Soap-Making Process Improvement by Using Ingredient X in the Formulation and Laboratory Validation of His Use

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Abstract – *Soaps are one of the major elements which we use in our everyday life and is produced by the saponification of a triglyceride (fat or oil). The ingredient X oil, a non-psychoactive compound that has long been used for its therapeutic benefits, which include soothing skin, treating joint injuries and easing chronic pain, and for those properties the ingredient X is used in the formulation of this soap to validate that can be a useful ingredient. The analysis chart of the tests made to the soap made with a mixture of a few oils, sodium hydroxide, water and Ingredient X demonstrate a few components presents in the soap. A chromatograph evaluated by HPLC showed presence of Ingredient X in it. The validation that the soap contains Ingredient X in it certifies that soaps can be made with Ingredient X and that can be beneficial for the health of the skin.*

Key Terms *- Natural ingredients, Natural Soap, Oils, Soap*

INTRODUCTION

Soaps are one of the major elements which we use in our everyday life. Basically, soaps are the sodium and potassium salts of higher fatty acids, such as oleic, stearic, lauric, and palmitic acids. These fatty acids are responsible for foaming and cleansing actions of soaps. Soaps are generally produced using edible oils, that is, palm oil, coconut oil, soybean oil, groundnut oil. However, usages of nonedible oils have advantages; it can significantly reduce the consumption of edible oils and ultimately the dependence on edible oils for soap manufacturing [1].

Soap can also be said to be any water-soluble salt of fatty acids containing eight or more carbon atoms. Soaps are produced for varieties of purpose ranging from washing, bathing, medication etc. The cleansing action of the soap is due to the negative ions on the hydrocarbon chain attached to the carboxylic group of the fatty acids. The affinity of the hydrocarbon chain to oil and grease, while carboxylic group to water is the main reason soap is being used mostly with water for cleaning purposes [2].

In addition to basic raw materials, other substances are added to the composition in order to improve its application. For examples soap made for medicinal purposes other medicinal importance ingredients are added to it to produce medicated soaps [2].

Other properties of the soap such as hardness are function of the metallic element present in the salt. For example, soap made up of Sodium salts shows little hardness compare to potassium salts soaps, provided the same fat or oil is used in both cases. These are characteristically different from soaps made from divalent metals such as magnesium, calcium, aluminum or iron which are not water soluble, Soaps are used for laundry and cleaning purposes, though the used of calcium soap in the formulation of animal feed have been reported [2].

It is generally known that soap is produced by the saponification of a triglyceride (fat or oil). In the process the triglyceride is reacted with a strong alkali such as; potassium or sodium hydroxide to produce glycerol and fatty acid salts. The salt of the fatty acid is called soap. The equation below represents typical saponification reactions:

$$C_3H_5(OOCR)_3 + 3NaOH \rightarrow 3NaOOCR + C_3H_5(OH)_3 \quad (1)$$

Where $C_3H_5(OOCR)_3$ represents fat, $NaOH$ represents the sodium hydroxide, $NaOOCR$ represents the soap, the $C_3H_5(OH)_3$ represents the glycerol and the R represents the hydrocarbon chain or alkyl group [2].

Fatty Acids are straight-chain monocarboxylic acids. The commonest fatty acid used in soap making contains a range of C10-C20 and most often have an even number of carbon atoms including the carboxyl group carbon. Examples of such saturated fatty acid is palmitic acid (CH3-(CH2)14-CO2H), while unsaturated fatty acids are oleic acid, C17H33COOH. The constituent component of fatty acids is chiefly oleic (C17H33COOH), stearic (C17H35COOH), palmitic (C15H31COOH), lauric (C11H23COOH) and myristic (C13H27COOH) acids; Hydrocarbon oils or paraffin are not suitable for soap-making, as far as chemical combination with the caustic alkalis is concerned. The oils and fats which form soap are those which are a combination of fatty acids and alkali. While glycerin is obtained as a by-product to the soapmaking industry [2].

