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Utilizing Used Cooking Oil and Organic Waste: A Sustainable Approach to Soap Production

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Abstract: This research examined the potential for utilizing waste materials generated during the production of dishes/meals and organic waste. Specifically, it evaluated the use of orange peel (OP), spent coffee grounds (SCG), and waste cooking oil in the production of soaps. For the purposes of this study, homemade soaps were made from used food oils using the cold saponification method using sodium hydroxide. During the soap preparation, spent coffee grounds and orange peel were added to the samples in increasing concentrations of 1%, 2.5%, and 5%. The quality of the individual types of homemade soaps was evaluated on the basis of physicochemical properties such as pH, moisture, total alkalinity, total fatty matter, malondialdehyde content, fat content, foaminess, and hardness. All soaps produced using the cooking oil met the ISO quality criteria and reveal a high TFM content, low moisture content, and also very good foam stability and satisfactory foaming stability. However, no relationship was observed between the use of OP and SCG in soap production and these parameters. However, according to the ABTS test, OP and SCG significantly contributed to the antioxidant properties of the soaps, while SCG-impregnated soaps performed slightly better in this respect. Soaps with SCG also had the highest levels of flavonoids. On the other hand, the fillers used for the soap formulation reduced their hardness. All soaps showed 100% solubility in water, thus confirming the biodegradability of the product. This study demonstrated the novel potential of incorporating waste products like orange peel, spent coffee grounds, and waste cooking oil into homemade soaps, highlighting their contributions to its antioxidant properties and water solubility while ensuring high quality standards.

Keywords: waste products; ecological production; waste cooking oil; orange peel; spent coffee grounds

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1. Introduction

Globally, there is a strong emphasis on enhancing waste management strategies, driven by the growing population on our planet. Based on projected population growth dynamics, it is expected that the global population will increase from 7.8 billion in 2020 to an additional 1.3 billion people by 2050 [1]. With the anticipated population growth and subsequent rise in waste production, various reverse logistics systems are being increasingly researched and proposed [2,3]. Significant waste also includes a group of so-called food organic waste, i.e., products that originated as part of the production of food and foodstuffs. Cooking oil is a type of vegetable oil that contains glyceride compounds derived from various fatty acids. During frying, harmful substances such as acrolein and malondialdehyde are created, posing risks to human health. These toxic products result from processes such as hydrolysis, thermal degradation, oxidation, and polymerization [4]. This is why it is unsuitable for cooking oil to be used multiple times for culinary purposes [5]. In addition to

households and restaurants, industrial food production also generates significant amounts of waste cooking oil. Unfortunately, the management of waste cooking oil from industrial food production is often inconsistent with basic environmental protection principles in some countries. Cooking oil from industry is frequently discharged into the immediate vicinity of food processing factories. This behavior is alarming and has a substantial negative impact on ecosystems [6]. There is a great effort to maximize the collection and subsequent recycling of waste oils in the countries of the European Union. It is known that the collection and recycling of oils originating from the food industry and restaurants is relatively successful. However, problems remain with waste oil originating from households, where a high percentage of this waste oil is still untreated and negatively affects wastewater treatment and the environment [7]. It is therefore important to change the strategies for processing waste cooking oil, so that we do not leave waste as the final product, but on the contrary can use the waste product as a basic material for further production and thus increase its economic value and current environmental benefits [6].

The potential of using waste oil as an economical raw material for producing animal feed, biodiesel, lubricants, building materials, soap, detergents, and other technical applications is being increasingly investigated [8,9]. Based on some studies, soap production appears to be one of the possible suitable alternatives for recycling edible oils. Various methods of soap production from used oil are described, whether it is the production of solid or liquid soaps [10–13]. Various methods for producing soap from used cooking oil include the cold process, hot process, liquid soap production, and rebatching. Cold and hot process saponification are advantageous for preserving the oil's properties and customization, with the cold process being particularly energy efficient. Liquid soap production offers convenience but is more complex, while rebatching allows easy recycling of soap scraps but depends on the quality of the base soap used. In the production of soaps by saponification, essentially no by-products are created, and at the same time, the production is almost energy-saving, which makes it an environmentally friendly technology [11,12]. However, according to some studies, soaps made from the recycling of edible oils can lead to a significantly faster degradation process caused by microorganisms [12,14].

