

Advice to Authors for an Editorial Contribution to CEE

Chemical Engineering Education publishes editorials, usually invited, on subjects of current relevance to the community. The topic is normally controversial and the author is encouraged to clearly state his or her opinion on the issue and the rationale for the stated opinion.

Recent editorial topics include:

Wankat	Winter 2003	"Tenure for Teaching?"
Edgar	Spring 2003	"ChE Curriculum of the Future: Re-Evaluating the Process Control Course"
Doraiswamy	Summer 2003	"Whither Chemical Engineering Research?"
Woods	Winter 2004	"For the Sake of Argument...Who Will Teach the Core ChemE Courses?"

The editorials are one journal page (~600 words) in length and should be submitted electronically to cee@che.ufl.edu. Although they are not reviewed, the editors do provide feedback on occasion. As always, CEE reserves the right to make editorial changes or to refuse to publish material that the editors consider to be inappropriate. The deadlines for inclusion in each issue are:

Winter issue	October 15
Spring issue	January 15
Summer issue	April 15
Fall issue	July 15

Note that the Fall issue is dedicated to graduate education. Should you have any questions, please contact us at cee@che.ufl.edu

Standards for Articles in CEE

Submissions to CEE continue to increase. This increase indicates that there is a high and increasing interest in improving chemical engineering education. CEE will best serve the education of chemical engineers if its standards remain high.

Educational papers should be prepared with the same care as technical papers; however, the style can be more informal and some use of the first person is often appropriate. Papers need to reflect knowledge of the engineering education literature in addition to the appropriate technical literature. As an absolute minimum, authors should search the literature, read any articles on related topics, and cite appropriate papers. Of course, more than the absolute minimum is normally expected, and authors should do a reasonably diligent search of other relevant engineering education journals such as the *Journal of Engineering Education* (<http://www.asee.org/publications/jee/index.cfm>), *Proceedings of ASEE Annual Conferences* (<http://www.asee.org/conferences/v2search.cfm>), *Proceedings of ASEE Frontiers in Education (FIE) Conferences* (<http://fie.engrng.pitt.edu/>), *International Journal of Engineering Education*, and *European Journal of Engineering Education* (<http://www.asee.org/publications/international.cfm>). If a large fraction of what the authors intend to cover has already been covered, they should consider reducing the paper to an Educational Brief or scraping the project altogether. Papers must be related to the teaching and learning of chemical engineering—purely technical papers are rarely published.

Can papers that have been published in a nonarchival medium such as the ASEE Proceedings, published as a Web page, or were presented as a lecture be submitted to CEE? Since CEE provides a different audience and is archived, the answer is yes as long as such publication would not violate copyright agreements. Of course, the original publication needs to be cited. If large parts of the paper are identical to the original paper, this overlap should be identified (e.g., "Parts of this paper were previously published in . . ."). Republishing is most appropriate when the original publication consisted of preliminary results that can be expanded upon in the CEE article. The original presentation venue of a lecture should be acknowledged (e.g., "presented as a Plenary Lecture at the International Conference on Engineering Education 2004 (ICEE04), University of Florida,

Gainesville, FL, Oct. 19, 2004"). Obviously, lectures need to be recast into the proper format for an article. All such submissions will undergo the normal editorial and peer-review processes. Articles that have been published in or are being considered by other archival journals should not be submitted to CEE.

Although copyright protection in a digital era is complicated, for the purposes of CEE the following standards should be followed. First, unless you know otherwise, assume that every source (even an e-mail message) has copyright protection. Authors who want to copy substantial portions from an article (a complete table or complete figure or a substantial portion of the text), any portion of a song, or any part of a poem are responsible for obtaining permission from the owner of the copyright. This is often the publisher, not the original author. Since copyright refers to the form of the presentation and not the underlying data itself, if you totally recast the data (e.g., convert a table into a plot) permission is not required. Of course, whether or not permission is required, appropriate quotation procedures should be followed and the sources must be appropriately cited. Even when you are the original author, you must ask the copyright owner for permission to reuse your own work. As a rule of thumb, if a copyright form was filled out, you probably transferred copyright to that publication.

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The object of this column is to enhance our readers' collections of interesting and novel problems in chemical engineering. Problems of the type that can be used to motivate the student by presenting a particular principle in class, or in a new light, or that can be assigned as a novel home problem, are requested, as well as those that are more traditional in nature and that elucidate difficult concepts. Manuscripts should not exceed 14 double-spaced pages and should be accompanied by the originals of any figures or photographs. Please submit them to Professor James O. Wilkes (e-mail: wilkes@umich.edu), Chemical Engineering Department, University of Michigan, Ann Arbor, MI 48109-2136.

MURDER AT MISKATONIC

Passion, Intrigue, and Material Balances: A Play in Two Acts

JAKE VESTAL

N.C. State University • Raleigh, NC 27695

Dramatis Personae

Dr. Alexander Verawood: Late and notable professor of engineering at the Miskatonic University of Arkham, Massachusetts.

Dr. Xavier Verawood: Alexander's younger brother.

Richard Pendleton: Alexander's personal assistant.

Gabriella de Morcef: Graduate student in the laboratory of Alexander.

Thaddeus Hardcastle: Powerful tycoon with ethically questionable business tactics.

Aurora Simonova: Alexander's mistress.

Zelda Verawood: Alexander's wife.

Dr. Errol Curry: Miskatonic engineering professor; colleague of Alexander.

Tommy Benedict: Custodian for the Miskatonic U. Chemistry and Engineering building.

I. THE SCENE

"Thank you Pendleton. Just park here and we'll go in together; it's much too cold for you to wait in the car."

Dr. Xavier Verawood and Richard Pendleton climbed the stone steps of the Miskatonic University Chemistry and Engineering building together. After passing through the large wooden doors at the entrance, they approached the



Jake Vestal is a chemical engineering senior at North Carolina State University and plans to earn a Ph.D. in ChE after he graduates. He enjoys studying Logic and Game Theory, in which he is earning a minor from the Mathematics Department. Jake is passionately fond of the board game "Go."

entrance to the Research Level, where the secretary greeted them, making a note of their arrival in the log, like this: "Verawood, Xavier, and Pendleton, Richard: in 8:40 a.m., 21 Nov. 1932." The pair continued past the desk through the well-lit hall and descended to the basement. Halfway down the corridor Xavier opened the laboratory door of the great Dr. Alexander Verawood, his brother and the reason for his visit, and stepped in.

The room was dominated by a huge copper tank, taller than Xavier and polished to a bright orange luster. It was feeding from a collection of tanks that surrounded it as it brooded in the center of the room. From the top and sides of the vessel sprouted a multitude of thin pipes that stretched out like tendrils from a vast and carnivorous jungle plant. Each line snaked its way to one of many contraptions on sturdy tables arrayed upon the flagstones of the lab's floor, or to a wall rack where an amber-colored liquid was subjected to the gauntlet of a multitude of delicate glassware and shiny metal, which looked to Verawood like some kind of glittering alchemy.

The place smelled, strangely, like a bakery next door to an auto garage, and it resonated with a humming sound and a regular pulse like a slow heartbeat. Every now and then pressure was released from somewhere in great puffs, and a large mercury thermometer on the wall proclaimed a warm temperature of 77 °F. The pair was hailed by a voice from across the room.

Xavier turned to see a very small, dark-haired young woman whose large brown eyes stood out starkly against the white of her lab coat. She was wheeling a cart that bore several very large tanks to a corner of the room. Xavier and Pendleton helped her wrestle the tanks off the cart and onto the floor, where they were safely chained to the wall.

"Thank you," said the woman, brushing away a strand of black hair. "Those empty CO₂ tanks were left somehow over there next to the full ones. It's lucky they were still on the cart; they're very heavy and quite cumbersome to move around. Are you here to see anyone?"

Pendleton noticed that a few of the tanks in the lab were painted a brilliant blue and sported proud labels stating "The Blue Boy: 2 ft³, Guaranteed to 3000 psi." He remembered how the Professor, his employer, had once performed a certain favor for Warrington Industrial, the maker of the Blue Boy tank, and as a result found himself supplied with gas storage practically to his heart's content.

"Yes, ma'am, I'm Xavier Verawood, brother of the Professor, and I think you must already know Richard Pendleton, my brother's assistant. I arrived from Georgia last night."

"Of course, Dr. Verawood, your brother has told me about you. Aren't you a professor of Medieval and Renaissance Literature at Emory? My name is Gabriella de Morcef; I'm your brother's graduate student. What can I do for you?"

"Well, I did come here to see the Professor, but if it's not too much of an inconvenience I was rather hoping you could explain this cyclopean monstrosity to us," Xavier said, meaning the huge copper tank. "It looks terribly interesting, but I'm afraid I must humbly confess to a deplorable ignorance of chemistry and mathematics. And, please, call me Xavier: I still haven't quite gotten used to 'professor' or 'doctor'."

At this, Gabriella lit up. "Of course! Follow me to the incubator: This is where it all starts."

Intrigued, the two men allowed themselves to be swept in the wake of the tiny scientist, who led them to the monolithic bronze tank in the center of the room. Opening a slide in the outer casing of the incubator, Gabriella exposed a view cell through which a churning amber liquid could be seen swirling against the glass.

"It's fortunate that you arrived on a collection day; otherwise you wouldn't have seen the lab in all its splendor," she said.

"Now, this yellow liquid that you see through the glass is really an aqueous solution of yeast cells, kept at 37 °C in a mixture containing one part tryptone and other proteins—that's milk; well, more or less," she added as an aside, before ticking off the remaining ingredients. "One part NaCl, some glucose, and a dash of MgCl."

"These tanks that you see surrounding the incubator all contain O₂ or CO₂. The CO₂ feed is part of a buffering system we use to keep our yeast cells producing their wonderful chemicals that this lab is dedicated to studying. Other reagents are mixed in with the solution, but they vary with the experiment that is being performed.

"As you must know, yeast cells are able to produce ethyl alcohol through fermentation; this is how beer is made. This lab is really quite like a plant, a brewery if you will, except we have found that we can coerce our little yeast cells to produce amazing hydrocarbons that can be modified for other uses, chiefly for fuel and industrial-lubricant applications. The trick is a combination of the proper choice of media and correct treatment of the cellular products. Essentially, we are using the machinery that already exists within the yeast cells to make our product! You can surely see how these very processes might drastically reduce our dependence on oil, and perhaps even eliminate it."

Here, Gabriella indicated the lines that radiated out from the incubator.

"On a collection day, the yeast solution, which by now contains our hydrocarbons of interest, is being sent off through this network of tubes to stations where various components alter the yeast feed. In the business, we call this a 'semi-batch' process. You can see on the wall there one of our distillation units; that particular component happens to be purifying the n-octane that the yeast cells have produced. The cells are be-

“As for the methane/ethane waste, it’s piped into that Blue Boy over there on the bench. Every Friday after he makes dinner the Professor tops it up to 1500 psig, then when he wants, he opens up a little valve under his desk, allowing fuel to flow from that tank to his office.”

ing lysed by a detergent, releasing the n-octane and forming a slurry of media, octane, and dead cell debris. From there, the mixture is heated and subjected to fractional distillation; thus, the octane is readily recovered. Each component that you see is doing essentially the same thing: It’s either autonomously synthesizing something or separating some important component of the virgin yeast feed that we might find to be a useful fuel or lubricant. It’s really a very simple process if you think of it in pieces.”

She smiled expectantly. Xavier tried not to let his eyes cross, but Pendleton simply nodded serenely. Xavier blushed, feeling like he had to ask something in order to avoid looking dim.

“Does nothing go to waste?” he blurted.

“Very astute, professor. Yes, the methane and ethane that we necessarily produce are useless. Stoichiometrically speaking, we produce about three times as much methane as we do ethane, and we used to just burn it all off to get rid of it. CO_2 is also produced; the yeasts respire just as you and I do. That extra CO_2 , however, is taken in tanks, on the cart that you helped me with, to photosynthetic algae that we grow on the roof in clear containers. There, the algae produce the sugars that we use in the yeast media in a CO_2 -rich environment. We could, of course, be purchasing or using atmospheric CO_2 , but why should we when we’re producing it right here in this room? Further, what we’re really trying to do is set up a working model of a plant that might someday operate on a near-closed cycle that couples the yeast/algae g-molecule exchange in order to supply us with cheap and virtually infinite fuels and lubricants. In the real plant, the CO_2 would probably just be piped into the algae tanks automatically with the rest of the yeast products.

“As for the methane/ethane waste, it’s piped into that Blue Boy over there on the bench. Every Friday after he makes dinner the Professor tops it up to 1500 psig, then when he wants, he opens up a little valve under his desk, allowing fuel to flow from that tank to his office.

“There, he has a clever little burner of his own design that he managed to coax into operating at 90% efficiency. He’s even constructed a marvelous little carburetor that draws the perfect amount of oxygen into the flame chamber; I saw it heat a pint of water up to boiling in only three minutes when I had coffee with him last week. The secret is that the reactor geometry allows the fuel to combust virtually completely. I only hate how he uses the thing with the door closed, but he claims that the little bit of CO_2 and water vapor that the burner produces is good for his office plants—one of his many eccentricities, I suppose.” She sighed. “He really is a brilliant

man, hates to waste anything at all, even though we do wind up having to dump what he can’t manage to reuse.”

“He’s been like that since childhood, Miss de Morcef, I can assure you,” said Xavier, glancing at the tank, whose gage now read 713 psig. “Is he in? I should very much like to see him; in fact, that’s the reason I came to Massachusetts. I stayed at his house last night, but you know how he gets so involved in his work that he winds up sleeping here.”

Gabriella nodded. “His new office is next door to the left. He took it because it’s close to the lab; it’s really not much more than a converted utility closet that was put into this basement when it was renovated. I would walk you there, but it’s never good to leave a high-pressure process unattended if one can help it, so you must forgive me. It was nice meeting you.”

The two men departed to Dr. Verawood’s office, which was out of the lab and next door, as they had been instructed. Xavier smelled the freshly painted walls and enjoyed the clomp-clomp of his shoes on the floor of the newly renovated hallway, while Pendleton silently noted the ventilation strips along the ceiling and floor g-molding. They were probably explosion-proof, he thought, and constantly running to prevent the buildup of dangerous gases in the basement.

The pair found a handsome hardwood door bearing the name “Dr. Alexander Verawood” on a golden strip. Both men noticed a strange smell that seemed to be leaking out from the large gap underneath the Professor’s door, no doubt put there in order to facilitate the slipping in of papers by students. Xavier knocked, but received no reply.

“Well Pendleton, he’s either engrossed in some great work or asleep. There’s no lock on this door; I’m sure he won’t mind if we go wake him up.”

“Of course he won’t mind, Dr. Verawood; he’s been very excited about your coming for two weeks now.”

Xavier turned the knob and pulled the door open. Immediately he uttered a choked gasp, cut short by the noxious rush of a malignant vapor, which smelled unmistakably like fuel gas. Pendleton coughed and sputtered as well, but retained the presence of mind to slap Xavier’s hand away from the light switch towards which it was creeping, thereby avoiding the chance of a quick spark followed by an unfortunate explosion.

“Ghastly smell, that. What’s the professor been...” he was stopped by the expression of horror on Xavier’s face. When Pendleton followed his gaze into the office, he saw what had upset the young professor.

There, on the floor of his own office, sprawled on the rug that he and Xavier had brought back from China, was the

great Dr. Alexander Verawood. He was cold, pale, and as dead as the flagstones he lay upon. Pendleton, falling to his knees from the onslaught of the horrible fumes and the sudden dull anguish that gnawed at his stomach, picked up the Professor's hand to check for a pulse. As he did so, Xavier noticed a pad of paper near the Professor's elbow bearing a single word:

"MURDER."

II. THE SUSPECTS

"Monstrous!" cried Xavier that evening, in his late brother's study at the Verawood family mansion. He was reflecting upon the day's events, of how they had moved his brother's body to the Verawood family vault with the help of a trusted family physician. First, though, Xavier had had to fumble for the supply valve in order to shut off the gas that was pouring forth from his brother's unlit burner. Luckily, they had been able to get the body out of the building by placing it in a black bag on a cart and claiming that it was a delicate piece of equipment that someone in the physics building had requested to borrow for a while.

Faraday, the family physician (to whom the Professor's last note was not mentioned), had performed an autopsy that afternoon with the conclusion that Verawood had died of a heart attack. Maddeningly, no traces of poisons, cuts, bruises, or any other trauma had been found anywhere on or in the late Professor's mortal coil.

"How could this have happened? What callous fiend could be responsible for such a heinous act! Oh, Pendleton, what are we to do?"

"Courage, Doctor, courage, there's hope yet. We won't find your brother's assassin by useless raving, but by logic. I must admit though, it's rather puzzling. Perhaps we should involve the police?"

"Not on your life, Pendleton, not with that Hardcastle around. He probably owns every policeman in Massachusetts by now. The murdering thug! I'm sure it was him, he's been hounding my brother since the beginning of the yeast project, and we know he was there the weekend of the murder!" Xavier was shouting.

"That's a circumstantial accusation, and you know it. Sit down and I'll have Doris bring us some wine and whatever is left from that roast we ate last night."

Xavier moved from the window to the soft suede chair beside the bookshelf, where his father used to read Goethe on rainy nights.

"Do you think there's such thing as the perfect murder, Pendleton?" he asked, despondently looking out at the cold trees.

"No. You should know that. Nothing in the real world corresponds exactly to its ideal counterpart, though it can get

very close. Perfection only exists in mathematics. No, your brother's murderer made at least one mistake, maybe more, and it's up to us to find it and exploit it."

Doris arrived with the roast and a bottle of Burgundy. The two men moved to the heavy wooden table near the short wall of the room, which was still somewhat cluttered by Alexander's notes and calculations. After setting down the food and drink, Doris slipped out, noticing that the two were deeply engaged in conversation. Xavier took a bite of his dinner.

"That feels better, I think we can get on with it now," he said.

Pendleton nodded, as Xavier took a solemn breath, then continued. "Let's start at the beginning. We conferred with the secretary and checked the log, and decided that only six associates of my brother were present in the C & E building between the time he was seen at his evening class on Friday, and 9:00 Monday morning, when we found him."

Xavier found a scrap sheet of paper and a pen, and started writing.

"There were six," agreed Pendleton, "All of whom had some kind of connection or business with your brother."

This is what Xavier wrote down:

Suspects:

- 1) Thaddeus Hardcastle
- 2) Aurora Simonova
- 3) Zelda Verawood
- 4) Errol Curry
- 5) Tommy Benedict
- 6) Gabriella de Morcef

"Why don't we start with old Thaddeus?"

"Very well. We know from the secretary's log that he visited the Professor on Friday evening, but did he have a motive?"

"A motive!" shouted Xavier. "Of course he had a motive! This is the baron himself we're talking about! He's always in the papers, in and out of court for the convenient and 'accidental' deaths involving anyone opposed to his juggernaut of an oil empire. He's the only one not affected by this Depression we're in, Pendleton—everyone needs oil. Only now, he gets to pay his workers less for the same amount of labor: more people are willing to strain away on his derricks and rigs because no one can find jobs. Alex's yeasts would have ruined his entire industry eventually, and he visited my brother on the weekend he was murdered!"

Xavier was becoming heated, and Pendleton thought it best to push along the conversation.

"Friday evening, from 5 to 6. Ample time, I should think. Let's move on to Miss Simonova."

"Well, I won't pretend I don't know what she was, but I think you know more than I do about her," admitted Xavier. "Alexander hardly talked about her in his letters. Look, she

was at the building from 8:30 to 9:30 p.m. on Saturday. Tell me about Aurora, Pendleton.”

“She was rather the climbing type, I came to believe, though I don’t insult your brother’s taste. Unfortunately, despite my personal opinions (which reflect somewhat poorly on Miss Simonova’s character), I can’t say much about her save one important detail: she thought she and Alexander were legally married.”

“What?!” scoffed Xavier. “How is that possible?”

“Apparently she convinced the late Dr. Verawood to enter into some kind of contract with her that she believed legally tied the Verawood estate to her name. I think a lawyer of dubious qualifications and reputation was involved, but the

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contract will never stand up in a court of law. You can be certain that your brother knew this. It is, I think, rather akin to what is often called the ‘Vegas marriage.’ The point is I’m rather convinced that at the time of the murder she truly believed that she stood to inherit all that was your brother’s upon his demise. Ironic, isn’t it, that someone so adroit in the arena of social interaction should be so naïve when it comes to even the simplest of legal issues?”

Xavier didn’t ask how the old servant knew all this; he didn’t want to put Pendleton in the position of admitting to being a snoop. Instead, he said:

“Well, the next one is obvious. Alex told me in a letter that Zelda found out about Aurora and that the resulting tempest was cataclysmic.”

Pendleton shuddered, remembering. “That’s not too far from the mark. Mrs. Verawood has been getting increasingly agitated since that day in August, and the onset of winter has not lifted her spirits. Before she left here on Friday to stay at her parent’s estate, she had taken to brooding in her rooms. As you know, she’s always been one to hold in her feelings until they compel her to rashness, and when she left she seemed elated, as if her melancholy had lifted upon the making of some decision. And she was there, at the C & E building, from 8–9 p.m. on Sunday.”

Xavier hung his head. “I wish that poor Alex’s married life had worked out, but the truth is that they just weren’t made for each other. Naturally I’d like to cite Zelda’s intractably

spoiled attitude; I thought her a right termagant when Alex first met her. That’s probably prejudice, though, and my brother ought not to have behaved as he did in his private life.” He shrugged.

“Dr. Errol Curry,” said Pendleton.

“Errol grew up with us, as of course you remember. I can only recall the keen jealousy he felt for my brother, who was always his better in school, sports, and social relations. Alex treated him very magnanimously, as far as I know, but I think that that only seemed to infuriate Errol even more. Alex wrote to me last month and told me that the department, like everything else these days, is having serious financial problems and that one of the faculty would have to be cut. I’m sure Errol was afraid for his life. The poor man hasn’t done very well recently, or truthfully, at all, and perhaps he became rather desperate.”

“Though I do remember the jealous admiration with which he regarded your brother in childhood, I cannot lay claim to any knowledge about his professional situation. Still, we must include him in our consideration, for he was present at the building from 9 a.m. to 12 noon on Saturday, and from 9 a.m. to 8 p.m. on Friday. But who is this Tommy Benedict?”

“He’s the janitor,” replied Verawood, “who was scheduled to work this Saturday from 8 p.m. to 11 p.m. in the basement while no one was around. We know that the labs are locked all through the weekend, and he had one of the four keys. Curry and Miss de Morcef have the other two, I believe, tied as they are to the university. Alex, of course, had the fourth.

“That’s a sketchy connection,” continued Verawood, “But Benedict certainly warrants consideration on circumstance alone. Now, you said you had something to tell me about Gabriella.”

Pendleton steepled his hands and took on a rather ironic expression.

“My dear young Master Verawood, you won’t believe what I am about to tell you,” he began, “but our Miss Gabriella de Morcef has an acute case of hallucinogenic paranoia.”

“What?! What does that even mean? Could she have killed him?”

“Not only that, but she might not remember it.” Pendleton waited for the implications to sink in.

“The late Professor received a letter from her mother in southern France. She is, by the way, the progeny of rather noble French stock on her father’s side, but her mother was an Italian serving girl with whom her father became completely enamored during a trip to Italy in his youth. She turned out to be quite a graceful, intelligent, and virtuous woman who adapted to her new life without a hitch and delighted everyone around her, much like her daughter Gabriella.

“Your brother always raved about Gabriella’s unsurpassed creative and analytical prowess, how she saw to the heart

of even the most complex problems with a quick and sure dexterity that rivaled his own.

“That letter from her mother, however, told of an incident that occurred while Gabriella and the family were vacationing in Nice one June. I think that Gabriella’s mother felt that Dr. Verawood should hear about it. The details are delicate and not pressingly relevant, but the end result was that Gabriella had an episode in which she felt that her life was in danger by unseen beings—beings, apparently, that gibbered to her in the sounds that a wind-flapped shingle made on the roof of her family’s seaside villa. Luckily her father found her before she could do any harm, for I’m given to understand that when he entered Gabriella’s room he found to his horror that her terrified little sister was physically restrained and in danger of receiving a mortal injury by Gabriella’s hand. Gabriella remembered nothing the next morning. Since, the young woman has been treated with medication, which has eliminated these violent episodes. What concerns me about this is that your brother told me once, in passing, that she feels her medicine curtails her higher and more subtle mental abilities, and that she does sometimes skip her pills.”

Xavier remembered the delicate, brilliant woman from the lab, so excited by her research and full of potential, and he felt a shiver well up.

“She was there on Friday from 9 a.m. to 5 p.m., and again Sunday evening from 5 p.m. to 12 midnight. When we asked her this afternoon, she said she spent Sunday in the library and Friday in the lab, and she doesn’t recall seeing the Professor after his evening lecture. Pendleton, what can we say about the fact that no one reported the Professor’s death at all? If Hardcastle was the one, then my poor brother lay in his office for the whole weekend while everyone else was coming in and out; surely someone must have seen the murder scene?”

“True,” replied Pendleton, thoughtfully. “True. I think that we must assume that every suspect that might have entered the building after the murder either 1) didn’t open the door of the Professor’s office or 2) did open the office door, but didn’t report what he or she found, and it’s not really too unlikely that one or the other situation actually occurred.

