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Quantification of Two Bleaching Agents: Hydroquinone and Clobetasol-17 Propionate in Lightening Creams and Body Milks Marketed in the City of Douala (Cameroun)

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Abstract: Hydroquinone (HQ) and clobetasol 17-propionate (17PC) are two medicinal agents commonly incorporated because of their skin bleaching properties into lightening cosmetics sold out in some sub-Saharan regions (Senegal, Togo, South Africa, Cameroon...). The aim of this study was to quantify these agents in lightening creams and body lotions marketed in Douala, the economic capital city of Cameroon. A preliminary investigation was carried out at the main cosmetics sales outlets (pharmacies, perfumeries and supermarkets) of the city, in order to come out the overall proportion of lightening cosmetics and to inventory the mainly bleaching agents listed as ingredients in these products. About forty lightening creams and body lotions were purchased and then analyzed in other to come out their respective hydroquinone and 17PC concentrations. The results have revealed a compliance rate of 79%, with 47.5% of the samples containing hydroquinone at a higher concentration than the one allowed in cosmetics ($\leq 2\%$). Clobetasol 17-propionate, forbidden in cosmetics, has been identified in all the samples with 77.5% having a dosage above the 0.05% accepted medicinal creams, what may lead to systemic repercussions when used chronically.

Keywords: hydroquinone, clobetasol 17-propionate, skin bleaching, lightening creams and body milks, Douala

INTRODUCTION

With a cosmetic market estimated to about 10.5 billion Euros (6825 billion CFA francs) for the year 2017, the African continent is considered by many experts as the future of the international cosmetic market [1-3]. However, since some couples of years, many brands have been gradually associated to a practice that has gradually become popular and polemic in black and sub-Saharan communities: "skin bleaching" and the use of illicit agents to lighten the skin.

Today, they are various types of bleaching products available on African and Cameroonian markets. It goes from classics lightening soaps and body milks to very specific products (concentrated serums; clarifying face lotions; super-lightening gels; masks...) and more often, the difference between "cosmetic" (use to keep a healthy skin) and "medicine" (use to heal or prevent a sickness) is difficult to establish. It is in a such context that the present study will first of all try to take a census of bleaching agents listed the most on lightening creams and body milks marketed in the city of Douala, economic capital of Cameroon and commercial leader of Central Africa sub-region. Then, it will last on two particular agents: hydroquinone and clobetasol 17-propionate, both used for their bleaching properties, in order to identify and quantify them in forty lightening creams and body milks.

MATERIAL AND METHODS Investigation running

A preview investigation was carried out at the 05 principals districts of Douala city (table I).

Table-I: Districts and neighborhoods investigated					
Districts Capital Neighborhoods investigated					
Douala 1	Bonanjo	Akwa, Mboppi, Bali, Bonapriso.			
Douala 2	New-bell	New-bell, Carrefour Anatole.			
Douala 3	Logbaba	Logbaba, Nyalla, Genie militaire, Yassa.			
Douala 4	Bonassama	Bonassama, Sodiko, Ndobo, Bonabéri.			
Douala 5	Kotto	Bonamoussadi, Logpom, Beedi, Ange Raphael, pk12.			

ETAME LOE Gisele et al., Saudi J. Med. Pharm. Sci., Vol-4, Iss-9 (Sept, 2018): 1046-1052

In each of these districts, the visited neighborhoods were chosen after a randomized selection and the three mains cosmetic sale outlets (pharmacies, perfumeries and supermarkets) were then investigated at a rate of five pharmacies and five other sale outlets (perfumeries and supermarkets) per district, which means a total of 50 investigated sale outlets [4]. The samples collected were analyzed at GENEMARK.SA quality control laboratory, located at Yassa (Douala-Cameroon).

Population

The study was concerning the two major types of cosmetic emulsions: creams and beauty (or body) milks. The including and excluding factors are resumed in Table II.

Table-11. Including an excluding factors							
	Including factors	Excluding factors					
1-Presentation :	Creams and body milks	Any other cosmetic different from creams					
2-Cosmetic	"Lightening" mention or synonyms: clarifying,	and body milks.					
properties :	bleaching	Example : lotions, soaps, gels were					
		excluded					

Table-II: Including an excluding factors

Forty lightening creams and body lotions were then purchased out of the total number of lightening cosmetics censed during the investigations.

Material and reagents

The various tests were carried out using the following equipment: a METLER AE240 balance (accuracy 10^{-3} g); an electronic pH-meter from « pHep » HANNA; a UV/Visible « GENESYS 10UV » spectrophotometer; a « STAR » UV lamp; 20 X 20 cm silica coated plates; an electronic hotplate and a thermometer. The following reagents were also needed: standard hydroquinone; hexane; acetone; ethanol 95°; sulfuric acid solution (0.05 M); a 0.05% 17PC standard medicinal cream (DERMOVAL®); samples of lightening cosmetics to analyze; Bromothymol Blue (BBT) and distilled water.

