Memorandum

Date: August 18, 2017

To: Lab Group

From: Terry A. Ring, Experiment Supervisor

Subject: Ring Experiment I-8 - Shell and Tube Heat Exchanger

The shell and tube heat exchanger in the laboratory has not been used for several months. Beehive Engineering would like you to measure the fouling resistance in this unit so that it can be used for a new design. To measure the fouling resistance you will need to first determine the overall heat transfer coefficient for the transfer of heat from the jacket to the liquid inside the shell. The wall conduction and inside and outside heat transfer resistances must be determined by predictions so that they can be subtracted from the overall heat transfer coefficient leaving the fouling resistance. In this process, there are errors in experimental measurements and errors in the various predictions which will have an effect on the accuracy of the fouling resistance.

For your safety review meeting you will have to establish a protocol for these measurements and a dimensionless number correlation for the shell side and tube side heat transfer coefficient that is reasonable for this type of equipment. Note, your Reynolds numbers may not be in the fully turbulent range. Also be prepared to discuss the propagation of error in all of the calculations needed for this lab.

For you laboratory report, you should compare your experimental results for the inside and outside heat transfer coefficients with theoretical correlations to generated credibility for using them in determining the fouling resistance. The final report should also clearly report the fouling factor.

Finally using this fouling factor and this shell and tube heat exchanger determine the flow rate for SiH_2Cl_2 which enters as a vapor at it boiling point at 10 psig and is condensed and cooled to -55 C using Syltherm XLT as the coolant operating from -95C to -60C. see

http://www.loikitsdistribution.com/files/sylther-xlt-technical-data-sheet.pdf

Be prepared in your oral quiz to address the following:

a) Safety issues with this experiment

b) Equipment operation

c) Data sheets

d) Other germane points with respect to this experiment discussed above

Please include this assignment in your report as an appendix but do not cite it in the body of your report.

Date: August 18, 2017
To: Lab Group
From: Terry A. Ring, Experiment Supervisor

Subject: Ring Experiment I-9 – Fin Fan Heat Exchanger

There are multiple ways to control the heat exchanged in a fin-fan heat exchanger. We want to take a look at two ways in this lab – one is to vary the air flow rate and the other is to vary the water flow rate. To be useful at various scales of operation for design purposes, you will need to understand the impact of these flow rates on the overall heat transfer coefficient. Develop a set of experiments varying the flow of water and the flow of air in the fin-fan apparatus. Use this data to determine 2 correlations one for the inside and the other for the outside heat transfer coefficients for both the stainless steel and the copper tubes. Compare your correlations to those available in the literature.

Use the best correlations you have found in this experiment to design a Fin-Fan unit to cool 260 lbmole/hr at 410 psia of a mixture given below from 330F to 120F before it goes into a flash followed by a distillation column.

Component	Mole %
H2	52
Silicon Tetrachloride	35
Trichlorosilane	13

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Memorandum

Date: August 18, 2017
To: Lab Group
From: Terry A. Ring, Experiment Supervisor
Subject: Ring Experiment I-10 – Glass Lined Reactor (HX)

The client needs a heat transfer correlations for a glass-lined reactor of odd geometry. Using the glass lined reactor determine the heat transfer coefficient for the transfer of heat from the steam jacket to the liquid inside the reactor and from the cooling water jacket to the liquid inside the reactor. Please make these measurements with the baffle in place and at various liquid levels in the vessel and stirring rates. For your oral exam you will have to establish a protocol for these measurements (including accuracy assessment) and a correlation (of the type Nusselt Number versus Reynolds number) from the literature that is reasonable for this type of equipment.

Based upon a reaction the saponification of ethyl acetate

Et-Ac + NaOH → NaAc + Et-OH
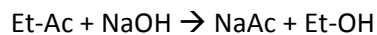
determine a design for this glass-lined reaction using this reactor. The kinetics of this reaction is reported in Hovarka, R.B. and Kendall, ;H.B. "Tubular reactor at low flow rates" CEP56(8),58-62(1960). In equimolar experiments they found this reaction to be second order overall. This reaction conversion may be limited by either kinetics or heat transfer. Assuming 1 M feed of both reactants and a conversion of reactants to the product of 0.85, determine the capacity (kg/hr of NaAc that the glass-lined reactor can produce.