“The former is not so hard to believe with Curry, Gabriella, and Tommy, who are likely to have not sought the professor out and would not have missed his absence. We can be certain that Hardcastle saw the Professor alive on Friday, though he may have murdered him at that time. If Hardcastle is not the murderer, then he simply left when his audience had concluded.

“As for Aurora and Mrs. Verawood ... well ... we can’t assume that they entered the Professor’s office just because we know they were in the building. To have both not murdered the Professor and to have discovered and not reported the Professor’s demise seems unlikely; therefore we can guess

that if one of these women is a murderess then the other one probably didn’t enter the room.”

Pendleton had finished and sat quietly, musing. Xavier’s gaze turned again to the window, through which he looked forlornly at the dark, flowing Miskatonic under the night sky. The minutes ticked by on the grandfather clock that had stood in the study since his grandfather’s time. Sighing, Xavier looked back at Pendleton. Curiously, Pendleton was staring intently at the suspect list, which had been flipped over to reveal some of the late Professor’s old calculation pages, scribbled on the paper. “What is it, Pendleton?” whispered Xavier.

“I know a little something about logic and engineering after all my years in this house. Xavier, the murderer can only be one person: I know how to find out who killed Dr. Alexander Verawood, and we can do it without leaving the room.”

SOLUTION

First, it is necessary to determine the old Professor’s cause of death. We only know that there were no marks or trauma on his body, that he knew he was being murdered (hence the note), and that a doctor gave “heart attack” as the cause of death.

Now, we must think back to a few details. The Professor’s office is a converted utility closet located underground; probably having poor ventilation and no windows. Gabriella, the lab assistant, wheels a cartload of empty CO₂ tanks into place when the two sleuths enter the laboratory; they help her unload them to the floor. The door to the Professor’s office has no lock on it, but opens outward.

Now we think back to the empty CO₂ tanks that Gabriella was moving. They were too large to move by hand and therefore unlikely to be misplaced by chance, which should set us thinking that CO₂, in very large quantities and in a small space, is as deadly as any poison.

The scene could have played out something like this: the Professor, minutes before he was killed, was cooking his evening meal on the little burner. Feeling a little hot under the collar, he gets up to stretch his legs, only to find that his shortness of breath gets worse. After a few minutes, he feels a pain in his chest and, desiring of fresh air, moves to his door, turns the handle, and pushes, only to find it blocked! Wondering what the obstruction could be, the Professor must have kept pushing with increasing worry as his head began to swim with lack of air. Sinking to the floor, he must have seen or heard the CO₂ being pumped into his office, decreasing the oxygen content of each of his breaths, and must have realized, at that moment, that his life was in danger. Before blacking out, he had time to leave his last note.

It was seemingly the perfect crime: no evidence, no fingerprints, no sign of a struggle, no need for the murderer to even enter the room. However, there was one more thing the murderer didn’t count on.

When the oxygen concentration of the room dropped, the Professor's life was not the only thing to snuff out! Indeed, the burner's pilot light could no longer continue to burn, but extinguished, allowing pure fuel gas to gush into the room. This is what Xavier and Pendleton smelled, and was why they didn't notice how thin the air was, for they were already holding their breath.

Now, the fuel gas was comprised of a 3:1 mol methane : mol ethane mixture and stored in a tank with a capacity of 2 ft³ (56.63 L). Making the reasonable assumption that the Professor's basement office is at room temperature (77 °F, 25 °C) and 1 atm, we use Yaw's Chemical Properties Handbook to calculate the heat of combustion at 958.9 kJ/g-mol, making the assumption that the water product of combustion is in the vapor phase.

Gabriella mentions that the burner is 90% efficient and heats one pint (0.473 L) water to boiling in 3 min. Therefore, the flow rate of fuel into the burner must be 0.0574 g-mol/min, assuming everything at roughly STP, assuming that the heat capacity of water is a constant 4.187 kJ/kg*K, and assuming that the density of water at 25 °C is 1 kg/L.

We also know that the 2 ft³ tank was filled up to 1,500 psig

(10,440 kPa abs) before the Professor started using it, and that it was at 713 psig (5,020 kPa abs) at 9:00 a.m. on Monday. So, we have only to find how long the fuel had been escaping, then count backward to find out when the flame went out.

That's easy: using the Ideal Gas Law at the lab temp, which we know is 25 °C, we find that the difference between the two pressures of the tank corresponds to a loss of about 124 g-mol fuel gas. Now, dividing 124 g-mol by 0.0574 g-mol/min tells us that the fuel had been escaping from the burner for about 36 hours (2,160 min).

Counting backward from 9:00 a.m. on Monday, that means that the fuel began escaping at 9:00 p.m. on Saturday. There were two people present in the building at that time: Tommy Benedict and Aurora Simonova, but only Tommy had the keys to the lab (which is locked on the weekends) to get to the CO₂ tanks!

It turns out that the Professor, to supplement his skimpy funds, was running a distillery as a part of his yeast experiments in the basement. This, of course, was a risky occupation during Prohibition, and so Tommy, who worked for a local mob boss, was persuaded to eliminate the competition. □

The object of this column is to enhance our readers' collections of interesting and novel problems in chemical engineering. Problems of the type that can be used to motivate the student by presenting a particular principle in class, or in a new light, or that can be assigned as a novel home problem, are requested, as well as those that are more traditional in nature and that elucidate difficult concepts. Manuscripts should not exceed 14 double-spaced pages and should be accompanied by the originals of any figures or photographs. Please submit them to Professor James O. Wilkes (e-mail: wilkes@umich.edu), Chemical Engineering Department, University of Michigan, Ann Arbor, MI 48109-2136.

EQUILIBRIUM-STAGED SEPARATIONS USING MATLAB AND MATHEMATICA

HOUSAM BINOUS

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Unit operations are ubiquitous in any chemical process. One of their main purposes is to achieve separation in order to recycle unconverted reactants or reach product purity specifications. If two phases are brought into intimate contact, a few components migrate from one phase to the other in every stage of the unit. Then, the two phases are separated. When we consider equilibrium-staged separations, we assume that both phases leaving each stage are in equilibrium and one is richer in one or more components. The most common unit operation is distillation. It is used, for instance, to separate crude petroleum into its various fractions such as kerosene and gasoline. In the pharmaceutical industry, liquid-liquid extraction is often employed. For example, antibiotics in aqueous fermentation are recovered using organic solvents. Finally, absorption and stripping are often involved in gas treatment plants—such as units used to remove acid gases (CO_2 and H_2S) from natural gas. In the present paper, we solve two problems while showing how graphical methods can be used to design the unit operations.

The first problem treats liquid-liquid extraction while the second problem deals with binary distillation.

EXTRACTION OF ACETIC ACID FROM WATER USING ISOPROPYL ETHER

Problem Statement

We want to separate^[1] acetic acid from water using isopropyl ether in a single-feed countercurrent extractor. Use the



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The author brings to the reader's attention that a program to determine the number of equilibrium stages using the Graphical User Interface (GUI) of Matlab is available.¹⁸ This GUI program, written by Claudio Gelmi, was inspired by the earlier code of Housam Binous.

Hunter and Nash²¹ Graphical approach to compute the number of equilibrium stages required. Assume equilibrium data, at 1 atm and 25 °C, given by Wankat.¹¹ Solvent and feed flow rates are equal to 1,000 kg/hr and 1,475 kg/hr, respectively. Feed, a binary mixture of water and acetic acid, contains 35 wt % acetic acid. We wish to obtain a raffinate with only 10 wt % acetic acid.

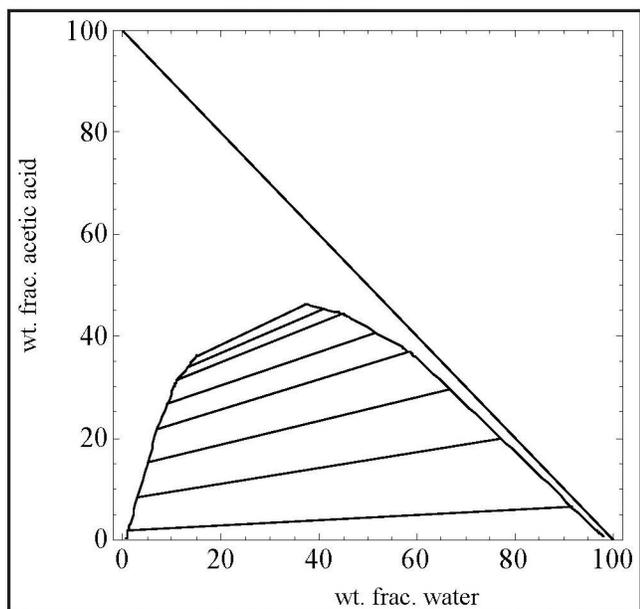
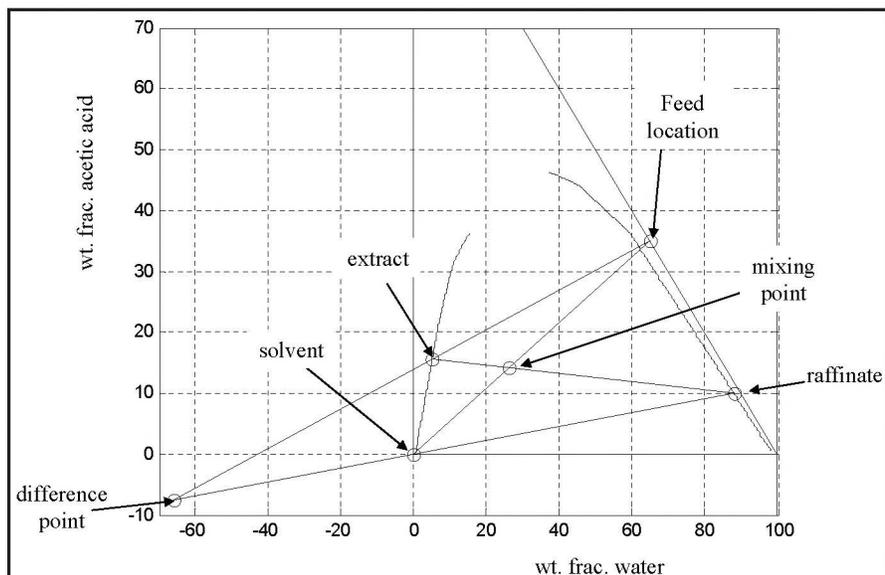


Figure 1. Equilibrium diagram for the ternary system obtained using Mathematica.



Then, we step off stages using alternatively the tie lines and the operating lines until we reach a raffinate composition lower than 10 wt %. This method is illustrated in Figure 3, obtained using Matlab. A similar figure was obtained by the author using Mathematica. We find that 5.35 equilibrium stages are required to achieve 10 wt % of acid in the raffinate. The Mathematica notebooks and Matlab programs are available from the author upon request or from the Mathematica Information Center⁴⁴ and Matlab File Exchange Center.⁵⁵

SEPARATION OF A MIXTURE OF BENZENE AND TOLUENE

Problem Statement

Consider a binary mixture composed of benzene and toluene. Assume a constant relative volatility equal to 2.45. Use

◀ **Figure 2.** Mixing and operating points obtained using Matlab.

Solution

To study the separation¹¹ of acetic acid from water using isopropyl ether for a single-feed countercurrent extractor, we will use the graphical method derived by Hunter and Nash²¹ and later by Kinney³¹ to obtain the number of equilibrium stages required to achieve a particular raffinate specification. This separation is usually difficult to realize with distillation due to the presence of a severe tangent pinch at high compositions of water, which prevent the distillate from being acid-free. A graphical solution performed by hand calculation has been presented by Wankat.¹¹ We will show how one can use Mathematica and Matlab to solve this problem more easily and accurately using the graphical approach of Hunter and Nash.²¹ Equilibrium data, at 1 atm and 25 °C, are given by Wankat¹¹ and can be used to plot Figure 1—where the tie lines have been drawn (obtained using Mathematica). The same figure was obtained by the author using Matlab. Note that the conjugate line can be easily constructed using the equilibrium points. The feed is 1,000 kg/hr of a solution composed of 35 wt % acetic acid and 65 wt % water. The solvent used to perform the extraction is pure isopropyl ether with a flow rate equal to 1,475 kg/hr. We require that the raffinate composition in acid not exceed 10 wt %. First, we locate the mixing point and the difference point (see Figure 2 obtained using Matlab). Then, we step off stages using alternatively the tie lines and the operating lines until we reach a raffinate composition lower than 10 wt %.

the McCabe and Thiele graphical approach to compute the number of equilibrium stages required. Assume feed, bottom, and distillate mole fractions are equal to 0.5, 0.1, and 0.9 mole % benzene, respectively. Take a reflux ratio 1.5 times greater than the minimum reflux ratio.

Solution

We want to separate a binary ideal mixture formed of benzene and toluene. The relative volatility in this ideal case is the ratio of the vapor pressures of benzene and toluene. A straightforward calculation shows that this mixture's relative volatility varies only between 2.35 and 2.6 for a benzene mole fraction ranging from 0 to 1. Thus, as a simplifying assumption, we take an average value of 2.45. The feed is a two-phase mixture with a feed quality equal to 0.85. The distillate, feed, and bottom mole fractions are 0.9, 0.5, and 0.1, respectively. The reflux ratio is taken to be 1.514, which is approximately 1.5 times larger than the minimum reflux ratio. To obtain the minimum reflux ratio, we require that the two operating lines, the feed line, and the equilibrium curve intersect, as shown in Figure 4—which was obtained using Mathematica. Then, we draw the McCabe and Thiele Diagram (see Figure 5 [next page], obtained using Matlab). A similar figure was obtained by the author using Mathematica. We conclude that the required

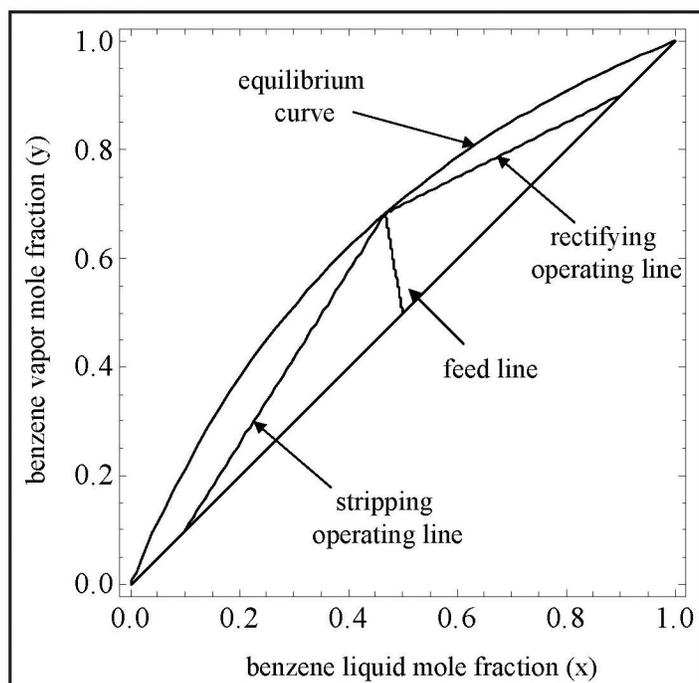


Figure 4. Finding the minimum reflux ratio using Mathematica.

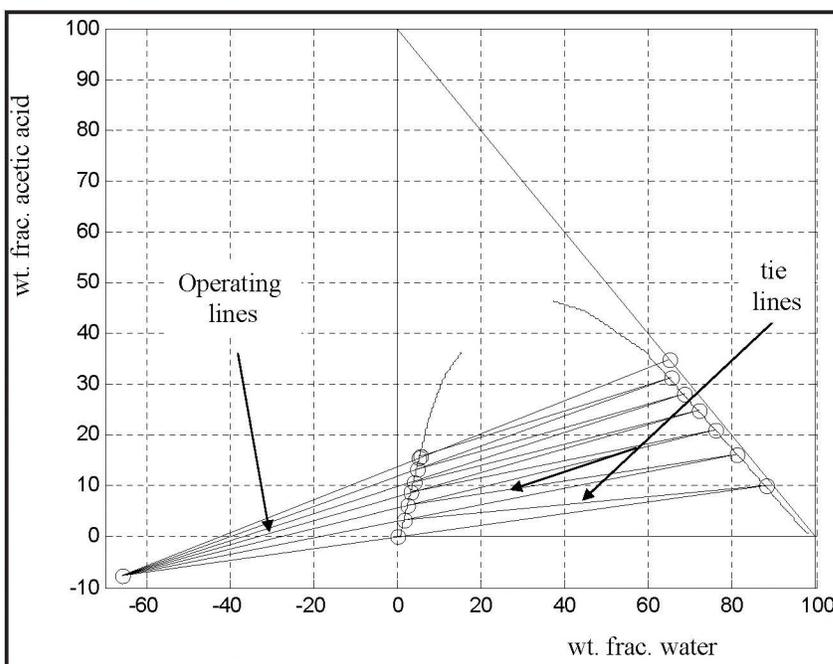


Figure 3. Hunter and Nash diagram obtained using Matlab.

number of theoretical plates, to achieve product and bottom specifications, equals nine. The Mathematica notebooks and Matlab programs are available from the author upon request or at the Mathematica Information Center^[6] and the Matlab File Exchange Center.^[7] The author brings to the reader's attention that a program to determine the number of equilibrium stages using the Graphical User Interface (GUI) of Matlab is available.^[8] This GUI program, written by Claudio Gelmi, was inspired by the earlier code of Housam Binous.

THE MANIPULATE COMMAND OF MATHEMATICA 6.0

Problem Statement

Use the new dynamic capabilities of Mathematica to prepare a demonstration where parameters such as relative volatility, feed composition, and solvent and feed flow rates . . . can be manipulated by sliders to study their impact on the number of equilibrium stages.

Solution

The output of Manipulate, a new function available in the new version of Mathematica (version 6.0), is an interactive object containing sliders. The output is not just a static result, it is an interactive program that allows the user to specify several selected simulation parameters using the sliders. The program then performs the computation and displays the result, usually in the form of a plot or a table. The slider is a control object, which can be dragged by the user in order to set a specified value of one parameter. The author has modified his previous program using Math-

ematica (version 5.2) to take advantage of this new feature. Figure 6 shows how the liquid-liquid extraction static simulation has been modified into an interactive program where one can change the solvent and feed flow rates, the raffinate

specification, and the acetic acid composition in the feed. Figure 7 presents the simulation where sliders have been set up for feed quality, feed composition, distillate and bottom specifications, and relative volatility. These programs are

available, among hundreds of demonstrations made with Manipulate, at the Wolfram Demonstration Project.^[9-11] In both cases the program displays the number of equilibrium stages.

MATHEMATICA AND MATLAB: TOOLS FOR TEACHING UNIT OPERATIONS

Teaching unit operations requires presenting to undergraduate students graphical methods such as the McCabe and Thiele or the Hunter and Nash model. These methods allow for the determination of the number of equilibrium stages for liquid-liquid extraction, distillation and absorption, and stripping problems. With powerful computer software such as Mathematica and Matlab, chemical engineering faculties can show readily how to compute the number of equilibrium stages for these classic junior- or senior-level problems. The author uses this opportunity not only to introduce students to separation science but also to convince them that Mathematica and Matlab can be applied to many other interesting chemical engineering and applied mathematics problems.

CONCLUSION

In this study, we show how Mathematica and Matlab can be used to solve problems that previously required tedious numerical and graphical techniques. First, we present the extraction of acetic acid from water using isopropyl ether. The graphical method, derived by Hunter and Nash, is used to obtain the number of equilibrium stages for a specific raffinate purity. Next, the McCabe-Thiele method is employed to give the number of equilibrium stages for a binary distillation problem. These classic problems are junior- and senior-level study material at the National Institute of Applied Sciences in Tunis. Students excel in these types of problems despite the fact that it is their first experience using Mathematica and Matlab.

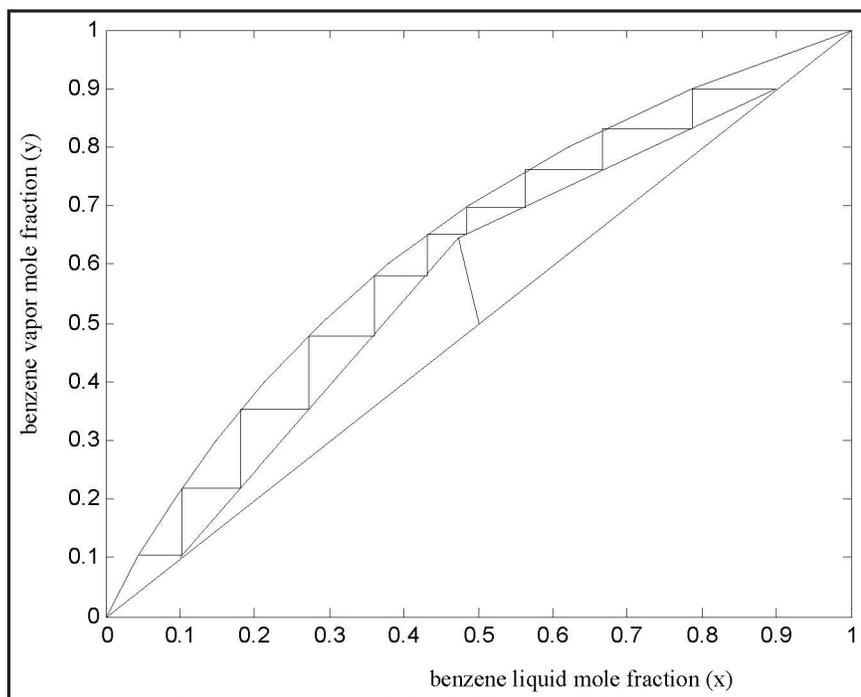


Figure 5. McCabe and Thiele Diagram obtained using Matlab.

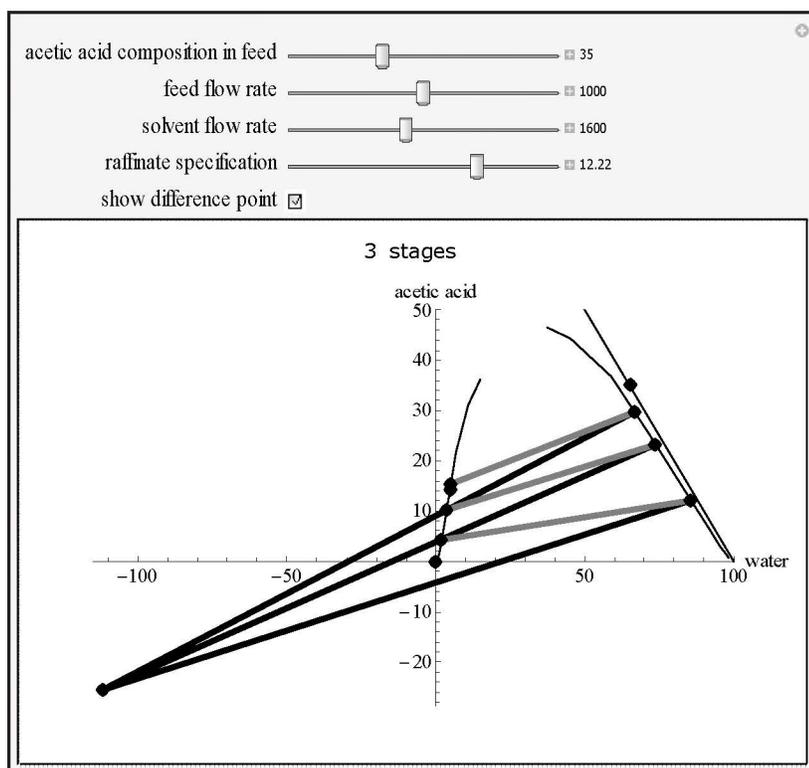


Figure 6. Liquid-Liquid extraction using the Manipulate command.

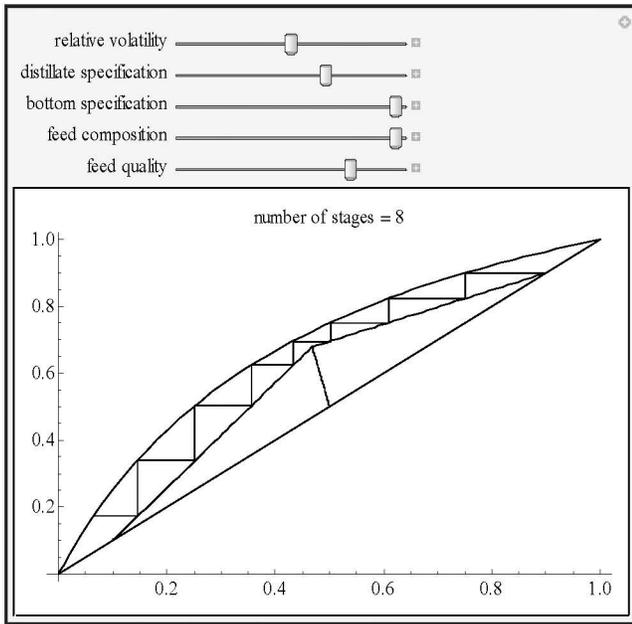


Figure 7. Distillation using the Manipulate command.

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XSEOS: AN OPEN SOFTWARE FOR CHEMICAL ENGINEERING THERMODYNAMICS

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Before computers, predictions of physical properties relied on graphical methods, correlations, and simple analytical models. Advances in molecular thermodynamics, statistical mechanics, and computer technology have changed this scenario forever. Thermodynamic models that take many details at molecular level into account are now available for several types of mixtures. Complexity usually prevents full derivations of these models in undergraduate classrooms, but it is possible to sketch their major assumptions and discuss how they translate into model capabilities and limitations. With this sort of background, students can apply these models to solve interesting computer-based chemical engineering thermodynamics problems.

