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HARNESSING SORBITOL ESTERS AND ETHOXYLATED SURFACTANTS FOR GRAPE SEED OIL MICROEMULSION SYSTEMS: PREPARATION AND EVALUATION

***[1]Harshal Patil, [1]Baliram Gadhve, [2]Manjiree Girdhar, [2]Chetan Pillai and [1]Jyotsna Waghmare**

[1]Department of Oils, Oleochemicals and Surfactant Technology Institute of Chemical Technology (ICT), Nathalal Parikh Marg, Matunga (E), Mumbai-400019, Maharashtra. India.

[2]Department of Chemistry, SVKM'S Mithibai College of Arts, Chauhan Institute of Science and Amrutben Jivanlal College of Commerce and Economics Bhaktivedanta Swami Marg, Gulmohar Road, Suvarna Nagar, Vile Parle (W), Mumbai 400056, Maharashtra India.

***Corresponding Author: Harshal Patil**

Department of Oils, Oleochemicals and Surfactant Technology Institute of Chemical Technology (ICT), Nathalal Parikh Marg, Matunga (E), Mumbai-400019, Maharashtra. India.

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ABSTRACT

This study has been conducted to develop stable microemulsion systems incorporating grape seed oil using sorbitol esters (UNITOP SMO) and ethoxylated surfactants (POLYSORBATE 80) as emulsifying agents. Grape seed oil was used because of its high amount of linoleic acid (62.246%) and the stability indices for the oil were excellent (acid value: 0.2, peroxide value: 1.3), which promoted its suitability for topical delivery. Four surfactant blends were studied on a pseudo-multipart model by varying the amounts of surfactants and creating the emulsion using phase diagrams using the water titration method with a hydrophilic-lipophilic balance (HLB) range of 9.7-12.9. The emulsions were characterized for particle size, electrical conductivity, pH, density, and stability parameters and significant particle size distributions were appropriately sized between O/W (oil-in-water) and W/O (water-in-oil) microemulsions with the O/W microemulsions using the higher HLB values (11.6-12.9) reporting 53.8-121.5 nm particle sizes and the W/O microemulsions using the higher HLB values (11.6-12.9) exhibiting particle sizes of 102.5-363.3 nm. The electrical conductivities were able to differentiate the microemulsions as the O/W microemulsions ranged from 177.5-184.2 µS/cm while the W/O microemulsions ranged from 69.3-89.3 µS/cm. All formulations underwent resolved thermodynamic stability (no phase separation was noted after centrifugation and 30-day studies) for stability characterization of the formulations. The use of all biodegradable ingredients complies with green chemistry principles and provides sustainable environmental alternatives to existing synthetic systems. Overall, the microemulsions produced in this study provide a viable approach for applications within cosmetics, pharmaceuticals, and nutraceuticals, most cases with an O/W microemulsion having significantly more potential.

KEYWORDS: - Grape seed oil, microemulsion, sorbitol esters, ethoxylated surfactants, Sustainable formulation.

1. INTRODUCTION

Grape seed oil, a natural lipid with high content of bioactive molecules, has been the focus of intense interest for its possible uses in pharmacy, cosmetics, and nutraceutical products. Nevertheless, its water insolubility and vulnerability to oxidation hinder its formulation and delivery in water-based systems. Microemulsion formulation of Grapeseed oil represents one of the effective solutions to the above drawbacks, due to the microemulsion systems' thermodynamic stability, transparency, and enhanced bioavailability. Grape seed oil possesses specific advantages for skin health because of its specific composition and characteristics. It is highly light and penetrates very easily into the skin, preventing residue and allowing deep penetration, thus ideal for sensitive skin types[1],[2],[3] Its rich linoleic acid content (65-72%), an essential unsaturated fatty acid that is not produced by the body, induces the reconstruction of membrane structures in skin cells, minimizes trans-epidermal water loss, and increases skin hydration. Additionally, grape seed oil is endowed with natural antioxidants, namely tocopherols and proanthocyanidins, which neutralize free radical damage and prevent aging signs like wrinkles and fine lines. Its antioxidant potential also shields the skin from damage caused by UV exposure and aids in maintaining collagen and elastin fibres, ensuring firmness and elasticity of the skin. Relative to other oils, lightness, high antioxidant levels, and capacity to stimulate tissue regeneration distinguish grape seed oil and make it particularly adept at inducing healthy, youthful skin and reducing the possibility of negative reactions.