Citrus peel is beneficial for soap production due to its natural properties. Its acidity can help suppress microbial growth, enhancing the soap's shelf life. Additionally, citrus peel contains essential oils and antioxidants, which can provide a pleasant fragrance and potential skin benefits. Utilizing citrus peel in soap production also offers an eco-friendly way to recycle this common waste product, reducing its environmental impact [13]. Coffee is also a very popular drink among consumers, and therefore coffee grounds are important organic wastes. Coffee grounds are the residual part of ground coffee that is created during the coffee brewing process, whether it is for home preparation or industrial coffee processing in the production of soluble coffee. The food industry, i.e., factories producing soluble coffee, must have a waste management plan, which sets out how this organic matter will be handled. Spent coffee grounds are most commonly used for composting, producing biodiesel, sourcing sugars, making activated carbon, or serving as a sorbent for removing metal ions [15,16]. SCG appear to be an interesting commodity for further evaluation, as they contain a large amount of organic (bioactive) compounds [15,17,18].

Spent coffee grounds (SCG) offer several advantages for soap production. Firstly, they provide gentle exfoliation, helping to remove dead skin cells and leaving the skin feeling smooth and refreshed. Additionally, SCG contain natural oils that can contribute moisturizing properties to the soap, promoting skin hydration. Furthermore, the caffeine content in SCG has been suggested to have antioxidant properties, potentially offering skin protection against free radicals. Utilizing SCG in soap production also aligns with sustainability efforts by repurposing a common waste product, thereby reducing its environmental impact [19,20].

Therefore, the presented study directly aimed to analyze the possibility of using the organic waste of orange peel, coffee grounds, and frying oil in the home production of homemade ("recycled") soap.

2. Materials and Methods

2.1. Soap Samples Preparation

The soap samples were prepared using cold saponification, incorporating used frying oil and adding orange peel and spent coffee grounds (SCG). The soap production process followed the cold saponification method originally described by Adigun et al. [21], with minor adjustments. Specifically, 130 g of filtered oil was mixed with 66.92 g of a 26% (*w*/*w*) NaOH water solution using a blender for 3 to 5 min. The orange peel and spent coffee grounds were added to this step of the soap preparation. The resulting mixture was then poured into molds and left to mature in the open air after 24 h. After a period of 4 weeks at room temperature (22 °C ± 2 °C), the soap samples were ready for analysis. SCG and orange peels were added during the soap preparation (in the following concentrations: 1%, 2.5%, and 5%) (Table 1).

Table 1. The description of the samples.

Samples	Ingredients
Control	130 g frying oil + NaOH
OP1%	130 g frying oil + NaOH + 1% orange peel
OP2.5%	130 g frying oil + NaOH + 2.5% orange peel
OP5%	130 g frying oil + NaOH + 5% orange peel
SCG1%	130 g frying oil + NaOH + 1% spent coffee grounds
SCG2.5%	130 g frying oil + NaOH + 2.5% spent coffee grounds
SCG5%	130 g frying oil + NaOH + 5% spent coffee grounds

2.2. Evaluation of the Physicochemical Properties of the Soap

The physicochemical parameters of the soap samples were studied, including pH, moisture, total alkali (TA), total fatty matter (TFM), malondialdehyde (MDA), fat content (FT), foaming, solubility and hardness.

The pH value was determined by measuring it in a 1% soap solution diluted with distilled water [22], using an Orion 4-star pH meter from Thermo Scientific, Waltham, MA, USA.

The total alkali content was determined through an acid–base titration method. Specifically, 10 g of the soap sample was dissolved in ethanol and then mixed with 5 mL of 1 N H_2SO_4 (aqueous solution). The excess acid was subsequently titrated with 1 N NaOH, using phenolphthalein as an indicator. The total alkali content was calculated using Formula (1) [23]. More precisely, 10 g of soap with a pH value of approximately 11.3 was dissolved in 60 mL of ethanol in a shaker overnight and then supplemented with 5 mL of H_2SO_4 (1.01 pH).

$$\%\text{Total alkali} = (V_{acid} - V_{base})/(m_{sample}) \times 3.1. \quad (1)$$

V_{acid}—volume of H_2SO_4; V_{base}—volume of NaOH; m_{sample}—mass of sample.

Foam production and stability were evaluated following the methodology described by Kempka et al. [24]. Specifically, 20 mL of a 0.5% soap solution underwent 30 s of homogenization within a 400 mL low-form glass beaker, using a HG-15A homogenizer manufactured by Witeg in Wertheim, Germany. The homogenization process occurred at approximately 13,500 RPM, employing the HT1025 dispersing tool (ETA a.s., Prague, the Czech Republic). Foam volume was measured immediately after mixing and again after 30 min. The foaming capacity (FC) was determined using the following Formula (2):

$$\%\text{Foaming capacity} = (V_{AH} - V_{IS})/V_{IS} \times 100 \quad (2)$$

V_{AH}—volume after homogenization; V_{IS}—volume of initial solution.

