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Developing Sustainable and Renewable Chemical Engineering Practices through Interning with a Private Chemical Company

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by

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Abstract

The objective of my Honors Thesis Project was to gain knowledge and experience in the means of chemical and bioprocess engineering by experiencing analytical and organic chemistry hands on in a laboratory setting and scaling up to an industrial chemical plant setting. It was determined that through thorough analysis via analytical chemistry techniques and innovation through research and development using organic synthesis, that sustainable practices can be developed and utilized within the chemical and bioprocess industries.

Table of Contents

Preface

I interned at RSA Corporation[1] the summer after my freshman, sophomore and junior years at SUNY-ESF[2]. While there, I was able to experience a little bit of every aspect of the chemical engineering industry. My first summer there, 2009, I studied with emphasis on the managerial and procedural side of the industry. In 2010, I spent the majority of my time studying the quality control and laboratory aspects of chemical processes. In 2011, I got to experience some innovative technologies by working in the research and development laboratory. While there, I studied sustainability and profitability through organic synthesis. Scale up from the laboratory setting, i.e. 2 liter flasks, to the chemical plant or more industrial setting, i.e. 500 gallon reactors, was also studied and executed.

Acknowledgements

To RSA Corporation:

I have been incredibly fortunate to have had the opportunity to work alongside some of the chemical engineering industry's finest for the past three summers at RSA Corporation. Being there, not only did I learn priceless lessons in chemistry and engineering, but also in life. Interning at RSA Corporation, I met many wonderful people that taught me unforgettable life lessons as well as more about who I am as an engineer and as a human being. My time spent there has truly shaped me into the person I am today and has prepared me for work within the chemical and bioprocess engineering industries. I cannot express my gratitude and thankfulness for having been given the opportunity to intern at RSA Corporation after only my freshman year at college, and then invited to come back for two more summers to further enhance my abilities as an engineer and scientist.

The time I spent at RSA will not soon be forgotten, as many of the technical skills I use on a daily basis and the life lessons that I extract from each day were bestowed upon me while studying at RSA.

To SUNY-ESF:

If I could go back to selecting a college senior year in high school, I can honestly say that I wouldn't change a thing. The time that I have spent at SUNY-ESF has been enriching beyond belief, insightful, and inspiring. I have learned more than I ever could have hoped to and have met incredible people, faculty and fellow classmates alike, that have made indelible imprints on my life. From getting an education at SUNY-ESF, I now know what I want to do with the knowledge and power that I have gained from being here. The pursuit of my internship at RSA Corporation and the development of this Honors Thesis were inspired by the desire that I have developed to make the chemical engineering industry more sustainable by adopting processes that work with renewable resources and recycling.

The faculty in The Bioprocess and Paper Engineering Department in Walters Hall has given me the awareness and understanding as to what bioprocess engineering is and how vital it is not only to the further growth of our country, but to the world as well. More sustainable and renewable processing and manufacturing practices need to be developed and the bioprocess and paper engineers will be some of the individuals to do so. SUNY-ESF has taught me not only to *Improve My World*, but to embrace it.

Thesis Project Body

Sustainability is the ability to endure a way of life in which the use of natural resources isn't destructive so that resources can be preserved. Renewable resources are materials that enhance sustainability. Currently, the chemical and chemical industries are fueling and helping to support and sustain the ways of life of the human race. Chemistry provides us with safe food and drinking water, medicine, clothing, our homes, transportation and just about every other aspect of what it means to live. While it is literally impossible to function without innovative chemistry, the chemical industry has been dependent upon petroleum for much too long and is slowly exhausting our finite supply of petroleum and polluting the Earth.

Since even proposing the idea of getting rid of or reducing the role of chemistry in our lives is absurd, something had to be developed: something innovative and different. That is where bioprocess engineering comes in. In the simplest definition, bioprocess engineering is chemical engineering without the petroleum, exactly what we all need to ensure a sustainable future on this planet. The internship that I had at RSA Corporation had such a positive impact on me in the means of developing sustainable chemical engineering processes that it has prompted me to develop it into my Honors Thesis paper.

I began my studies at RSA Corporation in May of 2009, soon after completing my freshman year at SUNY-ESF. I returned to RSA as a Laboratory Technician Intern in both 2010 and 2011. While my work in 2009 was important to building a foundation that would later lead to a more in depth understanding of the chemical engineering industry, the bulk of the chemistry and sustainable engineering that I performed was in 2010 and

2011. My studies, the projects I completed, and the concepts and principles I learned in regard to sustainable chemical engineering are described chronologically below.

2009

I started my time at RSA Corporation and the beginning of my Honors Thesis Project in May 2009. As it was my first summer interning there, I was involved with and studied mainly managerial and procedural aspects of the chemical engineering industry. I also learned a lot about the company itself and the various processes, projects and procedures that it was involved with.

Company History

RSA Corporation is a private chemical company that has been taking orders for and manufacturing organic chemicals and pharmaceuticals since 1935. The manufacturing facility and labs are FDA[(3)] inspected and its operations are conducted under cGMP[(4)] and GLP[(5)] guidelines. RSA takes on and produces many small order chemicals and products that larger companies can't invest the time and money in.

In 1935, this privately held, family owned company started out supplying quantities of a specialty organic chemicals selling to commercial or academic laboratories. Robert S Anthony founded the company in 1935. He and his wife ran the business in the beginning. He was a chemical engineer who graduated from Cooper Union[(6)]. She was an analytical chemist, graduated from Rutgers[(7)]. The original RSA Corporation operated out of leased space in several locations in New York City. In 1942, after a small fire at the NYC site, the company was moved to Ardsley, New York. RSA Corporation operated out of Ardsley from 1942 to 1993.

In 1965, Jan S. Anthony joined the company after receiving his degree in chemical engineering from Cornell University[8] in Ithaca, New York. He completed his post grad work at Columbia University[9] and Cooper Union. RSA Corporation continued to grow. It grew from producing small lab chemicals to larger scale custom synthesis and later, process development.