These characteristics make grape seed oil a superior option for numerous skincare uses, such as facial moisturizers, serums, and body lotions. It is versatile enough to be used by itself or in a blend with other ingredients to formulate powerful skincare products. Furthermore, grape seed oil's non-comedogenic properties prevent clogging pores, making it ideal for acne skin and balancing oil production in combination skin types. The dermal nourishing activities of grape seed oil go beyond its antioxidant activity.[2],[3],[4],[5],[6] The oil has high concentrations of linoleic acid, an omega-6 fatty acid that contributes to strengthening the skin barrier and enhancing water retention. Its light, non-greasy character makes it an excellent carrier oil for bringing other active ingredients deeper into the skin. In addition, grape seed oil is anti-inflammatory in nature and hence can be used to calm sensitive or irritated conditions of the skin like eczema and rosacea. Due to its soft nature, coupled with its skin-healing properties and anti-redness effects, it is an excellent component in products that are recommended for sensitive skin types. Its natural astringent properties also help to close pores and equalize skin tone, further making it effective in maintaining a clear and healthy-looking complexion.[5],[6],[7],[8],[8]

Microemulsions have several advantages over conventional emulsions, such as solubilizing hydrophilic and lipophilic compounds, enhancing drug bioavailability, and increased stability. These advantages make microemulsions useful in numerous fields, such as pharmaceuticals, cosmetics, food science, and enhanced oil recovery methods. The ability to impart microemulsion properties by changing composition and methods of preparation makes it possible to create specialized systems for specific needs and applications for both industrial and research purposes. Green microemulsions have more benefits for improved environmental sustainability than conventional microemulsions because they employ renewable, biodegradable, and less toxic components, which can include natural oils, surfactants from plant material, and eco-friendly co-surfactants.[10],[11] They also eliminate the dependency on petroleum-based synthetic chemicals which have a considerable carbon footprint, potential toxicity, and environmental persistence. Green microemulsions are also biodegradable and exhibit limited ecotoxicity, which will diminish the damage to ecosystems while also preventing bioaccumulation in life forms, allowing a faster degradation following use. Green microemulsions are also stable, efficient formulations that will deliver active ingredients using smaller amounts to reduce environmental impact after use. All of these factors make further argument to green microemulsions over traditional pharmaceutical, cosmetic, and food formulations.[12]

Sorbitan esters and polysorbates are non-ionic emulsifiers or surfactants that can stabilize oil-in-water or water-in-oil microemulsions. The sorbitan esters series is more lipophilic and is typically reserved for water-in-oil, while the polysorbates series is hydrophilic and is preferred for oil-in-water microemulsions. With the combination of these surfactants at the right ratios, one can modify the hydrophilic-lipophilic balance (HLB) to obtain the best microemulsion stability and stability performance across a wide range of microemulsion types.[13][14] Having this flexibility is critical to designing customized microemulsion systems for many applications such as drug delivery, topical agents in cosmetics, and improved oil recovery, while also providing excellent solubilization capacity for both hydrophilic and lipophilic compounds. Microemulsions are thermodynamically stable and optically transparent dispersions of oil and water stabilized by surfactants.[15][16][17] Microemulsions are generally composed of droplets possessing diameters of 10 - 100 nanometres, making them far smaller than even basic emulsions. Since microemulsions have unique properties such as high solubilization capacity, very low interfacial tension, and improved stability, they have become increasingly popular in multiple applications. The formation of a microemulsion requires specific surfactants and co-surfactants (and sometimes co-solvents) in order to reduce the interfacial tension between the oil and water phases. When the interfacial tension is reduced to zero, emulsion will occur spontaneously. A microemulsion system can exist as different phases including O/W, W/O, and bicontinuous characteristics depending on the formulation and environmental conditions.[18],[19],[20]

This study aims to develop and characterize stable emulsions for Grapeseed oil by utilizing the combined stabilizing/emulsifying power of sorbitol esters and ethoxylated surfactants. The effort is aimed at developing the surfactant mixture analytically to yield thermodynamically stable, clear, and nano dispersed emulsions for food, industrial, cosmetic, pharmaceutical and nutraceutical formulations.

