**Session 1526**

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

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## Abstract

As part of an integral approach in using hands-on teaching and learning, the Chemical Engineering Department at Rowan University is constantly developing unique experiments in which students learn fundamental principles through exploration of familiar objects, products, and processes.

The manufacturing and analysis of olive oil utilize a series of chemical processes and analytical techniques that are not traditionally covered in chemical engineering and/or chemistry curricula, but that are commonly encountered in industry.

In this project we will develop novel hands-on chemical engineering experiments by examining the manufacturing process steps. Each step will be a laboratory module. These modules will be integrated throughout the chemical engineering curriculum. The manufacturing steps range in complexity from fundamental engineering and science principles shown in gravity decantation of immiscible liquids to more complex principles required to describe filtration theory and identification of compounds using Nuclear Magnetic Resonance spectroscopy.

## Introduction

At Rowan, a hands-on minds-on approach to teaching and learning is used. Students are introduced to engineering principles and fundamentals starting at the freshman through what we called engineering clinics. Our program include interdisciplinary engineering clinic every semester.  Sharing many features in common with the model for medical training, the clinic provides an atmosphere of faculty mentoring hands-on, laboratory setting. These clinics allow students to work in multidisciplinary team with faculty of all four engineering disciplines (chemical, civil, mechanical and electrical engineering). Staring at the fall semester of the freshmen year, students are introduced to measurements, data analysis, and representation. The freshmen spring semester focuses on process and product design through reverse engineering. Students learn engineering tool and concepts by experimenting with real life processes and products such as automatic coffer makers, bread-making machines, electric toothbrushes, beer production and the human body. This approach has been very successful as is demonstrated by Hesketh [1] using the vehicle of a coffee machine. The introduction of SMET principles by application to familiar systems is extremely effective in attracting and retaining engineering students [2, 3, 4, and 5].

In addition to the clinic, specialized courses are taught to deliver a well-blended combination of theoretical and practical skills. This proposal strives to improve these programs and provide industry with engineers who can relate theoretical knowledge with tangible applications.

**Goals and Objectives**

Our goals and objectives in this project include:

1. Improve the learning process of fundamental physical and chemical processes by providing the students with innovative laboratory experiments that include solids size reduction, coalescence phenomena, filtration theory, liquid-liquid gravity and mechanical driven separations, adsorption, Nuclear Magnetic Resonance (NMR), and data acquisition and analysis.
2. Ensure that the equipment we purchase has an impact on a wide variety of courses in our curriculum including the interdisciplinary clinic sequence and core courses in the chemical engineering and chemistry programs.
3. Adapt ongoing research on olive oil properties (University of Wales, Istituto Sperimentale per la Elaiotecnica) to teach students about chemical flavors, and most important, the effect of monounsaturated fatty acids (olive oil contains around 70 % of this fatty acids) on human

cholesterol levels. These analytical experiments will improve the current biochemical and biomedical course contents.

4. Expand student teamwork experience by making group projects an integral part of the course structure.
5. Continue to improve written and oral communication skills of our students.

**Project Description**

The proposed project is intended to provide hands-on experience to enhance the learning process of fundamental principles and operations in chemical engineering. This project will adapt a commercial manufacturing line into a series of lab modules that teach separations processes (solid-liquid and liquid-liquid), data acquisition, chemical species recognition, and statistical analysis of the data.

Olive oil is manufactured using a series of batch processes which are characterized by a physical-chemistry operation [6]. This sequencing makes the process suitable for its vertical integration throughout the curriculum. Furthermore, the simple and safe nature of the manufacturing process readily makes it possible to transform these experiments into hands-on active learning experiences. The project will be integrated into the following Chemical Engineering and Chemistry courses: Engineering Clinics, Fluid Mechanics, Principles Chemical Processes, Separations Processes, Instrumental Methods, Unit Operations Laboratory, Process Optimization, Chemical Process Component Design, and Biomedical Process Engineering.

The proposed manufacturing line at Rowan will provide students with a unique interdisciplinary learning experience in which both, the Chemical Engineering and Chemistry departments will benefit from it.

**The Process and the Experiments**

Olive oil has gained an increasing market share because of its potential health benefits. This has resulted in an increase of research to determine what chemical components cause this health benefit [7, 8]. Currently, 486 millions of gallons is consumed worldwide every year. U.S.

demand increases about 20 % each year. The U.S. only produces 0.1 % of the total year production and takes nearly 30 % of the total worldwide imports (only outrun by the EC, which accounts for nearly 40 % of the imports).

The industrial process involve the following steps:

1. **Washing the olives**: removes dirt, stems, twigs and rocks.
2. **Milling/grinding the olives**: a paste is formed by size reduction, this is the first characteristic to be brought into a lab experiment.
    - *Experiments*: Product yield will be explored as a function of olive fragment size. The effect of the grinding time and grinding method in the final paste size will also be addressed. Students can easily relate these concepts by comparison with coffee beans grinding.
3. **Mixing**: this process is conducted to increase the size of the oil droplets. It is desirable to have large droplets, since they are easier to separate from the water and higher yields of oil result.
    - *Experiments*: Students will learn about surface tension forces responsible for the coalescing mechanism, mixing time, and temperature influences, and they will be able to compare results when a home beater mixer is used instead of the industrial spiral mixing blades unit
4. **Primary Separation**: in this step, the solid paste (pomace) is separated from the liquid mixture.
    - *Experiments*: Two processes will be analyzed- filter pressing and centrifugal decantation. Traditionally, chemical engineering curriculum includes one or more filtration experiments usually in the junior/senior Unit Operations Lab. In these experiments, a water-based solution of a salt (i.e. $CaCO_3$) [9] is filtered and the properties of the resulting cake analyzed. This type of experiment, although good in conveying filtration

theory information, does not offer insights on the importance that this operation has in the food and specialty chemicals industry. The filter press (figure 1) experiment proposed in this project will deliver the required filtration theory but it will also show students the role of this type of operation in the overall manufacturing process. Students will be capable of observing the effect that the applied pressure has on cake formation, moisture content and resistance, and volume of filtrate (oil-water mixture) as a function of time.

