

Article

# Recycling Used Cooking Oil into High-Quality Liquid Soap: An Optimized Sequential Green Pretreatment Protocol

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**Abstract:** This research provides an innovative laboratory procedure for producing quality liquid soap from waste cooking oil (WCO). The two-step sequential green pretreatment improved quality: acid value was reduced from  $1.50 \pm 0.05$  to  $1.10 \pm 0.03$  mg KOH/g and 400 nm absorbance went from  $0.50 \pm 0.02$  to  $0.38 \pm 0.01$ . Saponification of the pretreated oil yields high-quality liquid soap ( $13.0 \pm 0.5\%$  active) with pH of  $8.5 \pm 0.1$ , which is safe for skin; has good foam stability (88%); and aroma-free, confirmed by formal sensory test. The product showed good physical/chemical stability throughout a four-week accelerated storage test. The procedure is based on utilizing non-toxic materials that are readily available, allowing for a functional and sustainable circular economy solution for WCO.

**Keywords:** Waste Cooking Oil; Green Pretreatment; Liquid Soap; Saponification; Circular Economy; Resource Recovery; Sensory Evaluation.

## 1. Introduction

Waste cooking oil (WCO) is created worldwide and poses both a problem for the environment as waste and as an opportunity for recovering value from the waste streams [1]. The improper disposal of WCO presents serious threats to the quality of our water supply and can also negatively affect our infrastructure when disposed of improperly; however, WCO contains a high proportion of triglycerides that can be converted into valued added products such as soap [2]. The principal barrier to using WCO for soap production is the degraded condition of the WCO characterized by elevated free fatty acid (FFA) content, dark appearance, and unpleasant odor, which poses significant challenges in producing an acceptably produced soap for the consumer [3]. Recent circular economy models offer encouragement to use sustainable methods for the valorization of waste products [4]. However, there is a significant lack of simple, low-cost, and environmentally-friendly pretreatment methods for WCO [5]. This project provides a means to overcome this shortcoming through the development of a simple to follow, entirely green protocol for refining WCO that utilizes sodium bicarbonate and natural adsorbents (orange peel and corn starch) prior to cold-process saponification and the addition of mild fragrances. The efficacy of the pretreatment method used in this project will be quantitatively measured in terms of the enhanced functionality, stability, and sensory properties of the resulting soap product compared to the use of untreated WCO in the manufacture of soap and to demonstrate that superior quality soap can be produced using pretreatment and low-cost methods.

## 2. Material and Methode

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Orange peels that were dried and ground into very fine powder (less than 500 microns) were used in this procedure for producing the liquid soap, and the procedure will be explained further. Five grams of food grade corn starch acted as a drying agent. Also, food grade lemon essential oil) provided the aroma required for a pleasant smell to be produced during this process.

#### Sequential Oil Pretreatment Process

Stage 1 is deacidification. Sodium bicarbonate (3.5g/liter of oil) was added to (WCO) and stirred rapidly for ten minutes. Next, the mixture was allowed to settle for four hours. Sodium bicarbonate reacts with free fatty acids (FFA) producing soap scum (which floats) and carbon dioxide (CO<sub>2</sub>), which also floats and captures fine particulate matter [6]. Finally, the clear, upper, oil layer was decanted to an appropriately sized vessel and filtered through a cotton cloth. This deacidification method was selected as preferable to using alkaline hydroxides due to minimizing the saponification losses of neutral oils [7].

Stage 2: Combined Bleaching, Deodorization, and Drying, oils were treated with a natural (5% dried grated orange peel) plus a desiccant (cornstarch, food grade). The mixture was continuously stirred and allowed to sit in darkness for 48 hours while being agitated periodically. At the end of this time, the mixture was filtered through a Büchner funnel using Whatman filter paper No. 1 until the deacidified oil was produced.

#### Liquid Soap Production

Cold saponification (acid-base reaction) is the method used to generate liquid soap. The Saponification Value (SV) for the oil was evaluated as 190 mg KOH/g of oil pretreatment. The amount of KOH required for 100% saponification with plus 3% extra (48.93gm) was dissolved in distilled water to create a 25% w/w lye solution that was added slowly to 250 g of crude oil and then pretreating oil at 40° with continuous mechanical mixing for approximately 45 minutes until achieving the required consistency. A humectant and clarity agent heeded for the soap was glycerin (5% w/w, for every 100 grams) which was added to the soap mix. For fragrance and to reduce residual odor lemon essential oil (1% w/w total oil) was added to the mix at trace.