2. MATERIALS AND METHODS

2.1 Materials

Grapeseed oil was obtained from Bhagavati herbal & health care Pvt ltd, Gujarat. The surfactants were received from ROSSARI BIOTECH LTD, Mumbai. for the study, including non-ionic surfactants with different hydrophilic-lipophilic balance (HLB) values: UNITOP SMO (HLB = 4.3), POLYSORBATE 80 (HLB = 15.0). Additional reagents and chemicals used in the study were of analytical grade and obtained from reputable suppliers.

2.2 Methods

2.2.1 Characterization of Grapeseed Oil

Grapeseed oil was characterized through determination of various physicochemical properties including appearance, specific gravity, acid value, peroxide content, iodine value, and saponification value. These parameters were evaluated according to standard analytical methods to assess the quality and composition of the oil. Additionally, fatty acid composition was analysed using gas chromatography (GC) to determine the profile of saturated and unsaturated fatty acids present in the grapeseed oil.

2.2.2 Characterization of Emulsifier

The UNITOP SMO & POLYSORBATE 80 was characterized by measuring key properties including Appearance, specific gravity, hydrophilic-lipophilic balance (HLB), and surface-active properties. These parameters were determined to understand the emulsifying capacity and behaviour of the surfactant system.

2.2.3 HLB Blend Preparation

Surfactant blends with varying HLB values were prepared by mixing lipophilic and hydrophilic surfactants in calculated proportions. The blends were formulated using UNITOP SMO & POLYSORBATE 80. The HLB value of each blend was calculated according to the equation:

HLB = (% Surfactant A × HLB A + % Surfactant B × HLB B)

Where the percentages are based on the weight ratio of each surfactant in the blend. Required quantities of surfactants were weighed accurately and mixed thoroughly at ambient temperature until homogeneous blends were obtained. Different HLB values were achieved by varying the ratios of low HLB (UNITOP SMO) and high HLB (POLYSORBATE 80) surfactants. The prepared surfactant blends were characterized for pH, specific gravity, and surface tension prior to use in microemulsion studies.

2.2.4 Preparation and Characterization of Microemulsions

Pseudo-ternary phase diagrams were constructed for grapeseed oil systems using the water titration method at room temperature. Various surfactant blends with different HLB values, prepared from combinations of UNITOP SMO and POLYSORBATE 80, were employed as emulsifiers for each system. The oil and emulsifier were mixed in varying weight ratios ranging from 0:10 to 10:0, incorporating different hydrophilic-lipophilic balance (HLB) values. Deionized water was gradually titrated into these oil-surfactant mixtures until the onset of turbidity was observed. The percentage of water consumed relative to the total formulation, along with the corresponding oil and emulsifier percentages, was used to construct the phase diagrams. Microemulsion formulations were selected from the identified microemulsion regions within the phase diagrams for comprehensive characterization. The selected formulations were evaluated for various parameters including appearance, pH, viscosity, particle size, and electrical conductivity to determine their physicochemical properties and stability characteristics.

3. RESULTS

2.2.5 Characterization of the Microemulsions

A) Visual Assessment The homogeneity, clarity, and optical transparency of microemulsions were evaluated through visual examination against a black background at room temperature (25°C). Samples were assessed for phase separation, turbidity, and overall appearance to determine microemulsion formation and stability.

B) pH Measurement The pH values of different microemulsion formulations were determined at ambient temperature (25°C) using a calibrated pH meter (Equiptronics microcontroller pH meter model EQ 621, India). Measurements were performed in triplicate to ensure accuracy and reproducibility.

