Non-traditional Laboratory Experiments: Olive Oil Manufacturing and Testing.

Part I: Freshman Engineering Experiments

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Abstract

Olive oil manufacturing and processing involves the application of many fundamental chemical engineering principles and unit operations. These operations are not, however, traditionally explored in the chemical engineering curriculum. This paper presents the first set of experiments created as part of an NSF funded project whose goal is to incorporate the processes involved with the production and analysis of olive oil into undergraduate chemical engineering laboratories, illustrating the concepts of fluid mechanics, separations, process optimization, chemical analysis, experimental methods, food engineering, and many others.

In this paper, modeling of the filtration pressing of olives and oils properties were explored. Using a standard fluid mechanics model, fitted parameters representing the resistances of the filtering materials were determined to be dependent on applied pressure. The experiment based on the modeling of pressing can be modified, and applied to unit operations, process fluid transport, and advanced separations. The fluid physical properties of olive oil, density and viscosity, were also measured, as a function of temperature. A laboratory for Rowan University's freshman clinic course was developed as an introduction to measurement and laboratory techniques based on these experiments.

Background

Olives crops and the production of olive oils have been very important commercial activities in the Mediterranean countries since biblical times. Today, olive oil accounts for 3% of the total essential oil consumption, and 15% of the total world oil market. It is a staple component of the "Mediterranean Diet," revered by the rest of the world for its positive effects on the health of the region's residents. Ongoing research is currently focusing on the examination of the potential benefits of olive oil in the diet. This research includes examining its antioxidant properties, and its role in the prevention of numerous diseases such as cardiovascular diseases, cancer, arteriosclerosis and diabetes[1].

Figure 1: Folding mats with olive oil paste in between

Several primary steps are involved in the industrial olive oil pressing process. The first of these is grinding. The purpose of this step is to break open the oil-containing cells within the olive to ensure high yields. To further maximize oil yields, a mixing step is employed. Slow, extended mixing of the olive paste breaks up the oil-water emulsion and allows the small oil droplets to coalesce into larger ones[2]. The next step is primary solid-liquid separation. This is usually accomplished by pressing, the traditional olive oil separation method. The paste is loaded onto mats, and stacked between two plates or discs as shown in Figure 1.

Olive Oil Classification	% Oleic Acid
Extra Virgin	<1
Virgin	<2
Ordinary Virgin	<3.3
Lampante Virgin	>3.3
Refined	<0.3

Table 1: Olive Oil Classification

Pressure is applied mechanically in order to drive the oil-water mixture out of the paste and through the mats, which act as filters. The oil is then separated from the mixture either through decantation or centrifugation[1].

In addition to extraction of olive oil, quality control also plays an important role in the production of olive oil. Olive oil is classified based on the concentration of oleic acid, as shown in Table 1. Virgin olive oil is defined as oil that has been extracted through purely mechanical means, excluding processes using chemicals or thermal treatments that may affect the structure or composition of the oil[3]. If the concentrations of acid or volatiles, such as polyphenols, are too high, then the extracted oil must undergo a refining and deodorization step, as these components contribute to a bitter and rancid flavor (cite original proposal).

Theory

Filter pressing can be thought of in terms of a traditional fluid mechanics filtration operation[1, 4, 5], in which a fluid slurry is forced to flow through a filter by a pressure gradient. Suspended solids in the mixture above a certain size are deposited on the filter, and the fluid is allowed to pass through. In the case of pressing, the solid/liquid mixture is loaded onto the filter at the beginning of the process, resulting in an unsteady state process, and the pressure gradient is provided by a hydraulic press or crank. To represent the behavior of the filtration system, a modified form of the Hagan-Poiseuille, shown in Equation 1, was used[4,5],

$$V(t)=\frac{\beta A}{\alpha\omega}\left(1-e^{\frac{P\alpha\omega}{\eta\beta^2}t}\right) \quad (1)$$

where, t is time, P is pressure applied by press, V is volume of liquid extracted, A is the filter area, η is the viscosity of the liquid, β is the resistance of the deposited solid material, α is the resistance of the filter and ω is the amount of dry solids deposited per unit of extracted liquid. Using mathematical software and Equation 1, the extraction of olive oil over different loads or pressure (P) can be modeled, and α and β determined. The resistance of the cake, β, is not constant with pressure, and can be represented by Equation 2,

$$\beta = \beta' P^s \quad (2)$$

where β' is a constant and s is related to the compressibility of the cake (4, 5).