- Ultimately, students will identify the influence of these variables in product yield. In addition, different filtering materials can be tested and their effect in oil yields. Therefore, complete filtration theory can be addressed at this module.

Figure 1: Pressure Filter

5. **Oil-Water Separation**: In the olive oil industry, the conventional separation process is by gravity followed by decanting the lighter oil phase. In addition to performing experiments

using the conventional process, they will also perform experiments using a membrane process called Ultrafiltration (UF) [10].

- *Experiments*: The decanter experiments will be used to incorporate basic principles such as liquid miscibility concepts, density difference, and height effect in gravity separation. Ultrafiltration experiments will be conducted and the results compared to the conventional separation process. This set of experiments will show how an engineer can use new and innovative membrane technology to improve the performance of a process. Student teams will be established to conduct these tests and study the economic feasibility of these applications.

6. **Refining and deodorization**: Olive oil is a complex compound made of fatty acids, vitamins, volatile components, water soluble components and microscopic olive solids. The primary fatty acids are oleic and linoleic acid. The unique and delicate flavor of the oil is attributed to a number of volatile components. To improve the final product taste and odor, several volatile components need to be removed and acidity adjusted. Among the processes available for this task are: low temperature stripping and activated carbon adsorption.

- *Experiments*: an activated-carbon adsorption experiment will be used to introduce fundamental adsorption principles. The height of the carbon bed, column diameter, liquid flowrate, carbon properties and size distribution, and temperature are among the multiple variables that will be analyzed. Students will be able to establish the effect of these variables in the final product properties. Acidity will also be determined to assess its relationship with flavor and aroma.

7. **Chemical Analysis of Final Product**: flavor and aroma measure the quality of an olive oil. Results of current research suggest a good correlation between these properties and the polyphenol content [6]. Hydroxytyrosol, tyrosol, caffeic acid, coumaric acid, and p-hydroxybenzoic acid influence the sensory characteristics of olive oil. Hydroxytyrosol is present in good-quality olive oil, while tyrosol and some phenolic acids are found in olive oil

of poor quality. Various off-flavor compounds are formed by oxidation. Pentanal, hexanal, octanal, and nonatal are the major compounds formed in oxidized olive oil, but 2-pentenal and 2-heptenal are mainly responsible for the off-flavor. HPLC, Gas Chromatography, Nuclear Magnetic Resonance (NMR), and RAMAN Spectrometry are normally used to identify the main chemical species present in the oil. At Rowan, we have the capacity to perform HPLC, GC and NMR analysis.

- *Experiments*: a variety of olive oils will be given to the students to analyze and relate the presence of some of the aforementioned chemical compounds with the aroma and flavor of the oils. Student will also compare the characteristic of the in-house produced oil with the provided commercial oils.

8. **Olive Oil "Fingerprinting"**: Several European researchers [11, 12] have reported that the chemical composition of olive oil can be easily used to determine the geographical origin of such oil. These techniques are also used to identify olive oil adulteration [6, 8].

- *Experiments*: Another interested aspect of this project would be to develop experiments where students can "fingerprint" olive oil through its chemical composition. Students will be given a set of pre-analyzed olive oils of known origin and ask to perform the analysis using the existing NMR capacity at the Chemistry Department.

9. **Olive Oil and Health Related Issues**: Oleic acid is a monounsaturated fatty acid that makes up 55-85 % of olive oil, and linoleic acid isomers make up approximately 10 %. These two unsaturated fatty acids are linked to several beneficial effects in human health. Replacing dietary saturated fat with oleic acid is reported to lower levels of total plasma cholesterol and low density lipoproteins (LDL, also known as "bad cholesterol") without substantially reducing levels of high density lipoproteins (HDL, "good cholesterol") [13]. The so-called "Mediterranean Diet" is constantly appearing in the news [14, 15]. Quoting CNN, "People who eat a so-called Mediterranean diet are as much as 70 percent less likely to suffer a

second heart attack that those who follow a typical “Western” diet, according to new research”.

Oleic acid was also featured in the recent Hollywood film “Lorenzo’s Oil” (Universal Studios, 1992). In a rare genetic illness called adrenoleukodystophy (ALD), young boys suffer from an excess of harmful long chain ($C_{24}$-$C_{26}$) fatty acids which causes the myelin around nerves cells to deteriorate, leading to brain damage. They progressively lose more of their faculties, speech, movement, etc., until they eventually die. It was discovered that ingestion of large quantities of pure *oleic acid* triglyceride (mixed with around 25 % of the related $C_{22}$ acid) prevented the build up of the harmful longer chained acids, relieved all the symptoms associated with the disease, and allowed the sufferers to lead a normal life. This works by essentially keeping the enzyme responsible for biosynthesis of the acids busy metabolizing these harmless acids, and so they have no resources left to produce the harmful ones.

**Conclusions**

In this proposal we have developed several experiments that will allow teaching chemical engineering fundamentals and unit operations in a safe environment. The lab experiments will rise in complexity as advanced engineering topics are introduced as needed on specific courses and clinics. The utilization of a real manufacturing process tied to a common consumer product such as edible oils is expected to enhance the learning experience.

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