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PHYSICO-CHEMICAL CHARACTERISTICS OF SOME ECOLOGICAL PRODUCTS USED FOR TEXTILE CARE

BY

VALENTINA GABI MIRON (STĂNESCU)[1], NIKI VLAD MANCAȘI[1], VASILICA POPESCU[1,*] and ANDREI POPESCU[1,2]

[1]"Gheorghe Asachi" Technical University of Iași,
Faculty of Industrial Design and Business Management, Iași, Romania
[2]"Gheorghe Asachi" Technical University of Iași,
Faculty of Mechanical Engineering, Iași, Romania

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Abstract. Protecting the environment is an international issue that depends a lot on the involvement of each of us through actions to collect and recycle different used products. The purpose of this paper is to show that used cooking oils should be collected to be transformed into ecological laundry care products: soaps. The laundry soaps prepared by us were obtained through hot saponification processes, using the mixture of palm and coconut oils, unused and used/waste, in all 4 possible combinations. The characterization of the vegetable oils was carried out by determining the saponification value (SAP) and the iodine number (IN). The obtained soaps were characterized physically and chemically by determining pH, foam, color, density, cleaning/washing capacity inclusive the SAP, IN, INS (Iodine Number Saponification) values for oils mixture from soaps. The tested ecological soaps are excellent products for washing clothes because each has a low foaming and an excellent cleaning capacity.

Keywords: laundry soaps, palm oil, coconut oil, recycling, cleaning capacity.

[*]Corresponding author; *e-mail*: vasilica.popescu@academic.tuiasi.ro

1. Introduction

Saponification is the process of converting fats, oil, and lipid into soap using an aqueous alkali (Alum, 2024).

Soap and glycerine are the reaction products obtained at the end of the saponification process, which can be carried out at room temperature or at a maximum of 100°C. In the saponification reaction, the reactants are NaOH, H_2O and triglycerides, in the form of vegetable oils or animal fats. The steps in formulating a soap depend on the temperature during the saponification process:

-*hot saponification:* continuous mixing of the reactants as the temperature increases to 100°C; maintaining 1 h at this temperature; pouring into molds; standing for 2 h to harden the soap; use;

-*cold saponification:* continuous mixing of the reactants at room temperature (or at a maximum of 40°C to melt the animal fats); pouring into molds; standing for 2 h to harden the soap; ripening for one month (to complete saponification); use.

It can be said that soap can be manufactured in a single stage, if the hot saponification process is used, or in two stages (such as saponification initiation and ripening), when saponification is carried out cold.

The fats and oils used in saponification reactions can be virgin or waste. The collection and participation of waste oils in saponification reactions contributes to achieving a positive impact on the environment. Waste oils are highly polluting if disposed of improperly, because they contaminate soil and water. The solution to this problem is recycling because prevents the release of toxic substances into the environment and helps reduce greenhouse gas emissions. Recycling used oils is essential for protecting the environment and producing sustainable items in various industries, including textile care. Waste oils, from industrial and domestic sources, can be processed to create new products, thus minimizing the impact on the environment and contributing to development of the circular economy (Félix *et al*., 2017; Foo *et al*., 2022). Through recycling, waste oils can be transformed into new products, promoting a circular economy where resources are used more efficiently and sustainably, such as: soap, biodegradable detergents, shampoos, thread lubricant (Félix *et al*., 2017; Wolf *et al*., 2001; Giagnorio *et al*. 2017; Azme *et al*., 2023).

Figure 1 shows the stages of making soap from used oils, thus contributing to the development of the circular economy.

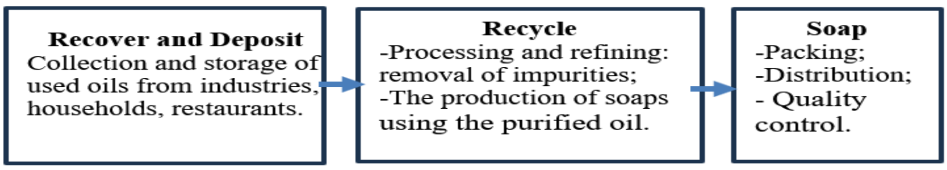

Fig. 1 – The stages of making soap from used oils.