Be prepared in your oral quiz to address the following:

a) Safety issues with this experiment

b) Equipment operation

c) Data sheets

d) Other germane points with respect to this experiment

Please include this assignment in your report as an appendix but do not cite it in the body of your report.

Beehive State Engineers

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Memorandum

Date: August 18, 2017
To: Lab Group
From: Terry A. Ring, Experiment Supervisor

Subject: Ring Experiment I-11 – Catalysis Experiment - Water gas shift rxn

The client needs to test a catalyst they have developed for the water gas shift (WGSR) reaction.

$$CO + H_2O \rightleftharpoons CO_2 + H_2$$

They would like you to determine a rate expression for this reaction on their proprietary catalyst and compare it to a very cheap substitute rusty Iron Grit that they are considering for a 3rd World client who struggled to pay for engineered catalysts. Your rate expression should be compared to those found in the catalysis literature. The following information on the reaction mechanism and accompanying figure comes from Wikipedia (Water-Gas Shift) page (https://en.wikipedia.org/wiki/Water-gas_shift_reaction).

"The water gas shift reaction is a moderately exothermic reversible reaction. Therefore with increasing temperature the reaction rate increases but the conversion of reactants to products becomes less favorable.[6] Due to its exothermic nature, high carbon monoxide conversion is thermodynamically favored at low temperatures. Despite the thermodynamic favorability at low temperatures, the reaction is kinetically favored at high temperatures. The water-gas shift reaction is sensitive to temperature, with the tendency to shift towards reactants as temperature increases due to Le Chatelier's principle. Over the temperature range 600 – 2000 K, the logarithm of the equilibrium constant for the WGSR is given by the following equation:[3]

$$\log_{10} K_{eq} = -2.4198 + 0.0003855T + \frac{2180.6}{T}$$

Two main mechanisms have been proposed: an associative 'Langmuir–Hinshelwood' mechanism, and a regenerative 'redox' mechanism. While the regenerative mechanism is generally implemented to describe the WGS at higher temperatures, at low temperature both the redox and associative mechanisms are suitable explanations.[7]

The typical composition of commercial Low Temperature Shift (LTS) catalyst has been reported as 32-33% CuO, 34-53% ZnO, 15-33% Al_2O_3.[3] The active catalytic species is CuO. The function of ZnO is to provide structural support as well as prevent the poisoning of copper by sulfur. The Al_2O_3 prevents dispersion and pellet shrinkage. The LTS shift reactor operates at a range of 200°C to 250°C. Low reaction temperatures must be maintained due to the susceptibility of copper to thermal sintering. These lower temperatures also reduce the occurrence of side reactions that are

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observed in the case of the High Temperature Shift (HTS) reaction. Noble metals such as Pt supported on ceria have been extensively used for LTS.[4]

The typical composition of commercial HTS catalyst has been reported as 74.2% Fe_2O_3, 10.0% Cr_2O_3, 0.2% MgO (remaining percentage attributed to volatile components).[5] The chromium acts to stabilize the iron oxide and prevents sintering. The operation of HTS catalysts occurs within the temperature range of 310°C to 450°C. The temperature increases along the length of the reactor due to the exothermic nature of the reaction. As such, the inlet temperature is maintained at 350 °C to prevent the exit temperature from exceeding 550 °C. Industrial reactors operate at a range from atmospheric pressure to 8375 kPa.[5]

In 1920 Armstrong and Hilditch first proposed the associative mechanism. In this mechanism CO and H_2O are adsorbed onto the surface of the metal catalyst followed by the formation of an intermediate and desorption of H_2 and CO_2. In the initial step, H_2O dissociates into a metal adsorbed OH and H. The hydroxide then reacts with CO to form a carboxyl or formate intermediate which subsequently decomposes into CO_2 and the metal adsorbed H, which ultimately yields H_2. While this mechanism may be valid under Low Temperature Shift conditions, the redox mechanism which does not involve any long lived surface intermediates is a more suitable explanation of the WGS mechanism at higher temperatures.