The foam stability was calculated using Formula (3):

$$\%\text{Foam stability} = V_{30min}/V_{AH} \times 100 \quad (3)$$

V_{30min}—foam after 30 min; V_{AH}—foam after homogenization

To determine the total fatty matter (TFM), a 10 g sample of soap was dissolved in hot ethanol (50 °C) that had been neutralized and was then filtered. The residue left on the filter indicated the portion of the material that was not soluble in alcohol, referred to as Matter Insoluble in Alcohol (MIA). Both of these methods were performed according to the previous research [25].

The MIA value was determined by drying (+105 °C, 24 h, in an oven: ECOCELL™) and weighing the filter, and the total fatty matter was calculated using Formula (4):

$$\%\text{TFM} = (100 - (\text{moisture content} + \text{MIA}))/1.085 \quad (4)$$

MDA (malondialdehyde) was determined using the modified TBA (thiobarbituric acid) method as per Khalifa et al. [26]. The soap sample (an amount of 1.5 g) was homogenized with 1 mL of EDTA (ethylenediaminetetraacetic acid, 0.3% water solution), 5 mL of BHT (butylated hydroxytoluene, 0.8% solution in hexane) and 8 mL of TCA (trichloroacetic acid, 10% water solution); the centrifugation was conducted for 5 min at 3000 RPM. The lower layer was filtered into a 10 mL volumetric flask and topped up to the mark with 10% TCA. A mixture was created by combining 4 mL of this solution with 1 mL of TBA, followed by incubation for 90 min at 70 °C in the dark. The mixture was then rapidly cooled to room temperature using an ice bath and allowed to stand for 45 min. Absorbance measurements were taken using a spectrophotometer (CE7210, Cecil Instruments, Milton, UK) at 532 nm. The results were calculated using a standard calibration curve.

The moisture content of the soap samples was measured in the following way: 5 g of the sample underwent desiccation within an oven set at 105 °C until achieving a state of consistent mass. Analytical balance model ALS 250-4A, Kern (Balingen, Germany), with a sensitivity of 0.1 mg, was used for weighing the samples. The percentage of moisture was measured according to the following Formula (5):

$$\%\ \text{moisture} = \left(\left(m_{\text{sample}} - m_{\text{dried sample}}\right)/m_{\text{sample}}\right) \times 100 \quad (5)$$

2.3. Determination of the Antioxidant Potential of the Soaps

Antioxidant potential was determined using 3 methods: ABTS, DPPH, and FRAP.

Using three methods to determine the antioxidant potential of the soaps provides a comprehensive and robust evaluation for several reasons. Each method assesses antioxidant activity through different mechanisms. The ABTS method measures the ability of antioxidants to scavenge the ABTS•+ radical, the DPPH method evaluates the reduction of the DPPH radical, and the FRAP method assesses the reducing power by determining the reduction of the Fe^{3+}-TPTZ complex. By using all three, this study captures a wider spectrum of antioxidant behaviors [27–29].

The antioxidant potential was evaluated based on the principle of comparing the antiradical activity of the sample with the antiradical activity of the substance Trolox by a modified method using fundamental principles according to Thaipong et al., [27]. Specifically, the quenching of the ABTS• + radical by the antioxidant in the sample was monitored using a spectrophotometer. Absorbance was measured at 735 nm. The calculation was according to Formula (6):

$$\text{ABTS}\ (\%) = \left[\left(\text{Abs}_{\text{ABTS}} - \text{Abs}_{\text{sample}}\right)/\text{Abs}_{\text{ABTS}}\right] \times 100 \quad (6)$$

Abs_{ABTS}—absorbance of ABTS solution; Abs_{sample}—absorbance of the sample.

The antiradical activity was evaluated based on the reaction of the substance with the stable radical DPPH (2,2-diphenyl-1-picrylhydrazyl). The DPPH radical is reduced, and this change is captured using a spectrophotometer (absorbance measured at 517 nm,

blank was ethanol). The method was carried out based on Brand-Williams et al., [28]. The calculation was according to Formula (7):

$$\text{DPPH(scavenging activity)}(\%) = \left[\left(\text{Abs}_{\text{DPPH}} - \text{Abs}_{\text{sample}}\right)/\text{Abs}_{\text{DPPH}}\right] \times 100 \quad (7)$$

Abs_{DPPH}—absorbance DPPH solution; $\text{Abs}_{\text{sample}}$—absorbance of the sample.

The detection of the antioxidant potential by the FRAP method consists of the determination of the redox properties of the substance based on the reduction of the Fe^{3+}-TPTZ complex by antioxidants in the sample. Changes in absorbance were measured by a spectrophotometer (593 nm, after zeroing the spectrophotometer using a blank). The method was according to the methodology of Behbahani et al., [29] and was lightly modified since the solvent was ethanol:water (1:1).