Soap is a means of cleaning the skin and textiles being a fundamental pillar of personal hygiene, providing an effective way to clean the body and clothing. The soap is an alkaline salt of a fatty acid or of a mixture of fatty acids, resulting from the alkaline hydrolysis reaction of fats (Azme *et al*., 2023). The fatty acids have 12 to 18 Carbon atoms in the chemical structure being the main components of some vegetal oils, fat or animal tallows.

In this article, the mixture of 2 oils (one cooking and the other waste) was converted into laundry soap during hot saponification.

Two types of waste oils were used in this work: coconut and palm. In both oils, the percentage of saturated fatty acids is higher than that of unsaturated ones, which gives the soap resistance to oxidation and hydrogenation reactions (Srivastava *et al*., 2018; Gema *et al*., 2022).

The objectives of this work are as follows:

1. Obtaining an ecological laundry soap, based on waste vegetable oils, such as palm and coconut oil;

2. Characterization of the composition and properties of waste vegetable oils, including their saponification (SAP) value, iodine number (IN) as relevant characteristics;

3. Testing and evaluating the effectiveness of the resulting soap in removing stains and cleaning laundry, compared to soap made with only virgin oils, using standardized evaluation methods. The characterization of the obtained soaps was done by determining their density, pH value, color and color difference compared to the soap obtained only from virgin oils. The quality of the soap was confirmed by the values of the indices that characterize the mixture of used oils (SAP and IN for the mixture) but also by the high cleaning capacity of engine oil stains on a white cotton fabric.

2. Materials and methods

Coconut and palm vegetable oils were used as materials for the production of soaps in the following variants:

1. Cooking palm oil;
2. Waste palm oil;
3. Cooking coconut oil;
4. Waste coconut oil.

The 2 cooking oils were manufactured in 2024 and have 2 years warranty for palm oil and 3 years for coconut oil. All the oils used in the formulation of laundry soaps were purchased from the "Atelierul de bere" restaurant in Iasi/Romania. This restaurant bought the vegetable oils from the company Dried Fruits, Bucharest/Romania. The waste oils come from the same lots as the cooking oils, but after 6 hours of use in the kitchen.

All substances (alcoholic KOH, HCl, phenolphthalein, CCl_4, Iodine/KI, $Na_2S_2O_3$ and starch) were purchased from Merck.

2.1. Laundry soaps preparation methods

The hot saponification method was used for the manufacture of soaps no.1-4 from Table 1. Variant 3 of soap was made using the cold saponification method and the analysis of this soap was made after 2.5 months of ripening.

The laundry soaps were made from 2 vegetable oils (palm and coconut) unused/cooking or waste, in all 4 possible combinations. The saponification recipe is included in Table 1.

Table 1
Soap preparation

<table>
<tr><th>Crt. no.</th><th>Saponification method</th><th>The soap component</th><th>Work recipe</th></tr>
<tr><td>1</td><td rowspan="4">Hot saponification
(100°C, 1 h)</td><td>cooking palm oil (PC)
cooking coconut oil (CC)</td><td rowspan="5">40 g Palm oil (83.33%)
8 g Coconut oil (76 degrees type) (16.67%)
7.14 g NaOH
18.24 g H_2O</td></tr>
<tr><td>2</td><td>cooking palm oil (PC)
waste coconut oil (CW)</td></tr>
<tr><td>3</td><td>waste palm oil (PW)
cooking coconut oil (CC)</td></tr>
<tr><td>4</td><td>waste palm oil (PW)
waste coconut oil (CW)</td></tr>
<tr><td>5</td><td>Cold saponification
(room temperature and 2.5 months ripening)</td><td>waste palm oil (PW)
cooking coconut oil (CC)</td></tr>
</table>

2.2. Characterization methods

The methods used for characterization aimed at two directions: A) characterization of the oils; B) characterization of the soaps.

2.2.1. Oils Characterisation

1. Determination of the saponification value (SAP) of palm and coconut oils

SAP values are determined for both a 1 g oil sample (palm or coconut) and a blank (no oil).

The method consists in adding a mass of 1 g of oil in an Erlenmeyer glass over which 25 mL of alcoholic KOH solution of 1N concentration is added. A refrigerant/condenser is attached. The glass is placed in the water bath, where it is boiled for 1 hour. Finally, remove the condenser, cool the beaker and then titrate the contents (a liquid soap) with 0.5 M HCl solution, in the presence of 0.5 mL of 1% alcoholic phenolphthalein (Odoom *et al*., 2015; Rambabu *et al*., 2020).