The regenerative 'redox' mechanism is the most commonly accepted mechanism for the WGSR. It involves a regenerative change in the oxidation state of the catalytic metal. In this mechanism, H_2O is activated first by the abstraction of H from water followed by dissociation or disproportionation of the resulting OH to afford atomic O. The CO is then oxidized by the atomic O forming CO_2 which returns the catalytic surface back to its pre-reaction state. Alternatively, CO may be directly oxidized by the OH to form a carboxyl intermediate, followed by the dissociation or disproportionation of the carboxyl. Finally H is recombined to H_2 and CO_2 and H_2 are desorbed from the metal. The principal difference in these mechanisms is the formation of CO_2. The redox mechanism generates CO_2 by reaction with adsorbed oxygen, while the associative mechanism forms CO_2 via the dissociation of an intermediate. The mechanism of decarboxylation is debated; it may involve β-hydride elimination, or it may require the action of an external base."

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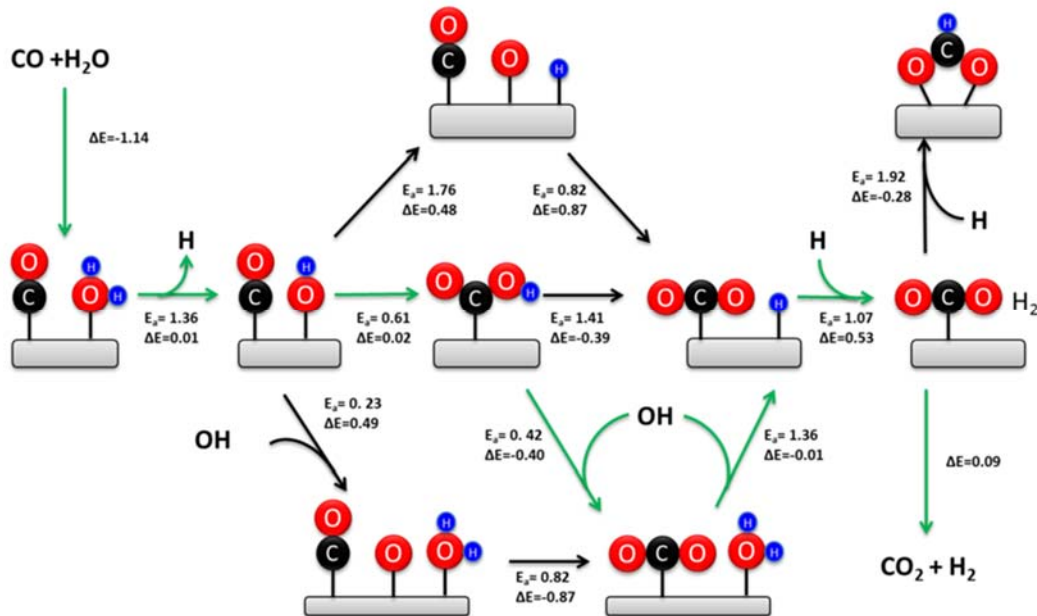
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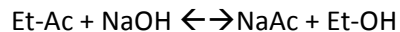
Memorandum

Date: August 18, 2017

To: Lab Group

From: Terry A. Ring, Experiment Supervisor

Subject: Ring Experiment I-12 – Stirred Tank Reactor

Our client has a CSTR saponification reactor operating in their plant. The saponification reaction used by our client is the saponification of ethyl acetate, (ET-Ac);

Et-Ac + NaOH ←→NaAc + Et-OH

followed by a separation process for sodium acetate, NaAc, from the mixture of the solvent water and the reaction byproduct ethanol (ET-OH).   They are having trouble with the reactor they have just installed and want to increase the reactor's yield. First of all, they would like to know if their stirred tank reactor is operating as an ideal reactor. They think that the impeller operating at 3 rpm may be too slow for ideal mixing. (Too small of a mixer motor seems to have been purchased and installed.) You will be required to develop a method of experimentally determining if the reactor is operating ideally and specifically determine just how far from ideal this behavior is.   Your laboratory work should make measurements of reaction conversion and compare the experimental results with your predictions.

A key factor in these experiments is the method used to measure the concentration of either reactants or products from the reactor. Develop an accurate method of chemical analysis. What size sample do you need to take from the reactor to give accurate analysis?

