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Memorandum

Date: September 20, 2017

To: Lab Group

From: Terry A. Ring, Experiment Supervisor

**Subject: Ring Experiment III-8 Shell and Tube Heat Exchanger – Control**

The shell and Tube Heat Exchanger has two control valves: one on the process fluid flowing to the tubes and one on the cooling water to the shell. Your task is to develop the best controller for each control valve using one of the following P, PI, PD or PID controllers. Step testing or process modeling can be used to better understand the unit operation's process dynamics to aid in determining the best controllers for these two situations. In the end you are required to show experimentally that the controllers you have selected can control the process. Finally, develop a method to quantitatively compare the various controller types you have performed experiments upon.

Be prepared to discuss the quantitative comparison method in the oral exam.

**Please include this assignment in your report as an appendix but do not cite it in the body of your report.**

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Date: September 20, 2017

To: Lab Group

From: Terry A. Ring, Experiment Supervisor

**Subject: Ring Experiment III- 9 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 pitting corrosion which is an unacceptable flaw in the part. If copper exceeds 75 ppm in the bath, the pitting corrosion starts to become a problem. In addition, you are to measure the column regeneration, i.e. where the copper ion is stripped from the loaded column using a 0.1M HCl stream determining the time to strip the column of copper and the amount of 0.1M HCl required.

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 be periodically regenerated when the copper concentration of 75 ppm breaks through. The client provided information includes:

Metal Etching rate = 1 kg/hr with 24 hr/day operation.

Bath Circulation rate =15 L/min

Alluminun Alloy 2014 has 1.29% wgt Cu in it and 1200 kg of metal is etch per hour.

Your task in this laboratory is to perform tests on the ion exchange column to see if Amberlite IRC 748i resin is capable of removing copper down to 25 ppm for how long in this system treating how much solution which is initially at 75 ppm. In addition perform tests on the regeneration of the resin using 0.1 M HCl.

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, 2) the flow conditions and time required for column during copper removal and 3) column regeneration with HCl.

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Date: September 20, 2017

To: Lab Group

From: Terry A. Ring, Experiment Supervisor

**Subject: Ring Experiment III-10 Glass Lined Reactor – Batch Kinetics**

The glass lined reactor is to be used to determine batch kinetics at both cooling water and stream temperatures. The saponification of ethyl acetate

$$\text{Et-Ac} + \text{NaOH} \rightarrow \text{NaAc} + \text{Et-OH}$$

is to be used for your experiments. It is necessary to perform experiments to determine the order of reaction with respect to ethyl acetate and with respect to sodium hydroxide as well as the activation energy for this reaction, hence the need to perform experiments at cooling water and steam temperatures.

For direct comparison purposes, the kinetics of this reaction are reported in Hovarka, R.B. and Kendall, ;H.B. "Tubular reactor at low flow rates" CEP56(8),58-62(1960). You are to compare your results to the results can be gleaned from this paper.

Design Problem: Using a sequential batch mode of operation, what is the production rate of sodium acetate for this batch reactor assuming each batch has at least a 98% conversion and the equimolar feed is 1 M in both Ethyl acetate and sodium hydroxide.

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Date: September 20, 2017

To: Lab Group

From: Terry A. Ring, Experiment Supervisor

**Subject: Ring Experiment III-11 Catalysis Experiment - Water gas shift rxn**

The client needs to design a catalytic reactor 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 $CuO_2$ catalyst. 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 ). With this rate expression, you are to develop a reactor designthat can produce 1,000 kg/hr of $H_2$ for a hydrogen production facility. Since $CO_2$ is easy to remove from the product stream using amine absorption and water is easy to remove by condensation, it is important to run the reactor with a high conversion and with a minimum of CO in the effluent stream. You are to determine the best reactor operating conditions to achieve these goals.