C) Particle Size Analysis The particle size distribution of microemulsion formulations was analyzed using dynamic light scattering with a Mastersizer 3000 Hydro (Malvern Panalytical, Worcestershire, UK) at room temperature. The mean particle diameter, polydispersity index, and size distribution were recorded to characterize the microemulsion droplets.

D) Electrical Conductivity The electrical conductivity of optimized microemulsion formulations was measured at 25°C using a conductivity meter LMCM20 (Labman Scientific Instruments Pvt. Ltd, India). Conductivity measurements were used to determine the microemulsion type, with high conductivity values indicating oil-in-water (O/W) systems and low conductivity suggesting water-in-oil (W/O) microemulsions.

E) Physicochemical Stability Studies Comprehensive stability testing was conducted over 30 days at room temperature to evaluate the long-term stability of microemulsion formulations. The stability assessment included monitoring changes in particle size, electrical conductivity, pH, and viscosity. Additionally, centrifugation stability tests were performed at 3,000 rpm for 30 minutes to assess the thermodynamic stability and resistance to phase separation of the microemulsions. All parameters were measured at predetermined intervals to track any deterioration or changes in the formulation characteristics.

3.1 Characterization of Grapeseed oil

Table -1: - Physicochemical parameters of Grape seed oil.

Sr. No	Parameter	Value
1	Physical appearance	Clear Yellow liquid
2	Specific Gravity	0.919
3	Acid Value	0.2
4	Iodine Value	140
5	Peroxide Value	1.3
6	Saponification value	186

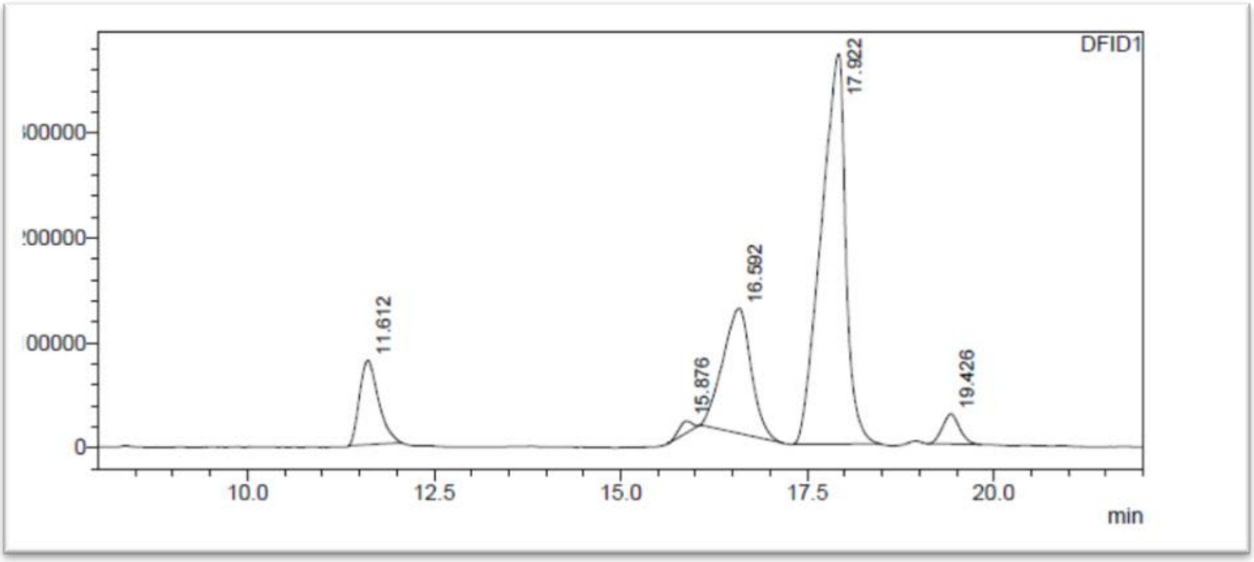

Figure 1: Gas Chromatography Graph of Grape Seed Oil.

