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Application and Stability Evaluation of Polymer blends in Cosmetics

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Abstract: *Objective: The use of polymers in the field of cosmetics has been popularized due to its properties of film-forming, emulsifiers, thickeners, rheology modifiers, stimuli-responsive agents, etc. The present work deals with the synthesis of polymer nanocomposites that has been incorporated into a base face mask formulation at a loading of 1% and subsequently studied for sensory attributes, primary skin irritancy and physical stability tests. Methods: The polymer nanocomposites were synthesised by the solution casting method and were incorporated in the base face mask formulation. Sensory evaluation was done using a ten member expert panel and the results were tabulated. The physical stability test was carried out at different storage conditions and the colour, odour, pH, viscosity and appearance were analysed using standard techniques. Results: The polymer nanocomposites were found to be stable in the face mask formulations for a period of 12 weeks. The sensory evaluation tests showed higher or equivalent results on most of the sensory parameters. The formulation showed no skin irritancy. Conclusion: The sensory and physical stability of the face mask formulation with the prepared polymer blend nanocomposite shows comparable and an equivalent match with base face mask formulation and also no irritation was found on using the face masks.*

Keywords: Polymer, Nanocomposite, Formulations, Stability, Synthesis, Sensory evaluation

I. INTRODUCTION

The advent of new technologies both in the field of polymer science and nanotechnology have led to an increased application in cosmetics. This has led to innovative advances in the creation of sophisticated scientific personal care products. The upsurge in the usage of polymers has mainly been to avoid volatile organic compounds in the cosmetic formulations. Polymers act as emulsifiers, film-formers, thickeners, modifiers, protective barriers and aesthetic enhancers [1].

Polydimethylsiloxane, Polyurethane/Silicone copolymers provide smoothness to the hair and this property finds applications in shampoos and conditioners [2]. Addition of polymers to water-based systems results in an increase in viscosity [3]. Starch has been modified by the incorporation of a hydrophobe in the long chain which results in efficient water repellancy [4]. Introduction of such hydrophobes also ensures good oil absorption. Clay based face masks works well for oily skin and aims at absorbing the natural sebum from the skin and leave it dry [5]. Polymethyl methacrylate is said to provide smoothness and fluidity to cosmetic products and have a transparent effect on the skin. Polyvinyl alcohol and polyvinyl acetate have excellent film-forming properties. In peel-off masks, it helps to moisturize the skin, remove dead cells and provide cleanness [6, 7]. Nanomaterials have gained momentum in this respect due to the properties of better UV protection, greater penetration in the skin, enhanced colour and long-lasting effects [8]. Zinc oxide and titanium oxide nanoparticles are found in sunscreens due to their excellent UV filtering properties [9]. Nanosilver has been extensively used in deodorants and creams due to its antibacterial properties [10, 11]. These deodorants claimed to provide antibacterial protection throughout the day and thus safeguarding against body odour. Similarly, Nanogold also known for its antibacterial properties finds applications in toothpastes as a disinfectant for the mouth [12]. Nanocapsules has an outer polymeric membrane and contains an inner liquid in the nanoscale level constitutes active ingredients in present day cosmetic formulations [13]. In spite of all these applications, the reliability of using nanoparticles is under debate due to the risks associated with it. Due to their small size, they provide deeper penetration into the skin and can enter the bloodstream causing complications [14]. They are also known to bind to the contaminating substances already present in the environment thereby increasing the risks of environmental pollution [15].

In the present work, a polymer composite has been prepared using polyvinyl alcohol (PVA), natural polymer, starch and a montmorillonite clay, bentonite. The polymer composite shows the combined properties of these ingredients in cosmetic applications. The individual properties are expected to be enhanced in the blended version of these polymers. An attempt has been made to further increase the applicability of this composite in the cosmetic field by the incorporation of nanoparticles in the polymer matrix [16]. The key to determining the usage of a particular composition in cosmetics is to study the stability of the composition over a prolonged period of time. The attributes or the variables included in the physical stability test decide the performance of the

particular composition and its benefits. The prepared blend and the nanocomposites are compared with the base to assess the properties and the various attributes. It is found to be stable over a period of 12 weeks and shows no skin irritancy. In most of the sensory parameters, the composites show better or equivalent match in comparison to the base mask formulations.

