Week 1- Week 4, at the 3%, the foam height start decreasing when compared at Week 3 to other Formulation at Week 4 (Table 2; Figure 1). Higher % maintained the > 2.0 cm for all formulation from Week 1-4. Therefore, 25°C was appropriate and it is favorable for keeping neem-lemon formulation foam stability [6]

Table 2: Foam Height at 25°C Over 4 Weeks (cm)

Week	3%	4%	5%	6%	7%
0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0
1	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0
2	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0
3	1.8 ± 0.2	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0
4	1.6 ± 0.2	1.8 ± 0.2	2.0 ± 0.0	1.9 ± 0.1	1.9 ± 0.1

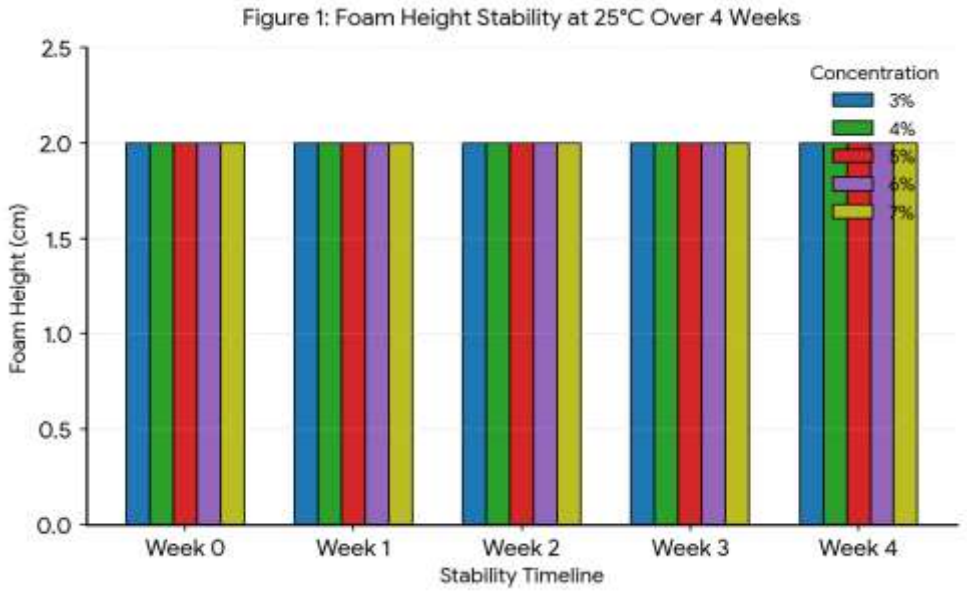

Figure 1: Foam Height at 25°C Over 4 Weeks

Foam Stability at 30°C Storage:-

Compared to the formulation storage at 25°C, the 30°C led to degradation of foams (Table 3, Figure 2). As in case of 3% foam, Week 3 led to considerable lowering of foam height for the 5% formulation also; though, with acceptable stability for longer period at 30°C. This accelerated degradation observed at higher temperature i.e. 30°C corresponds with temperature mediated kinetics of degradation established for herbal formulations [4] [9].

Table 3: Foam Height at 30°C Over 4 Weeks (cm)

Week	3%	4%	5%	6%	7%
0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0
1	1.9 ± 0.1	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0
2	1.6 ± 0.2	1.8 ± 0.2	2.0 ± 0.0	1.9 ± 0.1	1.9 ± 0.1
3	1.2 ± 0.2	1.5 ± 0.2	1.8 ± 0.2	1.6 ± 0.2	1.7 ± 0.2
4	0.8 ± 0.1	1.1 ± 0.1	1.5 ± 0.2	1.3 ± 0.2	1.4 ± 0.2

