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Comparative Physicochemical Evaluation of Some Marketed Hair Creams in the Nigerian Market

Omali I. C. Paul
Teacher, Department of Chemistry, S.S Simon & Jude Minor Seminary, Kuje, Abuja, Nigeria
Casimir Emmanuel Gimba
Lecturer, Department of Chemistry, Ahmadu Bello University, Zaria, Kaduna State, Nigeria
Stephen Eyije Abechi
Lecturer, Department of Chemistry, Ahmadu Bello University, Zaria, Kaduna State, Nigeria

Abstract:
Hair is one of the vital parts of the body and a protective appendage on the body and considered accessory structure of integument along with sebaceous glands, sweat glands and nails. Hair creams are widely used by the consumer of the cosmetic industries. Many brands are available in the market under different category for the purpose of cleansing, beautifying, promoting attractiveness, or altering the appearance and for anti-microbial activities. Ten hair creams brands were evaluated for their physicochemical properties, Physical characterization, pH, Iodine value, Saponification value, Peroxide value, Acid value and Free-fatty acid value. The leading brands of hair creams in the market with code names, APP, TS, PX, COC, SM, DM, S-8, SW, FEM and AT were standardized for their above physicochemical properties. The hair cream samples complied for the requirements by SON. Most of the hair cream's pH were close to the pH of the scalp mantle of 5.50, thus is likely to make the hair creams more scalp compatible and less likely to cause scalp problems. Exception is DM with pH 2.043 ± 0.006. Results from this study also showed that at 95% confidence level, $p<0.05$, there is significant difference among the physicochemical parameter values of all the hair creams under study.

Keywords: *Hair cream, pH, Iodine value, Saponification value, Peroxide value, Acid value and Free-fatty acid value*

1. Introduction

The development and marketing of cosmetics and personal care products has come a long way from the days when rose water was made and sold from the back of a covered wagon. Back then, product sales were driven more by promises than by any proven benefits, and there were no regulatory mechanisms in place to guarantee product efficacy or safety. Today, the cosmetics industry is subject to government oversight and consumers have come to expect that the products they use are safe and effective. While technological advances have spurred great innovations in product development and testing, few consumers are aware of the processes that manufacturers employ to ensure that products are safe for humans and the environment. Today, regulatory bodies around the world have set rigorous standards for product safety, while improved technology has made testing more efficient.

The safety and quality of a cosmetic product is the full responsibility of the manufacturer, the first importer into the market or the marketer. A cosmetic product put on the market must not cause damage to human health when applied under normal or reasonably foreseeable conditions of use [1]. As the quality of cosmetics formulations specifically hair-care formulation is the key parameter for safety and efficacy of cosmetics [2], it is important to assess them all the time especially here in Nigeria. Hair creams are widely used by the consumer of the cosmetic industries. Many brands are available in the market under different category for the purpose of serving as hair food.

The development and marketing of cosmetics and personal care products has come a long way from the days when rose water was made and sold from the back of a covered wagon. Product sales then were driven more by promises than by any proven benefits, and there were no regulatory mechanisms in place to guarantee product efficacy or safety. The cosmetics industry today is subject to government oversight and consumers expect that the products they use are safe and effective. Technological advances have spurred great innovations in product development and testing and regulatory bodies around the world have set rigorous standards for product safety. Ensuring the safety of cosmetics and personal care products is a top priority in the beauty care industry. The safety and quality of a cosmetic product is the full responsibility of the manufacturer, the first importer into the market or the marketer. A cosmetic product put on the market must not cause damage to human health when applied under normal or reasonably foreseeable conditions of use [1]. As the quality of cosmetics formulations specifically hair-care formulation is the key parameter for safety and efficacy of cosmetics, it is important to assess them all the time [3].

