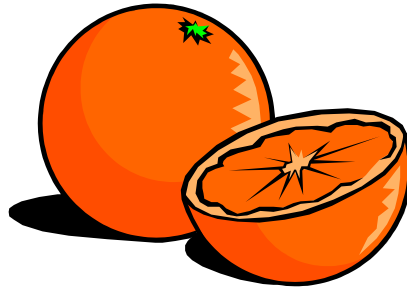


Polymers from Oranges:

Design and Feasibility of Polymer Production from Orange Oil Derivatives



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Executive Summary

This project presents the design and economic feasibility of a plant to produce a novel polymer made from orange oil derivatives and carbon dioxide. This new technology is desirable because of its use of carbon dioxide that would normally be emitted into the atmosphere and its use of renewable resources that reduce dependence on foreign oil. The polymer produced has properties similar to polystyrene, but the exact properties have not yet been determined experimentally. The feed products for the process are orange oil, tert-butyl hydroperoxide, and carbon dioxide. Carbon dioxide is a cheap feedstock because of its availability from power plants that wish to reduce emissions. The products of the process are the polymer polylimonene carbonate and tert-butyl alcohol. The process involves two main sub-processes, the production of limonene oxide from the orange oil and the production of the polymer from limonene oxide. Because of the large dependence of the process on the orange oil supply, this process should be approached as an addition to an orange processing plant in Florida. Florida also offers access to a carbon dioxide supplier and a styrofoam processing plant, making it an ideal location for the plant. A pilot plant should be constructed to insure the reliability of the product and to confirm reaction rates, which are based on limited lab-scale experiments using 30mL reactors. The equipment cost for the process was approximately \$1 million, resulting in a total capital investment estimate of approximately \$7.3 million. The product price necessary to achieve a ten percent return on investment is approximately \$1.18/lb. This corresponds to a Net Present Worth of \$1.1 million. This product price is not an unreasonable, but it is significantly higher than the current polystyrene price of \$0.90/lb. The largest risk to the process is the orange oil supply, which can have large fluctuations after natural disasters affecting the orange crop. Because of orange oil price risks, there is a 27% chance that at least a ten percent return on investment will be realized with a product price of \$1.18/lb. This was determined by creating a probability distribution for the price of orange oil with @Risk add-in for Excel. If further analysis of the properties of the novel polymer reveals advantages over conventional polystyrene, it is likely that this process will be able to compete with oil-derived polymers with current oil conditions. However, orange processors and the plastics industry should be aware of this technology and its developments as an alternative to oil-derived polymers.

Introduction

As the threat of the effects of global warming becomes more imminent, reduction of carbon dioxide emissions has become an important issue in industry. With initiatives like the Kyoto Treaty and the Clean Air Act attempting to curb the effect of atmospheric emissions become more popular, the need for emissions reduction technology is becoming a growing area of research and development. Another growing area of research is reducing dependence on foreign oil. Oil prices are vulnerable to political instability and the production rates determined by the major oil producing countries. Thus, reducing dependence on this oil provides more economic independence and stability for countries that do not produce large amounts of oil. Various technologies are becoming available for reducing emissions and reducing oil use. Some examples of these technologies are hybrid cars that use less gasoline and using oil reservoirs as carbon dioxide landfills rather than releasing it into the atmosphere.

Novel Polymer

Another desirable technology for reducing carbon dioxide emissions is using carbon dioxide as a feedstock for industrial production. As a feedstock, carbon dioxide would be abundant and fairly cheap because it is currently an undesirable side product. The carbon dioxide would also be “trapped” in the products made with it, and therefore would not be released into the atmosphere but rather put to a practical use. One such novel use of carbon dioxide as an industrial feedstock has been researched by Dr. Geoff Coates and his associates at Cornell University. They have developed a polymerization reaction which uses carbon dioxide and limonene oxide, a derivative of a chemical found in oranges, to make a new polymer, called polylimonene carbonate, which has properties similar to polystyrene. The properties of the novel polymer are still being analyzed and no information is available to the public at this time. This report outlines the design and economic feasibility of producing this polymer on an industrial scale.

