# Chemical characteristics of soaps obtained using red hot pepper seeds oil (*Capsicum annuum* L.)

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**Abstract.** Vegetable oils are an important raw material for soap production. They are widely distributed in nature, but only some of them are used in soap production. The search for alternative raw materials for oils from renewable sources is current worldwide. As a potential source of such oil, hot pepper seeds are considered. They are waste plant products that contain proteins, dietary fiber, lipids, minerals, bioactive compounds, etc. This study aimed to identify the optimal amount of hot pepper seed oil to include in the oil blend for cold process soap making. Four variations of soaps were made with hot pepper seeds oil, palm oil and coconut oil in different ratios. The values of indicators − total fatty matter content, free alkali, foaming ability, pH were determined. Soaps containing up to 10 % hot pepper seeds oil in the oil blend exhibit satisfactory quality characteristics. The findings indicate that hot pepper seeds oil is suitable for use in cold process soap making. Its proportion can be as high as 10 % of the total oils.

## 1 Introduction

The impressive development of cosmetic products in recent decades, and in particular of those for skin hygiene, imposes more and more requirements for increased functionality and consumer appeal of soap. This determines the use of new, non-conventional vegetable oils, which are the main raw materials for soap production. In recent years, there has been an increased interest in the utilization of oils from waste plant products. They contain various fatty acids (FAs), phytonutrients and biologically active substances, which opens up the possibility of their use as raw materials in the food, chemical, perfumery and cosmetic industries, including soap production.

The object of the present study is the use of non-traditional glyceride oil from the seeds of hot red pepper (*Capsicum annuum* L.), Cayenne variety, for the production of cold process soaps.

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In Bulgaria, pepper (*C. annuum*) is the second most important vegetable crop [1-2] and today occupies an area of 4035 ha, with an annual production of 50990 t of fruit [3].

In recent years, there has been an increased interest in the waste products of pepper processing. Red pepper (*C. annuum*) seeds constitute 4.14 % of the weight of fresh pepper [4] and 2.82 % for "Cayenne" variety [5] respectively, approximately 20 % of the dry weight of the whole fruit [6]. They are the main by-product which recycling or disposal is a challenge for industry [7-8].

Pepper seeds are a potential source of glyceride oil with nutritional properties comparable to high-priced vegetable oils [9]. Data in the literature show that the oil content of the pepper seeds of genus *Capsicum* varies from 14.6 to 35.9 % among cultivated species, and the oil content in the seeds of different *C. annuum* cultivars varies from 22.1 to 35.9 % [8]. The main FAs of the oil used are oleic (67.20 %) and linoleic (25.60 %). It is unsaturated oil and helps to balance the saturated fats in the soap formula. The unsaturated oils in the soap act as moisturizing, conditioning or skin nourishing ingredients. They are a factor that affects the quality and consumer perception of soap [10-12].

The color of the oil is reddish-yellow, which indicates the presence of carotenoids in it [13]. Apart from them, other important bioactive compounds - tocopherols and phytosterols - have been found in pepper seed oil [14]. They are associated with a higher antioxidant activity of the oil. Their presence contributes to the emollient properties of the soap and to improving the feel on the skin [12, 15]. They have an antioxidant, nourishing, protective and softening effect [16], which determines the cosmetic properties of the soap. The carotenoids give a characteristic color to the oil and to the soaps obtained from it.

From this point of view, taking into account the importance of the superior valorization of by-products, the use of non-traditional glyceride oil from the seeds of hot red pepper (*Capsicum annuum* L.), Cayenne variety, for the production of cold process soaps, was considered.

The aim of this study was to identify the optimal amount of hot pepper seed oil to include in the oil blend for cold process soap making. Also, the analysis of some physical-chemical parameters was carried out.

## 2 Materials and methods

### 2.1 Materials

Hot Pepper Seed Oil (HPO), Coconut Oil (CO) and Palm Oil (PO) in varying amounts are used to prepare the soap samples:

- Palm oil is supplied by "LLC Delta Wilmar", Ukraine.
- Coconut oil is supplied by "Cargill Palm Products", Malaysia.
- Hot pepper seeds oil is obtained by extraction with *n*-hexane of pepper seeds, Cayenne variety, grown in Bulgaria; farm – "Hot Farm" in the village of Strashimirovo, Varna region, harvest 2021.
- Sodium hydroxide (NaOH), 98-99 %, KLJ - ORGANIC QATAR W.L.L.