There are different approaches to using these models in undergraduate courses. A possibility is to ask students to program the models they need, requiring many hours of development and debugging. Another approach is to use professional software such as ASPEN, HYSYS, or Simulis Thermodynamics, in which students select the components, physical property package, and type of calculation, and get results immediately. They may be left, however, with little understanding of the steps involved in a phase or chemical equilibrium calculation and there is little or no access to the code—frustrating curious students. An intermediate approach is to use open software

for use in software like Matlab, Mathcad, or Excel, relieving students of the burden of writing long programs, yet enabling them to recombine parts of the code into new applications.

Many recent editions of textbooks on chemical engineering thermodynamics and related subjects provide computer programs as supplementary material (*e.g.*, Smith, et al.,^[1] Sandler,^[2] Elliot and Lira,^[3] Koretsky,^[4] Kyle,^[5] and Cutlip and Shacham^[6]). These programs often run in professional software such as Matlab, Mathcad, or Excel, or as stand-alone software, such as the well-designed ThermoSolver^[7] package that accompanies Koretsky's book.^[4] Another stand-alone software freely available is the VLE-Java program,^[8] which performs phase equilibrium calculations for binary mixtures



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using five different thermodynamic models.

An additional aspect, not to be neglected, is cost. Educational versions of professional software are available and affordable to many institutions and/or individuals, but this is not always the case—especially in developing countries. Availability and ease of use have made Excel a platform of choice for educational engineering software and, for example, its use in teaching chemical process design and integration has been recently discussed.^[9] Actually, Excel allows different levels of programming. Developers can implement their models in Visual Basic for Applications (VBA) as functions that users can easily call from the spreadsheet. For example, long calculations such as those of activity and fugacity coefficients and residual and excess properties can be programmed in VBA and made available as spreadsheet functions that students can use to develop procedures for calculations such as bubble and dew points and flashes, without extensive knowledge of programming languages.

Elliot and Lira^[9] followed this type of approach and developed excellent Excel spreadsheets freely available on the Internet. Some of the newest models, however, are not yet available in their spreadsheets. Another limitation is that some of the models are implemented for binary mixtures only or for mixtures with up to five components. Therefore, even though the Elliot and Lira spreadsheets are an outstanding educational tool, improvements are possible.

This work presents a new computational package, XSEOS, primarily intended for use in undergraduate courses but which may also be useful for graduate courses and research. XSEOS

—Excess Gibbs Free Energy Models and Equations of State —is a freely available program with many traditional and modern thermodynamic models. Moreover, XSEOS is an expandable platform with open source code that will hopefully contribute to the exchange of experiences in the teaching of chemical engineering thermodynamics.

XSEOS

The underlying concept in XSEOS is that students should have easy access to calculations using modern thermodynamic models to become aware of their existence and learn how to use them. The calculation experience, however, should not be limited to getting results from ready-to-use programs. Instead, students should engage in developing calculation procedures but not in long program development. For example, students can implement a bubble point algorithm using an Excel spreadsheet, and then use the functions available in XSEOS to compute the activity or fugacity coefficients required by their calculations. The XSEOS add-in does not contain ready-to-use VBA procedures for chemical equilibrium, flash, dew, and bubble point calculations, but XSEOS provides the physical properties required by these calculations. Furthermore, the XSEOS add-in does not contain VBA functions for ideal gas and ideal solution properties, as it is simple to program them in a spreadsheet and their implementation is a good exercise for students.

One of the major goals of XSEOS is to provide an environment where students can rapidly develop and test calculation procedures and compare the results of different models. Because ease of use was the major goal, the computer codes are not as efficient as they could have been if execution speed was the top priority. Nonetheless, speed of execution was never a limitation when XSEOS was tested in undergraduate and graduate courses at the United Arab Emirates University (UAEU), where students used the package in laptop computers to solve classroom and homework problems and develop course projects.

Table 1 presents the models and properties currently available in the program. In all cases, with exception of the Margules model, users can perform calculations with any number of components, only constrained by Excel, the operating system, and computer limits. The core procedures for all thermodynamic models were automatically written in VBA using Thermath,^[10] a program for the implementation of thermodynamics models based on Mathematica. They essentially implement formulas for many physical properties derived from a given thermodynamic model. The procedures that implement equa-

TABLE 1 Thermodynamic Models and Properties Currently Available in XSEOS		
	GE models	Equations of state
Models	Margules 2-, 3-, and 4- suffix* Regular solution theory Flory-Huggins Wilson TK-Wilson NRTL UNIQUAC UNIFAC Modified UNIFAC	van der Waals Redlich-Kwong Soave-Redlich-Kwong (SRK) Peng-Robinson Stryjek-Vera Predictive SRK (PSRK) Mattedi-Tavares-Castier
Properties	$\frac{g^E}{RT}, \frac{h^E}{RT}, \frac{s^E}{R}, \frac{c_p^E}{R},$ $\ln \gamma_i, \left(\frac{\partial \ln \gamma_i}{\partial T} \right)_x, n \left(\frac{\partial \ln \gamma_i}{\partial n_j} \right)_{T, n_{k \neq j}}$	$P, \left(\frac{\partial P}{\partial p} \right)_{T, x}, \left(\frac{\partial^2 P}{\partial p^2} \right)_{T, x}, \left(\frac{\partial^3 P}{\partial p^3} \right)_{T, x}, \ln \phi,$ $\ln \phi_i, \left(\frac{\partial \ln \phi_i}{\partial P} \right)_{T, x}, n \left(\frac{\partial \ln \phi_i}{\partial n_j} \right)_{T, P, n_{k \neq j}},$ $n \left(\frac{\partial V}{\partial n_j} \right)_{T, P, n_{k \neq j}}, \left(\frac{\partial \ln \phi_i}{\partial T} \right)_{P, x}, \left(\frac{\partial v}{\partial T} \right)_{P, x},$ $\frac{h^R}{RT}, \frac{s^R}{R}, \frac{c_p^R}{R}, \left(\frac{\partial P}{\partial T} \right)_{v, x}$

* The implemented Margules models work for binary mixtures only. All other models currently available in XSEOS work with any number of components.

tions of state also need a root-finding numerical method to determine molar volumes in the liquid or vapor phases: the Toplis, et al.,^[11] method was used because it is applicable to cubic and noncubic equations of state. These procedures are not used directly from the spreadsheet, as front-end procedures with more user-friendly calling protocols have been developed manually. The manual distributed with the program lists the procedures and explains how to use them.

SAMPLE CALCULATIONS

This section presents two sample calculations. In the figures that support this discussion, cell formats are simple. The sample spreadsheet distributed with XSEOS uses colors consistently to indicate cells with specified and calculated values.

Bubble pressures of the mixture methanol(1) + water(2) at 333.15 K

The objective of this example is to compute the bubble pressure of the mixture methanol(1) + water(2) at 333.15 K for several liquid phase compositions. Assuming ideal gas phase behavior, neglecting the Poynting correction and the fugacity coefficient at saturation, the total pressure (P) is given by:

$$P = x_1 \gamma_1 P_1^{\text{sat}} + x_2 \gamma_2 P_2^{\text{sat}} \quad (1)$$

where x_i , γ_i , and P_i^{sat} represent the mole fraction, activity coefficient, and vapor pressure of component i . Activity coefficients will be calculated using the UNIQUAC model. Figure 1(a) shows the spreadsheet before the beginning of calculations, with entries for the universal gas constant (in J/mol), temperature, vapor pressures of methanol and water, and UNIQUAC r , q , and binary interaction parameters (a_{ij}). Cell C10, which represents x_2 , is programmed as 1-B10 (i.e., 1- x_1) and, whenever the value of x_1 changes, the corresponding x_2 value is automatically recomputed. XSEOS has a function named `uniquac` that takes the universal gas constant, temperature, mole fractions, and UNIQUAC parameters as inputs to compute the logarithms of activity coefficients as outputs. This function, as well as many others in XSEOS, returns more than one output value. Functions of this type in Excel are called array functions. To use an array function, the user must mark the empty spreadsheet cells that will receive the output before

typing the function call. In Fig. 1(b), cells D10 and E10 are marked to receive the values of $\ln \gamma_1$ and γ_2 , and the function call is in preparation. In Fig. 1(c) the calling statement of function `uniquac` is ready but has not been executed yet. Execution occurs by pressing the keys CTRL+SHIFT+ENTER simultaneously. Figure 1(d) shows the results of this action and Fig. 1(e) shows the set of results for the row. In this figure, cells F10 and G10 were calculated by taking the exponential of cells D10 and E10, cell H10 implements Eq. (1), and cells I10 and J10 implement:

$$y_i = \frac{x_i \gamma_i P_i^{\text{sat}}}{P} \quad (2)$$

Having computed the results for the first liquid phase composition, it is simple to compute for a series of compositions. The value of cell B11 in Fig. 1(f) (the value of x_1 in the second point of the composition series) is defined as the value of cell B10 (the value of x_1 in the first point of the composition series) plus a step size, taken here as 0.25. One can then mark cells C10 to J10 and drag the Fill Handle (the small black square that appears in the bottom right corner of the marked region) to row 11. All the values, from x_2 to y_2 are automatically recalculated. By now marking cells B11 to J11 and dragging the Fill Handle to lower rows, results from x_1 to y_2 for additional compositions are computed. Figure 1(f) shows the results of these operations that can be used, for example, to prepare a Pxy diagram using the plotting facilities available in Excel.

Flash calculation by direct minimization of the Gibbs function

Minimization of the Gibbs function for a system with specified values of temperature, pressure, and initial amounts of each component is a fundamental criterion for thermodynamic equilibrium. In most undergraduate textbooks, however, the most common numerical approach is to solve the isofugacity equations together with the mass balances, often after considerable algebraic rearrangement. A flash calculation by minimization of the Gibbs function is interesting because it illustrates the direct application of a fundamental criterion in an actual computation.

	A	B	C	D	E	F	G	H	I	J
1	methanol(1)+water(2)									
2		R	T(K)							
3		8.314	333.15							
4	r	1.4311	0.92							
5	q	1.432	1.4							
6	a(i,j) (J/mol)	0	-1777		P1sat (kPa)	84.562				
7		2981	0		P2sat (kPa)	19.953				
8										
9		x1	x2	lngama1	lngama2	gama1	gama2	P (kPa)	y1	y2
10		0	1							

Figure 1 a-f: Bubble point calculations using the UNIQUAC model. (a) Spreadsheet before the beginning of calculations.

	A	B	C	D	E	F	G	H	I	J
1	methanol(1)+water(2)									
2		R	T(K)							
3		8.314	333.15							
4	r	1.4311	0.92							
5	q	1.432	1.4							
6	a(l,j) (J/mol)	0	-1777		P1sat (kPa)	84.562				
7		2981	0		P2sat (kPa)	19.953				
8										
9		x1	x2	lngama1	lngama2	gama1	gama2	P (kPa)	y1	y2
10		0	1	=uniquac(

(b) The user must mark the cells for $\ln\gamma_1$ and $\ln\gamma_2$ before typing the call of function UNIQUAC.

	A	B	C	D	E	F	G	H	I	J
1	methanol(1)+water(2)									
2		R	T(K)							
3		8.314	333.15							
4	r	1.4311	0.92							
5	q	1.432	1.4							
6	a(l,j) (J/mol)	0	-1777		P1sat (kPa)	84.562				
7		2981	0		P2sat (kPa)	19.953				
8										
9		x1	x2	lngama1	lngama2	gama1	gama2	P (kPa)	y1	y2
10		=uniquac(\$B\$3,\$C\$3,B10:C10,\$B\$4:\$C\$7)								

(c) UNIQUAC function call.

	A	B	C	D	E	F	G	H	I	J
1	methanol(1)+water(2)									
2		R	T(K)							
3		8.314	333.15							
4	r	1.4311	0.92							
5	q	1.432	1.4							
6	a(l,j) (J/mol)	0	-1777		P1sat (kPa)	84.562				
7		2981	0		P2sat (kPa)	19.953				
8										
9		x1	x2	lngama1	lngama2	gama1	gama2	P (kPa)	y1	y2
10		0	1	0.867	0.000					

(d) Numerical values after the UNIQUAC function call.

	A	B	C	D	E	F	G	H	I	J
1	methanol(1)+water(2)									
2		R	T(K)							
3		8.314	333.15							
4	r	1.4311	0.92							
5	q	1.432	1.4							
6	a(l,j) (J/mol)	0	-1777		P1sat (kPa)	84.562				
7		2981	0		P2sat (kPa)	19.953				
8										
9		x1	x2	lngama1	lngama2	gama1	gama2	P (kPa)	y1	y2
10		0	1	0.867	0.000	2.380	1.000	19.953	0	1

(e) Set of results for the specified liquid phase composition.

	A	B	C	D	E	F	G	H	I	J
1	methanol(1)+water(2)									
2		R	T(K)							
3		8.314	333.15							
4	r	1.4311	0.92							
5	q	1.432	1.4							
6	a(l,j) (J/mol)	0	-1777		P1sat (kPa)	84.562				
7		2981	0		P2sat (kPa)	19.953				
8										
9		x1	x2	lngama1	lngama2	gama1	gama2	P (kPa)	y1	y2
10		0	1	0.867	0.000	2.380	1.000	19.953	0	1
11		0.25	0.75	0.324	0.067	1.383	1.069	45.232179	0.6461642	0.3538358
12		0.5	0.5	0.109	0.191	1.115	1.210	59.215951	0.7960597	0.2039403
13		0.75	0.25	0.022	0.331	1.022	1.393	71.776916	0.9031892	0.0968108
14		1	0	0.000	0.478	1.000	1.612	84.562	1	0

(f) Complete set of results.

The objective of this example is to solve an isothermal flash of a ternary mixture of n-hexane(1) + n-octane(2) + n-decane(3) using the Peng-Robinson equation of state by direct minimization of the Gibbs function. Figure 2(a) presents the information required to formulate the problem, *i.e.*, the values of critical pressures and temperatures, acentric factors, binary interaction parameters (assumed equal to zero), temperature and pressure at equilibrium and the global number of moles of each substance in the system. The Gibbs function (G) of a system with two phases is:

$$G = \sum_{j=1}^2 \sum_{i=1}^c n_{ij} \mu_{ij} = \sum_{j=1}^2 \sum_{i=1}^c n_{ij} \left(\mu_i^{\text{ig,pure}}(T, P_0) + RT \ln \left(\frac{x_{ij} \hat{\phi}_{ij} P}{P_0} \right) \right) = \sum_{i=1}^c n_i \left(\mu_i^{\text{ig,pure}}(T, P_0) - RT \ln P_0 \right) + RT \sum_{j=1}^2 \sum_{i=1}^c n_{ij} \ln(x_{ij} \hat{\phi}_{ij} P) \quad (3)$$

where n_{ij} , x_{ij} , μ_{ij} , and $\hat{\phi}_{ij}$ represent the number of moles, mole fraction, chemical potential, and fugacity coefficient of component i in phase j . The symbols c , T , and P_0 denote the number of components, the temperature, and a reference pressure, respectively. The superscripts *ig* and *pure* refer to the ideal gas state and to a pure substance, respectively. From Eq. (3), it follows that:

$$\frac{\Delta G}{RT} = \frac{1}{RT} \left(\sum_{j=1}^2 \sum_{i=1}^c n_{ij} \mu_{ij} - \sum_{i=1}^c n_i \left(\mu_i^{\text{ig,pure}}(T, P_0) - RT \ln P_0 \right) \right) = \sum_{j=1}^2 \sum_{i=1}^c n_{ij} \ln(x_{ij} \hat{\phi}_{ij} P) \quad (4)$$

It can be shown that the minimum of $\frac{\Delta G}{RT}$ given by Eq. (4) corresponds to the phase equilibrium condition. Figure 2(b) shows the implementation of Eq. (4) with all the required intermediate steps. The numbers of moles of each component in the vapor phase (cells B16, C16, and D16) are taken as independent variables in the minimization problem, and the values in these cells in Figure 2(b) are just initial guesses. The

	A	B	C	D	E
1		n-C6(1)	n-C8(2)	n-C10(3)	
2	Tc(K)	507.5	588.8	617.7	
3	Pc(bar)	30.1	24.9	21.2	
4	omega	0.299	0.398	0.489	
5	kij	0.0	0.0	0.0	
6		0.0	0.0	0.0	
7		0.0	0.0	0.0	
8					
9	R (bar.cm³/(mol.K))	83.14			
10	Temperature (K)	450			
11	Pressure (bar)	4.5			
12					
13	Moles	n-C6(1)	n-C8(2)	n-C10(3)	Total
14	Global	40	30	30	100.000

Figures 2: Flash calculation of a ternary mixture using the Peng-Robinson equation of state. (a) Spreadsheet before the beginning of calculations.

numbers of moles in the liquid phase (cells B15, C15, and D15) are calculated from the mass balances, *i.e.*, by difference with respect to the specified total amounts (cells B14, C14, and D14). The mole fraction cells, from B19 to D21, are computed using the values present in the mole number cells. Two array functions, `prlnphil` and `prlnphiv`, use the Peng-Robinson equation of state to compute the logarithms of the fugacity coefficients in the liquid and in the vapor phases, respectively. Please refer to the first section under Sample Calculations for details on the use of array functions in Excel. The arguments in these functions are the universal gas constant, temperature, pressure, phase mole fractions (either liquid or vapor), and equation of state parameters (cells B2 to D7). The additional cells in the spreadsheet (B28 to D29, B32, and B33) represent the contribution of the liquid and vapor phases in the evaluation of Eq. (4). Cell B34 contains the cell to be minimized. Excel Solver is used to minimize cell B34 by modifying the values of cells B16, C16, and D16, as shown in Fig. 2(b). Figure 2(c) shows the spreadsheet after problem solution. By comparing Figs. 2(b) and 2(c), observe that the changes made by Excel Solver in the vapor mole numbers (cells B16, C16, and D16) propagate to the number of moles in the liquid phase, mole fractions, and all other physical properties. An interesting point to discuss in the classroom is that cells B28 and B29, C28 and C29, and D28 and D29 are pairwise equal because they are the natural logarithms of fugacities, *i.e.*, students can observe the isofugacity criterion for phase equilibrium appearing naturally from a numerical, as opposed to analytical, formulation.

USE OF THE PROGRAM IN COURSES

A preliminary version of the program, with different installation procedure but similar functionality, was used during the Spring of 2007 in the graduate course of Fluid Phase Equilibria (four students) and in two sections of the undergraduate course of Chemical Engineering Thermodynamics (about 25 students) at the UAEU. The current version was used in the only section of Chemical Engineering Thermodynamics (18 students) taught in the Fall of 2007.

Because experience with XSEOS in graduate courses is limited, the discussion focuses on the undergraduate course of Chemical Engineering Thermodynamics at the UAEU. The first half of this course consists of a review of the first and second laws of thermodynamics followed by a discussion about free energies, residual and excess properties, and fugacity calculations. The second half of the course focuses on formulating and solving phase equilibrium and chemical equilibrium problems. Students who take this course are usually familiar with using Excel to program formulas and prepare plots.

Use of the program consisted of two types of activities related to phase equilibrium and, to a lesser extent, chemical equilibrium calculations: (a) instructor-guided

classroom solution of problems with each student working on his/her own laptop computer, and (b) development of a course project in groups of two or three students. These activities roughly took place during the final third of the course. Table 2 (next page) presents some of the projects students developed using XSEOS. Intrinsic numerical procedures available in Excel, such as Goal Seek and Solver, were used in these projects, but instructors and students willing to develop Newton-Raphson-based calculation procedures will find the analytical derivatives required by many problems already implemented in the package (Table 1).

Student feedback about the preliminary version of XSEOS was informal. A formal anonymous survey about the current version was answered by 16 out of the 18 students who took the undergraduate Chemical Engineering Thermodynamics course in the Fall of 2007. Students should “strongly disagree” (1 point), “disagree” (2 points), “be neutral” (3 points), “agree” (4 points), or “strongly agree” (5 points) with each of seven statements presented to them.

Table 3 (next page) shows the statements and the corresponding average points. The first two statements were intended to estimate the level of understanding acquired by using the program. They originated scattered opinions. Because the survey was anonymous, it was not possible to

	A	B	C	D	E
13	Moles	n-C6(1)	n-C8(2)	n-C10(3)	Total
14	Global	40	30	30	100.000
15	Liquid	17.206	20.436	25.613	63.255
16	Vapor	22.794	9.564	4.387	36.745
17					
18	Mole fractions	n-C6(1)	n-C8(2)	n-C10(3)	
19	Global	0.400	0.300	0.300	
20	Liquid	0.272	0.323	0.405	
21	Vapor	0.620	0.260	0.119	
22					
23	In phi	n-C6(1)	n-C8(2)	n-C10(3)	
24	Liquid	0.743	-0.365	-1.438	
25	Vapor	-0.082	-0.149	-0.216	
26					
27	ln x(i) + ln phi(i) + ln P	n-C6(1)	n-C8(2)	n-C10(3)	
28	Liquid	0.945	0.009	-0.838	
29	Vapor	0.945	0.009	-0.838	
30					
31	delta G/(RT)				
32	Liquid	-5.009			
33	Vapor	17.952			
34	Global	12.943			

Figure 2 (c). Spreadsheet with the problem solution.

pinpoint the reason, but a possible explanation is that students who led their project groups developed a deeper understanding of the methods and calculations. Statement 3 tried to gauge the

reading and use of the program manual. Interestingly, students giving high grades in this statement tended to give high grades in the first two statements.

Statements 4 and 5 had to do with ease of installation in Excel 2003 and 2007, respectively. In the Spring of 2007, all students used Excel 2003 but, in the Fall, some students had already migrated to Excel 2007 and had installation problems. Statement 6 was intended to measure the effectiveness of the sample calculations provided in the package. Statement 7 was intended to assess the level of confidence for solving new problems and the results suggest that students are skeptical of their ability to do so.

In summary, for all statements, average points ranked between “neutral” and “agree.” Based on this

	A	B	C	D	E	F	G
13	Moles	n-C6(1)	n-C8(2)	n-C10(3)	Total		
14	Global	40	30	30	100.000		
15	Liquid	15.000	20.000	25.000	60.000		
16	Vapor	25.000	10.000	5.000	40.000		
17							
18	Mole fractions	n-C6(1)	n-C8(2)	n-C10(3)			
19	Global	0.400	0.300	0.300			
20	Liquid	0.250	0.333	0.417			
21	Vapor	0.625	0.250	0.125			
22							
23	In phi	n-C6(1)	n-C8(2)	n-C10(3)			
24	Liquid	0.744	-0.365	-1.438			
25	Vapor	-0.082	-0.149	-0.216			
26							
27	ln x(i) + ln phi(i) + ln P	n-C6(1)	n-C8(2)	n-C10(3)			
28	Liquid	0.861	0.040	-0.810			
29	Vapor	0.952	-0.031	-0.792			
30							
31	delta G/(RT)						
32	Liquid	-6.510					
33	Vapor	19.541					
34	Global	13.031					
35							
36							
37							
38							
39							
40							
41							
42							

Solver Parameters

Set Target Cell:

Equal To: Max Min Value of:

By Changing Cells:

Subject to the Constraints:

Figure 2 (b). Spreadsheet with initial guess before the minimization.

TABLE 2
Examples of Assignments and Course Projects Solved Using XSEOS

Assignment/Project	Thermodynamic Properties	Models
Calculation of octanol + water partition coefficients	Activity coefficients at infinite dilution	UNIFAC
Vapor-liquid equilibrium of amine + water mixtures for carbon dioxide absorption	Activity coefficients	UNIQUAC, NRTL
Properties of refrigerants that may replace CFCs (chlorofluorocarbons)	Fugacity coefficients, residual enthalpies	Cubic EOS
Compositional segregation of mixtures under gravitational and centrifugal fields	Fugacity coefficients	Cubic EOS
Vapor liquid equilibrium of chlorine + hydrogen chloride and nitrosyl chloride + ethyl acetate mixtures	Fugacity coefficients	PSRK EOS
Chemical equilibrium in multireaction system	Fugacity coefficients	PSRK EOS

TABLE 3
Opinion Survey of Undergraduate Students of Chemical Engineering Thermodynamics, Fall 2007, United Arab Emirates University
Point scale: "strongly disagree" (1 point), "disagree" (2 points), "neutral" (3 points), "agree" (4 points), or "strongly agree" (5 points)

Statement	Average Points
1. The XSEOS package helped me understand how to perform physical property and equilibrium calculations.	3.4
2. I understood the calculations I made using the XSEOS package.	3.7
3. I read or consulted the User's Manual provided with the XSEOS package.	3.4
4. Installation in Excel 2003 was easy and unproblematic (answer this question only if you used Excel 2003).	3.7
5. Installation in Excel 2007 was easy and unproblematic (answer this question only if you used Excel 2007).	3.1
6. The examples in the Excel spreadsheet are easy to follow.	3.5
7. I would know how to organize the solution of a new problem in XSEOS, similar but not identical to a problem of the sample spreadsheet.	3.2

feedback, the author has prepared movies (available on the Internet) that illustrate how to perform several types of calculations in XSEOS. This gives students the opportunity to review the calculation steps when needed. Furthermore, in future versions of the Chemical Engineering Thermodynamics course, XSEOS will be used not only for phase and chemical equilibrium calculations but also during the first half of the course. The residual and excess functions available in the package allow the solution of many problems related to the first and second laws, such as the design of heat exchangers, turbines, compressors, and expansion valves among many others. This will give students more time and opportunities to use and understand the thermodynamic models available in the program.

