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Development of Natural Castile Soaps from Vegetable Oils

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ABSTRACT

Objective: Castile soaps are believed to be natural, 100% biodegradable, non-toxic surfactants and in some cases, they have antimicrobial properties. As raw materials, they are used extensively in the cosmetic industry for personal and home care products. Natural castile soap bases are generally produced in super fat stoichiometry to protect skin from caustic damage. The super fat method optimizes the total amount of caustic required for the saponification of natural vegetable oils and keeps these oils at the maximum level in the final product. The aim of this study was to develop natural soap based raw materials and castile soaps from various vegetable oils such as olive oil, coconut oil, castor oil, almond oil and jojoba oil.

Materials and Methods: Castile soaps were produced using the super fat method by measuring the saponification values of the vegetable oils. Viscosity, pH, appearance, and microbiological tests were performed to determine their shelf life.

Results: The castile formulations did not contain any preservatives, no microbiological risk was observed during their shelf life. All soap based raw materials and castil formulations have a two-year shelf life. These castile soaps are 100% soluble in water.

Conclusion: This article documents the laboratory-scale production of six soap based raw materials and the development of five castile formulations for personal and household cleaning. Super fat method optimized the total amount of caustic required for the saponification of natural vegetable oils such as olive oil, coconut oil, castor oil and maintains the oils in the product at the maximum level without allowing the presence of free caustic in the final product. Super fat castile soaps are the safest personal and household cleaning products because they don't contain caustic residue, have their long shelf life and feature high solubility in water minimizing the risk of residue on skin and surface.

Keywords: Castile soap, super fat, vegetable oil, cosmetic products, preservative

INTRODUCTION

Traditional soap bases are obtained by reacting natural vegetable oils or fatty acids with caustic (sodium hydroxide or potassium hydroxide) in an aqueous environment. Although they are alkaline in aqueous solutions, they are the least damaging surfactants to the skin compared with classic petrochemical surfactants. Moreover, they do not change the pH of the skin (1-3).

The most important factor affecting soap formulation is high pH value. The basic pH value (pH: 10-11) has an antimicrobial effect in the final formulations, providing protection against harmful pathogens and viruses. This eliminates the need to add preservatives to the formulations (1).

Potassium salts of natural soap bases are widely used in personal care products, such as hand soap, shampoo and toothpaste as well as in household cleaning products, especially laundry and dishwashing soaps and surface cleaners (4, 5).

Castile soap is a traditional natural soap that can only be obtained by soaping vegetable oils, predominantly olive oil. The natural glycerine formed in the saponification reaction remains in the soap. Apart from olive oil, the most commonly used vegetable oils are coconut oil, palm kernel oil, hemp seed oil, castor oil and jojoba oil. Castile soap