II. MATERIALS AND METHODS

A. Preparation of Gold nanoparticles (AuNPs)

10mM standard solution of $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$ was diluted to 10 ml using milli-Q water and allowed to boil for 2-3 minutes. 0.5 % of trisodium citrate (stabilizing and reducing agent) was added and heated to boiling till the appearance of pale purple colour. The solution was stirred constantly and cooled to room temperature. The formation of gold nanoparticle (figure 1) was indicated by a change to wine red colour [17].

B. Preparation of ZnO nanoparticles (ZnONP)

Sol-gel method was used to prepare ZnO nanoparticles [18].

Zinc nitrate and Citric acid (1:2 ratio) was dissolved in minimum amount of water and made into a paste. The resultant precipitate was washed with double distilled water and heated up to 110°C using a heating mantle for an hour. White coloured ZnO nanoparticles (figure 2) was obtained.

C. Preparation of Nanocomposite Films

1) *PVA/Starch/Bentonite (PSB film)*: The composite films were prepared by solution casting method [19]. PVA, Starch and Bentonite were taken in the volume ratio of 3:2:1 and blended together by placing it in an ultrasonicator for 20 minutes. A homogenous mixture was obtained. This solution was casted in the petri dish to obtain the composite film. Curing of the polymer films (figure 3) was done at room temperature.

2) *PVA/Starch/Bentonite/AuNP (PSB-Au film)*: The prepared gold nanoparticle was used to synthesise the nanocomposite film by solution casting method. PVA, Starch and Bentonite were taken in the volume ratio of 3:2:1 and blended together by placing it in an ultrasonicator for 20 minutes. 5ml of AuNP was added to the above composition. Homogeneity of the solution was ensured by sonicating it for an additional 10 minutes. These solutions were casted in the petri dish to obtain nanocomposite films having different weight percentages of AuNPs. Curing of the polymer films (figure 4) was done at room temperature.

3) *PVA/Starch/Bentonite/ZnONP (PSB-ZnO film)*: The prepared ZnO nanoparticle was used to synthesize the nanocomposite film by solution casting method. PVA, Starch and Bentonite were taken in the volume ratio of 3:2:1 and blended together by placing it in an ultrasonicator for 20minutes. ZnONP was added to the above composition at a weight percentage of 1%. Homogeneity of the solution was ensured by sonicating it for an additional 10 minutes. These solutions were casted in the petri dish to obtain nanocomposite films having different weight percentages of ZnO nanoparticle. Curing of the polymer films (figure 5) was done at room temperature.

D. Preparation of the face mask formulation (Code-1)

The different ingredients in each of the phases along with their specific roles have been tabulated in Table I.

The total quantity of demineralised water along with the Phase A ingredients were added into a vessel slowly with homogenization. The mixture was heated to 70°C - 80°C and further homogenized. The phase B ingredients were weighed in a separate vessel and heated to 75°C .

Phase B was slowly added to phase A in vacuum. The mixture was homogenized at 3000 rpm and 650 Pa. After 30 minutes, phase D was added to the above mixture. It was homogenized and cooled. Once temperature reached to 50°C - 60°C , phase E was added into the vessel containing the above mixture. It was homogenized for and cooled. Once temperature reached to 40°C - 45°C , phase F was added and homogenized and was taken out of vacuum. BHT and Triclosan were dissolved in perfume. Once temperature reached to 40°C , phase G and Phase H was added to the above mixture and complete dispersion was ensured.

The final step was to add the polymer blend nanocomposites under homogenization to formulate the codes 2, 3 and 4.

E. Sensory Evaluation

The study was conducted using a 10 member expert panel of trained women who used a known amount of the test formulations and compared it to the conventional vanishing cream [20, 21]. The panellists were given coded samples in a randomized order. The sensory attributes of the formulations both during use and after use and based on this the results were tabulated in table II.

F. Physical Stability Test

Physical stability of all five products (one base and four samples) was assessed by keeping them at different storage conditions. 30g of each product was filled in HDPE tube and kept in different storage conditions such as at room temperature, 4^oC, 45^oC, 50^oC and 40^oC at a relative humidity of 70 %. The physical characteristics analysed and the methods of analysis are tabulated in table III [22].

