STUDENTS' PILOT LABORATORY FOR HOMOGENEOUS CHEMICAL REACTOR ANALYSIS AND DESIGN IN OLIVE MILL WASTEWATER TREATMENT

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In recent years, the Chemical Engineering Department at the University of Granada has endeavored to make a number of high quality, hands-on experiments available to undergraduate students enrolled in senior-level courses. In particular, the target is to familiarize our students with our latest research and also with scale-up of processes.

The present work is focused on giving the students a closer practical view of the treatment process of one of the most polluted agro-industrial effluents—that generated by the olive oil industry. This is especially interesting given the fact that the olive oil industrial sector is one of the most important ones in the economic framework of the Mediterranean countries and others—France, Serbia and Montenegro, Macedonia, Cyprus, Turkey, Israel, Jordan, the Middle East, Australia, the United States, and China—where olive oil production is also rapidly becoming an emergent agro-food industry. The treatment of olive mill wastewater (OMW) is already a task of global concern.

Direct disposal of these effluents to surface waters is often illegally practiced and causes severe pollution. OMW also cannot be used straight for irrigation.[1,2] OMW discharge contaminates soil, inhibits plant growth, and causes odor, underground leaks, water pollution, and hindrance of self-purification processes, as well as having negative impact on aquatic fauna and habitat.[3,4] The general attempted solution has been the construction of artificial lagoons for natural evaporation, like that shown in Figure 1. Over the years, this has become inefficient because of the low evaporation potential of these ponds, odor release to the surroundings, and hazardous underground leakages from deficiencies in pond construction.

Figure 1. *Artificial lagoons are used for storage and natural evaporation of olive mill wastewater.*

OMW exhibit several characteristics that make reclamation by conventional physicochemical treatments difficult.[2,5] The presence of phytotoxic refractory pollutants makes these effluents recalcitrant and thus hinders biological processes.[2] On the other hand, the acidic pH, low alkalinity, and low nitrogen content, as well as the presence of lipidic and phenolic fractions, make this wastewater potentially toxic for anaerobic treatments.[6]

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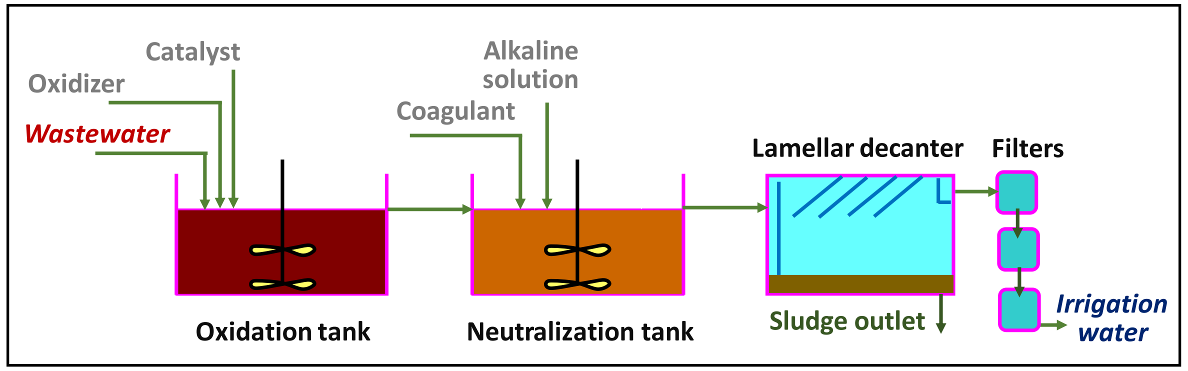