2.4. Textural Parameter and Solubility

The hardness of the soap samples was measured using a TA.XT plus texture analyzer (Stable Microsystems, Godalming, UK). A stainless P/5 cylindrical probe was used to penetrate the soap bars, which had dimensions of 50 × 50 × 20 mm, at five different points. The probe had a diameter of 5 mm, a penetration depth of 5 mm, and a test speed of 1 mm/s. The instrument was equipped with a 50 kg load cell. Hardness was defined as the force (in grams) required for the probe to create a 5 mm deep indentation in the soap sample.

The solubility was measured by immersing 2 g of the soap sample in distilled water (100 mL) and visually inspecting the solution after 24 h.

2.5. Statistical Analysis

The results are displayed in tables, including mean values and standard deviations. Statistical analysis to identify differences between the sample groups was conducted using one-way ANOVA with SPSS 20 (IBM, Corporation, Armonk, NY, USA).

3. Results and Discussion

The chemical characteristics of the experimentally prepared soap bars are summarized in Table 2; the presented scope of data includes information on the pH values, total alkali, total fatty matter, MDA, and fat content.

Table 2. Chemical characteristics of the soaps with the addition of orange peel and spent coffee grounds.

Samples	pH	Total Alkali (%)	Total Fatty Matter (%)	MDA (TBA µg/g)	Fat Content (%)
Control	$10.37 \pm 0.06^{a*}$	0.00 ± 0.00	91.17 ± 0.61	0.99 ± 0.29	8.69 ± 8.05
OP1%	10.29 ± 0.05^{acd}	0.00 ± 0.00	91.59 ± 0.26^{a}	4.84 ± 0.12	14.84 ± 5.00^{b}
OP2.5%	10.29 ± 0.04^{acd}	0.00 ± 0.00	91.90 ± 0.02^{a}	4.41 ± 1.28	19.41 ± 0.92^{b}
OP5%	10.25 ± 0.02^{d}	0.00 ± 0.00	91.83 ± 0.04^{a}	6.66 ± 1.67	18.83 ± 3.81^{b}
SCG1%	10.38 ± 0.03^{ae}	0.00 ± 0.00	90.61 ± 0.16^{b}	1.93 ± 0.06	17.90 ± 6.40^{b}
SCG2.5%	10.32 ± 0.03^{acd}	0.00 ± 0.00	91.55 ± 0.06^{a}	2.32 ± 0.00	14.36 ± 0.55^{b}
SCG5%	10.36 ± 0.01^{acd}	0.00 ± 0.00	91.16 ± 0.36	3.49 ± 0.55	18.95 ± 3.31^{b}
SCG	5.23 ± 0.02^{b}	NA **	NA **	3.55 ± 0.21	11.47 ± 0.21
OP	6.03 ± 0.04^{f}	NA **	NA **	1.26 ± 0.23	0.71 ± 0.12^{a}

* Different lowercase letters indicate statistically significant differences ($p < 0.05$) between rows. ** Not applicable.

The pH ranged from 10.25 ± 0.02 in sample OP 5% to 10.38 ± 0.03 in sample SCG 1%. The addition of 5% OP resulted in a statistically significant ($p < 0.05$) lower pH in comparison with the control soap samples prepared without the addition of SCG or OP. The lower pH of the orange peel was measured to be 6.03 ± 0.04. Our results are consistent with the values obtained by Sanaguano-Salguero et al. [30], where the pH of soaps produced from wasted oil recovered from fast-food restaurants ranged from 9.96 to 11.30. Compared to commercially available products, our results are similar to soaps with antibacterial

properties (pH values from 10.28 to 11.34) and some soap bars intended for children's care (pH from 7.41 to 11.55) [31].

The typical pH range of normal human skin is between 5.4 and 5.9. Introducing soaps with a higher pH can potentially impact the skin's protective function and microbiome. However, it has been shown that a considerable portion of commercial soaps have pH values significantly above neutral [31–34]. Total alkali is a frequently used quality parameter indicating the amount of all alkaline components, such as sodium/potassium hydroxides, carbonates, or bicarbonates. Generally, a lower TA (total alkalinity) value indicates a better quality of the soap. Excess alkalis can cause the saponification of the fats and oils naturally present on the skin, which form a protective coating. This protective layer is gradually washed away with water, leading to dryness and skin irritation [33,35]. Moreover, using soap with a high total alkaline content not only dries the skin but also causes scaling, making the skin more susceptible to fungal infections. Furthermore, it has been proven that skin exposure to the activity of an alkali agent such as NaOH increases the pH of the skin, and in consequence, leads to alterations in the activities of the enzymes responsible for filaggrin degradation [36]. Filaggrin is an epidermal differentiation protein that, under normal conditions, is proteolytically processed into free amino acids. These amino acids are further converted into highly hygroscopic structural components of the natural moisturizing factor (NMF), which maintains the *stratum corneum* at an optimal hydration level [37]. Research has shown that both genetic factors, such as mutations that result in the loss of function of the gene encoding filaggrin, and environmental factors, including exposure to alkaline agents that disrupt the processing of this protein, contribute to impaired skin function [38,39]. According to the standards set by the International Standardization Organization (ISO), the total alkalinity (TA) in good-quality soaps should not exceed 2%. In our study, the TA value was 0.00% for all experimentally produced soap bars. In a study conducted by Legesse, soaps made from used oil also showed a low, acceptable level of TA (ranging from 0.78 $\pm$ 0.02% to 1.09 $\pm$ 0.05%). However, these values were higher compared to the values obtained in our study [40].