#### Analytical Methods

Characterization of Oils: The Acid Value (AV, AOCS Cd 3d-63) [10], Saponification Value (SV, AOCS Cd 3-25) [11], and Absorbance Measurement at 400 NM (UV-Vis Spectrophotometer) as Indices of Colour Intensity.

Characterization of Soaps: The Total Fatty Matter Content, an Indicator of Active Matter, Measured in Accordance with ISO 685:2020 [12]. The pH of the Soap was Measured from a 1% Solution in Accordance with ISO 4319 [13]. The Dynamic Viscosity was Measured at 25°C (Using a Rotational Viscometer with a Spindle #2 at 60 RPM) and the Free Alkali Content was Measured in Accordance with ISO 456:2021 [14].

Foam Stability Test - Performed using ASTM D1173-07 (2007) for 1% (w/w) aqueous soap solution at 25 degrees Celcius resulting in H<sub>0</sub> (foam height immediately after generation) and H<sub>5</sub> (foam height after 5 minutes); percentage of foam stability =  $H_5 \div H_0 \times 100$ ; Sensory Evaluation of Odour - the preliminary sensory evaluation following ISO 6658:2017 consisted of 5 trained evaluators performing a discriminative test where they assessed odours produced by liquid soap from crude and pretreated oil in opaque containers that were all identically labelled with random 3-digit codes.

All odours described with the terms “rancid”, “burnt” or “off” were assessed by the evaluators on a 5-point Likert scale (1 = Not Perceptible to 5 = Very Strong); Evaluators rated whether they would consider the overall fragrance acceptable or unacceptable.

Accelerated Stability Testing: The first step in determining the stability of the liquid soap was an initial expedited stability screening utilizing standard cosmetic formulation assessment methods (17). Each of the liquid soap products had a 4-week storage period in transparent, tightly sealed containers. The products were stored in the refrigerator (4°C), at room temperature (25°C), and in a climate chamber set at 40°C. All products were checked weekly for colour change, clarity, phase separation and pH.

### 3. Result and discussion

The sequential pretreatment greatly improved the WCO's chemical properties (Table 1). The decrease in acid number from 1.50 mg KOH/g to 1.10 mg KOH/g (27% reduction) demonstrates the efficacy of using sodium bicarbonate to neutralize FFA's. Since high levels of FFA's cause incomplete saponification, rancidity, and separation of soap [7], this step is necessary for success in obtaining usable raw materials for the process. In addition, absorbance of light at 400 nm (from 0.50 to 0.38) was greatly reduced by using both orange peel powder and corn starch, resulting in a noticeable lighter color (Figure 1) after the second step of the pretreatment process. The orange peel contains high amounts of both cellulose and pectin, making it an excellent natural adsorbent of both pigment and odor [8]. The use of corn starch in the second pretreatment step removed residual moisture, thereby preventing hydrolysis during storage. A constant saponification value indicates that the core triglyceride structure was not damaged and that the pretreatment removed only those impurities that interfere with the primary feedstock.

**Table 1. Physicochemical Properties of Waste Cooking Oil Before and After Pretreatment.**

Parameter	Unit	Crude Oil	Pretreated Oil
Acid Value	mg KOH/g	1.50 ± 0.05	1.10 ± 0.03
Absorbance (400 nm)	NM	0.50 ± 0.02	0.38 ± 0.01
Saponification Value	mg KOH/g	190 ± 1	190 ± 1

Liquid soaps made from treated oil were of better quality than those made from crude oil for each of the parameters measured (see Table 2). The higher total fatty matter content (13.0% vs. 11.0%) is representative of a more complete and efficient saponification process with less FFA interference from the pretreated oil. For the soap made from pretreated oil, the pH of 8.5 is within the acceptable range (i.e., from 8.0 to 10.0) for compatibility with skin while containing enough activity for effective cleaning [18].