The SAP index is calculated using the equation 1.

$$SAP = \frac{(V_B - V_S)\cdot N_{HCl}\cdot 56.1}{W} \text{ [mg KOH/ 1 g oil]} \quad (1)$$

where: V_B and V_S are the volume 0.5 N HCl used in the titration; $V_B= V_{Blank}$ and $V_S=V_{Sample}$; N= normality of the HCl solution (0.5); 56.1= molecular mass of KOH used for oils saponification (g); W= mass of oil sample (g).

2. Iodine number (IN)

Weigh 0.3 g of the oil into a beaker and add 20 mL of CCl_4. Mix well then add 25 mL of Iodine/KI solution. The beaker is incubated in the dark for 30 minutes at room temperature.

Then add 200 mL of distilled water and after vigorous stirring start the titration with 0.1 N sodium thiosulphate solution ($Na_2S_2O_3$).

The titration is carried out until the yellow-brown color disappears. A violet coloration appears due to the formation of iodine during the titration reaction. To confirm the presence of iodine, add 0.5 mL of soluble starch solution (concentration 1%), which causes a blue color to appear. Using the same 0.1 N $Na_2S_2O_3$ solution, titrate until the blue color disappears and the solution in the beaker becomes colorless. The volume of thiosulphate used in the titration is read on the burette.

The IN index is calculated according literature (Chebet *et al*., 2016), with the equation 2.

$$IN= \frac{(V_B - V_S)\cdot N_{tiosulphate}\cdot 12.69}{W} \textit{ [mg iodine/ 1 g oil]} \quad (2)$$

3. SAP and IN values for the oil mixture

As any soap is based on mixtures of oils, it follows that the quality of a soap is also indicated by the SAP mixture and IN mixture values. The SAP and IN value obtained for a mixture of palm oil and coconut oil takes into account the combination percentages in the process of obtaining the soap. Thus, for a soap in which palm oil participated in a proportion of 83.33%, and coconut oil 16.67%, the $SAP_{mixture}$ and $IN_{mixture}$ values were calculated according to equations 3 and 4.

$$SAP_{mixture}=(83.33/100)\cdot SAP_{palm\ oil}+(16.67/100)\cdot SAP_{coconut\ oil} \text{ [mg KOH/1g oil]} \quad (3)$$

$$IN_{mixture}= 83.33/100\cdot IN_{palm\ oil}+16.67/100\cdot IN_{coconut\ oil} \text{ [g iodine/ 100 g oil]} \quad (4)$$

Another index called INS *(Iodine Number Saponification)* takes into account the SAP and IN values of each oil in the blend, according to equation 5.

$$INS = SAP_{mixture} - IN_{mixture} \quad (5)$$

2.2.2. Soaps characterization

1. The pH of the soap

To determine the pH of the soap, a solution of 50 mL of distilled water and 1 g of soap was made. It was mixed until dissolved. After a standing time of 30 minutes, the pH was measured with a pH meter.

2. The foaming capacity of the soap

A volume of 100 mL of a 1 g/L soap solution was placed in a 500 cm^3 graduated cylinder. It was shaken vigorously for 10 seconds and then allowed to stand for 10 minutes. The foam height was measured and noted.

3. The density of the soap

The soap density was determined by making the ratio between the mass of the soap (g) and its volume (cm^3).

4. CIELa*b* and delta CIELa*b* measurements of the soaps

A Datacolor portable spectrophotometer was used to determine the L* (lightness), a*, and b* values (the color positions of the soap on the **a** and **b** axes of the CIELab color chart), and their delta counterparts (delta L*, delta a* and delta b*). The delta CIEL* a* b* measurements were determined in comparison to soap made with only cooking/unused oils (PC and CC).

5. Cleaning capacity (washing)

The cleaning capacity was determined as follows: a 10x10 cm cotton sample is stained with very old, dark engine oil, and the stained area is marked with colored thread. The brightness of the cotton sample is measured both before and after soiling. The dirty samples are washed at 100ºC for 30 minutes, using 1g of soap each time (from the 5 types of soaps made from cooking/used palm and coconut oils) and 100 mL of distilled water; L* is measured on the washed and dried sample.