It is also noteworthy that the total alkalinity (TA) values of all soaps presented in our experiment are lower compared to some commercially available soaps [41]. Apart from TA, another important parameter specified in commercial transactions indicating soap quality is the total fatty matter content [24]. The term TFM (total fatty matter) refers to the absolute amount of fatty acids such as stearic acid, oleic acid, palmitic acid, and sodium oleate that can be released from the soap upon reaction with a mineral acid. Soaps with a high TFM value offer strong cleansing and lathering capabilities, effectively removing impurities and oil residues from the skin surface [42]. Besides, soaps high in TFM are less harmful, rehydrate the skin, and act as a lubricant throughout the day. Conversely, soaps with a lower TFM tend to absorb the skin's moisture, leaving it more sensitive and drier, and it is then prone to rashes, infections, and skin breakdown. Using soap with a low TFM may contribute to the occurrence of a skin condition known in medical terminology as xeroderma [43]. The TFM value is crucial for categorizing soap quality. Soaps with higher TFM levels are generally harder and of superior quality. In contrast, soaps with lower TFM, typically below 76%, are less desirable because they may contain higher levels of moisture and fillers. This is particularly true for commercial laundry soaps, where these factors can reduce their effectiveness.

According to the International Standardization Organization (ISO), high-quality soaps are expected to have a total fatty matter (TFM) content exceeding 76% [44]. These standards classify soap grades as follows: Grade 1 requires a minimum TFM of 76%, Grade 2 necessitates at least 70% TFM, and Grade 3 mandates a minimum of 60% TFM. In our experiment, soaps made from used vegetable oil achieved high TFM values ranging from 91.16% to 91.90%. These values exceed the 76% recommended for bath soaps, indicating their high quality, cleaning efficacy, and skin care properties. Additionally, these results surpass the TFM levels found in some branded soaps [33]. For example, washing commercial soaps, namely, Dove, Johnson, and Nivea achieved a TFM of 73%, 74%, and 84%, respectively [45].

Moreover, the addition of orange peel or spent coffee grounds did not affect the TFM level, since the TFM of the OP and SCG soaps did not differ from each other or the control sample ($p > 0.05$). Although, the sample with the addition of 1% of SCG had the lowest ($p < 0.05$) TFM (90.61 ± 0.16%). The obtained results are also more favorable compared to those achieved by Legesse and colleagues [40] when soaps prepared from oils collected from restaurants showed TFM values ranging from 75.42 to 88.53%.

Malondialdehyde is a highly reactive substance, formed as a secondary product of the peroxidation of unsaturated fatty acids, a process that intensively occurs during frying in oil rich in UFA [46]. For this purpose, MDA is commonly used as a marker of lipid peroxidation of edible oil [47]. In the tested samples, the lowest MDA level was in the control sample (0.99 ± 0.29 μg/g), and the highest value was reported for the OP5% (6.66 ± 1.67 μg/g). Little information can be found in science databases on the MDA status of soap prepared from frying oil, so it is difficult to relate our results to any relevant reference point. Nevertheless, there is a little evidence suggesting that edible oils with an MDA concentration of <0.576 mg MDA/kg are considered fresh, those with an MDA value of 0.65–1.44 mg MDA/kg are rancid but still within acceptable limits, and those with an MDA concentration > 1.5 mg MDA/kg are rancid. According to the results from the control soap sample containing only frying oil, it is challenging to attribute the source of malondialdehyde (MDA) in soaps with the addition of orange peel (OP) and spent coffee grounds (SCG) solely to the frying oil. This is because the MDA content in the control soap bar falls within acceptable limits. However, the extracts of SCG and OP showed higher concentrations of malondialdehyde, specifically 3.55 ± 0.21 μg/g for SCG and 1.26 ± 0.23 μg/g for OP.

The results for foaming properties, moisture content, solubility, and hardness are present in Table 3.