PROGRAM DISTRIBUTION

XSEOS is an Excel add-in with approximately 20,000 lines of VBA code at the time of its first public release. The Excel add-in, a sample spreadsheet, a manual with installa-

tion procedures, and instructional videos are available at: <<http://www.engg.uaeu.ac.ae/mcastier/downloads.htm>>.

After installation, all functions of the thermodynamic package become available. A spreadsheet that uses these functions in applications such as bubble points, flash calculations, chemical equilibrium, energy balances, and speed of sound is also provided. XSEOS has open source code and is distributed under the GNU General Public License, version 3. Therefore, it is an expandable platform and will hopefully contribute to the exchange of experiences in the teaching of thermodynamics. XSEOS runs in Excel, which is part of the Microsoft Office suite available from Microsoft resellers.

AREAS FOR COLLABORATION

Areas for collaboration can be broadly organized around three issues: expandability, usability, and portability.

Expandability

Given the large number of thermodynamic models available and their wide range of applications, the set of models currently available in XSEOS reflects an arbitrary choice based on the wide use of some models and on the personal experience of the author with some others.

Many extensions can be envisioned. The most direct is to include additional models, but there are classes of models more immediately necessary. Among these are models for electrolyte solutions, either excess Gibbs free energy (G^E) models or equations of state (EOS), and quantum mechanics-based models, such as COSMO or one of its variations, that may eventually replace conventional group contribution models for predictive phase equilibrium calculations. The creation of interfaces between XSEOS and external parameter databases for the models already available in XSEOS would be a useful extension to the package.

Usability

Examples that illustrate calculations such as the boiling point of pure substances, bubble points of mixtures, chemical equilibrium, speed of sound, and residual and excess properties are provided in the worksheets of the XSEOS spreadsheet files. User feedback may expand the set of examples available and contribute to improvements in interface and documentation.

Portability

The use of Excel as a development platform was primarily dictated by convenience because it is widely available as part of the Microsoft Office suite. The possibility of running XSEOS in freely available spreadsheets, such as Calc (part of Openoffice.org), requires porting the VBA code to other Basic dialects. Free online spreadsheet services, such as Google's spreadsheet, are currently unable to run XSEOS, but these services are evolving rapidly and may have the features required by the package in the future.

FINAL COMMENTS AND PERSPECTIVES

An Excel add-in, XSEOS, that implements several excess Gibbs free energy models and equations of state, has been developed primarily for undergraduate education, but may also be useful for graduate education and research. Several traditional and modern thermodynamic models are available in the package. Students of the United Arab Emirates University who used the package could quickly put together solutions to interesting course projects. XSEOS has open code and is freely available. This form of distribution will hopefully encourage other groups to contribute to the project. The package should be useful for instructors and students of chemical engineering thermodynamics courses.

ACKNOWLEDGMENTS

The author acknowledges Dr. Silvana Mattedi (Federal University of Bahia, Brazil) and the students of the Chemical Engineering Thermodynamics (Spring and Fall, 2007) and Fluid Phase Equilibria (Spring, 2007) courses at the United Arab Emirates University for testing the program and suggesting many improvements. The Research Affairs at the UAE University supported this work (Grant #05-35-07-11/06).

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LACTOSE INTOLERANCE:

Exploring Reaction Kinetics Governing Lactose Conversion of Dairy Products Within the Undergraduate Laboratory

JIMMY L. SMART

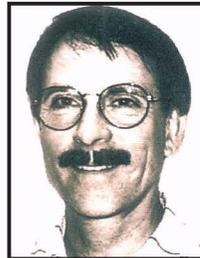
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Lactose intolerance is a condition suffered by an estimated 50 million Americans. Certain ethnic and racial populations are more widely affected than others. As many as 75 percent of all African-American, Jewish, Native American, and Mexican-American adults, and 90 percent of Asian-American adults, are lactose intolerant.^[1] Some populations in Africa are completely lactose intolerant, whereas some northern European populations are unaffected.^[2] Why so? Genetic evidence indicates that lactose intolerance arose over 5,000 years ago and spread among populations due to positive selection. Correlations have revealed lactose intolerance is prevalent in geographical areas of extreme climates and where there have been persistent occurrences of communicable diseases affecting cattle.^[3]

Lactose, or milk sugar, is a naturally occurring disaccharide found in dairy products and is normally converted to simpler sugars in the human digestion process. The lactase enzyme (β -Galactosidase) hydrolytically cleaves the lactose molecule to form monosaccharides, galactose, and glucose (see Figure 1). This enzyme is secreted by cells along the lining of the small intestine during normal digestion. In some people, this enzyme is absent or present in reduced concentrations. Interestingly,

most other mammals completely stop producing lactase at weaning, and are thereafter intolerant of milk. If, during the human digestion process, lactose is not converted to these simpler sugars, people may suffer symptoms of bloating, nausea, cramps, and diarrhea. Hence the term lactose intolerance. Fortunately, it is generally not life-threatening and can be controlled through proper diet. Lactase-deficient people can also ingest commercial supplements such as Lactaid and Dairy Relief with their meal to aid in digestion of dairy products.

This undergraduate laboratory experiment demonstrates the effect of catalytic action (Dairy Relief) upon skim milk and the subsequent conversion of lactose to glucose. An inexpensive



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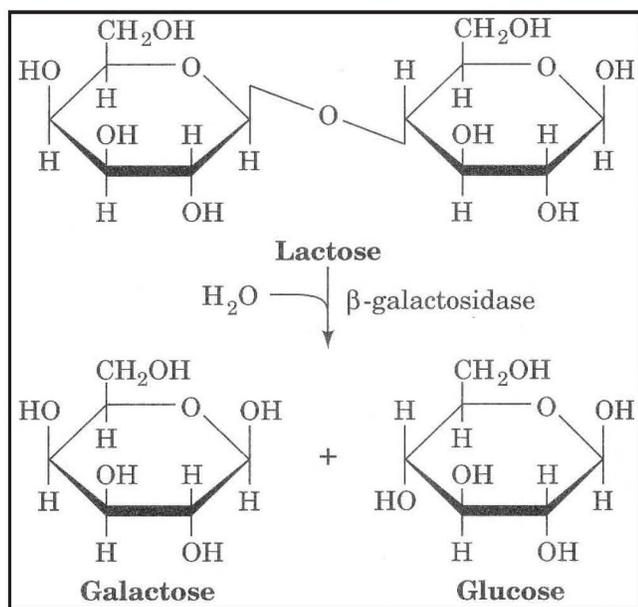


Figure 1. Conversion of lactose to monosaccharides.

blood glucose monitoring meter is used to record changes in glucose concentration over the course of the reaction. Two kinetic rate methods are offered to calculate reaction rates; these are the differential method and initial rate method. Effects of other reaction variables were studied, including pH, temperature, catalyst concentration, and catalyst accelerations/inhibition. A simple kinetic rate model is applied to demonstrate development of the Arrhenius expression. Finally, a more appropriate Michaelis-Menten model is applied to accurately model the complex enzyme kinetics.

CURRICULAR IMPACT

This experiment was primarily developed to be incorporated into a chemical engineering undergraduate unit operations laboratory (reactor design). Students could be required to develop reaction kinetics and use them to design/optimize a batch reactor to manufacture a lactose-free milk product based upon economic considerations. On the other hand, more fundamental portions, such as temperature and pH effects, could be assigned as part of an undergraduate semester project in a lecture class dealing with reactor design. Learning objectives might include determination of reaction rates, activation energy, optimum pH environment, catalyst deactivation temperature, Michaelis-Menten model parameters, and catalyst loading effects. The simplicity, portability, and quick variable response of this experiment also lend themselves well to K-12 audiences and other outreach activities. For these activities, it is recommended one omit the study of temperature effects (due to the inconvenience of transporting the control bath) and focus upon effects of pH and catalyst loading. The more advanced Michaelis-

Menten model might be appropriate for demonstration within a chemical engineering graduate lecture class. This experiment also has broad appeal to other disciplines, including biomedical engineering and food science curriculums.

EQUIPMENT, SUPPLIES, AND OPERATING PROCEDURE

Chemicals and Supplies

- Individual instrument test strips for glucose monitoring, \$0.60 ea.
- Skim milk, \$0.605/L
- Household distilled white vinegar, 5% acidity, \$2/L
- Household ammonia cleaning solution, dilute ammonium hydroxide, \$0.87/L
- pH 4.5 - 10.0 pH test strips (Fisher), \$0.09 ea
- Dairy Relief lactase tablet (9000 FCC units), \$0.06 ea

Procedure

1. Milk is a complex food containing over 100,000 different molecular species. Skim milk contains about 87% water, 3.7 - 5.1% lactose, and 0.75% minerals. It has a density of 1.03 and a viscosity of 2.1 cP @ 20 °C.^[4]
2. Fill the 1,000 ml glass-jacketed reactor with 490 ml of skim milk. Turn on the magnetic stirrer to a medium, reproducible setting (about 300 rpm). Set the temperature control bath to the desired temperature (between 12 and 38 °C) and wait for reactor temperature equilibration.
3. Initial studies can be conducted using the normal pH of skim milk, which is about 6.8. If runs will be made to examine effects of pH upon reaction rate, lower the pH of the milk with vinegar (about 10 ml for every pH unit) or raise the pH with ammonia cleaning solution (about 5 ml for every pH unit).
4. Calibrate the glucose meter according to the manufacturer's instructions. Once reactor conditions are at steady-state, measure the concentration of glucose in the skim milk (mg glucose/dL). Use a small bore pipette to deposit a single drop of sample mixture on a test strip. Each test requires about 60 - 75 seconds for total processing time.
5. Grind up two lactase enzyme tablets with a mortar and

TABLE 1
Equipment List

Equipment	Manufacturer	Cost
Blood glucose monitoring instrument	One Touch Basic (Johnson & Johnson, Lifescan Division)	\$60
Standard laboratory temperature-control bath	NESLab RTE-100	\$1,500
1000 ml glass-jacketed beaker	Ace Glass	\$120
Magnetic stirrer with stirrer bar	Fisher	\$200
—	TOTAL	\$1,880

pestle. Wet the powder with 10 ml distilled water to dissolve the powder. When ready to begin a reaction run, add the catalyst/water mixture to the skim milk.

6. If sampling across long periods of time (differential method of analysis), sample the reaction mixture twice and average the results. If using the method of initial rates, sample reaction mixture consecutively over a 10- to 15-minute time period as there is insufficient time to repeat samples.
7. To start the reaction at a higher glucose concentration, dissolve 1.0 g reagent glucose into 20 ml of distilled water and add it to 480 ml of skim milk. This will raise the reading of the glucose monitoring meter by about 100 mg/dL. Also, reagent-grade lactose can be added to skim milk to study the effect of disappearance of reactant, though starting concentrations of lactose in our milk were not precisely known (reported to be 3.7 - 5.1%).

EFFECTS OF pH AND CATALYST CONCENTRATION

The conversion rate of lactose in milk with the use of a lactase catalyst is a function of several variables, including lactose substrate concentration, lactase catalyst concentration, temperature, pH, mixing rate, and ion concentration. The effect of catalyst concentration upon reaction rate is shown in Figure 2. As expected, reaction rate is accelerated with increases in catalyst concentration.

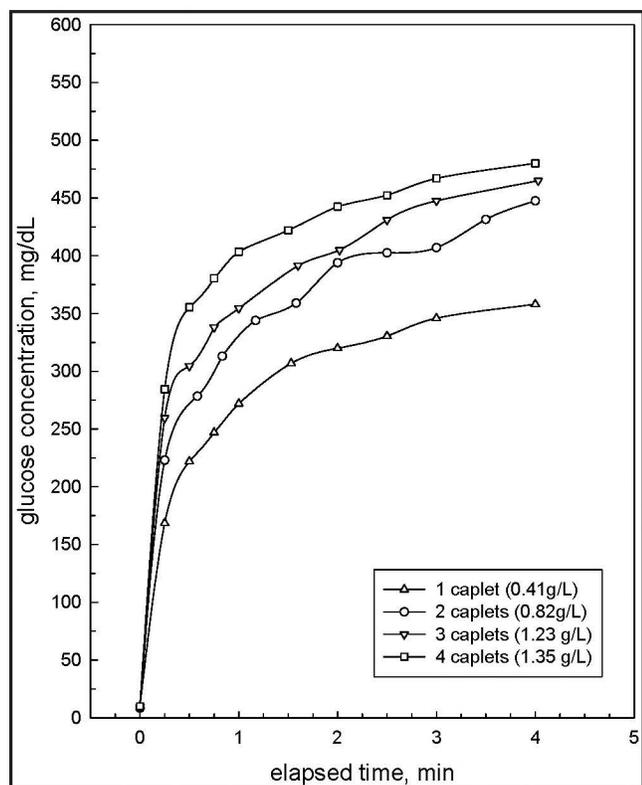


Figure 2. Effect of catalyst concentration upon reaction rate (22 °C, 300 rpm, 500 ml skim milk).

The activities of many enzymes vary with pH since the active sites of the enzyme contain significant acidic and basic groups. The influence of pH upon the reaction rate of lactose conversion to glucose is shown in Figure 3. A reduction in pH favors an increase in production rate until a pH of 4.5, where catalyst behavior becomes unstable. It has been reported that lactase in the Lactaid formulation is effective over the pH range of 3.5 - 8.5, with an optimum near 8.5.^[5] Classically, initial rate of this reaction would be expected to vary with a bell-shaped distribution, with lower rates occurring at low and high pH and a high median rate at some intermediate pH.^[6,7] Optimum pH for crystalline lactase is reported to be 6.6 - 7.7 at 20 °C.^[8] In this present investigation, however, reaction rates of lactase with milk appear to improve with decreasing pH—down to a pH of 5.0. Evidently, the Dairy Relief lactase formulation includes buffering agents to allow the lactase to operate in a more acidic environment, similar to that found within the stomach. At a pH of 4.5 (as measured by pH paper), the enzyme initially shows reduced efficiency. Finally after about 3/4 hour, it begins to behave erratically. Since the pH of stomach contents is commonly reported to be in the range of 2 - 4, this would seemingly be a problem for those people taking lactase to address lactose intolerance issues. The pH of the distal stomach contents, however, does not fall below a pH of 5 until some 60 - 75 minutes after the ingestion of a

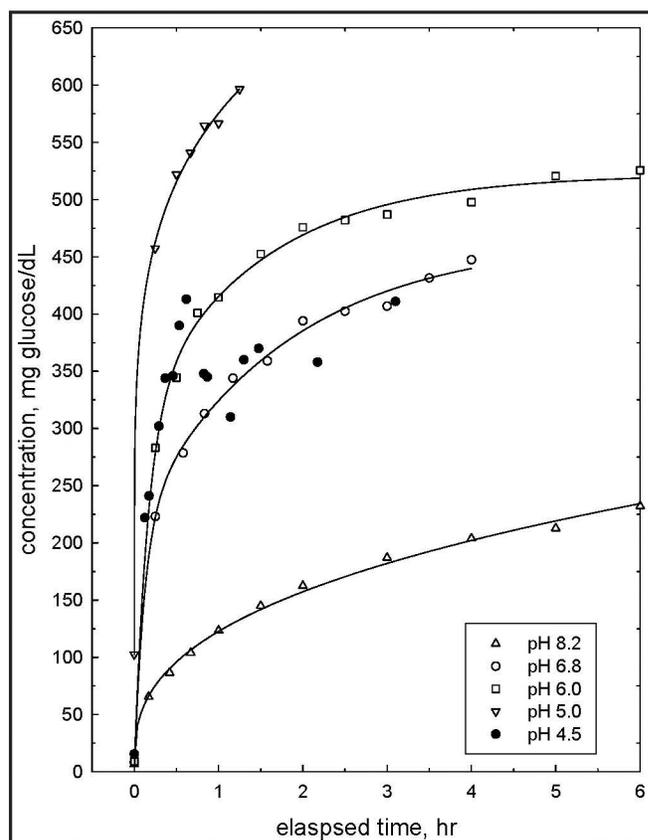


Figure 3. Influence of pH upon reaction rate (500 ml milk, 2 tabs catalyst, 22 °C, 300 rpm).

meal.^[9] According to the pH 5 curve of Figure 3, 75 minutes would provide time for a conservative conversion of about 1/3 of the lactose contained in a glass of milk. The stomach does not mix at a rate of 300 rpm, however, but it does operate at the higher temperature of 37 °C (310K).

TEMPERATURE EFFECTS (METHOD OF INITIAL RATES)

The constructed pH plot (Figure 3) was based upon laboratory data collected over several hours. From this data, a differential method of analysis, such as graphical or numerical differentiation, could be used (though not performed here) to determine the reaction rate law for each set of experimental conditions. These longer data-collection time periods, however, are usually not available or practical in a typical undergraduate laboratory setting. Therefore, as an alternative, the method of initial rates can be used to investigate reaction kinetics. For example, Figure 4 illustrates the effect of temperature upon reaction rate. As an assignment, students were asked to generate data similar to Figure 4 and determine the rate constants and order of reaction for conversion of lactose to glucose across various temperatures. Their first approach (not the correct one) was to treat the reaction as first order, in the sense, lactose → products. A mole balance on a liquid phase batch reactor for the appearance of glucose can be

written as:

$$\frac{dC_g}{dt} = r_g = kC_g^\alpha \quad (1)$$

where r_g is the reaction rate for the appearance of glucose, k is the specific rate constant, α is the order of reaction, and C_g is the concentration of glucose. Upon integrating Eq. (1), the final result is:

$$t = \left(\frac{1}{k} \right) \frac{C_g^{(1-\alpha)} - C_{g0}^{(1-\alpha)}}{(1-\alpha)} \quad (2)$$

Using Excel Solver, a nonlinear regression was performed on Eq. (2) to solve for the specific rate constant and the order of reaction. The order of reaction was approximately first order (varied between 0.7 - 0.9 across the temperature range of 12 °C - 37 °C). Using the values of the specific rate constants as determined from the nonlinear regression, an Arrhenius plot was constructed and the activation energy for the reaction was graphically determined to be 39.1 kJ/mole. Assuming conversion of lactose to glucose to be a single, irreversible reaction is unjustifiably simple. As it turns out, however (see below discussion of Michaelis-Menten kinetics), the lumping together of all reactions and use of the Arrhenius equation is a fairly good approximation to an otherwise complex reactive

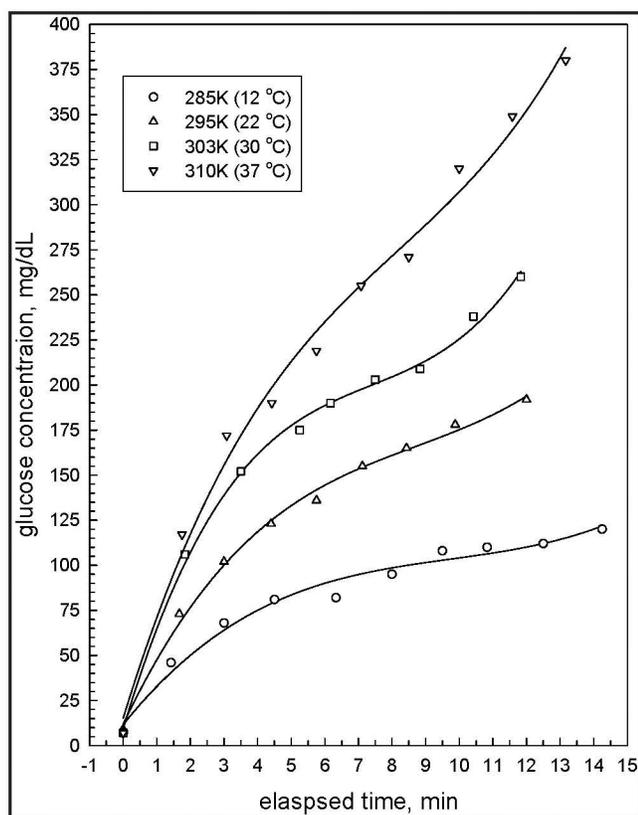


Figure 4. Use of initial rate method to assess effect of temperature (500 ml milk, 2 tabs catalyst, 300 rpm).

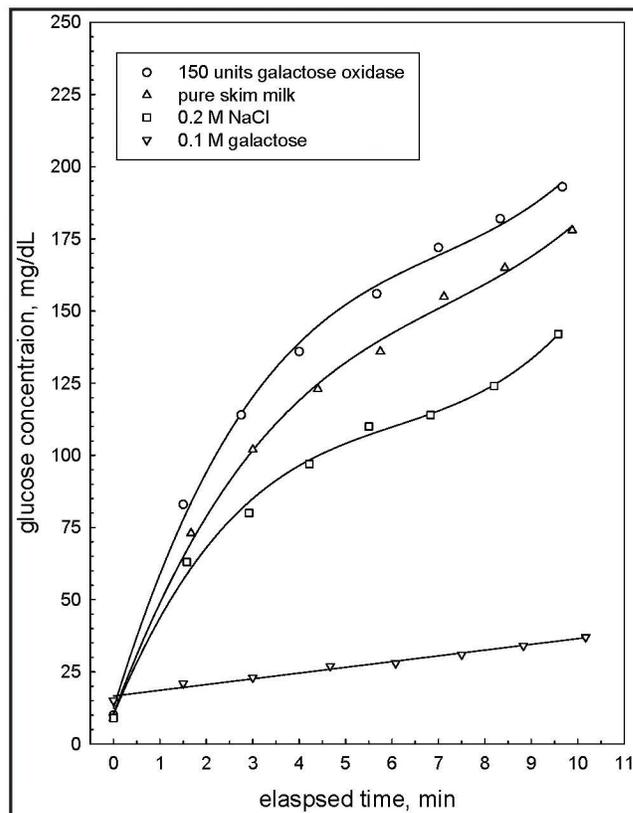


Figure 5. Use of initial rate method to assess impact of enzyme inhibition (22 °C, 500 ml skim milk, 300 rpm, 2 tabs catalyst).

Students were asked to generate data similar to Figure 4 and determine the rate constants and order of reaction for conversion of lactose to glucose across various temperatures. Their first approach (not the correct one) was to treat the reaction as first order, in the sense, lactose → products.

network (several reactions occurring with competing values of specific rate constants).

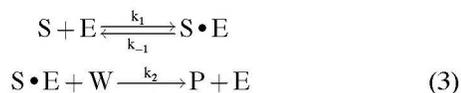
ENZYME INHIBITION

The surface of an enzyme contains many polar groups that are influenced by the surrounding ionic environment. It has been reported that sodium ions have a strong activating action on the lactase reaction.^[8,9] It is very likely a person might use significant quantities of table salt as they consume their hamburger along with a glass of milk. In this investigation, (see Figure 5, previous page) sodium chloride was not found to be an activating agent. At 0.2 M NaCl, the salt acted as a slight inhibiting agent. Using the initial rate method, the reaction rate with 0.2 M NaCl was 2.1×10^{-3} mol/L min. as compared to a rate of 2.4×10^{-3} mol/L min. with pure skim milk. At concentrations of 0.05 M NaCl and 0.05 M MgSO₄ (data not shown), there was no measurable change in the reaction rate. Other strong inhibitors of the lactase reaction are reported to be β-phenylthiogalactoside^[10] and heavy metallic ions,^[8] though these were not tested. Also, reported weak inhibitors of the reaction include some of the sugars, such as melibiose, galactose, glucose, and sucrose. According to Figure 5, galactose was shown to be a strong reaction inhibitor. When galactose was added to the reaction mixture (with no enzyme), the reversible reaction was slightly shifted to the right to produce higher concentrations of glucose—15 mg/dL vs. a routinely measured 9 mg/dL. This may indicate a reduction in normally available lactose in the initial reaction mixture of skim milk. Once the enzyme was added to the reaction mixture, the reaction proceeded at a much lower reaction rate (0.11×10^{-3} mol/L min vs. 2.4×10^{-3} mol/L min with pure skim milk).

Results with use of a reaction accelerating agent are also noted in Figure 5. Galactose oxidase converts any available galactose and drives the conversion of lactose into glucose at a slightly higher rate (2.9×10^{-3} mol/L min vs. 2.4×10^{-3} mol/L min with pure skim milk).

MICHAELIS-MENTEN KINETICS

The mechanism for conversion of lactose in milk to the products of galactose and glucose has been shown to follow the simple enzymatic reaction:



where S is the substrate, E is the enzyme, S · E is the substrate-enzyme complex, W is water, and P are the products. By writing simple rate laws and invoking the pseudo steady-state hypothesis, whereby the net rate of formation of the substrate-enzyme complex is assumed to be zero, the Michaelis-Menten equation can be expressed as:^[11, 12]

$$-r_s = \frac{k_{cat} C_{Et} C_S}{C_S + K_M} \quad (4)$$

where $-r_s$ is the net rate of disappearance of the substrate, $k_{cat} = k_2 C_W$, C_{Et} is the concentration of the total system enzyme concentration, C_S is the concentration of the substrate, and $K_M = (k_{cat} + k_{-1})/k_1$.