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Memorandum

Date: August 18, 2017
To: Lab Group
From: Terry A. Ring, Experiment Supervisor
Subject: Ring Experiment I-13 – Gas Absorber

WesTech has an Acid Mine Drainage Treatment System, see Figure 1, that initially takes Acid Mine Pool Water typically at pH 4 and conditions it with H_2SO_4 to lower the pH to pH 1. This stream is then fed to a "Forced Draft (air) Decarbonator" where CO_2 is removed in a packed bed. They do not know how to design the Decarbonator properly and we are to help them with this problem. There are several key questions that they would like answered given a flow of Acid Mine Pool water saturated with CO_2 at pH 4 and shifted to pH 1.

How much packing surface area is required for the removal of 95% of the dissolved CO2?

What diameter of the column and what size packing is suggested for processing 100 gal/min water supersaturated with CO_2 by 50% after acidification.

How much air should be supplied for the above separation?

In short, you are to design the Forced Draft Decarbonator stripping tower for them for this service using your experimental data and chemical engineering design tools.
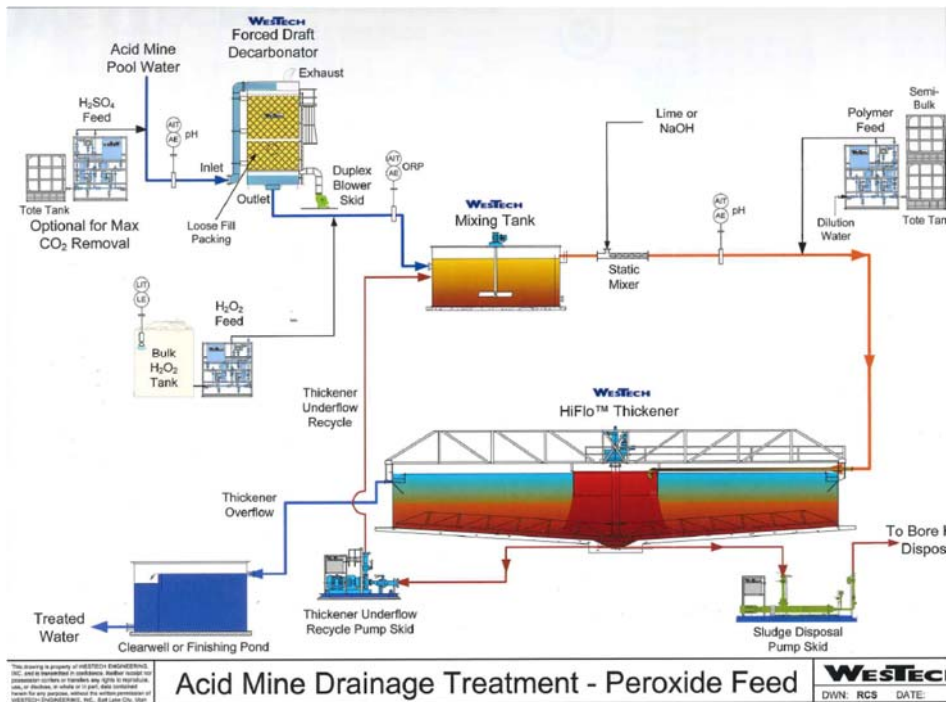

Figure 1 Acid Mine Drainage Treatment System

Memorandum

Date: August 18, 2017
To: Lab Group
From: Terry A. Ring, Experiment Supervisor

You can develop a test for CO_2 dissolved in water using the diamond ATR cell on the FTIR apparatus since CO_2 has an absorption peak at 4.27 microns, see

http://vitalsensorstech.com/PDF%27s/ATR%20spectroscopy%20method%20for%20dissolved%20CO2%20in%20beverages.pdf .

Please include this assignment in your report as an appendix but do not cite it in the body of your report.