From the time period of 1950 til 1965, the company operated up to 100 gallon and 200 gallon scale equipment. When Jan joined the company, they maintained the small scale capabilities of the company. When fine chemicals are being produced, as they are at RSA Corporation, the scale of production is limited to decrease possible losses, both monetary and in product. The longevity of RSA Corporation has been maintained by focusing on servicing the needs of clients while keeping in mind their humble beginnings. It was their roots in small projects and orders that ultimately got them to be the developed business that they are today.

During the 1980s, many small customer manufacturing chemical companies were either bought out by larger companies or closed down due to the inability to adapt and change with the changing times. It became increasingly difficult for small companies to comply with the ever growing regulatory requirements placed upon the chemical industry by the Clean Air Act[10], Clean Water Act[11], Occupational Safety Health Association[12], and the Environmental Protection Agency[13]. Larger companies that bought out some of the smaller companies, included companies such as DuPont[14]. The company was founded along the Brandy-Wine River in Delaware during the revolutionary war. Mr.

DuPont made gun powder for the soldiers. The company later grew and was named after its original founder, Mr. DuPont. Many large chemical companies are named after their original founders.

RSA Corporation was able to adapt to the changing conditions and with fewer competing companies, the business continued to grow and prosper. In 1989, the business continued to grow with the purchase of property in Danbury, Connecticut. A 16,000 square foot facility was designed and built specifically for the flexibility and diversity of equipment to accommodate the ever changing needs of custom, fine chemical synthesis and fine product development.

Much of the work currently carried out at RSA Corporation is done for major chemical and pharmaceutical companies under terms of confidentiality agreements. Because of RSA's relatively small size, its clients are secure in knowing that a company with the reputation and size of RSA can keep secrets. In large chemical companies, where staff members and workers come and go, technology often moves with the staff and information can easily be leaked out. One of the more useful points of information that I learned was that in industry, there is a fine line between general information and secrets.

At the present time, RSA Corporation employs 23 staff members. There are 4 chemists, 3 chemical engineers and 3 clerical staff. They balance work in the chemical plant or in the warehouse. Employment at RSA has been stable over several years.

Change within the Chemical Industry: Past, Present and Future

Small companies such as RSA are often bought out by larger companies. Since the beginning of chemical industries, this has been the case. RSA has been able to adapt without changing its core values, to change with the times and thrive.

In past years, simple chemistry has been exported to China and India. With simple chemistry moved out of the country, American chemical companies have been left with the more sophisticated chemistry and chemical processes. In order to carry out these processes, the American chemical companies have had to adapt to be technically competent. They had to develop the analytical abilities and attention to detail so that workers and companies could successfully complete work that isn't readily transferred overseas.

Environmental laws have also continually put stress on chemical companies. Environmental laws, many of which were implemented in the 1990s, have burdened many companies to the point of closure. Ever increasing regulations have aggravated companies. Companies have had to adapt to comply with these regulations yet remain competitive.

Public relations have also changed greatly within the chemical industry. Public relations provide a great deal of information about chemicals that are being used. This made it much more difficult to keep discoveries and developments secret. Negotiations began to take place to ensure certain levels of security. Public relations made it difficult to keep new technologies secret and with the advent of terrorism, it provided vital information access to potential terrorists. Local sites and materials that could be potentially targeted were left exposed. This exposure left many places vulnerable to attack and left a window of opportunity for the disruption of the American economy and way of life.

The public relations movement started with the incident at Bhopal, India[15]. At a government owned chemical plant, a chemical attack was carried out by an angry worker. The worker sabotaged a chemical drum, mixing it with water. This caused a deadly reaction, spewing poisonous gas and fumes everywhere. 3,000 people died because of this attack. As a response to this deadly accident, the “Community Right to Know” Act[16] was put into play. This act allowed the public to have access to information about where chemicals were and what type of chemicals they were. This provided terrorists and potential terrorists with all the information they needed to stage an attack. This caused a huge change in culture across the world. This act, while installed to try and protect the public, led to extending an unintentional invitation to terrorism everywhere.

As far as the environment is concerned, much change has occurred with the chemical industry. The government continually tightens the leash on environmental laws and regulations, often to the point of absurdity. The government itself is at fault for much of the false beliefs and public misunderstandings of the environment. When RSA Corporation was located in Ardsley, New York, there was a chemical spill. The chemical was washed into the nearby Saw Mill River[17] to neutralize the chemical and prevent any type of hazardous reaction. The Environmental Protection Agency (EPA) learned of what happened and placed a fine on RSA. It was then explained to the EPA that washing the chemical into the river was safer than letting it sit on the blacktop and react with the air. Washing the chemical into the river was actually better for the environment. As a matter of fact, the chemical was so harmless that washing the chemical into the Saw Mill River was equivalent to dumping 3,000 gallons of Coca-Cola[18] into the river. The EPA was appreciative of RSA's quick thinking and left them alone on the matter.

When I asked Jan Anthony to share his thoughts on the history and future of petroleum in the chemical industry and in society, he stated that our society undoubtedly runs on petrochemicals. He said that in order for chemistry and society to become more 'green', refineries would have to close and new 'state of the art' plants would have to open to produce organic and bio fuels. He told me an interesting story about how Italy uses wine byproducts to make biofuels. If companies and industries in United States could start a program similar to that, its likelihood of succeeding would be great. Because society depends on the chemical industry, Jan remarked, it is always safe, supplying jobs, and a great aspect of society to invest in. Petroleum or not, chemistry is the foundation of life.