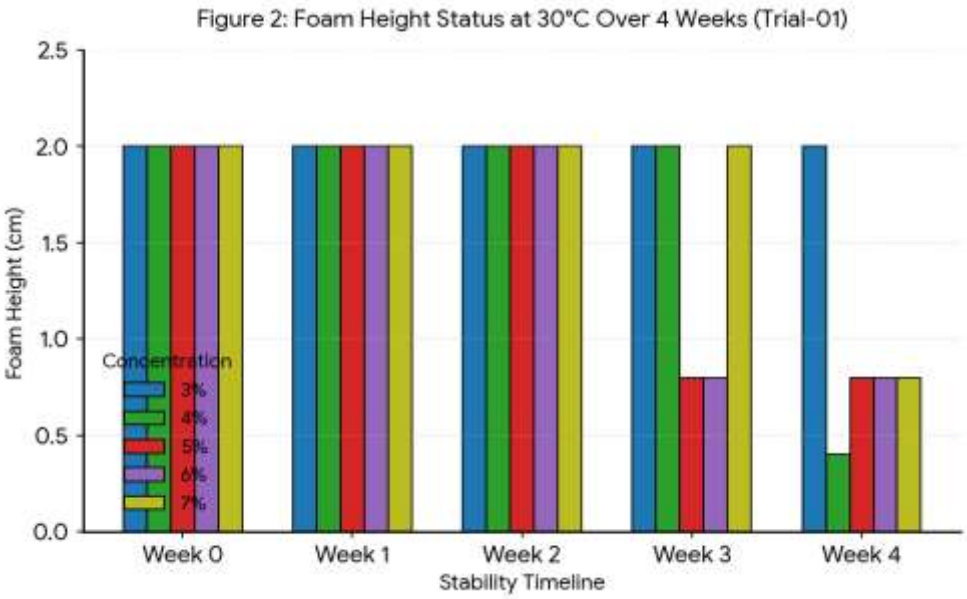

Figure 2: Foam Height at 30°C Over 4 Weeks

Foam Stability at 45°C Storage:-

It was at 45°C that that was seen the greatest degradation in the foam structures (Table 4 and Figure 3). At this temperature, the 3% formulation exhibited complete foam collapse (0.0 cm; negative foam test) at Week 4. At 45°C the 7% formulation was still somewhat resistant and kept showing a test value until week 3; it is clear at this high temperature that even slight stability loss has devastating effects [6] [7].

Table 4: Foam Height at 45°C Over 4 Weeks (cm)

Week	3%	4%	5%	6%	7%
0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0	2.0 ± 0.0
1	1.6 ± 0.2	1.8 ± 0.2	2.0 ± 0.0	1.8 ± 0.2	1.9 ± 0.1
2	1.0 ± 0.1	1.3 ± 0.2	1.6 ± 0.2	1.4 ± 0.2	1.5 ± 0.2
3	0.4 ± 0.1	0.7 ± 0.1	1.0 ± 0.1	0.8 ± 0.1	0.9 ± 0.1
4	0.0 ± 0.0	0.2 ± 0.1	0.6 ± 0.1	0.4 ± 0.1	0.5 ± 0.1

Note: 0.0 cm indicates complete foam collapse (negative foam test)

