AIChE®

2002
National Student Design Competition

If there are any questions about the design problem, student chapter advisors and design course instructors are asked to contact:

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Please read the rules on the following pages carefully before submitting a solution to AIChE.

American Institute of Chemical Engineers
3 Park Avenue, New York, NY 10016-5991

2002 AIChE NATIONAL STUDENT DESIGN COMPETITION

Reactor Design for Polyether Synthesis

DEADLINE FOR MAILING

Solutions must be postmarked no later than midnight, June 4, 2002.

RULES OF THE CONTEST

Solutions will be graded on (a) substantial correctness of results and soundness of conclusions, (b) ingenuity and logic employed, (c) accuracy of computations, and (d) form of presentation. Accuracy of computations is intended to mean primarily freedom from mistakes; extreme precision is not necessary.

It is to be assumed that the statement of the problem contains all the pertinent data for those available in handbooks and literature references. The use of textbooks, handbooks, journal articles, and lecture notes is permitted.

Students may use any available commercial or library computer programs in preparing their solutions. Students are warned, however, that physical property data built into such programs may differ from data given in the problem statement. In such cases, as with data from other literature sources, values given in the problem statement are most applicable. Students using commercial or library computer programs or other solution aids should so state in their reports and include proper references and documentation. Judging, however, will be based on the overall suitability of the solutions, not on skills in manipulating computer programs.

The 2002 National Student Design Competition is designed to be solved either by an individual chemical engineering student working entirely alone, or a group of no more than three students working together. Solution will be judged in two categories: individual and team. There are, however, other academically sound approaches to using the problem, and it is expected that some Advisors will use the problem as classroom material. The following confidentiality rules therefore apply:

1. For individual students or teams whose solutions may be considered for the contest: The problem may not be discussed with anyone (students, faculty, or others, in or out of class) before or during the period allowed for solutions. Discussion with faculty and students at that college or university is permitted only after complete final reports have been submitted to the Chapter Advisor.

2. For students whose solutions are not intended for the contest: Discussion with faculty and with other students at that college or university who are not participating in the contest is permitted.

3. For all students: The problem may not be discussed with students or faculty from other colleges or universities, or with individuals in the same institution who are still working on the problem for the contest, until after June 4, 2002. This is particularly important in cases where neighboring institutions may be using different schedules

Submission of a solution for the competition implies strict adherence to the following conditions:

(Failure to comply will result in solutions being returned to the appropriate Faculty Advisor for revision. Revised submissions must meet the original deadline.)

ELIGIBILITY

◊ ONLY AIChE NATIONAL STUDENT MEMBERS MAY SUBMIT A SOLUTION. Non-member entries will not be considered. If you would like to become a National Student Member, we must receive your membership application and check when you submit your solution, if not before. Application forms can be downloaded at: https://www.aiche-mart.org/memberapps/student.asp

◊ Entries may be submitted either by individuals or by teams of no more than three students. Each team member must meet all eligibility requirements.

◊ Each Faculty Advisor should select the best solution or solutions, not to exceed two from each category (individual and team), from his or her chapter and send these by registered mail, as per the below instructions, to the Institute.

TIMELINE FOR COMPLETING THE SOLUTION

◊ A period of no more than thirty days is allowed for completion of the solution. This period may be selected at the discretion of the individual advisor, but in order to be eligible for an award, a solution must be postmarked no later than midnight, June 4, 2002.

◊ THE FINISHED REPORT SHOULD BE SUBMITTED TO THE FACULTY ADVISOR WITHIN THE 30-DAY PERIOD.

REPORT FORMAT

◊ The body of the report must be suitable for reproduction, that is, typewritten or computer-generated. Tables may be written in ink. Supporting calculations and other appendix material may be in pencil.

◊ The solution itself must bear no reference to the students' names or institution by which it might be identified. In this connection, graph paper bearing the name of the institution should not be used.

SENDING THE SOLUTION TO AIChE

◊ Two copies of each of the solution(s) must be sent to the address below; original manuscript(s) must remain in the possession of the Student Chapter Advisor, or Faculty Advisor, sponsoring the student(s)

◊ There should not be any variation in form of content between the solution submitted to the Faculty Advisor and that sent to the AIChE office.