As times are changing and every day is full of uncertainties, I am often curious about where the chemical industry will end up 30 or 40 years from now. I was curious about what Mr. Anthony thought the future held in store for the chemistry industry. When asked what he thought, he responded with many ideas. He felt that product development and high volume and high value products that are being made overseas will be brought back into the United States to keep technologies and discoveries 'secret' and profitable. The quality in overseas production will also be increasingly questioned. Concerns will pop up more readily. With the prediction of increasing concerns and questions, Mr. Anthony predicted that a lot of work will come back to the United States, getting rid of a lot of the uncertainties in overseas production. Mr. Anthony also felt that business will be negatively impacted with Europe due to the new European REACH program[19]. The

REACH program is a new European Community Regulation on chemicals and their usage. REACH focuses on the Registration, Evaluation, Authorization and Restriction of CHemical substances, hence REACH. While this program was enabled to help the chemical industry and make it safer, it has so far done nothing more than inconvenience companies such as RSA. It has become increasingly difficult to get new products into Europe. The regulation went into effect in 2008 and has been issuing strict rules ever since. There is a strong fear that as more and more rules and regulations are issued, that many companies in the 'emerging world', such as China and India, won't comply with the rules and cause regulatory issues.

Products

RSA produces a line of many traditional chemical products, but is known for its production of specialty chemicals. RSA will often get a phone call from a company, perhaps a food manufacturer, asking if they could produce a new, exquisite flavor for a new food product. Individual, specialty requests such as that is what RSA specializes in. Since much of the specialty work is highly confidential, many of the specialty products cannot be listed. A list of products can be found on their webpage at www.rsa-corporation.com.

Projects

RSA has completed and worked on hundreds of projects throughout their years as a chemical company. A variety of projects include the synthesis and discovery of new chemicals and chemical properties. One project was in creating an adhesion promoter that would attach the bottoms on sneakers. Another was in creating a polymer that could be used to coat medical gloves. RSA has been involved in many impressive projects, some historical. The following timeline of projects gives a good idea of the projects that RSA has been involved with throughout the years.

In the 1970s, RSA Corporation worked with Exxon Mobil[20] in the production of rechargeable batteries and battery fluid that could be used in a battery run car. In 2000, RSA Corporation got a call from a large scale rechargeable battery company. They were looking for the same chemicals and rechargeable battery technology as Exxon had in the 1970s. A new company had actually bought the rechargeable battery technology from Exxon and wanted to re-spark the technology and chemistry.

In the late 1950s, early 1960s, RSA Corporation took on a project to produce penicillinase[21], the anti-dote to penicillin[22]. A special 500 gallon system was set up to produce millions of units of penicillinase. RSA Corporation was able to isolate the strain to grow penicillinase on the 500 gallon scale. The production of penicillinase was such a success, that the technology was sold to a drug company to set up the product for commercial production.

In the 1960s, RSA Corporation developed the process to isolate L-dopa[23], or Leva-dihydroxy phenalenine, from beans. L-dopa was found to have properties to treat Parkinson's Disease[24]. Before the synthetic synthesis of L-dopa was developed, RSA found it naturally. It was discovered that L-dopa was naturally occurring in African velvet beans, or vicia faba. These beans were about the size of jelly beans. RSA Corp. bought truckloads of the African bean. The beans arrived at RSA in burlap sacks and the experimentation then began. RSA was able to extract, isolate and purify the naturally occurring L-dopa. This material was then used for studies to prove the great potential of L-dopa in treating Parkinson's Disease. Synthetic L-dopa is now produced by DOW[25] pharmaceutical company. The synthetic process was developed shortly after RSA developed the natural process and had to be produced synthetically because the natural process wasn't practical. It took thousands and thousands of African velvet beans to produce just a small amount of L-dopa.

Another natural product that RSA worked with was L-Rhamnose[26]. L-Rhamnose is isolated from rutin, which is extracted from Australian eucalyptus. Rhamnose is a sugar, which occurs in free form in poison sumac, and has the property of being metabolized by only certain organisms. Humans don't metabolize Rhamnose. Rhamnose has the same degree of sweetness as regular table sugar. If Rhamnose could be made at sufficient low cost, it would be the ideal dietetic sweetener, being natural, with the same sweetness as traditional sugar, and having zero calories. The biggest problem with Rhamnose, however, was that it wasn't widely occurring enough to be economical or practical.

At the present, much of the work carried out by RSA is highly confidential, specializing in process development for pharmaceutical intermediates. Many of these materials are high value, added materials that are produced on a relatively small scale, approx. 50 to 100 kilos, but costing multi-thousands of dollars per kilo. On the other end of the spectrum, RSA produces close to one million pounds per year of inexpensive foam stabilizers used principally in firefighting applications. Many projects are 'one time' synthesis projects, where quantities vary from a few kilos to a few thousand pounds. Over the years, RSA has produced more than 3,000 different products and has developed quite a broad range of technologies.

The most commonly produced chemicals at RSA are quaternary ammonium compounds.

One of the more interesting projects that was carried out at RSA was in developing an aqueous based tablet coating. Tablet coatings used to be solvent based in open coating pans. The coating was dissolved in organic solvent, applied to the tablet and dried. While this process worked, it had many negatives. The drying process that was used released solvent, and therefore wasted money, and contributed to air pollution. A professor at Purdue University[27] came up with an idea for water based coatings in the form of latex. This was similar to latex paints. The idea went to RSA and became a reality.

Another interesting project carried out at RSA was in producing the flavoring that went into Campbell's[28] campfire pork and beans. It was a fairly simple process that was carried out for months and months. However, in the final months of production, the flavoring increased the cost of a can of the product by a quarter of a cent. Due to this price increase, Campbell's decided to discontinue the product.

One product that was produced truly shows RSA's connection to the bigger picture and the world. They developed and produced the lubricant that oils the gears and motors in self-guided missiles. The production of the lubricant was tricky, because the lubricant had to be able to withstand intense heat and conditions, but only for a period of about ten to fifteen minutes. Once the product was developed and produced successfully, the product and its technology were sold to a private company and contracted with the government.

