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A Cosmetic Analysis in Compliance with the Legislative Requirements, Halal and Quality Control

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Abstract: Cosmetic products must be safe for use by consumers. It is also regulated and required by the legislation of countries all over the world. The safety aspect is also in line with and fulfills the halal and toyyiban requirements under the Syariah Law that requires products to be unharmful to the consumer and user. In order to ensure that the cosmetic products meet the safety requirements, quality control measures in physico-chemical and microbiological analysis as well as *in-vitro* testing of skin irritation are carried out and presented in detail.

Keywords: *Cosmetic, safety, physico-chemical analysis, halal, microbiology, skin irritation*.

Introduction

Cosmetic products in Malaysia are regulated under the Control of Drugs and Cosmetic Regulations 1984. It is under the jurisdiction of National Pharmaceutical Control Bureau (NPCB), Ministry of Health. It is defined as the national regulation and legal aspects of cosmetic control. The Bureau is responsible for the registration, licensing and surveillance of all cosmetic products. The Regulations has been amended in 2007 to include the amendment of the notification procedure and transpose the ASEAN Cosmetic Directive (ACD) into the Guidelines for Control of Cosmetic Products in Malaysia (1).

According to the definition in the Guidelines, cosmetic product shall mean any substance or preparation intended to be placed in contact with various external parts of the human body (epidermis, hair system, nails, lips and external genital organs) or with teeth and mucous membranes of the oral cavity, with a view exclusively or mainly to cleaning them, perfuming them, changing their appearance and/or correcting body odors and/or protecting them in good condition.

In modern society, the main purpose for using cosmetics are for personal hygiene, enhance attractiveness through makeup, to improve self esteem and promote tranquility, protect skin and hair from damaging ultraviolet light, pollutants, and other environment factors, prevent aging and in general to help people enjoy a full and rewarding life.

In daily life, cosmetics are becoming very important; they are used daily and regularly by increasing numbers of people and the quantities consumed are increasing each year. In the year 2005, Malaysian total retail sales of cosmetic products are valued at RM3.2 billion, of which the skincare sector showed the highest sales (RM800 million), followed by hair care (RM600 million), bath and shower (RM500 million), oral care and color cosmetics (2). The total percentage value growth of cosmetic products from year 2000 to 2005 was 25.7%, whereas the cumulative average growth rate (CAGR) is about 5%.

Safety standards for cosmetic products vary between different parts of the world. However since globalization, Malaysia and ASEAN countries have put in place a relatively stringent regulatory framework to ensure cosmetic safety. Cosmetic products are not allowed to be placed in the market unless their safety has been scientifically proven. The production of cosmetic and personal care products has to meet several objectives before they could be released for sales. One of the most important objectives is to comply with the regulation enforced in the country and meet the quality control requirement. If it is required to be certified as a halal product, then it must follow the halal specification requirement as in the Malaysian Standard MS 2200: Islamic Consumer Goods – Part 1: Cosmetic and Personal Care Products – General Guidelines (3).

Before the cosmetic products are put in the market, the industry has to comply with the Cosmetic Regulation by notifying the regulatory authority. The notification is to ensure that their products do not cause any damage to human health and to establish the product information file (PIF). The PIF is a file consisting of the qualitative and quantitative composition of the product (name and percentage of the ingredients), raw materials specifications, and manufacturing method as in Good Manufacturing Practice (GMP) Guidelines (4), assessment of the safety for human health of the finished product, its ingredients, its chemical structure and its level of exposure. It should also include data on the undesirable effects on human health resulting from the use of the product, supporting data for claimed benefits (efficacy assessment), certificate of analysis to check the ingredients and microbiological control.

In Malaysia, the certification body responsible for granting Malaysia Halal logo is the Islamic Development Department of the Prime Minister Department. For cosmetic and personal care products that require halal certification, the products shall follow the Malaysian Standard MS 2200: 2008 requirement.

Halal is things or actions permitted and allowed by Syariah Law. The Syariah Law is Islamic Law based on the Quran, Hadith, Ijma' and Qiyas together with religious ruling (fatwa) issued by competent Islamic Authority. In every aspect of halal, the toyyiban aspect is included. Besides fulfilling the Syariah Law, which is a must for Muslims, the safety factor plays a significant role and contributor in determining the toyyiban i.e. quality aspect of products. In fact, the halal and toyyiban can be synonymous with halal quality. The product has quality if it is clean, pure, nutritious, hygienic and healthy. Therefore the halal products also meet the safety and quality requirement to fulfill the customer needs.