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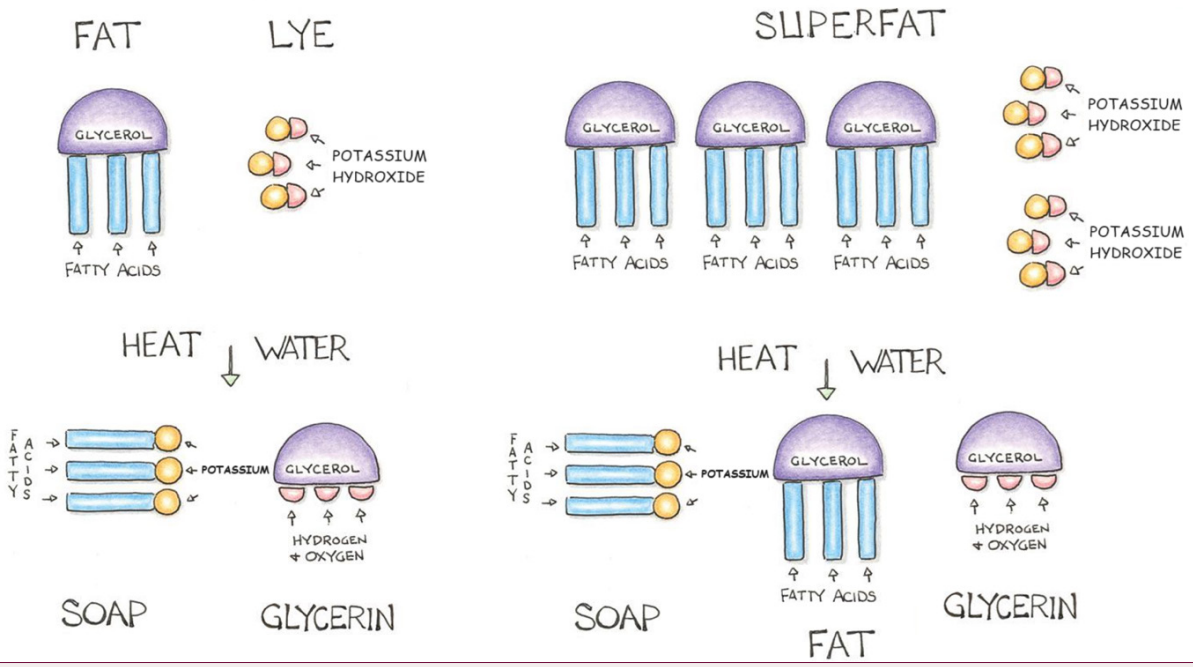

Figure 1. Super fat saponification (https://infinitalab.com/product-testing-service/laboratory-tests-to-determine-ingredients-in-soap/).

does not contain animal fats or synthetic ingredients. It is a concentrated soap and does not contain thickeners. It is 100% biodegradable (environmentally friendly) and can be used for hand and body cleaning; house, baby and pet washing. The natural pH of castile soap is alkaline (pH: 10), thus it provides an environment in which bacteria and viruses cannot live (4- 6).

The super fat method optimizes the total amount of caustic required for the saponification of natural vegetable oils and maintains the oils in the product at the maximum level without allowing the presence of free caustic in the final product (Figure 1). There are several reasons why soaps may become slightly oilier. Human skin contains a certain amount of sebum (oil), which varies depending on the region. Soaps are made to be oilier to maintain the necessary sebum balance after washing hands with natural liquid soap. Super fat liquid soap maintains the sebum balance after washing hands, while the film formed by the sebum (oil) layer formed in the hand area makes it difficult for water to evaporate from the skin, allowing the skin to remain moist for a long time (7, 8).

Coconut oil and coconut fatty acid are frequently used in soaps. The benefit of coconut oil is that it contains high levels of mono-unsaturated fatty acids, such as palmitic, capric, caprylic, oleic, stearic, and linoleic acids. The lauric acid content of approximately 44-52% ensures that it is oxidation resistant. The main reason for using coconut oil in soap is that it is an extraordinary cleaner with a very high foam quality. Coconut oil is one of the best cleaning substances to be used in soaps. In addition, it has antibacterial and anti-inflammatory properties. It works well with skins that have reddish infections, such as eczema-prone dry skins and acne-prone oily skins. Coconut oil is also a strong antioxidant. It is a natural moisturizer and has great benefits for the skin (9-14).

Olive oil has been used since Ancient Egypt and Greece and is primarily rich in oleic acid and other monounsaturated fatty acids. In addition to fatty acids, it contains strong antioxidant substances such as polyphenols, squalene and flavonoids. The phenolic substance complex combined with vitamin E synergistically strengthens its preservative effect. Olive oil also has a high antioxidant capacity. Soaps manufactured using olive oil clean skin without irritation. Their rich components provide day-long care for the skin and ensure it stays moisturized throughout the day. Natural soaps based on olive oil help the skin to renew its natural oils to ensure lasting protection, and they do not contain any of the hard chemicals and synthetic odors found in many soaps (15-18).