Quality control for efficacy and safety of hair cream cosmetics is of paramount importance. Quality can be defined as the status of a drug that is determined by identity, purity, content and other chemical, physical, or biological properties or by manufacturing process. It's a term that refers to processes involved in maintaining quality and validity of a manufactured product. Different parameters for quality control of hair creams are organoleptic evaluation like color, odor, touch and clarity also determination of pH, Iodine value, Saponification value, Peroxide value, Acid value and Free-fatty acid value.

2. Materials and Methods

Ten majorly marketed hair creams labeled (PX, AT, COC, DM, FEM, APP, SW, SM, S-8 and TS) were used for this study, and for each of these ten hair creams selected, three batches comprising of three samples & making a total of thirty samples for this study were collected for this study. The samples were bought from major markets (Sabon-Gari central market, Samaru general market, Tudun wada and Zaria city markets) in Zaria metropolis, Kaduna state shown in the map below. The country of manufacture was ascertained as all the hair creams contained the serial number for the registration of the cosmetic product by National Agency for Food and Drug administration and Control (NAFDAC) in Nigeria. The Container label information on collected hair cream samples is given in Table 1.

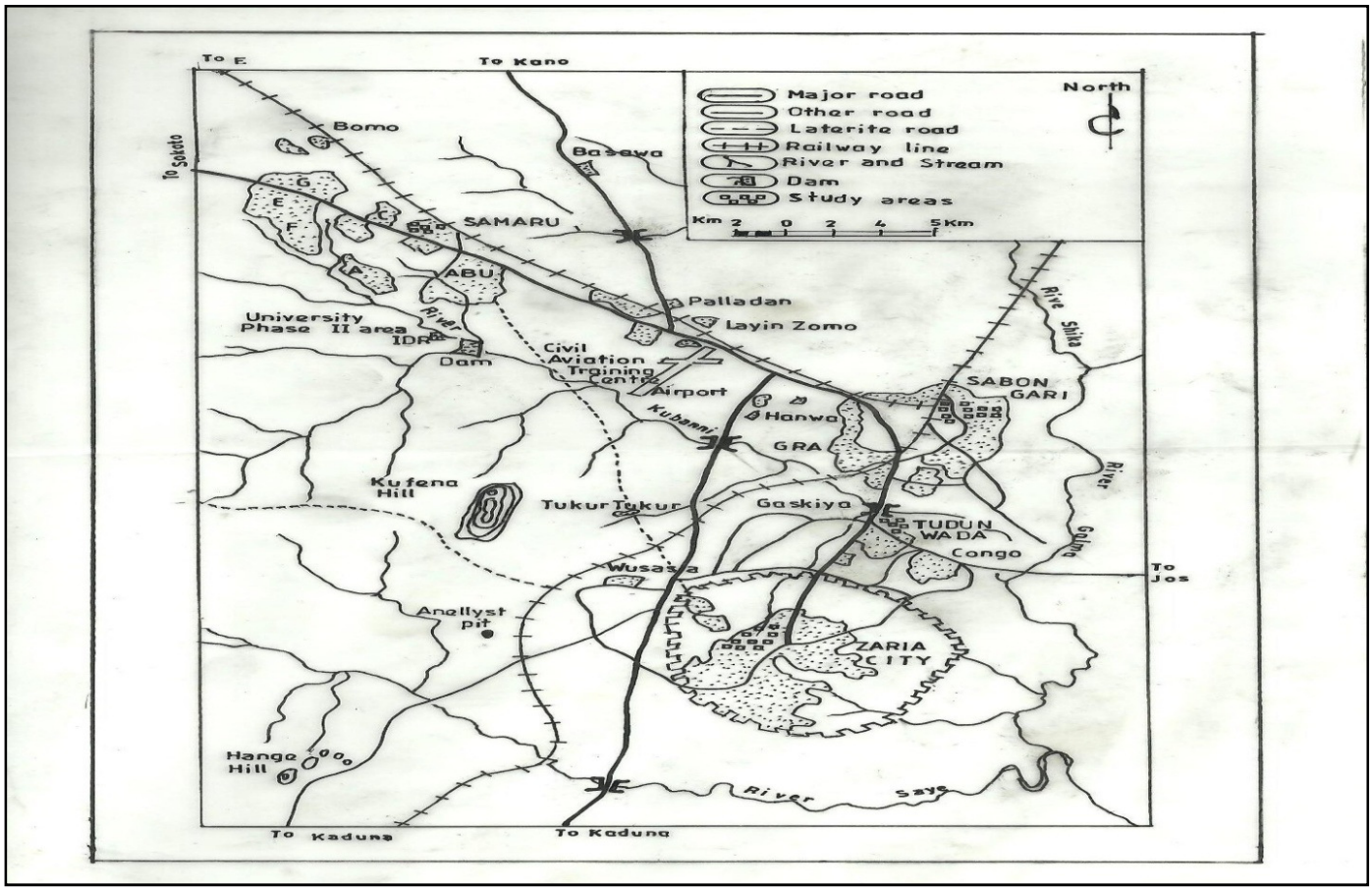

Figure 1: Map showing the sampling area

Product Code	Manufacture date	Expiry date	NAFDAC No.	Batch No.
PX	+	+	+	+
AT	-	-	+	-
COC	-	-	+	-
DAM	+	+	+	+
FEM	+	+	+	+
APP	+	+	+	+
SW	-	-	+	-
SM	+	+	+	+
S8	-	-	+	-
TS	-	-	+	-

Table 1: Container label information on collected hair cream samples
(+) Label disclosure provided, (-) Label disclosure not provided

2.1. Materials

Chemicals of analytical grade purity and distilled water were used in the preparation of reagents. All glassware used were washed and rinsed with distilled water before drying in the oven. The reagents required were purchased from Stevemoore Chemicals, Emanto, Zaria, Kaduna State, Nigeria. The Jenway digital pH meter model 3505 was used for pH determination. All the experiments were performed in triplicate and the average values were reported. Statistical analysis was carried out using SPSS to describe the pattern of distribution of the physicochemical parameter in all the samples under study &ANOVA was used to compare test parameters using the SPSS 16 software.

