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COSMETIC EVALUATION OF SOME IRANIAN COMMERCIAL NORMAL HAIR SHAMPOOS AND COMPARISON WITH NEW DEVELOPED FORMULATION

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ABSTRACT: Four commercial normal hair shampoos produced by four famous Iranian companies were chosen for evaluating their characteristics based on scientifically measurable properties and were compared with our new developed formulation. Selection of the evaluation parameters of shampoo is a challenging test, because of the multitude of both subjective and instrumental test methodologies available for this purpose. The following characteristics were tested: physical appearance/visual inspection evaluation, stability, pH, foam quality, ability to reduce the surface tension, ash value, wetting agent and viscosity. The results of tests given much information about quality and characteristics and sensorial attributes related with famous Iranian market shampoos, so that they fit the demands and the needs of Iranian consumers. Also, a newly developed formulation for normal hair shampoo was introduced with high foaming, good wetting ability, detergency power and so on.

INTRODUCTION: Since, ancient era, people have been using herbs and ash for cleaning, beautifying and managing of their hair because hair is considered as an integral part of human beauty ^{1, 2} and hair cleansing is very important to obtain a satisfying aspect. The main constituent of hair is keratin. Keratin is a remarkable protein which is resistant to wear and tear. A Shampoo may be described as a cosmetic product for washing of hair and scalp, packed in a form convenient for use. Its first function is cleansing the hair of accumulated sebum, scalp debris and residues of hair-grooming preparations. The added functions of shampoo include lubrication, conditioning, medication and so on ^{3, 4}.

Most of the shampoos are formulated as aqueous solutions or as emulsions, both containing mixture of surfactants (synthetic or natural) as cleansing and foaming agents, other excipients (viscosity controlling agents, emollients, preservatives, *etc.*) and active ingredients ^{3, 4}. The surfactants are responsible for the cleaning and lathering ability of the shampoo as well as its skin tolerance.

The shampoo formulations must be medically safe for long term usage. Evaluation of shampoos comprises the quality control tests including visual assessment and physiochemical controls such as pH, density, viscosity, surface tension, foam volume, ash value, detergency and wetting agent ⁵. Detergents based on sodium lauryl ether sulfate are the most common, but the concentration will vary considerably in a different brand and even within a manufacturer's product range ³. From ancient time beyond memory, the Persian people (Persian is the ancient name of Iran) have been using abundantly from nature to care for their health, skin, and hair, as natural ingredients that have preventive,

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protective and corrective and detergency action such as *Acanthophyllum*, *Ziziphus*, *Lawsonia* and ash and so on. However, the first industrial production of detergent in Iran was happened in 1964 by Tolid Daru Company. At present, there are many local companies that are producing cosmetics and toiletries in there. Four famous Iranian companies which normal shampoos chose were from them were Kaf, Pakshoo, Negin Behdasht Ariyan, and Cinere Natural Cosmetics companies.

Iran is a country that is located in the Middle East with seventy-five million populations. This country is one of the biggest markets for cosmetics and toiletries and has seven ranking in the world. There are many cosmetics and toiletries from international and local companies in this market. Not only there are many cosmetics and toiletries produced by local companies in this country but also the export their different products to some other country. One of the important products in this country is normal hair shampoo that there are indifference brands.

This study aimed to evaluate some characteristic of several famous commercial shampoos for normal hair that are producing in Iran, based on scientifically measurable properties. The data of this article show scientific information of some famous and bestsellers shampoo that can be used for different aims, from consumers to cosmetics and toiletries producing companies, and also proposed new formulation with better qualification for normal hair shampoo such as foam volume, ability to reduce the surface tension, detergency power and wetting time and other physiochemical quantities.

MATERIALS AND METHODS: Four kinds of shampoos, chemicals, shampoo ingredient, a piece of cotton, canvas, velvet and Indian ink was purchased from the market. Some hair tresses were obtained from the salon.