Customers

Because RSA Corporation is such a unique company, they have a wide range of unique customers. Large manufacturers, small companies, and independent businesses all go to RSA for their chemical needs. Many of the Fortune 500[(29)] chemical companies, including Pfizer[(30)] and DuPont, use RSA as a chemical supplier. Sigma Aldrich[(31)], a combination manufacturer and distributer, is a bigger chemical company located in Milwaukee. The company buys chemicals from RSA and sells them all over the world. Proctor and Gamble[(32)] is another customer that purchases chemicals from RSA to be used in their paper and hygiene products. The products that they produce and sell include organic chemicals, reagents and active pharmaceutical ingredients. They also do business with the FFI[(33)], Food and Fragrance Industry, producing flavors and fragrances.

RSA does business both nationally and internationally. About 80% of its business is domestic and 20% of its business is international. As far as international business is concerned, RSA does business with China, Spain, and a large majority of its international business with Central America, South America and the Caribbean. The reason for this is because most other chemical companies won’t do business with those parts of the world. Often times, companies in those parts of the world will not follow through with monetary transactions, delivery, or proper communication. RSA, however, has developed strong, trustworthy relationships with companies in that part of the world. Often times, RSA will have products delivered to Miami or Texas and then shipped south. This way, the shipping and deliveries stay within the United States, and the company that is receiving the product needs to arrange for transport from the United States location to its final destination. Also, no product is shipped before payment is made. That way, there are no monetary discrepancies.

Plant Site Processes

The current facility is located in an industrial facility in Danbury, Connecticut. The facility is on 6 acres of land and consists of two buildings. One building consists of offices, the warehouse, chemistry labs and 2500 square feet of lab space. The second building is the plant, which contains approximately 20 reactors ranging in size from 30 gallons to 2,000 gallons. Most equipment is glass lined with variable speed agitation and heating/cooling capabilities. Each reactor is equipped with overhead condensers for distillation or reflux. The heat exchangers are made of corrosion resistant materials which include stainless steel, hastelloy, tantalum and glass. The various combinations of equipment and accessories enable the company to handle a wide variety of corrosive and reactive materials.

The plant has three levels. The top level provides access to the nozzles, controls, instrumentation and man ways on the reactors. The middle level provides access to the bottom of reactors and serves as a service floor where much of the material is brought to the equipment to be used. The lower level has other processing equipment, such as centrifuges for isolating solid materials and dryers for product drying and finishing.

The plant operates in a process flow known as batch processing. In batch processing, the product being made moves in stages. The other form of processing, known as continuous processing, produces product in a steady stream. The type of fine, chemical processing that RSA does requires batch processing. It is the most logical processing to use with fine, small scale processing.

In the plant, many processes take place. Unit processes include dry and liquid blending, centrifugation, crystallization, atmospheric distillation, vacuum to 10 microns, air and vacuum drying, filtration, fractional distillation (72 L. with 4" x 4' packed column), hydrogenation (low pressure (<500 psig)), evaporation and high pressure (to 800 psig, normal operating temperature range: 0°C-250°C), normal operating pressure range: 10 microns to 800 psi.

Along with the unit processes, many reactions also take place in the plant. Reactions include the following:

Acetylation
Acid / Base Reactions
Acid Chlorides
Acylation
Aldol Condensation
Alkoxylation
Alkylation
Amidation
Amination
Ammonolysis
Arylation
Benzoylation
Birch Reduction
Bromination
Bischler-Napieralski
Cannizzaro Reaction
Carboxylation
Chloromethylation
Claisen Condensation
Condensation
Cyanation
Cyclization
Dealkylation
Decarboxylation
Dehalogenation
Dehydration
Dehydrohalogenation
Diazotization
Dieckmann Cyclization
Diels-Alder
Dimethyl Sulfate Reactions
Distillation
Doebner Knoevenagel
Epichlorohydrin Chemistry
Epoxidation
Esterification
Etherification
Ethoxylation
Ethylation
Fenton Reaction

Fermentation
Friedel Crafts
Gabriel Synthesis
Grignard
Halide Exchange
Halogenation
Hofmann Degradation
Hydrogenation
Hydrohalogenation
Hydrolysis
Hydroxylation
Imidization
lodination
Knoevenagel Kolbe
Condensation
Liquid Ammonia
Methylation
Michael Addition
Multistep synthesis
N-alkylation
Neutralization
Nitration
O-alkylation
Oxidation
Oximation
Phosgenation
Polymerization
Propoxylation
Quaternization
Reduction, catalytic
Reduction, chemical
Reformatsky Reaction
Rosenmund Reaction
Ring Closure
Sandmeyer Reaction
Saponification
Substitution
Tosylation
Transesterification
Williamson Synthesis
Wittig Reaction

When I toured the plant, I was impressed by not only the layout, but also the organization. When I think of a chemical plant, I imagine an old building that is intimidating and disorganized. The chemical plant at RSA couldn’t have been further from that. I was immediately impressed by the fact that Jan Anthony had designed the plant. It was interesting to see the three different levels of the plant, with certain parts of different reactors poking through each level. The layout of the plant was key for optimal chemical production, having all necessary production equipment and allowing enough room in the plant for potential additions.

Raw Materials Used

The raw materials used at RSA consist of a wide variety of commercial, organic chemicals. Most reactions are run in solvents, such as methanol, acetone, toluene and heptane. Acids and bases, such as sulfuric acid, hydrochloric acid and sodium and potassium hydroxide are also used. There is quite a long list of reactive chemicals that are used to make the various products at RSA. The materials are handled in drum quantities. Materials generally aren’t handled in bulk quantities. Since RSA’s primary business is in custom manufacturing, the list of processes would look like the table of contents in an organic chemistry textbook.

Departments

RSA is a small, privately owned and operated company. It is organized into five main areas: administrative, manufacturing area, process development lab, quality control lab, and the warehouse. I worked primarily in the administrative portion of the business while being introduced to the various aspects and projects carried out in the other departments.