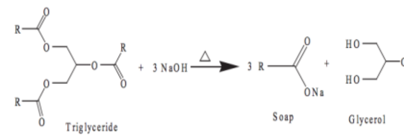
Several chronic inflammatory skin disorders, including atopic dermatitis (or eczema), psoriasis, cutaneous infections, and familial cold autoinflammatory syndrome reduce significantly the patient's quality of life, health outcomes and work productivity. [3]

The traditional systemic treatments present often some limitations, including cumulative toxicity of target organs and potential drug interactions. In recent years, several researchers analyzed the use of Ingredient X in the treatment of these dermatologic disorders, including also melanoma and nonmelanoma skin cancer, melasma, and seborrheic dermatitis, and reported its anti-inflammatory properties and inhibitory effect on rapidly proliferating tumorigenic cell lines. [3]

Currently, Ingredient X oil is a widespread ingredient in skin care products formulated as body oils, moisturizers, lotions and lip balms, but scientific studies on topical safety and efficacy of this extract are lacking. [3]

Concise Chemistry of Soap Making

Chemical reactions in soap making/saponification. Saponification is the main reaction of soap making process. In this reaction (see Figure 1), esters are broken down into alcohols and salts of carboxylic acids as shown in the next reaction:



Soap making (saponification) reaction. Here, R is the long chain hydrocarbon.

Figure 1
Reaction #1

The word "saponification" comes from the Latin word "saponins" which means soap. Saponification is also widely used in more general terms to refer alkaline hydrolysis of any type of esters. The overall reaction of triacylglycerol saponification is carried out in two steps. Three fatty acid molecules and glycerol are produced by the hydrolysis of ester linkages in first step:

$$\text{Fat or oil} + 3H_2O_3 \rightarrow 3\text{fatty acids} + \text{glycerol} \quad (2)$$

The second step involves an acid–base reaction where the fatty acid molecules and a base (usually NaOH) are subjected to produce water and salts:

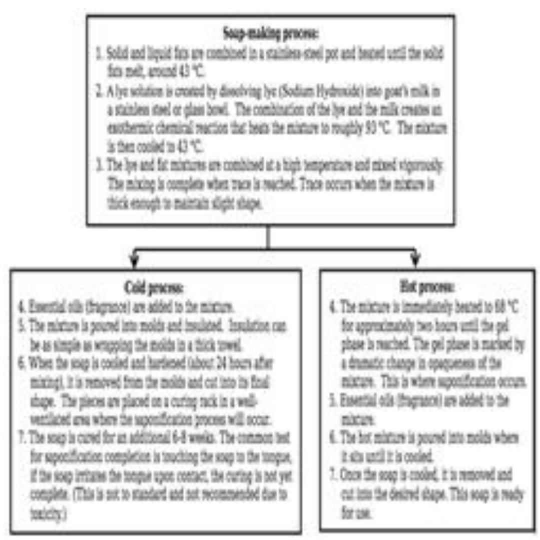


Figure 2
Soap Making Process for Both Hot and Cold Processes [4]

$$3\text{fatty acids} + 3\text{NaOH} \rightarrow 3\text{fatty acid salts} + 3H_2O \quad (3)$$

There are two types of operation involved in soap making (Figure 2). One is continuous operation which is used for industrial production and another is batch operation for small-scale production. Both operations involve three different processes: cold process, semi boiled or hot process, and fully boiled process. In cold process, the reaction takes place substantially at room temperature; in hot process, the reaction takes place at near boiling point; and in the fully boiled process, the reactants are boiled at least once, and the glycerol recovered. Among these processes, simple cold process is common and widely applicable [2]. The Figure 2 explains the soap making process for both hot and cold processes [3].