Interesting aside: Leonor Michaelis (1875-1945) was father of the permanent hair wave and Maud Menten (1879-1960) was one of the first women in Canada to earn a medical doctorate.^[13]

The quantity k_{cat} is called the turnover number of the enzyme because it represents the maximum number of substrate molecules converted to products per active site per unit time, or the number of times the enzyme “turns over” per unit time. K_M , with units of mol/L, is called the Michaelis constant and measures the attraction of the enzyme for its substrate. K_M is often called the affinity constant. If we let $V_{max} = k_{cat} C_{Et}$, then the Michaelis-Menten equation takes the final familiar form of:

$$-r_s = \frac{V_{max} C_S}{K_M + C_S} \quad (5)$$

where V_{max} is the maximum rate of reaction for a given total enzyme concentration, with units of mol/L sec.

According to the basic rate equation of enzyme kinetics as shown in Eq. (3), there are three rate constants. Each of these constants will obey the Arrhenius law and there will be three activation energies associated with each of the reactions. Sometimes, however, the Arrhenius law will follow a quite complicated enzyme process that involves several rate constants.^[14] The Arrhenius law is reported to be obeyed for application of lactase to a variety of substrates over the range of 0 - 37 °C, with activation energies in the range of 12 - 20 kcal/mol.^[15] Initially, the reaction rate increases with temperature and then begins to decrease at higher temperatures. At much higher temperatures, the complicated molecular structure of the enzyme begins to unfold and lose its catalytic properties. This is called thermal denaturation.^[16] The point of thermal denaturation for crystalline lactase is reported to be

40 °C.^[8] As a related example, Sizer^[17] evaluated the thermal denaturation of the catalase-hydrogen peroxide system. According to Figure 6, the activity of this catalyst follows an Arrhenius relationship up to about 53 °C and then follows an inactivation curve at higher temperatures.

Investigators have found the conversion of lactose to glucose to follow the Michaelis-Menten relationship. This is true when $k_2 \ll k_{-1}$ and there is thermodynamic equilibrium between the initial free reactants and the enzyme-substrate complex.

Eq. (5) could be inverted and a Lineweaver-Burk plot constructed to graphically evaluate the slope of the plot as K_M/V_{max} and the intercept as $1/V_{max}$.^[11, 18] This is not helpful, however, as the reaction rate for the disappearance of the reactant, $-r_g$, is not known.

A more useful method to evaluate the Michaelis parameters is to substitute Eq. (5) into Eq. (1) and integrate. Note, for every mole of reactant lactose disappearing, one mole of glucose is produced. For the appearance of glucose, the final result upon rearrangement is:

$$\frac{1}{t} \ln \frac{C_g}{C_{go}} = \frac{C_g - C_{go}}{K_M t} - \frac{V_{max}}{K_M} \quad (6)$$

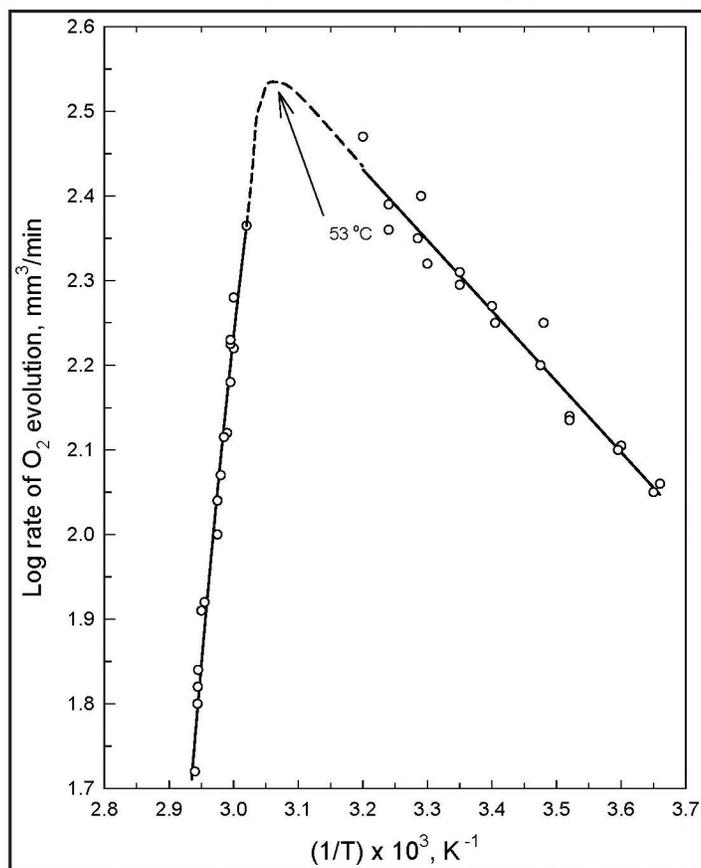


Figure 6. Thermal denaturation of the catalase-hydrogen peroxide system (from Sizer, I.W., "Temperature Activation and Inactivation of the Crystalline Catalase-Hydrogen Peroxide System").^[17]

Using data from Figure 3 and plotting terms from Eq. (6), the slope of the line is $1/K_M$ and the intercept is $-V_{max}/K_M$ (see Figure 7, next page). From this plot, the Michaelis-Menten constant, K_M , assumes different values at different pH levels (also will vary with temperature), but V_{max} is only a function of enzyme concentration. According to Table 2, the enzyme is increasingly attracted to the substrate at lower pH (higher K_M), but V_{max} is relatively steady across the pH range (enzyme concentration was not varied).

The Michaelis-Menten constant, K_M , for lactose substrate is reported to be 10^{-3} moles/L and for maximal velocity, V_{max} , to be 3×10^{-3} $\mu\text{m/ml/min/std conc of enzyme}^{[10]}$ (9.0×10^{-4} mol/L sec for conditions of this investigation). Another investigator reports K_m and V_{max} for crystalline lactase acting on lactose substrate to be 3.85×10^{-3} mol/L and 1.9×10^{-5} mol/L sec, respectively, at 20 °C.^[15]

GLUCOSE MONITORING ISSUES

In the interest of appealing to high school outreach programs and funding-challenged college undergraduate departments, an inexpensive means was sought to monitor the reaction kinetics associated with conversion of lactose to glucose in milk. The obvious choice to monitor the reaction would have been to adopt a standard glucose analyzer (Beckman, YSI, etc.) commonly found in research and hospital laboratories. This complete set-up, however, would cost over \$10,000. Other methods considered were based upon refractive index, freeze point depression, use of a lactose probe, chromatography,^[19] a colorimetric procedure,^[20] a differential pH technique,^[21] and various other wet chemistry methods.^[22, 23] Finally, an inexpensive blood glucose monitoring meter, commonly used by diabetics to measure glucose levels in blood, was selected as a candidate for monitoring lactose conversion in milk.

Glucose monitoring meters are portable medical devices available from several manufacturers for use by diabetics as a quick, easy, and inexpensive means to monitor blood glucose levels. The meters are not recommended by the manufacturer for testing glucose in solutions other than blood. Currently, two different technologies that govern the operation of these meters are: (1) electrochemistry (amperometry), and (2) reflectance photometry. With reflectance photometry technology, the test strip for the

pH	K_M , mol/L	V_{max} , mol/L sec
8.2	1.35×10^{-3}	2.26×10^{-4}
6.8	3.46×10^{-3}	2.63×10^{-4}
6.2	4.39×10^{-3}	2.63×10^{-4}
5.0	12.5×10^{-3}	2.88×10^{-4}

meter contains a combination of glucose oxidase catalyst and a dye mixture. As a drop of blood is placed on the test strip, glucose and oxygen react in the presence of the catalyst to produce gluconic acid and hydrogen peroxide. Hydrogen peroxide, in turn, with mediation by peroxidase, oxidizes a dye impregnated in the test strip to produce a blue color. The meter senses the intensity of this blue color, which is proportional to the glucose concentration of the sample. The method is based upon Trinder's glucose oxidase method and the two coupled reactions are called Trinder's reaction.^[24] Additional references^[25, 26] can be consulted for more information about bioelectrochemical analytical methods.

The Bayer *Ascensia Contour* glucose meter (electrochemistry technology) was tested and found to be unsuitable for purposes of these experiments. Next, the Lifescan *One-Touch Basic* meter (reflectance photometry) was tested. Range of the meter is 0 - 600 mg glucose/dL (0 - 33.3 mmol/L). Normal range of glucose concentrations in blood for fasting humans is 70 - 110 mg/dL. Operation of the meter involves inserting a coded test strip within the meter and placing a small sample drop on the strip. After about 60 seconds, the meter provides a digital readout of glucose concentration. This meter was responsive when measuring glucose levels in skim milk across varied temperature and pH ranges, and was selected for use in this experimental investigation.

METER ACCURACY AND PRECISION

The blood glucose monitoring meter is designed to measure glucose concentrations in blood, but not necessarily glucose concentrations in milk. A typical meter measurement of raw skim milk is 9 - 10 mg glucose/dL. The actual range of glucose in nonfat milk as measured by other sophisticated laboratory instruments has been reported to be 2.2 - 27.0 mg/dL.^[27, 28] Even though our meter measurement was within this range, it is not known how accurate the meter is at the lower end of its range (0 - 600 mg/dL). In an effort to check the bias and accuracy of the glucose monitoring meter over its entire range, reagent-grade glucose was added to skim milk to make up various standard concentrations. According to Figure 8, there is significant deviation between actual concentration of glucose in skim milk and concentrations measured by the meter. Also, the meter was unable to reproduce concentrations of glucose in buffered deionized water solutions. When glucose was added to bovine blood, meter readings were accurate and linear. As noted above, the glucose monitoring meter measures glucose concentrations in blood based upon the result of two reactions with reagents contained on a standardized test strip (Trinder's reactions). Blood is a very complex mixture of numerous compounds, including various acid-base buffers to control its normal pH within the range of 7.3 - 7.4. It is well documented that Trinder's reactions are subject to bias by numerous interfering reagents, such as uric acid, cholesterol, hemoglobin, ascorbic acid, and maltose.^[29, 30]

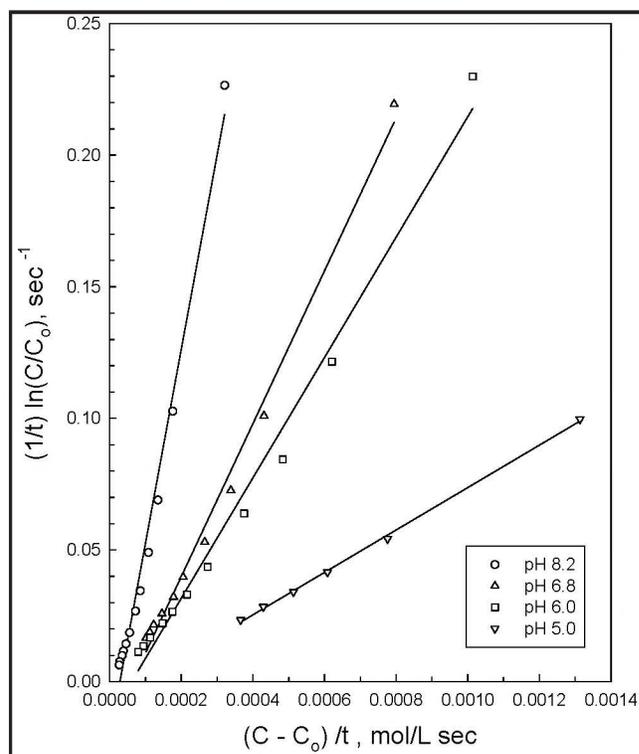


Figure 7. Determination of Michaelis-Menten parameters for various pH (500 ml milk, 22 °C, 2 tabs catalyst, 300 rpm).

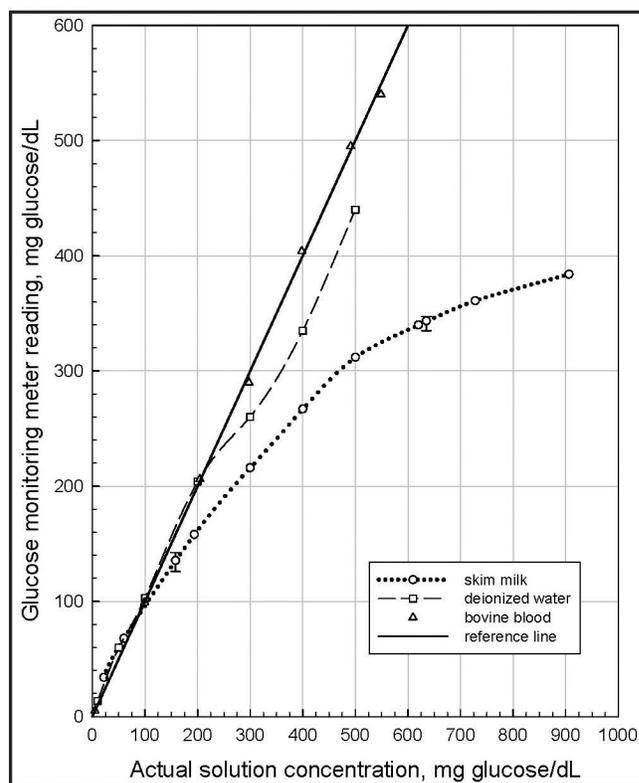


Figure 8. Calibration of glucose monitoring meter (Lifescan One-Touch Basic Meter).

Skim milk is a very complex mixture, containing many compounds that may interfere with use of Trinder's reactions to analytically quantify concentrations of glucose. It is believed matrix interferences between glucose and skim milk solutions contributed to nonlinearities in experimental meter measurements (see Figure 8). Unfortunately, these were unable to be resolved. Obvious interferences were investigated, including effects of pH (buffered solutions), solubility, viscosity, red color, and various ion concentrations. Glucose concentrations in milk could be linearized with the transform $y = -1.553e + 4 + 2022 \text{ Ln}(x + 2184)$, where x is the actual glucose concentration and y is the meter reading. This approach would be acceptable if all other variables were to remain relatively steady, such as temperature and pH (which is the case with use of the meter to monitor glucose levels in human blood). The purpose of the use of this meter, however, is to evaluate the effects of changes in operating variables upon reaction rates in milk mixtures. Therefore, for use as a student exercise, these matrix interferences were ignored. Unfortunately, during the laboratory experiments, meter reading data does not correspond to true concentrations of glucose in milk mixtures.

The precision of the meter was found to be satisfactory for student testing. Error bars based upon results of 10 samples at various concentrations are shown in Figure 8 to demonstrate reproducibility. At the lower range, sample mean was 134.1 mg/dL with a sample standard deviation of 8.2. At the upper range, sample mean was 341.1 mg/dL with a sample standard deviation of 6.2. Typical precision can be roughly estimated to be about $\pm 10\%$. For laboratory runs occurring across long periods of time, students were required to test each sample two times and average the results. When using the initial rate method (occurring across about 10 minutes), there is insufficient time to repeat samples. Each test requires about 60 to 75 seconds to perform.

CONCLUSIONS

An inexpensive glucose monitoring meter has been shown as an effective tool for measuring relative rates of reaction for the conversion of lactose to glucose in skim milk. A number of variable effects can be studied using the meter, but absolute numbers associated with variable effects cannot be relied upon because the meter has proven not to be linear across ranges of glucose in skim milk. Trends can be studied, however, and the meter serves as a valuable teaching aid for use by undergraduates in the chemical engineering laboratory.

NOMENCLATURE

α	reaction order, dimensionless
C_{Et}	total system enzyme concentration, mol/volume
C_g	glucose concentration, mg/dL
C_{go}	initial glucose concentration, mg/dL
C_w	water concentration, mol/volume

t	time, sec
k	specific rate constant, time^{-1} , if $\alpha = 1$
k_{cat}	turnover number = $k_2 C_w$
r_g	reaction rate for the appearance of glucose, mol/L min
$-r_s$	reaction rate for the disappearance of the substrate, mol/L min
V_{max}	maximum rate of reaction for a given total enzyme concentration, mol/L sec
K_M	Michaelis constant (affinity constant), mol/L

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GROUP PROJECTS IN CHEMICAL ENGINEERING USING A WIKI

JEFFREY J. HEYS

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Group projects are frequently assigned in chemical engineering courses to achieve a number of different objectives. First, we want to improve students' understanding of group dynamics so that they can work more effectively in teams.^[1] Second, a group of students can frequently achieve a greater depth of understanding on a given topic than a single student working alone. Third, grading a single group project normally requires less time than grading four (or more) individual projects. There are many other advantages to group projects for specific courses, but these three advantages are nearly universal and apply to any course. There are also a number of disadvantages or perceived disadvantages often cited by either the students or the instructor. First, it is difficult to determine the workload distribution among the students in the group—making it a challenge to give individual grades.^[2] Second, some groups are unable to schedule enough meetings when every group member can be present. Third, the collecting of the individual pieces of a project into a single coherent document or presentation can be difficult and frustrating for some groups.

Despite the disadvantages, group projects are an important and integral part of the educational experience within the

chemical engineering curriculum. Team projects in an industrial setting, however, are changing because geographically dispersed teams are becoming more common.^[3,4] Often, the team members can be located in two or more different cities or countries. Of course the team members will meet at important times during the project, but much of the day-to-day work will be completed by communicating over the phone or Internet (*e.g.*, e-mail, instant messenger). At Arizona State University (ASU), we recently began using a “wiki” for group projects in the Introduction to Chemical Engineering: Mass and Energy Balances course to overcome some of the difficulties associated with traditional (paper) group projects and to



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give the students experience in working on a group project where the group members are not necessarily located in the same physical location.

The term “wiki” is a Hawaiian word meaning “quick,” but the term is used here to refer to a specific type of Web site. The definition in this case, taken from Wikipedia (<www.wikipedia.org>), is that a wiki is a Web site that allows visitors to add, remove, and edit content. It is frequently used as a collaborative technology for organizing information on Web sites. One of the most commonly cited examples is Wikipedia, an online encyclopedia where entries can (normally) be edited by anyone using a modern browser such as Internet Explorer (from Microsoft), Firefox (from the Mozilla Foundation), or Opera (from Opera Software). Wikis are used for collaborative or group projects for two main reasons: (1) they are easily modified (you do not need to learn HTML programming) using any modern browser instead of requiring every group member to use the same software (how many group projects have been interrupted by incompatible software versions or operating systems?), and (2) the changes made are immediately available to other group members. A group project completed using a wiki results in a Web page, and, since it is a wiki, that Web page can be edited using the same software that is used to view it.

Depending on the type of wiki used (see the next paragraph), a wiki can overcome many of the difficulties given above that are normally associated with group projects. First, the computer server that hosts the wiki can store every change to the individual project Web page, and students can be required to log in to make any changes. This means that when grading the wiki, the exact contributions made by each and every student are available to the instructor. This includes every change made and when the change was made. Second, editing the project Web page only requires that a student have Internet access, so the group does not need to meet face-to-face as frequently to work on the project. Their discussions and editing can happen online instead. Third, instead of having group members produce individual pieces that are glued together the night before the project is due, the group project can ideally be produced over time with each member editing the contributions of others to produce a more coherent final document. A few other advantages include

- *The instructor always has access to the current state of the group project while it is being completed.*
- *Group projects from the past are easily available to later classes for their reference.*
- *Students can be required to edit and/or view other group projects.*

SETTING UP A WIKI

There are basically three options when setting up a wiki for group projects.

- (1) *Many colleges and universities have servers and*

software in place for hosting a wiki. For example, at ASU the Web page <wiki.asu.edu> allows you to create a wiki for group projects with a simple click of a button on the Web page. You only need to give the wiki a name (e.g., CHE 211 Projects) and the students can then create and edit their own group project Web pages on that wiki.

- (2) *There are a number of companies that host free wiki Web sites (e.g., <www.wikia.com>).*
- (3) *Install MediaWiki (<www.mediawiki.org>), which is the software behind Wikipedia, on your own computer or Web server. We used a basic desktop computer (Dell computer with an Intel Xeon processor) running Linux (Red Hat Enterprise Linux 4), and the installation of MediaWiki took about one hour. In hindsight, we recommend running the Ubuntu or Fedora flavors of Linux because the one small difficulty we encountered was specific to RHEL.*

Options 1 and 2 are clearly simpler and avoid the need for a continuously running desktop computer with Linux, but there are also some important advantages to option (3). First, running your own copy of MediaWiki provides outstanding flexibility in terms of what is possible to include on the group project page. For example, we used the “math” add-on extensively because it provides a straightforward method for displaying complex mathematical equations on the project Web page using a syntax that is similar to LaTeX (*i.e.*, this is equivalent to the equation editor in Microsoft Word). Similar add-ons include displaying chemical reactions and including animations or movies. A second advantage to option (3) is that it provides a fine level of control over access to the wiki’s Web pages. We chose to require a log-in for editing of the Web pages, but no log-in was required to view the pages. This means that students can modify the project of any group they want, but those changes are logged under their name and can be “undone” at any time. While the advantages to option (3) listed so far do not require any programming knowledge whatsoever to implement, the final advantage to option (3) is that MediaWiki is largely a collection of PHP files that can be edited to achieve any desired behavior. Just purchase a PHP book at the local bookstore and any missing feature or any desired change can be made by the instructor.

The upper part of the “Main Page” for the wiki used in the Mass and Energy Balances course is shown in Figure 1 (see Reference 5). At the top of the page is an introduction explaining the goals of the project and a list of important dates for the project during the semester. Also on the Main Page, but not shown in Figure 1, are a list of links to the various group project pages and some instructions on using the wiki. Near the upper-right corner in Figure 1 is a link to “Log in/create account.” The wiki was set up so that users were required to log in to edit any of the Web pages, but account creation was disabled. Accounts were created for all registered students in the class using a unique ID. By clicking on the “Log in” link,

students could enter their unique ID and have a randomly generated password e-mailed to their registered e-mail address. Also note the “edit” links near the top of the page and along the right edge. Clicking on these links allows the entire page or subsections to be easily edited.

Clicking on an edit link brings up the editing window that is similar to a normal word processing window. We chose to require a log-in before the editing of any pages, and the upper right corner of the page indicates which user is logged in. The editing process is similar to using any word processor, and the

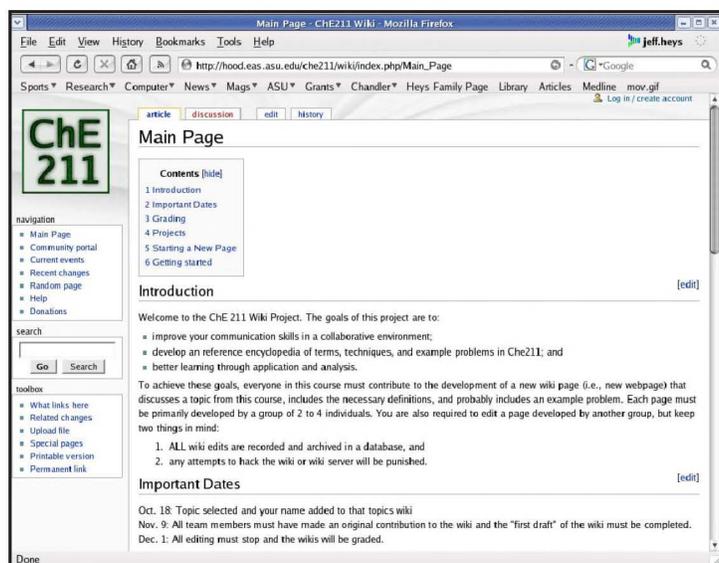


Figure 1. Upper part of the Main page for the ChE 211—Mass and Energy Balances wiki.

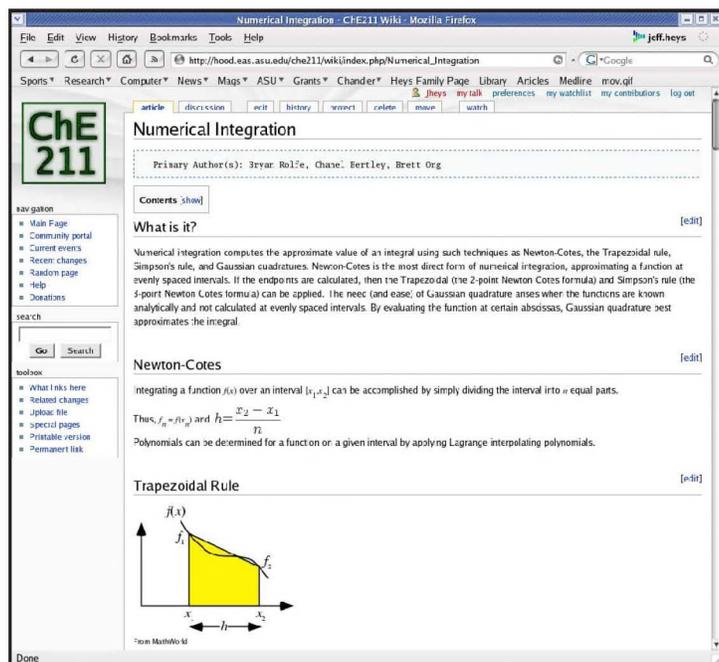


Figure 2. An example of a group project report on Numerical Integration. The figure shows less than 10% of the final project, but it illustrates a typical result.

buttons above the editing window allow for the insertion of objects such as mathematical equations, lines, bold face text, and links to other Web pages. The “discussion” tab, shown in Figure 1, is a similar window where group members can discuss their plans with each other and the instructor can leave comments for the group.

Clicking on the “history” tab in any window brings up a page showing a detailed list of the changes that have been made to the Web page. For example, the history tab for the main page shows that the page has mostly been modified only by the instructor, “Jheys.” It also shows, however, a student changed the name of a project (the only part of the project appearing on the main page) and another student corrected the instructor’s spelling and grammar (oops!).