The administrative department does everything from the pricing and purchase of raw materials, documenting of shipments, keeping track of accounts payable and accounts receivable, and the preparation of administrative and safety documents and programs. All external contact begins and ends in the administrative office. Inquiries are then passed on throughout the company. The administration of the safety information is in support of both the evaluation of new projects and the safety of operations on projects brought into the facility. All communication and record keeping aspects of the business occur in the administration.

The quality control/analytical labs take care of mandating basic quality. This includes the testing of raw materials, along with associated record keeping. The labs also carry out in process checks which help the plant personnel keep track of and see, in the most timely fashion available, the progress of the chemistry that is occurring. The lab tests, documents and approves quality before any product is shipped. The analytical lab supports all aspects of what occurs in the plant and provides the plant personnel with the information that is needed to assure that the processes go smoothly and produce the desired final product of the proper quality. The quality control labs are another part of the support function, supporting production in the plant and making sure that the chemistry is of a certain quality.

The development labs perform a wide range of tasks. Before anything is run in the plant, it is run through the lab to give hands on experience to verify certain process parameters and to identify potential processing problems. Often, difficulties that could occur in completing a process are discovered in the small scale lab runs. In this way, many problems can be solved at a small scale to avoid potential health, safety, and economic issues.

Another function of the development labs is to solve unanticipated problems that do occur in the plant. While I was spending time at RSA this summer, a process was run successfully in the lab with a 43 hour reaction time. When the same process was run on a larger scale in the plant, at the 43 hour mark, the reaction had only proceeded 20%. Samples were taken from the batch and brought to the lab. After a few days work, it was determined that the particle size of one of the reactants was too large. Since the reaction was heterogeneous, the particle size of the key reactant, a solid, was reduced by micro-pulverizing, producing particles of about 10 microns. The reaction was then continued and completed successfully.

Another area that the development labs cover is in discovery. The lab technicians make discoveries through experimentation and find new and more efficient ways to make new molecules desired by customers.

In the plant, which is staffed by three chemical engineers, one chemist, and ten chemical operators, the actual chemical processes are carried out. A broad range of operations, whatever is needed for a particular synthesis, such as distillation, extraction, filtration, centrifugation, drying or grinding, are used. All of these processes support whatever form of chemistry is carried out in the reactors. Reactors range in size from 30 gallons to 2,000 gallons. Depending on the complexity of the synthesis, a process could be completed from as little as one day to as long as three months for a more sophisticated, multi-step preparation. A typical process runs three to five days. Products of a sophisticated nature might be produced in the ten to fifteen kilo range with pricing from $3,000 to $5,000 per kilo. The company produces two or three larger scale products in the range of 5,000 to 50,000 kilos, but priced in the $10 to $20 per kg range. The plant provides the bulk of income for the company.

The warehouse stores the products that are produced in the plant. It is run in support of the manufacturing operation, dealing with and keeping track of raw materials and finished products/projects. The warehouse has a staff of two material handlers.

Economic Aspects of the Chemical Industry

With all startup companies, in the early years, profits fluctuated greatly. Some years, business was increasingly profitable, while in other years, the founders had to supply capital to maintain the economic vitality of the company. The first major change occurred in 1953. When the company was in a position to provide a worldwide supply of piperazine citrate[34], used as a material to fight parasites in cattle, for a period of about a year, the company jumped at the opportunity and operated round the clock, producing little more than the piperazine citrate. By doing this, the company produced substantial excess profits. As good as this situation was, it also had significant negatives. On one hand, the company was able to eliminate all debt and purchase new and larger equipment to help them grow and prosper. On the other hand, in meeting this demand for a single product, other business opportunities were lost and when the piperazine citrate business came to an end, there was a dramatic drop off in sales.

RSA Corporation took this experience and learned from it. The company learned that if you put all of your eggs in one basket, so to speak, that sooner than later, the bottom of the basket will fall out. It took RSA almost three years to recover from the piperazine citrate project and get back on a normal business schedule.

Since the piperazine citrate incident, the company has grown from a handful of employees to its present level of 23 employees. The growth of the company has been limited, in a good way, by the conservative philosophy of the owners.

The basic philosophy of RSA and its owners is that ‘our success is based on quality, attention to detail, and responsiveness.’ If the company got too big, these core principles could get easily lost and would be sacrificed. Over the years, by sticking to these principles, RSA has enjoyed steady growth and has been able to weather the bumps in the road along the journey.

RSA's core principles have helped them become and stay as successful as they are today. The company believes in being open to change, trying to predict the future, or predict which potential customers and projects will be profitable, and also, and maybe most importantly, never forgetting where the roots of the business began. Since RSA began a small company and is still a company it is often vital for its members and staff to remember this.

The second major change in RSA Corporation occurred in the early 1990s. The company relocated to Danbury, CT. At the time of the transition, the company went from 12 to 13 employees and about three million dollars in sales to 23 employees and more than double the sales volume. The company has enjoyed excellent profitability since its move to Danbury, CT and expects this to continue into the future.

Work Performed

By completing various projects throughout my internship at RSA Corporation, I learned many new aspects about chemicals and chemical properties. Each project that I was involved in covered a different aspect or learning experience from the company. I learned about all aspects of the business, ranging from the certain processes that are used to create and classify chemicals, to the procedures that the administrative offices use to ensure quality company performance.

The biggest project that I was involved in at RSA Corporation was in updating Material Safety Data Sheets (MSDS reports)[35] and converting them into the new, European format. The MSDS reports were updated using various chemical information databases, such as Sigma

Aldrich at (www.sial.com), Acros Organics[36] at (www.acros.com), TSCA[37] at (http://www.epa.gov/lawsregs/laws/tsca.html) and EISC[38] at (http://ecb.jrc.ec.europa.eu/esis/).

