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Oil recovery through deemulsification research : separation of water from emulsified oil

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WERC International Environmental Design Competition Responsibilities

Aaron Russell

The purpose of this project was to develop an industrial process to separate an emulsion of oil and water that is used as a lubricant and coolant in copper rolling mills. The process developed was supposed to be designed in such a way that the maximum amount of each component could be recovered in an environmentally friendly way and subsequently recycled. Once a process was developed, the teams were required to construct a working, bench scale model of the process, as well as formulate a full scale design. I was one of six team members and I served as the research coordinator for the team. As the research coordinator, my main responsibilities included planning and conducting laboratory experiments and developing and demonstrating the bench scale apparatus.

I was also involved initially in the research that was done regarding known emulsion separation techniques. Through our research we were able to determine six separation techniques that we would be able to both test in the laboratory and adapt into a full scale design. The separation techniques we decided to test were thermal separation, centrifugation, acidification, hydrophobic ultrafiltration, hydrophilic ultrafiltration, and evaporation. The majority of the work that I did involved coordinating and performing the experiments to test the effectiveness of each of these technologies when applied to samples of the emulsion. The testing showed thermal separation, centrifugation, and hydrophobic ultrafiltration to be unable to achieve a significant degree of separation of the oil and water. Acidification, evaporation, and hydrophilic ultrafiltration each achieved excellent separation and, considering the environmental and economic implications of each technology, we ultimately decided to use a combination of hydrophilic ultrafiltration followed by evaporation. Also, after discovering the contaminants present in the water phase, we decided to treat the resulting water phase with a reverse osmosis system.

The next major responsibility that I had was the development and optimization of the bench scale system. Fortunately for us, most of the equipment that we need to put our system together was readily available within the department. Once the system was put together, many optimization tests had to be performed. These tests allowed us to determine the conditions at which we needed to run the system to ensure that we were able to meet the quality requirements of the competition while also staying within the time limit.

When it finally came time for the competition, my final responsibility came in the form of the presentation of our results. I was one of four people responsible for writing and giving a section of our presentation before a panel of 12-15 judges. I also helped in the development and presentation of a poster that summarized our process. My final responsibility was to demonstrate our bench scale system to produce samples of our products for judging. After three days of competition against seven other teams, our team took home first place.

ORDER

WERC TASK 5

SPRING 2008

Oil Recovery through De-Emulsification Research

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Separation of Water from Emulsified Oil

WERC Task 5

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PROJECT O.R.D.E.R.
Oil **R**ecovery through **D**e-**E**mulsification **R**esearch

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EXECUTIVE SUMMARY

In an effort to improve the environment, there is a need to recover and reuse the oil and water components of lubricating emulsions used in copper drawing and rolling processes. The Freeport-McMoRan Copper and Gold Inc. copper rod mill located in El Paso, TX was chosen as the site location for this project. It is one of the largest rolling and drawing operation facilities in the world, and it meets the established criteria set by Project ORDER. A large facility generates an average of 8,400 gallons of spent lubricant per day. The WERC emulsion sample contains 98 v% water and 2 v% lubricating oil and contains metal debris that would negatively impact water quality if it were discharged into surface waters. Oil and water are valuable resources and their maximum recoveries are desired. Project ORDER successfully recovers more than 90 v% of the water and essentially all of the oil. The recovered water could be recycled for fresh lubricant production within the facility, eliminating almost all water discharge and reducing water intake. The recovered oil will be sent to oil recyclers, lowering discharge expenses.

Project ORDER has carefully evaluated several water recovery, oil recovery, and metal recovery technologies to design the commercial process. The first processing step of Project ORDER is an ultrafiltration (UF) membrane that recovers 90 v% of the water in the spent emulsion sample. As water permeates the membrane, the concentration of oil in the emulsion increases from about 2 v% to 30 v%. The second processing step removes essentially all of the water from the UF concentrate using an evaporator, which operates by passing low pressure steam through a jacketed, agitated vessel. The third processing step removes metal debris from the oil using a depth filter. The fourth processing step utilizes a reverse osmosis (RO) membrane to purify the UF permeate water for recycle. The fifth processing step reduces the amount of waste from the RO reject using an evaporator, which also operates by passing low pressure steam through a jacketed, agitated vessel. The evaporator removes essentially all of the water in the RO reject and the remaining waste is sent for disposal. The evaporated water from both evaporation units is condensed and combined with the RO permeate to be recycled.

Based on a spent emulsion production rate of 8,400 gal/day, it costs $793, 000 per year for current disposal by incineration. For Project ORDER the fixed capital investment is $899,000, the yearly operating cost is $528,000, and the net present worth is $413,000 with a 24% discounted rate of return. After the initial investment is recovered, Project ORDER results in a net savings of $265,000 per year. This project is a promising process to achieve all the goals

of Task 5. It produces oil with less than 3% water content, produces maximum water yield, minimizes waste solution, avoids the use of harmful materials and is cost and energy efficient.

The health and safety of all individuals involved and the environmental impact of Project ORDER is of utmost importance throughout the construction and life of the project. The facility will ensure that all processes will comply with regulations outlined by the Environmental Protection Agency (EPA), Occupational Safety and Health Administration (OSHA), the Resource Conservation and Recovery Act (RCRA), and Texas State and El Paso County regulations. All operations and company procedures will comply with The Emergency Planning and Community Right-to-Know Act of 1986.

The following report provides a detailed proposal for an oil and water recovery system, including experimental research results, process optimization, full-scale design, economic analysis, and environmental, health and safety considerations.

INTRODUCTION

The industries that produce copper rod and copper wire utilize both metal rolling and drawing processes. Drawing processes require lubricants in order to reduce friction between the dies and the hard copper metal. Rolling processes require lubricants not only to reduce friction at the roll bites, but also to prevent metal-to-metal contact between the work rolls and the work piece, as well as to control the temperature of the work rolls[1]. However, due to the increasing price of lubricating oils, the rolling lubricants currently used in industry are oil-in-water emulsions, whose oil compositions can range from 1 to 10 weight percent. Commercial oils used to make these lubricating emulsions generally consist of a light mineral oil and a surfactant to emulsify the mixture[2].

Task 5 requires the separation of a waste emulsion from a copper rolling mill into its oil and water components. In this task, the waste emulsion is 2 v% mineral oil and 98 v% second pass reverse osmosis (RO) water, where the oil phase is actually a mixture of two different lubricating oils used in roughly equal proportions. The first mineral oil contains an amine compound that acts as an anti-oxidant and a fatty acid ester as a surfactant. The second mineral oil contains sodium 2,3-dinonylnaphthalene-1-sulfonate as a surfactant. The surfactant is pre-blended with the oils, so the preparation of the emulsion can be performed by simply combining the oil and water with agitation.

In copper rolling operations, copper particles flake off into the emulsion as the steel rolls move over the softer, hot copper metal. The fatty acid esters in the rolling oils chemically combine with the copper debris and form metallic soaps, such as copper stearate, copper oleate, or copper abietate, which results in foaming and lower efficiency of the emulsion as a lubricant in the rolling process[3]. This foaming problem is diminished by adding a foam inhibitor to the lubricant. The emulsion for this task contains calcium formate as a foam inhibitor at a ratio of 2.5 pounds per 2800 gallons.

Copper particles from the work piece also flake off into the lubricant during drawing operation, which requires the eventual filtration of the emulsion. Although both drawing and rolling operations degrade the lubricant to some extent, the degradation process for rolling operation is much more rapid and complex. This is primarily due to the high operating temperatures of the rolling process[4].