Experimentations

Some organoleptic properties such as sample's color, smell and texture were checked. Their physicochemical properties like pH and emulsion's types (O/W-oil in water- or W/O -water in oil-) were also determined using respectively an electronic pH-meter and the "colorants method" (dissolution or no of an aqueous solvent- BBT- in the samples) [5].

Hydroquinone identification and quantification

Hydroquinone was identified by thin layer chromatography (TLC), following the procedure described by Siddique S *et al* in 2012 [6]. It quantification has required a sulfuric UV spectrophotometric dosage, at the wavelength of λ =302nm, using the 2013 Kipngetich E. method [7].

Clobetasol 17-propionate detection and quantification

It has also required a UV spectrophotometric method (λ =239nm), but this time in an ethanolic environment and using the process described by Neelam D *et al.* in 2016 [8].

RESULTS

Investigation on bleaching agents found the most in lightening creams and body lotions marketed in Douala

A total of 297 different lightening creams and body lotions were identified out of the 681 censed (lightening and non-lightening) during the investigations, what gives a global proportion of 43.6% lightening cosmetics for 56.4% non-lightening. This proportion has been also estimated in each district and the results were reported on figure 1.

ETAME LOE Gisele et al., Saudi J. Med. Pharm. Sci., Vol-4, Iss-9 (Sept, 2018): 1046-1052



Fig-1: Proportion of lightening and non-lightening cosmetics over the 05 investigated districts

The figure 2 is representing the distribution of lightening cosmetics according to their presentations:

cream tubes (5%), cream pots (14%) and body milks (81%).



Fig-2: Distribution of lightening cosmetics according to their presentations

The most often countries listed as origins of these bleaching products were reported in decreasing order in figure 3 and the most cited bleaching agents were also censed and reported in decreasing order in figure 4.



Countries mentioned on cosmetic's labels (%)





bleaching agents mentioned on cosmetics labels (%)

Fig-4: Distribution of lightening cosmetics according to the bleaching agents mentioned on their labels

Concerning the physicochemical and organoleptic properties, samples colors varied from white to pink, with a smooth texture. Their smell was going from floral notes (rose, lavender...) to fruity smell (orange, lemon, vanilla, coconut, cocoa...). All of them were O/W (oil in water) emulsions (dissolution of BBT in all the samples) with pH between 4 and 6.9.

Over the 40 analyzed samples, 24 simultaneously had the both agents and 16 only had 17PC.

Hydroquinone's identification and dosage

The TLC analyze has permitted to identify HQ in 24 samples, while 16 revealed no trace (table III). The obtained concentrations were reported in table IV and V.

Hydroquinone and 17PC research and quantification in samples

Table-III : Hydroquinone TLC identification result							
	HQ presence	HQ absence	TOTAL				
Samples	24	16	40				
Frequency (%)	60	40	100				

	Table-TV: Hydroquinone concentrations of analyzed samples																	
% HQ ± 0,22	8. 35	7. 5	6. 85	6. 6	5. 6	4. 85	4. 2	4 .3	2. 95	2. 65	2. 35	2. 16	1. 9	1. 55	1. 15	0. 8	0. 15	0
Effectives	1	1	1	2	2	3	1	4	1	1	1	1	1	1	1	1	1	16

Table IV. Hydroguinene concentrations of analyzed complex

Table-V: Hydroquinone concentrations gaps

HO concentration (%)	0	10:21	12:51	15:10]	TOTAL
Samples	16	5	12	7	40

The compliance rate of hydroquinone concentrations to the standard ($\leq 2\%$) was 52.5% (table VI).

Table-VI: Compliance rate of hydroquinone concentrations to the standard (≤2%)

HQ concentration	Compliant	Non compliant	
Frequency	52,5%	47,5%	

17PC Détection and dosage 17-propionate de clobétasol

The spectrophotometric analyze has revealed 17PC's presence in the 40 samples and their concentrations were reported in tables VII and VIII.

Table-VII: 17PC's concentrations in analyzed samples												
% 17PC± 0,0033	0.12	0.11	0.1	0.09	0.08	0.07	0.06	0.05	0.04	0.03	0.02	0.01
Effectives	2	2	2	6	10	7	2	1	3	3	1	1

Table-VIII: 17PC's concentrations gaps							
17PC concentration (%)	[0,01;0.05]] 0.05 ; 1.2]	TOTAL				
Samples	9	31	40				
Frequency	22,5%	77,5%					

What means a 0% compliance rate, as 17PC is forbidden from cosmetics (table IX).

Table -IX: Compliance rate of 17PC's concentrations to the standard

17PC concentration	Compliant	Non-compliant
Frequency	0%	100%

From these results, the veracity of sample's labels has been checked (table X) and those with true allegations (mentioned and effectively present + non-

mentioned and effectively absent) or false allegations (non-mentioned but effectively present + mentioned but absent) were identified and reported in table X.

