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Original Article

Evaluation of the Behaviour of Violet Acid Pigment 43 in Formulations of Shampoos and Conditioners in Bleached Hair

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ABSTRACT

Objective: The objective of this work was to evaluate the behavior of Acid Violet 43 pigment shampoos and conditioners formulations and when applied to hair through 10 formulations. **Method:** 10 formulations were developed, these being 5 shampoos and 5 conditioners. Among 5 shampoos, 4 were pigmented in different concentrations and 1 served as control. In the same way it was done for the conditioners, being 4 conditioners pigmented in different concentrations and 1 conditioner for control. The samples were subjected to organoleptic and physicochemical characterization for 15 days, being evaluated appearance, color and odor, behavior after centrifugation, determination of pH, viscosity and foam content (for shampoo). **Result:** The samples showed satisfactory results in the tests, because their organoleptic and physicochemical characteristics remained stable throughout the analysis period. As for the behavior of the pigment on hair shaft, hair samples were bleached and washed with the formulations developed. Prewash and post wash solutions (wash water) were prepared at the same concentrations and were also read in a spectrophotometer at 570nm. For white, the depigmented products were used. It was observed that both the shampoos and the conditioners deposit pigment in the wires, however we verified by visual analysis that the conditioners are more effective. **Conclusion:** The wicks were also subjected to moisture content tests to verify if pigment interfered with the deposition of water in hair.

Key words: Acid violet 43, Bleaching, Semi-permanent hair dye, Hair dye, Shampoo.

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INTRODUCTION

New technologies have been sought not only to increase the offer of colors and simplification of processes, but also to make them more effective and faster; but also seeks to minimize damage to hair and human health.[1,2] Zanatta *et al.* discuss the strong trend in the world market for hair care. Several cosmetic technologies have been applied to hair dyes, making pigment agents accessible and increasing the variety of products as permanent, semi-permanent and temporary dyes.[3] Bleaching and exposure to oxidative dyes can also change the physical properties of the hair, as in these processes the disulfide bonds can be broken.[4] There is no great range of analytical methods for determining acid or basic hair dyes that involve s semi-permanent or temporary dyes. Most studies develop methods for the analysis of permanent dyes.[5]

Due to the popularity of products containing semi-permanent dyes, especially those containing Acid Violet 43, and the scarcity of methods of analysis to evaluate the action of the product on the hair yarn, it is fundamental to study the behavior of the product when applied to hair and when Incorporated into formulations of shampoo and conditioner.

Zhao describes that Acid Violet 43 is a semi-permanent dye used in hair dyes.[6] Andrisano reports that this type of tincture lasts around four to six washes by interacting to a moderate degree with the hair stem.[7]

MATERIALS AND METHODS

The research was developed mainly at the Pharmacy School of Federal University of Ceara (UFC), in the Laboratory of Pharmaceutical Technology and Laboratory of Clinical Analyzes of the same university.

Materials

The hair wicks used were Caucasian, light brown. They were purchased by the UFC Pharmacy School through the company Bella Hair ®. The sample of Acid Violet 43 (Lavanya Rebecca) was granted by the company Colormix® pigments.

Formulas and preparation techniques

Shampoo

Table 1 describes the qualitative and quantitative composition of the formulations. The shampoos were prepared under slow mechanical stirring under normal conditions of temperature and pressure.

100g of each formulation were produced, being different only about the pigment concentrations incorporated in the formulas. The shampoo 1 was formulated at 0.001% pigment, shampoo 2 at 0.0005%, shampoo 3 at 0.00025% and shampoo 4 at 0.0001%. The formulations were characterized for their organoleptic properties (aspect, color and odor), centrifugation test, viscosity and pH. These analyzes correspond to the first day (24 hr after production).

Conditioner

Table 2 describes the qualitative and quantitative composition of the conditioners. The products were handled according to the usual phase inversion technique. The aqueous and oily phases were heated separately and then the aqueous phase was slowly poured under the oil phase, both at 70-75°C under constant mechanical stirring. 100g of each formulation were produced, these being different only about the pigment concen-

Table 1: Percent composition (% w / w) of the raw materials used in each shampoo formulation.

Components (INCI)	Standard shampoo (%w/w)
Sodium Laureth 2 Sulfate	30%
Cocamidopropyl Betaine	5%
Cocoamide DEA	5%
Glycol Distearate	3%
Citric Acid	I.N.
Phenoxyethanol	0,05%
Sorbitol	3%
Propylene glycol	3%
Cyclomethicone	0,2%
Panthenol	1%
Destiled Aqua	q.s. 100ml
Acid Violet 43	-

Subtitle:

I / N: If necessary.