As the lubricating emulsion is applied to the rollers, it is exposed to high surface temperatures, ranging between 1,570 to 1,620 degrees Fahrenheit, in addition to the high friction loads of the rolling operation. This causes the oil to thermally and mechanically degrade, lowering the lubricity and effectiveness of the emulsion. This degradation is similar to the breakdown that occurs with motor oil in an engine. Moreover, in addition to metal flakes, the emulsion also collects copper oxides during the rolling process. The copper exposed to the air becomes oxidized, forming an undesired copper oxide film on the surface of the metal[5]. This film grows thicker due to the elevated diffusion of oxygen and continuous reactivity with metal in the open atmosphere. The emulsion reduces this effect by cleaning debris, mostly metal fines and oxides, from the surface.

As the emulsion collects copper oxides and other metal particles, it can be filtered and recycled until it is degraded to a point where it can no longer be used. Although most copper oxides can be easily filtered, there are some of the particles which are so fine that they collect at the interface between the oil droplets and the water phase, making them almost impossible to remove by filtration. The smallest particles accumulate in the emulsion over time and bridge between the steel rolls and the copper work piece, reducing the lubricating effectiveness. An obvious indication that the emulsion is no longer usable is the buildup of copper debris on the steel rolls. This debris buildup, along with oil degradation, eventually reduces the lubricating effectiveness to the extent that the emulsion must be replaced.

The emulsion cannot be discharged to the sewer; consequently other disposal methods are used. The conventional disposal method is incineration[7]. Incineration is not economical or environmentally friendly: the energy costs are high and the oil and water components are wasted. There is a need for a disposal method that recovers and recycles these valuable resources.

SITE SELECTION

Project ORDER was designed as a battery limits addition to an existing copper rolling and/or drawing process. The primary criteria used to determine a logical location for this process addition were: (1) The use of an emulsion lubricant in a copper rolling and/or drawing process and (2) the need for the recovery and reuse of valuable resources from the spent emulsion discharge.

Freeport-McMoRan Copper and Gold Inc., the world's largest publicly traded copper company, uses a similar emulsion as the lubricant in their copper drawing and rolling processes. This company's operation nearest to the University of Arkansas is located in El Paso, Texas[6]. The El Paso copper rod mill was chosen as the site location for this project, as it is one of the largest rolling and drawing operation facilities and meets the established criteria.

PROCESS SELECTION

De-emulsification Techniques

After losing its effectiveness, lubricating emulsions used in copper drawing and rolling operations must be separated to recover the oil and water components. While several methods of separation were considered, the most common techniques for separating oil-in-water emulsions are listed in Table 1.

Hydrophobic ultrafiltration, heating, and solvent dilution techniques proved ineffective in lab tests when used alone, largely due to the surfactants in the emulsion. While acidification will successfully separate the emulsion, it results in the production of undesired by-products, such as metal salts, and involves higher health and environmental risks. Also, established by lab testing, acidification results in the formation of a third, emulsified phase that would require additional separation. Although no single, viable method for complete separation was presented, hydrophilic ultrafiltration followed by another technique was determined to be a promising option.

A hydrophilic ultrafiltration (UF) membrane exhibits an affinity to water. Due to its selectivity, water in the feed emulsion can be forced by pressure to permeate the membrane. The remaining water, oil, and metal fines can be re-circulated to pass through the membrane loop again. The emulsion can then be concentrated to approximately 30 v% oil and 70 v% water. This composition allows more than 90% of the water from the feed emulsion to be recovered in the UF permeate. The remaining emulsion concentrate can then be separated more easily due to its reduced water content. Ultrafiltration is also an economical option due to its low operating pressures.

Table 1. Techniques for the Separation of Oil-in-Water Emulsions

Treatment Method	**Advantages**	**Disadvantages**
Ultrafiltration (hydrophilic)	• Allows for more than 90% water recovery • Non-chemical option for separation • Inexpensive	• Separation is usually not complete • Membrane fouling can occur
Ultrafiltration (hydrophobic)	• Complete oil recovery is completed in one step • Non-chemical option for separation • Inexpensive	• Presence of a surfactant can prevent complete separation • Membrane fouling can occur
Acidification	• Surfactant is de-activated for more effective separation	• Higher environmental and health risks • Heat is required for reaction to occur • A third phase may result, requiring additional separation • Large raw material cost
Heating	• Non-chemical option for separation	• Presence of a surfactant can prevent complete separation • High cost due to energy requirements
Solvent Dilution	• Entire oil phase is dissolved, lowering viscosity, and resulting in quick separation • Requires little or no heat.	• Higher health risk • Presence of a surfactant can prevent complete separation • High energy cost due to evaporating the solvent

Water Recovery

Hydrophilic ultrafiltration was chosen as the first step in the process due to its high recovery for water as well as its environmental and economic benefits. The water recovered from the UF membrane must then be treated by a reverse osmosis (RO) membrane unit so it can be recycled. This combination of ultrafiltration and reverse osmosis completes the water recovery technology for Project ORDER.

In order to maximize the amount of resources recovered from the spent lubricant, complete separation is required. Separation techniques for oil and copper recovery were explored.

Oil Recovery

While the oil may have undergone degradation, if it can be reduced to less than 3 v% water, oil recyclers may purchase the oil for its remaining energy content. Several technologies were evaluated to achieve the water content goal, including acidification, evaporation, hydrophobic ultrafiltration, and the use of coalescers.

The use of coalescers and hydrophobic ultrafiltration were unsuccessful in breaking both the original spent emulsion and the concentrated emulsion in lab tests. This failure is most likely due to the highly effective surfactants present in the oils. Acidification was proved in lab tests to successfully separate the emulsion, however, as mentioned previously, it entails higher environmental and health risks. Evaporation with agitation proved to be effective in lab testing, removing essentially all of the water from the ultrafiltration concentrate. Due to small concentrate volumes, energy costs are relatively low for evaporation and it is an economical option. For these reasons, evaporation was selected as the oil recovery method for Project ORDER.

Copper Recovery

Copper is an economically valuable material and should be recovered from the spent lubricant. Most of this debris is left in the recovered oil stream. In lab tests, depth filtration was shown to be a successful method for removing copper from the oil recovered by evaporation.

Copper cementation was also considered; however, this method can only be used after acid addition. Due to environmental and health concerns, this option was discarded.

BENCH-SCALE TESTING AND DESIGN

Lab Testing

The experiments performed by Project ORDER tested the effectiveness of six different emulsion separation technologies, while evaluating their viability on the industrial scale. The experimental results of each technology option are discussed below. The ultimate decision was based on four main considerations: separation effectiveness, total recovery, economic feasibility, and environmental ramifications.

1) *Thermal Separation* - The first experiment conducted tested heat as a possible means of separating the emulsion. This involved heating a sample of the spent emulsion to 200 ^{o}F

for 20 minutes. A thin, reddish-brown, liquid layer formed at the top of the emulsion, which indicated that some separation occurred. However, the bottom layer maintained the same consistency throughout the experiment which indicated that heat, alone, does not allow a complete separation.