Table-X: Labels veracity on the presence or not of HQ and 17PC in analyzed cosmetics

	Real allegations	Wrong allegations
HQ	85%	15%
17PC	73%	27%
GLOBAL	79%	21%

DISCUSSION

The investigation carried out through various cosmetic sale outlets of Douala city has revealed that the proportion of lightening cosmetics (43.6% against 56.4% non-lightening), lower than non-lightening cosmetics proportion, is nevertheless significant enough from a statistical and epidemiologic viewpoint to aware public authorities on skin bleaching.

By looking closer to these results within the different districts, it appears that lightening creams and body milks consumption is higher in commercial areas (Douala 2 district for example, which includes central market, women market, Congo market..., records the higher proportion of lightening cosmetics: 62%) than in manufacturing areas (Douala 3 and 4 districts including respectively the Yassa and Bonaberi industrial zones, have recorded the lower lightening cosmetic proportions: 38,8% and 40% respectively).

Body milks were the favorite types of lightening cosmetic emulsions (81%), more available than creams. This can be due to the fact that in skin bleaching practice, milks represent the base of "homemade preparations", in which various quantities of highly concentrated lightening creams or lotions are added [9,10].

Concerning the origin of these products, the forefront place of France had already been cited in past years (DASS, 2003) [11]. It can also be explain by the actual economic context. In fact, since the publication in 2012of the *Euromonitor international* study's results, many cosmetic giants (UNILEVER© L'OREAL © ...) settled down in Africa and particularly in Cameroon, in order to facilitate the establishment of their labels in nearby countries (Gabon, Equatorial Guinea...) [1, 2]. However, the second place occupied by Cameroon following our results, indicates that local companies (BIOPHARMA©, LANA COSMETICS© ...) are exerting a strong competition on all these international brands and intend to bring the trend back to local consumption [2].

Natural bleaching agents like AHA (alphahydroxylated acids) were the most mentioned on cosmetic's labels (13%), while synthetic agents as hydroquinone and 17PC respectively represented 10.5 and 3%. This proportion associated to those of all the other natural bleaching agents (kojic acid, licorice extracts, vitamin E...), demonstrates that manufacturers today prefer to put ahead the natural side of their cosmetics as a commercial tool. The analyzed samples have revealed no major non-compliances concerning their organoleptics characteristics. About their physicochemical properties, all the samples were having a pH between 4 and 6, in agreement with skin's pH. In fact, a weak acid pH constitutes one of the favorable conditions for skin absorption, just like particle sizes, skin's state or thickness [5]. They were also all O/W (oil in water) emulsions, what can be explain by the fact that in this kind of formulation, active ingredients are in the internal phase and so better protect form diverse alteration process like oxidations [12].

Among the two researched agents, hydroquinone has been identified in 60% of the samples, each time in association with 17PC. The obtained concentrations were going from 0.15 to 8.35 %, with 19 values higher than the 2% authorized in cosmetics, which mean 47.5% of samples with medicinal concentrations of hydroquinone. Such results are demonstrating that cosmetics, normally intended for esthetic use, are now turned away with medicinal concentrations which may lead to diverse diseases [13-19]. In 2003, such results were published in Geneva-Switzerland where the presence of hydroquinone was revealed in 28.6% of the samples at toxic concentrations going to 21.4 % [11]. Recently, Metogbe et al. published an article listing concentrations going from 2 to 6 % in cosmetics bought in eastern Africa and Canada [20].

Clobetasol 17-propionate, forbidden from cosmetics according to cosmetic regulation authorities, has been identified in all the analyzed samples with only 22.5% having a concentration lower or equal to therapeutic dosages [19]. The concentrations found were going from 0.01 to 0.12%, two times higher than the 0.05% allowed medicinal creams. These results are closes to those from a similar study carried out in Ivory Coast by Ake *et al.* They had found 17PC in almost all the analyzed samples (92.22%), with only 25.3% of obtained concentrations \leq 0.05% and 0,102% as maximum concentration [21].

At last, 79% of cosmetic labels were true: 85% of labels were right about the presence or not of hydroquinone in their formulation and 73% for 17PC, so a global rate of 21% false allegations.

However, 10% of these cosmetics had an unknown composition, and 20% did not even mention in which country they were manufactured. This should be an alarming report when all the short and long term side effects of skin bleaching are reminded, and knowing that some countries that ban and report the use of hydroquinone and 17PC as bleaching agents, do export them in some African nations [22].

CONCLUSION

The data obtained have permitted to finally conclude that lightening creams and body lotions marketed in the city of Douala are potentially dangerous products. The various bleaching agents that they contain (hydroquinone and clobetasol 17-propionate), used at too high concentrations and for extended periods, may perhaps lead to serious complications (kidney deficiency, diabetes, high blood pressure...) for user's health.

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