***Figure 2.** Fenton-like oxidation flow-scheme.*

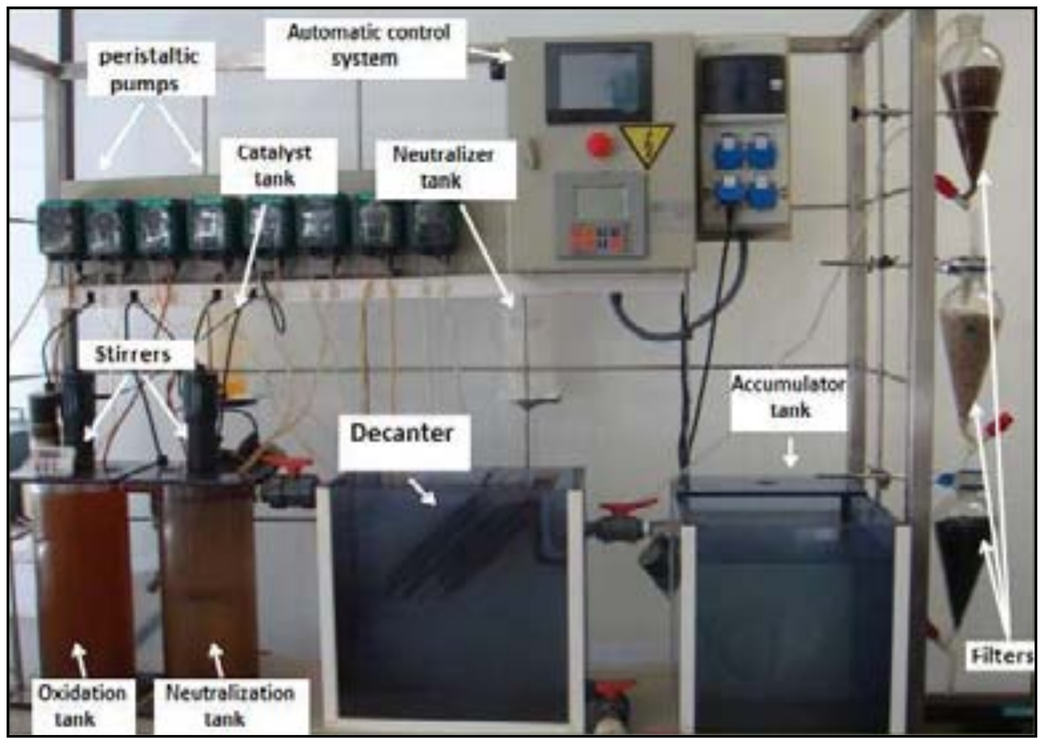

***Figure 3.** Fenton-like oxidation pilot-scale plant.*

A wide variety of alternative stand-alone and integrated processes for the treatment of OMW have already been proposed and developed but have not yet led to completely satisfactory results in terms of cost-efficiency, such as natural evaporation and thermal concentration,[2] composting,[7] treatments with clay[8] or lime,[9] and physicochemical procedures including coagulation-flocculation,[10] electrocoagulation,[11] and biosorption.[12]

In this respect, chemical remediation strategies known as "advanced oxidation processes" (AOPs) are required for its depuration; these include ozonation,[13] Fenton's reaction[14] photocatalysis,[15] electrochemical treatments,[16] and hybrid processes.[17]

Currently, there is a scarcity of literature in chemical engineering education international journals referring to teaching of treatment of bio-refractory wastewaters by advanced oxidation processes. This is a need, since AOPs have recently turned out as a feasible solution for the depuration of recalcitrant wastewaters.[13,14,18] For this purpose, the TEP-025 Research Group, Chemical and Biochemical Processes Technology, has set up a wastewater treatment plant on a pilot scale on the premises of the Chemical Engineering Department of the University of Granada. It allows the students to not only observe but also fully interact with the wastewater treatment plant devices, in a scale similar to the real industrial one.

In this sense, it is worth underlining the need to strengthen experimental learning by chemical engineers on a pilot scale, based on active learning.

EXPERIMENT

Wastewater depuration pilot plant and practical activities

The pilot plant used in this work was located on the premises of the Department of Chemical Engineering at the University of Granada. The flow-scheme of the treatment process is shown in Figure 2. A photograph of the pilot plant is given in Figure 3. The process undertaken at the plant consisted of the following:

1. *Chemical oxidation was carried out in a continuous stirred tank reactor (CSTR). The reactor (vertical cylindrical vessel with flat bottom) was 16 cm in diameter and its overall height was 38 cm. The working volume and the overall volume of the reactor were 7.0 L and 7.4 L, respectively*
2. *Neutralization was carried out in a stirred tank similar to the reactor described above (with 7 L capacity)*
3. *Solid–liquid separation was conducted in a lamellar settler (22 L capacity)*
4. *Six storage tanks: (i) for OMW (32 L capacity); (ii) for the oxidant reagent (3 L capacity); (iii) for the catalyst (3 L capacity); (iv) for the neutralizing agent (3 L); (v) for the preparation of the flocculant provided with agitation system (1 L capacity); and (vi) for the final treated water (50 L capacity)*
5. *Nine peristaltic pumps*
6. *Pneumatic level sensors*
7. *Electronic control system to work whether in batch or continuous mode*
8. *Three stirring systems formed by overhead stirrers installed in the oxidation reactor, neutralization tank, and flocculant tank. The stirring equipment consists of three impellers set at 60 rpm. A three-bladed mixing propeller was placed above the bottom of the reactor, the other two turbines with four blades above were installed in the shaft. Each impeller was 5 cm in diameter, the vertical distance between the impellers was 12 cm, and the lower impeller was located 5 cm from the bottom of the reactor*

For the experiments of the Fenton-like process, $FeCl_3$ was used as catalyst, as in the study by Martinez, et al.[5] who compared three different reagents—ferric perchlorate, Mohr's salt, and ferric chloride—concluding that $FeCl_3$ is the best catalyst from the point of view of effectiveness and economic cost. Moreover, the $FeCl_3$ catalyst can be recovered and reused, boosting the economic efficiency of the process.

To evaluate the effectiveness of the proposed Fenton-like process for the treatment of OMW, the influences of the main variables that control the oxidation process were examined: pH, $[FeCl_3]/[H_2O_2]$ ratio, and temperature. Once the wastewater entered the reactor, a certain amount of $FeCl_3$ was added and then the hydrogen peroxide solution, keeping agitation at 60 rpm (following Martínez, et al.[5]) and this moment was considered as zero time. During the experiment, samples were taken at regular intervals during three hours of operation. In all experiments, 5% (w/v) hydrogen peroxide solution was added, since according to Martínez, et al.[5,14] it offers the highest percentage of chemical oxygen demand (COD) reduction.

Three sets of experiments were carried out. In the first set, $FeCl_3$ concentrations, $[Fe^{3+}]$, from 0.04 to 4 g/L were tested, while hydrogen peroxide dose $[H_2O_2]$ was kept at 20 g/L. In the second set, three $[H_2O_2]$ concentrations were used (20, 100, and 200 g/L), and iron concentration was maintained at 4 g/L. Then, different experiments with the following $[FeCl_3]/[H_2O_2]$ ratios were prepared: 0.002, 0.003, 0.010, and 0.020 (using OMW with initial COD = 1700 mg/L) and other $[FeCl_3]/[H_2O_2]$ ratios (0.020, 0.090, and 0.2) using wastewater with initial COD value equal to 4137 mg/L. In the third set of experiments, three temperatures were used (10, 20, and 30 °C) while the $[FeCl_3]/[H_2O_2]$ ratio was maintained at 0.090 and the initial COD value of the wastewater was equal to 4150 mg/L.

Chemicals

The different reagents used in this work were hydrogen peroxide (30% w/w H_2O_2), sodium hydroxide (98% w/w NaOH), hydrochloric acid (37% w/w HCl), and ferric chloride (30% w/w $FeCl_3$), all them purchased from Panreac S.A; phenol (99% w/w C_6H_5OH) provided by Sigma-Aldrich; and Nalco 77171 flocculant (anionic polyelectrolyte oil-based) bought from Nalco España S.A. (Barcelona, Spain).

Analytical methods

Measurements of COD, residual H_2O_2 concentration, total suspended solids (TSS), ashes, total phenols, total iron, electrical conductivity (EC), and pH were carried out following standard methods.[19] A Helios Gamma UV-visible spectrophotometer (Thermo Fisher) was used for COD, total phenols, and total iron measurements.

EC and pH measurements were performed with a Crison GLP31 conductivity-meter and a Crison GLP21 pH-meter, with temperature autocorrection (25 °C). Buffer standard solutions for EC (1,413 µS/cm and 12.88 mS/cm) and pH (pH 4.01, 7.00 and 9.21) measurements were also supplied by Crison.

COD was measured by photometric determination of the concentration of chromium (III) after 2 h of oxidation with potassium dichromate/sulphuric acid/silver sulfate at 421 K (German standard methods DIN 38 409-H41-1 and DIN ISO 15 705-H45).