QS: Quantum sufficit

(-): Absence of product in formulation

trations incorporated in the formulas. Conditioner 1 was formulated at 0.001% pigment, conditioner 2 at 0.0005%, conditioner 3 at 0.00025% and conditioner 4 at 0.0001%. They were characterized for their organoleptic properties (aspect, color and smell), centrifugation test, viscosity and pH. These analyzes correspond to the first day (24 hr after production).

Centrifugation test

Before carrying out physicochemical characterization the formulations were centrifuged on the first day. At day 15 the samples were also centrifuged. All shampoos and conditioners were subjected to test at 3000 revolutions per minute (rpm) for 30 minutes as established.[8]

Organoleptic evaluation

The organoleptic evaluation was done according to the Guide of Stability of Cosmetic Products of the National Sanitary Surveillance Agency. The evaluation was made visually, verifying if there was any change in appearance, color and odor or indication of phase separation.

Physicochemical evaluation
pH determination

Samples were submitted to pH evaluation using a potentiometer. First, the electrode was calibrated with buffer solutions of pH 7.0 and 4.0, respectively. After the calibration, the pH was read by introducing the electrode directly into the sample.[9]

Viscosity determination

The viscosity was determined on day 1 (24 hr post-production) using rotational viscometer, using the spindle of number 3 for the conditioner formulations and spindle of number 1 for the shampoos. The results are expressed as centipoise (cP).

Shampoo foam content

The foam characterization assay was adapted from the cylinder agitation method described by Klein (2004).[10] In this method, a fixed amount of diluted shampoo is poured into a graduated cylinder. A stopper is placed over the cylinder and the system is inverted a fixed number of times and then the foam volume is measured.

For the content and maintenance of foam, a 10% (w / w) purified solution of the shampoo was prepared from the shampoo, 25 ml of the solution was poured into a closed 100 ml beaker and shaken manually and vertically, five consecutive times, measuring the foam at the end of the shaking and after five minutes.

The test was performed to ascertain whether the presence of pigment Acid Violet 43 would interfere with the shampoo's foam-keeping ability.

Evaluation of acid violet 43 in discolored hair wicks
Hair wick's bleaching

The hair was discolored by adapting the method described by Nakano (2006), with the hair wicks being treated with commercial decolorizing powder and hydrogen peroxide 30 volumes, the proportion of the mixture between decolorizing powder and hydrogen peroxide being 1: 2 (m / m), respectively. After preparation, the mixture was applied to the hair wicks and held for 40 min. The process was repeated, totaling two discolourations.[11]

Prewash solutions preparation: A solution of each of the produced formulations was prepared in volumetric flask. The solutions were prepared at the concentration of 0.1 g of product in 100 ml of distilled water. These calibration solutions were read in a spectrophotometer at 570nm and the absorbance's were compared to absorbance's from the wash water reading of the bleached hair wicks to verify if there was pigment deposition in the hair.

Hair wicks washing and preparation of post wash solutions: The hair wicks were washed in a similar manner to conventional washing, like occurs in the bath. The method described by Cuelho, Bonilha and Canto (2013) was adapted, being weighed 4 samples of bleached hair and 1 of natural hair, each containing 0.1 g of hair.[12] each sample was previously washed with standard shampoo and then dried naturally under the light. Then the hair wicks were submitted to a treatment (shampoo and conditioner), with 1000, 500, 250, 100 pigment concentration in the formulas (μg / ml).

The washes were given as follows: 0.1 g of hair was previously wetted in distilled water and then 0.1 g of shampoo was applied, with gentle movements between the thumb and middle fingers, from the root to the opposite end of the wires. So, the entire length of the hair was impregnated by the product. The process was carried out for 3 min. After that time, the sample was rinsed with 100mL of distilled water, this water being separated into a beaker, to make comparative reading with the comparison solutions.

The hair wicks were treated with 0.1 g of conditioner. After 3 min, the samples were rinsed with 100mL of water, this water being separated, to make comparative reading with the calibration solutions.

Study of pigment deposition content in the hair wicks

After the prewash and post wash solutions preparation the absorbances were checked at 570 nm in quartz cuvettes. The procedures were done in triplicate. One blank was made up of a standard shampoo dilution for the shampoo readings, and the other was the standard conditioner dilution for the conditioner readings. The dilutions of the whites were made in water in the ratio of 1: 1000, in the same concentration of the pre and post wash solutions of samples 1, 2, 3 and 4.

Through the application of the equation of the straight line of the shampoo and the conditioner the concentration of the Acid Violet 43 deposited in the hair was found.