Experimental Setup

Pressing

For the pressing operation, the FirstPress® filter press obtained from The Olive Oil Source, Greenbrae, CA, shown in Figure 2, was used. The unit was characterized by simple construction, consisting of a stack of slotted plates and nylon filter cloths, which were pressed between two steel plates by a 40,000lb hydraulic jack. The liquid from the pressing flows out of the plates into a catch tray, and out a drain hole in the rear of the unit. Also obtained from The Olive Oil Source were a hammer mill and a mixer, shown in Figure , for the grinding and mixing operations.

Figure 2: Unmodified Pressing Unit

Instrumentation

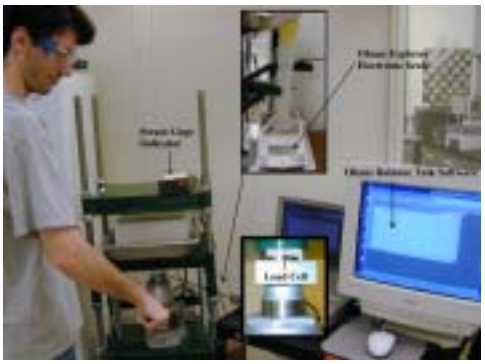

Figure 3: Modification and instrumentation of the pressing unit

Several modifications were required in order to collect the data necessary for the modeling of the oil extraction process. A load cell and strain indicator, obtained from Omega Instruments, shown in Figure 3, were placed between the jack and lower press plate to measure the force applied by the press. This can be divided by filter area to obtain pressure, which is required for the model. A scale and large graduated cylinder, shown in Figure 3, were placed beneath the drain hole in the catch tray to collect the extracted liquid. The scale, obtained from Ohaus, was connected to a computer with data logging software, obtained from Ohaus, to read the amount of oil collected with time. All other model parameters could be measured before or after the pressing, or determined from the experimental data.

Fluid Property Measurement

The Canon-Fenske Routine Viscometer (Figure 4) was acquired from Fisher Scientific to measure the viscosity of the oil extracted from the pressing unit. This equipment is for use only with Newtonian fluids and within a certain range of kinematic viscosity. Three viscometers with three different kinematic viscosity ranges were purchased. Because this viscometer requires a constant temperature, an acrylic casing was built to fit the viscometer inside of the water bath.

Figure 4: Cannon-Fenske routine viscometer in an acrylic casing.

Materials and Methods

Olives for the pressings were obtained from The Olive Oil Source. Because they were purchased early in the olive season, before the crop was ripe, low oil yield is expected.

First, the olives must be made into a uniform paste by grinding. The olives are washed to remove dirt, stones, etc to avoid potential damage to the equipment. They are then grinded in the hammer mill to break up the skins and produce a uniform paste for pressing. The resulting olive paste is then placed in a mixer, shown in Figure 2, for one hour. This helps the oil droplets coalesce to increase the yield of extraction. The paste is then loaded onto the mats and plates. 347g of paste were loaded onto one layer. The plates were then placed with the collection tray into the press, and the hydraulic jack was used to set the operating pressure. Because of the compression of the cake during the operation, the jack must be continuously pumped to keep the constant pressure required for use of the model. Oil from the extracted liquid was separated by centrifugation. The physical properties of the extracted oil were examined using the Cannon-Fenske viscometer for viscosity and a small volumetric flask for density. A freshman laboratory experiment exploring the analysis of olive oil fluid properties is enclosed in the appendix.

Results

Figure 5 illustrates the results obtained from the extraction of oil/water mixture from olive paste at different loads. These results show that as the press pressure increases, the rate of extraction also increases, but not in proportion to the applied pressure. This is in agreement with the suggestion that the resistance of the paste increases with pressure.

α (Resistance of the filter medium) were determined by fitting Equation **Error! Reference source not found.** to the data using nonlinear regression software. The modeling results are shown in Figure 6 and Figure 7. It is seen in Figure 7 that both resistance terms of the model increase significantly with increasing pressure. A study of lower pressures will give a more complete view of the relationship between applied pressure and the model resistance terms, and enable a determination of β'.