Residual H_2O_2 concentration was determined following the colorimetric method using titanium sulfate, given its simplicity and accuracy. Titanium sulfate reacted with the H_2O_2 present in the solution, forming a yellow complex with a maximum absorbance around 410 nm.[19]

Iron ions were reduced to iron ions (II) and—in a thioglycolate medium with a derivative of triazine—formed a reddish-purple complex that was determined photometrically at 565 nm (Standard German methods ISO 8466-1 and German DIN 38402 A51).

Total phenols and phenol derivatives reacted with a derivative thiazol, giving a purple azo dye, which was determined photometrically to 475 nm (Standard German methods ISO 8466-1 and DIN 38402 A51).

RESULTS AND DISCUSSION

The reason for setting up the pilot plant was the scarcity of teaching about bio-refractory wastewaters by AOPs found

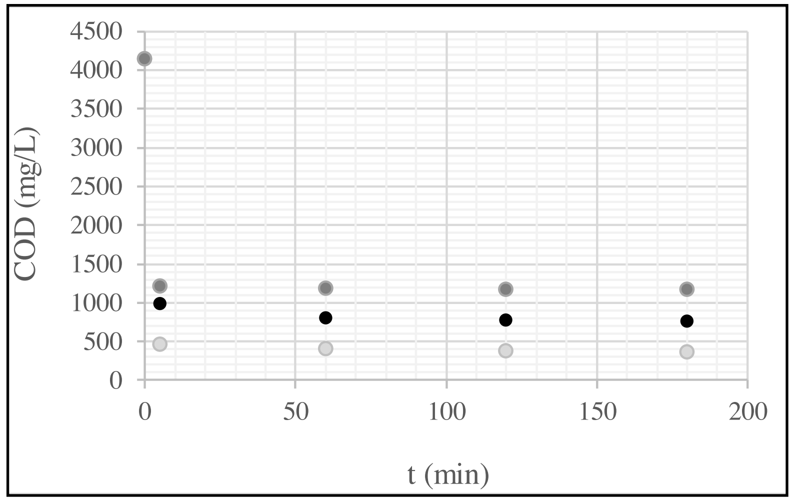

Figure 4. *COD concentration in the Fenton oxidation CSTR vs. time. Experimental conditions [Fe^{3+}]/[H_2O_2] ratio: = 0.02 (dark gray dot), 0.09 (black dot) and 0.2 (light gray dot); pH = 3 and temperature 20 ° C.*

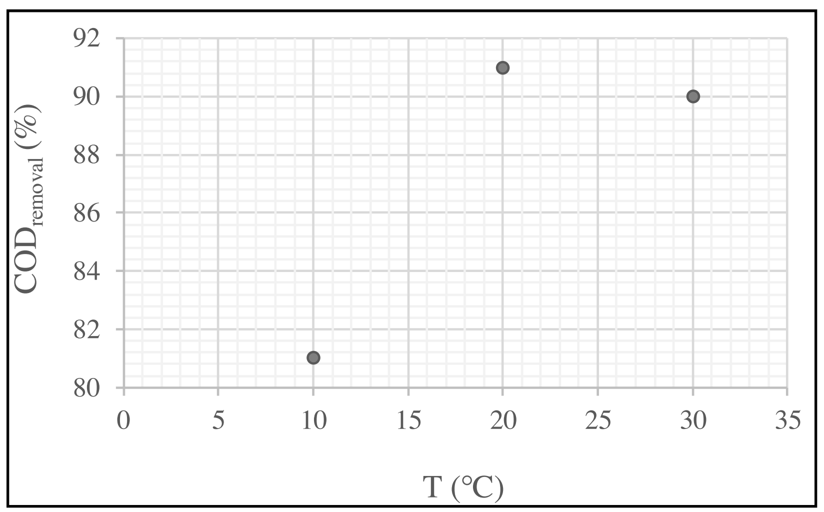

Figure 5. *COD removal efficiency in the Fenton oxidation CSTR vs. T: 10, 20, and 30 ° C. Experimental conditions: [H_2O_2] = 45 g/L, [Fe^{3+}] = 4 g/L, pH = 3.*

in chemical engineering education international journals. Similarly, Ganley, et al.[20] developed and implemented an experimental module for senior-level reaction engineering/reactor design students to characterize the kinetics of dye neutralization by household bleach in a plug-flow reactor. This is indeed a need, since AOPs—including ozonation, Fenton's reagent, wet oxidation, and photocatalytic processes, as well as electrochemical, solar-driven and hybrid treatments—have been pinpointed as the most feasible solution to date for the depuration of recalcitrant wastewaters.[13,14,18]