Humidity

According to Silva (2012), when the hair sample is heated between 25 °C and 200°C refers to the dehydration of the more superficially bound water.[13] To determine the humidity and evaluation of cuticle damages, the water loss by desiccation was determined, adapting the method described in 5th ed. Of the first volume of the Brazilian Pharmacopoeia.[14]

The dried samples were weighed on analytical weighing-machine (PRECISA® MOD 205 TO SCC) and wet in distilled water for 1 min interval. Then, for another 1 min the samples were placed on absorbent paper to remove excess water. Wet samples were weighed and dried for a period of 2 hr in a drying oven at 102°C. After the drying oven period the samples were accommodated to cool in a desiccator. After cooling the samples were weighed.

Visual analysis

After washing and drying, the samples were arranged side by side from the samples submitted to the standard treatment to visually analyze the possible visual changes. The samples submitted to the treatments 1, 2, 3 and 4 were compared to the samples treated with standard treatment (without pigment).

RESULTS

Stability evaluation

The Figure 1 shows the results of the centrifugation test of the standard shampoo samples, shampoo 1 (1), shampoo 2 (2), shampoo 3 (3) and shampoo 4 (4) on day one (24 hr post- production), before storage in drying oven and at room temperature. At the end of the 15th day samples of both shampoo and conditioner were centrifuged.

Organoleptic evaluation

None of the 5 shampoo and conditioners formulation presented characteristics that would affect the stability of the formulations, according to the evaluated criteria. During a period of 15 days the formulations were submitted to the same temperature variation stress. They remained the same homogeneous, pearly white or violet color. In the color analysis the samples did not show any alteration, the pigmented samples presented a pearly violet coloration, varying in intensity according to the pigment concentration in the formulation. Already the standard shampoo formulation remained pearly white.

Physicochemical evaluation

pH: The pH values were obtained directly in the formulations maintained at room temperature. The values are presented in Table 3.

Viscosity and foam content: The shampoos' viscosity values were determined 24 hr after the formulations were produced. The number 1 spindle had been used and 2 rpm has been established. The results are expressed in centipoises: 2578 (1); 2010 (2); 1831(3); 1819 (4); 1813 (standard shampoo). Concentration of pigment versus viscosity of the shampoos and conditioner presented in Figure 2.

Like what happens in shampoo, more viscous conditioners are the more concentrated and as the pigment concentrations go down, the viscosity also decreases. The variation of foam height and viscosity in formulations was presented as: H1 (post inversion foam height), H2 (foam height after 5 min) and ΔH = (H2-H1).

Shampoo 1: 60cm (H1), 56cm (H2), 4cm (ΔH). Shampoo 2: 58cm (H1), 53cm (H2), 5cm (ΔH). Shampoo 3: 60cm (H1), 57cm (H2), 3cm (ΔH). Shampoo 4: 57cm (H1), 54cm (H2), 3cm (ΔH). Standard shampoo: 56cm (H1), 52cm (H2), 4cm (ΔH).

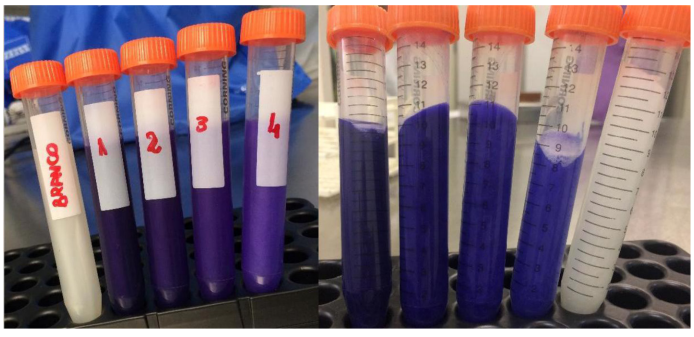

Figure 1: Shampoos and Conditioners after centrifugation.

Study of the efficiency of pigment deposition in bleached wicks

From the mathematical application of the equation contained in Figure 3, it was possible to obtain the amount of pigment present in the washing solutions and by the difference in the concentration of the prewash and post wash solutions, the amount of pigment deposited on the yarn after washing with shampoo.

The higher the concentration of the shampoo, the higher the concentration of pigment in the hair, but when working with pigment retention capacity, the results suggest that shampoo 2 may be the best pigment concentration option, because half the amount of shampoo 1 was used and the retention percentage was practically the same. Deposited pigment concentration after washing with shampoo and conditioner presented in Table 4.

HUMIDITY CONTENT

The results were described by the mass in the sample in: initial mass (Im), final mass (Fm) and Δ m.