Table 3. Foaming properties, moisture content, solubility and hardness of the experimentally produced soaps.

Samples	Foaming Capacity (%)	Foam Stability (%)	Moisture (%)	Solubility (%)	Hardness (g)
Control	129 ± 27 ^{a}*	93.65 ± 2.59 ^{a}	5.43 ± 0.34 ^{a}	100	3380 ± 87 ^{f}
OP1%	148 ± 27.53 ^{a}	94.44 ± 3.12 ^{a}	4.98 ± 0.26 ^{a}	100	3047 ± 173 ^{cd}
OP2.5%	123 ± 16.58 ^{a}	92.74 ± 1.95 ^{a}	5.62 ± 1.08 ^{a}	100	2127 ± 121 ^{a}
OP5%	126 ± 23.93 ^{a}	94.45 ± 1.32 ^{a}	5.87 ± 0.54 ^{a}	100	2115 ± 78 ^{a}
SCG1%	125 ± 9.128 ^{a}	94.46 ± 2.19 ^{a}	4.10 ± 0.32 ^{a}	100	3115 ± 123 ^{cde}
SCG2.5%	105 ± 16.83 ^{a}	94.46 ± 1.47 ^{a}	5.74 ± 0.05 ^{a}	100	2855 ± 181 ^{c}
SCG5%	125 ± 33.41 ^{a}	94.45 ± 1.89 ^{a}	5.71 ± 0.63 ^{a}	100	2467 ± 122 ^{b}
SCG	NA **	NA **	7.45 ± 0.35 ^{a}	NA **	NA **
OP	NA **	NA **	10.9 ± 0.46 ^{b}	NA **	NA **

* Different lowercase letters indicate statistically significant differences ($p < 0.05$) between rows: (a, b); ** Not applicable.

Foaming is a critical factor that enhances the washing properties of soap. It is defined as a dispersion system consisting of gas bubbles coated with a layer of liquid [48,49]. The fatty acid composition of the oils utilized in soap formulation contributes to its foamability. The presence of saturated fatty acids (SFA) with short carbon chains like lauric acid and myristic acid allows the production of a fast-forming fluffy lather with high cleansing power [40,50].

On the other hand, the aforementioned fatty acids are more soluble in water and, therefore, will not provide a long-lasting product. Nevertheless, palmitic and stearic acids with long carbon chains form solid soap bars with longer lifespans and create the stable and creamy lather desired for shaving soaps. Unsaturated fatty acids (UFA), such as oleic and linoleic, have long hydrophobic carbon chains in their structure; thus, such soaps are characterized by poor water solubility and are hard to solidify. Additionally, because UFA

compounds contain a double bond between at least one pair of carbon atoms, soaps rich in UFA are more susceptible to rancidity [51].

In the presented study, the highest foaming rate was recorded for the samples with the addition of 1% orange peel (OP1%) and the lowest after the addition of 1% spent coffee grounds (SCG2.5%), where the FC (foaming capacity) reached 148% and 105%, respectively. Orange peel, in addition to its odor-masking properties in the process of producing soap from frying oil, also acts as a soap booster. A moderate effect of orange peel used in soap production on the soap's foaming potential has been observed previously [52]. However, in our analysis, there was no correlation between the amount of orange peel used and the foaming power. The greatest foaming ability was demonstrated in the latest work described by Antonic and colleagues [25]. Their soaps, produced from different frying and cooking oils, exhibited FCs ranging from 123% to 408%, depending on the type of oil. The lowest FC was for soaps produced from fried palm oil (FC from 123% to 235%), whereas the sunflower-based soaps revealed FCs ranging from 250% to 408%, and soaps produced from rapeseed oil reached FC values between 262% and 350%. On the other hand, in the same study, soaps prepared from palm oil showed the highest foam stability (61% to 78%). This may be because palm oil is rich in palmitic and stearic acids, ensuring greater foam stability [53]. In our study, the soaps with spent coffee grounds and orange peel addition had significantly higher foam stability (92.74% to 94.46%). Although, compared to the control sample, there is no clear relationship between the type and amount of the additive used and the foam stability. Consequently, it can be concluded that the stability of the foam is influenced by the composition of fatty acids contained in the oil.

The moisture content indicates the amount of free water in the soap, making it an important parameter for predicting the soap's shelf life. Excess water can lead to soap spoilage due to hydrolysis of un-saponified fat, producing free fatty acids and glycerin [33]. The moisture contents shown in Table 3 ranged from 4.10 $\pm$ 0.32% for the sample SPC1% to 5.87 $\pm$ 0.54% for sample OP2.5% and were lower than the results of the earlier findings [54]. No statistically significant difference ($p > 0.05$) was found between these values. Therefore, it can be assumed that the enrichment of the soap with the mentioned components did not affect its moisture. Most commercial soaps are traded with the specification of MC max 14%; however, the value depends on the final purposes of the soap bar [44].