Table 2: Fatty acid composition of Grape seed oil.

Sr. No	Fatty acid Composition	Percentage
1	Palmitic caid	10.382
2	Stearic acid	1.014
3	Oleic acid	23.004
4	Linoleic acid	62.246
5	Linolenic acid	3.354

3.2 Characterization of Emulsifier

Table 3: Physicochemical Parameters of Surfactant.

Sr. No	Parameter	UNITOP SMO	POLYSORBATE 80
1	Physical appearance	Brownish yellow	Clear Viscous liquid
2	Specific Gravity	1.01	1.09
3	Saponification value	158	53
4	Hydroxyl value	195	70
5	Moisture content	0.4	1.2
6	Surface tension	44.9 mN/m	40.32 mN/m
7	Critical Micelle Concentration.	0.2 mM	0.015 mM
8	HLB value	4.3	15

Table 4: Composition and Calculated HLB of UNITOP SMO and POLYSORBATE 80 Blends.

Blend No	Surfactants used	UNITOP SMO	POLYSORBATE 80	HLB BLEND
1	UNITOP SMO + POLYSORBATE 80	20%	80%	12.9
3	UNITOP SMO + POLYSORBATE 80	32%	68%	11.6
4	UNITOP SMO + POLYSORBATE 80	40%	60%	10.7
5	UNITOP SMO + POLYSORBATE 80	50%	50%	9.7

Table 5: Characterization of Sorbitan Monooleate/Polysorbate 80 Blends.

Blend no	pH	Specific Gravity	Surface Tension (mN/m)	CMC (mM)
1	6.5	1.07	43	0.014
3	6.7	1.06	41	0.035
4	6.9	1.05	39	0.055
5	7.0	1.04	37	0.080

3.3 Construction of Phase Diagram for microemulsion

Microemulsion formulations were prepared by mixing the grape seed oil with the surfactant blend systems. Four surfactant blends of UNITOP SMO/POLYSORBATE 80 with HLB values from 9.7 to 12.9 were blended with the oil phase, then slowly agitated with distilled water. Surfactant blends studied were Blend 1 (20% UNITOP SMO + 80% POLYSORBATE 80, HLB 12.9), Blend 3 (32% UNITOP SMO + 68% POLYSORBATE 80, HLB 11.6), Blend 4 (40% UNITOP SMO + 60% POLYSORBATE 80, HLB 10.7), and Blend 5 (50% UNITOP SMO + 50% POLYSORBATE 80, HLB 9.7).

Figures 2, 3, 4 and 5 display the pseudo-ternary phase diagrams of the systems under investigation, involving grape seed oil, UNITOP SMO/POLYSORBATE 80 surfactant blend mixtures, and water. The areas filled with shading indicate zones of microemulsion formation detected at room temperature. The respective phase diagrams are related to the particular surfactant blend compositions to enable systematic investigation of the effects of HLB values on the formation and stability of microemulsions in the ternary system.

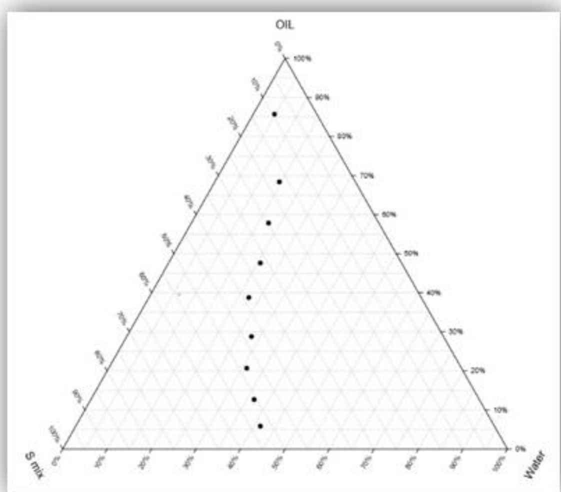

Figure 2: Grape Seed Oil Microemulsion With HLB 9.7 .