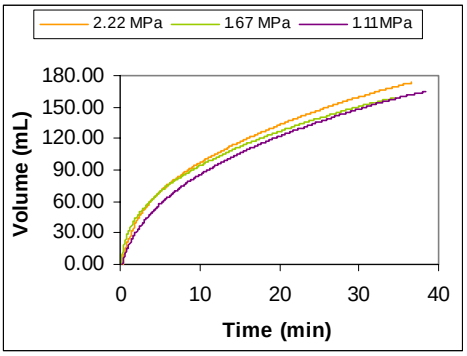

Figure 5: Olive oil extraction at three loads

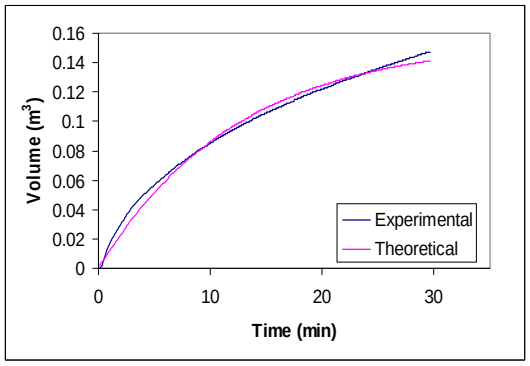

Figure 6: Modeling results of olive pressing 1.67 MPa

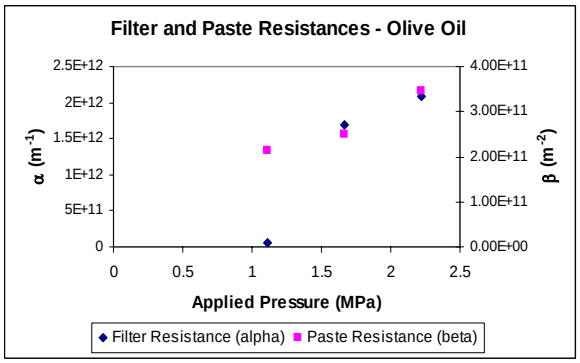

Figure 7: α and β from three different trials.

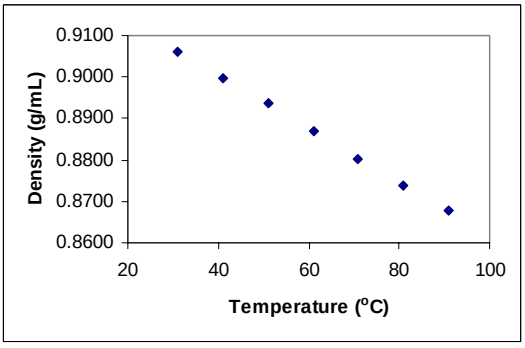

Figure 8: Olive oil density as a function of temperature

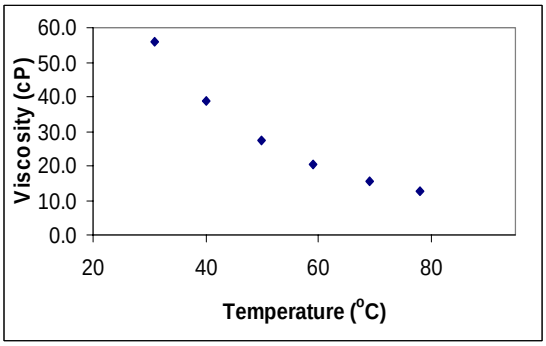

Figure 9: Olive oil viscosity as a function of temperature.

The density and viscosity of olive oil as a function of temperature are shown in Figure 8 and Figure 9, respectively. These properties were determined using the procedure outlined in the freshman clinic laboratory experiment enclosed in the appendix.

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Biographical Information

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Mariano J. Savelski received a Chemical Engineering degree from the University of Buenos Aires, Argentina in 1991, a M.E. from the University of Tulsa in 1994 and a Ph.D. from the University of Oklahoma in 1999. Dr. Savelski has seven years of industrial experience working first as process engineer for SADE Skanka and later on as quality and technology engineer for Kellogg. He joined the Chemical Engineering Department at Rowan University as Assistant Professor in 1999.

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(1964) Grasas Aceites 15, 299-307

Appendix – Freshman Clinic Laboratory Handout

Chemical Engineering Department – Rowan University

Analysis of Olive Oil Physical Properties

Objectives

In this experiment two physical properties, density and viscosity of olive oil, will be analyzed. The results will be helpful to understand how these properties behave at different temperatures. Also, light absorption will be used to determine oil concentrations.

Introduction

Two of the most known fluid properties by Chemical Engineers are density and viscosity. When dealing with pipes, turbines, pumps, reactors, and many others, the behavior of a fluid (gas or liquid) is a major concern.

Density (ρ) is a measure of how much mass is contained in a given unit volume, or in other words, density is a measure of how tightly a mass is packed together. It is defined as:

$$\rho = \frac{mass}{volume} \text{ (SI: Kg/m}^3\text{, CGS: g/m}^3\text{, BI: lb}_m\text{/ft}^3\text{)} \quad (1)$$

When this mass is heated the collision between molecules increase and more space is necessary to contain the same initial mass. Therefore, the density is usually inversely proportional to the temperature.