In this experimental practice at pilot scale, the different stages of the process were shown *in-situ* and explained: first, Fenton-like chemical oxidation carried out in a continuous stirred tank reactor (CSTR), followed by a coagulation flocculation process; next a decanting stage to achieve solid-liquid separation; and finally filtration-in-series system through sand, active carbon, and olive stones.[12,14] In this way, students can observe the effluent passing through the several stages and understand which stage is attained in each operation.

Furthermore, the different components of the pilot plant involved in the Fenton-like process were shown and their specific function could be fully explained, including peristaltic pumps, different stirrers (propeller and turbine), automatic control system, pneumatic level sensors, and pH and temperature gauges.

This teaching tool not only lets the students learn about this physicochemical treatment and become familiar with the basic wastewater purification stages and devices by carrying out experimental practices, but also encourages them to be concerned about the environmental problem related to the generation and disposal of these heavily polluted liquid effluents.

In the Fenton process normally Fe(II) salts are used as the catalyst with hydrogen peroxide (H_2O_2) as the oxidant agent.[14] Among the wide range of advantages it presents we can pinpoint its equipment simplicity and operational ease—since it may be conducted at ambient temperature and pressure conditions—and also its high performance for the oxidation of multiple organics and its non-toxicity, given that H_2O_2 can break down into environmentally safe species like H_2O and O_2.

During the experiments the students filled a workbook that included an introduction to the olive oil production process and the generated effluents, the problems and necessities in relation to wastewater treatment, the flow diagram of the Fenton-like oxidation process (Figure 2), reagent preparation and characteristics, some calculations regarding the latter as well as the efficiency of the process, and a series of activities and graphical representations in relation to the depuration process. As examples, the COD concentrations in the Fenton oxidation CSTR vs. time for different [H_2O_2]/[Fe^{3+}] ratios studied are given in Figure 4, whereas the COD removal efficiency in the Fenton oxidation CSTR vs. various temperatures (10, 20, and 30 °C) is reported in Figure 5.

It was observed that increasing the [Fe^{3+}]/[H_2O_2] ratio did not lead to major COD removal, as can be seen in Figure 4. Moreover, it should be noted that when the ratio of [Fe^{3+}]/[H_2O_2] > 0.01 acidification of the reaction medium was not required, because of the fact that [H^+] generation lowered OMW pH immediately from 6-7 down to 2-3.

Otherwise, it was confirmed by the students that higher COD removal efficiencies could be attained upon incrementing the reaction temperature, up to a value of 20 °C (ambient temperature), such that further increase of the reaction temperature did not lead to cost-efficient improvement of the Fenton oxidation reaction, as it can be seen in Figure 5. It was therefore confirmed that, among the different AOPs, Fenton's process appears to be the most economically advantageous since it can be conducted satisfactorily at ambient temperature and pressure conditions, and also due to its equipment simplicity and operational ease.

Students may also simulate reactor response for different reactor volumes and species' residence times over a range of operating conditions, and perform a material balance on a particular pollutant or global COD, assuming perfect reactor mixing.

Students may take into account a range of reactor variables, such as the reactor volume, conversion, total volumetric flow rate, temperature, pH, and the initial concentrations of each reactant. Some example design problems include:

- *Study of the required maximum or minimum solution feed rate required to achieve a given pollutant or global COD conversion, with all other reactor conditions fixed (temperature, volume, feed concentrations).*
- *Determining the range of reactor conversion resulting from the adjustment of the temperature and reactants feed streams over a specified range.*

To sum up, the learning activities performed using the experimental apparatus for each stage of the process comprise the following:

- AOP reaction:

- *use of different catalysts (aluminium sulphate, aluminium chloride, Mohr's salt, ferric per-chlorate, ferric chloride)*
- *catalyst dosage*
- *oxidant dosage*
- *catalyst/oxidant ratio*
- *effect of pH*
- *effect of T*

- Flocculation process:

- *different flocculants: QG-2001, QG-2002, DQGALFLOC-130H, and Nalco- 77171 (commercial anionic or cationic polyelectrolyte-based)*
- *different flocculant dosages: 0.5 - 150 mg/L*
- *effect of stirring rate and time on flocculation performance*
- *effect of pH and control on flocculation performance*

- Settling process:

- *Lamellar decanter*
- *Truncated decanter*
- *Effect of flowrate*

- Filtration process:

- *Filtrating material (gravel or sand, olive stones, activated carbon)*
- *Effect of granulometry*
- *Effect of filtrating material load*
- *Effect of filtrating sequence*
- *TSS removal efficiencies of the different processes*

The learning objectives pursued in each process stage were :

- AOP reaction:

√ *Study of the effect of the operating variables on the AOP performance*

√ *Thermodynamics and kinetics (COD, phenols) of AOP on the effluent treatment*

√ *Effect of the reagents type and dosages on the AOP performance*

√ *Effect of residence time*

- Flocculation process:

√ *Study of the effect of flocculant type and dosage on the flocculation efficiency*

√ *Effect of the operating conditions and residence time*

- Settling process:

√ *Study of the efficiency of different types (geometry) of settlers*

√ *Study of the settling velocity*

- Filtration process:

√ *Study of the efficiency of different types of filtrating materials*

√ *Effect of granulometry and load*

√ *Optimization of the filtration sequence*

Moreover, overall experiments conducted using the entire apparatus served to examine the entire set of processes, with the following objectives:

√ *Effect of the main operating variables (flowrate and temperature) on the overall process performance and kinetics*

√ *Effect of pH on the overall process performance*

√ *Study of the type and dosage of reagents of the overall process performance*

Prior to performing the pilot-scale experiments, students were provided with a detailed document that includes a

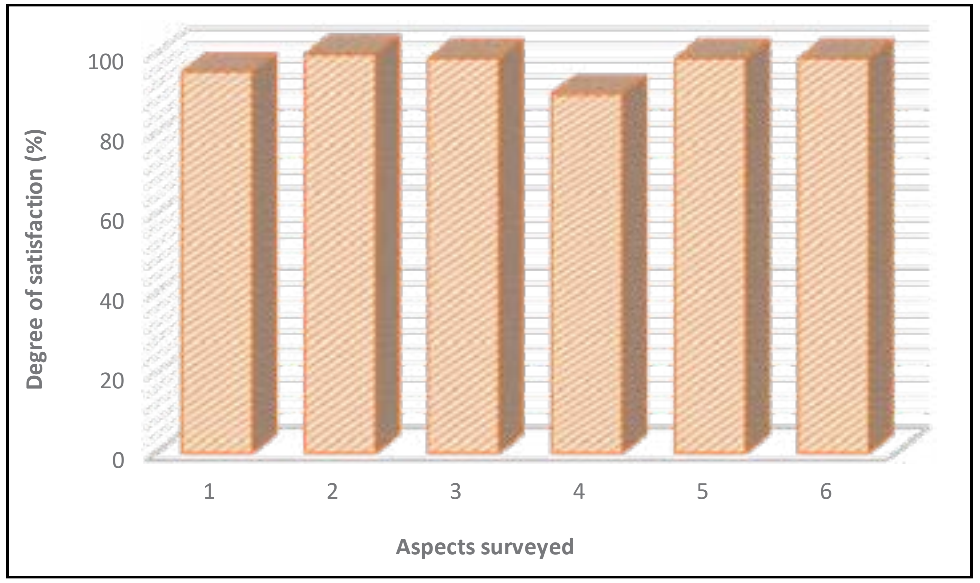

Figure 6. *Survey made to students of subjects Chemical Industries and Chemical Engineering (4th and 3rd courses, degree in chemistry) at the end of the pilot experience. 1: adequate grade of difficulty; 2: good interaction; 3: improved knowledge of automatic control systems; 4: versatility of the pilot plant; 5: improved vision of industrial scaling; 6: overall satisfaction with the experience.*

basic explanation of the nature of the experiment, sequential operating instructions and data acquisition systems, and a short discussion of the significance of the collected data of operation. Students divide their efforts in the laboratory work and data analyses, and are formally divided into teams to compare results.

During the performance of the experiments, any questions and clarifications made by the students—above all, those requested at the beginning of the lab—received immediate feedback and this helped the students carry out the experiments with net confidence. Also, each team of students was observed to be actively assisting the other teams during the laboratory session. This important fact leads to a very productive and cooperative environment, and could even make it possible to carry the laboratory without the direct intervention of the supervisors.