Sigma Aldrich is a chemical company that buys and sells products all over the world. One of the companies that they buy their more specialized products from is RSA Corporation. Sigma Aldrich buys the product and then sells it to customers. Because they purchase product from RSA, Sigma Aldrich's chemical database contains much information about the products that RSA produces. This makes it very easy to update the MSDS reports. Acros Organics is another chemical company that supplies an online chemical database. While Acros doesn't list as much information as Sigma Aldrich does, it still is very helpful in obtaining information on certain chemicals. TSCA stands for Toxic Substances Control Act and is another online chemical database that provides users with important information on chemical listings. TSCA will show a user what 'lists' certain chemicals are on and what regulations and rules must be followed.

ESIS stands for European chemical Substances Information System and offers some insight into how European countries group and classify their chemicals. It lists information on chemicals related to EINECS[39] (European Inventory of Existing Commercial chemical Substances), ELINCS[40] (European List of Notified Chemical Substances), NLP[41] (No-Longer Polymers), BPD[42] (Biocidal Products Directive), PBT[43] (Persistent, Bioaccumulative, and Toxic) or vPvB[44] (very Persistent and very Bioaccumulative), C&L[45] (Classification and Labeling), Export and Import of Dangerous Chemicals[46], HPVCs[47] (High Production Volume Chemicals) and LPVCs[48] (Low Production Volume Chemicals), including EU Producers/Importers lists[49], IUCLID Chemical Data Sheets[50], IUCLID Export Files[51], OECD-IUCLID Export Files[52], EUSES Export Files[53], Priority Lists[54], and Risk Assessment[55] process and tracking system in relation to Council Regulation[56]. While this information is more relevant to Europe, the fact that MSDS reports are now in the European format is making the above listed information more relevant to what is happening here in the United States.

Chemicals and chemical products can be easily searched online by using a CAS # [57]. Each chemical has a unique CAS, or Chemical Abstracts Service number. Searching by a chemical or product's CAS number is like searching by its ID number. Products also have EINECS numbers, which is the European version of the CAS system, and stands for European INventory of Existing Commercial chemical Substances (EINECS). RSA also gives its products their own unique RSA company code. With three unique codes given to each chemical and each chemical product, it is easy to distinguish one chemical from the other.

Once each MSDS report was updated, using one of the above described methods, the MSDS was converted into the new, European format. The European format is more widely used and accepted by companies and is much more user friendly and organized. The new formats are also more detailed and provide a wider range of vital information to the user. The European format for MSDS reports is longer, but also is more thorough and could provide vital information in the event of an emergency.

Updating and converting MSDS reports exposed me to a lot of information on various types of chemicals, ranging from harmless food flavorings to toxic benzene. I learned how certain chemicals, depending on their properties, should be handled and stored and how the chemical should be treated in an emergency situation. Reading the MSDS report for a chemical is probably the best way to learn the most about any chemical and the properties it possesses. Please see the appendix for some sample MSDS reports.

I worked with Stephanie Weber on a project that included reviewing the internal audit system within RSA Corporation. Every year, the company completes an internal audit to ensure quality and that the company isn't lacking in certain departments. I think this is a great system that keeps the company on track and open to suggestions by its peers. My involvement in the internal audit project was in reviewing what had been done in the past and making suggestions for what should be done in the future.

I reviewed past reports that had been written up by other companies as well as RSA. Using the other company's audits, I made suggestions as to what RSA should add to their audit check list, questions section, and follow up reports to make the audit more thorough. I then went through past RSA audits and made comments about items such as the questions being asked and the organization of the reports. Once the RSA audits were reviewed, I typed up a list, suggesting new questions to ask and new sections to add to the audit itself. I also made suggestions as to which departments should audit other departments. It's more efficient and thorough to have a department that knows very little about another department, say the lab and the plant, audit the other.

I must say that I was very impressed by the fact that RSA chooses to conduct internal audits. They are not mandatory by industry. The fact that they conduct internal audits shows that RSA is responsible and truly interested in striving for the best that their company can produce. By conducting company run internal audits, the staff members also learn and understand more thoroughly what is going on in other parts of the company. Someone in the office who is auditing the warehouse would learn about what is happening in the warehouse and how certain procedures are carried out. The more everyone knows about the company, the greater the level of understanding is amongst workers. Please see the appendix for the audit reflection.

PHA reports[(58)], or Processing Hazards Analysis reports, were reviewed and completed with the help of Brad Andrews, one of the chemical engineers stationed in the plant. PHA reports list the components of a chemical product, their individual hazards, the hazards of the chemical process, dangers to the environment, health hazards, and ‘if’ questions that tell the operator what to do in case of a mechanical failure or emergency. These reports are vital and must be included with each chemical product.

In learning about PHA reports, I reviewed several reports for various chemical products and processes. After I had a good understanding of how to interpret the chemical processes and read the reports themselves, I wrote and revised a few reports. These reports were reviewed by Brad and then filed in the corresponding chemical folder. Please see the appendix for sample PHAs.

I also worked with Brad on developing an efficient way in which to receive and access information and regulatory updates. This way, the company would be aware when new and updated rules and regulations were issued. By reading through information on chemical companies, chemical databases, government rules, regulations and organizations and by browsing the internet, I was able to put together a resources list. The list is composed of various websites and links that offer information on updates and information on the chemical industry.

Another project that I was involved in included looking at hazardous materials and waste. I helped classify and identify hazardous materials and wastes with help from Chad, the other chemical engineer stationed in the plant. My role was in identifying chemicals based off the chemical name, identifying the CAS #, and then identifying the certain hazards of that chemical. Depending on how hazardous the chemical was depended on how the material was disposed of.

2010

This was my second summer at RSA Corporation, continuing my Honors Thesis Project and developing it to involve more chemistry and sustainability. I spent the majority of my time studying the quality control and laboratory aspects of chemical processes.