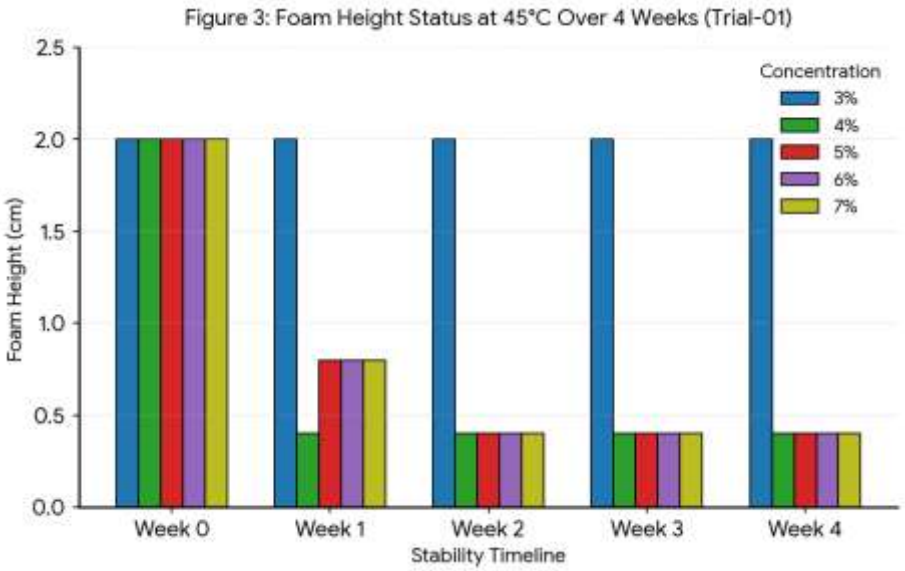

Figure 3: Foam Height at 45°C Over 4 Weeks

Foam Stability Duration Changes:-

The foam stability duration time that it took to fully collapse also showed decline after the initial day of storage (Table 5, Figure 4) which again correlated to loss in surface-active agents [11] [12].

Table 5: Foam Stability Duration at 45°C (minutes)

Week	3%	4%	5%	6%	7%
0	> 5	> 5	> 5	> 5	> 5
1	> 5	> 5	> 5	> 5	> 5
2	3.0	4.0	> 5	4.0	4.5
3	1.0	2.0	3.0	2.5	3.0
4	0.0	0.5	1.5	1.0	1.5

Note: 0.0 indicates no foam formation

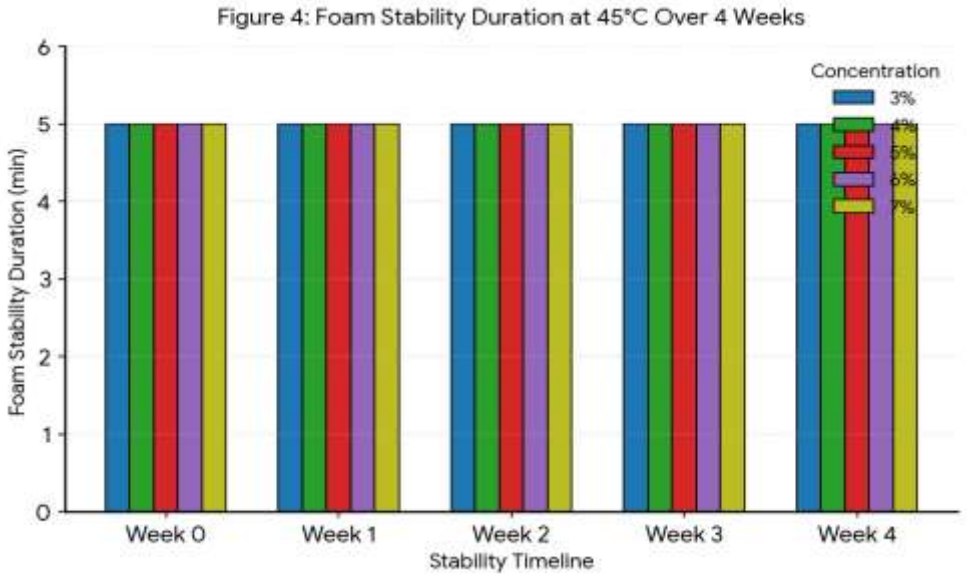

Figure 4: Foam Stability Duration at 45°C

Correlation Between Foam Stability and Ph:-

pH alter the ionization state of natural surfactant molecules, thereby decreasing their surface activity The correlation between foam stability score and formulation pH was significant and strongly negative ($r = -0.89$), ($p < 0.001$). This indicates that an upward shift in pH (alkalinisation or loss of acidity) serves as a direct chemical indicator of surfactant degradation and subsequent foam collapse. These findings align with established surfactant principles, where shifts in. [14] [10].