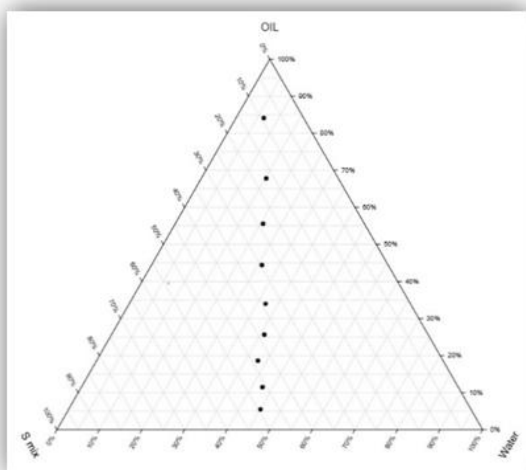

Figure 3: Grape Seed Oil Microemulsion with HLB 10.7.

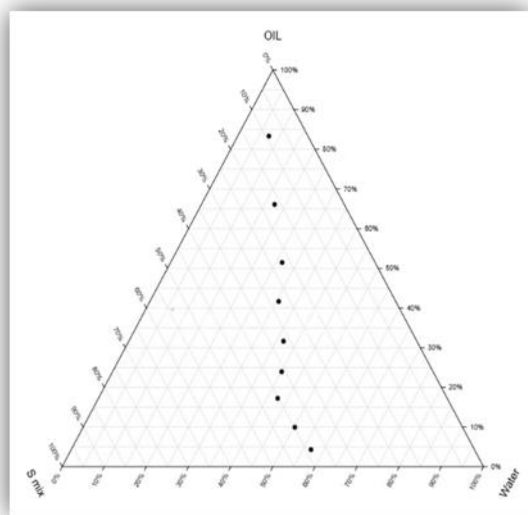

Figure 4: Grape Seed Oil Microemulsion With HLB 11.6.

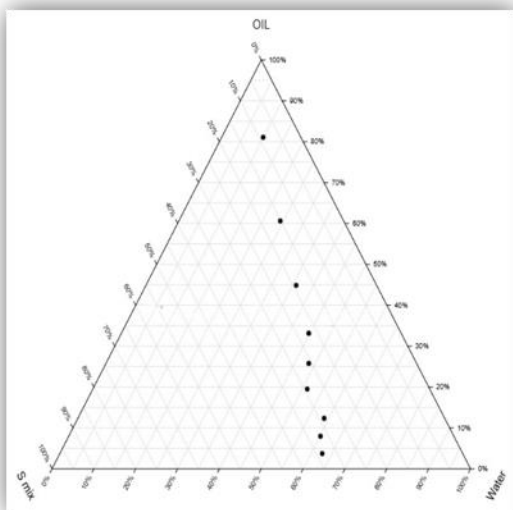

Figure 5: Grape Seed Oil Microemulsion with HLB 12.9.

Table 6: Grapeseed oil Microemulsion with different HLB.

Sr. No	Formulation	HLB	OIL	S MIX	WATER	TOTAL
1	GSO -1	HLB 9.7	5.85	52.63	41.52	100.00
2	GSO -2	HLB 10.7	4.31	38.79	56.90	100.00
3	GSO -3	HLB 11.6	4.31	38.79	56.90	100.00
4	GSO -4	HLB 12.9	3.73	33.58	62.69	100.00
5	GSO -5	HLB 9.7	38.76	38.76	22.48	100.00
6	GSO -6	HLB 10.7	25.64	38.46	35.90	100.00
7	GSO -7	HLB 11.6	23.95	35.93	40.12	100.00
8	GSO -8	HLB 12.9	19.51	29.27	51.22	100.00

Table 7: Grapeseed oil Microemulsion With Different HLB Physical Parameter.