In Malaysia, the certification body responsible for granting Malaysia Halal logo is the Islamic Development Department of the Prime Minister Department. For cosmetic and personal care products that require halal certification, the products shall follow the Malaysian Standard MS 2200: 2008 requirements. According to the MS 2200, cosmetic products must be safe and not hazardous to users and consumers. It also means the products have certain quality and meet the requirement of consumers in terms of its use and efficacy. In brief, the halal cosmetics are products that must not have human parts or ingredients derived from thereof; not contain any animal forbidden to Muslim or are not slaughtered according to Syariah Law; no genetic modified organism (GMO) which are decreed as najs; no alcohol from alcoholic drinks (khamar); no contamination from najs during preparation, processing, manufacturing and storage; safe for consumer and comply with legislation in force in Malaysia.

Quality control of cosmetic and personal care products aims to provide assurance that the products will be of consistent quality appropriate for their intended use. The involvements of all concerned at all stages are mandatory towards the achievement of this objective, from the start of manufacturing to the distribution of the finished products. In general, requirements of the Cosmetic Regulation will cover physico-chemical properties, safety, microbiology and efficacy, whereas the halal compliance will include the safety, microbiology, efficacy, alcohol and animal origin ingredients detection.

The physico-chemical properties such as solubility, pH, viscosity and color are established during product formulation and production as a means of quality control (5). It is also important parameters indicating usefulness and purpose of products (6, 7). The finished products of cosmetic and personal care will be analyzed for safety, which include the toxic metal contaminants, skin and eye irritancy and microbiology testing. With regards to allergic and safety, the eye and skin irritation testing is carried *in vitro* before it can be used for the human evaluation (*in vivo*).

In this paper, the common cosmetic analysis carried out by the producers and the cosmetic R&D laboratory in compliance with the legislative requirement, halal and quality control will be highlighted and discussed.

Materials and Methods

Chemicals and Materials

Samples of cosmetic products (body scrub, body cellulite gel, thigh cellulite gel and body firming lotion) were developed in SIRIM laboratory. Lead, arsenic and mercury standards, nitric acids, sulphuric acid and hydrogen peroxide were obtained from Sigma Aldrich. Media for enumeration of bacteria and fungi were purchased from Difco. All other reagents used were of analytical grade.

Analytical equipment

The analysis of toxic metals contaminant for mercury were carried out using a Perkin-Elmer PE Optima 4300 DV Atomic absorption spectrometer – flow injection atomic spectrometer (AAS-FIAS) whereas lead and arsenic, Perkin-Elmer PE AAnalyst 300 optima inductive couple plasma- optical emission spectrometer (ICP-OES). For skin irritancy, a Dynex Technology Elisa reader was used.

Physico-chemical analysis (pH, viscosity and color)

The pH value of the cosmetic products was detected by a Mettler Toledo pH meter (S20 + Crison). Determination of viscosity was carried out using a Brookfield Viscometer (DV-II + Pro) applying spindle number S25 at 12 rpm speed. Colour was determined using a chromameter (Minolta CR-300) and reported using 'L', 'a', 'b' system.

Skin irritation analysis

The skin irritation analysis was conducted using the Dermal Irritection Test kit from Invitro International (Irvine, CA 92614, USA). This is a biochemical (*in vitro*) test that mimics an acute dermal irritation test. To perform this standardized assay, the test sample is applied to a synthetic biobarrier composed of a semi-permeable membrane containing a keratin-collagen matrix coated with a dye. Following application, the sample is absorbed by and permeates through this synthetic biobarrier to gradually come into contact with a proprietary solution containing highly-ordered globulins and glycoproteins. Reaction of the test sample with these proteins and macromolecular complexes promotes conformational changes that may be readily detected as an increase in the turbidity of the protein solution. In addition, the dye that has been dissociated from the biobarrier during transit of the applied sample may be detected spectrophotometrically at a wavelength of 450 nm.