The dirt visibility after washing (H_s) is calculated with equation 6:

$$H_s = (L^*_{before\ soiling} - L^*_{after\ washing}) / (L^*_{after\ washing}) \cdot 100 \ \ (\%) \qquad (6)$$

3. Results and discussion

1. Characterization of oils: SAP and IN

The SAP results obtained for the oils are highlighted in the Table 2.

Table 2

Results obtained for oils: saponification value (SAP)

Oil name (abbreviation)	SAP	
	(mg KOH/ 1 g oil)	(mg NaOH/1 g oil)
cooking palm oil (PC)	*192.50*	*137.25*

waste palm oil (PW)	*181.66*	*129.52*
cooking coconut oil (CC)	*256.23*	*182.69*
waste coconut oil (CW)	*243.80*	*173.83*

The literature specifies the following values for SAP:

-*when SAP is expressed in mg KOH/1 g oil:* for cooking palm oil 199 (http://www.soapcalc.net/calc/OilList.asp) and 191.324 (Abdulkadir and Jimoh, 2013); for cooking coconut oil, the SAP value is 257 (http://www.soapcalc.net/calc/OilList.asp);

- *when SAP is expressed in mg NOH/1 g oil:* 142 for cooking palm oil and 183 for cooking coconut oil (http://www.soapcalc.net/calc/OilList.asp).

From the analysis of these data, it results that the cooking oils used in this article have SAP values close to those in the literature, namely 192.5 for palm oil and 256.23 mg KOH/ 1 g oil. For waste palm and coconut oils there are no indications in literature, regarding the SAP values recorded after 6 hours of use in the kitchen.

Table 2 shows the values SAP for waste oils: 181.66 mg KOH/1g palm oil and 243.88 mg KOH/1g coconut oil. This fact confirms the power/high capacity of involvement in the saponification operation.

The IN results obtained for the oils are highlighted in the Table 3. The IN values of the oils used have values between 4.23 and 48.521(g iodine/100 g oil) which means that these oils contain fewer double bonds than other oils, therefore reducing the risk of browning due to oxidation.

Soaps formulated with these oils achieved higher hardness.

For palm oil, the IN values in Table 3 are in the range of the standard (45-55 g iodine/100 g palm oil), indicating the presence of a normal level of unsaturated fatty acids in triglycerides (Abdulkadir and Jimoh, 2013). For coconut oil (76 degrees type), the IN value of the standard was slightly exceeded, which normally ranges between 6-11 g iodine/100 g oil.

The data from literature indicate IN values of 53 (http://www.soapcalc.net/calc/OilList.asp) and 53.87 g iodine/100 g palm oil (Abdulkadir and Jimoh, 2013); for coconut oil 76 degrees, IN takes values of 10 (http://www.soapcalc.net/calc/OilList.asp) or 6.3-10.6 g iodine/100 g oil (Odoom and Edusei, 2015).

For palm waste, the literature indicates a decrease in the IN value to 45.58 mg KOH/1 g oil, after 6 hours of use in frying various foods (Chebet *et al*., 2016). For used coconut oils, there are no indications regarding the IN value recorded after 6 hours of use in the kitchen.

Table 3

Results obtained for oils: IN

Oil name (abbreviation)	IN		
	(mg iodine/1g oil)	(g iodine/ 1g oil)	(g iodine/100 g oil)
cooking palm oil (PC)	*485.21*	*0.48521*	*48.521*
waste palm oil (PW)	*465.30*	*0.46530*	*46.530*
cooking coconut oil (CC)	*115.36*	*0.11536*	*11.536*
waste coconut oil (CW)	*42.30*	*0.04230*	*4.230*

2. Characterization of oils mixture

The characterization of the oils mixture was carried out by determining the indices: $SAP_{mixture}$, $IN_{mixture}$ and $INS_{mixture}$. The values obtained for these indices are included in Table 4.