Castor oil obtained from the seeds of the *Ricinus communis* plant, castor oil is a multipurpose herbal product used by people for thousands of years. Castor oil has several medical, industrial, and pharmaceutical applications. It is generally used as an additive in foods, medications, and skin care products. Castor oil is rich in ricinoleic acid, a mono-unsaturated fatty acid. These types of oils play the role of moisturizers and are used to hydrate the skin. Humectants prevent the loss of water from the external layer of the skin, thus maintaining moisture inside. Castor oil is commonly used in cosmetics to support hydration and is generally added to cosmetic products, such as lotions, make up products and cleaners. Castor oil increases foam in soap and gives it a creamy texture, resulting in an

Table 1. Starting materials for castile soap bases

Starting Material	Trade Name/Supplier	Saponification Number (Calculated)
Coconut Fatty Acid	Distilled Coconut Fatty Acid DNF 300/Evyap	263
Olive Fatty Acid	Greenfac 7986/GreenOleo	196
Pure Olive Oil	Verde N6362/4K Kimya	189
Castor Oil	Pressed Castor Oil/Interfat	167
Gum Rosin	Gum Rosin WW Grade/Hatkim	180
Sweet Almond Oil	Almond oil/Interfat	196
Nettle Seed Oil	Nettle Seed Oil/Mecitefendi	186
Avocado Oil	Organic Avocado Oil/Interfat	186
Jojoba Oil	Jojoba Organic Golden Oil/Jojoba Desert	97

abundant foamy sensation. A ratio of approximately 10% castor oil in soaps has been shown to have beneficial effects (19-21).

Gum rosin also known as colophony or Greek pitch (Latin: pix græca), Gum rosin is a solid resin produced by heating fresh liquid resins to vaporize volatile liquid terpene compounds. Gum rosin is obtained from pine trees and other coniferous plants. It is semi-translucent and its color changes from yellow to black. The term 'Colophony' comes from colophonia resina, the name of an ancient Ionian city. It has a mild pine scent. Gum resin is made from residues obtained by distillation of oleoresin from pine trees. Rosin and its chemical derivatives are usually used to make pitch for soaps, lacquer, seal wax, printing ink, dryers, paper dyes, glues, binding agents, solder flux, shiny grease for dyes, and barrels. Furthermore, rosin can be turned into triethylamine acetate to kill algae, bacteria, mold, mollusca and other pests (22-26).

The present study aimed to develop natural soap based raw materials and castile soaps from various vegetable oils such as olive oil, coconut oil, castor oil, almond oil and jojoba oil.

Materials and Methods

Castile soaps were produced using the super fat method by measuring the saponification values of the vegetable oils. Viscosity, pH, appearance, and microbiological tests were performed to determine their shelf life.

pH measurement: The pH electrode was immersed in the solution (2% soap-water dilution) to be measured.

Equipment: Mettler Toledo Seven Excellence; pH Electrode: InLab Expert Pro-ISM

Viscosity measurement: A 100 g sample was poured into a 150ml beaker and a suitable viscometer spindle was dipped into the beaker. Readings were taken at the selected rotation speed (rpm) to measure the unchanging viscosity value for 5 minutes.

Equipment: Brookfield DV3T viscometer; Spindles: RV03, T-Bar C93, T-Bar D94

Calculation of Saponification Number:

Five g oil sample for analysis was weighed and placed into a 250 ml glass conical flask (the exact amount is noted), and 50 ml (approximately 42.43 g) of 0.5 N KOH-EtOH solution was added. The solution was boiled under reflux for 60-90 minutes. After boiling, the solution was allowed to cool to room temperature. 2-3 drops of 1% phenolphthalein solution were added to the solution and titrated with 0.5 N HCl solution (the colour changes from pink to light yellow). The same process was repeated using a blank test.

Preparation of 0.5 N KOH-EtOH: 33 g potassium hydroxide was dissolved in 100ml of pure water. Approximately 900mL of ethyl alcohol is added and the volume is completed in a 1 L volumetric flask.

Saponification value = 28.05 x (a-b)/c

a: 0.5 N HCl mL in blank test (50mL KOH-EtOH)

b: 0.5 N HCl mL during saponification

c: amount of oil (grams)

Synthesis of Soap Bases

All syntheses were carried out by heating natural vegetable oils (triglycerides) or fatty acids with a caustic in an aqueous solution. When oil is used, glycerin is formed as a by-product of the saponification reaction. When fatty acids are used, only a soap base is formed (Table 1).