III. RESULTS AND DISCUSSION

The sensory parameters were studied at the time of use and after use. On comparing the parameters in Table II, the creamy feel, thickness and ease of spreading increases for the sample having the ZnO nanoparticles (code 4) as compared to the base face mask formulation (code 1). The blend without the presence of any nanoparticles (code 2) shows a nearly equivalent match with the base. Incorporation of nanoparticles enhances some of the sensory parameters of the polymer blend. The addition of gold nanoparticles to the polymer composite (code 3) is shown to reduce the skin irritation during use and feels smooth on the skin. After use of the formulations, the entire area of the facial skin projects an even skin tone and a non-oily look more effectively on usage of Code 4. Code 3 with the gold nanoparticle shows greater skin brightness and lesser skin irritation. The composition coded 4 shows an increase in most of the sensory parameters as compared to the base face mask and also the polymer composite coded 2. This is in accordance with the already existing cosmetic-active properties of ZnO nanoparticles. The complete sensory evaluation at the time of use and after use is represented in the form of a cluster bar graph 1 and 2.

The physical stability is estimated on the basis of colour, fragrance, appearance, viscosity and pH. Colour, appearance and fragrance are estimated at the temperature ranges of 4-7^oC, 25-27^oC, 45^oC, 50^oC and at a relative humidity of 70 %. It is found to be stable for a period of 12 weeks for all the codes. It was observed that viscosity of the samples remains the same over a period of 12 weeks. The pH of the composition at room temperature is found to be in the range of 5.2-5.9.

IV. CONCLUSION

The prepared nanocomposites have been incorporated in the base face mask formulations and the studies reveal that these nanocomposites show very good physical stability over a period of 12 weeks. The sensory parameters show enhanced properties for code 4 as compared to the base face mask formulations that is coded 1. No irritation was found before and after using the face masks. This paves the way for an effective usage of these compositions in face masks to enhance the properties with only a 1% loading of the sample into the base face masks.

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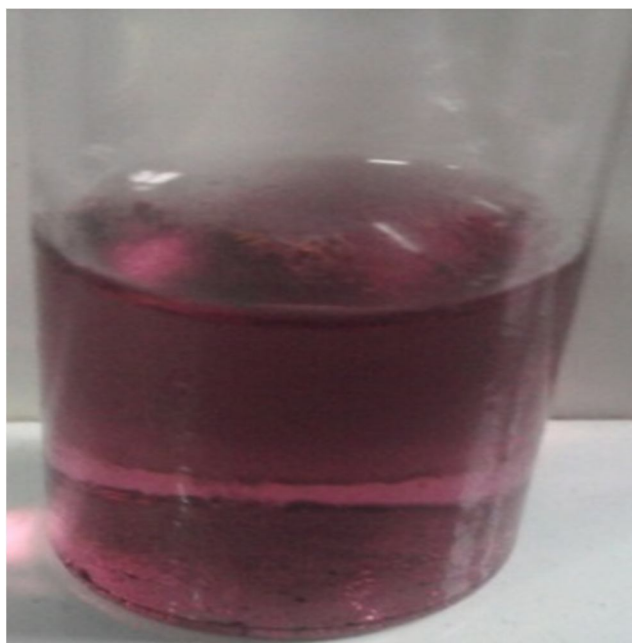