2.2. Physicochemical Analysis

Determination of pH was done according to Standard Organization of Nigeria (SON) protocol for pH determination in hair creams while Iodine value (I.V), Saponification value (S.V), Peroxide value (P.V), Acid value (A.V) and the Free-fatty acid value (F.F.A) were carried out using the American Standard for Testing Material (ASTM) Method-(D5558-95).

2.3. Physical Evaluation

2.3.1. pH Determination

Exactly 5g of the sample was weighed into a 100ml beaker. 45ml of water was added and the hair cream was dispersed in it. p^H of the suspension was determined at 27^{o}C using the Jenway pH meter model 3505 model [4].

The sensory properties were also determined by physical examination of color, odor, touch and clarity of all the samples.

2.4. Chemical Evaluation

2.4.1. Acid Value

Exactly 2g of each hair cream sample were weighed into 250ml conical flask. 50ml of neutralized ethyl alcohol was added. The mixture was heated on a water bath to dissolve the sample. The solution was titrated against 0.1M KOH using phenolphthalein as indicator. The acid value was determined after which the free fatty acid was calculated respectively as follows:

$$Acid\ value = \frac{AxMx56.1}{w}$$

Where: A = ml of 0.1M KOH consumed by sample, M = Molarity of KOH, W = Weight in grams of the sample. Then

$$Free\ fatty\ acid = \frac{acid\ value}{2}$$

2.4.2. Iodine Value

Exactly 0.5g of hair cream was weighed into conical flask and 20ml of carbon tetrachloride was added to dissolve the hair cream. 25ml of Wij's reagent was added to the flask using a measuring cylinder in a fume chamber. Stopper was then inserted and the content of the flask was vigorously swirled. The flask was then placed in the dark for 35minutes. At the end of this period, 20ml of 10% aqueous potassium iodide and 100ml of water were added using a measuring cylinder. The content was titrated with 0.1M sodium thiosulphate solution. Few drops of 1% starch indicator were added and the titration continued by adding the sodium thiosulphate drop wise until coloration disappeared after vigorously shaking. The same procedure was used for the blank test. The Iodine Value (I.V) is given by the expression:

$$Iodine\ value = \frac{12.69C\ (V1 - V2)}{M}$$

Where: C = concentration of sodium thiosulphate, V_1 = volume of sodium thiosulphate used for the blank, V_2 = volume of sodium thiosulphate used for determination, M = Mass of sample, 12.69 = constant