### 2.2 Qualitative analysis of oils

The quantitative analysis of oils is performed as follow:

- Saponification value (SV) is determined titrimetrically, according to the conditions of ISO 3657:2020 [17]
- Iodine value (IV) is determined by the Weiss method, ISO 3961:2018 [18].

• Acidity is determined titrimetrically, according to ISO 660:2020, and the results are presented as a percentage of oleic acid [19].
• Determination of FA composition. It is defined according to ISO 12966-1:2014 [20] and ISO 12966–2:2017 [21]. Methyl esters of fatty acids (FAMEs) were prepared by the standard method and analyzed by gas chromatography (GC). A Hewlett Packard 5890 A apparatus with a Supelco 2560 capillary column (75 m × 0.25 mm × 18 μm) and a flame ionization detector (FID) was used for GC analysis under the following conditions: column temperature 130 °C (4 min) with heating from 15 °C/min to 240 °C (5 min); injector and detector temperature were 250 °C, carrier gas - hydrogen, at a speed of 0.8 $cm^3$/min; split ratio was 50:1. FAs were identified by comparing retention times with those of a standard mixture of FAMEs (FAME mix, 37-component, Supelco Inc., USA).
• Preparation of soap samples with red hot pepper seeds oil. Glyceride oils are saponified with sodium hydroxide, the amount of which is calculated on the basis of SV. The technology for preparing the soaps follows the method described in [22]. Four variants of soap (№1-4) were prepared. The quantitative ratios of the oils are shown in Table 1. The alkaline solution has a concentration of 16% (22 °Bè – Baume degree). The finished viscous mass is poured into soap mold.

**Table 1.** Oil blends used for soap variants.

| Oil components | Oil blends № | | | |
|---|---|---|---|---|
| | **1** | **2** | **3** | **4** |
| Hot pepper seeds oil (HPO) | - | 5 | 10 | 20 |
| Palm oil (PO) | 80 | 75 | 70 | 60 |
| Coconut oil (CO) | 20 | 20 | 20 | 20 |

### 2.3 Qualitative analysis of soap

The quantitative analysis of soaps is performed as follows:
• The moisture content is determined by drying at 105°C to constant mass, the results being expressed as a percentage, based on the weight of the soap, ISO 672:1978 [23].
• The ISO 456:1973 method is used to determine the percentage of free caustic alkali content [24].
• Determination of total fatty matter (TFM) content is carried out in accordance with ISO 685:2020 [25].
• Unsaponifiable and unsaponified matter are defined according to ISO 1067:1974 [26].
• Determination of foaming ability. It is determined according to the methodology of ISO 696:1975 [27].
• The pH of aqueous solutions of the soap samples was determined potentiometrically according to the conditions of the method ISO 4316:1977 [28].

## 3 Results and discussion

The findings of the chemical parameters of the oils utilized in this investigation, namely acidity, SV and IV are summarized in Table 2.

The oil exhibits a relatively low acidity level (0.25 % oleic acid) and complies with the specifications for crude oils, which typically allow for acidity levels up to 1 %. This result is comparable to the data obtained by [13] for red pepper (Urfa pepper) seed oil – 0.22 %.

The SV value for HPO is 187.80 mg KOH/g and is close to those of olive oil – 190.09 mg KOH/g [29] and peanut oil – 187.70 mg KOH/g [30], which were used for soap production. This indicates that oil can be included in their composition.

An important indicator that reflects the degree of unsaturation and the potential oxidation sensitivity of oils is IV. Its value for HPO is 93.00 g $I_2$/100 g oil, relatively high due to the higher amount of unsaturated linoleic acid.

**Table 2.** Properties of the used oils.

| Vegetable oil | Sapunification value (mg KOH/g) | Iodine value, (gI2/100g) | Acidity, (%) |
|---|---|---|---|
| Hot pepper seeds oil (HPO) | 187.8 ± 0.12a | 93.0 ± 1.00 | 0.25 ± 0.020 |
| Palm oil (PO) | 203.4 ± 0.29 | 55.0 ± 0.20 | 0.20 ± 0.025 |
| Coconut oil (CO) | 258.8 ± 0.34 | 11.0 ± 0.10 | 0.60 ± 0.017 |

[a.] All data are presented as mean ± standard deviation (n = 3).

This result for the IV is closely align with literature values for red pepper seed oil (Urfa pepper), which typically range around 94.26 g $I_2$/100 g oil [13].

Table 3 displays the fatty acid composition of HPO, PO, CO and the prepared oil blends.