2) *Gravity Separation* - This experiment tested the effects of gravity on emulsion separation. A sample was centrifuged from 5 to 20 minutes at 14,336 G's. This resulted in the appearance of three phases: an oily phase, the emulsion, and a small solid phase. While it was apparent that a small amount of separation had occurred, the emulsion layer remained unchanged in consistency and appearance indicating that gravity separation would not be sufficient.
3) *Acid Separation* **-** This experiment utilized acid to break the spent emulsion. The emulsion was treated with HCl to a concentration of 0.3 w% and then heated to 200 ^{o}F for approximately 40 minutes. The sample was then allowed to cool and settle for several hours. This technique was tested on both the original emulsion and the concentrate from the hydrophilic UF membrane (discussed below). This procedure appeared to result in a clean separation of the oil and aqueous layers. However, as the sample was decanted, it became apparent that a third, "rag" layer had formed in between the oil and water layers. The rag layer made complete separation extremely difficult. Several different methods were tested to handle this layer including gravity, coalescers of varying types, and solvent extraction. None of them were completely effective. The acid itself presented an additional problem with this technique. The aqueous stream from either sample would have to be neutralized and treated further before it could be recycled or disposed, creating more waste.
4) *Hydrophobic UF Membrane* - This experiment involved the use of a flat sheet polytetrafluoroethylene (PTFE) hydrophobic membrane to allow only the oil phase to permeate the membrane and, in turn, concentrate the water phase and any contaminants. This method was tested on three different samples: the original spent emulsion; the concentrate from the hydrophilic membrane (discussed below), and the oil and rag layers from an acidified sample. This technique failed to complete the desired separation for all three samples.

5) *Hydrophilic UF Membrane* - The method that produced the best results was the use of a flat sheet hydrophilic membrane to allow only the water phase to permeate and to concentrate the oil phase and contaminants. Using this membrane, over 90% of the volume of the original emulsion could be removed as permeate, leaving a concentrate that was approximately 30 v% oil. The aqueous permeate was then treated with an RO system to purify it to the point that it could be recycled.
6) *Evaporation* **–** Simply evaporating the water from the spent emulsion in its original state would not be economically feasible. However, once the emulsion was processed through the hydrophilic membrane, evaporation proved to be the most effective method for separating the remaining water from the concentrate. This process results in steam that can be immediately condensed and recycled directly, and an oil stream that can simply be filtered to remove solid copper contaminants and sent to oil recyclers.

Bench-Scale Design

The bench-scale process uses a hydrophilic UF membrane (1) to recover approximately 90 v% of the water from the original spent emulsion. The specific membrane for this unit is a flat sheet, 10,000 molecular weight cut-off, polyethersulfone membrane with an active area of 33.7 in^2. The UF unit is a commercially available bench top system with an operating pressure range of 0-75 psi and a recirculation flow rate of up to 12 liters per minute (LPM). The UF system is shown in Figure 1. The UF permeate is then processed by an RO system (2) where 90 v% of the water is recovered as RO permeate. The RO membrane in the bench-scale is a spiral wound element composed of cellulose acetate with an active area of 333 in^2. The RO system uses a 0.5hp pump capable of 1750 rpm, and this system is shown in Figure 2. The concentrate from the UF is then evaporated on a hot plate (3) until all remaining water has been removed from the oil. Finally, the oil is passed through a syringe packed with filter media (4) to remove the solid contaminants. This procedure demonstrates the ease with which it can be filtered, and produces a clean, recyclable oil product. The process flow schematic is presented as Figure 3.