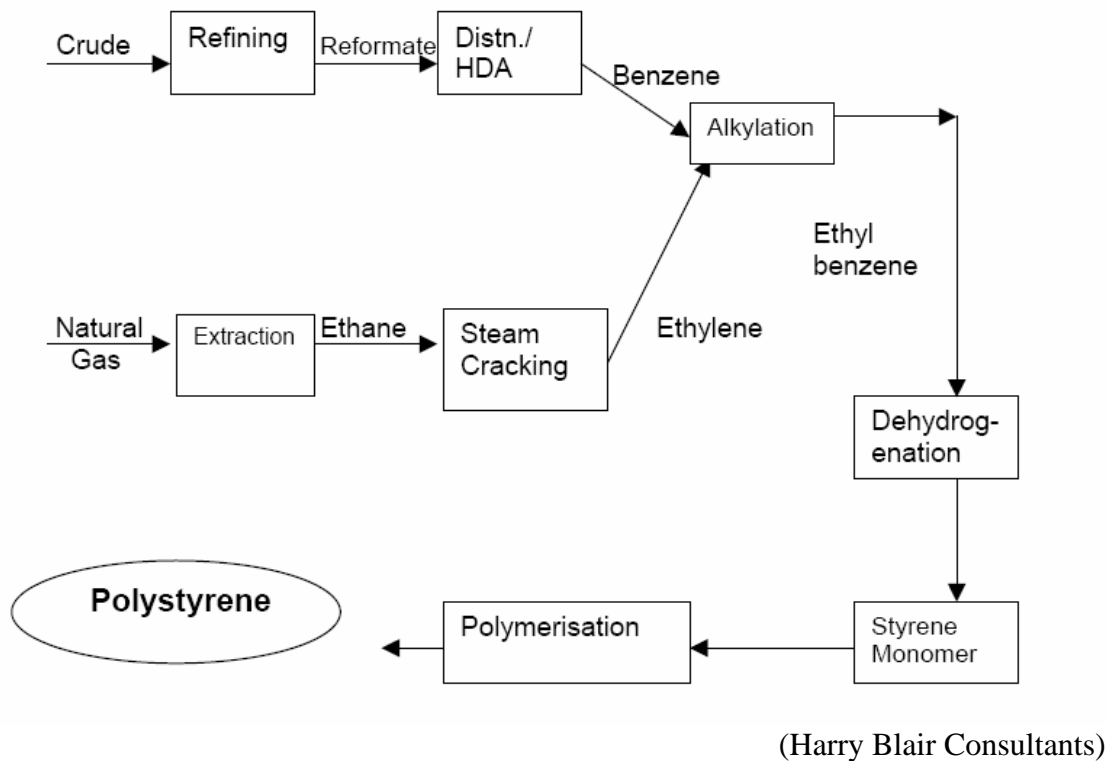
Conventional Polystyrene Production

The beginning of the polystyrene production chain is crude oil and natural gas. Ethane is produced from the refining process of a typical oil refinery and is one of the major products of natural gas. From there, the ethane is run through a steam cracker and ethylene is produced. Crude oil is run through a hydroalkylation and refining process and benzene is produced. At this point the benzene and ethylene are fed to a reactor and an alkylation takes place to form ethylbenzene. Ethylbenzene is then dehydrogenated into the styrene monomer. Polystyrene is produced by the polymerization of the styrene monomer. It is a highly exothermic reaction and is effectively initiated by adding a free radical provider such as benzoyl peroxide and providing modest heating of the solution.