New Shampoo Formulation: With the different ingredients and varying concentrations of them, several different shampoos formulations for normal hair were prepared and visual assessment and physiochemical controls tests such as Physical appearance/visual inspection, pH, dirt dispersion, viscosity, surface tension, foam volume, and foam

stability, solid content, ash value, detergency, and wetting agent were done. The best quality formulation was chosen that has been shown in **Table 1** (F5). The procedure of preparing shampoos involves mixing water with EDTA and guar, glycerin, decyl glucoside, cocamide propylene betain, sodium lauryl ether sulphate and ammonium lauryl ether sulphate and cocamide diethanolamine until solvation respectively and followed by heating to 70 °C and then EGMS was added. After reaching room temperature panthenol, opacifier, triethanolamine, preservative, and NaCl was added slowly with continuous stirring respectively.

Shampoo Evaluation Method:

Physical Appearance / Visual Inspection: All chosen shampoos were evaluated for physical appearance such as color and odor with smelling and visual inspection by ten volunteers ^{6,7}.

Determination of pH: The pH of 10% shampoo solution in distilled water was determined at room temperature 25°C ^{5,8,9}.

Rheological Evaluations: The viscosity of the shampoos was determined by using Brookfield Viscometer (Model DV-1 Plus, LV, USA) set at different spindle speeds 20 rpm. The viscosity of the shampoos was measured by using spindle T95 at speed 20 rpm. The temperature and sample container's size was kept constants during study ^{5,10}.

Solid Content Determination: 10 g of each shampoo was weighed in a tare evaporating dish and kept in a hot air oven at 105 °C. Repeated the drying until the constant weight loss was observed after 4 h. The solid content was calculated for each sample ^{5,10,11}.

Ash Value: Total ash: 2 g of each shampoo was taken and placed in a tarred silica crucible and kept in a muffle furnace at 45 °C until free from carbon, this was then cooled and re-weighed. Finally, the percentage of total ash was calculated ^{5,14}.

Stability Studies: The thermal stability of shampoos was studied by placing in glass tubes, and they were placed in a humidity chamber at 45°C and 75% relative humidity. Their appearance and physical stability were inspected for one month ⁹.

TABLE 1: THE NEW SHAMPOO FORMULATION (F5)

Ingredient	Use (g)	Mass %
Water	Solvent	Until 100
EDTA	Complexing Agent	0.1
Guar hydroxypropyl trimethyl ammonium chloride	Conditioning	0.3
Glycerin	Humectant	1.5
Decyl Glucoside	Detergent (Nonanionic Surfactant)	2
Sodium Lauryl Ether Sulphate (70%)	Detergent (Anionic Surfactant)	11
Ammonium Lauryl Ether Sulphate (30%)	Detergent (Anionic Surfactant)	5
Cocamide Propyl Betain (28%)	Detergent (Zwitterionic Surfactants)	5
Coconut fatty acid diethanolamide (85%)	Viscosity Increasing and Foam Booster	3.5
Ethylene Glycol Diestearate	Pearlazing Agent	0.3
DexPanthenol	Vitamin and Emollient	0.5
Styrene/Acrylates Copolymer (8%)	Opacifier	1
Triethanolamine	pH Conditioner	Needed to pH 6-7
NaCl	Viscosity Increasing	1.2
Methylchloroisothiazolinone 1.5%	Preservative	0.1

Surface Tension Measurement: Measurements were carried out with a 10% shampoo dilution in distilled water at room temperature with dropper method^{10, 15}. Thoroughly clean the dropper using chromic acid and purified water because surface tension is highly affected by grease or other lubricants. The data calculated by the following equation bellowed:

$$R_2 = (W_3 - W_1) n_1 / (W_2 - W_1) n_2 \times R_1$$

Where, W_1 is weight of empty beaker, W_2 is the weight of beaker with distilled water, W_3 is Weight of beaker with shampoo solution, n_1 is some drops of distilled water, n_2 is a number of drops of shampoo solution, R_1 is the surface tension of distilled water at room temperature and R_2 is the surface tension of the shampoo solution.