GROUP PROJECTS USING A WIKI

The group projects from our first semester of using wikis (Fall 2006), can be found at <<http://hood.eas.asu.edu/che211/wiki>>. We chose to give the students the option of using one of the suggested project topics or proposing their own topic, and >90% of the groups chose one of the suggested topics. The topics loosely fall into four different categories.

- (1) *Advanced topics that are not covered in lecture, but are presented in the textbook (e.g., commercial process simulation packages).*
- (2) *Topics covered in class that students frequently struggle to understand, and the goal is to provide future classes with an additional reference on this topic (e.g., recycle streams).*
- (3) *Topics covered in other courses that may be useful to the students in Mass and Energy Balances (e.g., numerical integration).*
- (4) *Popular topics related to the course material (e.g., future energy sources).*

For category (1) projects, the students typically read the material in the textbook and then obtained some of the books and articles referenced by the textbook.^[6] For commercial process simulation packages, company Web sites also provided important information. Category (2) projects involved the students presenting course material in a new perspective that they found more intuitive, and they often included additional example problems. Some excellent projects resulted from topics in categories (3) and (4), but the students often recycled material from other classes, limiting the amount of new learning. This is not a limitation of using a wiki, but it is a common difficulty in all types of group projects. Figure 2 shows the very beginning of the numerical integration group project as an example. The entire numerical integration project as well as the other 19 projects competed in Fall 2006 are available on the Web site given above.

Based on the results of our first semester of using a wiki for the group project and on feedback from the students, we offer a couple suggestions for using this format. First, because students are often unfamiliar with wikis, it is important to set early milestones or due dates for parts of the project. For example, we required students to visit the Web page and get their password during the first month of the semester, before they were prepared to choose a topic. We also required each group to create a nearly blank page containing only their names early on so they could get a little experience editing a wiki. To facilitate this project, a sample page was created in class so that students could see the process for themselves—which greatly reduced the level of intimidation. A second suggestion is to provide students with multiple links to documentation for the use of wikis in general and MediaWiki in particular. During the Fall of 2007, a group of students at Arizona State University created a wiki entitled “How to Create a Wiki-Project,” and this wiki, as well as many other examples, can be found on the ChE 211 site given above.

Two problems were noted when the group projects were moved to a wiki format. First, the level of plagiarism appeared to increase because students found it incredibly easy to cut and paste from existing Web pages. This problem was identified early in the semester, and in-class warnings appeared to reduce the level of abuse. Numerous tools are available (*e.g.*, Ephorus) that can be used to detect plagiarism with little effort. Second, some of the students had extensive experience in using HTML (thanks to <myspace.com>, etc....) and this knowledge allowed them to introduce specialized features to the Web pages beyond what is possible using the normal “editing” features. This is not a problem, except those individuals would dominate the actual editing of the group’s Web page—reducing the instructor’s ability to determine individual contributions. This difficulty was avoided by requiring students entering material for other students to note that they were doing so in the “discussion” area of each page.

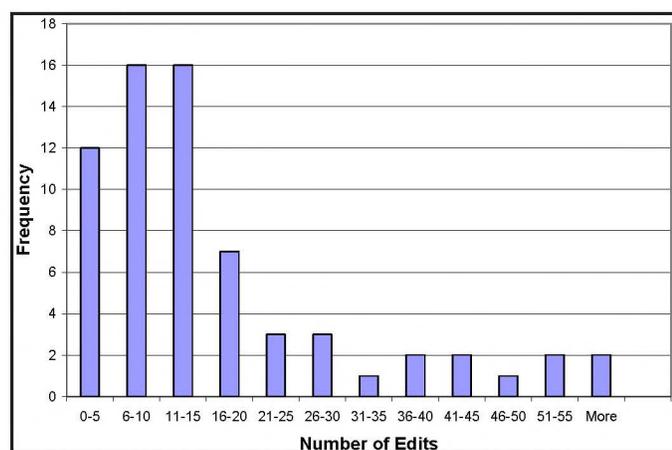


Figure 3. The number of students (frequency) making different numbers of edits. The average number of edits made was 18.

We have found the wiki format to be useful for projects in a Mass and Energy Balances course, but this does not mean that this is a useful format for projects in other courses. For example, the rigorous formatting requirements of senior laboratory reports, such as flowsheets, equipment diagrams, and numerous chemical and mathematical formulas, would make the use of a wiki difficult. Also, if a project report is more than 10 pages, the wiki format may not be appropriate because it requires that the person preparing the report be actively connected to the Internet during the entire time they are writing. In summary, we tend to prefer using wikis in larger, undergraduate courses where they can be an interesting and useful way for groups of students to prepare a report that is equivalent to 5-10 pages.

LEVEL OF PARTICIPATION

One of the advantages to using a wiki is that each contribution by a student is recorded in a database. The individual changes may be as small as correcting a spelling mistake or adding a period, and the changes may be as large as adding numerous paragraphs to the group report. On average, an individual change was equivalent to adding one or two sentences. In the Fall of 2006, there were 67 students enrolled in the ChE 211 course at ASU, and they completed 20 group projects. On average, projects received about 61 edits each, and the average student made about 18 edits or individual changes to his/her group project Web page. The standard deviation for the average edits per student, however, was 20, indicating that the students varied greatly in the number of edits made. In fact, a few students only made two or three edits, but a few other students made more than 100 edits. Figure 3 is a histogram showing the number of students making edits over different ranges.

This data should not be interpreted as an exact measure of the distribution of work performed by the various group members because an “edit” could be a large or small change.

(There have been a number of studies that have tried to examine work distribution among members of a groups, and studies on improving group performance.^[7,8]) There is some correlation, however, between the number of edits and the amount of work done, and we were surprised at the large distribution. Typically, a group of four would have one or two individuals making 20 or 30 edits and one or two individuals making less than 10 edits. This observation encourages more study on the distribution of work in group projects and ways to measure that distribution. It also motivates us to more closely monitor the work that every individual in the class is doing on the group project throughout the semester so that students can be alerted when they are not doing their share of the work. We also plan to increase the percentage of the final grade that is tied to the individual’s contributions to the group project from about 25% to 40-50%. Historically, we have used the

smaller percentage because individual contribution was difficult to measure, but now that the data is available, we plan to increase the percentage.

CONCLUSIONS

Using a wiki as a medium for group projects has advantages and disadvantages over the traditional oral and paper mediums. The advantages include

- *The ability to continually monitor and measure the work being done by each group and individual student.*
- *Straightforward methods for archiving the projects and making them available to future classes.*
- *Providing the students with experience in working as part of a virtual or Web-based team.*

Disadvantages include:

- *The need to invest time setting up the wiki (especially if the instructor elects to install MediaWiki).*
- *Possibly an increased potential for plagiarism.*
- *A few students are intimidated by the software.*

Based on our experience, the advantages were greater than the disadvantages, and we plan to continue using the wiki. The only significant change we plan to make is to more closely monitor the contributions of individual students throughout

the semester so that we can alert students when they are not contributing sufficiently to the overall group effort.

ACKNOWLEDGMENT

Thank you to Luke Olson for showing me the potential of wikis and helping with the installation of MediaWiki.

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Random Thoughts . . .

ON-THE-JOB TRAINING

RICHARD M. FELDER

North Carolina State University

Sheila: Good morning, Reggie—great to have you with us. I'm Sheila Conner, head of Process Engineering.

Reggie: Nice to meet you.

S: I was really hoping you'd join our group—I saw your file and you don't see 3.9 GPAs every day, especially coming from *your* university.

R: Thanks. I'm looking forward to using what I learned there.

S: Well, you'll get plenty of chances to do that here. As they probably explained to you, we get involved in almost everything that happens in the plant—designing new processes and products, retrofitting, troubleshooting, you name it. How does that sound?

R: Um, good, I think. I just took plant design last spring, so I'm probably more up on that than on that other stuff.

S: I see—well, anything you're not sure of, just ask anybody here. As a matter of fact, there's something we could use your help on over in hydrides...the efficiency of one of their packed absorption towers has been falling for the last week and they can't figure out what's going on. Go over to Building 293 and ask for Ben Whitman—he'll fill you in, and then see if you can figure out anything.

R: OK.

* * *

R: Excuse me, are you Ben?

Ben: Yeah—what can I do for you?

R: I'm Reginald Bunthorne. Sheila from Process Engineering sent me . . . something about an absorption tower?

B: Oh, yeah. Come into the office, kid, and I'll show you what we got. OK, here's a GC trace on the off-gas line from about two weeks ago—this peak is CMPH, and it's down around 2.5 where it's supposed to be. It started to creep up on us last week, and here's yesterday's trace—the peak is up to 7, which means we're absorbing a lot less CMPH than we're supposed to. Unless we can fix this, we're going to have to take the process off line and break down the tower to see what's going on, and a lot of people across the street will be very unhappy if we do that. Got any ideas for us, kid?

R: Um . . . I think we once used the diffusion equation and Henry's law—or maybe it was Raoult's law—to analyze a continuous packed absorption tower. I could try doing that.

B: Say what?

R: Of course I can only do it if the column is isothermal. It is, isn't it? If it isn't, I think I'd also have to write a differential energy balance equation, and that's farther than we ever went in that course. I can give it a shot, though—you do have Matlab here, don't you?

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- B:** (Looks suspiciously at Reggie and says nothing)
- R:** Let's see—I think I'll also need to know the diffusivity of—what was that, CPMH?—and the Henry's law solubility. Do you happen to know what they are?
- B:** Uh, tell you what, kid—we've got a few ideas we want to try first, so let's hold off on that stuff for now. I'll check in with Sheila and tell her we'll call again if we need you.
- R:** OK, and don't forget the diffusion coefficient.
- B:** Trust me, I won't forget it.
- * * *
- S:** So I hear you had a session with Ben yesterday. How do you think it went.
- R:** Fine—I'm surprised I haven't heard back from him.
- S:** Hmm. Well, we've got another job over here I'd like you to take a look at. It's a distillation column we need to design for a pretty tough separation—think you can handle it?
- R:** No problem—I did a couple of them in mass transfer . . . I think we also did one in plant design, but one of my teammates handled that part.
- S:** Um . . . yes . . . fine. Here's the folder with the design specs . . . let me know if you need anything else.
- * * *
- S:** Yes, Reggie—got something for me on that column?
- R:** I get that you need eight theoretical stages.
- S:** What?
- R:** Eight theoretical stages—here's the McCabe-Thiele diagram.
- S:** Uh . . . how'd you do McCabe-Thiele with nine components in the feed?
- R:** I just used two components I could look up data for in Perry's Handbook and used Raoult's law to come up with an equilibrium curve and then did McCabe-Thiele.
- S:** For a multicomponent system with nine highly polar compounds?
- R:** Well, they never really talked about systems like this in mass transfer—the professor said that equilibrium separations were trivial and we spent most of the course solving differential equations for rate-based processes . . . and I tried finding vapor pressure data for those other feed compounds but they weren't in Perry, so I just made some simplifying assumptions.
- S:** Ah, some simplifying assumptions. OK, why don't you let me have the file back and we'll take it from here. Let's see—you know any process control?
- R:** Yeah, we learned how to calculate transfer functions for linear systems.
- S:** But real-time control for nonlinear systems?
- R:** They said they didn't have time to cover that.
- S:** What about statistical quality control?
- R:** Um, no—we had a couple of weeks of statistics in the unit ops lab . . . means and standard deviations and t-tests and that stuff, but they never really explained what you do with it.
- S:** Ever size a pump?
- R:** No—pumps were in the fluids syllabus, but the prof. took so long on the Navier Stokes equation that we never got to them.
- S:** Know anything about separation process synthesis?
- R:** Um . . . not really.
- S:** Heat exchanger networks?
- R:** No—we only did single exchangers.
- S:** Using overall heat transfer coefficients and log-mean temperature differences, right?
- R:** Uh, yes. We were supposed to look at solving more complex problems using a simulator, but the prof. said we needed to learn the fundamentals before getting into black box simulations and I guess we never got past the fundamentals.
- S:** Equipment cost estimation?
- R:** My design teammate did that part.
- S:** I see . . . OK, to tell you the truth, Reggie, I'm not sure there's a good fit between your skills and the kind of things we do around here. I'm going to talk to Human Resources about finding you a more suitable position.
- R:** All right . . . but in the meantime, what should I do while I'm here?
- S:** Know how to use a coffee maker?
- R:** Um . . . □

All of the *Random Thoughts* columns are now available on the World Wide Web at
http://www.ncsu.edu/effective_teaching and at <http://che.ufl.edu/~cee/>

PID CONTROLLER SETTINGS

Based on a Transient Response Experiment

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Process control and automatic control systems play an important role in the design and operation of modern industrial plants, resulting in economical and safe plant operation. Among other topics in the field, controller tuning is particularly important because controller settings severely affect the performance of the closed-loop system. In addition, accurate settings from experimental dynamic data are extremely useful when processes are too complex to be modeled from fundamental principles.^[1] Chemical engineering instruction should emphasize these facts. The unit we propose to study has a nonadiabatic plate-heat exchanger, complex internal geometry, and nonlinear dynamics coupled and reciprocally interacting with a heater tank without agitation. In this unit, a monitoring-control system using Labview^[2] has been implemented. Multivariable control examples were proposed, but the emphasis in this work was toward showing the new, improved computer interface and the pedagogical potential of the experimental unit. The PCT23 unit has also been used as the main tool for developing advanced control tracking in the controller.^[3] In that work, a solution strategy was developed based on a set of partial differential equations, reflecting the complexity of the system model. In our case, an experimental reaction curve combined with tested sintoniza-

tion techniques is considered as a viable method for tuning up a PID controller. Similar laboratory pilot-scale experiments have appeared elsewhere in the literature.^[4, 5] These include computer-controlled units with a standard shell and pipe heat exchanger and a steady external heat source.^[4] The controlled temperature is the exit hot stream, and the manipulated vari-

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able is the cold stream flow rate with additional disturbance introduced in the hot stream flow. The system identification is carried out in the frequency domain by building an experimental Bode plot diagram. This method has two drawbacks. First, it's complex in that poor students lack understanding due to the frequency domain analysis requirements. Furthermore, frequency response analysis is not practical for systems with time constants—in the order of hours as in this laboratory unit—because it needs five to six experimental runs using a sinusoidal signal to obtain a complete Bode diagram. In this experiment, the manipulated variable is the power to a heater tank source and the disturbances are introduced in the cold flow stream. The open-loop identification is performed using the classic reaction curve with only a single experimental run, with a power step perturbation inside the heater tank. The resulting reaction curve is fitted directly in the time domain using readily available worksheet program tools.^[6] Other experiments^[5] focus on a theoretical homework profile, which is more appropriate for advanced courses without experimental requirements. Undergraduate students in the Department of Chemistry at the University of Aveiro receive lectures on fundamentals and applications of process dynamics, simulation, modeling, and control as part of four courses as follows: Instrumentation and Process Control, Laboratory EQ4, Chemical Process Modeling and Simulation, and Advanced Process Control. This paper focuses on the controller tuning experiment of Laboratory EQ4.

This is a weekly, six-hour laboratory where students are divided into groups of three. Each experiment lasts two weeks. In the first week, students carry out the lab exercise. In the second week, they do the numerical calculations and simulations that require computational support available in the PC computer laboratory near the wet laboratory. Assessment is based on an individual oral quiz and a report prepared by each group. At the end of semester, a selected group has the opportunity to present a longer report and an oral presentation. In this final report, the students can collect the experimental results from the other parties and prepare a review of the accumulated results.

In our opinion, there are two ways to train students to tune a PID controller: i) through the use of computer simulators with powerful numerical libraries embedded in commercial software such as Matlab/Simulink, Mathcad, and Hysys or other specifically tailored applications;^[7-9] or ii) by direct experimentation^[9]—as in this experiment. With this experiment, chemical engineering students have the opportunity to study a small process unit to determine the PID controller settings using the Process Reaction Curve Method combined with Internal Model Control or Ziegler-Nichols tuning relations. These theoretical parameters are tested via closed-loop experiments and the results are compared.

EXPERIMENTAL SET-UP

Experiments are carried out on PCT23 Process Plant Trainer equipment shipped by Armfield.^[10] It is a very flexible apparatus that integrates a small pasteurization unit, and offers a wide range of operating possibilities such as manual operation, data logging with PC, direct digital control, and industrial PID and PLC.^[12] This equipment, schematically shown in Figure 1, consists of a cold water feed tank, heater tank, plate-heat

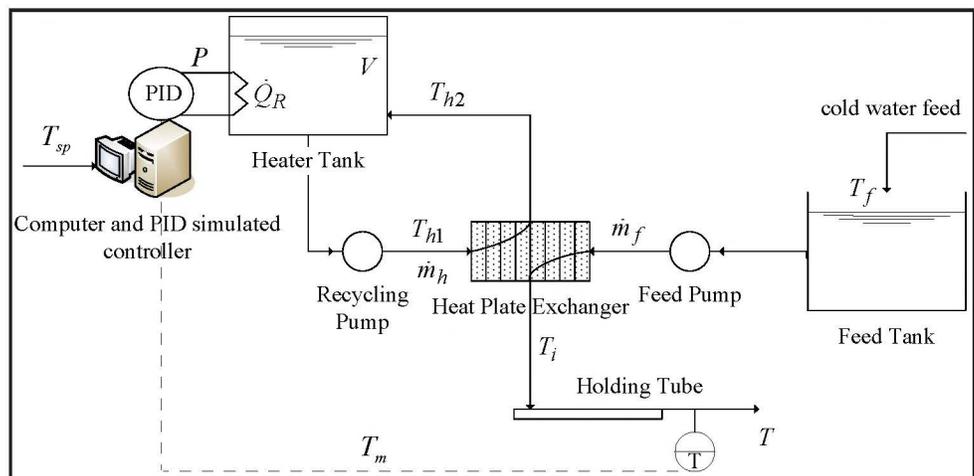


Figure 1. Schematic of the experimental equipment.

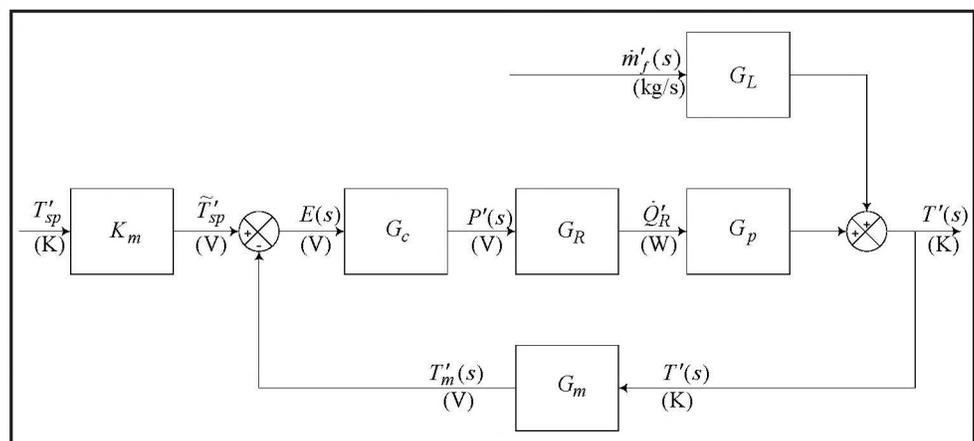


Figure 2. Block diagram of the feedback control system.

exchanger, holding tube with insulation, recycling pump, and feed pump. A PC-based system logs all signals and uses the Genesis software package where the PID control algorithm is implemented.^[10]

The objective of this set-up is to heat the feed water from temperature T_f to an exit temperature, $T = T_{sp}$, using the plate-heat exchanger and a hot stream from the heater tank at T_{h1} . Therefore, the control strategy consists of measuring T and adjusting the power to the electric heater, \dot{Q}_R , so that, regardless of disturbances, the exit temperature returns to T_{sp} . The holding tube is insulated so a pure time delay results; \dot{m}_f and \dot{m}_h denote the water mass flows of the feed and the heating circuits, and V is the liquid volume inside the heater tank.

THEORY

Using the described control strategy, the controlled variable is T , whose set-point is $T = T_{sp}$, and the manipulated variable is \dot{Q}_R . In this experiment, a disturbance is introduced by changing the cold water feed flow rate, \dot{m}_f , using a peristaltic pump. Figure 2 (previous page) is the resulting block diagram of the feedback control system, where: G_c , G_R , G_p , G_m , and G_L are the transfer functions for controller, electrical resistance heater, process, thermocouple-transmitter combination, and load, respectively; K_m is the gain that converts the set-point, T'_{sp} , to a voltage signal, \tilde{T}'_{sp} , that is used internally by the controller; E is the error signal; P' is the controller output; and T'_m is the measured temperature. Apostrophes identify deviation variables calculated from the original steady state values, for instance: $T'_m \equiv T_m - T_m^0$, $\dot{Q}'_R \equiv \dot{Q}_R - \dot{Q}_R^0$, etc.

In this experiment two different controller tuning techniques

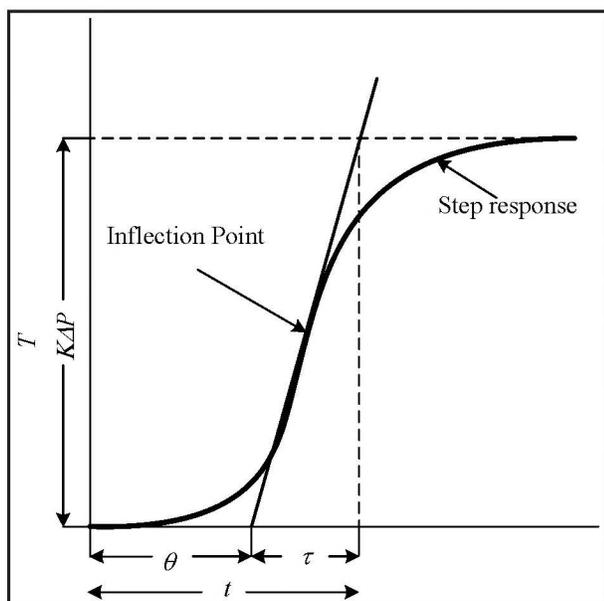


Figure 3. Step response of a first-order system with time delay, and the graphical analysis^[1, 11] required to obtain the parameters for the Ziegler-Nichols tuning rules.

are tested: i) Process Reaction Curve Method combined with an Internal Model Control Method (IMC) tuning rule; and ii) Process Reaction Curve Method combined with Ziegler-Nichols (ZN) tuning relations.^[1, 11]

The Process Reaction Curve Method is an established procedure for determining the parameters of an open-loop transfer function with a single experimental test carried out with the controller in manual. A step change in the controller output (ΔP) is introduced and the measured process response, reaction curve $T(t)$, is recorded. In this experiment, a first-order plus time delay model is selected and can be written in the Laplace or frequency domain as:

$$\frac{T'(s)}{P'(s)} = G_R G_p G_m = \frac{K e^{-\theta s}}{\tau s + 1} \quad (1)$$

Eq. (1) includes the transfer functions for the electrical resistance heater, the process, the thermocouple, and the temperature transmitter, for a step input of magnitude ΔP :

$$P'(s) = \Delta P / s \Rightarrow T'(s) = \frac{K \Delta P e^{-\theta s}}{s(\tau s + 1)} \quad (2)$$

Taking the inverse Laplace transform, the time domain response is:

$$\Delta T(t) = K \Delta P (1 - e^{-(t-\theta)/\tau}) \quad (3)$$

Note the system gain K , the time delay θ , and the time constant τ are the model parameters. Process gain is the ratio of the change in the steady state value of ΔT to the size of the step change ΔP [from Eq. (3): $\Delta T^\infty = \Delta T(t = \infty) = K \Delta P$], and τ may be found using several graphical methods.^[1, 11] Alternatively, the three parameters may be estimated by nonlinear regression.

There are many methods to select the PID controller settings. The ideal PID controller equation illustrates the three required parameters (gain K_c , integral time τ_I , and derivative time τ_D):

$$P = P^0 + K_c \left[E(t) + \frac{1}{\tau_I} \int_0^t E(t) dt + \tau_D \frac{dE(t)}{dt} \right] \quad (4)$$

For a first-order plus dead time model, the IMC provides the following tuning relations:^[11]

$$K_c = \frac{1}{K} \frac{\tau + \frac{\theta}{2}}{\tau_c + \frac{\theta}{2}}, \tau_I = \tau + \frac{\theta}{2}, \tau_D = \frac{\tau \theta}{2\tau + \theta}, \quad (5)$$

where τ_c is a design controller parameter normally chosen as $\theta \leq \tau_c < \tau$.

Alternatively, controller settings may be determined using the ZN tuning rules,

$$K_c = \frac{1.2}{\theta S^*}, \tau_I = 2\theta, \tau_D = 0.5\theta, \quad (6)$$

where θ and $S^* = S/\Delta P = K/\tau$ are determined by graphical analysis of the process reaction curve shown in Figure 3.

Accordingly: i) process gain K is the ratio of the change in the steady state value of T divided by the step change ΔP ; ii) the root of tangent line drawn at inflection point S is the time delay θ ; iii) S is the slope of tangent line; and iv) extending the tangent at inflection point to the steady state line $T = K\Delta P$, the intersection point corresponds to $t = \theta + \tau$.

The performance of the resultant feedback control system has to be tested and evaluated prior to start-up of an industrial plant, by studying the dynamic and steady state characteristics of its response to some perturbations. This task is accomplished using two laboratory experiments, one for each set of suggested controller settings [Eq. (5) or Eq. (6)].