**Table 3.** Fatty acid composition of HPO, PO, CO and oil blends.

| МК % (w/w) | | HPO | PO | CO | № 1 | № 2 | № 3 | № 4 |
|---|---|---|---|---|---|---|---|---|
| | | | | | PO:CO | HPO:PO:CO | | |
| | | | | | 80:20 | 5:75:20 | 10:70:20 | 20:60:20 |
| UFAs | Myristoleic C14:1 | 0.11 ± 0.00 | -b | - | - | 0.005 | 0.011 | 0.022 |
| | Pentadecenoic C15:1 | 0.60 ± 0.00 | - | - | - | 0.030 | 0.060 | 0.120 |
| | Palmitoleic acid C16:1 | 0.62 ± 0.03 | - | - | - | 0.031 | 0.062 | 0.124 |
| | Heptadecenoic C17:1 | 0.37 ± 0.00 | - | - | - | 0.018 | 0.037 | 0.074 |
| | Oleic C18:1 | 15.50 ± 0.23 | 39.20 ± 0.70 | 6.50 ± 0.10 | 32.66 | 31.48 | 30.28 | 27.13 |
| | Linoleic C18:2 | 42.80 ± 0.40 | 10.10 ± 0.40 | 2.10 ± 0.00 | 8.50 | 10.14 | 11.77 | 15.92 |
| | Linolenic C18:3 | 0.45 ± 0.00 | 0.40 ± 0.02 | < 0.10c | 0.32 | 0.33 | 0.33 | 0.33 |
| | Gadoleic C20:1 | 0.25 ± 0.00 | - | - | - | 0.01 | 0.03 | 0.05 |
| | Erucic C22:1 | 0.95 ± 0.01 | - | - | - | 0.05 | 0.10 | 0.19 |
| SFAs | Caprylic C8:0 | 0.10 ± 0.00 | - | 6.20 ± 0,05 | 1.24 | 1.25 | 1.25 | 1.25 |
| | Capric C10:0 | - | - | 6.50 ± 0.05 | 1.30 | 1.30 | 1.30 | 1.30 |
| | Lauric C12:0 | 0.35 ± 0.00 | 0.50 ± 0.00 | 49.00 ± 0.80 | 10.2 | 10.18 | 10.19 | 10.11 |
| | Myristic C14:0 | 1.05 ± 0.01 | 1.00 ± 0.01 | 17.70 ± 0.60 | 4.41 | 4.41 | 4.42 | 4.41 |
| | Pentadecanoic C15:0 | 0.12 ± 0.00 | - | - | - | 0.01 | 0.01 | 0.02 |
| | Palmitic C16:0 | 25.70 ± 0.32 | 44.00 ± 0.80 | 9.10 ± 0.10 | 37.02 | 36.00 | 35.18 | 33.36 |
| | Margaric C17:0 | 0.10 ± 0.00 | - | - | - | 0.01 | 0.01 | 0.02 |
| | Stearic C18:0 | 6.75 ± | 4.70 ± | 2.90 ± | 4.44 | 4.46 | 4.55 | 4.75 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | 0.06 | 0.07 | 0.01 | | | | |
| | Arachidic C20:0 | 1.10 ± 0.02 | - | - | - | 0.05 | 0.11 | 0.22 |
| | Behenic C22:0 | 3.00 ± 0.03 | - | - | - | 0.15 | 0.30 | 0.60 |
| % UFAs | | 61.68 | 49.70 | 8.60 | 41.48 | 42.18 | 42.69 | 43.96 |
| % SFAs | | 38.32 | 50.30 | 91.40 | 58.52 | 57.82 | 57.31 | 56.04 |

b - Not detected. c - Not quantified.

For the mixtures FAs content is calculated based on the percentage of the oils used in the individual mixtures.

Oil blends differ mainly in the content of oleic, linoleic and palmitic acids. As the percentage content of HPO in the oil mixture increases, the content of linoleic acid in it increases significantly, and the amount of oleic and palmitic acid decreases.

Figure 1 and Figure 2 depict the distribution percentages of saturated fatty acids (SFAs) and unsaturated fatty acids (UFAs) in both the individual oils and the prepared oil blends, respectively. As the content of HPO in the soap formulation increases, the content of UFAs increases and that of SFAs decreases by about 2.5 %. As seen from the data in Figure 2, the amount of polyunsaturated fatty acids (PUFA) increases much more sharply by 7.5 %, at the expense of monounsaturated fatty acids (MUFA), which decrease by about 5 % in oil mixture № 4.