Natural hair: 1,73g (Im); 1,29g (Fm); 0,44g (Δm). Standard treatment: 1,94g (Im); 1,04g (Fm); 0,9g (Δm). Treatment 1: 1,85g (Im); 1,00g (Fm); 0,85g (Δm). Treatment 21,87g (Im); 0,99g (Fm); 0,88g (Δm). Treatment

Table 2: Percent composition (% w / w) of the raw materials used in each conditioner formulation.

Components (INCI)	Standard conditioner (%w/w)
Cetyl Alcohol(1)	3,5%
Cyclomethicone(2)	5%
Polysorbate - 80(2)	3%
Citric Acid	I.N.
Tetrasodium edta (1)	0,05%
Sorbitol(2)	5%
Propylene glycol(2)	5%
Cetrimonium Chloride(2)	3%
Panthenol(2)	1%
Phenoxyethanol (2)	0,1%
BHT(1)	0,05%
Acid Violet 43(2)	-

Subtitle:

- Absence of the product in the formulation
(1) Components of Oily Phase
(2) Water Phase Components
I / N If necessary
QS Quantum sufficit

Figure 2: Graph of concentration of pigment versus viscosity of the shampoos and conditioner.

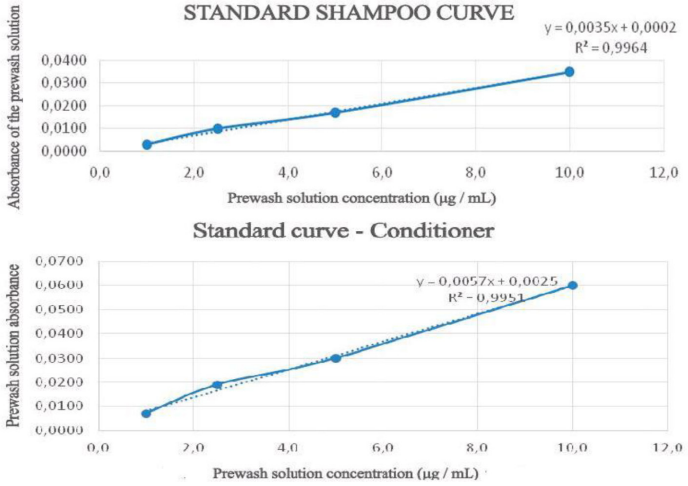

Figure 3: Standard Shampoo Curve.

Table 3: Shampoos' and conditioners' pH values on days 1, 7 and 15.

Formula	Standard Shampoo			Shampoo 1			Shampoo 2			Shampoo 3			Shampoo 4		
Day	1	7	15	1	7	15	1	7	15	1	7	15	1	7	15
pH	6,0	6,1	6,1	6,3	6,2	6,0	6,0	5,9	5,0	6,2	6,3	6,3	6,3	5,5	6,4

Formula	Standard Conditioner			Conditioner 1			Conditioner 2			Conditioner 3			Conditioner 4		
Day	1	7	15	1	7	15	1	7	15	1	7	15	1	7	15
pH	4,5	4,5	4,6	4,3	4,2	4,2	4,5	4,5	4,5	4,4	4,5	4,5	4,3	4,4	4,5

Table 4: Deposited pigment concentration after washing with shampoo and conditioner.

Product	[ ] prws mcg/mL	[ ] pows mcg/mL	µg/mL	% Deposited pigment
Shampoo 1	10,0	3,9998	6,0	60,57142857
Shampoo 2	5,0	1,714085714	3,3	66,85714286
Shampoo 3	2,5	1,9998	0,5	22,28571429
Shampoo 4	1,0	Out of detection limit	3,6	-

Product	[ ] prws mcg/mL	[ ] pows µg/mL	mcg/mL	% Deposited pigment
Conditioner 1	10,0	8,418552632	1,6	20,1754386
Conditioner 2	5,0	4,032587719	1,0	14,03508772
Conditioner 3	2,5	1,401008772	1,1	15,35087719
Conditioner 4	1,0	0,699254386	0,3	7,368421053

Subtitle:
[ ] Concentration
Prws: Prewash solution
POWS: Post wash solution
Δ [ ]: Variation in concentration / amount of deposited pigment

3: 1,70g (Im); 0,98g (Fm); 0,72g (Δm). Treatment 4: 1,98g (Im); 1,04g (Fm); 0,94g (Δm).

To determine the humidity and evaluation of cuticle damages, was determined the water loss by desiccation, adapting the method described in 5th ed. of the first volume of the Brazilian Pharmacopoeia.[14] By applying the mathematical formula: (Δm) x 100) / im, where Δm is the mass variation (final mass - initial mass) and im corresponds to the initial mass, we find the humidity content of the samples.