◊ Each copy must be accompanied by the enclosed ENTRY FORM giving each contestant's name, AIChE membership number, college or university, Faculty Advisor name, address, home address, home telephone number, and student chapter, lightly attached to the report. The executive director of the Institute will retain this form for identification.

◊ DEADLINE: Entries must be postmarked no later than midnight, June 4, 2002. As soon as the winners have been notified, original manuscripts must be forwarded to the office of the executive director as soon as possible.

SEND TO:
Awards Administrator
American Institute of Chemical Engineers
3 Park Avenue
New York, New York 10016-5991

DEADLINE: JUNE 4, 2002

2002 AIChE Student Design Competition

Reactor Design for Polyether Synthesis

I. Objectives and Scope

Polyethers are an important family of products that are found in many consumer products, such as, detergents, foaming agents, defoaming agents, rigid foams (imitation wood and insulation), flexible foams (rug underlays, seat cushions), and food additives. This diverse set of properties is the result of adding different ratios of ethylene oxide (EO) and propylene oxide (PO) to the polyether (as heterics {PO and EO}, as block polymers {PO or EO}, or as caps to a block polyether) and also as a result of adding the polyethers to other chemicals. The current industrial process for manufacturing polyethers is a semi-batch process. In this design problem you are asked to design a semi-batch reactor system to manufacture a 3000 molecular weight, straight propylene oxide polyether. The primary emphasis of this problem is to develop a safe design.

In plant designs, there is usually a balance based on engineering judgment including the appropriate use of technical standards, industrial standards, the company's acceptable level of risk, and the investment. The best design solutions will be based on the appropriate and balanced application of the principles of design and safety. The winners will not necessarily have the lowest investment.

II. Specific Objectives of this Design Project

1. Determine the best design for this reactor system: a) determine the capital investment (installed not operating cost) for a semi-batch polyether reactor system, and b) include and emphasize the appropriate use of design and safety features.
2. Only size the safety relief valves for the reactor or reactors and only for the runaway reaction scenario.
3. Conduct and document a safety review that contains two parts: a) Hazards: a clear description of the major hazards of this process and what design features are incorporated to circumvent these hazards and b) Inherent Safety: highlights of one or more of the following inherent safety concepts: i) design features for easier and effective maintainability, and ii) design features using inherent safety concepts that circumvent accidents even when instruments fail or operators make mistakes.
4. Develop recommendations for the best design and state your reasons for these recommendations.
5. Your report must be formatted as specified below, containing each item listed.

III. Report Table of Contents Format

A. Title Page – Include author/authors names, and the school
B. Table of Contents
C. Executive Summary – A summary (maximum of one page) that highlights the important findings, including the investment for this process, the recommendations (based on the investment and safety considerations), and precautions (based on the safety reviews).
D. Introduction – Summarize a problem statement, including project background and design objectives.
E. Process Results – Provide a description of the process, the functions of the major equipment components and controls, stream connectivity, safety, and other design features. Provide results, using figures, tables, and graphs to support your claims. Results and support calculations should be in English Units.
F. Process Flow Diagram (PFD), Piping and Instrument Diagram (P&ID), and Layout – The PFD should include the major equipment, stream numbers, mass and energy balances, major controls, pressures, temperatures, flows, etc. The P&ID should include a detailed process description (more detailed than the PFD), the specifications for the major equipment components, and the detailed controls. The Layout should include the reactor and other parts of the process that are not part of this specific design, e.g., location of the control room, catalyst removal station, propylene oxide storage vessels, product storage vessels, etc.
G. Safety Review Sections– i) Hazards, and ii) Inherent Safety. Place the MSDSs in the appendix A3.
H. Assumptions List – Provide a numbered list of the assumptions made and the justifications for making them. Clearly indicate where the assumptions were applied in the development of the process design.
I. Economic Summary – Include a table that describes the installed investment for the design, and include the economic methods that were used to compute the investment.
J. Discussion of Results – Provide an analysis of the process in relationship to the investment and safety. Include a discussion of the alternative process designs that were considered; especially discuss the unique design features that you may have discovered, including the cost and the safety features of the final design.
K. Conclusions - This should be an enumeration of your conclusions (clear and concise).
L. Recommendations - Your recommendations should be based on all of the above results.
M. References – List references in the standard format found in your textbooks.