There are three main types of polystyrene (PS), crystal PS, impact PS, and expandable PS. Each has its own unique set of advantages, disadvantages, and uses. Crystalline PS is a clear, hard polymer and is typically used for CD cases, electronics cases, and medical applications. While crystalline PS is typically produced by batch reaction of styrene monomers, there has been an increasing inclination to use continuous processes to make crystalline PS. Additional properties are added to the regular polymer such as UV

resistance, flame retardant, and anti-static mechanisms. Impact PS is essentially regular polystyrene that has had some polybutadiene elastomers added to the polymer. Impact PS is more resistant to collision than traditional PS and as a result is used in places that impacts would have serious deleterious effects such as, household appliances, and electrical casings like calculators and computers. Expandable PS is most commonly thought of as packing peanuts or disposable cups and plates. It is produced in the same general method as the other two types of PS except that toward the end of the polymerization reaction, a saturated hydrocarbon is added to the mixture. This hydrocarbon is absorbed into the beads of polymer and acts as a blowing agent. The polymer is then sent to a steam expander where the density of the polymer is reduced significantly by addition of air pockets. It is this last type of PS that we will try to substitute with the novel polymer. Regardless of the type of polystyrene produced, oil and natural gas are the principal ingredients of the polymer. With oil prices increasing every year, it is important for industry to attempt to find alternative sources for plastic products which can be produced via other methods. This will extend the life of our non-renewable resources such as oil and natural gas.

Figure 1: Conventional Polystyrene Process Flow Diagram

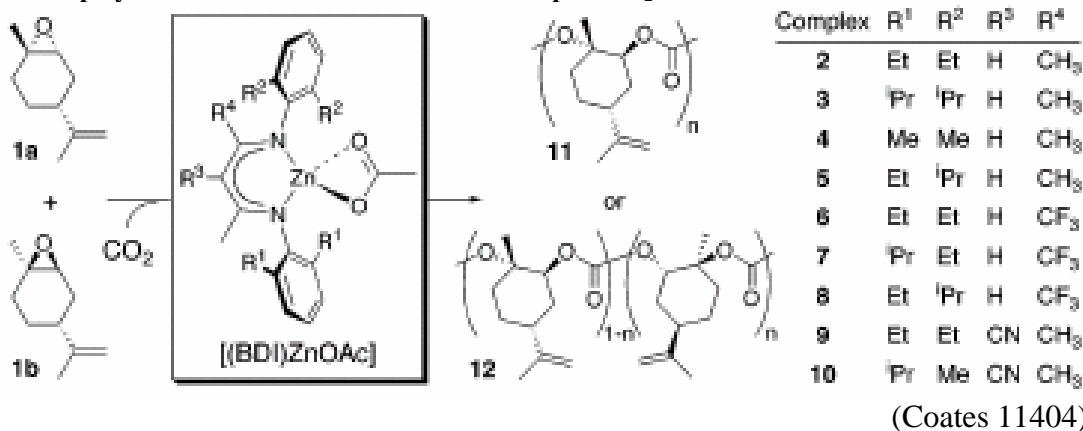


Process Design

Polymerization Reaction

The reaction investigated by Dr. Coates and his associates at Cornell is a copolymerization reaction involving pressurized carbon dioxide gas and trans-1,2-limonene oxide with a novel catalyst produced specifically for the process. Trans-1,2-limonene oxide is an epoxide which is one product of the oxidation of R-limonene, the primary component of orange oil. The catalyst is a beta-diiminato zinc complex which is dissolved in the limonene oxide solution. This reaction is stereoselective and results in a regularly patterned polymer with only one type of monomer. In the reaction shown below, the catalyst is selective for reactant 1a to produce the polymer 11. The catalyst to be used is given as complex 3 in the reaction below because this catalyst shows the best selectivity and activity. The reaction occurs at the optimum rate near 100 psi carbon dioxide and 25°C. In the investigation of the reaction, temperature, pressure, and catalyst type were varied to find the optimum rate and selectivity. The polymer is soluble in the limonene phase, but the addition of methanol causes the polymer to precipitate out of the liquid, which stops the reaction and reactivates the catalyst. The polymerization reaction results in a narrow molecular weight distribution with number averaged molecular weights around 9 kg/mol (Coates 11404-5).