General Synthesis Procedure

Addition of caustic solution: 500 g of fatty acid or vegetable oil was taken into the reaction vessel. 30% potassium hydroxide

Table 2. Reaction conditions

Soap Base	Weight of the Starting Material	Superfat Ratio	Reaction Time (hour)	Reaction Temperature	pH	Total Weight of Soap Base
Potassium Cocoate	500g Coconut Fatty Acid	1.0	1.5	80°C	11.2	1600 g
Potassium Olivate	500g Olive Fatty Acid	0.88	1.5	80°C	10.7	800 g
Potassium Olivate	500g Pure Olive Oil	0.88	5	90°C	10.7	800 g
Potassium Castorate	500g Castor Oil	0.92	5	80°C	9.2	750 g
Potassium Rosinate	500g Gum Rosin	1.0	4	90°C	10.4	1500 g
Natural Soap Base for Hair Products	A mixture of 430g Sweet Almond Oil, 40g Nettle seed Oil, 15g Avocado oil and 15g Jojoba Oil	0.90	5	90°C	10.7	750 g

solution was added according to the saponification number and the super fat ratio.

Reaction Monitoring

Heat was applied for one and half hours, and the solution was slowly stirred at approximately 80°C. The pH of the solid soap base in aqueous solution (2%) was measured to determine whether the reaction was complete (Table 2).

Dilution of the Soap Base

The hot solid soap base is then dissolved in hot water and allowed to cool to room temperature. The total weight of the soap is adjusted by adding cool water.

Caustic Formulation

Weight of potassium hydroxide solution = (Weight of Starting Material) x (Saponification Number) x (Super fat ratio) / (% concentration of potassium hydroxide x 10)

Castile Soap Based Formulations

Various amounts of the soap base (potassium cocoate, potassium olivate, etc.) mixtures were created depending on the intended use of the formulation. The potassium cocoate content is kept high when the intention is to increase its cleaning properties. The rate of potassium olivate, which has a high moisturizing function, is high in viscous products that have a lot of contact with the skin. Natural essential oils (lavender, tea tree lemongrass, orange and eucalyptus) were used in the formulations to increase the antimicrobial effect and for odour-related reasons (Table 3). In addition, various inorganic salts (sodium bicarbonate, sodium carbonate, and sodium citrate), white vinegar and citric acid were used to increase the cleaning properties of the formulations and to adjust their viscosity.

Hand Soap

Potassium olivate, potassium cocoate and potassium castorate were used in this gel formulation. A certain amount of orange oil or lavender oil was added to enhance the antibacterial effect. In addition, white vinegar and baking soda were used to enhance the cleaning properties (Table 4).

Laundry Soap

Only potassium cocoate was used in this liquid formulation. Sodium carbonate and sodium citrate were added to reduce

Table 3. Natural essential oils

Natural Essential oils	INCI/Supplier	Use Level (Product)
Lavender Oil	Lavandula Angustifolia Herb Oil/Sensient	0.2% (Laundry Soap)
Orange Oil	Citrus Sinensis Peel Oil/Ventos	0.4% (Hand Soap)
Lemongrass Oil	Cymbopogon Flexuosus Oil/Sensient	0.25% (Dishwashing Soap)
Tea Tree Oil	Melaleuca Alternifolia Leaf Oil/Sensient	0.05% (Hair Soap)
Eucalyptus Oil	Eucalyptus Globulus Leaf Oil/Nuka	0.05% (Surface Cleaner)

Table 4. Hand soap formulation	
Ingredients	**% (w/w)**
Potassium Olivate	23.00
Potassium Cocoate	7.00
Potassium Castorate	1.00
Fragrance (Natural Essential Oils)	0.1-0.50
Sodium Bicarbonate	0.25-0.50
Citric Acid or White Vinegar	0.1-0.50
Pure Water	67.5-68.5

the effect of lime in water. In addition, essential oils such as lavender oil or orange oil were used to enhance the antibacterial effect (Table 5).