IV. Appendices

A1 Equipment Specifications List – Provide a list of all the equipment used in the process, including type, description, function, materials of construction, size, operating conditions, purchased cost, and all important specifications.

A2 Investment analysis that was summarized in the body of the report. Include spreadsheet calculations showing formulas, sample calculations, and sources of the methods used.

A3 Safety Review– Enclose the MSDSs to this Appendix A3.

A4 Computer process simulators and other programs – The students should use generally available process simulators (ASPEN®, etc.) together with spreadsheets and student developed computer programs. Include a description of when and where each simulation package was used, and the input and output sheets. Tie this information (via the appropriate labels) to the PFD or the P&ID.

A5 Back-up/Support Data and Calculations – Provide documentation for the calculations made by hand at least on a sample basis, but preferably include all hand calculations made for the design. Include flow charts where possible. All special purpose computer programs must be documented by including a brief description, input, and output files. This means that cell formulas must be included for your spreadsheet calculations.

V. Process Description

General. The manufacturing process for polyethers consists of building chains by adding propylene oxide or ethylene oxide (or a mixture of the two) to a multiple functional alcohol, such as glycerin or propylene glycol (or one of many available alcohols). Adding oxide to an initiator, however, is not sufficient to have a reaction take place. You need a catalyst that is potassium hydroxide; it must be present for the reaction to occur. The first step in the preparation of a polyether is to activate an initiator with some type of catalyst. In this project we will use only a glycerin initiator, potassium hydroxide catalyst, and propylene oxide. The activated initiator is actually a unique chemical species, specifically potassium glycerate. The generation of an activated initiator and the reaction with propylene oxide is illustrated in Figure 1.

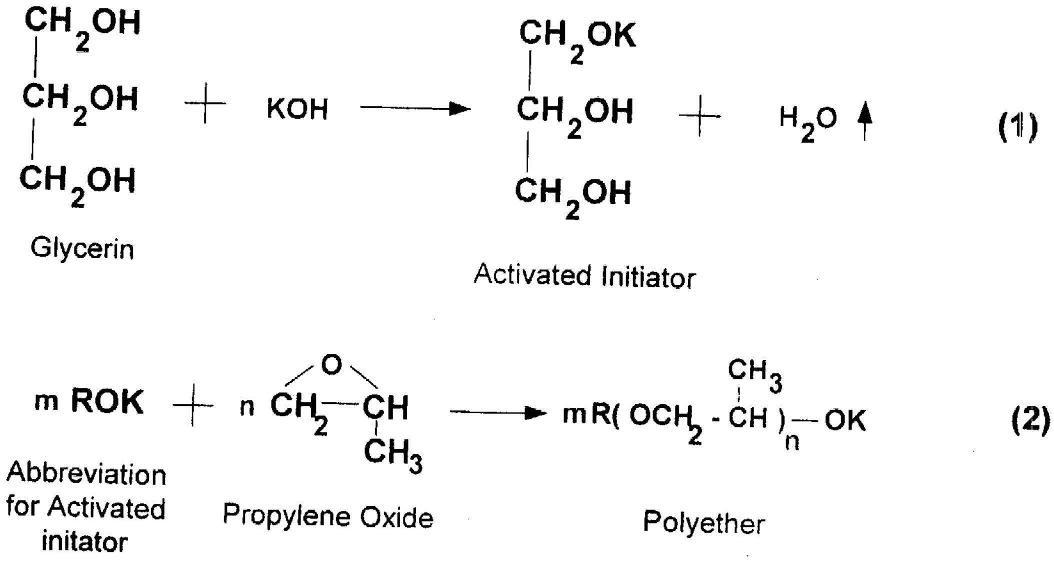


Figure 1 Polyether Reactions

In this project you will work with both reactions. Assume that reaction (1) is fast, and there are no heat effects. Notice that the potassium does not saturate the available hydroxyls. The potassium, however, migrates within the reaction mass from one hydroxyl to another within a single molecule and from one molecule to another. The final product has approximately equal length chains on each molecule. The molecular weight of a typical polyether may range from a few hundred to thousands. The reactor of this project will manufacture a 3000 molecular weight polyether.