Finally, the students shared their results, impressions, and the knowledge acquired with the teachers and rests of students, and their workbook was evaluated by the corresponding supervisor to correct any erroneous concepts and consolidate the learning process.[21-23] Students apply the various writing and reporting techniques to produce concise reports of significant analytical depth, to comply with the learning objectives and outcomes related to the pilot module.

A survey was made to the students of the operative subjects Chemical Industries (4th course, degree in chemistry) and Chemical Engineering (3rd course, degree in chemistry) at the end of the experience (total number of surveyed students = 60), covering different aspects of the quality and satisfaction that comprised the grade of difficulty, ranking of interaction, improved knowledge of automatic control systems, versatility of the pilot plant, improved vision of industrial scaling, and overall satisfaction with the experience. The survey asked about six items, and each item was based on a subset of scores to denote satisfaction on a usual 5-point scale with a final open "observations and suggestions" question; the results are shown in Figure 6. The obtained feedback encouraged continuing with the implementation of this kind of setup, as all aspects surveyed were valued above a 90% degree level of satisfaction. The obtained feedback encouraged continuing with the implementation of this kind of setup, as all aspects surveyed were valued above a 90% degree level of satisfaction. The overall quality of the student work resulting from this lab experience and student feedback regarding the laboratory module has been generally very positive. The attitudes and responses observed in the students toward the pilot-plant module indicated very good student satisfaction, and that students appreciate the flexible application of the pilot plant.

Furthermore, the experimental hardware and the instructional documentation have been successively refined after each of the courses for which the experiment has been used, in part based on the feedback received from students. In this sense, the length of the practices was adjusted, and the sets of experiments among the different possibilities were chosen with regard to students' suggestions of areas where they felt they needed more training.

In this context, it is worth highlighting the major focus on experimental learning rather than lecture-based learning. As reported by Keyser[24] lecture-based learning is considered unsuccessful in many cases due to poor student attention, simplified examples, and too much material presented at once. Students' attention is believed to decrease as the lecture proceeds and only lower-level learning information is acquired.[25]

On the other hand, properly structured laboratory projects may be classified as inductive learning experiences. Laboratory projects have been demonstrated to enhance the development of discipline-specific skills and general research skills. If adequately conducted they may be a kind of active learning that enhances student learning and improves retention of information.[26-28] As suggested by Chickering and Gamson,[29] the importance of active learning lies in the fact that it may involve higher-order thinking activities comprising analysis, synthesis, and evaluation. In active learning students are involved in more than listening; attention is focused on developing students' skills.[25] Meyers and Jones[30] highlighted that active learning permits "students to talk, listen, read, write, and reflect through problem-solving exercises, informal small groups, simulations, case studies, and role-playing." Active learning may also engage multiple intelligences, self-reflection, and dialogue with others.[31] As underlined by Patterson,[25] active learning does not mean lack of lectures or instructor involvement; instructors should still play an influential role in increasing students' interest.[32]

One more drawback observed in many chemical engineering experimental subjects is that most chemical reaction practices are carried out at lab scale, whether in vessels and beakers magnetically stirred or in small lab or bench-scale devices purchased from commercial parties.[33] This is indeed not enough nor satisfactory for a chemical engineer's maturation, as in those commercial devices there is a lack of scalable pumping and stirring systems, deflectors, and automatic control loops, etc. This limits chemical engineering students' ability to visualize, become familiar, and perform reaction experiments in the most real-scale approach possible, as can be done in pilot-scale experiments. Moreover, bench-scale devices provided by commercial firms do not permit modification, disassembly, and reassembly with the goal of being more versatile as well as allowing the students to analyze the components, pumping and pipe systems, valves, and gauges, and to test different configurations.

On the contrary, by using pilot plants such as the one described here, students are allowed, for instance, to test different propeller and impeller stirrers and follow the reaction conversion and efficiency as a result. In the constructed pilot plant an automatic control loop was implemented, allowing the measurement and adjustment of the pH, temperature, stirring, feedstream and reagents flowrate, and tank volume (it was also equipped with minimum and maximum tank levels controlled by pressure gauges to avoid excessive tank overflow). In the set-up plant presented here, any process sequence may be studied, allowing, for example, testing different types of solid/liquid separation operations after the oxidation reaction, comprising conic or lamellar decanters and different types of filtration (with activated carbon, gravel, and/or biosorbents like olive pitches, in different configurations and sequences). See Figure 3.