Daily Activities

My time spent at RSA Corporation in 2010 was an 8am to 5pm, Monday through Friday commitment. Daily responsibilities and duties performed included calibration of the analytical balances, Karl Fischer water content analysis, GC analysis (Gas Chromatography), HPLC analysis (High Performance Liquid Chromatography), AA analysis (Atomic Absorbance), assays via HCl, NaOH and $AgNO_3$ titrations, halide determination, density analysis, pH analysis, refractivity, percent solids analysis, weight loss on drying, melting point determination, and standardization of solutions.

The daily calibration of the analytical balances in the quality control laboratory was completed first thing at 8 am every morning. It is considered an SOP[(59)], or Standard Operating Procedure, to maintain the balances daily by calibrating them to 0.0000 decimal points and cleaning them as well. 50 gram, 1 gram and 200 milligram weights were used to calibrate each of two analytical balances.

Karl Fischer[(60)] water content analysis is an important analytical evaluation that measures the amount of water in a wet or dry sample. As products were made in the chemical plant, samples, both solid and liquid, would be brought over and tested for water content. During the summer in particular, it was difficult to test hygroscopic solid materials for the moisture content accurately. Hygroscopic materials are sensitive to the moisture in the air and quickly liquefy at room temperature because they absorb the moisture from the air.

A special procedure had to be developed in order to accurately measure the moisture content of hygroscopic materials as to not waste products. Hygroscopic materials were dried at approximately 60 degrees Celsius for about an hour before being used for tests such as the Karl Fischer analysis and the melting point test. The precautionary procedure worked most of the time resulting in data that allowed the product to fall within specification.

Another procedure that was utilized on a daily basis was GC analysis. GC stands for gas chromatography and is used to check a product for particular components. Liquid samples would be brought over from the chemical plant and 2 to 4 micro liter samples would be injected onto the gas chromatography column. Traveling through the column, with assistance from temperature and pressure programming, the components that make up each product are separated on the column. A chromatogram is then produced to show the resultant residence times and heights and areas of each peak. Waste water samples from waste streams were tested weekly via this method.

AA analysis, or atomic absorbance, was less commonly used in the analytical laboratory. This method determines the presence and concentrations of certain elements. AA analysis was used to analyze particular liquid samples. A blank containing distilled water was always used to clear or 'level' the wavelength measurement.

The most common day to day activity was in performing assays via HCl, NaOH and AgNO3 titrations. HCl titrations were performed in concentrations of 1.0 N and 0.1 N. NaOH titrations were also performed in concentrations of 1.0 N and 0.1 N. The most commonly performed titrations were using 1.0 N AgNO3, or silver nitrate titrations. These titrations were used to assay products that were halide based and also to test for the presence of halides in products that were supposed to be halide free. The silver nitrate titrations caused the product being titrated against to turn yellow and precipitate out a salt.

As far as calculating the assays were concerned, the NaOH and HCl titrations utilized color changes that were made visible using indicators such as methyl red and phenolphthalein. The AgNO3 titrations were a bit more complicated in that millivolt and silver nitrate probes had to be used to indicate when the endpoint for the titration had been reached. Also, the sample was dissolved in nitric acid for the AgNO3 titrations, were the sample was simply dissolved in distilled water for the HCl and NaOH titrations.

Halide determination was particularly important in the analytical lab, as many products would fail to meet their specifications if halides were found to be present. Halides that were commonly found included chlorides, bromides and iodides. If the concentration of such elements were found to be too high, the product had to go back to the plant for further purification.

Density analysis was frequently done on liquid products to identify the presence of unwanted components, especially salts and solid precipitates. Density testing was very simple, consisting of blanking a 10 mL volumetric flask on the analytical balance and then filling the flask to the etched line indicating 10 mL on the flask. The weight of the product was then recorded and divided by the volume, 10 mL, to produce the respective density. If the density was high, then there were components in the product that still needed to be filtered or separated out via purification techniques. Rarely was the density lower than expected. On occasion when the density was lower than expected, it indicated that the reaction utilized to create the product hadn’t gone to completion and that the uncompleted product had to be put back into the reactor.

Along with calibrating the analytical balances every morning, the pH probe was also calibrated daily. This was essential because the pH was taken on every sample that was brought over. This testing is important because pH plays an important role in the characteristics of a product. Solutions of pH 4, 7 and 10 were used to calibrate the pH probe. The pH probe was also flushed and refilled with filling solution once a week.

On certain liquids, refractivity was measured. Refraction is the bending of any wave, such as a light, as it passes from one medium into another of varying optical density. It impacts the dispersion and absorption properties of the material and affects how it will interact with other materials.

Two final procedures that were frequently used in the lab were melting point determination standardization of solutions. Testing the melting point of solids revealed how pure the material was. If the observed melting point was the same as the accepted melting point value, than the substance was said to be pure according to that method. If the observed melting point was lower than that of the accepted melting point, then impurities were present. Impurities damage the internal structure of the product, making it weak and therefore lowering the melting point. The more the observed melting point deviates from the accepted value, the more impurities that there are present.

The standardization of solutions was vital to the integrity of the data that was output by the lab. Solutions that required standardization included 1.0 N NaOH, 0.1 N NaOH, 1.0 N HCl, and 0.1 N HCl. The exact normalities of these titrants had to be known in order to properly calculate the assays that were performed via titration. Standards had to be re-standardized monthly.

2011

This was my third and final summer at RSA Corporation, allowing me to finish the third and final and most important component of my Honors Thesis Project. The summer of 2011 was when all of the background knowledge and technique that I had learned from the previous two summers got put to the test. I conducted three research and development projects that tested the limits of sustainability within chemical engineering.

The first project that I conducted involved the recycling of a waste stream from the production of a material to extract a desired compound to then be reused to produce yet another product that could be sold for profit. This was a multi-faceted project, as there were many different chemicals that were tested to see which material yielded the highest purity and greatest quantity of product. Because this research and development project was done through a private company, details of chemicals and processes used cannot be disclosed. The particular chemical that was used in the reaction and purification of the chemical waste stream, however, was simply manipulated to see what chemical would be ideal for the recycling process.