Sr.no	Formulation	HLB	Type of emulsion	pH	Density	Conductivity	Particle size
1	GSO -1	HLB 9.7	O/W	7.1	0.98	183.50	121.5
2	GSO -2	HLB 10.7	O/W	7.23	1.00	179.40	110.4
3	GSO -3	HLB 11.6	O/W	7.34	1.01	184.20	58.9
4	GSO -4	HLB 12.9	O/W	7.15	1.03	177.50	53.8
5	GSO -5	HLB 9.7	W/O	7.2	1.02	69.32	363.3

6	GSO -6	HLB 10.7	W/O	7.54	1.01	71.89	257.7
7	GSO -7	HLB 11.6	W/O	7.61	1.01	89.32	119.4
8	GSO -8	HLB 12.9	W/O	7.54	1.02	76.32	102.5

Table 8: Grapeseed oil Microemulsion Centrifuge Stability Data.

Sr. No	Formulation	HLB	Centrifuge stability Before	Centrifuge stability After 30 days
1	GSO -1	HLB 9.7	Stable	Stable
2	GSO -2	HLB 10.7	Stable	Stable
3	GSO -3	HLB 11.6	Stable	Stable
4	GSO -4	HLB 12.9	Stable	Stable
5	GSO -5	HLB 9.7	Stable	Stable
6	GSO -6	HLB 10.7	Stable	Stable
7	GSO -7	HLB 11.6	Stable	Stable
8	GSO -8	HLB 12.9	Stable	Stable

4. DISCUSSION

4.1 Grape Seed Oil Characterization

Physicochemical analysis of grape seed oil validated the high quality and amenability for formulating microemulsion. The oil possessed desirable qualities such as low acid value (0.2), reflecting low free fatty acid content and adequate storage stability. The peroxide value (1.3) was appropriately within acceptable limits, validating the absence of appreciable oxidative spoilage. These are the parameters upon which the stability of microemulsion depends, as oxidized oil may undermine system integrity and therapeutic effectiveness. In the analysis of the fatty acid profile, linoleic acid occupied 62.246% of the total fatty acid content, justifying the oil's nutritional and cosmetic worth. This high linoleic acid value is especially important in skin care usage, where this fatty acid is vital in the restoration of barrier function, reduction of trans-epidermal water loss, and hydration. The oleic acid content (23.004%) also increases the ability of the oil to penetrate, making it a prime candidate for topical delivery systems.

4.2 Surfactant Selection and HLB Optimization

The strategic choice of UNITOP SMO and POLYSORBATE 80 as the surfactant system was extremely efficient in the formation of microemulsions. The great disparity between their HLB values (4.3 and 15.0, respectively) offered the required flexibility to adjust the hydrophilic-lipophilic balance over a broad range (9.7 to 12.9). This strategy allowed the formation of oil-in-water and water-in-oil microemulsions, illustrating the versatility of the surfactant system. The character data showed that the higher HLB value blends (11.6 and 12.9) gave more effective emulsification with lower surface tension values (39-37 mN/m) and lower critical micelle concentrations. The reduction of surface tension is positively related to the capacity for stable microemulsion formation and increased solubilization. The stepwise lowering of the CMC values from 0.080 mM to 0.014 mM with rising HLB reflects better surface activity and better micelle formation.

4.3 Phase Behaviour and Microemulsion Formation

The pseudo-ternary phase diagrams accurately located stable microemulsion regions for all the HLB blends that were tested and validated the thermodynamic stability of the systems. The development of large microemulsion regions in the phase diagrams reflects the strong nature of the surfactant blends and their convergence with grape seed oil. The capacity to produce stable microemulsions at varying oil-to-surfactant ratios allows for flexibility in formulation across different applications demanding varying oil loading capacities. The analysis of phase diagrams identified that increased HLB blends (12.9) enabled the formation of microemulsions with increased water content (maximum 62.69%), whereas reduced HLB systems (9.7) were optimized to include higher concentrations of oil (maximum 38.76%). The observed behavior is congruent with the principle that increased HLB surfactants prefer oil-in-water systems, while decreased HLB surfactants result in water-in-oil microemulsions.