Surface Cleaner

Potassium rosinate, potassium cocoate, potassium olivate and potassium castorate were used in this liquid formulation. The amount of potassium rosinate was optimized to effectively clean the surfaces and form an anti-pollution layer. In addition, eucalyptus oil was added to enhance the antibacterial effect (Table 6).

Table 5. Laundry soap formulation	
Ingredients	**% (w/w)**
Potassium Cocoate	80.00
Fragrance (Natural Essential Oils)	0.1-0.5
Sodium Carbonate	0.01-0.10
Sodium Citrate	0.01-0.10
Pure Water	19.30-19.88

Table 6. Surface cleaner formulation	
Ingredients	**% (w/w)**
Potassium Olivate	2.50
Potassium Cocoate	4.00
Potassium Castorate	0.25
Potassium Rosinate	0.25
Fragrance (Natural Essential Oils)	0.05-0.10
Sodium Citrate	0.01-0.05
Pure Water	92.85-92.94

Dishwashing Soap

Potassium cocoate, potassium olivate and potassium castorate were used in this gel formulation. The proportion of potassium cocoate is kept high to maintain the high cleaning properties of dishwashing soap. In addition, sodium carbonate, sodium bicarbonate, white vinegar and lemongrass oil were added to increase the viscosity, rheology and cleaning properties of the product (Table 7).

Hair Soap

A natural soap base consisting of sweet almond oil, nettle seed oil, avocado oil and jojoba oil, potassium cocoate, potassium olivate and potassium castorate was used to provide care for hair. Tea tree oil was used to prevent dandruff (Table 8).

Table 7. Dishwashing soap formulation	
Ingredients	**% (w/w)**
Potassium Olivate	18.50
Potassium Cocoate	25.00
Potassium Castorate	1.00
Fragrance (Natural Essential Oils)	0.1-0.50
Citric acid or White Vinegar	0.1-0.50
Sodium Carbonate	0.75
Sodium Bicarbonate	1.50
Pure Water	52.25-53.05

RESULTS

Table 8. Hair soap formulation	
Ingredients	**% (w/w)**
Potassium Olivate	20.0-25.0
Potassium Cocoate	5.0-10.0
Potassium Castorate	1.0-2.0
Mixture of Sweet Almond Oil, Nettle Seed Oil, Avocado Seed Oil and Jojoba Seed Oil Potassium Soap	2.0-4.0
Fragrance (Natural essential oils)	0.05-0.50
Sodium Bicarbonate	0.25-0.50
Citric Acid or White Vinegar	0.10-0.50
Pure Water	57.5-71.5

Table 9. Physical properties of soap bases

Soap Base	Density at 20°C	Total solid (calculated)	Water solubility at 20°C (w/w, soap/water)
Potassium Cocoate	1.0 ± 0.1 g/cm³	33%	1.28
Potassium Olivate (from Fatty Acid)	0.9 ± 0.1 g/cm³	72%	0.31
Potassium Olivate (from Oil)	0.9 ± 0.1 g/cm³	70%	0.33
Potassium Castorate	0.9 ± 0.1 g/cm³	70%	0.5
Potassium Rosinate	1.0 ± 0.1 g/ cm³	40%	1.0
Natural Soap Base for Hair Products	0.9 ± 0.1 g/cm³	70%	0.33