Figure 1: Gold Nanoparticle

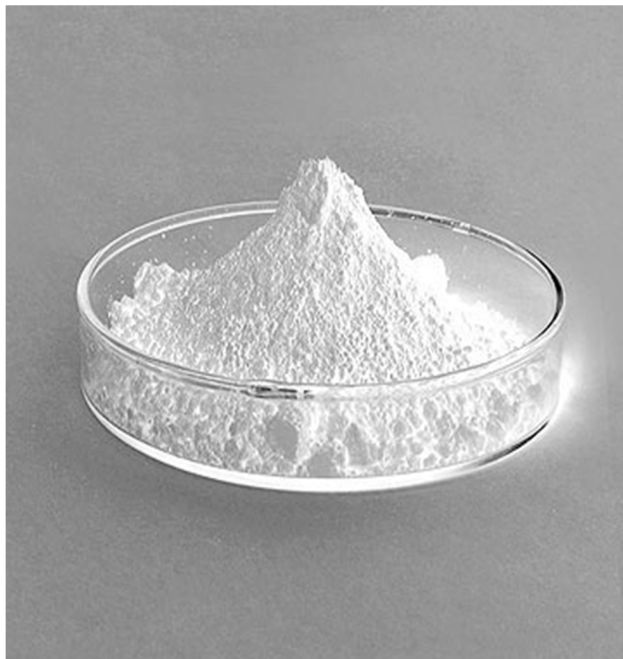


Figure 2: Zinc Oxide nanoparticle



Figure 3: PSB film



Figure 4: PSB-Au film



Figure 5: PSB-ZnO film

Table I: Raw Materials used in different

Phase	Raw Materials	Functions	Code 1	Code 2	Code 3	Code 4
Phase "A"	Aqua/Water	Diluent	Qs to 100	Qs to 100	Qs to 100	Qs to 100
	Magnesium Aluminum Silicate	Stabilizer	1.00	1.00	1.00	1.00
	Multani mitti	Filler	30.0	30.0	30.0	30.0
	Bentonite	Stabilizer	5.0	5.0	5.0	5.0
	Allantoin	Skin soothing agent	0.10	0.10	0.10	0.10
	Titanium Dioxide	Opacifier	3.00	3.00	3.00	3.00
	Disodium EDTA	Chelating agent	0.050	0.050	0.050	0.050
Phase "B"	Cetostrearyl Alcohol	Thickener	15.00	15.00	15.00	15.00
	Polysorbate-60	Emulsifier	5.00	5.00	5.00	5.00
	Isopropylmyristate	Emollients	1.00	1.00	1.00	1.00
Phase "C"	Glycerin	Moisturiser	10	10	10	10
	Xanthan Gum	Thickener	0.40	0.40	0.40	0.40
Phase "D"	Methylparaben	Preservative	0.25	0.25	0.25	0.25
	Propylparaben	Preservative	0.15	0.15	0.15	0.15

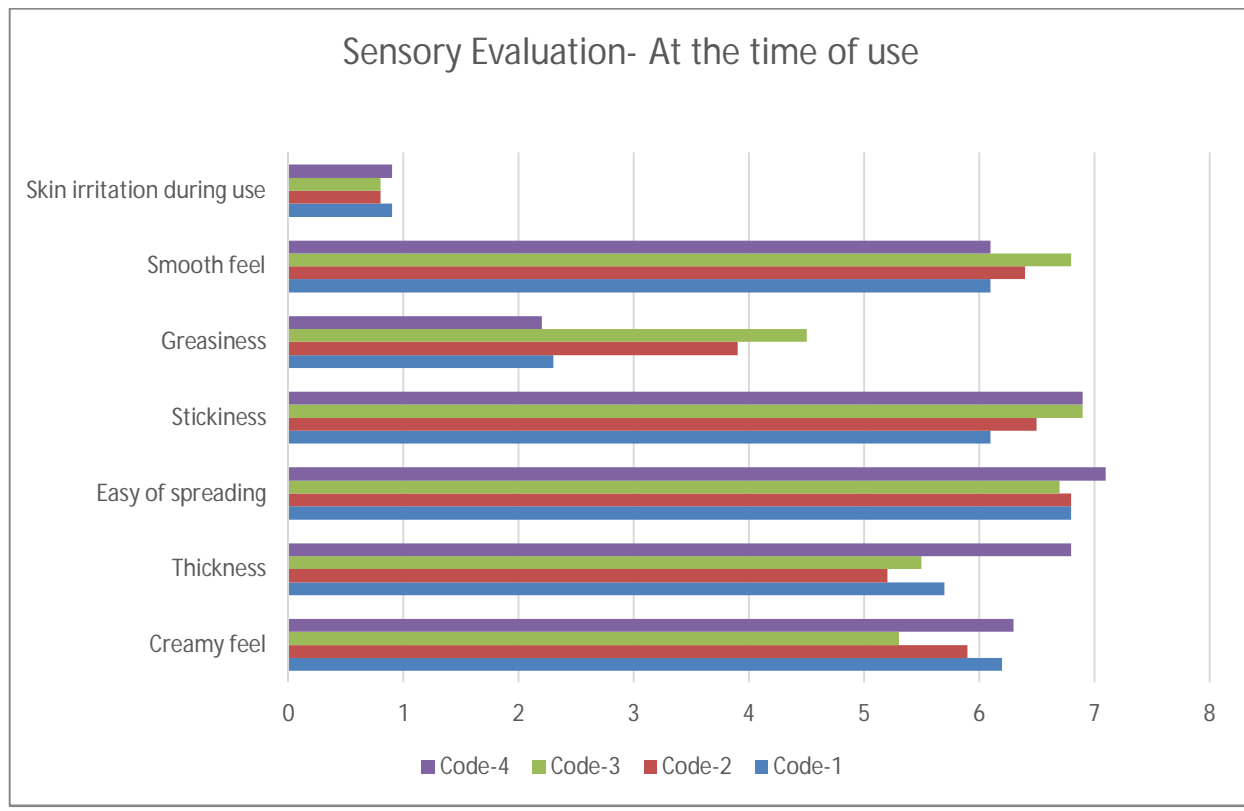
	Phenoxyethanol	Preservative	0.50	0.50	0.50	0.50
	Propylene Glycol	Moisturiser	0.50	0.50	0.50	0.50
Phase "E"	Sodium laureth sulfate	Cleaning agent	1.0	1.0	1.0	1.0
	DMDM Hydantoin	Preservative	0.30	0.30	0.30	0.30
Phase "F"	Licorice (glycyrrhiza glabra) concentrate	Melanin Inhibitor	0.10	0.10	0.10	0.10
	Banana(Musa sapientum) concentrate	Skin Nutrient	0.10	0.10	0.10	0.10
Phase "G"	Perfume	Fragrance	0.30	0.30	0.30	0.30
	Triclosan	Anti bacterial agent	0.1	0.1	0.1	0.1
	BHT	Anti oxidant	0.03	0.03	0.03	0.03
Phase "H"	CI:19140	Colourant	0.10	0.10	0.10	0.10
Phase "I"	PSB	Nano active	0.00	1.00	0.00	0.00
	PSB-Au	Nano active	0.00	0.00	1.00	0.00
	PSB-ZnO		0.00	0.00	0.00	1.00