Table 4

Indices that highlight the quality of the oils used for manufacturing the laundry soaps

Oil name	Oil percentage (%)	$SAP_{mixture}$ [1] (mg KOH/1g oil)	$IN_{mixture}$ [2] (g iodine/100g oil)	$INS_{mixture}$ [3]	Verification $INS_{mixture.}$ [4]
PC	*83.33*	*203.12379*	*42.35560*	*160.7681*	*160.76819*
CC	*16.67*				
PC	*83.33*	*201.05171*	*41.13769*	*159.9140*	*159.91401*
CW	*16.67*				
PW	*83.33*	*194.09081*	*40.69650*	*153.3943*	*153.39431*
CC	*16.67*				
PW	*83.33*	*192.01873*	*39.47859*	*152.5401*	*152.54014*
CW	*16.67*				
PW	*83.33*	*this soap was made by cold saponification method, as early as 2.5 months ago*			
CC	*16.67*				

[1]$SAP_{mixture}$= Σ [(oil percentage/100)·SAP]; (mg KOH/ 1 g oil);
[2]$IN_{mixture}$ = Σ [(oil percentage /100)·IN]; (g iod/100 g oil);
[3]$INS_{mixture}$ = $SAP_{mixture}$ – $IN_{mixture}$;
[4]$INS_{mixture}$ = Σ (oil percentage ·INS).

It is observed that the $IN_{mixture}$ are lower than 42.35 g iodine/100g oil, and the $INS_{mixture}$ varies between 152.5 and 160.77, which indicates that the soaps made from a mixture of cooking oil (unused) and waste oil behave very similar to those obtained only from unused/cooking oils.

3. Soaps characterization

The physical characteristics of the 5 laundry soaps made are indicated in Table 5.

Table 5
Assessment of the quality of laundry soaps: Physical characteristics

Saponification method	Soap code	Density (g/cm^3)	pH	Foam (cm)
Hot saponification	*PC+CC*	*1.326*	*9.78*	*10*
	PC+CW	*1.119*	*9.89*	*9.5*
	PW+CC	*1.164*	*9.81*	*10.5*
	PW+CW	*1.315*	*10*	*9.5*
Cold saponification	*PW+CC*	*0.847*	*9.40*	*8.5*

The results in Table 5 indicate the following variations: density between 0.847 and 1.32 g/cm^3, pH varies from 9.4 to 10 and foam height varies from 8.5 to 10.5 cm.

In the recipe for obtaining our laundry soaps, palm oil predominates (83.33%). No other researcher has used the combination of the 2 oils (palm and coconut 76 degrees) in the unused/used forms in the manufacture of laundry soaps. For this reason, the comparison of the characteristics of our laundry soaps can only be done by referring to the data in the specialized literature, relating to soaps that have palm oil as a predominant component. Thus, soaps with Kernel palm oil generate a pH of 9.88-10 upon dissolution, and a strong foaming capacity (Muhammed *et al*., 2022). Exceeding the pH value of 10 leads to a decrease in the stain removal capacity (Nchimbi, 2020).

In our soaps, the pH takes values of 9.78-10 when the saponification process took place hot and 9.40 when the saponification was done cold. The foaming capacity is moderate (the height of the foam in the graduated cylinder does not exceed 10.5 cm), which indicates a good laundry soap, and can be used without problems in the household washing machine.

Regarding the density, the lower value of our cold-made soap can be explained by the fact that during the ripening (of one month) water is eliminated, which causes a decrease in the weight per unit volume of this soap.

The color of the laundry soaps is indicated by the brightness L*=90.14-91.93 for those obtained by hot saponification and L*=45.73 for the soap made by cold saponification and matured for 2.5 months at room temperature (laboratory).The yellowish tinge indicated by the size b* is weak, because b*=8.89 and 9.79 for the hot-made soaps and b*=14.08 for the cold-made and matured for 2.5 months (Table 6).

Table 6
*Color evaluation of soaps by CIELa*b*and delta CIELa*b* measurements*

Saponification method	Soap code	CIEL*a*b*			Delta CIEL*a*b* (wih PC+CC as standard)		
		L*	a*	b*	ΔL*	Δa*	Δb*
	PC+CC	*90.14*	*-1.28*	*9.64*	*0*	*0*	*0*

Hot saponification	*PC+CW*	*91.93*	*-0.51*	*9.79*	*1.78*	*0.76*	*0.16*
	PW+CC	*90.88*	*-1.35*	*9.04*	*0.73*	*-0.07*	*-0.6*
	PW+CW	*90.3*	*-1.4*	*8.89*	*0.16*	*-0.13*	*-0.75*
Cold saponification	*PW+CC*	*45.73*	*-1.5*	*14.08*	-	-	-

4. The cleaning power of soap

To indicate the cleaning capacity of soaps, some researchers (Azme *et al*., 2023; Cheng *et al*., 2024) made subjective assessments (giving grades from 1 to 5 or ratings such as removes well/does not remove the stain) and indicated a good ability to remove stains caused by soy sauce and lipstick but the inability to remove oil stains.