The overall outcome of the recycling of the waste stream turned out to be very successful. The ideal chemical compound to be used to react, isolate, purify and ultimately recycle the desired material was identified. Once this chemical compound was identified, it was used on various chemical waste streams, ranging from highly contaminated to barely contaminated at all. The usability of the chemical was identified by testing its ability to isolate, purify and recycle the desired material in the waste streams that varied in concentration of contamination. It turned out that the chemical was able to successfully isolate, purify and ultimately recycle, both quantitatively and qualitatively, the desired product. The more contaminated waste streams, however, did take a longer time to process and required a higher temperature. This is most likely due to the fact that unwanted, foreign 'bodies' were attached to the desired material. It took a higher temperature at a longer, sustained period of time to eliminate the excess of contamination.

By the end of the summer, this project had left me in the organic research and development laboratory and found its way to the chemical plant. There, the chemical engineers scaled up the entire recycling procedure so that the process could be carried out large scale in the reactors. The chemical that I had identified as the optimal component for isolation and recycling was continually used throughout the rest of the summer to recycle the chemical waste streams to isolate and purify the desired material. This material, at zero cost because it comes from a waste stream, was then marketed and sold for profit, adding to the economic growth of the company for the summer. This type of sustainability through recycling is vital to the chemical industry because it provides for a greater economic turnover and overall income for the company.

The second project involved the production of a fluorosurfactant lacking halides. A company sent this in as a special request to RSA Corporation. The fluorosurfactant would be incorporated into a coating that would be applied to household furniture. It is important for fluorosurfactants to be free of halides because halides are highly flammable. Having a flammable material in a fluorosurfactant coating is completely counterproductive and weakens the integrity of the coating. Again, due to copyright and secrecy agreements, the details of the material produced cannot be discussed.

There were multiple steps to completing this project. The two main steps were to determine the percentage of halide present in the material and then to further purify the material until little to no halide remained. The purification process consisted of combining the material with water and another chemical to act as a catalyst to react and bind with the halides, causing them to fall out of solution. This method effectively lowered the halide concentration until virtually no halides existed within the coating material. The halide concentration was determined by diluting a 7 gram sample of the material in 100 ml of dilute nitric acid and then titrating the solution with silver nitrate until the endpoint was reached. There is no color indicator involved in this titration, so a millivolt meter was used to indicate when the endpoint had been reached. The millivolt value of 250 mV was used as the endpoint value. Once this point had been reached, the solution as titrated at least 100 mVs higher to be sure that the titration reaction had gone to completion.

Overall, the second project was very successful. It taught me valuable experience in analyzing material, collecting data and then from that data, determining what needs to be done to achieve the final desired product. I also learned a many useful calculations during this project that aided in the prediction of how much water should be added to the material and how much water needed to be stripped off to generate a certain percentage concentration of material with minimal halides. I also learned how to troubleshoot with this project. Sometimes a certain step, such as adding X amount of water and distilling over for X minutes, didn’t produce the logical, calculated result. Looking at chemical processes in both a theoretical and actual aspect is important. I also found it to be important to seek after and understand why certain processes didn’t produce certain results. The more that can be understood about a process, the better.

The third project that I was involved in was in reacting and purifying two chemicals to reach a particular percentage value for each chemical present in the final product; very similar to the second project. Due to copyright and secrecy agreements, the details of the material produced cannot be discussed. This project, of the three, was the most simple. I used the technique of reacting, purifying and analyzing the material via titration to determine the actual percent concentrations of the two main chemicals in the material. The titration that was performed for this project, however, was different from the one that was performed in the second project. This titration was via 1.0 N HCl, as the material was basic. For the HCl titration, the endpoint is reached when there is a color change from yellow to red. Methyl red was used as the indicator.

The reaction and purification was process was almost identical to the process used in the second project. A simple distillation set up was used and the material that was distilled off was tested via 1.0 N HCl titration for the percentage concentration of the two target chemicals. Once the actual percent concentrations were calculated, water was added to the round bottom flask holding the material in the distillation set up. The water was then distilled off to a certain indicated volume, which was indicated via calculation. Adding water to the material allowed for adjustment in the percent concentrations of the chemicals present, providing for the achievement of the ideal percent concentrations in the final product.

This project also resulted with great success. After a bit of trial and error and much calculation, the target concentrations of each chemical in the solution were reached. This project helped to fine tune my skills in operating the distillation column and in calculating projected steps to take to reach a final goal. The biggest lesson that I learned in completing this project is that it is important to be persistent and to be diligent in calculations and sticking to them. If a certain amount of water had to be distilled off of the solution and too much came over, I'd have to start over again. Patience and being observant were vital.

After spending three summers at RSA Corporation and experiencing work in many of the various aspects of a chemical engineering company, I learned valuable lessons in the art of developing chemical engineering to make it sustainable and renewable. Just because chemical engineering is based in petroleum doesn’t mean that chemical processing can’t be made more sustainable, or that waste streams and byproducts can be recycled to produce material that can be reused or sold for profit. The ultimate revolution and future of the chemical industry lies within bioprocess engineering. Once bioprocess engineering replaces chemical engineering, chemical processing as we know it will have been made as sustainable as possible. In the meantime, a bit of critical thinking, an observant eye and a calculation or two of economic cost analysis, as I learned while at RSA, makes for a more sustainable and renewable chemical industry.

Sources Cited and Consulted

As all of the content in this thesis is based on personal experience, the only reference that I have is to RSA Corporation. The business can be most easily accesses and viewed through the below citation.

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Appendices

Please see Dr. Shijie Liu in 302 Walters Hall for my laboratory and experience archives from summer 2009, summer 2010 and summer 2011. Also, RSA Corporation can be contacted directly to retrieve data and information from my laboratory notebooks that are kept at RSA Corporation for copyright and secrecy agreements.

Plant Site Map

A: Offices

B: Laboratories

C: Chemical Warehouse

D: Chemical Plant