Solubility was 100% for all samples (with and without the addition of OP and SCG), which indicates that the non-soluble waste of cooking oils that were fortified with SCG and OP was turned into a 100% water-soluble product [55]. Hardness is an important physical characteristic that determines the quality of the soap bar. Overall, a higher hardness level is preferable for solid soaps as it allows them to maintain their shape. From the obtained results, it can be concluded that the hardness of soap is influenced by the incorporation of plant additives such as OP and SCG. The highest level of hardness was demonstrated by the control sample (3380 $\pm$ 87 g), while infused soaps showed a lower level of hardness; there is also a noticeable correlation indicating that an increasing amount of these additives resulted in a hardness level decrease. Adequate dependence was noticed in the study performed by Purwanto et al., where the addition of an extract of dragon fruit peels affected the hardness of the soap [56]. Besides, hardness can be influenced by other factors such as moisture content, meaning that a lower moisture content would result in a higher hardness level and vice versa [57]. Furthermore, the level and profile of the fatty acids also influence the physical properties of the soaps. It was observed that SFA, such as palmitic fatty acid, formulate soaps much harder than UFA. The explanation for this is the fact that SFAs have much less limited rotation around each C-C bond, unlike UFAs, and can therefore be more easily packed into a solid form [58].

The antioxidant potential of the produced soaps was assessed using various assays, including FRAP, DPPH, and ABTS. Moreover, phenolic and flavonoid content was also evaluated. The obtained results are summarized in Table 4.

Table 4. The content of the polyphenols and flavonoids and antioxidant properties of produced soap samples.

Samples	TPC (mg gallic acid/g)	TFC (mg epicatechin/g)	FRAP (%)	DPPH (%)	ABTS (%)
Control	5.16 ± 7.36 ^{acdf*}	0.08 ± 0.00 ^{a}	96.67 ± 4.38 ^{a}	4.03 ± 0.11 ^{a}	3.54 ± 0.54 ^{ac}
OP1%	4.30 ± 0.50 ^{ad}	0.25 ± 0.03 ^{cf}	67.77 ± 0.26 ^{c}	5.67 ± 0.55 ^{a}	4.07 ± 1.07 ^{cd}
OP2.5%	3.64 ± 0.06 ^{a}	0.22 ± 0.01 ^{cdf}	68.63 ± 3.48 ^{b}	5.35 ± 0.05 ^{a}	5.07 ± 0.51 ^{d}
OP5%	4.03 ± 0.82 ^{ad}	0.11 ± 0.01 ^{e}	74.45 ± 7.50 ^{bd}	17.63 ± 1.75 ^{c}	5.43 ± 0.35 ^{d}
SCG1%	3.23 ± 0.06 ^{ac}	0.27 ± 0.03 ^{fg}	86.63 ± 7.77 ^{ba}	3.74 ± 0.24 ^{a}	4.72 ± 0.59 ^{cd}
SCG2.5%	4.12 ± 0.36 ^{ab}	0.31 ± 0.01 ^{g}	90.09 ± 7.16 ^{da}	5.26 ± 0.13 ^{a}	5.33 ± 0.74 ^{d}
SCG5%	4.81 ± 0.30 ^{bd}	0.31 ± 0.01 ^{hg}	78.47 ± 9.02 ^{eab}	7.69 ± 0.78 ^{a}	6.29 ± 1.07 ^{d}
SCG	23.53 ± 0.33 ^{e}	0.17 ± 0.00 ^{i}	137.4 ± 7.88 ^{f}	-	30.61 ± 1.94 ^{c}
OP	15.68 ± 0.16 ^{f}	0.02 ± 0.00 ^{j}	69.61 ± 3.80 ^{bd}	84.66 ± 1.94 ^{b}	22.86 ± 1.47 ^{f}

* Different lowercase letters indicate statistically significant differences ($p < 0.05$) between rows: (a, b, c, d, e, f).

Orange peel and spent coffee grounds, apart from containing essential oils that provide a pleasant fragrance, also contain substances that can suppress the oxidation of UFAs present in the soap, prolonging its durability, and also can have a protective effect on the skin. The main pigments present in orange, namely carotenoids, in addition to their color, which can positively contribute to the visual appearance of soap, have been associated with strong antioxidant properties. Out of the three isomers of carotene, namely alpha, beta, and gamma, the beta isomer (β-carotene) is the most active and acts as an antioxidant, effectively quenching reactive oxygen species [59]. Besides, β-carotene is a precursor of the retinol known as vitamin A, capable of inhibiting lipid peroxidation and possesses the ability to scavenge the singlet oxygen, neutralizing thiol radicals, and stabilizing peroxyl radicals [60–62]. Hesperidin belongs to a group of flavonoids found abundantly in orange peel and is considered a promising substance for use in skin care products due to its antioxidant, anti-inflammatory, and anti-cancer properties [63]. Spent coffee grounds can be reused as a raw material for soap production, providing skincare benefits due to their richness of phytochemical components that exhibit various protective effects. The high quantities of phenolic acids such as chlorogenic, ferulic, caffeic [64], ellagic, gallic, tannic, and p-coumaric acids [17] with well-known antioxidant and antimicrobial activities make SCG a valuable substrate for soap formulation.