Your design should facilitate a six step process: 1) add glycerin to the reactor and then add the potassium hydroxide catalyst (3 wt % KOH or 0.03 X [lb. of glycerin + lb. of KOH]) to the reactor, 2) heat the mass to the reaction temperature, 3) remove the water down to 0.5% water by vacuum stripping to form the activated initiator (the water is removed via the vapor pressure of water over the mass; assume the partial pressure is equal to the weight fraction times the vapor pressure), 4) add the propylene oxide, remove the heat of the exothermic reaction to control the temperature, and continue the addition to form the 3000 mol. wt. product, 5) at the end of the addition period, react the free oxide down to 1%, and 6) transfer the hot product to the next process step for catalyst removal and the addition of an oxygen inhibitor package (the catalyst removal and the addition of the inhibitor are not a part of this design project).

When you start the reaction, you only have a small quantity of glycerin in the vessel. You should investigate using more then one reactor in series (e.g., make a 500-2000 molecular weight polyether in a first reactor and the 3000 molecular weight material in the second). Also notice that the material leaving the last reactor has 1 % free oxide, therefore you will need to add approximately 1 % more oxide to the reactors compared to the stoichiometric requirement.

A batch reactor cannot be used for polyether reactions because it is prohibitively hazardous; that is, the large amounts of free oxide at the beginning of the reaction could easily become an uncontrolled runaway reaction. Industry uses a semi-batch reactor for polyethers; i.e. PO is added to the reactor at a uniform rate over the addition period to limit the amount of free propylene oxide (in the liquid phase) in the reactor. A continuous reactor is not recommended because a single reactor usually produces 50 to 100 different products in a single year; this would be difficult with a continuous reactor because the transition from one product to another would generate prohibitively large losses.

VI. AIChE Design Project Specifications

1. Capacity: Manufacture 100 million pounds per year of a 3000 mol. wt. polyether that is made with glycerin, a potassium hydroxide catalyst (3 % potassium hydroxide) and propylene oxide.
2. Chemistry: Start with glycerin, catalyst, and propylene oxide. All of the KOH reacts with the glycerol. As a design basis, the potassium hydroxide is limited to 3 % because potassium hydroxide may precipitate as a solid when the concentration exceeds approximately this 3% limit. Use the jacket as illustrated in Figure 2, to bring the mass up to the reaction temperature and use the external heat exchanger to remove the heat of reaction. This reaction is highly exothermic.
3. Reactor Configuration. Determine the capital investment for this system; i.e., the semi-batch reactor system. This reactor configuration is illustrated in Figure 2. Notice that we are specifying an external heat exchanger for cooling. Internal coils are sometimes used, but the use of coils is a complication that is not justified for this student problem. Coils, for example, condense vapors on the area that is not submerged in the liquid. The relatively small jacket on the vessel (on the lower portion of the vessel) is only for heating the initial charge of the glycerin and catalyst, and for removing the water by evaporation. Assume that the oxide comes from an existing storage vessel. In your design, you will need to provide a pump and the controls to appropriately add the oxide into the reactor, and to control the temperature. In Figure 2, only one reactor is shown. Your design may have more than one reactor.
4. Utilities: Assume that the following utilities are available at any capacity; i.e., water at 75 ^{O}F, steam at 125 psig, electrical power, instrument air at 85 psig, and nitrogen at 85 psig.
5. Process conditions and constraints: Control the maximum temperature of the reaction at or below 260 ^{O}F (it is known that the product degrades at or above this temperature). If the temperature is too low (say lower than 212 ^{o} F) then the reaction rate is very low, and the free oxide will increase to prohibitively high concentrations. Use stainless steel to prevent

contamination. Transfer the product to the catalyst removal station after you react the free oxide down to 1 %, and at the reaction temperature.