The cleaning power of all the soaps tested in this work is very high because after washing off the extremely used (black) engine oil dirt, the stained spot has disappeared and the visibility of the dirt is imperceptible, the visibility of the dirt being detected only by a spectrophotometer Data Color. According to the calculation with relation (6) the dirt visibility takes values of 1.19% in the case of PW+CC oil laundry soap and 5.67% when the soap is based on PC+CW mixture (Table 7).

Tabel 7

The cleaning power of dirt stains on textiles

Saponification method	Soap code	$L^*_{\text{before soiling}}$ (%)	$L^*_{\text{after washing}}$ (%)	Visibility of dirt after washing (%)
Hot saponification	*PC+CC*	*95.21*	*92.69*	*2.72*
	PC+CW	*96.33*	*91.16*	*5.67*
	PW+CC	*95.47*	*94.35*	*1.19*
	PW+CW	*95.68*	*93.49*	*2.34*
Cold saponification	*PW+CC*	*96.55*	*93.84*	*2.89*

4. Conclusions

By using 2 vegetable oils that have synergistic effects, excellent results are obtained with an optimized recipe, obtained after many experimental attempts to formulate the soap. Palm oil (83.33%) gives an excellent bar of soap (cleansing, moisturizing and smell) and coconut oil (16.67%) increases cleansing, foaming capacity and confers good antibacterial and antimicrobial properties.

The obtained results in this work confirm the following aspects: saponification values of oil mixtures in laundry soaps are 192-203 mg KOH/1 g oi land the iodine number of the oil mixture used in laundry soaps is low (IN<43), which indicates that our soaps are characterized by rigidity, a very important aspect for any laundry soap.

The resulting soaps have INS <160, which indicates that they are harsh on the skin but excellently clean the dirt from the laundry.

The prepared soaps are hard in consistency (density 0.9-1.32 g/cm^3), light in color and can successfully replace laundry soaps obtained from unused fats.

The studied soaps have good capacities for cleaning oily dirt, which other soaps fail to do; they have an alkaline pH of 9-10, a moderate foaming power (7-10 cm), which is why they can be used as laundry soaps, even in automatic washing machines.

The soaps tested have high washing capacities because very low values were obtained for the visibility of dirt, after washing the samples stained with engine oil (H_s= 1.19-5.67%).

Tested ecological soaps can successfully replace soaps made from unused oils, having extremely close characteristics.

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CARACTERISTICI FIZICO-CHIMICE ALE UNOR PRODUSE ECOLOGICE UTILIZATE PENTRU ÎNGRIJIREA TEXTILELOR

(Rezumat)

Protejarea mediului este o problemă internațională care depinde foarte mult de implicarea fiecăruia dintre noi prin acțiuni de colectare și reutilizare a diferitelor produse uzate. Scopul acestei lucrări este de a arăta că uleiurile de gătit uzate trebuie colectate pentru a fi transformate în produse ecologice de îngrijire a rufelor: săpunuri. Săpunurile de rufe preparate de noi au fost obținute prin procese de saponificare la cald, folosind amestecul de uleiuri de palmier și cocos, nefolosit și uzat/deșeuri, în toate cele 4 combinații posibile. Caracterizarea uleiurilor vegetale a fost efectuată prin determinarea valorii de saponificare (SAP) și a numărului de iod (IN). Săpunurile obținute au fost caracterizate fizic și chimic prin determinarea pH-ului, spumei, culorii, densității, capacității de curățare/spălare inclusiv a valorilor SAP, IN, INS (Iodine Number Saponification) aferente amestecului de uleiuri din săpunuri. Săpunurile ecologice testate sunt produse excelente pentru spălarea rufelor deoarece fiecare are o spumare redusă și o capacitate excelentă de curățare.