Table 10. Stability results of soap bases

	Stability (25°C, 60% humidity) 2 years			Stability (40°C, 75% humidity) 6 months		
Soap Base	**pH**	**Viscosity**	**Appearance**	**pH**	**Viscosity**	**Appearance**
Potassium Cocoate	11.2-11.0	50-55 cP	light yellow liquid	11.2-11.0	50-55 cP	yellow liquid
Potassium Olivate (from fatty acid)	10.7-10.5	50000-55000 cP	yellow-orange semi-solid	10.7-10.4	50000-60000 cP	orange semi-solid
Potassium Olivate (from oil)	10.7-10.5	50000-53000 cP	yellow-orange semi- solid	10.7-10.4	50000-55000 cP	orange semi-solid
Potassium Castorate	9.2-9.3	150000-170000 cP	orange-yellow semi-solid	9.2-9.3	150000 -180000 cP	dark orange semi-solid
Potassium Rosinate	10.4-10.5	50-65 cP	dark orange-red liquid	10.4-10.5	50-80 cP	dark red liquid
Natural Soap Base for Hair Products	10.7-10.4	75000-80000 cP	orange semi-solid	10.7-10.4	75000 -85000 cP	dark-orange semi-solid

Six natural soap based raw materials were created in liquid or semi-solid physical form.

When fatty acid forms of vegetable oils are used in saponification reactions, the reactions take one and half hours to complete (using coconut fatty acid or olive fatty acid). For vegetable oils, the reaction time was approximately five hours.

The physical properties of the soap based raw materials are summarized in Table 9. Total solid content is calculated using the following equation. The water solubility of the natural soap based raw materials was observed to be higher than 0.3.

Total solid percentage = (weight of the starting materials) / (total weight of the soap base) x 100

The stability results of the soap bases are summarized in Table 10. Only a slight change was observed in the color and viscosity of the soap bases during the two-year period.

Viscosity Measurement: Potassium cocoate (RV03 Spindle, 35 rpm), potassium olivate (T-Bar C93 Spindle, 5 rpm), potassium castorate (T-Bar D94 Spindle, 5 rpm), potassium rosinate (RV03 Spindle, 35 rpm), natural soap base for hair products (T-Bar C93 Spindle, 5 rpm).

Five liquid or gel soap-based formulations were developed using these soap based raw materials.

Pure water (deionized water: <5 µS/cm) must be used in all reactions and formulations to prevent inconsistency in the appearance of the formulations.

One hour of mixing (1000rpm, IKA RW20) is sufficient for the formulations. Transparent appearances were obtained by allowing them to rest for approximately one hour.

The shelf life of natural soap bases and castile soaps is approximately 2 years under room conditions. Viscosity, pH, appearance and microbiological tests were performed to

Table 11. Stability results of formulations

Formulation	Stability (25°C, 60% humidity) 2 years			Stability (40°C, 75% humidity) 6 months		
	pH	Viscosity	Appearance	pH	Viscosity	Appearance
Hand Soap	10.2-10.3	700-800 cP	yellow gel	10.2-10.3	700-850 cP	yellow-orange gel
Laundry Soap	11.2-11.0	10 cP	light yellow liquid	11.2-10.8	10 cP	yellow liquid
Surface Cleaner	10.5-10.2	<10 cP	orange-red liquid	10.5-10.0	<10 cP	orange-red liquid
Dishwashing Soap	10.5-10.2	1500-2000 cP	yellow gel	10.5-10.0	1500-2200 cP	orange gel
Hair Soap	9.9-9.8	1200-1300 cP	orange gel	9.9-9.7	1200-1350 cP	dark orange gel

Table 12. Microbiological test results

Formulation	Water Content (%)	Preservative	Bacteria/mL for 2 years (ISO 21149)	Yeasts and Mould/mL for 2 years (ISO 16212)
Hand Soap	~68	No	< 1	< 1
Laundry Soap	~19	No	< 1	< 1
Surface Cleaner	~92	No	< 1	< 1
Dishwashing Soap	~52	No	< 1	< 1
Hair Soap	~60	No	< 1	< 1

determine the shelf life. The stability results of the formulations are summarized in Table 11. Only slight changes were observed in the color and the viscosity of the formulations during the two-year period.

Viscosity Measurement: Hand soap (RV03 Spindle, 65 rpm), laundry soap (RV03 Spindle, 35 rpm), surface cleaner (RV03 Spindle, 35 rpm), dishwashing soap (RV03 Spindle, 30 rpm), hair soap (RV03 Spindle, 55 rpm).