The irritancy potential of a test sample is expressed as a Human Irritancy Equivalent (HIE) score. This score is defined by comparing the increase in optical density (OD_{450}) produced by the test material to a standard curve that is constructed by measuring the increase in OD_{450} produced by a set of Calibration substances provided with the testing kit. These Calibrators have been selected for use in this test because their irritancy potential has been previously documented in a series of *in vivo* investigations. The results are computed from Invitro International Irritation software provided by the manufacturer

Toxic metals analysis

For lead and arsenic determination – the sample (5 - 10 g) is dried on slow heat on sand bath, and then ashed at 450°C for 4 hr. A 10 ml of 10% nitric acid was added to the sample, filtered, and the lead and arsenic content was determined using Perkin Elmer ICP-OES instrument.

For Mercury determination – The sample (0.5 g) was added with 10 ml 4:1 sulphuric acid solution and left overnight. Then 5 ml of hydrogen peroxide was added drop-wise, followed by digestion in a water-bath shaker for 2 hr at 70°C, cooled and added with 20 ml 5% nitric acid. The solution was mixed thoroughly, homogenized, filtered and determination of the mercury content carried out by AAS-FIAS spectrometer instrument.

Microbiological analysis

The contamination of cosmetic products by microorganisms can be detected by determining the presence of bacteria via the aerobic plate count (APC) and the presence of other microorganisms via yeast and mould count (YMC). The total microbial count is calculated from the total count of APC and YMC.

Samples were prepared accordingly and serially diluted in modified leetheen broth. Each dilution (0.1 mL) was aseptically pipette and spread onto the surface of modified leetheen agar for APC and potato dextrose agar for YMC. Plates were incubated for 48 hours (for APC) and 7 days (for YMC) at 30º C (8). The colony forming units for each diluted sample was then counted and recorded.

Results and Discussion

Determination of pH, viscosity and color

The pH, viscosity and color measurements are important components of physico-chemical properties that determine the specification of a finished product and also raw materials. Results of pH measurement for the cosmetic products (body scrub, body cellulite gel, thigh cellulite gel and body firming lotion) were within the optimal range of pH 5.0 to 6.5 (Table 1). The pH results complied with the company specification requirements. This range of pH is suitable for the skin as the pH of the healthy skin is 5.5 and as person becomes older, the skin would turn more neutral (9). Measurement of pH can reflect the stability of a product because they are indicative of chemical reactions taking place (9). For example, compounds such as esters may hydrolyze and release free acid that could produce adverse effects. Such chemical changes can be monitored by measuring the pH. It is assumed that cosmetic products at the skin pH range are more compatible with the body (10). Furthermore, pH measurement can provide information about the quality of the product and gives clues as to the functionality of the product. Some materials will function differently as the pH changes.

The results of viscosity of the cosmetic products were within the range of the company specification (Table 1). Only the body scrub was highly viscous (63120 cps) as it was a paste and therefore it was thicker. Viscosity is another revealing parameter commonly used to evaluate the quality of finished products. It determines the proper consistency and can indicate product stability (11). Viscosity measurement is also an indicator of the thickness and flow properties of the products.

The color measurement of the cosmetic products was undertaken using the ‘L’, ‘a’, ‘b’ system, with only ‘L’ values presented (Table 1). The color of the finished product was recorded to detect the change of color when the product is undergoing stability test. In general, the company will set allowable maximum color difference after stability test and usually it did not exceed 3-5 ‘L’ units. The small color difference change implied that longer storage times and moderate increases in temperature only slightly affected the color (10).

Skin irritation analysis

The irritancy potential of a test sample for the skin is expressed as a human irritancy equivalent (HIE) score. The predicted in vivo classification, based on this scoring system is shown in Table 2. The results of skin irritation of body scrub and body firming lotion were 0.83 and to 0.79, respectively and can be classified as non-irritant (Table 3). The body and thigh cellulite gels gave HIE score 1.12 and 1.09 which were classified as mild irritant. All these readings comply with the company’s specification and also meet the requirements of quality control, safety and the regulation. Such products (body and thigh cellulite gels) tested results as mild irritant can be accepted (complied) as an indicator of a preliminary irritancy testing and used as a screening tool. In addition, we have to look at the function of cosmetic products. Cellulite products are meant to be mild irritant due to some ingredients like chili pepper that cause the burning sensation. The formulators have a choice to relook at the ingredients used and reformulate; and further reconfirming it by carrying out the human subject evaluation (*in vivo* evaluation). Conversely, if the products tested resulted as irritant, the products shall not be released for sale as it is not safe for consumer use. Therefore, the products will be destroyed and a new formula developed or reworked job is carried out to reduce the irritation risk.