6. Basis for investments: Use the factor method [1] to determine the total investment based on the cost of the major equipment, including: vessels, heat exchangers, controls (or control loops), pumps, vacuum system, relief valves, and catch tanks.
7. Kinetics: The kinetics of a propylene oxide to polyether reaction that is catalyzed with potassium is shown below:

$$r = \frac{dC_O}{dt} = -kC_K C_O \tag{1}$$

$$k = 9.84 \times 10^{11} \exp\left(\frac{-15099}{T}\right)$$

$$k = \frac{pounds_{total}}{(pounds_{potassium})(hour)}$$

where T is degrees Rankin, C_K and C_O are weight fraction of potassium (not potassium hydroxide) and propylene oxide, respectively, and r is:

$$r = \frac{pounds_O}{(pounds_{total})(hour)}$$

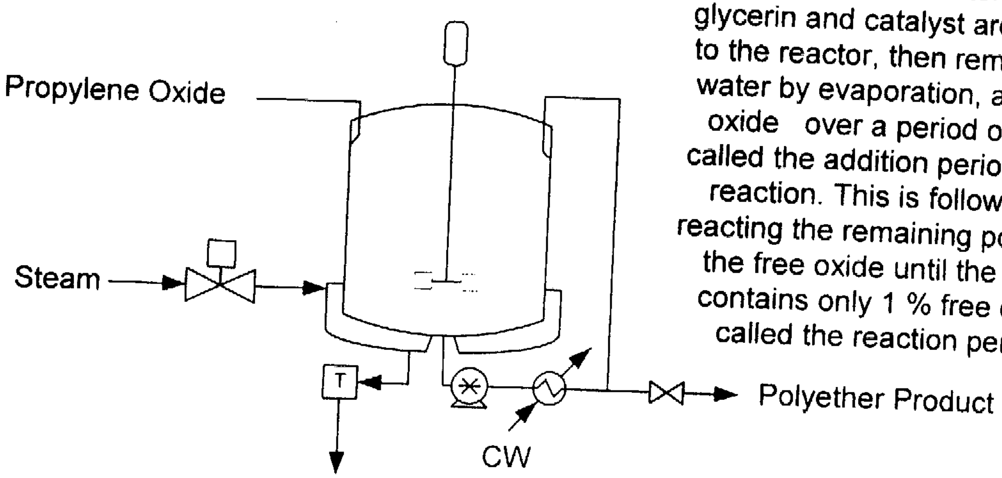
8. Heat Generation and Removal: The heat released by the reaction and the heat removed with the heat exchanger are given by Equations 3 and 4, respectively. The maximum temperature in the reactor is 260 ^{O}F and the minimum is 212 ^{O}F; temperature is a degree of freedom.

$$\frac{dQ_R}{dt} = (W_{total})(-\Delta H_R)(-r) \tag{3}$$

Where: $$\frac{dQ_R}{dt} = (pounds_{total})\left(\frac{BTU}{pound}\right)\left(\frac{pounds_O}{(pounds_{total})(hour)}\right)$$

$$\frac{dQ_L}{dt} = UA\,\overline{\Delta T_L} \tag{4}$$

where: $$\overline{\Delta T_L} = \frac{\Delta T_2 - \Delta T_1}{\ln\left(\frac{\Delta T_2}{\Delta T_1}\right)}$$



Semi-Batch Reactor: The glycerin and catalyst are added to the reactor, then remove the water by evaporation, and add oxide over a period of time, called the addition period of the reaction. This is followed by reacting the remaining portion of the free oxide until the mass contains only 1 % free oxide, called the reaction period.