Viscosity can be thought as the internal stickiness of a fluid. It is directly linked to the rate of deformation of a fluid[1]. Viscosity is highly dependent on temperature and its relation is often found to approximate:

[1] Mechanics of fluids. 3rd edition. Merle C. Potter, David C. Wiggert.

$$v = A \cdot e^{(B/T)}, \quad B = k / Ev^2 \tag{2}$$

and used over a large temperature range, where v is the kinematic viscosity, k is the Boltzmann's constant, and T the temperature. The constants A and Ev (known as the activation energy for viscous flow) exhibit a large variation between different fluids.

Absorption is physical phenomena related to the absorption of ultraviolet (UV) or visible (Vis.) light by different compounds. The equation that represents the absorption of light in a cuvette of length b is generally the Beer-Lambert Law:

$$A = \varepsilon \cdot b \cdot C \tag{3}$$

Where A is absorbance (Unit less)

ε is the molar absorbtivity with units of L mol^{-1} cm^{-1}

b is the path length of the sample and is usually in cm.

C is the concentration of the sample in moles/L

Absorption can be represented as the difference between the light that is striking a sample and the light that is leaving the sample as shown in Figure 1.

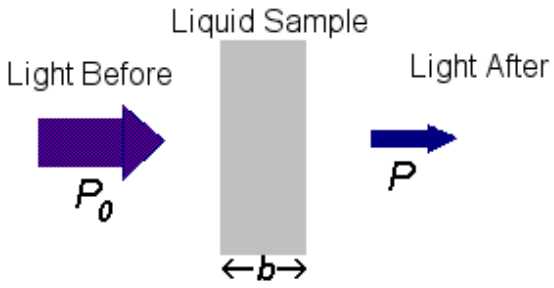


Figure 1. The difference between the "Light Before" and "Light After" is how much light was absorbed by the sample.

Density and Viscosity Experiments:

Materials

The following materials will be provided for the Density and Viscosity experiments:

- Olive Oil
- 25mL volumetric flask (Figure 2)
- Water bath
- Stop watch
- Analytical balance

- 200 Cannon-Fenske Routine Viscometer in a acrylic casing (Figure 3)
- Methanol rinse bottle

Procedure

1- Density:

1.1- Setup a water bath to 30°C as shown in Figure 2.

1.2- Weight an empty 25mL volumetric flask and fill it with olive oil up to the white line.

1.3- Place the glass stopper in the flask and be sure that the hole through the stopper is filled with oil.

1.4- Place the flask in the water bath using the clamps so that the flask neck is outside the water level. Leave the flask inside the water bath for approximately 6 minutes.

Figure 2. 25mL volumetric flask in water bath.

1.5- Clean the top of the stopper before removing the flask from the water bath. Remove the flask and rinse it with methanol. Dry the flask and weight it in the analytical scale placed in the back of the lab.

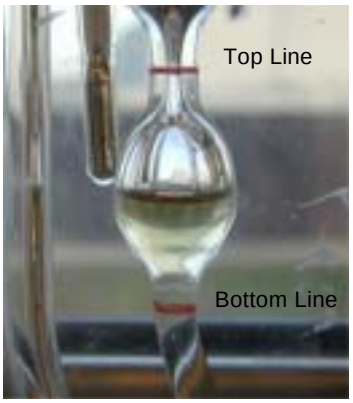


Figure 3. Cannon-Fenske viscometer

1.6- Repeat this procedure for four temperatures and record the results in Table 1.

2- Viscosity:

2.1- Clean the viscometer with methanol and let it dry.

2.2- Pour approximately150mL of olive oil in a clean beaker.

2.3- To charge the sample into the viscometer, invert the instrument and place the thinner tube into the beaker. Apply suction to the opposite end until the fluid slightly passes the bottom line (Figure 3 and Figure 4).

Figure 4. Procedure to charge the viscometer

2.4- Place the viscometer in a water bath at 30°C and connect it to the vacuum line. **Gently** turn on the vacuum until the water reaches the desired level inside the acrylic casing. Turn off the vacuum and wait until the temperature inside the acrylic casing reaches the desired temperature (if the acrylic casing is not perfectly sealed keep the vacuum valve slightly open).

2.5- Evaluate the viscosity at four different temperatures and record the results in Table 1 (Refer to the Appendix for calculation instructions).

Table 1. Density and viscosity of olive oil at different temperatures

Temperature (°C)	Density (g/mL)	Viscosity (cP)
30		
45		
60		
75		

2.6- **Before you leave!!** Write your results in the board and copy the result from other groups, this will be useful to compare the inconsistency of the results.