Today, there is a growing consumer demand for antioxidants derived from natural sources. This trend is driven by the effectiveness of natural antioxidants, their safety, and the health implications associated with synthetic alternatives. Synthetic antioxidant compounds used in soap production, such as butylated hydroxyanisole (BHA) or butylated hydroxytoluene (BHT), may offer higher antioxidant effectiveness. However, they have been documented to potentially cause skin issues such as irritation or allergic reactions [65,66]. In the present study, total polyphenol content in the produced soap samples ranged from 3.23 ± 0.06 mg/g (SPC1%) to 5.16 ± 7.36 mg/g (control sample). Surprisingly, both types of soaps infused with OP or SCG did not achieve higher TPC values compared to the control soap sample without any additives. Overall, the soaps with OP and SCG fortification achieved a comparable level of TPC. However, considering the TPC values of stand-alone OP (15.68 ± 0.160) and SCG (23.53 ± 0.33), the SCG soaps retained the proportionally lowest level of antioxidants in the saponification process. This observation indicates that some polyphenols are more susceptible to degradation under the difficult conditions occurring during the saponification process. Our observations confirm the results obtained by Adigun and colleagues [21] according to which, in the cold saponification process, soaps enriched with extracts of wild Newfoundland blueberries retained the lowest level of antioxidants among the four wild blueberries.

Polyphenols are gaining increasing interest in cosmetology due to their dual action as antioxidants and antimicrobial agents [67,68]. Flavonoids are a group of phenolic compounds that include various subclasses such as anthocyanins. Their antibacterial, antifungal, and anti-inflammatory properties make them potentially beneficial for enhancing

the skin care properties of soap [63,69]. Soaps enriched with both OP and SCG showed significantly ($p < 0.05$) higher TFC values compared to the control sample. The addition of spent coffee grounds (SCG) to the soap showed a slightly stronger impact on the flavonoid content compared to orange peel (OP). However, the concentration of flavonoids did not consistently correlate with the percentage of OP and SCG in the soap composition, and this relationship did not demonstrate a clear trend. Further characterization indicated that fortifying soaps with OP and SCG enhanced their antioxidant properties. A dose-dependent effect of OP and SCG on the soap's antioxidant capacity was observed in the ABTS test. According to the ABTS results the highest antioxidant capability was revealed for the SCG5% sample, which is consistent with the antioxidant capacity of the SCG extract (30.61 ± 1.94). Inconsistent results were observed using the FRAP assay, which indicated that infusing soap with orange peel (OP) and spent coffee grounds (SCG) reduced the antioxidant properties of the soaps. This disparity can be attributed to the different mechanisms of action employed by each method. The ABTS and DPPH assays primarily involve electron transfer reactions to assess antioxidant activity, whereas the FRAP assay evaluates the antioxidant's ability to provide reducing power [70]. Additionally, the ABTS assay offers greater flexibility as it can be applied to measure antioxidant activity across various pH levels. In contrast, the DPPH method is sensitive to acidic conditions and may not perform consistently under different pH environments [71].

4. Conclusions

The reported work provides evidence that used oil, which is considered a waste product and a significant source of environmental pollution, can be repurposed into soap. Physicochemical analysis revealed that our soaps meet the criteria established by the ISO requirements, such as the total fatty matter and total alkali. Moreover, the soaps exhibited 100% solubility in water, indicating their degradability, which is an important ecological advantage as soap prevails over oil, which is difficult to degrade as waste. The addition of other organic waste to soap, such as orange peel and spent coffee grounds, showed a higher concentration of flavonoids and antioxidant activity compared to soap without additives, which translates into protective and caring properties—the advantages of the produced soaps. The soap formulation from used edible oil proposed in this study through cold saponification is simple, offers products of high quality, and therefore stands out as a favorable solution for small entrepreneurs. This approach not only mitigates the environmental impact of waste oils but also adds value by creating functional, eco-friendly products. Future research could explore optimizing the concentration of additives to balance their hardness and antioxidant properties further and investigate the potential marketability and consumer acceptance of these sustainably produced soaps.

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