Figure 2 Polyether Semi-Batch Reactor

9. Semi-Batch Kinetics: The standard reaction sequence for this reaction in a semi-batch reactor is divided into two steps or periods:
   - the addition period (propylene oxide is added and reaction occurs) $0 \leq t \leq \theta_A$
   - the reaction period (propylene oxide feed is shut off and reaction continues)

   In this semi-batch reactor, the catalyst concentration changes as the oxide is added as shown in Equations 5 and 6.

$$C_K = \frac{C_{K1} W_B}{W_{total}} \tag{5}$$

$$W_{total} = W_B + \frac{W_O}{\theta_A} t \tag{6}$$

where C_{K1} is the pounds of potassium per pound of glycerin plus potassium

W_B is the total pounds of glycerin plus the pounds of potassium

W_O is the total pounds of propylene oxide added over the entire addition period

θ_A is the addition period in hours.

The addition rate is constant. The W_O is the total amount of oxide that is required to manufacture a 3000 molecular weight polyether. During the addition period the maximum free PO (C_0) is limited to 20 % (this is a design constraint).

The reaction sequence is to add all of the required oxide during the addition period using Equation 7, followed by reacting the oxide down to the 1 % level during the reaction period using Equation 8. Equation 7 and 8 are used to compute the oxide concentration in the liquid phase. The oxide in the vapor phase is a function of its partial pressure. Assume that the oxide in the vapor phase is a part of the 1 % free oxide that leaves the reactor at the end of the reaction period.

$$\frac{dC_O}{dt} = \frac{(W_O/\theta_A)}{W_{total}} - \frac{kC_O C_{K1} W_B}{W_{total}} - \frac{C_O(W_O/\theta_A)}{W_{total}}$$

(7)

Addition Reaction Dilution

Note that Equation 7 ignores the vapor phase mass.
During the reaction period, all the oxide has been added, therefore:

$$C_K = \frac{C_{K1} W_B}{W_B + (W_O/\theta_A)\theta_A} = \frac{C_{K1} W_B}{W_B + W_O}$$

and

$$\frac{dC_O}{dt} = -\frac{kC_O C_{K1} W_B}{W_B + W_O}$$

(8)

In this project, use weight fraction instead of mole fraction because in this polymerization reaction the moles of the product (polymer) is constant throughout the entire reaction. The use of weight fraction, in this case, is also the normal practice within industry.

10. Relief Valve for the Reactor, and a Control System: The worst case scenario for a polyether reactor is a runaway reaction, therefore, the other commonly used relief scenarios for vessels can be ignored, i.e. fire around the vessel, failure of the air and nitrogen regulators, etc.

    Only size the relief valve for the reactor or reactors and only for this runaway scenario using the Fauske monogram method [2]. This relief sizing methodology is based on two-phase flow; that is, it is known that the runaway release from a polyether reactor has two-phases (liquid polyether, and propylene oxide and inert gases). The relief's set pressure is set at some nominal pressure above the normal operating pressure (say 10-15 psi above), and below the maximum allowable working pressure (MAWP) of the reactor. Also assume that the maximum allowable pressure in the reactor during a relief is approximately the MAWP plus 10 % of the MAWP.

    Venting this relief material to the atmosphere is hazardous due to the toxicity and flammability of propylene oxide. Therefore, you have two options: a) total containment (use a vessel with an MAWP high enough to withstand the maximum pressure during the runaway), or b) containment via treating the effluent from the relief via a cyclone separator with the liquid discharging to a large quench tank containing an acid to stop the reaction by the neutralization of the active catalyst end groups and the vapors discharging to a flare (see reference 3 for details).

11. Properties: To simplify this problem, only use the physical properties shown below in Table 1. Assume that the mixture properties are a simple function of the weight fractions.