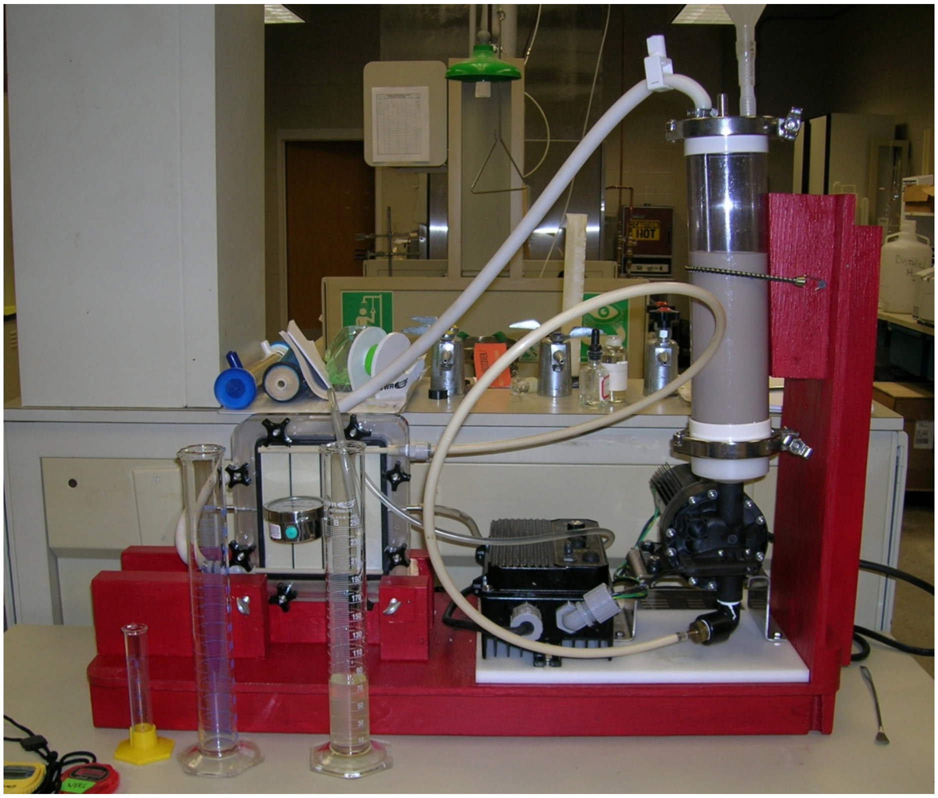

Figure 1. Hydrophilic UF Bench-scale System

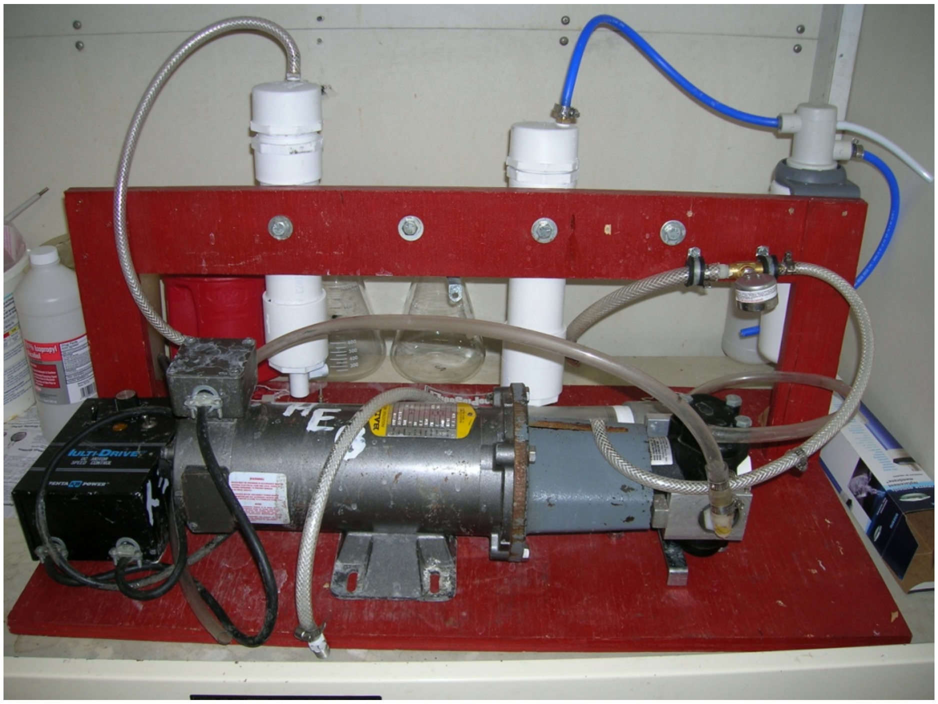

Figure 2. RO Bench-scale System

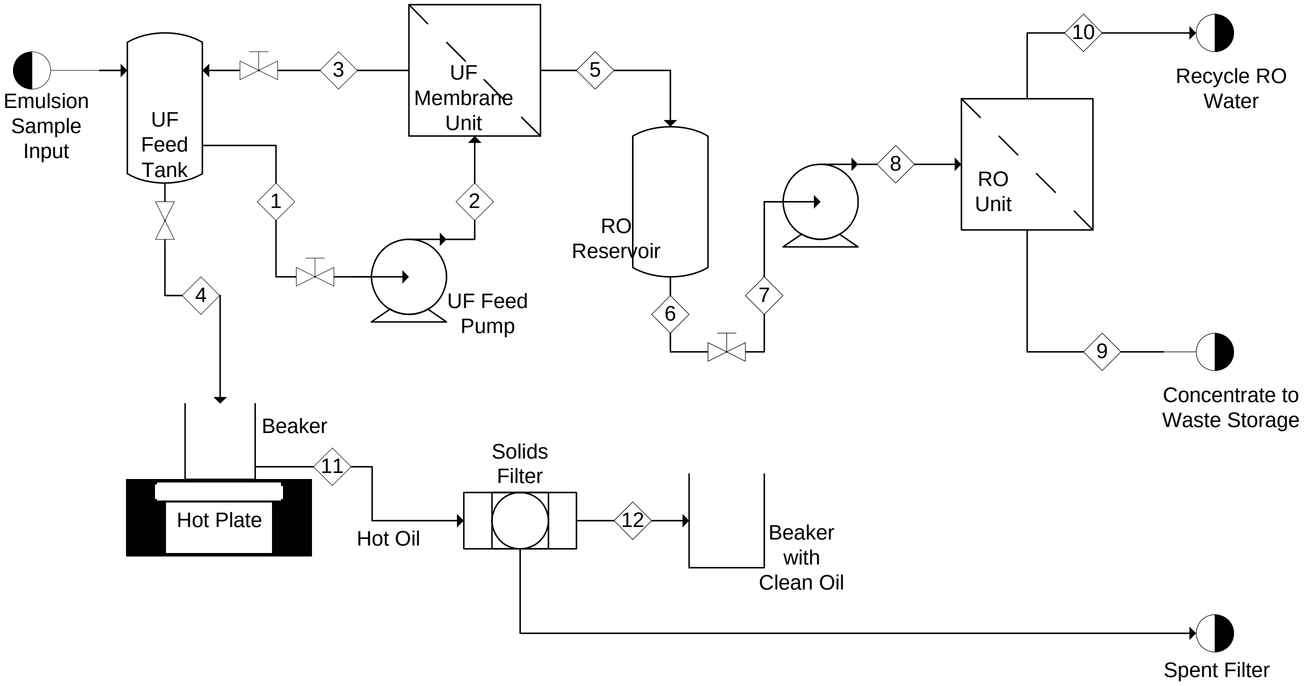

Figure 3. Process Flow Schematic for Project ORDER Bench-Scale Process

PROCESS OPTIMIZATION

RO Optimization

Major considerations affecting the design of the RO unit are the final copper and chlorine concentrations in the permeate stream. Thin-film composite (TFC) polyamide spiral elements with high rejection are the only viable membranes able to deliver ultra low concentrations of both copper and chlorine. For this small system, 4” diameter, 40” long elements provided the most economical and effective solution. Selection of the RO bank array was based on number of elements, membrane flux, permeate concentrations, and pump requirements. RO simulation software, Fluid systems ROPRO 6.0, was used to determine the optimal membrane configuration. Table 2 presents design parameters calculated by the simulation software for several potential configurations. The 4:3:2:1 case provides the most favorable operating conditions even though it does not contain the fewest number of elements. It is the optimal case due to the lower pressure and pumping requirements and thus was the chosen configuration for the Project ORDER RO unit.

Table 2. Design Parameters for RO Unit

Array	Total Elements	P (psig)	Flux (gfd)	Reject TDS (ppm)	Permeate TDS (ppm)	Beta*	Copper (ppm)	Chlorine (ppm)
1 bank	50	446.6	20.5	1374.55	1.38	1.155	0.56	0.7
2 : 1	48	382.5	21.3	1376.26	1.21	1.216	0.49	0.61
2:1:1	48	388.3	21.2	1377.17	1.1	1.347	0.46	0.57
3:2:1:1	42	480.4	24.4	1379.16	0.92	1.068	0.38	0.47
4:3:2:1	50	385.4	20.5	1377.59	1.1	1.081	0.44	0.55
5:3:2:1:1	48	396.6	21.3	1378.07	1.03	1.08	0.42	0.52

*Concentration polarization coefficient (Value should not exceed 1.13).

FULL-SCALE DESIGN

The process flow diagram is presented as Figure 4. The plant system consists of: (1) an existing spent emulsion storage tank, (2) an emulsion feed pump, (3) a UF feed tank, (4) a UF feed pump, (5) a UF membrane unit, (6) an evaporator feed pump, (7) a water evaporator, (8) an evaporated water condenser, (9) a filter feed pump, (10) an oil filter, (11) an oil cooler, (12) an oil product storage tank, (13) a permeate surge tank, (14) an RO reservoir, (15) an RO feed pump, (16) an RO unit, (17) a recovered water surge tank, and (18) an RO concentrate evaporator.

The spent lubricating emulsion is held in a 10,000 gallon, flat-bottom polyethylene storage tank. The emulsion exiting the storage tank is pumped by a 350 gpm, 50 ft head centrifugal pump into a 10,000 gallon polyethylene UF feed tank. As the emulsion is concentrated by the UF membrane unit, the concentration of oil in the UF feed tank increases from about 2 v% at the beginning of the UF batch to 30 v% at the end of the UF batch. This effectively recovers approximately 90 v% of the feed water for reuse.