EXPERIMENTAL PROCEDURE

Table 1 shows the operating conditions, useful notes, and necessary data as part of the laboratory controller tuning experiment in steady state conditions. Super-scripts 'o' and ' ∞ ' stand for original and final steady state conditions, respectively [e.g., $P^o = P(t = 0)$ and $P^\infty = P(t = \infty)$]. The computer interface of the PCT23 Process Plant Trainer shows the controller output as a percentage. Experimental procedure is as follows:

- a) Initialize experimental set-up and let system reach a steady state.
- b) Carry out an open-loop experiment (controller in manual) to measure the process reaction curve by introducing a step change

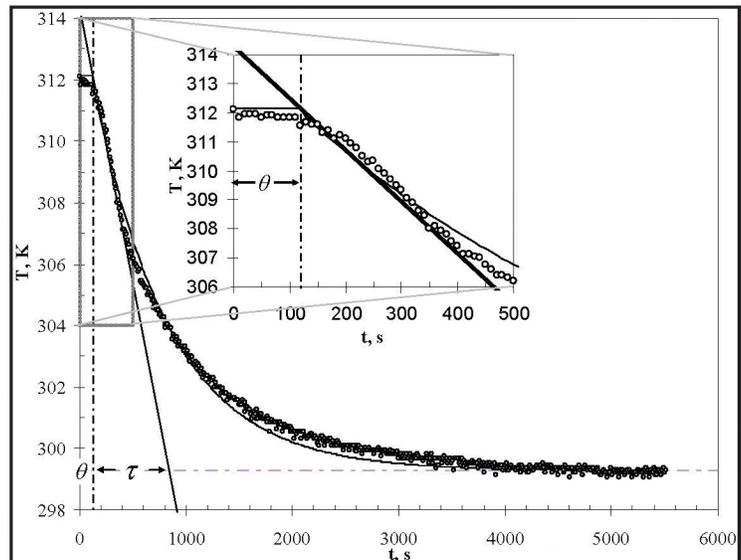


Figure 4. Process reaction curve for operating conditions shown in Table 2, and first-order plus time delay model fitted to experimental data. The expanded area of the first 500s is given in the inset.

Step	Task	Experimental Conditions	Register	Notes
a	Reach steady-state	$P = 30\%$ $\dot{m}_f = 4 \times 10^{-3} \text{ kg/s}$ $\dot{m}_h = 6.2 \times 10^{-3} \text{ kg/s}$	T^∞	<ul style="list-style-type: none"> $T^\infty = T(t = \infty)$, etc. Computer interface shows P in percentage
b	Open-loop experiment to measure process reaction curve	$T^o = T^\infty$ (step a) Input step in P : $\Delta P = -15\% \Rightarrow P = 15\%$	$T(t)$	<ul style="list-style-type: none"> Put controller in manual mode Initial steady-state is the final condition of step a
c	Wait for steady-state (e.g., same as step a)	$P = 30\%$ $\dot{m}_f = 4 \times 10^{-3} \text{ kg/s}$ $\dot{m}_h = 6.2 \times 10^{-3} \text{ kg/s}$	T^∞	<ul style="list-style-type: none"> There is enough time to optimize model parameters [Eq.(3)], carry out graphical analysis (Fig.3), and calculate controller settings using Eqs. (5) and (6)
d	Closed-loop experiment (controller settings obtained by Internal Model Control in step c)	$T_{sp} = T^\infty$ (step c) $P^o = 30\%$ K_c, τ_I, τ_D Input step in \dot{m}_f : $\Delta \dot{m}_f = (4 \rightarrow 2) \times 10^{-3} \text{ kg/s}$	$T(t)$	<ul style="list-style-type: none"> Initial steady state is final condition of step c Set-point is T^∞ from step c P varies with time
e	Reach same steady-state of step c	$P = 30\%$ $\dot{m}_f = 4 \times 10^{-3} \text{ kg/s}$ $\dot{m}_h = 6.2 \times 10^{-3} \text{ kg/s}$	T^∞	—
f	Closed-loop experiment (controller settings obtained from Ziegler - Nichols equations in step c)	$T_{sp} = T^\infty$ (step e) $P^o = 30\%$ K_c, τ_I, τ_D Input step in \dot{m}_f : $\Delta \dot{m}_f = (4 \rightarrow 2) \times 10^{-3} \text{ kg/s}$	$T(t)$	<ul style="list-style-type: none"> Initial steady state is final condition of step e Set-point is T^∞ from step e (or step c) P varies with time

in the controller output (ΔP). Plot temperature T vs. time.

c) Operate system to attain a steady state. During this time

period, optimize the parameters of the first-order plus time delay model [Eq. (3)] using a spreadsheet program and least-squares method. Plot the resultant correlation on the same plot as the experimental data. Next, calculate the PID controller settings using Eq. (5) (IMC). Using the required graphical analysis of Figure 3, compute the controller settings using the ZN tuning rules [Eq. (6)].

d) Perform a closed-loop experiment with the PID controller settings obtained in step c) using IMC, introducing a step perturbation in the feed mass flow. Plot the temperature T vs. time.

e) Return the system to attain the same initial steady state (*i.e.*, final condition of step c).

f) Perform a closed-loop experiment with the PID controller settings obtained from the c) ZN approach. Again introduce the same step perturbation in the feed mass flow and plot the temperature T vs. time on the same graph of step d).

NOTE: In order to reduce experimental time duration, the reaction curve can be given to students before the laboratory session. For a reduced two-week laboratory session, students can perform the reaction curve and PID tuning calculation

in the first week, and the closed-loop experiments in the second week.

TABLE 2	
Experimental Conditions, Observations, and Calculated Results	
Open-loop identification experiment (Table 3, step b)	
Experimental conditions: $\dot{m}_r = 4 \times 10^{-3} \text{ kg/s}$, $\dot{m}_h = 6.2 \times 10^{-3} \text{ kg/s}$, $T^\circ = 39^\circ\text{C}$	
Input step in P : $\Delta P^\circ : 30\% \rightarrow 15\%$	
Experimental observations: $T = T(t)$ shown in Figure 4.	
Calculated results from process reaction curve	
Model parameters [Eq.(3): $K = 0.8476 \text{ K}/\%$, $\theta = 120 \text{ s}$, $\tau = 717.3 \text{ s}$ (from nonlinear fitting AAD = 0.073%)	
PID controller settings from Internal Model Control Method [Eq.(5): $K_c = 5.10 \text{ } \%/K$, $\tau_i = 777.3 \text{ s}$, $\tau_D = 55.4 \text{ s}$	
Experimental parameters (from Figure 4): $ S^* = 0.0014 \text{ K/s}$; $\theta = 120 \text{ s}$, $\tau = 730 \text{ s}$	
PID controller settings from Ziegler-Nichols relations [Eq.(6): $K_c = 7.14 \text{ } \%/K$, $\tau_i = 240.0 \text{ s}$, $\tau_D = 60.0 \text{ s}$	
Closed-loop experiments (Table 3, steps d and f)	
Experimental conditions: $\dot{m}_h = 6.2 \times 10^{-3} \text{ kg/s}$, $P^\circ = 30\%$, $T_{sp} = T^\circ = 39^\circ\text{C}$	
Input step in \dot{m}_r : $\Delta \dot{m}_r : (4 \rightarrow 2) \times 10^{-3} \text{ kg/s}$	
Experimental observations for each set of PID parameters $T = T(t)$ shown in Figure 5.	

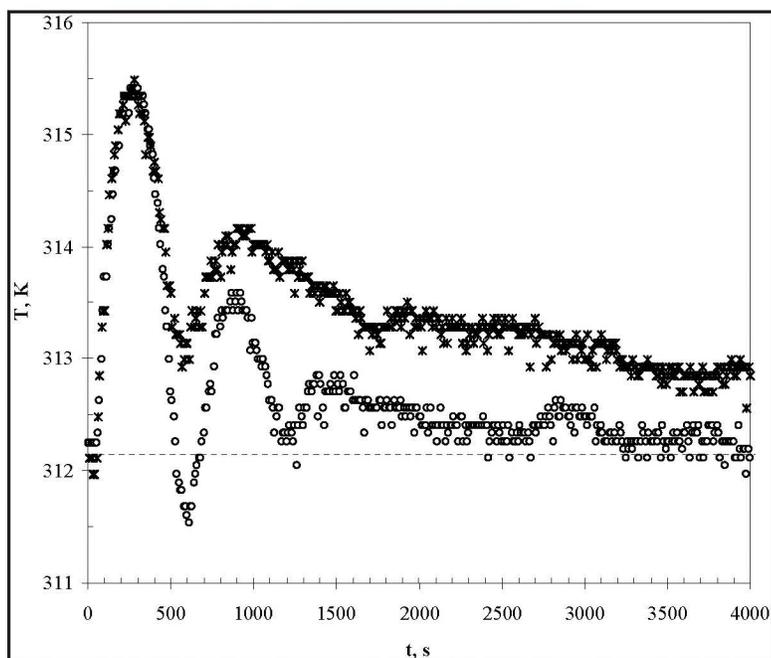


Figure 5. Closed-loop responses obtained for the PID controller settings determined by Internal Model Control tuning relations [Eq. (5)], (xxx); Ziegler-Nichols relations [Eq. (6)], (ooo). Experimental conditions and parameters are shown in Table 2.

TYPICAL RESULTS AND DISCUSSION

After the experimental work proposed, the students collected one reaction curve and two closed-loop experiments with IMC and ZN tuning settings. Table 2 summarizes the experimental conditions, observations, and calculated results. The obtained process reaction curve is presented in Figure 4 (previous page), as well as the results from a first-order plus dead time model [Eq. (3)]. The results of using the ZN rules are also shown in Figure 4. The small deviation (AAD = 0.073%) does show the excellent fit of the data. Furthermore, students can also conclude that the process is self-regulating since its reaction curve is bounded and reaches a new steady state after a step change at $t = 0$. It should be noted that the student must realize the reaction curve can be inverted, resulting in the standard diagram (Figure 3).

Figure 5 shows the typical underdamped-load responses of the closed-loop PID controlled system.

For the same experimental conditions (Table 2), these results emphasize the effect that different controller settings have on system response. The ZN tuning rules result in a faster response (more aggressive). This response is due to a combination of higher K_c and lower τ_I values:^[11] 7.14 %/K and 200.0 s (ZN), respectively, compared to 5.10 %/K and 777.3 s (IMC). Students need to be aware of this fact. The feedback control system with IMC parameters exhibits a remarkably sluggish response, that of the set-point intersected at 7600 s, compared to ZN at 535 s.

Students should conclude there is no perfect controller tuning method. The goal is to have good preliminary education that can provide a starting point for additional field tuning, especially when available process information is incomplete or inaccurate.^[1,11]

FURTHER REMARKS

The proposed experiment is mainly oriented toward system dynamics and controller tuning. Other useful tasks may be considered by instructors, such as carrying out a thermal analysis of the system to allow the estimation of heat losses and global heat transfer coefficients. The same experiment may be implemented using the industrial PID and console available in the PCT23 unit. This set-up allows us to change control strategy to a cascade control scheme.

At the end of this work students answer a survey, allowing instructors to figure out the benefits and difficulties found during its execution. Following a procedure suggested by other authors,^[12] the set of questions listed in Table 3 was assessed from 1 (strongly disagree) to 5 (strongly agree). The results suggest that this experimental work accomplishes the main objectives in general and does contribute to students' understanding and interest in the field of process control.

CONCLUSIONS

PCT23 Process Plant Trainer equipment provided by Armfield^[10] is used to teach PID controller tuning. The experiments introduce and solidify theoretical concepts by obtaining approximate transfer functions or time domain models from a process output response to some step input, and by calculating the PID controller settings from typical industry-developed tuning rules. They also provide the means for experimental validation of controller performance. Students do recognize that there are no unique methods to estimate satisfactory controller settings and that additional field tuning may be required.

ACKNOWLEDGMENTS

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TABLE 3
A Proposed Survey for Assessing the Usefulness of This Experimental Work

The answers for all questions were classified as: strongly disagree (1 point); disagree (2 points); somewhat agree (3 points); agree (4 points); strongly agree (5 points). Results refer to 32 students in 2005-2006.

Question	Mean	Standard Deviation
1. Were the concepts previously acquired on process control sufficient to allow you to carry out this work?	3.29	0.61
2. Is this experimental work connected with the theory taught in other control courses?	3.29	0.61
3. Is the available bibliography sufficient?	4.00	0.68
4. Were you able to do the experimental work without difficulty?	2.29	0.91
5. Were you able to do the calculations without difficulty?	2.00	0.78
6. Were you able to interpret and discuss the results without difficulty?	2.50	0.65
7. Do you think you improved your skills in controller tuning?	3.43	0.51
8. Did the practical work motivate or increase your enthusiasm about process control?	3.36	0.50
9. Did you find the experimental work important for the understanding of chemical process control?	3.71	0.83
10. Do you feel stimulated to take future new challenges in the field of control?	3.43	0.65

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NOMENCLATURE

AAD Absolute Average Deviation

IMC Internal Model Control

$E = T_{sp} - T$, Error signal

G_c, G_R, G_p, G_m, G_L Transfer functions for controller, electrical resistance heater, process, thermocouple-transmitter, and load

K Process gain

K_c Controller gain

$K_m = T'_{sp} / \tilde{T}'_{sp}$, gain to express set-point (K) as voltage signal (V)

\dot{m} Mass flow, kg/s

P Controller output

PID Proportional-Integral-Derivative

\dot{Q}_R	Power of electrical heater, W
t	Time, s
T	Temperature, K
V	Liquid volume inside heater tank, m ³
ZN	Ziegler-Nichols

Greek letters

Δ	Deviation relative to initial steady state value; Step change
τ	System time constant, s
τ_i, τ_D	Integral and derivative time, s
θ	Time delay, s

Superscripts

$^{\circ}, \infty$	Initial and final steady state conditions.
\sim	Variable expressed as voltage
\wedge	Deviation variable

Subscripts

F	Feed
H	Heating circuit
M	Measured value
Sp	Set-point

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INTEGRATING ENVIRONMENTAL MANAGEMENT

By Introducing an Environmental Management System in the Student Laboratory

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The most efficient way to achieve an environmental and sustainable development culture and prevent pollution from industrial processes is to educate students by our own example. For this reason, the university must be a reference for future professionals who are to lead the industrial processes. These leaders will be chemical engineering students who, in the near future, will perform engineering tasks. Therefore knowledge, principles, and basics related to sustainable development should be introduced within the curriculum.^[1, 2] These principles could be easily introduced in subjects related to Environmental Science and Technology, which constitutes a very important aspect of chemical engineering education. Engineering educators are responsible for achieving this goal, but in order to do so, the scientific and technological fundamentals of Environmental Science should be explained on the basis of sustainable development. These explanations can be applied using a tool that is now more common in industry and a subject in the chemical engineering studies curricula:^[3] the environmental management system.

An environmental management system is defined by the International Organization for Standardization (ISO) as “the part of the overall management system that includes organizational structure, planning activities, responsibilities, practices, procedures, processes, and resources for developing, implementing, achieving, reviewing, and maintaining the environmental policy.” Environmental policy is defined as the “statement by the organization of its intentions and principles in relation to its overall environmental performance, which provides a framework for action and for the setting of environmental objectives and targets.”

There are a number of standards upon which one can model different environmental management systems. The ISO 14001 environmental management system standard is the most widely recognized framework, and many entities have their environmental management systems certified as conforming to ISO 14001.^[4] The main features of these systems are:

- *A voluntary approach oriented to developing positive long-term objectives and progressing toward achieving them, rather than applying stiff penalties for failing to comply with many itemized requirements.*

- *A continual step-by-step improvement in the environmental performance of enterprises and industries.*
- *A complete integration of the principle of sustainable development.*
- *An easy and natural integration with other standards such as ISO 9000, the Quality Management System.*

The first objective of these systems is the same as that of chemical engineering educators: To enhance a positive attitude about environmental protection and management. Achieving this objective will result, on a long-term scale, in more sustainable development within society.^[5, 6] The second objective is to design elements and implementation steps. Both of these objectives involve orientation and working familiarity with environmental management system guidelines. This is not always easy to achieve for workers and/or students because they do not usually feel involved.

Further, the selected objectives should be achieved within the more abstract concept of sustainable development. This concept demands that the three main aspects of development—economic, social, and environmental—be considered as



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a whole.^[7-9] Attempting to do so often produces conflicting interests, and the solution—a successful integration—implies an essential change from commonly held views on progress and growth in human society. This integration is not explicitly included in ISO 14001 systems. For this reason, educators must provide the knowledge, concepts, and approaches of sustainable development to students—who will soon contribute to society’s material progress and growth.

Using the environmental management system as a tool, we introduced the concept of sustainable development in chemical engineering education within the unit operations laboratory, which is the facility most similar to an industrial facility. In this way students become conscious in every session of the fact that they are participating in an environmental management system and trying to achieve further objectives related to environment that will contribute to the sustainable development of society. Students must be aware of generated hazardous waste and how adequate management of it is fundamental to avoiding problems in basic utilities such as drinking water, wastewater, or solid waste treatment. Students must learn to try to minimize waste production and the use of reagents while considering the social, economic, and environmental importance of this minimization. Students must work according to environmental and security regulations not only because it is their obligation legally but because they are conscious of its importance for society. To do so, students must be supplied with the necessary procedures and instructions. In this paper we show how the system has been developed and implemented in the laboratory, what role students have played, the main problems identified, and how those problems have been solved.

CONTEXT

Academic context

This work was undertaken in the Chemical and Nuclear Engineering Department of the Technical University of Valencia. This department teaches many subjects within engineering studies such as industrial engineering, chemical engineering, and materials engineering. Many of these subjects are related to environmental science, including environmental science and technology, environmental technology, water pollution, air pollution, solid wastes, environmental analytical techniques, environmental management, environmental impacts, radioactive pollution, and drinking water treatments. These subjects are scheduled for the later years of a student’s studies as they build upon earlier knowledge to introduce concepts and techniques related to water pollution, air pollution, and hazardous wastes, the management of these concepts, and the main preventive tools used in industry.

The plethora of subjects related to the environment taught by our department was the basis for our objective of introducing sustainable-development principles and objectives throughout the students’ academic tasks.

PREVIOUS EXPERIENCE

Among the various engineering studies, the Chemical and Nuclear Engineering Department mainly works with the Industrial Engineering School. This higher education center has been certified under the ISO 14001 Environmental Management System.^[10]

The principal objective of this system is to improve the center’s activities from an environmental point of view, to properly treat its wastes (paper, plastics, hazardous wastes, etc.), to reduce its consumption of raw materials, to create a positive attitude toward the environment, and to integrate the principles of sustainable development.

As in all environmental management systems, the people affected should be integrally involved, including not only the staff of the center but also the students. Although the students are the largest part of the Industrial Engineering School, it has been demonstrated that they do not know what the environmental management system is, what it implies, or what their role is within it. Usually their only contribution to the environmental management system consists of putting waste in the appropriate container as a rote action, without being conscious that they are part of a global management system trying to achieve broader objectives. For this reason, and because we are responsible for environmental subjects in engineering studies, it is our duty to involve students in the environmental management system and to use it to introduce the principles of sustainable development.

APPLICATION TO THE DEPARTMENT

In Spain, the docent responsibilities and student laboratories depend on the various departments within the university. Based on the implementation of an environmental management system in the Industrial Engineering School, the Chemi-

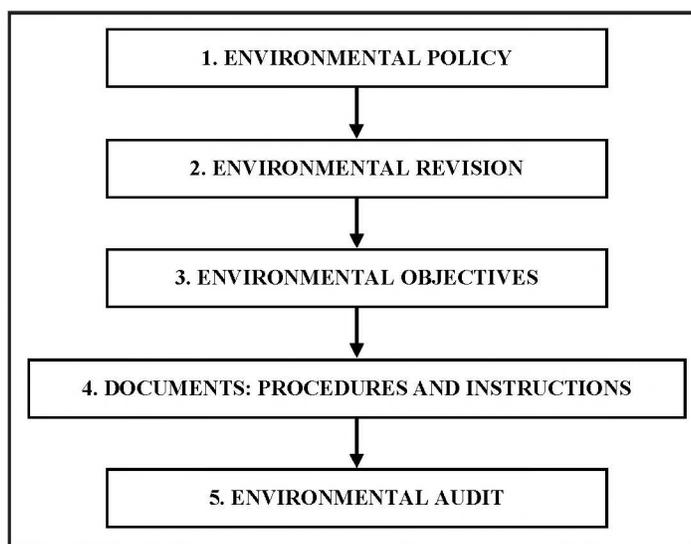


Figure 1. Steps to implement an environmental management system.^[11]

cal and Nuclear Engineering Department is implementing an environmental management system in accordance with the ISO 14001 specifications. The steps toward implementation are contained in Figure 1. Referring these steps to the student's laboratory, we have:

1. Environmental Policy

The first step was approving the department's environmental policy. This environmental policy is presented in Figure 2. Many points of the environmental policy are directly related to students, and according to it our duty as engineering educa-



Ingeniería Química y Nuclear

ENVIRONMENTAL POLICY

The Chemical and Nuclear Engineering Department is aware of the need to incorporate the environmental ethics to all its activities and, in accordance with the Environmental Policy of the Polytechnic University of Valencia, it has decided to assume that responsibility. The University, and by extension its Departments, have, as a main target, the creation, development and transmission of Science, Technology and Culture. They are an instrument for the advance of our society and for intellectual development, as well as for the promotion of the freedom of thought. Through all this, it is possible to influence society by introducing improvements in the relation of the human activity with nature and in the management of the natural resources.

The Department assumes the contents of the Agenda 21 document from the United Nations. It assumes the responsibility to generate science, technology and culture, according to solidarity principles with all the contemporary world and under sustainability criteria in order to extend it towards the future generations.

As part of a Higher Education Institution, the Department tries to raise awareness in all its members, as well as in the students, for the preservation and improvement of the Environment. It is conscious that through instruction and research it plays an exceptional role in the transformation of society.

As an instrument to reach these aims, the Chemical and Nuclear Engineering Department is committed to implement an Environmental Management System: UNE-EN-ISO 14001 and consequently to try to maintain the continuous improvement of its environmental practices. In particular:

1. Analyzing and evaluating the activities developed in the Department, according to the Environmental Management System, and trying to determine and to diminish the environmental impacts that can be derived from them.
2. Providing an environmental instruction adapted to all the students and workers.
3. Providing to the members of the Department appropriate training and environmental information related to their activity.
4. Fulfilling all the environmental legal requirements, trying to go beyond the prescribed minimums.
5. Rationalizing the consumption of raw materials, resources and energy.
6. Preventing the possible pollution and avoiding, as far as possible, the spills, the emissions and the wastes generated in the different activities.
7. Managing the wastes generated according to laws.
8. Informing of its objectives to the university community, favoring its participation in the environmental instruction of the students.
9. Working with the companies, institutions, and people that develop their activity with the department, to help them and to commend them for improving environmental performances.

To carry out these commitments, challenging environmental objectives will be established. They will be public and, as far as possible, quantifiable, evaluating continuously the goals.

Annual reviews will be made that will contain a revision of the environmental performances of the department and these will become public, spreading the expected objectives to all the university community

The Chemical and Nuclear Engineering Department Director

Figure 2. Environmental Policy of the Chemical and Nuclear Engineering Department of the Technical University of Valencia.

tors is to provide and foment environmental instruction on the basis of the sustainable development principles adapted to all students (points two and eight). In the laboratory, students are the main consumers of raw materials, resources, and energy. Therefore, according to point five, they must rationalize the consumption of these resources. They have to prevent possible pollution and avoid, as best as possible, the spills, emissions, and wastes generated in different activities (point six). Finally, according to point seven, they must manage the wastes properly in accordance with environmental laws. As faculty, we have to supervise and stimulate them to achieve these objectives, as well as to not only be cognizant of the Environmental Policy but also to develop new attitudes and aptitudes more consistent with the principles of sustainable development.

2. Environmental Revision

The second step was to analyze the department's environmental situation, including how it manages wastes, the consumption of raw materials, energy, etc. This analysis unveiled environmentally strong and weak points used to establish environmental goals to be achieved. In-lab discussion was used to alert students to these goals and how they relate to sustainable-development objectives, thereby encouraging student participation in the continuous improvement process required by ISO 14001.

3. Environmental Objectives

The third step was to establish objectives to be achieved. These objectives were related to different activities, but one of the most important objectives was to involve the students in the environmental management system in order to develop the principles of sustainable development.

4. Documents: Procedures and Instructions

The last step was the preparation of a manual with the necessary procedures and instructions. The most important in the student's laboratory are the following:

Procedures

- ▶ IQN-P-001: Procedure for the identification of significant impacts. This procedure aims to identify and quantify the impact of laboratory management on the environment, society, and the economy.
- ▶ IQN-P-002: Procedure for the identification and updating of the legal requirements.
- ▶ IQN-P-003: Procedure for environmental instruction. This procedure shows how to develop informative tasks in the laboratory. This information given to the students will not only be related to environmental issues, but also to cooperation and sustainable development.
- ▶ IQN-P-004: Procedure for writing and controlling environmental management system documents.
- ▶ IQN-P-005: Procedure for control of the wastes. This procedure includes classification of the waste indicating its hazard, the possible risks of inadequate control of it, and recommendations to reduce expenses derived from its management.
- ▶ IQN-P-006: Procedure for management of raw materials and natural resources. This procedure includes recommendations for reducing consumption of raw materials and natural resources, and for reducing expenses derived from their use.
- ▶ IQN-P-007: Procedure for environmental control of dealers. This procedure encourages dealing with companies that perform their activities according to the principles of sustainable development.
- ▶ IQN-P-008: Procedure for emergency situations.
- ▶ IQN-P-009: Procedure for detecting and correcting inadequate environmental management system performance and implementing future preventive actions.
- ▶ IQN-P-010: Procedure for internal and external environmental communication.
- ▶ IQN-P-011: Procedure for internal audits of the environmental management system.
- ▶ IQN-P-012: Procedure for revision of the environmental management system.