4.4 Characterization of Microemulsions: Structure, Stability, and Phase Behavior

Particle size analysis and conductivity measurements were used to analyze the structural and stability characteristics of the prepared microemulsions. The findings showed an unequivocal correlation between droplet size distribution and hydrophilic-lipophilic balance (HLB) values. Oil-in-water (O/W) microemulsions had noticeably smaller particle sizes (53.8–121.5 nm) than water-in-oil (W/O) systems (102.5–363.3 nm). The smallest droplet size (53.8 nm) was obtained with an HLB 12.9 formulation (GSO-4), suggesting maximum surfactant efficacy in minimizing interfacial tension and maximizing thermodynamic stability. The nano-scale sizes validate the presence of true microemulsions, which will be able to enhance bioavailability and skin permeation in topical formulations. Electrical conductivity measurements also facilitated structural characterization, successfully discriminating between O/W and W/O microemulsions. O/W systems showed the high conductivity (177.50–184.20 µS/cm) as a result of the permanent aqueous phase, while W/O systems showed lower conductivity (69.32–89.32 µS/cm), which is typical for oil-rich structures. These results confirm the successful production of different types of microemulsions and offer a solid method of phase identification under the course of formulation optimization. Stability testing proved outstanding performance for all formulations, with no phase separation after centrifugation testing and 30-day storage. This outstanding stability can be explained by the microemulsion's thermodynamic character and the right choice of surfactant. The preserved stability against varied environmental conditions indicates an excellent suitability for commercial use with a need for long shelf life. In addition, all preparations demonstrated pH values within the physiologically acceptable range (7.1–7.61), compatible with topical applications without skin irritation. Density measurements (0.98–1.03 g/cm³) continued to be in line with normal emulsion values and further substantiated the creation of stable disperse systems. Inversely related HLB to particle size in O/W microemulsions indicates that increased-HLB surfactants promote interfacial activity to create smaller, more stable droplets. These rigorous characterization data emphasize the important role of HLB in controlling microemulsion properties while illustrating their superior stability and appropriateness for pharmaceutical and cosmetic use.

4.5 Environmental Sustainability and Green Chemistry Aspects

In terms of sustainability, naturally-derived grape seed oil, and biodegradable surfactants reflect the emerging trend of increasing demand for green formulations in the cosmetic and pharmaceutical industries. The sustainability benefits combined with the enhanced functionality of these microemulsions also provide them with alternatives to traditional synthetic systems. Since the oil phase and surfactants are all biodegradable, they have a limited footprint on the environment and can support sustainable practices for manufacturers.

4.6 Applications and Commercial Potential

The applications described in this work, show the level of flexibility. The O/W microemulsions with lesser particle sizes (GSO-3 and GSO-4) are ideal for facial serums and moisturizers which are associated with general improved penetration and lighter application. The W/O systems may have a use in more occlusive formulations or in some types of specific delivery formulations where the orientation of the active ingredients can be manipulated. The ability to vary the level of oil while retaining the stability of the system provides a product that has variations in both feel and concentration of active ingredients. This flexibility is valuable to the cosmetic industry since consumer preference for texture and application characteristics can vary widely between product categories.

5. CONCLUSIONS

This study developed stable grape seed oil microemulsions using sorbitol esters (UNITOP SMO) and POLYSORBATE 80, offering sustainable solutions for cosmetics, pharmaceuticals, and nutraceuticals. Grape seed oil's high linoleic acid (62.2%) and stability (acid value: 0.2, peroxide value: 1.3) make it ideal for topical delivery. Optimizing HLB (9.7–12.9) enabled both O/W and W/O microemulsions, with higher HLB (11.6–12.9) yielding smaller particles (O/W: 53.8–121.5 nm; W/O: 102.5–363.3 nm). Pseudo-ternary phase diagrams confirmed thermodynamic stability. Conductivity distinguished O/W (177.5–184.2 µS/cm) from W/O (69.3–89.3 µS/cm). Formulations showed excellent stability. Biodegradable ingredients align with green chemistry. O/W microemulsions suit lightweight cosmetics, while W/O systems may enable controlled release. This research advances eco-friendly microemulsion technology, providing a foundation for sustainable formulations in multiple industries.

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