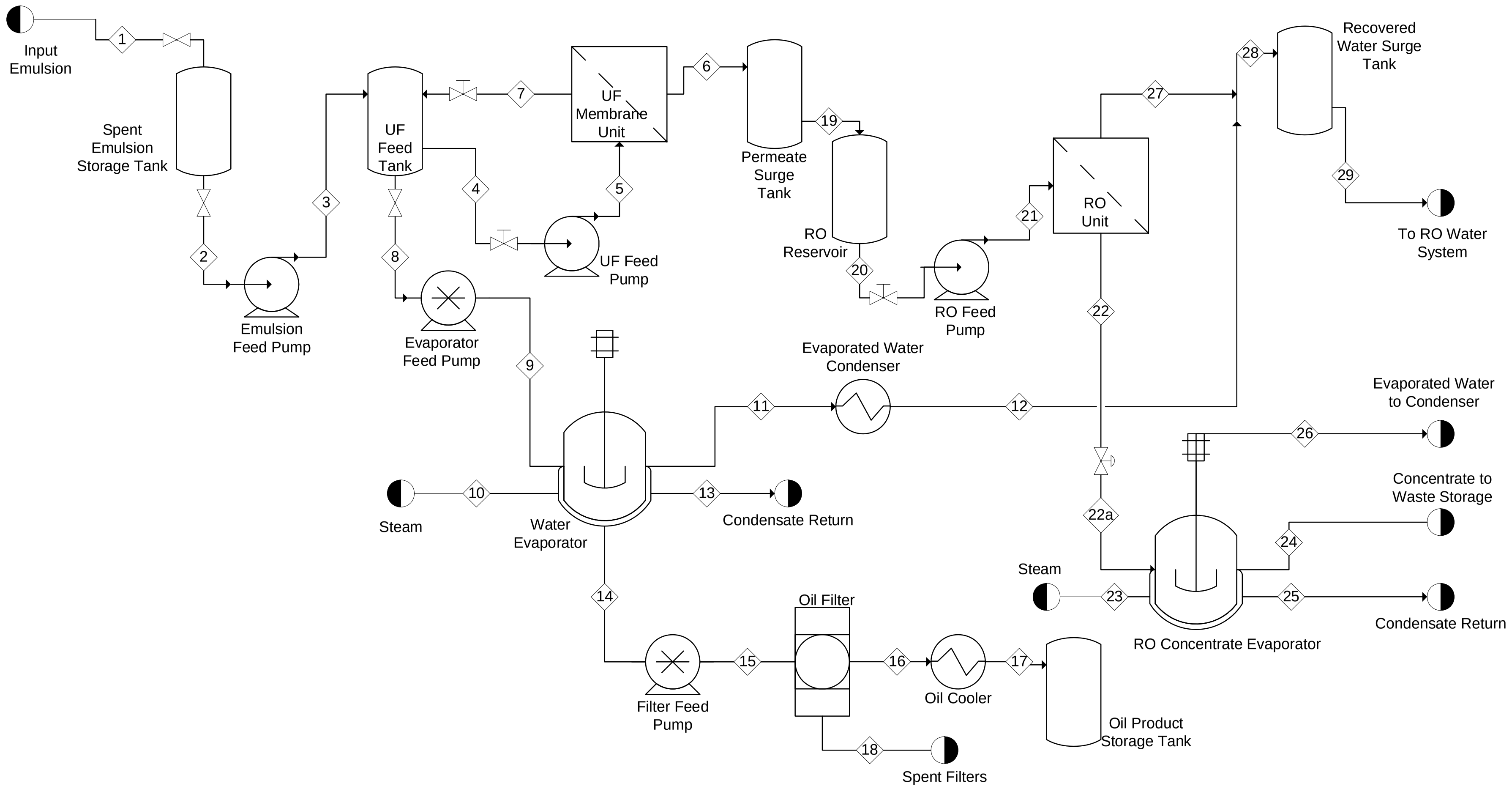

Figure 4. Process Flow Schematic for Project ORDER

Table 3. Process Flow Diagram Stream Table

	Stream Number														
Component (lb/day)	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
Water	68573	68573	68573	68573	68573	65307	3265	3265	3265	0	3265	3265	0	0	0
Oil Type 1	700	700	700	700	700	0	700	700	700	0	0	0	0	700	700
Oil Type 2	700	700	700	700	700	0	700	700	700	0	0	0	0	700	700
Calcium Formate	8	8	8	8	8	8	0	0	0	0	0	0	0	0	0
Copper (I) Oxide	31	31	31	31	31	2	30	30	30	0	0	0	0	30	30
Steam	0	0	0	0	0	0	0	0	0	253	0	0	253	0	0
Total	**70011**	**70011**	**70011**	**70011**	**70011**	**65316**	**4695**	**4695**	**4695**	**253**	**3265**	**3265**	**253**	**1429**	**1429**
Pressure (psia)	10	10	22	15	35	20	20	20	30	50	35	30	30	20	30
Temperature (F)	100	100	100	100	100	100	100	100	100	300	212	100	212	275	275

	Stream Number														
Component (lb/day)	**16**	**17**	**18**	**19**	**20**	**21**	**22**	**22a**	**23**	**24**	**25**	**26**	**27**	**28**	**29**
Water	0	0	0	65307	65307	65307	6531	6531	0	65	0	6465	58776	62042	62042
Oil Type 1	700	700	0	0	0	0	0	0	0	0	0	0	0	0	0
Oil Type 2	700	700	0	0	0	0	0	0	0	0	0	0	0	0	0
Calcium Formate	0	0	0	8	8	8	8	8	0	8	0	0	0	0	0
Copper (I) Oxide	0	0	30	2	2	2	2	2	0	2	0	0	0	0	0
Steam	0	0	0	0	0	0	0	0	465	0	465	0	0	0	0
Total	**1399**	**1399**	**30**	**65316**	**65316**	**65316**	**6540**	**6540**	**465**	**75**	**465**	**6465**	**58776**	**62042**	**62042**
Pressure (psia)	25	15	0	18	15	380	335	10	50	35	35	30	15	25	20
Temperature (F)	275	150	100	100	100	100	100	100	300	275	212	212	100	100	100

Water is removed batch-wise, over a 12-hour cycle, from the spent emulsion by circulating the emulsion through the membrane loop. A batch process was selected over a continuous feed-and-bleed process due to varying production volumes with a maximum of 8,400 gal/day. In the event that the production volume for a day is less than 8,400 gallons, the emulsion will be stored until it reaches this volume. The emulsion is pumped by the UF feed pump at a pressure of 35 psig and 350 gpm. The UF membrane is a 10,000 molecular weight cutoff polyethersulfone spiral-wound membrane with a required area of 1,100 ft^2. The 8" diameter by 4' long selected elements each have 200 ft^2 of active area, so six elements are required. This hydrophilic membrane allows water, calcium formate, and dissolved salts to permeate the membrane.

At the end of a UF batch, the UF feed tank contains 280 gallons of concentrated emulsion. This emulsion is pumped at 23 gpm by the evaporator feed pump into the water evaporator.

The water evaporator is an agitated, jacketed, steam-heated (50 psig steam), 400 gallon vessel. Water is evaporated from the UF concentrate over a 12-hour period at which time the batch temperature starts rising above 212 F. When the batch temperature reaches about 275 F, all the water has been removed and the evaporator batch is finished. Because the remaining oil contains large amounts of metal debris, the heated batch is then pumped through the oil filter. The filter feed pump is a positive displacement gear pump with a pressure capability of 100 psig.

The polyester depth filter element is 4.5" diameter by 50" long with a pore size of 1 micron. At 7 gpm, the pressure drop in a fresh filter is approximately 1.5 psig. As solids are collected, the pressure drop will rise and the element will be changed when the pressure drop exceeds 25 psig. The spent filter and collected solids will be sent to waste disposal.

For safety reasons, the hot filtered oil is cooled by the oil cooler to below 150 F prior to being sent to the storage tank. The oil cooler is a shell-and-tube heat exchanger with a required area of 52 ft^2 with a duty of 267,968 BTU/hr. It operates at an approximate feed rate of 60 lb/day and over a 20-minute period.

After leaving the cooler, the oil is sent to the oil product storage tank, which collects 170 gallons of oil each day. The oil product storage tank is a carbon steel tank with a capacity of 3,500 gallons. Because an oil recycling truck can transport up to 3,200 gallons of oil, the recovered oil from each service is stored in the storage tank until the volume reaches nearly 3,200 gallons. A pickup will be scheduled every two weeks or as needed.