Table 1: Color, pH and viscosity of cosmetic products

	Cosmetic products Results			Company specification		Remark
Cosmetic Product Samples	Color	pH	Viscosity (cps)	pH	Viscosity (cps)	
Body scrub	90.31	5.62	63120	5.0-6.5	10000-50000	complied
Body cellulite gel	26.27	5.04	6999	5.0-6.5	5000-10000	complied
Thigh cellulite gel	24.49	5.05	6999	5.0-6.5	5000-10000	complied
Body firming lotion	32.49	5.03	8958	5.0-6.5	5000-10000	Complied

Table 2: Relationship of human irritancy equivalent (HIE) score for skin to irritancy classification

Human irritancy equivalent (HIE) score for skin	Predicted skin irritancy classification
0.0 – 0.90	Non-irritant
0.90 – 1.20	Mild irritant
1.20 – 5.00	Irritant

Table 3: Skin irritation analysis of cosmetic products

	Skin irritation			
Cosmetic Product Samples	HIE score	Predicted skin irritancy score	Company specification	Remarks
Body scrub	0.83	Non-irritant	Non-irritant	Complied
Body cellulite gel	1.12	Mild irritant	Mild irritant	Complied
Thigh cellulite gel	1.09	Mild irritant	Mild irritant	Complied
Body firming lotion	0.79	Non-irritant	Non-irritant	Complied

The skin irritation analysis is a quantitative *in vitro* test that utilizes changes of relevant macromolecules to predict the acute skin irritancy of cosmetic formulations and ingredients. This method is an alternative method to Draize skin irritation test in rabbits according to OECD TG 404 (12). The assay is based on the principles that chemical compounds that cause skin irritation are known to induce alterations in the structure of keratin, collagen and other dermal proteins. The processes of conformational change that are induced in this *in vitro* assay mimic the effects that are produced when these types of irritants are applied to the skin.

One aspect of safety that concerns consumers can be described as mildness which is the product ability to perform its intended function without irritating the skin or eyes. Products that are harsh can cause irritation. Irritation, or commonly known as irritant contact dermatitis (ICD), the symptoms include erythema (redness), burning, itching and flaking. Skin irritation is one of the most common adverse effects of cosmetic products in humans depended in many factors, including the concentration, duration and frequency of exposure, exposed skin site, rate of penetration and intrinsic toxic potential of the substance in the product (13). In order to make sure that the product is safe and

does not cause any irritation to the user, skin and eye irritation analysis is carried out.

Only product that comes in contact with the eyes is subjected to eye irritation analysis. Consequently, the *in vitro* skin irritation test is a reasonable strategy to determine the potential skin irritancy and to predict the *in vivo* toxic effect of chemicals, cosmetic ingredients and formulations and new cosmetic products (14). The test serves as an extremely useful screening tool that facilitates all stages raw materials selection, formulation development and final product selection. This will ensure the safety aspect of the cosmetic products from causing irritancy has been eliminated and reduced.

Toxic metals contaminants

The toxic metals of primary toxicological concern in cosmetics are lead, arsenic, mercury, cadmium and antimony. However, the Malaysian Cosmetic Guidelines and the ASEAN Cosmetic Directive impose heavy metal specification of cosmetic with the maximum limit of lead 20 $mgkg^{-1}$, arsenic 5 $mgkg^{-1}$ and mercury 1 $mgkg^{-1}$. Cadmium and antimony are not being regulated. Table 4 showed the results of the toxic metal analysis (lead, arsenic and mercury) on cosmetic products were complied with the ASEAN specification. The detection of lead, arsenic and mercury level for all cosmetic products analyzed were 0.006, 0.004 and 0.1 $mgkg^{-1}$, respectively. The results were well below the maximum limit allowed under the regulation. Therefore, the products are safe and not cause harmful effect to consumer.