Determining of Anionic Surfactant: The total anionic surfactant content of each shampoo was determined by the titration method with 0,00447 N of cetrimide. Cetrimide is an antiseptic which is a mixture of different quaternary ammonium salts including cetrimonium bromide (CTAB). Under brand name cetavlon. The consuming volume of cetrimide was a criterion for anionic surfactant content of shampoos.

Foaming Ability and Foam Stability: To evaluate the foaming ability of formulations prepared, the Ross-Miles foam column method was used. Briefly, one percent aqueous solutions of each formulation were prepared at room temperature, and 200 ml of them were poured into a glass column containing 50 ml of the same solution from

a height of 50 cm at room temperature. The height of the foam generated is measured immediately and their volumes were calculated. To evaluate the foam stability, the same procedure was performed and foam volume after 20 min was also determined^{10, 11}.

Dirt Dispersion: One percent solution of each shampoo (1 g of the sample in 10 ml of water) was taken. 1 drop of India ink was added; test tube was a stopper and shook it ten times. The amount of ink in the foam was estimated as None, Light, Moderate, or Heavy^{14, 16}.

Wetting Time: Wetting time was measured by Drave's test when some kind of weighed skein was allowed to sink through a wetting solution in a 500 ml graduated cylinder, the time is taken for sinking was given as wetting efficiency. Some kind of skeins such as cotton, canvas, and velvet was chosen for wetting time test. The result showed that velvet skein sinking had more repeatability than another one (not shown) and also it is a more accurate time-saving method.

Since, this method can be chosen as an alternative to canvas disc method. Therefore the velvet cut into 1-inch diameter discs having an average weight of 0.30g was chosen for wetting test of shampoos. The lower time required for sinking, the wetting efficiency is higher. The disc was floated on the surface of shampoo solution 1% w/v, and the stopwatch started^{1, 17, 18, 19}. The time required for the disc to begin to sink measured accurately and noted as wetting time.

Detergency Ability: There are several methods for the determination of detergency ability of shampoos. Earlier work on detergency evaluation was done using greasy wool yarn by brand and powers¹⁹. Later the method changed to evaluating detergency by using 10 mg hair swatch. Thompson method used tresses soil with artificial sebum for determination of detergency power and using of gas chromatography data²⁰. Also in many articles, the surface tension of ten percent shampoo solution is attributed to detergency power, the less surface tension, the more detergency power.

In this work, the detergency power was evaluated by inspiring of brunet and powers method, but there are some improvements on that for a better and repeatable response that are near to real hair washing by consumers. For detergency power evaluation a crumple of hair was washed with a 5% sodium lauryl sulfate (SLS) solution, then dried and cut into 10 inches, 2 g swatches. Swatches were suspended in an n-hexane solution containing 10% artificial sebum (soya oil 20%, coconut oil 15%, stearic acid 15%, oleic acid 15%, paraffin wax 15% and lanolin 20%) and the mixture was shaken for 15 minutes at room temperature. Swatches were removed, the solvent evaporated at room temperature, and the dried hair swatch weighed to determine the sebum load.

In one percent solution of each shampoo, a soiled swatch was washed with 250 ml of one percent solution of each shampoo for 150 seconds in 90 rotate per minutes by rotary in 40 °C respectively. It was then dried using a hair dryer and further dried in an oven at 60 °C for 4 h to ensure uniform moisture content and re-weighed. Finally, the percentage of detergency power was calculated using the following equation: in which, DP is the percentage of detergency power C is the weight of sebum in unwashed swatches, and T is the weight of sebum in the swatches after washing.

$$DP = 100 (1-T/C)$$

RESULTS AND DISCUSSION:

Physical Appearance/ Visual Inspection: The color and odor of five shampoos are acceptable by ten volunteers; however, they think the physical appearance of F4 and F5 are more acceptable than others that shown creamy pearl white appearance is more popular.

pH Value: The pH of shampoos is important for improving and enhancing the qualities of hair, minimizing irritation to the eyes and stabilizing the ecological balance of the scalp. The pH is one of the ways to minimize damage to the hair. Mild acidity prevents swelling and promotes tightening of the scales, thereby inducing shine. As seen from **Table 2**, all the shampoos were ranged 6 to 7, which is near to the skin pH.

Rheological Evaluations: Viscosity is the thickness or stickiness of a liquid. The viscosity of shampoo is related at least in part to the number of solids that are present. Product Viscosity plays an important role in defining and controlling many attributes such as shelf life stability and product aesthetics such as clarity and ease of flow of product for packing and spreading ability of shampoos on hair and product consistency in the package. The viscosity of five shampoos change from 2100 to 12800 cps that are acceptable, however, F2 shampoo has lower viscosity and seems watery and F3 shampoo have relatively high viscosity for shampoo. The volunteers choose F4 and F5 more proper in viscosity viewpoint.

Solid Content: If a shampoo has too many solids, it will be hard to work into the hair or too hard to wash out. If it doesn't have enough, it will be too watery and wash away quickly. The solid content of a good shampoo will be between 20% – 30% by weight. Between five candidate shampoos, only F2 is out of range. It must be pointed out that F2 shampoo has lower viscosity and seems more watery between five candidate shampoos.

Ash Value: The total Ash values of five shampoos were found to be between 2.94- 4.04 w/w **Table 2**. The results showed that F2 and F4 formulations have the lowest and highest ash value respectively and new shampoo formulation has acceptable ash value.

Stability Study: Stability and acceptability of organoleptic properties (odor and color) of formulations during storage period indicated that they are chemically and physically stable. The stability of each shampoo was inspected for one month is listed in **Table 1**.

Surface Tension Measurement: It has been approved that a proper shampoo should be able to

decrease the surface tension of pure water to about 40 dynes/cm¹². Surface tension reduction is one of the mechanisms implicated in detergency and the reduction in surface tension of pure water from 72.8 dynes/cm to range of 30-40 dynes/cm of 10%

dilution for each shampoo is considered as a good detergent¹³. The results are shown in **Table 1**. The only surface tension of F2 formulation is out of range, and also our new formulation has the lowest surface tension between five candidate shampoos.

TABLE 2: EVALUATION OF PHYSIOCHEMICAL CHARACTERISTICS OF SHAMPOOS

Parameter	F1	F2	F3	F4	F5
Physical Appearance	Pearl-Yellow	Brownish Yellow	Pearl-Light Yellow	Pearl-White	Creamy Pearl-White
Fragrance	Acceptable	Acceptable	Acceptable	Acceptable	Acceptable
pH (10% solution)	6.5	6.0	6.8	7	7
Viscosity	3900	2100	12800	6800	6300
Solid Content%	21.03	22.08	15.14	20.71	23.30
Ash Content%	3.78	2.94	3.97	4.04	3.73
Stability	stable	stable	stable	stable	stable
Surface Tension	36	43.2	33.1	35.5	32.6
Consuming Cetrinide Volume	6.35	5.05	6.10	6.40	-

Determining of Anionic Surfactant: The total anionic surfactant content of commercial shampoos (F1-F4) was determined by a titration method with 0,00447 N of cetrinide and shown in **Table 1**. The box for F5 is blank because the total anionic surfactant content is shown in **Table 1**.

Foaming Ability and Foam Stability: Although, foam generation has little to do with the cleansing ability of shampoos, it has high importance to the consumer and is, therefore, an important criterion in evaluating shampoos. All the five shampoos showed foaming volume between 272-386 cm³ in distilled water **Table 3**. All five shampoos showed comparable foaming properties. This value being stable almost 10 min, so all these products were good regarding this parameter.