Figure 2: Copolymerization Reaction at 25°C and 100 psia CO₂



Feeds

Limonene Oxide

R-(+)-Limonene Oxide is one of the two feedstocks for the polymeric process described in this report. It is one of several oxidation products that can be formed from limonene, which is the major component of orange oil. Oxidation of limonene is a natural occurrence when limonene is exposed to air, but it is undesirable because it contaminates the limonene and yields a mixture of several oxidation products. Limonene oxides are produced in relatively small amounts for some odorizing and flavoring applications. However, the amounts and processes of currently produce limonene oxide would not be able to sustain the rates at which this feedstock will be needed to produce a moderate amount of polymer. Thus, it is desirable to produce R-(+)-Limonene oxide from

limonene, which is a much more abundant substance. Limonene, which is present in orange oil as 90-96% R-limonene, can be combined with the tert-butyl hydroperoxide (TBHP) at ambient pressure and temperature to produce about 88% trans-1,2-limonene oxide with the aid of a titanium catalyst an organic solvent. This chiral epoxidation process is a developing one, which will likely become more popular industrially if green chemistry continues to grow in popularity. It is likely, therefore, that the industrial scale technology of this process will develop in parallel with the industrial scale technology of the polymerization process itself.

Limonene

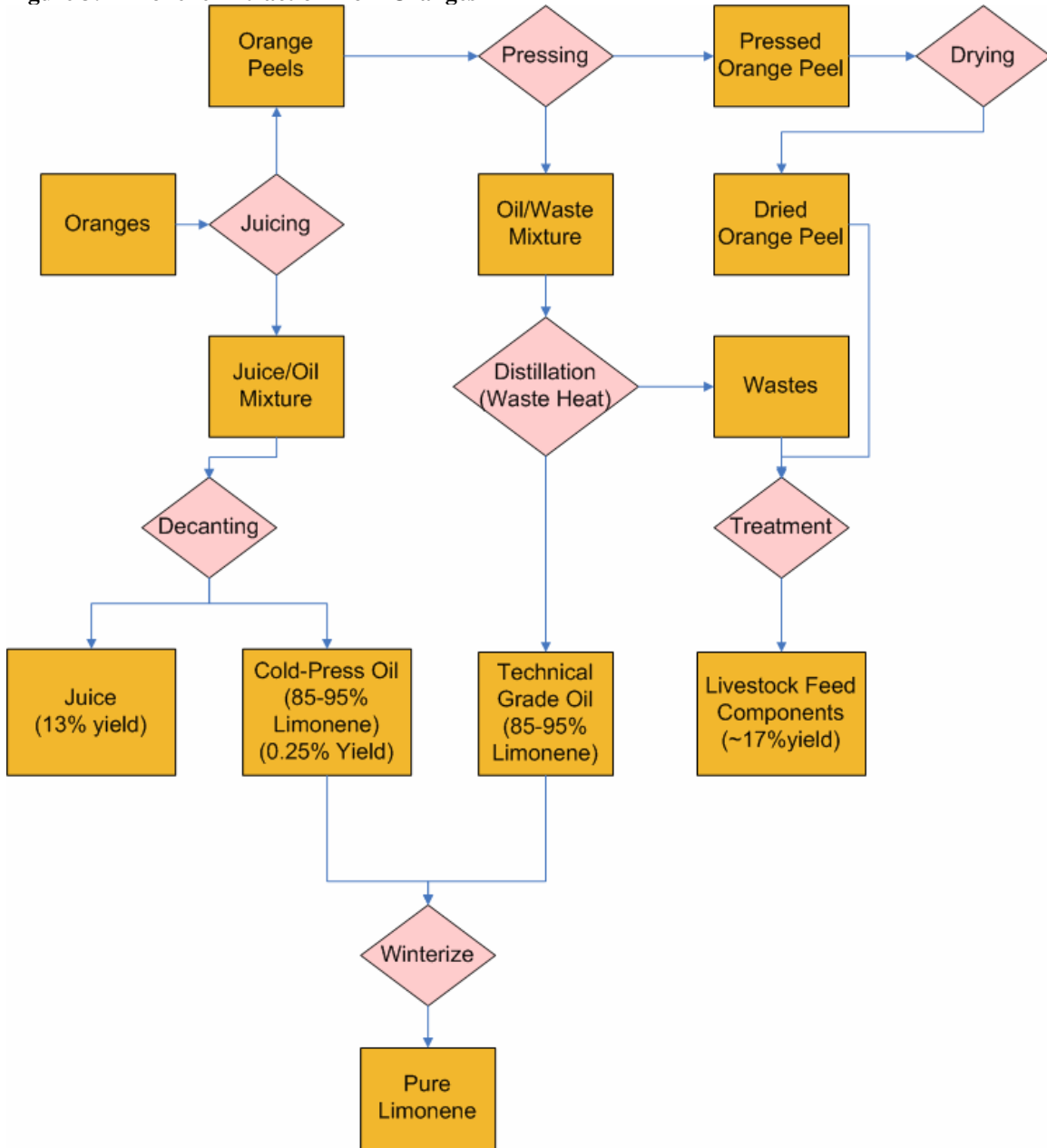
The primary source of limonene in the world today is oranges. Limonene is produced from oranges as a by-product of the orange juicing process. When an orange is juiced, it produces not only orange juice but it also produces a small amount of orange oil, which is separated from the orange juice to make the juice more pure and called cold press orange oil. Orange oil is also produced from the orange peel treatment process by steam extraction, producing oil which is called expressed orange oil. The remainder of the orange peels are usually processed to produce livestock feed. The orange oil from juicing and peel processing consists of approximately 90-95% limonene, and this is currently the largest source of limonene production in the world. The other components of the oil can be separated from the limonene by freezing the oil in settling tanks. These other