Natural hair: 25, 44%. Standard treatment: 46, 39%. Treatment 1: 45, 94%. Treatment 2: 47, 05%. Treatment 3: 42, 35%. Treatment 4: 47, 47%

Figure 4: Hair wicks post treatment.

VISUAL ANALYSIS

After washing and drying, the samples were arranged side by side, from the samples submitted to the standard treatment to visually analyze the possible visual changes. The samples submitted to the treatments 1, 2, 3 and 4 were compared to the samples treated with standard treatment (without pigment).

In view of the treatments applied to the hair, it was observed that the samples washed with treatment 1 presented a grader color, when compared with the others, proving that the higher the concentration of pigment Acid Violet 43 in the formulations, the greater the masking yellowish tones capacity, as can be observed in Figure 4.

DISCUSSION

The centrifugation promotes a gravitational force on the sample, causing stress against the formulation, increasing the mobility of the particles, making it possible to anticipate possible changes in stability.[8] The test performed on day 1 and day 15 showed that none of the shampoo and conditioner formulations presented physical instability. All the samples maintained the same homogeneous appearance after the preparation, thus demonstrating that the pigment did not interfere with the stability of the product. There were no large pH variations of the shampoo and conditioner preparations and the shampoo formulas remained within the range (5.0 and 7.0) as suitable for these formulations, demonstrating the stability of the formulation to this factor.

It is important that the formulation has a viscosity that allows the product to spread well through the scalp and does not drain from the hands during application.[15] The pigment concentrations in the formulations are directly proportional to the viscosity of the formulations. The shampoo 1, which is more concentrated, has a higher viscosity and as the pigment concentration in the formulations decreases, so does the viscosity drop. It was noted that the standard (non-pigmented) conditioner had a viscosity that was slightly higher than the 0.0001 concentration conditioner, as can be seen in Figure 3.

Considering the linearity of the viscosity / concentration ratio of the other formulations, it is believed that this slight difference results from some variation in the pigment incorporation technique.

The presence of a sodium atom in the structure of Acid Violet 43 may be the explanation for this phenomenon of increase of viscosity, since in the conditioners it can be ionically bound to a chloride ion of Cetrimonium Chloride, forming NaCl, an important donor of viscosity.

For the shampoos, Gomes and Pires (2014) affirm that sodium chloride can be obtained in the formulations through a chemical reaction of sodium lauryl ether sulfate, which was used in standard shampoo formulations 1,2,3,4. The combination with anthraquinones sodium may cause a reaction, thus increasing the viscosity of the shampoos.[16]

Couto *et al.* reports that as the viscosity increases the foam content also increases. The phenomenon was not clearly observed in the shampoos formulations, since the density variations are discrete, in addition to the fact that the test is carried out by manual agitation, and there is no constancy in the force applied at the time of the inversion of the system.[17] Although the hypothesis that the pigment interferes with the viscosity of the formulations and that the viscosity interferes with the foaming is confirmed, it has not been found that the pigment Acid Violet 43 has the ability to improve foam ability.

The same procedure was performed to verify the pigment concentration after application of the conditioner, however, since it contained both the pigment of the shampoo application and conditioner's, the post wash solution of the conditioners was more concentrated than expected to a post wash solution when only the conditioner is applied, disrupting the test's fidelity. In view of the application of the formula, it can be concluded that washing with the conditioner would be less effective than washing with the shampoo, however, during the washing process, it was visually found that the pigment acted more efficiently by applying the conditioner.

The outermost layer of the yarn, the cuticle, is the structure responsible for providing chemical resistance in addition to controlling the amount of water in the structure.[18]

Under normal conditions, the hair has 10% of water retained, but this content varies according to the relative humidity of the air. When wet the hair absorbs an amount equivalent to 30% of its weight.[18]

The results show that the sample of natural hair (not discolored) presented lower humidity content when compared to the other samples, which refers to the better control of water retention in the hair.

The standard treatments, 1, 2, 3 and 4 were applied to bleached hair and the results were very similar to each other however they showed a difference of up to 22.03% in relation to the natural hair humidity.

The obtained quotients show that the pigment Acid Violet 43, even in different concentrations, does not interfere in the humidity content of the hair, nor in the structure of the cuticle, confirming the hypothesis that the bleaching process, not the pigment itself, who damages the cuticle, leading to an increase in water retention in the hair.

CONCLUSION

The pigment Acid Violet 43 was stable in formulations and effective in neutralizing orange and reddish tones of bleached hair. In the organoleptic evaluation the pigment did not interfere in the odor or in the appearance of the products.

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CONFLICT OF INTEREST

None.

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