Cold Process

For each soap formulation, a NaOH solution is added directly with the fat/oil in the ratio 1:1 (v/v) into a stainless-steel container. The fats/oil is warmed gently and pours into the stainless-steel soap making container followed by the alkali solution to form an intimate mix. Then stir frequently for 10–15 min. The additives such as perfumes and other ingredients can be added before pouring the saponification mixture into the molds. The mixture into dies turned into soap, and the soap is allowed around 24 h to harden by air-drying to obtain bar soap. These soap bars can then be observed for color, lathering, texture, and cleaning power. Overall, better products can be obtained by the combination and proper mixing of ingredients, accuracy, and stirring at proper temperature. [1]

Sodium Hydroxide

Sodium hydroxide (NaOH) is manufactured in the chloro-alkali industry, where a range of related chemicals, including sodium hydroxide, chlorine, sodium carbonate, etc., are all produced. Both chlorine and NaOH are generated simultaneously via the electrolysis of sodium chloride (brine) solution. The overall chemical reaction for the electrolysis of the brine solution proceeds in accordance with the following equation:

$$2NaCl_{(aq)} + 2H_2O_{(l)} = Cl_{2(g)} + H_{2(g)} + 2NaOH_{(aq)} \quad (4)$$

The sodium hydroxide solution is evaporated to leave solid NaOH.

Solid sodium hydroxide is commonly encountered as pills, flakes and cast blocks. The solid melts without decomposition at 318°C (614°F) and boils at 1,390°C (2,534 °F) and is not flammable nor does it not support combustion. Solid NaOH is extremely soluble in water (1,260 g/l at 20°C (68 °F)) reacting exothermically to produce strongly alkaline solutions, e.g. pH 14 at 50 g/l at 20°C (68 °F).

Aqueous solutions of sodium hydroxide are both colorless and odorless. Both concentrated and dilute solutions of sodium hydroxide feel slippery on skin contact due the saponification occurring with NaOH and the natural oils present on the skin. NaOH has a lower solubility in polar solvents such as ethanol, methanol, etc., and is insoluble in ether and other non-polar solvents. Strong solutions of sodium hydroxide have a characteristic high viscosity that adds to practical operational difficulties during storage, transfer, etc. [5]

When oil and lye are combined, however, both are consumed in a chemical reaction called saponification. Each molecule of oil may react with as many as three molecules of sodium hydroxide to produce as many as three molecules of soap. This three-to-one ratio means that there is a definite relationship between the weight of oil used in a soap recipe and the amount of sodium hydroxide needed to turn it completely into soap. If the soap maker adds “too much” lye, three molecules of sodium hydroxide react with each molecule of oil until the oil is completely consumed and turned into soap and the excess sodium hydroxide remains in the soap. Unlike the cake example, the soap is not simply a little more alkaline - it is caustic and potentially dangerous. We cannot dole out lye and oil one molecule at a time, but because each molecule has a specific weight, we can determine the weight of sodium or potassium hydroxide required to exactly saponify a given weight of oil. This is generally expressed as the number of milligrams of potassium hydroxide required to completely saponify one gram of oil. Because different oils contain different oil molecules, the saponification value (SAP or SV) for palm oil differs from that for coconut oil or olive oil. Worse than that, it may differ from one sample of palm oil to another; the values tabulated in soapmaking books are simply averages over many samples of each kind of oil [6].

Oils in Soap Making

Fats and oils are esters of different fatty acids and glycerol. Fats and oils are divided into three classes, fixed oils, mineral oils and essential oils. Fixed oils form the main raw materials for soap making as they decompose into fatty acids and glycerol when strongly heated and can be easily saponified by alkali. Fixed oils, which include both animal and vegetable fats and oils, are further classified according to its physical properties as follows:

- Nut oils: These oils are characterized to be having large proportion of fatty acids with low molecular weight, especially lauric and stearic acid. Examples of these oils are coconut oil. These oils, when used in toilet soaps are the chief foam-producing ingredients. They usually saponify easily with strong alkali solution. Once these oils have begun to saponify, the process proceeds rapidly with the evolution of heat. They require very large quantities of strong brine to grain their soaps, and the grained soaps tend to carry more salt than other soaps. These oils are more suitable for the making of cold process soaps.
- Hard fats: The hard fats contain appreciable quantities of palmitic and stearic acids. Examples of these oils are palm oil, animal tallow and hydrogenated fats. These oils produce slow-lathering soaps, but the lather produced is more resistant over long periods of time than the nut oils. In soap making, they are first saponified with weak alkali, and in the final stages with stronger alkali solutions.
- Soft Oils: These oils have substantial amounts of unsaturated acids, namely oleic, linoleic and linolenic acids. The soap making properties of these oils vary with their fatty acid composition, and their physical and chemical properties of the acids. Examples of these kind oils are groundnut, cotton seed, fish oil and olive oil. These oils cannot produce a very hard soap when used alone for soap making. They are generally blended with nut oils. Their soaps lather freely and have very good detergent properties. [7]

Fragrances and Essential Oils

An essential oil is a mixture of secondary metabolites from plants that are responsible for its characteristic odor. It’s a combination of different aromatic and aliphatic compounds, frequently terpenes and terpenoids, which composition depends on the plant species, variety, geographic origin, soil and season. Current cosmetic products use synthetic fragrances while high quality cosmetics’ manufacturers choose essential oils. However, due to their high volatility, organic fixative molecules such as sucrose, sucrose derivatives, sodium carboxymethyl chitosan, among others are needed. Following this principle, the use of an essential oil is increased with the use of a further additive which is not essential for soap cleaning function [8].

Ingredient X

The market is always ready to jump at a new oil trend. Enter ingredient X oil, a non-psychoactive compound that has long been used for its therapeutic benefits, which include soothing skin, treating joint injuries and easing chronic pain. Studies have confirmed what has been thought and realized by traditional cultures for thousands of years. A 2014 scientific report found that because of its anti-inflammatory and sebum-reducing properties, ingredient X and can have a positive effect on acne-prone skin. Additionally, results from multiple trials have also concluded that ingredient X strongly regulates the proliferation, migration and neurogenesis of Mesenchymal Stem Cells (MCSs). These adult stem cells can differentiate into the main types of precursor cells and have demonstrated anti-inflammatory, immune, metabolic and self-renewal properties. Dermatologists agree that ingredient X anti-aging and anti-inflammatory benefits are clinically proven. Ingredient X oil is reputed to be the most unsaturated oil derived from the plant kingdom, so it is less pore clogging and a great moisturizer for dry, cracked skin.

This is the new frontier in skincare, and the companies and the practitioners using their products are paving the way. The industry has spoken: Ingredient X is the new super oil!

In the beauty industry's experiments with ingredient X, several early adopters have confirmed the oil's incredible moisturizing and skin soothing properties, due to its high levels of essential fatty acids.

Since the days of Cleopatra, who used castor oil from the castor seed to nourish her skin, people have been obsessed with the notion that moisturizing oils are essential for beauty care and continue to be a beauty market craze, appearing in everything from your shampoo to your facial cleanser. Now medical practitioners are even using it in your therapeutic massages and medical facial treatments [9].

Method of the Soap Making

The general soap production method is divided into the following major steps.

1. The oils used for the soap have to be weighted depending on the percent of each oil in the mixture and the quantity of water and sodium hydroxide as well.
2. When weighing the oils and ingredients for the soap you start mixing the oil/fat including the Ingredient X. Some of them need to melt in order to be used because they have to be in his liquid state.
3. In a separate bowl the water and the sodium hydroxide are mixed because of the exothermic reaction the water and the sodium hydroxide make. And when the temperature drops can be added to the mixed oils.
4. Using an immersion blender, the oils and the mix of the water and sodium hydroxide are blended for a few minutes making sure all ingredients are dissolved in the mixture. Until the mixture achieves a thick consistence.
5. If adding colorants and perfumes they can be added when all of the oils are blended.
6. When having all ingredients mixed the solution can be putted in the mold to be used for the shape of the soap.
7. After putting the soap mixture in the molds, they need to be for at least 1 day there in order to dry and having the soap ready to use.

Results and Discussion

The results are presented in two laboratory evaluations. For the evaluation that the soap is suitable for use a laboratory results are presented. This result presents every component presented in the soap that can be either harmful or not harmful for the skin. The second laboratory evaluation presents the identification of Ingredient X by HPLC method.