> "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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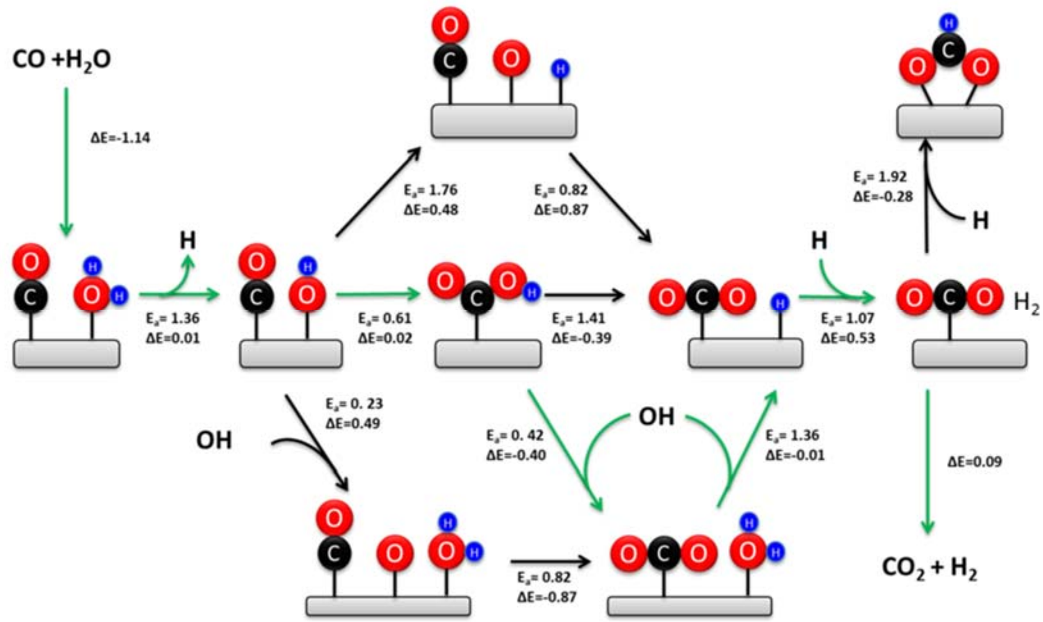


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To: Lab Group

From: Terry A. Ring, Experiment Supervisor

**Subject: Ring Experiment III-12 Gas Absorber – Evaporative Cooling Service**

In dry climate,s air can be cooled by evaporating water into it (your typical Utah swamp cooler). The heat of water evaporation is removed from the air thus cooling the air. In the process of cooling the air, it is also simultaneously humidified. As a result this can be monitored by both the temperature and the relative humidity of the air. A relative humidity sensor is available in the laboratory and temperature sensors are mounted and data logged on the system.

You are to study this simultaneous mass and energy balance problem with the gas absorber. For future design purposes, you are to determine a correlation for the mass transfer coefficient as a function of gas and liquid flow rate. Quantitatively compare your correlation to those available in the literature.

Please use the design information you developed for the following problem.

A standard 2,000 $ft^2$ home requires 50,000 BTU/hr of cooling. Assuming that summer time air is 90F, how big (in $ft^2$) a home can Column 2 in the lab cool? What is the flow rate of water for Column 2 in this situation?

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Date: September 20, 2017

To: Lab Group

From: Terry A. Ring, Experiment Supervisor

**Subject: Ring Experiment III-13 Heat Control**

The heat control is an apparatus with a long residence time which makes developing a controller for it a time consuming operation. As a result, you should plan a limited experimental program to be used for process modeling purposes and another for controller verification. Using control station, fit the experimental data to a process model. Use the process model to develop only two controllers be they P, PI, PD or PID and determine the appropriate coefficients for the two best controllers. After you have developed the controllers using control station implement the controllers on the heat control apparatus and test your two controllers. Compare the accuracy of the two best controllers on the apparatus to those same controllers on the process model. Develop a method to quantitatively compare the various controller types you have performed experiments upon. Be prepared to discuss the quantitative comparison method in the oral exam.

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Date: September 20, 2017
To: Lab Group
From: Terry A. Ring, Experiment Supervisor
**Subject: Ring Experiment III-14 Liquid Level Control**

The liquid level control experiment measures the pressure head in a tank and controls either the inlet flow rate or the outlet flow rate from the tank. The object of this experiment is to test both system configurations with a controller (either P, PD, PI or PID) with an optimized set of tuning parameters. Determine which configuration is the best, to control level in the tank. You are also to specify the controller type (e.g. P, PD, PI, PID) and optimum tuning parameters for the experimental system, which best controls level by manipulations of the flow rate in or out of the tank. Develop a method to quantitatively compare the various configurations (both valve location and various controller types) you have performed experiments upon. Be prepared to discuss the quantitative comparison method in the oral exam.

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Memorandum

Date: September 20, 2017

To: Lab Group

From: Terry A. Ring, Experiment Supervisor

**Subject: Ring Experiment III-15 pH Control**

The objective of this experiment is to develop a pH controller for the mixing of dilute NaOH solution with dilute HCl solution. This type of controller is prone to difficulties as the signal measured, pH, is highly non linear with concentration as a result the control of a valve or pump in response to the error in pH measured compared to that of the set point is not linearly related but logarithmically related. Your objective is to use the tricks you learned in your Control Class to deal with this problem and come up with a pH controller that can control the pH on both the acid and base sides and control to < 0.8 pH units variability. You are to experimentally demonstrate this by 1) a shift of set point from the acid side to the base side, and 2) a shift of set point from the base side to the acid side, keeping to <0.8 pH after the initial process shift.

Be prepared in your oral quiz to address the following:

a) Safety issues with this experiment

b) Equipment operation

c) The definition of pH. Any Temperature effects?

d) Other germane points with respect to this experiment

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