2.4.3. Saponification Value

Exactly 3g of the hair cream was weighed into the Erlenmeyer flask or conical flask. 25ml of 0.5M ethanolic KOH was added and the resulting mixture was refluxed for 60 minutes. The resulting solution was subsequently titrated against 0.5M HCl with phenolphthalein as indicator. The resulting end point was obtained when the pink colour changed into colourless. The same procedure was used for the blank. The Saponification value (SV) was then calculated using the expression:

$$S.V = \frac{56.1(B - S)xM\ of\ HCl}{weight\ of\ sample}$$

Where: B = ml of HCl required by blank, M = Molarity of KOH, W = weight in grams of the sample, 56.1 = Molar mass of KOH

2.4.4. Peroxide Value

Exactly 5g of the hair cream sample was weighed into a 250ml stoppered Erlenmeyer flask by graduated cylinder, added 30ml of acetic acid-chloroform solution in the ratio 2:3, shook to dissolve the hair cream sample and added 0.5ml of saturated potassium iodide solution. Exactly 0.5ml saturated Potassium Iodide solution was then added, shook for 1minute and added 30ml of water; this was titrated with 0.01M sodium thiosulphate, adding the titrant slowly with continuous vigorous shaking, until the yellow color was almost discharged. 5ml of starch solution was added and continued the titration, shaking vigorously, until the color is discharged (n1

ml of 0.01M sodium thiosulphate). A blank test under the same condition was carried out (n2 ml of 0.01M sodium). It was made sure the volume of 0.01M sodium thiosulphate used in the blank did not exceed 0.1ml.

$$Ip = \frac{10(n1 - n2)}{M}$$

Where: n_1 = ml of 0.01M sodium thiosulphate, n_2 = ml of 0.01M sodium thiosulphate consumed by blank, M = weight of the sample

3. Result and Discussion

3.1. Physical Characterization

The physical characterization of hair creams is unique for every preparation. Inconsistencies in the properties for a particular preparation would mean that the preparation has been adulterated [5]. The distinctive properties of each category of sample analyzed were the same for all the batches as shown in Table 2 below implying that there was consistency in producing the hair creams analyzed.

3.2. Physicochemical Analysis

p^H indicates the hydrogen ion concentration of a solution and it is the measure of the alkalinity or acidity of the solution. Uncontrolled amounts of pH in preparations can pose health problems [4]. Some of these health problems that can result from uncontrolled pH are acidosis which is a condition of decreased alkalinity of the blood tissues. The symptoms of acidosis may include sickly sweet breath, headache, nausea, vomiting, disturbances of tissues and Central Nervous System (CNS) functions. If the pH is above or below the acceptable limits or range for a particular preparation, it can also damage the kidneys and the liver, can irritate the skin or tissues, and can cause inflammation of skin and tissues. It can also penetrate the skin to cause deep sores. Ideally, the pH of hair creams should be close to the pH of the scalp mantle which is 5.50 [6]. Because of the inclusion of various substances in the formulation of hair creams such as soaps that are alkaline in nature as emulsifiers, the cosmetics may not have the slightly acidic pH that is required. As a result of this, the hair creams formulations are usually titrated to an acceptable pH by the addition of components such as phosphoric acid and citric acid [7]. From Table 4 of pH values, pH of the hair creams under study ranges from 5.783 ± 0.006 for SW to 2.043 ± 0.006 for DAM, with the trend in decreasing pH given as: SW > TS = AT > SM > S-8 > PX > COC > APP >FEM > DAM. Most of the hair cream's pH was close to the pH of the scalp mantle of 5.50, this is likely to make the hair creams more scalp compatible and less likely to cause scalp problems. Exception is DAM with pH 2.043 ± 0.006. The high acidic pH of DAM may be attributed to the fact that the manufacturer directly or indirectly skipped titrating the hair cream formulation to acceptable pH which is likely to irritate the scalp with long time usage, showing that the manufacturer failed to achieve a scalp compatible pH. ANOVA summary Table 3 shows that at 95% confidence value, $p<0.05$, implying that there is a significant difference among the pH values of all the hair creams under study.