The evaporated water is condensed by a shell-and-tube heat exchanger with a required area of 16 ft^2 and a duty of 272,113 BTU/hr using 54 gpm of cooling water. The condenser reduces the temperature of the water to about 100 F. After being condensed, the liquid water combines with the water permeate from the RO unit and is recycled to produce fresh emulsion.

The water permeate from the UF membrane is stored in the 9,000 gallon polyethylene tank and later enters the 9,000 gallon polyethylene RO reservoir, which allows the water to be continuously pumped through the RO unit. The purpose of the RO unit is to increase the purity of the UF permeate water by removing salts. The water is pumped by the RO feed pump at 50 gpm, 1155 ft head, and 380 psig. The RO membrane is a proprietary TFC polyamide spiral-wound membrane with a fiberglass outer wrap. This system requires 50 elements with 64 ft^2 of active membrane area and provides a chloride rejection of 99.6% as well as a copper rejection of 99.5%.

The RO permeate combines with the condensed water from the evaporator and is stored in the 9,000 gal polyethylene water surge tank. Ten percent of the UF permeate, consisting of water, copper chloride, and other salts, enters the RO concentrate evaporator where 99% of the concentrate is evaporated.

The RO concentrate evaporator is an agitated, jacketed, steam-heated (50 psig steam), 1,000 gallon vessel. It is operated with a 600 gal heel to insure that a proper amount of heat transfer area is maintained. Only 50 gallons of fresh feed is added every hour to the evaporator and the temperature rises above 212 F as the water is evaporated. When the batch temperature reaches 275 F, all the water is removed and the evaporator batch is finished. The remaining concentrate is purged at a rate of 50 gallons per hour and can be sent to disposal.

ECONOMIC ANALYSIS AND BUSINESS PLAN

As mentioned previously, incineration is the current disposal method for the spent lubricant. Based on a spent emulsion production rate of 8,400 gal/day and a batch time of 12 hours, the energy required to incinerate the emulsion is 5.8 million BTU per hour. Therefore, utilizing incineration, disposal costs are $792,000 per year[7]. In addition to being uneconomical, incineration wastes the valuable water and oil components of the emulsion. After the initial investment is recovered, Project ORDER results in a net savings of $265,000 per year.

Project ORDER recovers 90% of the water from the spent emulsion, which amounts to 7,448 gallons per day. The cost of water in El Paso after the first 2,000 gallons is $0.0061/gallon[8]. Therefore, recovering 90% of the water saves $45 per day, or $17,000 per year.

The majority of the lubricating oil is recovered from the emulsion, which is approximately 170 gallons per day. Used oil can be sold to oil recyclers for about $0.10 per pound[9]. This results in $51,000 additional revenue per year. Total revenue for Project ORDER is $861,000 per year including the avoided incineration and water costs.

Equipment prices were obtained through manufacturer and literature estimates and total $217,000. Utility costs are $18,000 per year. Operating cost is $528,000 per year, with $200,000 of the cost for operating labor. Fixed capital investment is $899,000 and working capital is $47,000.

Several assumptions were made in order to complete the economic analysis. The tax bracket for a company is determined by overall profit, thus it can vary widely. A tax rate of 35%, a project life of 20 years, and a plant startup period of 2 years[10] were assumed. It was also assumed that 60% of the fixed capital investment will be spent in the first year of startup, with the additional 40% used in the second year. The 6-year MACRS depreciation method and a discount rate of 15% were used for the analysis.

The cost of the project will be recovered in 5.5 years according to the discounted payback period calculated. Furthermore, the net present value of the entire project is $413,000. Through this economic analysis, it has been determined that this project is economical and environmental friendly. All costs for Project ORDER are summarized in Table 4.

Economy of Scale

In addition to evaluating economic costs for a spent emulsion production rate of 8,400 gal/day, Project ORDER also determined the minimum operating scale for this process. Minimum operating costs for this process are approximately $500,000 per year. At a production rate of less than 5,400 gal/day, it is more economical to incinerate the spent emulsion.

Table 4. Summary of Project Costs

Delivered Equipment Cost	*Material*	*Capacity/Area*	*Unit Cost $*
UF Feed Tank	polyethylene	10000 gal	5,267
Permeate Surge Tank	polyethylene	9000 gal	4,379
RO Reservoir	polyethylene	9000 gal	4,379
Water Evaporator (w/ jacket & agitator)	stainless steel 304	400 gal	12,000
Recovered Water Surge Tank	polyethylene	9000 gal	4,379
Oil Product Storage Tank	carbon steel	3500 gal	4,500
RO Concentrate Evaporator (w/ jacket & agitator)	stainless steel 304	1000 gal	20,000
Oil Cooler	stainless steel	62 ft^2	7,439
Evaporated Water Condenser	stainless steel	21.2 ft^2	3,480
Emulsion Feed Pump (centrifugal) A/B	bronze	390 gpm at 50' head	4,422
Evaporator Feed Pump (positive displacement) A/B	cast iron	23 gpm	1,098
UF Feed Pump (centrifugal) A/B	bronze	390 gpm at 50' head	4,424
RO Feed Pump (centrifugal) A/B	stainless steel	50 gpm at 1155' head	15,482
Filter Feed Pump (positive displacement) A/B	cast iron	7 gpm	1,256
Oil Filter (depth filter)	polyester fiber	7 gpm	535
Ultrafiltration (spiral-wound) [automated]	polyethersulfone	1100 ft^2	30,327
RO (spiral-wound) [automated]	proprietary TFC polyamide	73 ft^2	93,333
Subtotal			216,701
Equipment Installation Direct Cost	*Cost Estimation Basis*[11]		*Cost $*
Purchased Equipment Installation	30% Delivered Equipment Cost		65,010
Electrical Systems Installed	11% Delivered Equipment Cost		23,837
Services Facilities Installed	15% Delivered Equipment Cost		32,505
Equipment Delivery	10% Delivered Equipment Cost		21,670
Instrumentation & Controls	8% Delivered Equipment Cost		17,336
Building Services	18% Delivered Equipment Cost		39,006
Piping Systems	68% Delivered Equipment Cost		147,357
Subtotal			346,721
Additional Project Cost	*Cost Estimation Basis*[11]		*Cost $*
Engineering and Supervision	33% Delivered Equipment Cost		71,511
Construction Expense	41% Delivered Equipment Cost		88,847
Buildings (including services)	6% Delivered Equipment Cost		13,002
Site Preparation	5% Delivered Equipment Cost		10,835
Legal Expense	4% Delivered Equipment Cost		8,668
Contractor's Fee	22% Delivered Equipment Cost		47,674
Contingency	44% Delivered Equipment Cost		95,348
Subtotal			335,886

Fixed Capital Investment		899,309
Working Capital	5% of Total Capital Investment	47,332
Total Capital Investment		946,641