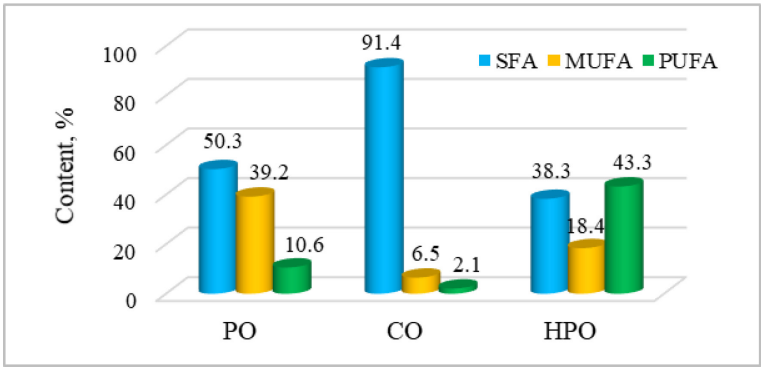


**Fig. 1.** Quantitative distribution of saturated, monounsaturated and polyunsaturated fatty acids in PO, CO and HPO.

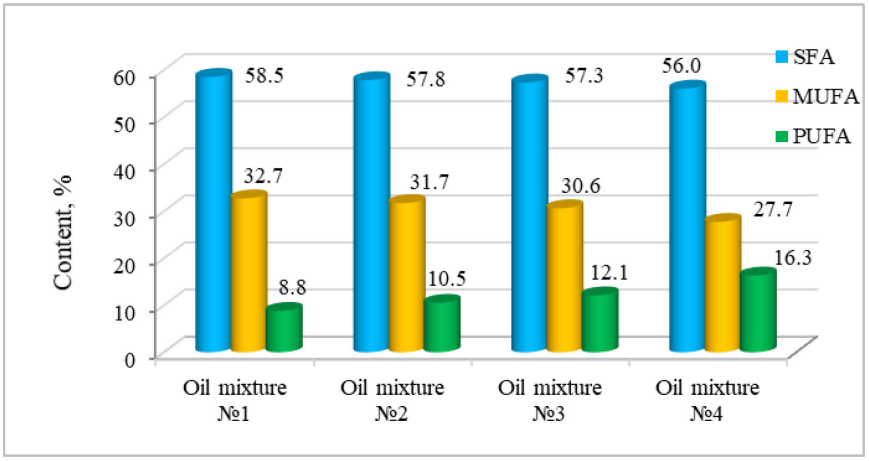


**Fig. 2.** Quantitative distribution of saturated, monounsaturated and polyunsaturated acids in the four oil blends.

In soap production, the optimal ratio of UFAs to SFAs is typically around 40/60 [31-34]. Such is the ratio of FAs in the control sample № 1 (PO: CO / 80: 20). All samples showed a similar ratio of unsaturated and saturated fatty acids. In sample № 4 the ratio is 44/56 (HPO: PO: CO = 20: 60: 20). The amount of HPO used in the soap formula is limited by the high proportion of linoleic acid in it, which is a prerequisite for oxidation processes and changes in the finished product.

The steps in making the soaps are illustrated in Figure 3. It is clearly seen that the use of HPO in the oil mixture contributes to the coloring of the base soap mass.

The four soap samples obtained were subjected to analysis based on the primary soap indicators, and the findings are detailed in Table 4.

The moisture content of these samples ranges from 30.87 % to 33.50 %, aligning with literature references (18.50-35.70 %) for cold process soaps [35-37].

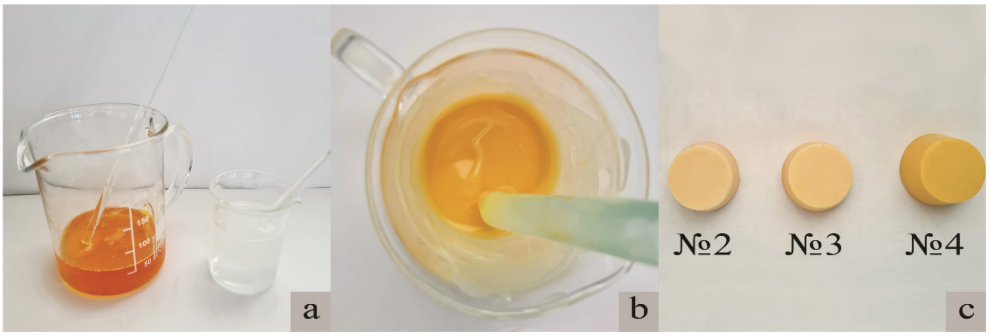


**Fig. 3.** Preparation of soaps with HPO: a – oil mixture and alkaline solution; b – thickened soap mass; c – finished soaps – samples № 2, № 3 and № 4.