Oils Mixture Laboratory Results

The Figures 3 and 4 show the analysis chart of the tests made to the soap made with a mixture of a few oils, sodium hydroxide, water and Ingredient X.

The charts (Figures 3 and 4) show the units of each parameter as well as the result obtained by different methods with the method detection limit (MDL). The intent of the method detection limit is to define the smallest concentration of analyte that can be detected with no guarantee about the bias or imprecision of the result. [10] This test ensures that the physical and chemical properties of the oil ingredients in the soap are thoroughly examined. Those results ensure the quality of the soap.

The aerobic plate count (Figure 3) determines poor sanitation or problems with process control or ingredients. The aerobic plate count in the results was less than 1 meaning the soap can be an antibacterial soap as it inhibits microorganism or bacterial growth.

REPORT OF ANALYSIS

Certificate Number: CERT - 41467

May 12, 2021

Customer Name:	Feria Cientifica	**Custody Number:**	109103
Contact:	KATHIA RIVERA DEL CUETO	**Sampled Date:**	Wednesday, April 21, 2021
Customer Address:	Puerto Rico	**Sampled Time:**	0900 hrs.
	Puerto Rico Puerto Rico 00731	**Received Date:**	Wednesday, April 21, 2021
Phone/Fax:	787-835-4242	**Received Time:**	1000 hrs.
Contact Email:	kathia.riveradc@gmail.com	**Sample Matrix:**	Solid
Sampled By:	kathia Rivera	**Sample Type:**	Grab
Sample Received By:	S. Aponte	**Temp Rec at Lab:**	Room Temperature
Sample Delivered By:	Rafael Ballester	**Lab. Sample Number:**	AT-21-2126
Project and Sample Description:	Feria Científica - Trabajo de Investigación Jabón Artesanal		

Parameter	Units	Result	Method	Method Detection Limit	Analysis Date	Analysis Time	Analyst
Aerobic Plate Count	CFU/10g	<1	AOAC 990.12	---	April 21, 2021	1540	JV
Ammonia	mg/kg	1,148	SM 4500 NH3-C	80	April 23, 2021	1530	JS
Cadmium	mg/kg	ND	EPA 200.7	0.17	May 4, 2021	1139	CV
Calcium	mg/kg	27.6	SM 3111B	440	May 4, 2021	1514	CR
Chloride	mg/kg	335	EPA 300.0	160	April 24, 2021	0218	FO
Copper	mg/kg	2.95	EPA 200.7	0.23	May 4, 2021	1139	CV
Iron	mg/kg	4.22	EPA 200.7	0.27	May 4, 2021	1139	CV
Lead	mg/kg	ND	EPA 200.7	0.24	May 4, 2021	1139	CV
Magnesium	mg/kg	5.53	SM 3111B	0.50	May 4, 2021	1355	CR
Mold	CFU/10g	<1	AOAC 997.02	---	April 21, 2021	1540	JV
Nitrate Plus Nitrite	mg/kg	<12.3	Calculated	12	May 6, 2021	1330	FO
pH	S.U.	10.53	AOAC 981.12	---	April 21, 2021	1540	JV

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Test results in this report meet ISO 17025 requirements