The Iodine Value is the number of grams of Iodine that combines with 100g of oil or fat in the hair cream. An iodine solution is violet in color and any chemical group in the substance that reacts with iodine will make the color disappear at a precise concentration. The amount of Iodine solution thus required to keep the solution violet is a measure of the amount of Iodine sensitive groups. Superior shelf life of cosmetic base oil is essential to store the cosmetics over a longer period. Perusal of data presented in Table 4 shows that mean Iodine value (g/100g) of the hair creams varied from 39.163 ± 0.144 for DM to 9.656 ± 0.028 g/100g for Coconut oil (less than 100), with the trend in decreasing iodine value given as: DM > FEM > TS > AT > SW > S-8 > SM > APP > PX > COC. These values not only show that all the oil components of the hair cream samples are stable and resistant to oxidative rancidity conferring them a longer shelf life but also fall within non-drying oils (iodine value less than 100) and SON's standard of low unsaturation which are useful in the manufacture of soaps, hair creams and shaving creams [8]. The mass of Iodine in gram absorbed by 100 g the oil/fat in the natural state is known as Iodine number/Iodine value. It is an index of the degree of unsaturation of the fat or oil of the hair creams. Iodine value represents true unsaturation of fats only when double bonds are unconjugated and addition is not interfered by other groups. The higher the iodine value, the more unsaturated fatty acid bonds are present in a fat/oil. Greater the numbers of double bonds, the more prone the oil or fat component of the hair cream is to oxidation via the double bonds. The iodine values obtained in this study indicate that the oil component of the hair creams contain appreciable level of unsaturated bonds. Storage procedure used should ensure protection of oil from oxidative deterioration. ANOVA summary table shown in Table 3 shows that at 95% confidence value, $p<0.05$, implying that there is a significant difference among the iodine values of all the hair creams under study.

The saponification value had been reported to be inversely related to the average molecular weight of the fatty acids in the oil fractions of the hair creams [9]. Oil fractions of hair creams with saponification values of 200mgKOH/g and above as stipulated by SON, (S-8, FEM, SW, COC, AT and SM) as shown in Table 4, had been reported to possess low molecular weight fatty acids [9]. According to [10] a saponification value above 200 mg KOH/g indicates high proportion of fatty acids of low molecular weight. This shows that the oil may have a potential for use in soap making, cosmetics industry and for the thermal stabilization of poly vinyl chloride (PVC). Table 4 reveals that oils constituents of the hair creams under study with saponification values between 278.006 ± 2.856 for SM to 148.353 ± 1.079 mg KOH/g for APP have very low molecular weight fatty acids reason their oils are also used for making specialty soaps, shampoos, hair conditioners, moisturizers and shaving creams. Oil component of hair creams with saponification values less than 200 mg KOH/g like DM (186.970 ± 0.095), PX (186.576 ± 1.698), TS (165.183 ± 1.649) and APP (148.35 ± 0.623) will have a lower potential for use in soap making compared to those with saponification values greater than 200 mg KOH/g as shown in Table 4. The higher the Saponification number of a fat free from moisture and Unsaponifiable matter, the more soluble the soap that can be made from it. By implication the oil constituent of Soul mate with 278.006 ± 2.856 mg KOH/g will have more potential for use in

soap making and cosmetics industry compared with Apple having the lowest saponification value of 148.353 ± 1.079 mg KOH/g. The trend in decreasing saponification value is given as SM > AT > COC > SW > FEM > S-8 > DM > PX > TS >APP. Saponification value of oil serve as important parameters in determining the suitability of the oil in soap making, these type of oil is grouped among those yielding soaps of soft consistency. Hence the oil constituents of all the hair creams under study could be used for making hair creams since it requires oils of soft consistency. ANOVA summary table shown in Table 3 shows that at 95% confidence value, $p<0.05$, implying a significant difference among the saponification values of all the hair creams under study.