Memorandum

Date: August 18, 2017
To: Lab Group
From: Terry A. Ring, Experiment Supervisor
Subject: Ring Experiment I-14 – Ion Exchange

The client from LA has an etching bath that uses a strong pH 10 NaOH solution to etch surface damage off forged parts made from aluminum alloys. Some aluminum alloys have significant concentrations of copper in them which builds up in the bath. After a while the growing concentration of copper in solution starts to cause localized corrosion by first precipitating out a copper particle on the part surface and then initiating galvanic corrosion as shown in Figure 1 that results in aluminum hydroxide clusters filling pits in the corroded aluminum part's surface.
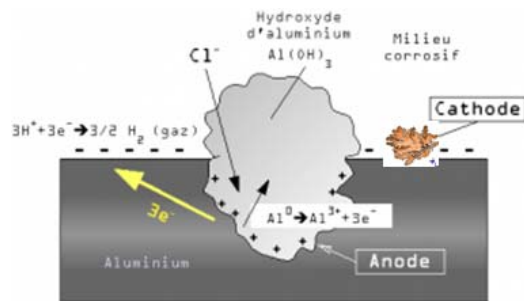

Figure 1 Localized Corrosion caused by excess copper ion in solution

The client wants to find a way to remove the copper ion from the etch bath solution. Since the 1000 L bath is circulating through a heat exchanger, we have suggested that an ion exchange column be placed in this line and periodically changed when it breaks through. The client provided information includes:

Metal Etching rate = 2 kg/hr with 24 hr/day operation.

Bath Circulation rate =15 L/min

Alluminun Alloy 2014 has 1.29% wgt Cu in it.

If copper exceeds 75 ppm in the bath corrosion starts to become a problem. Your task in this laboratory is to perform tests on the ion exchange column to see if either Amberlite IRC 748 or IRC 748i resins are capable of removing copper down to 75 ppm and for how long in their 1000 L circulating system.

With this laboratory information and product information available, you are to design the size and shape of the ion exchange column to be used for this application by the client. Please include: 1) the pressure drop as the pump

that is presently being used may not be adequate with the ion exchange column in the flow loop and 2) a means to regenerate the column for reuse.

Please include this assignment in your report as an appendix but do not cite it in the body of your report.

Memorandum

Date: August 18, 2017
To: Lab Group
From: Terry A. Ring, Experiment Supervisor
Subject: Ring Experiment I-15 – Spray Dryer

For some upcoming DoE research, we need to know the drying capacity of the pilot spray drier in our lab. Consider the following:

(a) In a paper (Gluckert, F. A., "A Theoretical Correlation of Spray Dryer Performance," AIChE Journal 8,460-466 (1962), the claim is made that the drying capacity of a spray drier can be calculated by means of a general correlation equation. Are there alternative or better correlations? You are to check the Gluckert assertion by performing appropriate tests on the laboratory spray dryer.
(b) Operate the dryer with a distilled-water feed with inlet gas temperatures of 200 and 300°C and determine the maximum drying rate under these conditions.
(c) Operate the dryer with a solution feed of detergent soap at a concentration of 10% and the same inlet gas temperatures as under (a) above or select a better temperature if literature indicates differently. Select an appropriate solution concentration and determine the maximum drying rate under these conditions. Collect samples of the dried product for microscopic examination (get some photos) to determine the maximum diameter of the dried product.
(d) Compare your results to the other correlations. If your results do not agree with the Gluckert correlation or other correlations you have found, your report must contain a quantitative explanation of the likely cause of the differences. This explanation must be supported by sample calculations.
(e) From your results, recommend the size, number and operating conditions for spray drying 100 pounds/hour of Copper sulfate for the DoE research.

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Memorandum

Date: August 18, 2017
To: Lab Group
From: Terry A. Ring, Experiment Supervisor

GLÜCKERT EQUATION

$$Q = \frac{6.38\, K_f\, v^{2/3} \Delta T}{D_m^2} \frac{w_1}{\rho_1} \sqrt{\frac{\rho_a}{w_a V_a} \frac{w_a + w_1}{w_a}}$$

Q = rate of heat transfer to spray, BTU/hr

K_f = thermal conductivity of gas film surrounding the droplet
BTU/(hr · ft)(° F · ft)(at avg. temperature between dryer gas and drop temperature)

v = volume of dryer chamber, ft3

Δt = temperature driving force, °F ($T_{wb-inlet} - T_{exhaust}$)

D_m = maximum drop diameter, ft

w_1 = weight rate of liquid flow, lbm/hr

w_a = weight of atomizing air flow, lbm/hr

$\rho 1$ = density of liquid, lbm/ft^3

ρa = density of atomizing air, lbm/ft^3

V_a = velocity of atomizing air at atomizer, ft/hr.

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