For ISO 17025 Scopes of Accreditation refer to www.altolenterprises.com

Figure 3

Report of Analysis

REPORT OF ANALYSIS

Certificate Number: CERT - 41467

May 12, 2021

Parameter	Units	Result	Method	Method Detection Limit	Analysis Date	Analysis Time	Analyst
Potassium	mg/kg	9.17	SM 3111B	0.47	May 4, 2021	1038	CR
Selenium	mg/kg	<0.40	EPA 200.7	0.40	May 4, 2021	1139	CV
Silica (as SiO2)	mg/kg	32.3	EPA 200.7	26	April 28, 2021	1305	CV
Silver	mg/kg	ND	EPA 200.7	0.100	May 4, 2021	1139	CV
Sodium	mg/kg	47,452	SM 3111B	2000	May 4, 2021	1205	CR
Sulfate	mg/kg	804	EPA 300.0	130	April 24, 2021	0218	FO
Surfactants As MBAS	mg/kg	136	SM 5540C	15	April 23, 2021	0817	AR
Total Phosphorous	mg/kg	5.23	EPA 200.7	1.3	May 3, 2021	1405	CV
Yeast	CFU/10g	<1	AOAC 997.02	---	April 21, 2021	1540	JV
Zinc	mg/kg	1.38	EPA 200.7	0.37	May 4, 2021	1139	CV

ND = Not Detected

Saira Vázquez Báez

Laboratory Operations Director

Licensed Chemist #5471

A 1844132

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Test results in this report meet ISO 17025 requirements

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Figure 4

Continuation of Report of Analysis

The ammonia shows a result of 1,148mg/kg and compared to the detection limit is high (Figure 3). The ammonia is considered safe to the skin but can irritate it. The sensitive and dry skin should use ammonia free soap.

Cadmium and Lead are considered heavy metals and potential carcinogenicity of human exposure. [11] The laboratory results show that there wasn't detected any of them in the soap (Figure 3).

Although the amount of Calcium analyzed compared to the method detection limit is less (Figure 3) still works as a moisturizer. Calcium works with the epidermis to produce sebum, a natural skin-coating substance that helps skin retain moisture. [12]

Copper has two key properties that endow it as an excellent active ingredient to be used in products, which come in contact with the skin, aiming to improve the skin's well-being. Copper plays a key role in the synthesis and stabilization of skin proteins, and it also has potent biocidal properties. [13] The soap showed a high content of copper compared to the method detection limit having a 2.95mg/kg content (Figure 3). This show that the mix used formed high content of copper which is very beneficial for the skin.

The next parameter found in the soap was Iron that is in a high content with 4.22 mg/kg compared to the method detection limit which was 0.27 mg/kg (Figure 3). This content of Iron is not harmful for the skin because Iron is essential for healthy skin, mucous membranes, hair and nails [14].

Magnesium was as well a content of the soap in a concentration of 5.53mg/kg being higher than the LDL which was 0.50 mg/kg (Figure 3). But the magnesium in recent studies have claimed that transdermal magnesium is absorbed through sweat glands. Of relevance, topical application of a cream containing 2% magnesium reduced diaper dermatitis and diaper rash in children [15].

The pH of the soap resulted to be 10.53 (Figure 3) being alkaline and a good level for the use in the skin. Normal healthy skin has potential of hydrogen (pH) range of 5.4-5.9 and a normal bacterial flora. Use of soap with high pH causes an increase in skin pH, which in turn causes an increase in dehydrative effect, irritability and alteration in bacterial flora [16].

The tests result of a low concentration of Selenium and Yeast that is less than 1mg/kg (Figure 4). These results are so low that neither affect of benefit the skin so his content doesn't harm.

Results show a 32.3 mg/kg content of Silica and compared to the MDL its difference is not too high (Figure 4). The content of Silica is beneficial on the skin because it is suggested that silicon is important for optimal synthesis of collagen and for activating the Hydroxylation enzymes, improving skin strength and elasticity [17].

As for this mixture of soap was used sodium hydroxide in the final reaction there is sodium formed. In the laboratory results the amount of sodium found was 47,452 mg/kg and compared to the MDL is two times higher (Figure 4). The sulfate content as the laboratory show is 807 mg/kg being very high compared to the MLD. This concentration of sulfate can be harmful to the skin and for persons that are allergic to it. The biggest issue with sulfates is that they can cause varying levels of skin and eye irritation, which gets worse the longer the product is in contact with the skin. People with sensitive skin should avoid sulfate, but as the sulfate cause the foamy effect in soap the sulfate free products wont probably foam up, so they'll produce a mora hydrating, creamy wash. [18]

Surfactants are perhaps the most important of all cosmetic ingredients. In cosmetics, surfactants are used for cleansing, foaming, thickening, emulsifying, solubilizing, penetration enhancement, antimicrobial effect, and other special effects. One of the most common applications of surfactants in cosmetics is for cleansing formulations. [19] The soap made showed a concentration of 136 mg/kg of Surfactant as MBAS (Methylene blue active substances) very high compared to the MDL that was 15 mg/kg (Figure 4). This concentration of surfactants allows the soap to be a good cleansing effect.