Table II: Sensory evaluation for codes 1, 2, 3 and 4

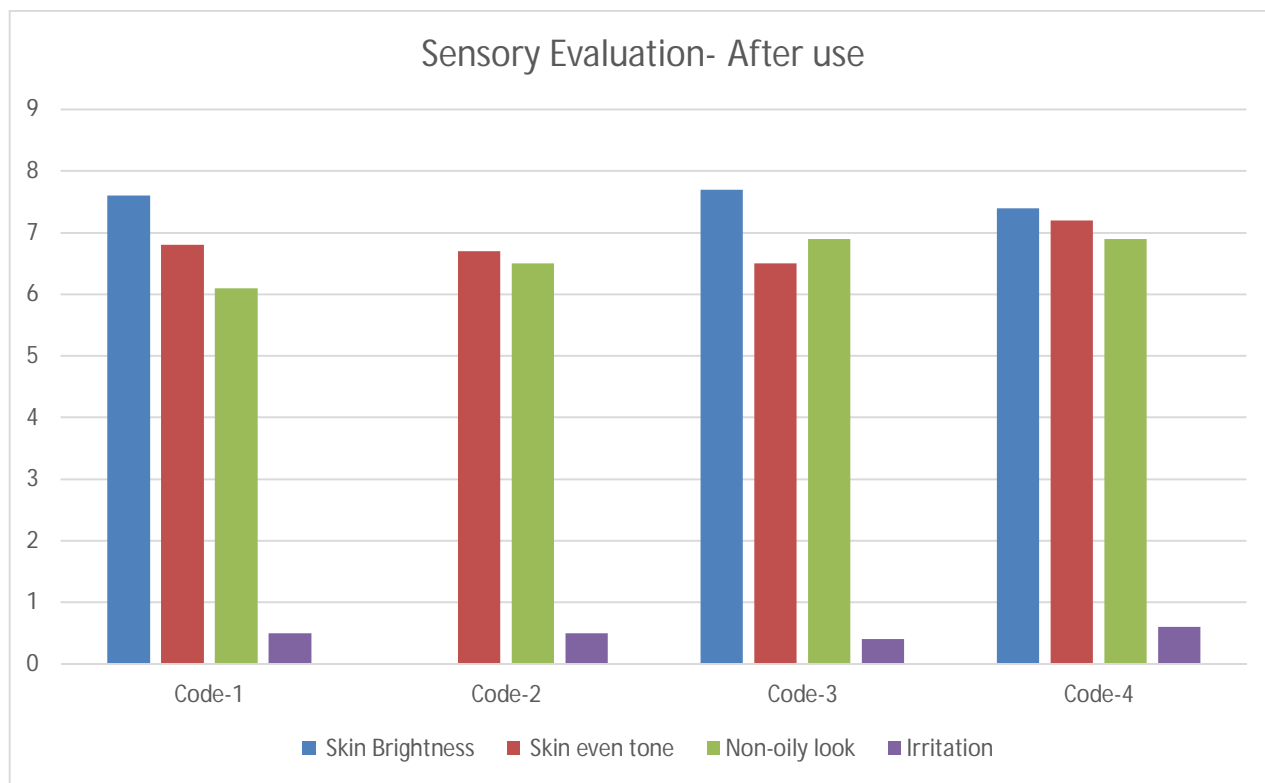
Sensory parameter	Code 1	Code 2	Code 3	Code 4
At the time of use				
Creamy feel	6.2	5.9	5.3	6.3