It has been seen that the new formulation shampoo showed more foam volume than commercial shampoos that can be more acceptable for consumers. This fact is maybe due to the presence of foam stabilizing ingredient in shampoos such as cocamide monoethanolamide and cocamide diethanolamine. A point to be noted here is that there does not seem to be any direct correlation between detergency and foaming, which only confirms the fact that a shampoo that foams well need not to clean well.

Dirt Dispersion: Shampoo that causes the ink to concentrate on the foam is considered poor quality; the dirt should stay in the water. Dirt that stays in

the foam will be difficult to rinse away. It will redeposit on the hair. Four shampoos don't show dirt on foam, and F2 shampoo shows light foam in the dirt. These results indicate almost no dirt retained in the foam; so new and marketed formulations are satisfactory.

Wetting Time: The rate of wetting or wetting ability of the surface-active agent is commonly used to determine their comparative efficacies. Wetting phenomena are complex and depend upon several processes and factors such as diffusion, surface tension, concentration and the nature of the surface is wet. Each wetting agent has to reduce surface tension. By comparing of results in **Table 3** it can be seen that F3 and F5 have minimum wetting time respectively and F2 showed the more wetting time. These results are corresponding with surface tension data.

Detergency Ability: Some cosmetic chemists think that shampoo should not be so powerful a detergent as to strip all natural secretions from the hair and scalp. Although cleaning or soil/sebum removal is the primary aim of a shampoo, experimental detergency evaluation has been difficult to standardize, as there is no real agreement on a standard soil, a reproducible soiling process or the amount of soil a shampoo should ideally remove. The targets of these testing shampoos are normal hairs which have moderate soil on themselves. The detergency ability of normal hair shampoos not to be so high or very low. The detergency ability of

normal hair shampoo must be higher than dry hair shampoos and lower than greasy hair shampoos; in other words, the normal hair needs normal detergency. As seen from the results, there is a ring in the amount of sebum removed from 62% to 80% by the marketed shampoos. The results of detergency studies showed that new formulation (F5) has significantly medium detergency ability

when compared with the marketed formulations. It was found between the detergency power range of marketed normal hair shampoo. Therefore new formulation shampoo could be classified as 'shampoo for normal hair.' If detergency ability range of this marketed shampoos for normal hair were chosen as the standard. The results are presented in **Table 3**.

TABLE 3: EVALUATION OF SHAMPOOING ABILITY

Parameter	F1	F2	F3	F4	F5
Foam Volume (cm ³)	272.90	288.20	311.15	334.11	385.12
Foam Stability %	94	94	95	96	96
Dirt Dispersion	None	Light	None	None	None
Wetting Time (s)	31.6	41.3	26.5	29.3	27.2
Detergency Ability %	80	62	75	78	72

CONCLUSION: The result of this evaluation give many information of four famous Iranian market normal hair shampoos characteristics like organoleptic evaluation, stability and physico-chemical evaluation, solid content, pH, ash value, foaming, surface tension, dirt dispersion, wetting time and detergency power. However, it is difficult to arrive a conclusion as which is the best shampoo amongst those tested because it is improbable that anyone formulation will fare better than the others in the entire test performed. It is also difficult to give a relative order to import to the tests, as each is important in its own right. It was seen that many characteristics of these shampoos are in standard range although in some cases out of range were observed.

However, they are bestseller normal hair shampoos in this country, and they continue to enjoy market popularity. Also, a new formulation for normal hair shampoo was introduced that have acceptable properties. It's all tested characteristics are in standard rang and some characteristics of this shampoo such as foam volume, wetting time, surface tension, physical appearance, and detergency are so good and can be introduced as the developed formulation of available normal hair shampoo.

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CONFLICT OF INTEREST: Nil

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