Operation Cost excluding Utilities	*Cost Estimation Basis*			*Cost $*
Maintenance	2% Delivered Equipment Cost[11]			4,334
Insurance	1% Fixed Capital Investment[11]			8,993
Laboratory Charges	Sample testing cost			10,000
Operating/Cleaning Supplies	15% Maintenance Cost[11]			650
Filters/Membrane	Replacemtent Cost			24,112
Membrane Cleaning	Cleaning solution and water			20,793
Waste Treatment	RO Concentrate Evaporator Waste/Oil Filter			13,200
Subtotal				82,082
Utility Cost	*Required*	*Unit Cost $* [10]	*Daily $*	*Yearly $*
Water Evaporator (LP Steam)	253 lb/day	16.22/1000kg	1.86	679
Oil Cooler (Cooling Water)	6297480 Btu/day	0.354/GJ	2.35	858
Evaporated Water Condenser (Cooling Water)	6530712 Btu/day	0.354/GJ	2.44	890
Emulsion Feed Pump (Electric)	7.5 hp	0.06 kWh	8.06	2,940
Evaporator Feed Pump (Electric)	3.5 hp	0.06 kWh	3.76	1,372
UF Feed Pump (Electric)	7.5 hp	0.06 kWh	8.06	2,940
RO Feed Pump (Electric)	17.5 hp	0.06 kWh	18.80	6,861
Filter Feed Pump (Electric)	1.5 hp	0.06 kWh	1.61	588
RO Concentrate Evaporator (LP Steam)	465 lb/day	16.22/1000kg	3.42	1,249
Subtotal			50.34	18,376
Labor Costs	*Cost Estimation Basis*			*Cost $*
Operators	$50,000/yr x 4			200,000
Worker Benefits	25% of operating labor cost			50,000
Additional Supervision Cost	15% of operating labor cost[11]			30,000
Subtotal				280,000
General Expenses	*Cost Estimation Basis*[11]			*Cost $*
Financing	5% of Fixed Capital Investment			44,965
Plant Overhead	50% of operating labor and maintenance			102,167
Subtotal				147,132

Total Yearly Cost	$527,590

REGULATORY CONSIDERATIONS

Project ORDER will conduct business in a manner which promotes environmental quality, employee safety, and community awareness. Project ORDER will ensure that all processes comply with regulations outlined by the Environmental Protection Agency (EPA), Occupational Safety and Health Administration (OSHA), Resource Conservation and Recovery Act (RCRA), and Texas State and El Paso County regulations.

Environmental

After an environmental review is conducted by the EPA, the plant will either: (1) be required to submit an Environmental Impact Statement (EIS) or (2) be issued an FNSI (Findings of No Significant Impact) in the case that this project has no significant impact on the environment. As Project ORDER does not present a significant environmental impact, an EIS will not be required.

The standards for the management of used oil are outlined in section 3014 of RCRA and part 279 of title 40 of the Code of Federal Regulations (CFR)[12]. The recovered oil must meet the specifications outlined in the CFR to be recycled, and these specifications are summarized in Table 5. The oil recovered through Project ORDER bench-scale testing has been proven to meet these specifications. In the full scale process, regular sample testing will be performed in order to ensure compliance with these specifications.

Table 5. Used Oil Specifications for Recyclers[12]

Constituent/property	Allowable Level
Arsenic	5 ppm maximum
Cadmium	2 ppm maximum
Chromium	10 ppm maximum
Lead	100 ppm maximum
Flash point	100 °F minimum
Total halogens	1000 ppm maximum
Note: Applicable standards for the burning of used oil containing PCBs are imposed by 40 CFR 761.20(e).	

The recovered oil, evaporated RO concentrate, and the spent oil filters will be taken by a licensed waste disposal or recycling firm, which will have the responsibility of handling the material according to all federal, state, and local regulations. Project ORDER will investigate the disposal methods used by the waste disposal firm to insure that they meet all federal, state, and local regulations.

Health and Safety

Regulations set by the OSHA Department of Labor in Title 29 of the CFR will be followed. Employees will be required to complete all safety training relevant to their position in early employment. Workers will also be required to repeat safety training on an annual basis or as required, since procedures may change. The most valuable resource is the personnel, so safety and health programs are considered an investment. It is also recognized that compliance with regulations alone will not ensure the highest attainable safety standards. Safety and health considerations will be integrated into all other functions of the organization.

OSHA requires that all personnel be trained on chemical safety, emergency procedures, and OSHA guidelines. In addition, OSHA requires that Material Safety Data Sheets (MSDS) for each chemical must be on site and available at all times. The facility is required to provide personal protective equipment (PPE) for all operators and maintainers, as outlined by 29 CFR 1910[13]. Employees will be required to wear appropriate PPE in all operating areas, and it will be made readily available at all times and kept in sanitary condition. All equipment will be properly isolated and grounded to remove electricity hazards. Proper safety techniques will be provided through instruction and followed by all personnel.

Community Right-to-Know

Congress passed the Emergency Planning and Community Right-to-Know Act of 1986 (EPCRA) in order to provide United States citizens with information regarding exposure to hazardous chemicals. Regulations regarding the management of toxic substances are outlined in title 40 of the CFR and under section 302 of the EPCRA and the subsequent reporting requirements of sections 304 and 311-313.

Chapter 116 under title 40 of the U.S. code for Community Right-to-Know will be satisfied. MSDS's and other appropriate forms for any hazardous chemicals listed under the OSHA Act of 1970 [29 U.S.C. 651 et seq.] used in the process will be submitted to the appropriate local emergency planning committee, the state emergency response commission, and the designated fire department. This information will also be made available for anyone, public or private, who requests the information. There are currently no hazardous chemicals used in Project ORDER, but these guidelines will be followed if any future chemical additions are made. At the completion of the process addition, the fire department will conduct an inspection of the site and be directed to the specific locations of hazardous chemicals (if present) at the facility.

CONCLUSIONS AND RECOMMENDATIONS

1) Project ORDER has determined that ultra-filtration followed by evaporation is the most cost effective and environmentally conscious method of separating the emulsion.
2) This specific process recycles essentially all of the water and virtually all of the oil.
3) The opportunity also exists for recovery of the copper that is filtered from the oil following evaporation of the water.
4) The Project ORDER process can be constructed as an addition to an existing mill without impairing the current established processes.
5) The recovered oil meets the specifications outlined in part 279.11 of title 40 of the CFR; therefore, the oil is acceptable for recycling.

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March 15, 2008

Ms. Jessica Nichols
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Department of Chemical Engineering
Fayetteville, AR 72701
email: jenicho@uark.edu

Re: WERC Audit: Oil recovery through de-emulsification research (aka Project **ORDER**)

Dear Ms. Nichols,

Your report is well-written and easy to follow. There are some grammatical faux-pas, which I'm sure you'll find in a final editing. Your overall work presents a degree of process engineering sophistication that is unusual in undergraduate students. I commend you and your team on the accomplishment. In general, the process design seems workable...is someone planning on piloting it?

You requested that I review your draft report with special attention to economic, health, and legal issues. I am not an expert in any of the three areas you mentioned, what follows are my stream of thoughts as I read through the report.

The summary contains some hyperbole. One should always be mindful of the uncertainty involved and express your numerical assessments without adjectives such as maximum and minimum, which imply absolutes. Be consistent referring to Task 5 or Task five in your text. You refer to sodium sulfonate as a surfactant, do you actually mean sodium lauryl sulfate, or some generic alkyl aryl sodium sulfonate? Did your lab tests include emulsion samples with metal particulates (it was uncertain from the discussion)? Particulates (especially metal fines) could cause material damage to membranes in crossflow filtration. It is likely that these particles would need to be removed if present. Tests of centrifugation requires the radius dimension in order to actually calculate the force exerted on the colloidal particles, the centrifuge's rpm alone is insufficient to determine whether this processing approach is viable.

In the Process optimization section you should identify what RO simulation software you used. What is "β" in Table 2? Did you include an allowance for membrane flux decline and cleaning?

In Full-scale design section, what about the cooling duty for the water condenser and the oil cooler? Both of those could be <u>new pieces of equipment</u>. Why choose a batch process, versus a continuous, feed-and-bleed? Will the overhead from the evaporation processes contain volatile organics? If so, the condensed water would need a further polishing.