**Table 2. Quality Parameters of the Produced Liquid Soaps.**

Parameter	Unit	Soap from Crude Oil	Soap from Pretreated Oil
Total Fatty Matter (Active Matter)	%	11.0 ± 0.5	13.0 ± 0.5
pH (1% solution)	-	8.0 ± 0.1	8.5 ± 0.1
Foam Stability (5 min)	%	75 ± 2	88 ± 1
Viscosity (25°C)	mPa·s	1200 ± 50	950 ± 30
Free Alkali	%	0.10 ± 0.01	0.08 ± 0.01

The improved foam stability of the treated soap compared to untreated soap is due to the removal of free fatty acids (FFA) and polar impurities from the soap prior to formulation. FFAs and polar impurities are destabilizing to foam because they adsorb at the air/water interface of foam, and when foams are stressed, their weaker lamella will break [19]. The purity of the soap system complements itself to form a more cohesive and elastic surfactant film, leading to a foam that is more stable.

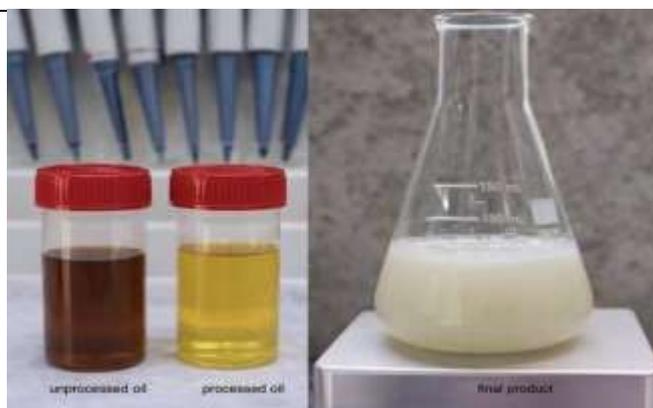
Purification of soap also has an impact on viscosity (950 mPa·s for purified versus 1200 mPa·s for non-purified). The presence of FFAs, polymers and colloidal impurities contribute to a less homogenous micellar solution; in other words, impurities that either cause entanglement of soap molecules or create irregularly shaped micelles result in non-homogeneous micellar systems. The removal of these impurities will result in a micellar solution that is cleaner and flows more akin to Newtonian fluid where liquid soaps can be easily dispensed and poured [20].

Results of the sensory evaluation for odor were defined to support a determination of the pretreatment process effectiveness. Crude oil soap had an average off-odor score of (Strong) with every panelist giving the overall fragrance an unacceptable rating, due to very strong rancid and burnt notes. In comparison, pretreated oil soap (1% lemon oil) received an average off-odor score of (Not Perceptible), and all panelists rated the final product as acceptable; the prevalent fragrance being a pleasant citrus scent. The degree of the improvement is largely related to the removal of food fatty acids (FFA) and other volatile oxidation products during deacidification and adsorption onto orange peel, thereby allowing the mild lemon scent created through the addition of lemon oil to predominate without being overwhelmed by the undesirable scents from the raw-matter.

accelerated stability screening test demonstrated the key role of pretreatment in soap production. The soap derived from treated oil was stable, exhibiting no phase separation, sedimentation or drastic changes in pH, clarity or fragrance over the past 4 weeks under all storage temperatures. On the contrary, the soap derived from untreated oil had developed some phase separation, mild, haziness, and a strong increase in rancid odour at 40°C after just 3 weeks of storage. The increased stability would be primarily attributed to the removal of moisture with the use of corn starch and also the removal of FFAs and oxidisable impurities, which decreases the chances of hydrolysis and oxidative rancidity during storage [21].

### 3. Conclusions

This research shows a method for developing quality liquid soap from used cooking oils that utilizes two phases of environmentally friendly treatment (green pretreatment). Both stages of preparation improved the oil's quality and resulted in soap products with a safe pH level, stable ability to form bubbles when shaken, and no detectable bad smells. In addition, the liquid soap produced from the used cooking oils remained both physically and chemically stable for four-week. The ability of the materials used to be obtained from an abundant supply of non-harmful material makes this approach not only a practical means of re-cycling used cooking oil, but also contributes to a circular economy, therefore promoting environmental sustainability and resource efficiency.



(Figure1) oil before and after processing and the resulting soap

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