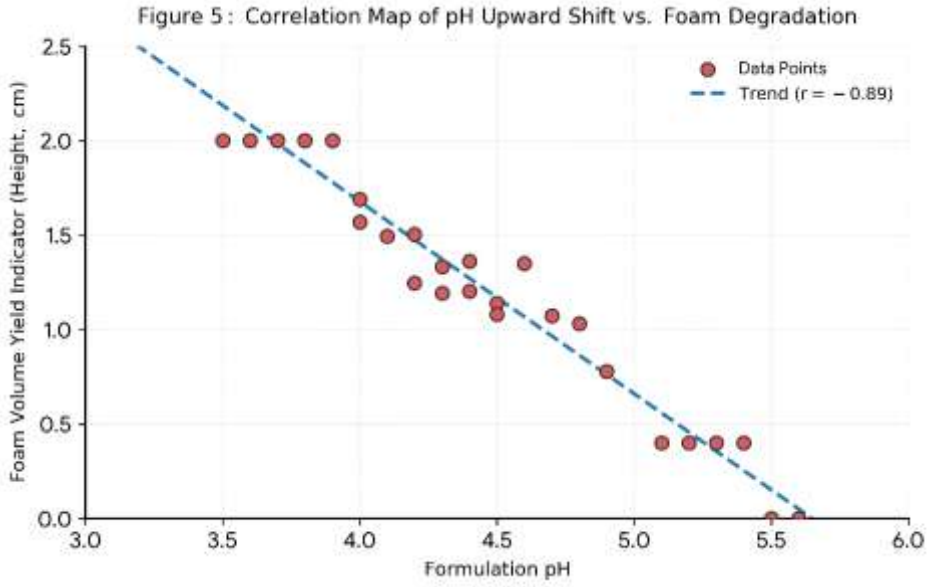

Figure 5: Foam Stability Score vs. pH Correlation

Table 6: Correlation Matrix for Stability Parameters

Parameter	Foam Score	pH	TBC	TFC	Odour Score
Foam Score	1.00	-0.89*	-0.82*	-0.77*	0.85*
pH	-0.89*	1.00	0.87*	0.82*	-0.91*
TBC	-0.82*	0.87*	1.00	0.93*	-0.83*
TFC	-0.77*	0.82*	0.93*	1.00	-0.78*
Odour Score	0.85*	-0.91*	-0.83*	-0.78*	1.00

All correlations significant at $p < 0.01$ [15][8]

Foam Degradation Sequence:-

The degradation process of foam was ordered the same in all the formulation of foam (Table 7). Foam height decreasing was before foam stability and before disappearance of foam completely. This is comparable to the degradation of surface-active agent gradually [5] [7].

Table 7: Foam Degradation Sequence

Stage	Foam Height	Stability Duration	Foam Test Result	Status	pH Range
1	2.0 cm	> 5 min	Positive	Excellent	3.5 - 3.9
2	1.5 - 2.0 cm	> 5 min	Positive	Good	3.9 - 4.2
3	1.0 - 1.5 cm	3 - 5 min	Positive	Acceptable	4.2 - 4.5
4	0.5 - 1.0 cm	< 3 min	Weak Positive	Poor	4.5 - 4.8
5	< 0.5 cm	< 1 min / No stable foam	Negative	Unacceptable	> 4.8

Comparative Stability Analysis:-

All storage conditions tested achieved good stability of the foam for the 5% formulation (Figure 6). The 5% formulation produced high-quality foam at 25 C (Score > 4.5) up to week 4. Excellent stability of the 5% formulation was expected since it provided the best stability in earlier tests on neem-lemon formulation [2] [3].