HPLC identification of Ingredient X

The Figures 5, 6 and 7 show the chromatograph evaluated by HPLC for three samples of the soap to identify the Ingredient content in it The chromatographs were obtained using HPLC and they identify the Ingredient X presence in the soap. They identified a series of peaks, each one representing a derivative compound of Ingredient X passing through the detector and absorbing UV light. Each test was evaluated by tree dilutions the Figure 5 presents a dilution of 0.562g of the soap, the Figure 6 presents a 0.271g and the Figure 7 a 0.108g using a volume of 10mL and dilution factor of 250/500. Even though each dilution decreases in concentration in each chromatograph the presence of Ingredient X is present as Ingredient X (E).

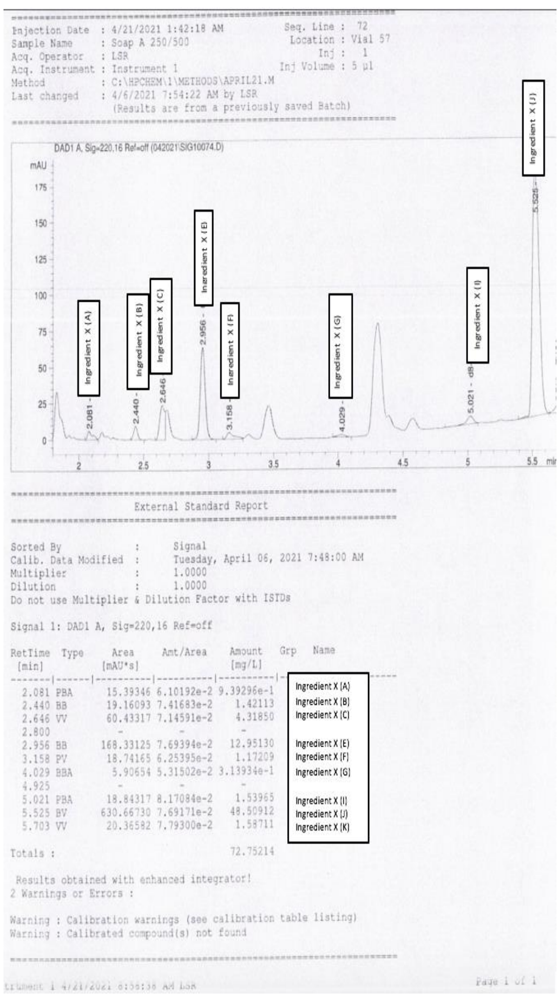

Figure 5
Chromatogram from Sample A

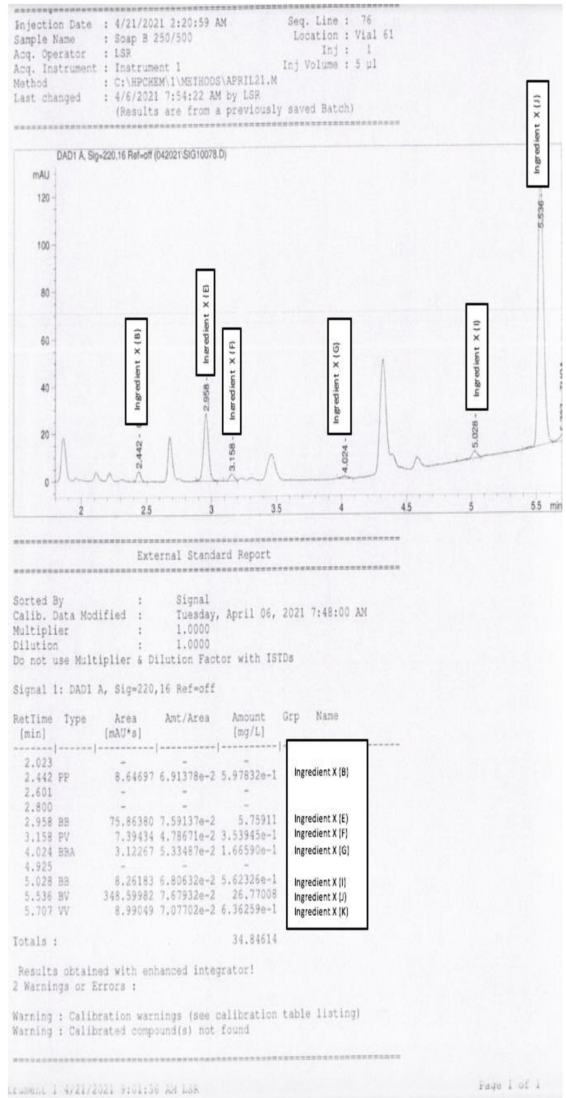

Figure 6
Chromatogram of Sample B

Ingredient X has a lot of derivatives and in this soap are a few of them present as showed in each chromatograph but as each compound have different migration rates, they are present in different retention time across the chromatograph. In previous studies the retention time of Ingredient X fluctuates between 2.2 [20] to 3.5 [21] and this is because of the dilution used and method in the solution. The concentration that is given by the chromatogram is 12.95 mg/L (Figure 5) and is the usual concentration that only variation could be depending on the units used because another concentration can be 1.35μg/mL. [20]