Peroxide values of the hair cream samples ranges from 9.790 ± 0.017 for SM to 4.193 ± 0.011 mg KOH/g for TS. The trend in decreasing peroxide value is given as: SM > FEM > PX > DAM > AT > SW > COC > APP > S-8 > TS. The peroxide values of the oil component of the hair creams under study were low which means just minimum oxidation has occurred and low aldehydes resulted although they may have volatilized. Low Peroxide values provide a clear guideline relating their oil component good shelf life. Peroxide value is an index of rancidity; thus the high peroxide value of oil indicates a poor resistance of the oil to peroxidation during storage [11]. The peroxide values of all the hair cream samples were below the maximum acceptable value of 10 meq/KOH/g set by the Standard Organization of Nigeria [4]. Peroxide value is an indication of level of deterioration of oil. The low peroxide value further confirms the stability of the oil of these hair creams. Fresh oils have values less than 10meq/kg. Higher values between 20 and 40 results to a rancid taste [12]. The low acid and peroxide values are indicators of the ability of the oil to resist lypolitic hydrolysis and oxidative deterioration [13]. ANOVA summary table shown in Table 3 shows that at 95% confidence value, $p<0.05$, implying a significant difference among the peroxide values of all the hair creams under study.

Acid value ranged between 4.703 ± 0.015 for DM to 1.684 ± 0.002 for TS same trend follows for Free-fatty acid values in Table 4. The trend in decreasing Acid value is given as: DM > S-8 > SM > COC > AT > PX > SW > APP > FEM > TS. Same trend also follows for free-fatty acid values. Among all hair creams under study, SM (4.130 ± 0.242), DAM (4.703 ± 0.015) and S-8 (4.470 ± 0.060) were slightly above the acceptable limit of 4 mg KOH/g by Standard Organization, 2004. These Acid value shows that there might have being an in vivo hydrolytic activities in the oil component of the hair creams or a slight incidence of hydrolysis of the triglycerides of the oil components of these hair creams which is an indicator of inadequate processing and storage conditions (i.e., high temperature and relative humidity, tissue damage) of these hair creams same applies for the free fatty acid values of same three hair cream samples SM (2.065 ± 0.121), DM (2.351 ± 0.007) and S-8 (2.235 ± 0.0304). The FFA can be reduced or removed by alkaline refining to increase the shelf-life of the oil component of these hair cream samples. FFA is a source of flavors and aromas. On one side, we have short chain FFA which tends to be water soluble and volatile with characteristic smell. On the other side, we have long chain saturated and unsaturated fatty acids. The later are more prone to oxidation in their free form and their breakdown products (aldehyde, ketones, alcohols, and organic acids) provide characteristic flavors and aromas. In most cases these flavors and aromas are considered a defect in oils, fats, and foods that contain them. However, there are instances where hydrolysis of triglycerides and oxidation of FFA is a key in the development of desirable flavor and aroma in foods. This is the case of aged cheeses and some processed meats. ANOVA summary table shown in Table 3 shows that at 95% confidence value, $p<0.05$, implying that there is a significant difference among the Acid value and Free-fatty of all the hair creams under study.