Table 1 Properties of Pure Components and Pertinent Design Parameter

Component	Property	Units	80 °C	100 °C	140 °C	180 °C
PO	Vapor Pressure	psia	60	100	220	440
Polymer	Vapor Pressure	psia	0	0	0	0
PO	Heat Capacity	cal/g/°C	0.33	0.34	0.37	0.42
Polymer	Heat Capacity	cal/g/°C	1.0	1.1	1.2	1.3
PO	Heat of Vaporization	cal/g	120	98	82	55
Polymer	Heat of Vaporization	cal/g	0	0	0	0
PO	Density	g/cc	0.76	0.74	0.66	0.55
Polymer	Density	g/cc	0.90	0.85	0.80	0.75
PO	Viscosity	Centipoises	0.25	0.25	0.25	0.25
Polymer	Viscosity	Centipoises	300	250	200	150
PO	Thermal Conductivity	cal/cm/s/°C ($\times 10^{-5}$)	35	33	32	32
Polymer	Thermal Conductivity	cal/cm/s/°C ($\times 10^{-5}$)	50	45	40	35
PO	Heat Transfer Coefficient	BTU/hr/ft^2/° F	50	60	70	80
Polymer	Heat Transfer Coefficient	BTU/hr/ft^2/° F	20	25	30	35
The heat of reaction is 660 BTU/pound of PO.						
Flammability Regions; see Figure 3						

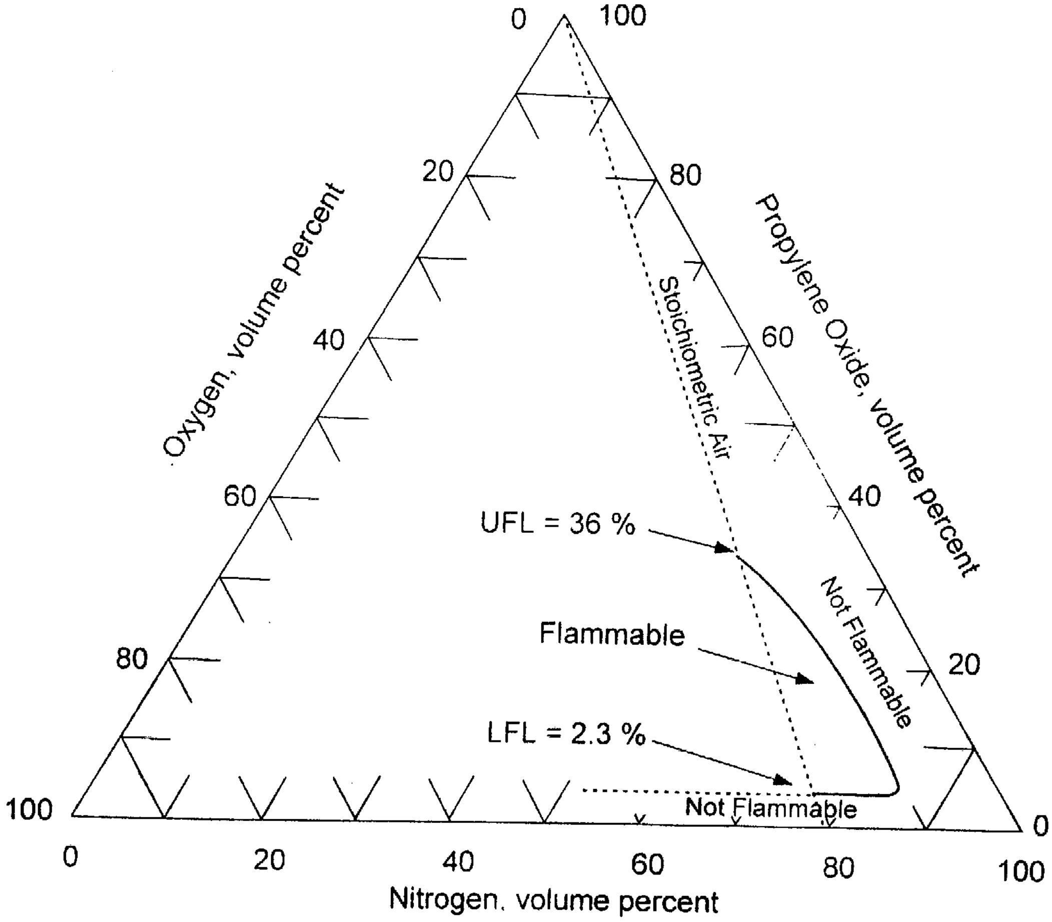


Figure 3 Flammability Limits of Propylene Oxide

Assume that the polymer has no vapor pressure. The partial pressure of propylene oxide over the reacting mixture (P_0) can be approximated using:

$$P_0 = 2.175 \times C_0 \times P_0^{Sat} \qquad (9)$$

where P_0^{Sat} is the propylene oxide vapor pressure; P_0 and P_0^{Sat} have the same units.