Microbiological test results of formulations are summarized in Table 12. Generally, microbiological risk is very high in personal and home care formulations with high water content. Although these formulations contained high water content, no microbiological growth was observed during the shelf life. Microbiological tests were performed according to iso standards (27, 28).

DISCUSSION

While there are many publications on industrial surfactants (sls, sles etc.), there are very few studies in the literature on natural soaps. There is also no literature study comparing industrial surfactants with castil soaps. The word soap is used very incorrectly in the cosmetic industry. Every product with surfactants is named soap, but natural soaps are castile (traditional) soaps obtained by the saponification reaction of natural vegetable oils.

This study demonstrates the viability of producing natural soap based raw materials from vegetable oils and fatty acids to reduce costs and increase efficiency when manufacturing personal and household cleaning products.

Castile soap surfactants obtained as a result of natural vegetable oils or fatty acids reacting with a caustic are widely used in cosmetic products and the chemical industry. The trend towards natural and non-toxic raw materials recently has increased the importance of this soap based raw materials. In line with the updates in the Natural Cosmetics Standards (COSMOS, ISO 16128), these soap based raw materials can be called 100% natural (29-31). Although the saponification of vegetable oils using a caustic is a chemical reaction, in Figure 2, the value in the NNI and SyMo columns specified by the standard is 0, and these soap based raw materials are considered 100% natural (or Natural origin index:1).

Natural castile soap formulations have a basic pH (10-11) due to their chemical structure. High pH values have antimicrobial effects and are effective against harmful pathogens and viruses, thus eliminating the need to add preservatives, which often have toxic effects, to the final products. Although the castile formulations did not contain preservatives, no microbiological growth was observed during their 2-year shelf life.

CERTIFICATION LEVEL: COSMOS APPROVED RAW MATERIAL (SCOPE 2)

Commercial name	INCI	Function	%PPAI	%CPAI	%NNI	%SyMo	Restriction	Approved since
S-Base Hair	Potassium Oleate, Potassium Linoleate, Potassium Palmitate,Potassium Stearate	Surfactant	0	100	0	0		04.04.2024
S-Base PO-FA	Potassium Olivate	Surfactant	0	100	0	0		04.04.2024
S-Base PC	Potassium Cocoate	Surfactant	0	100	0	0		04.04.2024
S-Base PO-HP	Potassium Olivate	Surfactant	0	100	0	0		04.04.2024
S-Base PCS	Potassium Castorate	Surfactant	0	100	0	0		04.04.2024
S-BASE PR	Potassium Rosinate	Surfactant	0	100	0	0		04.04.2024
S-BASE MIX	Aqua, Potassium Olivate, Potassium Cocoate, Potassium Castorate, Vinegar, Sodium Bicarbonate	Surfactant	0	31,5	0	0		04.04.2024

Figure 2. ETKO Cosmos Certificate for soap bases.

CONCLUSION

Synthetic surfactants are generally poorly soluble in water. They remain on dishes, clothes and surfaces after washing, and build up over time. When they come into contact with our bodies, they can cause irritation and allergic effects on the skin surface. In contrast, natural castile soaps are 100% soluble in water, minimizing the risk of residue. This makes it easier to develop personal and household cleaning products that are safer for consumer health.

This article documents the laboratory-scale production of six soap based raw materials and the development of five castile formulations for personal and household cleaning. Super fat method optimized the total amount of caustic required for the saponification of natural vegetable oils maintains the oils in the product at the maximum level without allowing the presence of free caustic in the final product. Super fat castile soaps are believed to be the safest personal and household cleaning products and they have been used safely for centuries.

Ethics Committee Approval: Ethics committee approval was not required because the study used no material or experimental animals that would require approval.

Peer-review: Externally peer-reviewed.

Author Contributions: Conception/Design of Study - G.O.; Data Acquisition - G.O., A.B.; Data Analysis/Interpretation - G.O., A.B.; Drafting Manuscript - G.O.; Critical Revision of Manuscript - G.O.; Final Approval and Accountability - G.O.

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