From the survey, regarding an open question to students asking which competencies they considered to have improved with the present practical set-up, the following answers were highlighted: improvement in the knowledge of equipment and wastewater plant design; optimization of operating variables of different unit operations of plants; understanding of the spatial connection among different parts of a wastewater treatment process and the effect in the overall efficiency; and reclamation and reuse of secondary streams.

This theme is very interesting for the students of these scientific areas, given the fact that the olive oil industrial sector is one of the most important ones in the economic framework of Mediterranean countries, highlighting Spain as the main olive oil producer worldwide currently. Moreover, the Fenton process can be applied not only to this kind of particular wastewater (OMW), but also for the treatment of several types of industrial and agro-food industries, such as textile effluents,[34] dye wastewater,[35,36] cosmetic effluents,[37] laboratory wastewater,[38] pharmaceutical wastewater,[39] cork cooking,[40] pulp mill effluents,[41] pesticide wastewaters,[42] OMW,[14] and phenolic wastewaters.[43] Therefore, the analysis, full understanding, and learning of this procedure not only at simple lab-scale but at pilot scale, may also help students to understand this treatment process for those effluents and analogous ones, since the knowledge acquired may be easily extrapolated.

The pilot-scale plant was also used for Science Week, an event that takes place each academic year within the first week of November (<http://fciencias.ugr.es/en/home/32-cursos/la-noche-de-los-investigadores/1043-depuracion-de-aguas-residuales-de-laindustria-oleicola>). During this week students choose to perform a variety of experimental scientific practices. For the setup of the experimental experience, students were divided in two groups of five to eight, in two sessions daily for a total number of 10 sessions during the week. Daily sessions were supervised by two lecturers, meaning 10 lecturers during the experience throughout the week. Before the implementation of the activity, the necessary reagents (see "Chemicals" subcategory in the "Experiment" section) were purchased or provided by the TEP-025 Research Group "Chemical and Biochemical Processes Technology," as well as the analytical equipment (see sub-section on analytical methods).

At the end of Science Week, qualitative surveys were given to the students to evaluate their degree of satisfaction and gather their suggestions to improve the experience. The following year, the experience was also implemented during the Summer Scientific Campus of the University of Granada (Figure 7), as well as in the European's Researchers Night (<http://fciencias.ugr.es/estudios/34-noticias/1739-nochede-los-investigadores-2014>), and received positively.

Currently, this material is widely used in chemical engineering, chemistry, food technologies, and environmental sciences lessons at the University of Granada. Moreover, it is used every year in the BioTic Summer Scientific Campus of the University of Granada, as well as in the Science Week and in the European Researchers Night, and ultimately in the Proyecto de Iniciación a la Investigación e Innovación en educación Secundaria en Andalucía (Project for the Initiation of Research and Innovation at High School in Andalucía) experiences.

CONCLUSIONS

In recent years, the Chemical Engineering Department at the University of Granada has endeavored to make a greater number of high-quality, hands-on experiments available to undergraduate students enrolled in senior-level courses. In particular, the target is to familiarize our students with our latest research and also with scale-up of processes.

The present work focuses on giving students a closer practical view of the treatment process of one of the most polluted agro-industrial effluents—the wastewater generated by the olive oil industry. This theme is very interesting for the students of these scientific areas, given the fact that the olive oil industrial sector is one of the most important ones in the economic framework of Mediterranean countries, highlighting Spain as the main olive oil producer worldwide nowadays.

The overall quality of the student work resulting from this lab experience is excellent, and student feedback regarding the laboratory module has been generally very positive. The attitudes and responses observed in the students toward the pilot-plant module indicated very good student satisfaction, and that students appreciate the flexible application of the pilot plant. Furthermore, the experimental hardware and the instructional documentation have been successively refined after each of the courses where the experiment has been in use.

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Figure 7. *Experimental workbook prepared for the students of the BioTic Scientific Summer Campus of the University of Granada (<http://biotic.ugr.es/estudia/campuscientificos- de-verano>).*

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