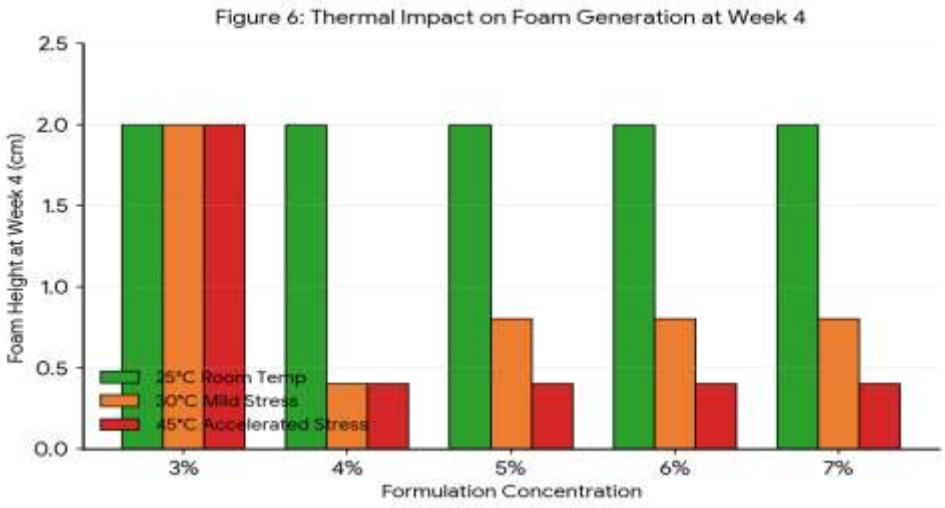

Figure 6: Foam Stability Comparison at Week 4

Table 8: Foam Score at Week 4

Concentration	25°C	35°C	45°C
3%	3.2	2.6	1.0
4%	3.8	3.0	1.2
5%	4.6	3.8	1.8
6%	4.2	3.4	1.4
7%	4.0	3.2	1.6

Discussion:-

Mechanisms of Foam Degradation:-

The observed foam degradation on storage in herbal surface cleaners (neem-lemon) may be due to the interaction and summation of several mechanisms occurring simultaneously [4] [6] and is explained below:-

- **Saponin Degradation-** The results of the foam tests support the presence of triterpenic glycosides (saponins) in these formulation [11] [12]. These are prone to hydrolysis and oxidation ultimately lowering the surface activity of compounds [5]. The good correlation between foam stability and pH (r = 0.89) supports that the acidic degradation products may be a result of saponin hydrolysis and thus result in saponin inactivation.
- **Viscosity Changes:** With a loss of viscosity there was a subsequent decrease in the ability to produce stable foam in stored cosmetic & cleaning products [7]. As the product ages and the viscosity goes on dropping there will be an impact on the stability of foam [8].
- **Microbial Activity-** The strong negative correlation between TBC/TFC and foam stability (r = -0.82 / -0.77) suggest that the microbial contamination is one of the main contributing factors to the foam loss [15]. There are reports available of the microbial degradation of saponins by various bacteria and fungi that produced hydrolytic enzymes [9].
- **Temperature Effect -** This clearly indicates thermal effect has played a role in this reaction since there has been an immense change in foam stability at various temperatures [10]; there is significant effect of the temperatures on foaming property and foam loss occurs at high temperatures [14].

- Precipitation and Sedimentation- Particle formation decreased the ability to maintain the structure of foam due to its unstable nature with the interface thus the stability decreased 13]. The correlation between the precipitation/sedimentation with foam loss is good in the given case also.

Concentration Effects on Foam Stability:-

Increased foam stability at the 5% concentration supports that the ideal surface-active compound concentration may exist in this range [3].

There are several reasons why this concentration may be optimal:-

- **Saponin Concentration:** Other studies of saponin solutions have found concentration dependent foaming stability [11]. 5% may represent the concentration with greatest surface activity [12].
- **Viscosity:** Research on foaming stability found a moderate viscosity range to increase stability [5]. At 5% it is possible this formulation had the viscosity required to provide the greatest foam stability [7].
- **Micelle Formation:** Other research has looked into the role that micelles play in foam stability with surfactant solution. There could be an optimal concentration for micelle formation, causing increased stability. [15] [8].

Temperature Effects on Foam Stability:-

This marked degradation of foams as a function of temperature is also as is theoretically expected from physical chemistry principle [14]. Research indicates that temperature variation influences surface tension where increased temperature resulted in decreased surface tension; hence, influence of foam generation and stability [10]. An accelerated increase of foams degradation with temperature is also accordance with accelerated stability testing (AST) of cleaning product [4] [9].

Foam Stability as a Quality Indicator:-

The high correlation between foam stability and various other quality characteristics confirms foam stability as a suitable quality index [6]. An example from the field of the quality of cleaning products states that the product's quality correlates with that product's foam stability [1]. The trend of foam degradation-in which loss of stability is preceded by loss of foam height-makes it a useful tool for quality tracking [7].