Table 4: Heavy metals contaminants of cosmetic products

Cosmetic Products	Lead (Pb) ($mgkg^{-1}$)	Arsenic (As) ($mgkg^{-1}$)	Mercury (Hg) ($mgkg^{-1}$)
Body scrub	0.006	0.004	0.1
Body cellulite gel	0.006	0.004	0.1
Thigh cellulite gel	0.006	0.004	0.1
Body firming lotion	0.006	0.004	0.1

**Note: ASEAN Cosmetic Specification Limit for lead (Pb) is 20 $mgkg^{-1}$, arsenic (As) is 1 $mgkg^{-1}$ and mercury (Hg) is 5 $mgkg^{-1}$.*

Toxic metals are naturally occurring, are present in the environment and make their way in trace quantities into raw materials (15). These substances end up in the products we consume and use every day. Skin exposure is expected to be the most significant route for cosmetic products since the majority of cosmetics are applied to the skin. Skin absorption of toxic metals is fairly minimal, with adsorption of individual elements influenced by a number of factors including physico-chemical properties of the mixture (16). Oral exposure can occur in the cosmetics used in and around mouth, as well as from hand-to-mouth contact after exposure to cosmetics containing toxic metals impurities.

Microbiological analysis

The total microbial counts for all cosmetic products analyzed were < 100 $cfug^{-1}$ and complied with the ASEAN microbial limit test (Table 5). In addition to the microbial limit specified, no product shall have a microbial content recognized as harmful to the consumer. The compliance to the recommended microbial limit in the finished products showed that the products are adequately preserved.

The ASEAN microbial limits recommended for microbial specification (1) are as follows: 1) product around the eyes and mucous membrane not more than 500 $cfug^{-1}$ or $cfumL^{-1}$; 2) baby products less than 3 years not more than 500 $cfug^{-1}$ or $cfumL^{-1}$; and 3) for all other products not more than 1000 $cfug^{-1}$ or $cfumL^{-1}$. Baby products and product around the eyes and mucous membrane require stringent control, therefore the microbial limit is lower (not more than 500 $cfug^{-1}$ or $cfumL^{-1}$). Other recommended tests include the identification of *Pseudomonas aeruginosa*, *Candida albicans* and *Staphylococcus aureus*, however, many manufacturers do not carry out identification on these organisms as their normal quality control procedure.

Table 5: Microbiological analysis of cosmetic products

Cosmetic Product Samples	Total microbial count[a] ($cfug^{-1}$ or $cfumL^{-1}$)
Body scrub	< 100
Body cellulite gel	< 100
Thigh cellulite gel	< 100
Body firming lotion	< 100

Note: [a]Total microbial count included the APC and YMC.

**The ASEAN Microbial Limit Test (MLT) for eye, mucous membrane products and baby products less than 3 years old is 500 $cfug^{-1}$ or $cfumL^{-1}$; the other cosmetic products is 1000 $cfug^{-1}$ or $cfumL^{-1}$.*

The microbiological analysis is an important component in the quality control program of cosmetic and personal care products. Its requirement is similar as in the requirement of halal and legislation that is to ensure the safety aspects of the consumer during its use. Its analysis is indispensible for substances susceptible to contamination. Microorganism will grow wherever conditions are favorable (suitable temperatures, nutrients in abundance, and a moist environment). Many cosmetics, particularly the emulsion-type formulations, provide good media for the growth of bacteria and fungi (17).

Means of inhibiting their growth are therefore essential to prevent deterioration of the product and to ensure safety of the consumer. Therefore, the microbiological analysis in the quality program to meet the halal and legislative requirement is to ensure the product that reaches the consumer is free from numbers and types of microorganisms that could affect product quality and consumer health. During normal product use, the quality of the product will not be affected by microbial activity.

Conclusion

Cosmetic analysis is an important component in conforming to the requirements of halal and quality without violating the regulatory compliance. Physico-chemical test are important in maintaining the stability of products, whereas toxic metals and skin irritation are for safety of consumers. The microbiological analysis is for both stability and safety of consumers. Safe products are the basic requirement of regulatory, halal and quality control parameters in producing cosmetic products. All cosmetic products shall use safe ingredients, manufacture under GMP and undergo adequate safety assessment for the finished products to comply with regulatory requirement, halal and quality control before they could be released to the market.

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