Thickness	5.7	5.2	5.5	6.8
Easy of spreading	6.8	6.8	6.7	7.1
Stickiness	6.1	6.5	6.9	6.9
Greasiness	2.3	3.9	4.5	2.2
Smooth feel	6.1	6.4	6.8	6.1
Skin irritation during use	0.9	0.8	0.8	0.90

After use				
Skin Brightness	7.6	7.5	7.7	7.4
Skin even tone	6.8	6.7	6.5	7.2
Non-oily look	6.1	6.5	6.9	6.9
Irritation	0.5	0.5	0.4	0.6



Graph 1: Sensory evaluation-At the time of use



Graph 2: Sensory evaluation-After use

Table III: Methods of analyzing the physical characteristics

S. No.	Physical Characteristics	Method of Analysis
1	Colour	Visual
2	Appearance	Visual
3	Odour	Olfactory
4	pH	BIS:6608:2009 Annex B
5	Viscosity @30°C, TF spindle S95 0.60 RPM /45 sec	BIS:13360(Part 1 1/Set ISO 2555 : 1060)

Table IV: Physical Stability- Colour

S. No.	Colour	Weeks	Code 1	Code 2	Code 3	Code 4	Code 5	Remarks
1	Stability at 4-7°C	1	Stable	Stable	Stable	Stable	Stable	No change in Colour
		2						

S. No.	Appearance	weeks	Code 1	Code 2	Code 3	Code 4	Code 5	Remarks
1	Stability at 5-7 ⁰ C	1	Stable	Stable	Stable	Stable	Stable	No change in appearance
		2						
		4						
		8						
		12						
2	Stability at 25 – 27 ⁰ C	1	Stable	Stable	Stable	Stable	Stable	
		2						
		4						
		8						
		12						
3	Stability at 45 ⁰ C	1	Stable	Stable	Stable	Stable	Stable	
		2						
		4						
		8						
		12						
4	Stability at 50 ⁰ C	1	Stable	Stable	Stable	Stable	Stable	
		2						
		4						
		8						
		12						
5	Stability at 45 ⁰ C 70 % RH	1	Stable	Stable	Stable	Stable	Stable	
		2						
		4						

Table V: Physical

		8					
		12					

Table VI: Physical Stability- Fragrance

S. No.	Fragrance	Weeks	Code 1	Code 2	Code 3	Code 4	Code 5	Remarks
	Stability at	1	Stable	Stable	Stable	Stable	Stable	
		2						
S. No.	Viscosity	Weeks	Code 1	Code 2	Code 3	Code 4	Code 5	Remarks
1	Stability at 5-7 ⁰ C	1	Stable	Stable	Stable	Stable	Stable	
		2						
		4						
		8						
		12						
2	Stability at 20 ⁰ C	1	Stable	Stable	Stable	Stable	Stable	
		2						
		4						
		8						
		12						
3	Stability at 45 ⁰ C	1	Stable	Stable	Stable	Stable	Stable	
		2						
		4						
		8						
		12						
4	Stability at 50 ⁰ C	1	Stable	Stable	Stable	Stable	Stable	
		2						
		4						
		8						
		12						

Table VII: Physical Sta

Table VIII: Physical S

No significant change in viscosity



pH	Weeks	Code 1	Code 2	Code 3	Code 4
Stability at 25 ⁰ C-27 ⁰ C	1	5.92	5.26	5.31	5.62
	2	5.90	5.50	5.32	5.62
	4	5.88	5.67	5.35	5.61
	8	5.80	5.80	5.30	5.60
	12	5.89	5.92	5.32	5.60



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