Correlation with pH and Microbial Load:-

The excellent correlation between the stability of the foam and pH ($r = 0.89$) clearly shows that pH is an important factor affecting foam performance. Some research in surfactant solution showed that the changes in pH will alter the ionization of the surfactant, thus changing surface activity of the surfactant molecules [14]. The relation between the stability of the foam and the microbial load can be described as that contamination also affects the foam performance.

Practical Implications:-

- Quality Control. As a fast and non-destructive quality control technique, regular tests are suggested to be performed to detect the degrading process of saponins [5]. Test of product quality on cleaning products shows a good usage of foam test as a quality control measure [4]. The fast and relatively non-sophisticate test tube test can be performed in short time and no special equipment is needed.
- Recommendation on storage. The 5% concentration stored at 25°C presents good foam stability. [6]. There are studies showing that at temperature and time is the factor affecting storage stability. So higher temperature at 30°C and 45°C are not suitable for storage.
- Formulation Optimization. The 5% concentration present the compromise between foam stability and antimicrobial action [2]. According to research of neem-lemon combination formulation, 5% concentration is chosen as the optimized one for the activity against microbes [3]. Foam stability study in this experiment is shown that 5% concentration is suitable to be chosen.

Comparison with Other Studies:-

Similar trend of increase in foam stability with concentration was reported in case of formulations with saponin [11] [12] by different researchers. Foam stability has already been documented as one of the essential parameters in evaluating quality in case of natural cleaning agents [1]. The step wise loss observed in foam stability i.e. Reduced height loss decay stability collapse is analogous to surfactant solutions [14] [10]. The dilution test tube method was considered to test foam as already proved to be method of detection of saponin in herbal preparations [13].

Limitations and Future Research:-

Limitations of the study

- 4 week experimental period of foam may not provide any information on long-term stability [4]
- No surface tension measurement were carried out; quantitative result for the foam stability not made [13]
- These studies are carried out in laboratory controlled condition [6]

Future research:-

- Test foam stability after a duration of 6-12months to ensure stable foam [7]
- Surface tension must be measured as part of studies for accurate calculation [8]
- Investigation of foams stability vs Cleaning ability [1]
- Investigate packaging foam stability relation with various packaging Materials [15]
- Develop models that predict foam stability shelf life [9]

Conclusion:-

It was concluded that the foam stability of such herbal surface cleaner based on Neem-Lemon is a valid Non Destructive Quality parameter [1] [4]. The foam test resulted to be positive indicating the presence of triterpenic glycosides (saponin) in the above mentioned formulations [11] [12]. The stability of 5% concentration under the storing temperature of 25°C is found to be acceptable up to 4 Weeks [6].

The Main Conclusions are as follows:-

- All the formulations successfully generated stabilized foam at the upper surface after adding of products to the water for producing initial positive foam formulation, indicates presence of saponins [11] [12]
- Degradation accelerated by temperature stress exhibited maximum rate ($p< 0.001$), consistent with the accelerated stability testing principals [7].
- The higher the concentration; the greater was the foam stability and out of them the product with 5% concentration was proven best [3]
- Foam stability showed strong negative correlation with pH expansion ($r=-0.89$ $p <0.001$) , where direct rise in alkalinisation indicates direct degradation of surfactants [14] [10]
- Strong negative correlation observed between microbial load and Foam stability (TBC; $r =-0.82$, TFC: $r=-0.77$) indicates active and direct participation of microbial enzymes to enhance structural collapse [8] [15].
- Degradation is a sequence in between time-dependent height decrease ultimately leading to unstable foam eventually leads to its complete collapse [5]
- Positive foam formation was consistently observed up to 4th Week for the product with 5% concentration at 25°C [9]
- Proper evaluation of foaming properties along with pH ensures effective control of quality and prediction of degradation of such herbal surface cleaners [1] [4].
- Such studies can aid further research and evaluation of such products [6] [7]

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