You should provide an estimated material and energy balance including the temperatures and pressures on your process flow diagram.

I know that there is some debate on this issue but, most of the rest of the world is metric, and the US government requires metric units, so intellectual rigor suggests that you should present your results in metric units (with English in parenthesis if you so desire).

In the Economic analysis and business plan, isn't the water for creating the emulsion supposed to be RO-permeate quality, not out-of-the-tap, so the value of recovered water may be higher. The total revenue quoted doesn't follow from the two inputs discussed, so the reader becomes confused. You should provide a separate graphic that plots the cumulative costs and credits over time, that makes it easier to identify the economic payback and cash flow.

Referring to the items in Table 3: i) one usually puts in spares of key pieces of equipment with moving parts (like pumps); ii) utility costs should be part of a separate section on operating costs (note: kWh, not KWh) and are these the yearly or per batch costs, or?; iii) is that membrane replacement cost per year?...seems high...membranes are usually cleaned and would have at least a 5 year lifetime, or 3 years in harsh duty; iv) shouldn't the operators be getting benefits, that is, indirect costs (retirement, medical, vacation and sick leave) besides supervision; and v) costs of laboratory analysis seem low, unless the lab equipment and facility already exists at that location.

You really need to include a table of your key assumptions both process design-based, and economic analysis, and provide some rationale as to why you used it. For example, why a 15% discount rate?

In the Environmental, Table 3 is 4, etc. How does Project ORDER present greater community health risks (that is what you said)? It would seem that it presents less than existing practices (air polution due to incineration).

Your Conclusions state that 99% of the water is internally recycled, but the Summary said 90% is recovered?

References: When citing online sources it is customary to include the date accessed because internet content is ephemeral. Given a date, it is possible to retrieve sources from content archival services.

Please contact me if you need any clarification on the points I've mentioned above. Good luck to you and your team.

Best regards,

John Pellegrino
Res. Assoc./Adj. Prof.

Southwire Company, One Southwire Drive, Carrollton, Georgia 30119, USA

Jessica,

Since March 11th, I only had a chance to briefly scan your report. The deadline of 3/17 is not something by which I can generate a written review/audit in the example format.

The following, however, are some random suggestions:

1) The El Paso facility has a relatively very high use of lubricant, reported at 8400 gal/day. One might conclude this is the result of the size (annual capacity) of the El Paso operation. Other rod mills may not use 8400 gal/day. Consequently, you might consider further evaluating the treatment rate capacity vs project economics. For example, what would the project economics be at a use rate of, say, 200 gals of the 2% emulsion per day? Would a perceived wider facility need for Project O.R.D.E.R. diminish?

2) At the El Paso facility there might be uses for the UF produced water {i.e., as is} without the need for the RO. Such might bear investigating to reduce needed capital and emulsion treatment operating expenses.

3) The 2 to 6 months reported as the time for an emulsion solution to degrade may perhaps be rapid relative to experience at some other copper rod mills.

4) The oil surge tank's capacity in relationship to the recycling truck's transport capacity should be evaluated. Perhaps several extra days' 170 gal/day collection rate should be added to the size of the oil surge tank. Under the conditions specified, the transport truck must make a pick up within one day of 3000 gallons being produced, else the treatment must cease because the oil surge tank will be overfilled.

5) I suggest not reporting "All of the lubricating oil is recovered from the emulsion"

For example, there will be some residual oil in the spent filters

6) I suggest a minor rearrangement in the Summary of Project Costs by moving the section listing Utility Cost after Working Capital and Total Capital Investment sections. The Utility Cost is a figure in the summed Total Yearly Cost. This was somewhat difficult for me to follow until I examined the figures and their approximate sums closely.

7) I have concern the necessary "regular" sampling to assure compliance with the specifications of 40 CFR 279.11 will exceed the laboratory charges listed as $600 yearly. A stated sampling frequency that will be necessary might help alleviate my concern. However, should sampling be necessary for each truck shipment $600 may vastly under state the annual cost of testing.

Good luck with the competition and the career you pursue.

David Hutcheson

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Seth W. Snyder, Ph.D.
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18 March 2008

WERC Task 5 Team
Ralph E Martin Department of Chemical Engineering
University of Arkansas
Fayetteville, AR 72701

Dear Team

Jessica Nichols requested that I audit the report: "PROJECT O.R.D.E.R. - Oil Recovery through De-Emulsification Research". The project is focused on developing a commercially viable process to recover lubricating oils, water, and metals in the copper processing industry. Water management is industrial processes is growing as an economic and environmental concern. The type of issues addressed in this report will emerge in many other settings.

The critical technical challenge is to achieve a highly efficient process for handling and separating the emulsions. The team considered several potential systems including a range of membrane processes, gravity driven, chemical and temperature treatments. In the analysis the team considered overall recovery efficiency, capital and operating costs, energy use, physical space, and environmental and societal impacts.

Taken together the team did a very thorough job considering technological solutions. The work included initial laboratory testing where they evaluated comparative advantages and disadvantages of the proposed processes. After considering the experimental results, the team developed a preliminary plant design optimized for efficient oil, water, and metals recovery. To the best of my knowledge, the systems were selected based on meeting all environmental requirements whiles maximizing economic return. I was very impressed with the ability of the students to carry the project from a process optimization through process flow schematic, and economic analysis and business plan. As the move from the academic setting into their graduate educations or professional careers they will be well prepared to join teams and become immediate contributors.

I include a few pointers to improve the report and the analysis.

<u>Suggested modifications to the report:</u>

All reports are limited in space. The information presented in the Full Scale Design section should be presented in a spreadsheet or table format. The narrative should focus on the important issues in the design.

The author should not assume that the reader knows all terms. Process terms were defined but others such as "TFC polyamide spiral elements" were not. In general the authors should reference materials or provide names of vendors.

The never defined the role of “Task 5” in terms of a whole project. The auditor was not clear if there were other Tasks that had to coordinate with Task 5 or if the other tasks were focused on different projects.

Suggested modifications to the analysis:

The experimental section does not specific if the work was done on actual process samples or on synthetic samples.

The number of runs, uncertainties, and ranges were not provided for any of the experimental runs.

Time and costs for piloting the technologies weren’t considered.

The economic impact for a single plant was provided. There was no information provided on the potential for similar installations at other plants.

Membrane fouling was identified as a potential challenge. Frequency of membrane cleaning cycles was not provided. A cost of 15 % was considered for membrane replacements. This suggests membrane lifetimes of 6 – 7 years. That might be too long.

Summary:

The Task 5 team presented a strong report surveying potential solutions to an important environmental issue in the metal processing industry. The team considered the merits and challenges to several technology solutions, and based on limited experimental results, designed a process that should meet the environmental and society requirements while providing maximum economic return.

About the auditor:

Seth Snyder received a B.A. from the University of Pennsylvania and a Ph.D. from the University of Virginia. After a postdoctoral fellowship, he worked in the pharmaceutical industry. He joined Argonne in 1998 and after three years in administration took over leadership of the Chemical and Biological Technology Section in the Energy Systems Division. After a consolidation in 2008 he also took over leadership of the Process Technology Section. His work focuses on scaling process engineering systems from the bench through the demonstration scale. The work incorporates fermentation, enzymatic conversion, and membrane processes into water treatment, biofuels, and CO_2 management.

Sincerely,

Seth Snyder

Seth W. Snyder, Ph.D.