When handling PO in a reactor, the vapor phase over the reaction mass needs to be controlled to below the flammability region by adding nitrogen. See Figure 3.

When removing water by evaporation, assume that the partial pressure is equal to the weight fraction times the vapor pressure.

As a design basis, assume that the density of the reaction mixture is constant throughout the entire reaction cycle (addition and reaction periods). Assume a mixture density of 57-lb./cu. ft.

12. Equipment Sizing Basis and Purchased Costs: For this project, use the factor method [1] for determining the installed cost or the capital

investment. The major equipment should include: vessels, heat exchangers, pumps, vacuum system, reactor relief valves, and control loops. If your equipment sizes are different compared to those shown below, use the Lang Factor relationship [1] to compute your costs.

Vessels. The vessels should be stainless steel. The purchased cost of vessels is a function of the size and the pressure rating. For this project, assume the following:

Purchased cost of Stainless Steel Vessels					
Size	Pressure Rating (psig)				
(gallons)	100 psig	150 psig	200 psig	300 psig	400 psig
5000	$ 60,000	$70,000	$ 80,000	$90,000	$120,000
10000	$100,000	$120,000	$140,000	$160,000	$190,000
20000	$170,000	$190,000	$220,000	$250,000	$300,000

Heat Exchangers and pumps: The heat exchangers and pumps should also be stainless steel. The purchased cost is a function of the size and pressure as illustrated below:

Heat Exchangers, Stainless Steel		
Size (ft^2)	150 psig	300 psig
500	$23,000	$27,000
1000	$40,000	$48,000
2000	$65,000	$78,000
4000	$100,000	$120,000

Purchased Cost of Pumps (Stainless Steel)) Including the Motor	
Size in gpm x psi	Purchased Cost ($)
1,000	$20,000 to $50,000
10,000	$100,000 to $150,000

Note regarding the pumps: The ranges of these pumps can be utilized such that low flow and high head pumps would be at the high end of the range, while high flow and low head pumps would be at the low end of the range. For example: a 5 gpm pump with a 200 psi head would be $50,000, while a 200 gpm pump with 5 psi head would be $ 20,000.

Relief Valves: The purchased cost of relief valves is shown below:

The Purchased Cost of Relief Valves		
Relief Size Area (in.^{2})	Flange Size Inlet(inch) x Outlet(inch)	Cost ($)
1.29	2 x 3	1400
1.84	3 x 4	1600
2.86	4 x 6	2300
11.1	6 x 8	4400

The Purchased Cost of Control Loops	
Each Control Loop	$ 10,000

The Purchased Prices for Vacuum Pumps, Stainless Steel, Mechanical Water Ring Units		
Size in HP	Cost	Pumping Rate in CFM
10	$ 40,000	50
50	$ 80,000	400
100	$ 175,000	1000
200	$ 300,000	1500
Each of these vacuum systems levels off at 3 mm Hg absolute.		

Note for all costs: Assume that these purchased costs are for freight on board (FOB).

VII Economics

1. Purchased Equipment: Use the costs as described above.
2. Capital Investment: Use the methods from Peters and Timmerhaus [1], or any other generally acceptable documented method.

References

1. Peters, M. S. and Timmerhaus, K. D., *Plant Design and Economics for Chemical Engineers*, 4th Edition, (New York: McGraw-Hill, 1990).
2. Fauske, H. K., "Generalized Vent Sizing Monogram for Runaway Reactions," *Plant/Operations Progress*, Vol. 3, No. 4, 1984.
3. Grossel, S. S., "Design and Sizing of Knockout Drums/Catchtanks for Reactor Emergency Relief Systems," Plant/Operations Progress, July 1986.