Proposed Questions

1- **Share your data and gather all the results from other groups.** Set a table in Excel with all the results.
2- Calculate the average, the standard deviation, and the confidence (95%) for the results obtained. Discuss the meaning of this error analysis.
3- Plot Density vs. Temperature. How does the density of olive oil behave with the change of temperature?
4- Plot Viscosity vs. Temperature. How does the Viscosity of olive oil behaves with the change of temperature?

Absorption Experiment:

Materials

The following materials will be provided for the Absorption experiment:

- Two sets of five diluted samples of either Olive Oil, Grapessed Oil, Corn Oil, or Soybean Oil.
- 5 to 10 Methyl Acrylate cuvetts.
- One unknown sample (mixture of two oils).
- 10mL of Cyclohexane.
- Latex gloves.

Procedure

1-1 Wear the provided latex gloves and safety goggles before starting this experiment.

1-2 **Do not touch the transparent sides of the cuvetts!** Hold them from the rough sides and fill them with the given samples (remember to correctly label the samples by their concentration)

1-3 Fill one cuvette with cyclohexane and label it as "B" (Blank).

1-4 Take the samples to the spectrometer.

1-5 Make sure the spectrometer is setup for "Absorption" and that the wavelength is 330nm.

1-6 Test the sample labeled as "B" first. If the reading is different from zero calibrate the spectrometer.

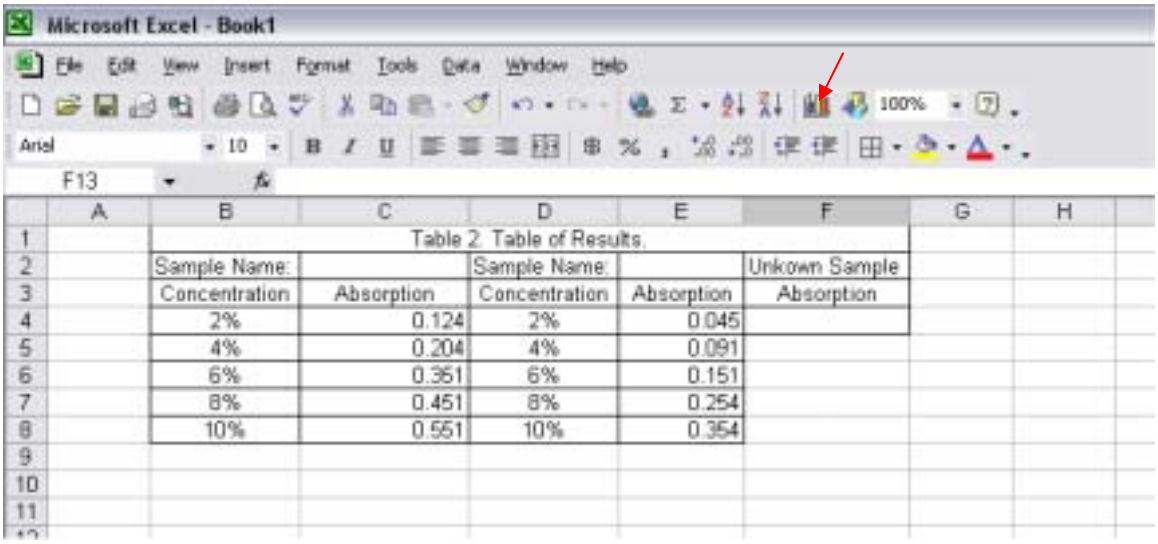
1-7 Test all the samples and write down the results in Table 2.

Table 2. Table of Results.

Sample Name:		Sample Name:		Unknown Sample
Concentration	Absorption	Concentration	Absorption	Absorption
2%		2%		
4%		4%		
6%		6%		
8%		8%		
10%		10%		

Proposed Questions:

1- Plot the Absorbance vs. the concentration (label you graph) for the two sets of samples using Microsoft Excel. For this, enter the concentration in one column and the absorption in the opposite column (See Figure 1). Click on the "Chart Wizard" (red arrow shown in Figure 1), choose "XY (Scatter)", click on the first option and hit "next". On the next screen, click on the "series" tab and define your X and y-axes. Click "next" and finalize the setup.



	A	B	C	D	E	F	G	H
1		Table 2. Table of Results.						
2		Sample Name:		Sample Name:		Unkown Sample		
3		Concentration	Absorption	Concentration	Absorption	Absorption		
4		2%	0.124	2%	0.045			
5		4%	0.204	4%	0.091			
6		6%	0.351	6%	0.151			
7		8%	0.451	8%	0.254			
8		10%	0.551	10%	0.354			
9								
10								
11								

Figure 1. Using excel.

2- After plotting the graph print it and locate where the unknown sample would be.
3- Indicate the concentration of the sample and its composition.