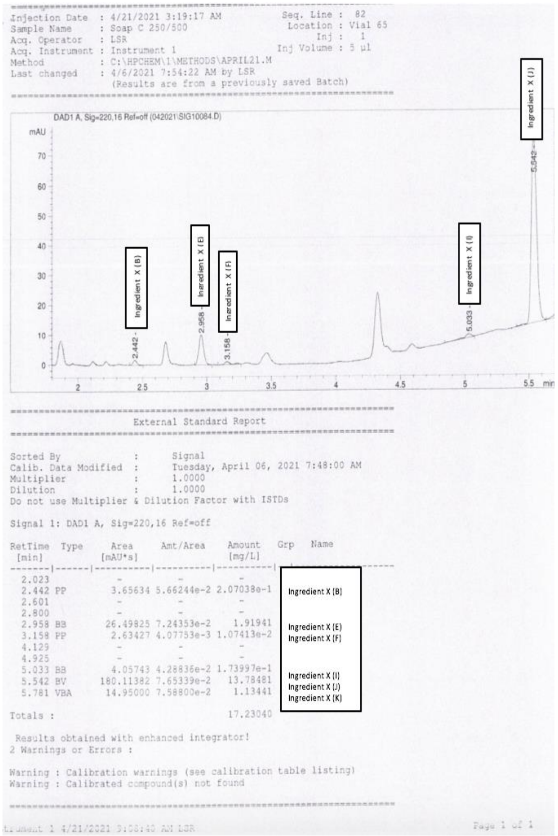

Figure 7
Chromatogram of Sample C

Conclusions

In our daily basis we use soaps so it's important to know the ingredients that are in it. Several persons suffer of allergies and some of them are to chemicals or ingredients. Natural soaps are made with natural ingredients but can cause allergy to specified persons. When buying a natural soap usually we don't know the content of it and for that the laboratory evaluation provided the specific content of ingredients in it.

The Ingredient X is an innovative oil that is very beneficial for the skin but there are few studies about it. The validation that the soap contains Ingredient X in it certifies that soaps can be made with Ingredient X and that can be beneficial for the health of the skin. There could be more products that can be made with this Ingredient X with the proper studies and evaluation and can be very helpful for some conditions in the skin. And not only the Ingredient X but with some research the derivatives of Ingredient X can be used as well for the production of many others products that can be very useful for the skin.

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