Over time, moisture levels decrease notably due to the evaporation of free water in the soap, resulting in a reduction in weight [34-35].

The highest FA content is observed in the control sample № 1 – 52.37 %, and in the other three samples it is lower. The values TFM of samples № 2, № 3 and № 4 have insignificant differences, respectively – 50.60; 50.10 and 49.82 %. They are close to the values of soap with shea butter – 58.00 % [38] and higher than those of soap with onion seed oil – 36.66 % [39]. The values of TFM are influenced by the soap preparation method and are corroborated by literature findings [37].

**Table 4.** Results of the analyzed soap samples

| Samples (% HPO) | Moisture content (%) | Total fatty matter content (%) | Foaming ability ($cm^3$ after 30 s) | pH | Content of unsaponified fats and unsaponifiable substances (%) | Free alkali (%) |
|---|---|---|---|---|---|---|
| Sample № 1 0 % | 30.87 ± 0.06[d] | 52.37 ± 0.21 | 230.00 ± 5.00 | 10.30 ± 0.10 | 3.27 ± 0.06 | 0.003 ± 0.006 |
| Sample № 2 5 % | 32.30 ± 0.36 | 50.60 ± 0.35 | 120.70 ± 4.70 | 10.20 ± 0.06 | 4.20 ± 0.05 | 0.03 ± 0.002 |
| Sample № 3 10 % | 33.05 ± 0.22 | 50.10 ± 0.31 | 100.30 ± 4.60 | 10.20 ± 0.06 | 4.50 ± 0.06 | 0.04 ± 0.001 |
| Sample № 4 20 % | 33.50 ± 0.16 | 49.82 ± 0.34 | 70.60 ± 3.20 | 10.20 ± 0.07 | 5.10 ± 0.07 | 0.04 ± 0.003 |

[b.] The table shows the mean values ± standard deviations of the data from the measurements of the given indicators.

Regarding the foaming ability, the best results are for the control sample № 1 – 230.00 $cm^3$. As the content of HPO in the formulation increases, the foaming ability of soap samples № 2, № 3 and № 4 decreases linearly, respectively – 120.70; 100.30 and 70.60 $cm^3$. For the four soap samples, the pH is in the range of 10.20-10.30. There were no statistically significant differences in the pH values. Close to the pH data are those of the soaps obtained by the cold method and analyzed by [40-42], respectively 9.53-9.96, 10.01-10.57 and 10.40.

Control sample №1 has the lowest content of unsaponifiable matter and unsaponified fat (3.27 %), while in samples № 2, № 3 and № 4 their amount is significantly higher (4.20, 4.50 and 5.10 %). Close to these values for unsaponifiable matter (3.375-5.897 %) were reported by [43] for soaps produced by the cold method and containing algae extract. In other soaps studied, a high percentage of unsaponifiable matter was also determined, from 0.96 to 9.86 % respectively [44].

The higher HPO content is responsible for the presence of more unsaponifiable matter in the finished soaps. These components, in turn, have a negative impact on the foaming ability of soaps.

The results obtained indicate that the soaps adhere to the standard specifications concerning the free alkali content [45, 46], with levels in the soap being minimal, below 0.05 %. Lower values reflect higher quality in soap. NaOH can almost completely react with PO and CO, as well as with HPO, since no free alkalis above the norms are found.

The sample soaps have a different color, which is evident from Figure 3. The deep orange color is due to HPO, the higher its percentage in the formulation, the more intense the color of the resulting soaps.

## 4 Conclusion

The study suggests that hot pepper seeds oil holds promise for incorporation into soap production using the cold process method. Samples containing up to 10 % hot pepper seeds oil exhibit satisfactory values across investigated parameters that determine soap quality, including total fatty matter content, free alkali content, foaming ability, pH, color and content of unsaponified fats and unsaponifiable substances. This indicates the feasibility of producing high-quality soap with the inclusion of hot pepper seed oil, up to 10 % of the oil composition, while maintaining a balanced ratio between unsaturated and saturated fatty acids in the oil blend.

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