Instructions

- ▶ IQN-I-001: Instructions for proper hazardous wastes management.
- ▶ IQN-I-002: Instructions for management of waste paper.
- ▶ IQN-I-003: Instructions for management of waste glass.
- ▶ IQN-I-004: Instructions for management of used electronic equipment.
- ▶ IQN-I-005: Instructions for hazardous substances identification in the shopping process.
- ▶ IQN-I-006: Instructions for work in the laboratory according to environmental and sustainable principles.
- ▶ IQN-I-007: Instructions for the development of new, practical, laboratory classes according to environmental and sustainable principles.

For the students, the most important instructions and procedures presented in the first class are: IQN-P-001, IQN-P-003, IQN-P-005, IQN-P-006, IQN-P-008, IQN-P-009, IQN-I-001, IQN-I-002, IQN-I-003, and IQN-I-006. All allow students to actively participate in the environmental management system because they mainly relate to: the proper management and minimization of wastes and reagents; the critical analysis of the system in relation to sustainable-development principles; and carrying out adequate sustainable development and environmental work.

APPLICATION TO THE STUDENTS' LABORATORY

The most active student participation is developed in the laboratory. For this reason we emphasize applying the environmental management system to the teaching laboratory. In this way students are more conscious in every session that they are participating in an environmental management system. They learn that the principles that guide this system can be applied afterward in industry and that these principles are the same as those from sustainable development. Among other things, students must try to minimize waste production and reagent consumption, and be sure to work according to environmental regulations and the sustainable-development principles. To achieve these goals, we supply students with adequate explanations, concrete examples, and the necessary procedures and instructions.

Two problems identified with applying the environmental management system to the student laboratory were: 1) the proper management of varied wastes generated in the chemistry laboratory due to the great diversity, and 2) the sequence of courses. Subjects related to environmental science are upper-level courses. Experimental subjects, however, are pursued in the student's laboratory from the first year of the student's studies. At this level, they only have a general sense of what an environmental management system is and what the principles of sustainable development are.

To solve these problems, the following actions were taken:

- ▶ *The first day in the laboratory, students receive a theoretical explanation of the department's environmental management system. The explanation includes how the objectives of this system are related to the principles of sustainable development, and how students themselves could apply these principles in society after finishing their studies. The most important procedures and instructions used in the laboratory are also explained to the students.*
- ▶ *In each laboratory class, students have to write a report of their results that includes an environmental and sustainable evaluation of their work. This evaluation addresses the identification of the reactants used and the wastes generated. They must evaluate the cost of the experiment as well as determine the composition of the wastes and characterize them according to environmental laws. Then, they have to decide if the waste can be recycled, disposed of down the drain or in the general garbage, or if it must be collected in specific containers. To make this work easier, students receive a handout (shown in Table 1, next page) with an example from a chemistry laboratory exercise. They also use this page to gain awareness of the type and quantity of reagents used and the waste produced. In this way, it is easier for them to evaluate the work from an environmental and economic point of view. Additionally, they reference the bylaws pertaining to water pollutants and proper*

hazardous-waste management.

- ▶ *In each session, students must propose alternatives to improve the practical laboratory class from the perspective of sustainable development. Concretely, they have to propose alternatives, when it is possible, for the used reactants by looking for other chemicals with a lower environmental, economical, and social impact. They also have to propose alternatives for proper waste management, and they can even make suggestions about the entire process. Initially these suggestions are more focused on the minimization of wastes, but later they consider the entire process from an environmental, social, and economical point of view.*
- ▶ *The last day in the laboratory, students have to discuss the different alternatives. This is probably the most active participation from students in the environmental management system because they usually find new environmental solutions and make new environmental suggestions related not only to the generated wastes but also to the development of the environmental policy, the inclusion of new facts in the environmental revision, and the criticism and development of the procedures and instructions to be more adequate for the practical laboratory classes. As an example of these proposals, students have suggested completing Statement 7 of the environment policy by adding the necessity of going beyond the legal obligations. These suggestions are made according to procedure IQN-P-012.*

The last two points are the most difficult for students—particularly for students in the first year of their studies—as it is much easier for last-year students who have already taken numerous classes related to Environmental Science and Technology. In addition, students in last year of their studies are more interested and active in the practical laboratory classes. They become more conscious that they are a part of the environmental management system and that the application of this system can contribute to sustainable development.

Nevertheless, the results obtained are very encouraging. In fact, we have approximately quantified the consumption of reagents per student and the production of wastes per student in some experiments carried out in the laboratory, and we have compared them between the first and the last years of study. Table 2 (next page) shows an example for the titration of hydrochloric acid (a typical titration necessary for various laboratory classes). The results show that, excluding accidents, there is a clear decrease in the consumption of reagents and the production of wastes in a student's last academic year. This decrease can be attributed not only to students' greater experience, but also to their greater awareness.

Therefore, the environmental management system is a tool that can easily be applied to introduce the principles of sustainable development in all the aspects of the future process that will be controlled by the students when they join the work force.

TABLE 1			
Illustrative Form to Be Filled in By Students After Each Practical Laboratory Class			
LABORATORY		VE16010	
SUBJECT		Chemistry laboratory	
STUDIES		Chemical Engineering	YEAR 2nd
CLASS No.	5	Redox titration	
OBJECTIVES:		Determination of the iron concentration present in a problem solution by means of a redox titration, using MnO_4^- and $\text{Cr}_2\text{O}_7^{2-}$ as oxidant agents.	
REAGENTS USED			
Reagent	Mass (g) or Volume (ml)	Concentration	Comments
$\text{Na}_2\text{C}_2\text{O}_4$	0.22 g/100 ml	2.201 g/l	
H_2SO_4	10 ml		
KMnO_4	70 ml	0.1 N	
Problem solution (Fe^{2+} , Fe^{3+})	20 ml		
SnCl_2	-		In excess
HgCl_2	10 ml	0.25 M	
Zimmerman solution	25 ml		(Mn^{2+} , H_2SO_4 , H_3PO_4)
H_3PO_4	5 ml		
$\text{Cr}_2\text{O}_7^{2-}$	50 ml		
WASTE PRODUCED			
LIQUIDS	Mass (g) or Volume (ml)	Concentration (ppm)	Type
$\text{CO}_2 + \text{Mn}^{2+} + \text{H}_2\text{SO}_4$	140 ml	$[\text{Mn}^{2+}] = 257.9$	Hazardous waste
$\text{Fe}^{3+} + \text{Sn}^{4+} + \text{Cl}^- + \text{Hg}_2\text{Cl}_2 + \text{Zimm.} + \text{Mn}^{2+}$	85 ml	$[\text{Fe}^{2+}] = 10259.3$; $[\text{Cl}^-] = 1044.12$; $[\text{Mn}^{2+}] = 475.2$	Hazardous waste
$\text{Fe}^{3+} + \text{Sn}^{4+} + \text{Cl}^- + \text{Hg}_2\text{Cl}_2 + \text{H}_3\text{PO}_4 + \text{H}_2\text{SO}_4 + \text{Cr}_2\text{O}_7^{2-}$	80 ml	$[\text{Fe}^{2+}] = 10970$; $[\text{Cl}^-] = 1044.12$; $[\text{Cr}^{3+}] = 866.6$	Hazardous waste
GASES	Mass (g) or Volume (ml)	Concentration (ppm)	Type
SOLIDS	Mass (g) or Volume (ml)	Concentration (ppm)	Type

TABLE 2		
Evolution of the Consumption of Reagents Per Student and the Production of Wastes Per Student in the Titration of Hydrochloric Acid		
Reagent	Mass (g) or Volume (ml)	
	2nd academic year	5th academic year
Na_2CO_3	~0.85 g	~0.45 g
HCl ~ 0.1N	~100 ml	~30 ml
Phenolphthalein	~6 droplets	~4 droplets
Distilled water	~500 ml	~300 ml
PRODUCTION OF WASTES		
Waste	Mass (g) or Volume (ml)	
	2nd academic year	5th academic year
$\text{Na}_2\text{CO}_3 \sim 4.5 \text{ g/l}$	~150 ml	~75 ml
HCl	~80 ml	~20 ml
$\text{NaHCO}_3 + \text{NaCl} + \text{phenolphthalein}$	~70 ml	~60 ml

CONCLUSIONS

It is possible to introduce the concept of sustainable development in engineering education by implementing an environmental management system in the students' laboratory.

In the laboratory, students learn to reduce the consumption of raw materials, resources, and energy; to prevent pollution; to reduce the generation of wastes; and to manage the wastes generated according to environmental laws.

Also in the laboratory, students learn to propose alternatives for its management to improve the laboratory work from an environmental, economical, and social point of view.

Finally, through all these aspects, students are more conscious of the fact that they are participating in an environmental management system and that its objectives and methods can easily be applied to their future jobs with respect for the principles of sustainable development.

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This column provides examples of cases in which students have gained knowledge, insight, and experience in the practice of chemical engineering while in an industrial setting. Summer internships and co-op assignments typify such experiences; however, reports of more unusual cases are also welcome. Description of the analytical tools used and the skills developed during the project should be emphasized. These examples should stimulate innovative approaches to bring real-world tools and experiences back to campus for integration into the curriculum. Please submit manuscripts to Professor W.J. Koros, Chemical Engineering Department, Georgia Institute of Technology, Atlanta, GA, 30332-0100.

Challenges of Implementing **A JOINT INDUSTRIAL-ACADEMIC RESEARCH PROJECT**

As Part of a Nontraditional Industrial Ph.D. Dissertation

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To many people the notion of a distance-education doctorate in engineering may seem ridiculous enough by itself, but add the fact that the prospective student intends to maintain full-time employment as a senior process engineer with a chemical manufacturing company, and the ridiculous notion appears to become an impossibility. Indeed, convincing a university and a corporation, two notoriously inflexible institutions, to do as much “outside the box” thinking as was required for this endeavor was no small task. Despite the potentially daunting obstacles, however, and through a unique partnership between Evonik Degussa Corporation in the United States, Evonik Degussa GmbH in Germany, Auburn University, and the University of South Alabama, this seemingly impossible proposition has become a reality.

This article will describe the conception and organization of this unique and very nontraditional research project from the perspective of the student who is close to completing this

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unusual journey. At each step along the way, compromises from all parties have been required, but in the end an environment was established whereby everyone stood to gain from the process. As each step of the process is described, it will be accompanied by some first-hand lessons learned from the experience.

A WIN-WIN-WIN SCENARIO

The real key to making this unlikely dissertation project a reality was to negotiate a scenario where all parties involved — the student, the universities, and the company — had something to gain from a successful collaboration. For the student the benefit is clear, the chance for achieving the Doctor of Philosophy degree while maintaining a career. For the universities, the benefits include exposure to industrially relevant research and processes and the opportunity to apply theory to real applications. Additionally, universities can benefit greatly from the improved intellectual diversity stemming from adding industrial experience to academic research groups. For the company, the benefits come from the creative thinking required of the Ph.D. student, which can lead to an increased potential for innovation. Additional company benefits come both from the infusion of fresh mindsets into the research and development process and from establishing a relationship with the university to identify talented students for future employment, as well as the opportunity for corporate branding. By establishing a win-win-win scenario, everyone is motivated to find a way to ensure a successful outcome.

RESEARCH OBJECTIVES

The research project was originally conceived as a conceptual process-development project for the production of an industrial chemical from renewable, bio-based feed stocks.^[1] The original objectives included the development of a conceptual process and an assessment of its economic viability. In addition, laboratory experiments were to be carried out to gather the data needed for the process-design activities. This industrial project in and of itself, however, would not necessarily have contained sufficient academic rigor for a Ph.D. dissertation. To address this potential shortcoming, the original project has been incorporated into the other systems-engineering research work at Auburn University. In this way, the project could be given a broader scope, yet the original goals were preserved by including the industrial application as a case study.^[2]

In nontraditional projects like this one, it is incumbent upon the research advisor to ensure the resulting dissertation has sufficient depth. This was accomplished by ensuring that there was enough flexibility in the scope to allow the student to follow leads and dive more deeply into avenues of interest that arose as the project progressed. Because of this, the project evolved significantly as it moved along. In fact, the final results were quite different from what was initially

envisioned. This means that care must be taken in choosing a research topic. The project must have a clear focus, but be open enough to allow the creativity that is vital to intellectual development of the student. Particularly projects that include the development of generally applicable methodologies or mathematical techniques are good candidates. In this way the development of the methodology or technique can be openly published, whereas the results of the application of the methodology or technique to the specific industrial research topic can remain proprietary. By framing the research in this way, the practical outcome desired by the industrial partner and the scientific contribution desired by the university can both be accommodated.

With any collaborative research project, the different parties involved do not necessarily share the same objectives. This project is no different; therefore it is important to ensure that the objectives of both the university and the industrial partner are defined and addressed. The academic objectives are usually based on a “process of discovery” where scientific merit is favored over specific results. From the perspective of the university, the ultimate goal is the advancement of scientific knowledge on a research subject. The objectives of the corporation, however, usually have a different focus. In industry, the objectives are usually based on predefined deliverables with economic viability being considered at each step. From the perspective of the corporation, the ultimate goal is a positive return on the research investment. Framing the research project to incorporate the objectives of both the university and

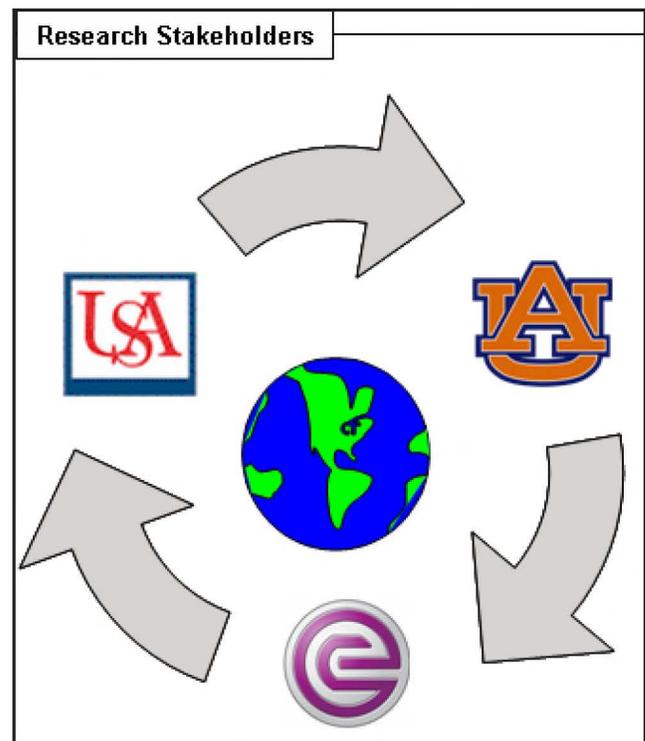


Figure 1. Stakeholders in the proposed research project.

the industrial partner is important for successful collaboration. Developing a clear scope of work with the principle objectives defined improves the likelihood of satisfying the expectations of all the research parties.

ORGANIZATIONAL CHALLENGES

When defining the scope of the research project, several organizational challenges had to be addressed to ensure a successful collaboration. First and foremost is establishing the need for flexibility from the student, the university, and the industrial partner. Research is a fluid process and it is nearly impossible to force the process to fit into a rigid schedule. From the student, flexibility with regard to work and study schedules is needed; from the university, flexibility with regard to residency requirements and in what order the individual requirements of the Ph.D. degree are met is needed; and from the industrial partner, flexibility with regard to work hours and vacation schedules is tremendously beneficial.

Nearly as important as establishing the need for flexibility is establishing channels of communication so no one is left out of the loop. With research partners in two universities and on two continents, good communication is critical. Determining a schedule for update meetings and status reports early in the process can help to ensure that everyone involved is informed regarding the progress of the research. In order to ensure that company goals are met, a representative from Evonik Degussa was included on the research committee.

Another key item considered during the organization of this project was determining where and how this research fits in with current research being done at Auburn University. Since this research was conceived outside the university, organizing funding for the project could have been a challenge. In this case, the research is wholly funded by Evonik Degussa. It may at first glance seem surprising that a company would be willing to fund such an endeavor solely to benefit an employee. Since this research was going to be conducted by Evonik Degussa regardless, however, including it as part of a Ph.D. dissertation adds tremendous value. Since the industry researcher and the student are one and the same in this case, the potential for creative discoveries is greatly improved. With this arrangement, the student is motivated not only by career goals, but by the chance to achieve the highest degree in the field. For others considering a similar path who work for companies without such a well-developed research history, however, grant money may also be a potential source for funding.

Regardless of the source, it is important to accurately forecast the total cost of the project. By spending the time to estimate the total cost over the life of the project, the issue of running out of money before all the academic requirements are met can be avoided. Some lessons learned from the budgeting process of this project are listed in Figure 2.

Finally, since this research must fit in with the normal job

Lessons Learned: Budget

- ◆ Accurately determine the expected duration of the research project. The dissertation will include additional elements beyond the industrial portion of the research, so it is important to include these when estimating the project duration.
- ◆ Decide how university tuition and fees will be paid. Since the research was not conceived at the university, funds for tuition waivers or stipends may not be available.
- ◆ Determine who will be responsible for travel costs for both the student and academic advisor. Additional costs for attending technical conferences and seminars may also need to be included.

Figure 2. Some lessons learned regarding the budgeting process.

function of the student, it is important to organize how hours during the work day will be allocated between the research project and other job functions. This research project has been organized as an Evonik Degussa engineering project with a fixed budget to account for research hours spent during the work week. The original budget was based on allowing approximately 30% of the week to work on the research project. Depending on the phase of the project, however, more or less time was spent as required.

Despite the time allowed during the work week, a significant amount of the student's own time is required. In this regard, the effort is similar to the effort required for a traditional Ph.D. Although the time commitment is significant, a student with several years in industry can draw on this experience with regard to both problem solving and time management in general.

One potential problem is drawing the line between what part of the research is focused on meeting the goals of the company and what part is solely focused on meeting academic requirements. Clearly, activities such as completing coursework requirements are solely academic. Since the development of a Ph.D. candidate requires time to be spent exploring various avenues of research, it can be difficult to determine what work is being done to meet the company goals and what work is being done to meet the additional requirements for academic rigor demanded by the university. Therefore it is important to establish research goals and milestones up front so progress can be easily measured.

In summary, it is important to realize that balancing work, school, and sanity is not always easy. Any student considering this path must have advanced time-management skills. In other words, personal organization is as important as the

Lessons Learned: Conflict

- ◆ Definition of pre-existing intellectual property: It is important to establish what each side already knows with regard to the proposed research.
- ◆ Ownership of any patents that arise from the research: Patent ownership can be a huge source of conflict; negotiating an ownership and royalty agreement up front may help to avoid this potential conflict.
- ◆ Publication of research findings. Since the research may contain business-sensitive information it is important to determine what can and cannot be published and who will make this decision.

Figure 3. Some lessons learned regarding identifying potential sources of conflict.

organization of the project. Without good organization, it is easy to feel overwhelmed by the demands of school and career. Understanding of these demands from supervisors at work and from the academic advisor can help make this burden manageable.

POTENTIAL SOURCES OF CONFLICT

Even with the most well-organized projects, conflict between the partners will arise. To help minimize this potential conflict, it is important to consider where conflict might arise and take preemptive action to avoid it. One primary source of potential conflict is over the ownership of intellectual property generated as a result of the research. In the case of this project, a thorough research contract has been entered into between Evonik Degussa and the two universities. In the process of negotiating these contracts, it became clear that there were two competing interests regarding intellectual property: The university does not want to “give away” its innovations, and the company does not want to pay royalties to use its own technology. Clearly, addressing intellectual property (IP) issues before they become conflicts is wise. In the case of this research project, the scope was divided between academic objectives and industrial objectives to help settle any intellectual property conflicts that may arise. By dividing up the anticipated intellectual property before it is generated, serious conflict can be avoided. In addition, all department faculty members were included in the IP agreement. This allowed frank and open discussions regarding the research without compromising confidential research results.

Another potential source of conflict is the duration of the research project. Because the Ph.D. student must be allowed time to develop creative solutions to problems and explore alternate avenues of research, the pace of the project may be slower than the pace to which industry is accustomed. To

avoid a possible conflict, it is important to outline a schedule up front, including milestones and a general timeline. In the case of this research project, a three-year timeline was proposed to meet the industrial and academic requirements. This timeline was acceptable to Evonik Degussa, and reasonable for a Ph.D. student already holding a master’s degree. Some lessons learned regarding identifying potential sources of conflict are listed in Figure 3.

Conflict is a normal part of business, and this collaborative research project is no exception. Even though conflict can not be avoided entirely, brainstorming to look for possible sources of conflict and addressing them from the onset can help to streamline the process. Although there is no universal right way to address potential conflicts, it is well worth the time to look for and discuss them before any money or time is invested on collaborative research.

PUBLICATIONS AND DISSEMINATION OF RESULTS

A primary part of the academic experience is publication in technical journals and presentation at conferences. As natural as this process is in academia, it is often discouraged in industry for reasons of protecting business-sensitive information. These two conflicting interests can also cause problems for the industrial Ph.D. student. For this research project, the importance of publications and technical presentations was discussed before any work began. The agreement reached allowed publication and presentation of general research findings, while specific data was withheld. This agreement was an acceptable compromise for all sides. As a result of this agreement, the total scholarly output from this research so far includes four peer-reviewed publications and 10 technical presentations at national and international conferences.

COURSEWORK REQUIREMENTS AND RESEARCH PROGRESS

Since the requirements for the Doctor of Philosophy degree include coursework as well as research, being located so far from campus presents challenges for a prospective student. Obviously, driving more than 200 miles just to attend class is not practical. Fortunately in this case, all graduate courses in engineering at Auburn University are available through distance education. The distance education option is really a necessity for a nontraditional student. Another avenue to explore is transfer credit. Since many universities allow at least some transfer credit, it may be possible to take some courses at a nearby university to avoid having to take all classes via distance education. Auburn University allows up to 12 hours of transfer credit to count toward the 30 hours of graded coursework required. In addition to the 30 hours of traditional graded coursework, Auburn University requires a further 30 hours of ungraded coursework as well. This requirement was met by research and dissertation as well as directed study credit.

Lessons Learned: Final Notes

- ◆ Negotiate as much as possible up front.
- ◆ Brainstorm early and often to anticipate potential roadblocks.
- ◆ Secure management commitment up front.
- ◆ Be prepared for surprises along the way!

Figure 4. Some final lessons learned regarding the non-traditional Ph.D. process.

Not only does the geographic distance create challenges for completing coursework, it can also be an obstacle for the normal interaction between a student and advisor that is so critical to the student's development. To help bridge this gap, it is critical that both the student and the advisor be comfortable using long-distance collaboration tools such as e-mail, video conferencing, instant messaging, and Web-based meeting tools. If used effectively, these tools can create an open forum for communication and discussion.

Despite advances in technology, the use of long-distance collaboration tools can never completely take the place of face-to-face conversations. Because of this, regular visits to Auburn by the student and to Evonik Degussa by the advisor are important. Initially, three or four face-to-face meetings were scheduled to discuss project planning, resolve intellectual property issues, and determine the overall direction of the research. Once the research work began, two or three meetings per semester were planned to review the progress with the other research committee members at Auburn University. Meetings were held both at Auburn and on the Evonik Degussa plant site. As the research progressed, fewer face-to-face meetings were required while more extensive use was made of long-distance collaboration tools.

PARTICIPATION IN A RESEARCH GROUP

In order to truly develop as a Ph.D. candidate, participation in a research group is also important. From the academic side, this research is folded into the research group of the academic advisor. This was a tremendous advantage of the partnership with Auburn University. Since the research project fit well with other research already being done at Auburn, there has been ample opportunity to learn from, and contribute to, other research in this area. Because of this it is important to spend time interacting with the other students in the research

group, not just the advisor, during scheduled visits to campus. Interacting with other students can enhance creativity due to an exchange of ideas from others working on similar projects. In addition to campus visits, participation in technical conferences and seminars with other members of the research group is a good way to achieve this interaction.

CONCLUSIONS AND FINAL THOUGHTS

Integrating industrially relevant research topics into an academic setting is an important goal for providing balance to a chemical engineering department. Through collaboration with an industrial partner on an academically interesting and industrially important research topic, this goal can be achieved. Although this project is unique for all parties involved, the results of the collaboration have so far been successful. This experience can serve as a model for other manufacturing companies looking to bring an academic perspective to a research project, and for universities looking to bring an industry focus to chemical engineering education. Although many of the points raised in this contribution may appear obvious when they are put in print, making such a project work in practice is no trivial matter.

In conclusion, this unique and nontraditional research project has been a tremendous learning opportunity for both the student and the advisor. Although sometimes challenging, the success of this project has been the result of hard work, careful planning, and good communication along the way. Based on this experience, some final lessons learned are listed in Figure 4.

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