S/N	Sample	Batch	Color	Odor	Touch	Clarity
1	**PX**	PX_{123}	Yellow	Choking	Smooth	Opaque, N. V
2	**AT**	AT_{123}	Yellow	Choking	Smooth	Opaque, N. V
3	**COC**	COC_{123}	Colorless	Sweet	Smooth	Opaque, N. V
4	**SW**	SW_{123}	Colorless	Slightly Choking	Smooth	Opaque, N. V
5	**TS**	TS_{123}	Yellow	Sweet	Smooth	Opaque, N. V
6	**S-8**	$S8_{123}$	Yellow	Choking	Smooth	Opaque, N. V
7	**SM**	SM_{123}	Yellow	Sweet	Smooth	Opaque, N. V
8	**DM**	DM_{123}	Orange	Choking	Smooth	Opaque, N. V
9	**APP**	APP_{123}	Yellow	Choking	Smooth	Opaque, N. V
10	**FEM**	FEM_{123}	Orange	Sweet smell	Smooth	Opaque, N. V

Table 2: Physical Characteristics of the hair cream samples
Subscript $_{123}$: Stands for the three batches comprising each of the hair cream samples under study
N.V: Non-viscous

Parameters					
pH	**Iodine value**	**Saponification value**	**Peroxide value**	**Acid value**	**Free-fatty acid value**
P<0.05	P<0.05	P<0.05	P<0.05	P<0.05	P<0.05

Table 3: ANOVA summary table for physicochemical parameters

S/No	Parameter	App	TS	PX	COC	SM	DAM	S-8	SW	FEM	AT
1	pH	4.583± 0.003	5.393± 0.006	4.626± 0.003	4.613± 0.003	5.386± 0.007	2.043± 0.006	5.226± 0.004	5.783± 0.006	4.316± 0.003	5.393± 0.003
2	I.V(mg/g)	17.596 ± 0.300	29.943 ± 0.505	11.666± 0.005	9.656± 0.028	22.816± 0.015	39.163± 0.144	26.560± 0.294	29.183± 0.025	34.843± 0.305	29.430± 0.036
3	S.V(mg KOH/g)	148.35 ± 0.623	165.183 ± 1.649	186.576± 1.698	242.476± 0.623	278.006± 1.649	186.976± 0.095	205.386± 0.542	223.773± 0.544	222.830± 1.433	260.553± 2.159
4	P.V(meq/KOH /g)	5.386± 0.015	4.193± 0.011	8.023± 0.152	6.043± 0.075	9.790± 0.017	7.783± 0.020	4.203± 0.025	6.216± 0.021	8.590± 0.010	7.070±0.12 1
5	A.V(mg KOH/g)	2.243± 0.001	1.684± 0.002	3.363± 0.001	3.926± 0.001	4.130± 0.242	4.703± 0.015	4.470± 0.060	2.903± 0.277	1.700± 0.010	3.893± 0.020
6	F.F.A(mg KOH/g)	1.121± 0.0005	0.842± 0.0013	1.681±0.00 12	1.963±0.00 08	2.065±0.12 13	2.351±0.00 76	2.235±0.03 04	1.451±0.13 89	0.850±0.00 50	1.946±0.01 04

Table 4: Mean ± S.D values for physicochemical parameters of hair creams under study

4. Conclusion

The leading brands of hair creams in the market, APP, TS, PX, COC, SM, DM, S-8, SW, FEM and AT were standardized for their physicochemical properties and some parameters required by SON. The observations were as shown in Table-4 above. All the samples complied with the requirements of Physical characterization, pH, Iodine value, Saponification value, Peroxide value, Acid value and Free-fatty acid value. In addition to this the containers for packaging of all the samples were light weight and made up of transparent polymer. Most of the hair cream's pH was close to the pH of the scalp mantle of 5.50, this is likely to make the hair creams more scalp compatible and less likely to cause scalp problems. Exception is DAM with pH 2.043 ± 0.006. The high acidic pH of DAM may be attributed to the fact that the manufacturer intentionally skipped titrating the hair cream formulation to acceptable pH which though is likely to irritate the scalp with long time usage, will play a vital role in the preservative efficacy of DAM. These methods are simple and reproducible. Present findings suggest the selected parameters may be used in the standardization of hair creams.

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