components are water and waxes which are generally considered undesirable and therefore not sold. Since limonene has a lower freezing point than the other components of the oil, the other components freeze and settle to the bottom of the settling tanks. The amount of oil in an orange can vary from 0.25-0.5wt% and varies depending on orange type, weather, and maturity. Limonene is also present in lesser amounts in all citrus fruits and also in some trees, but the amount of limonene which can be produced from these alternate sources is less than that of oranges. The limonene in these alternate sources is also usually present as part of a complex mixture of organic substances, making separations more difficult. Another benefit of oranges as a source of limonene is that they primarily produce only the R-diastereomer of limonene, which is the stereoisomer of interest in the polymerization process. This eliminates most of the waste of the unused diastereomer in the process and increases the yield compared to a stereoisomer mixture (Guenther).

Carbon Dioxide

Carbon dioxide is an unwanted waste product of many chemical reactions, most notably the combustion of fossil fuels by power plants. Because of the contribution of industrial emissions of carbon dioxide to the greenhouse effect, companies are working to reduce their carbon dioxide emissions. Thus, carbon dioxide is a cheap and abundant substance which it is desirable to use in profitable processes that prevent it from being released into the atmosphere. Because companies are so eager to reduce their carbon dioxide emissions, the carbon dioxide can most likely be purchased from companies at the cost of compressing and transporting the gas. Since many environmental activist groups are currently suggesting a tax on carbon dioxide emissions, it is possible that in the future it would be more economical for companies to sell the carbon dioxide rather than pay the taxes on it. This might lead to an even cheaper source of carbon dioxide. The amounts

of carbon dioxide needed could easily be supplied by even a single plant of one of the large electric power plants, such as those owned by the Progress Energy in Florida. Since Progress Energy has spent millions of dollars on technology to reduce emissions, it is likely that they would be a good candidate for carbon dioxide supply (Progress).

Figure 3: Limonene Extraction from Oranges

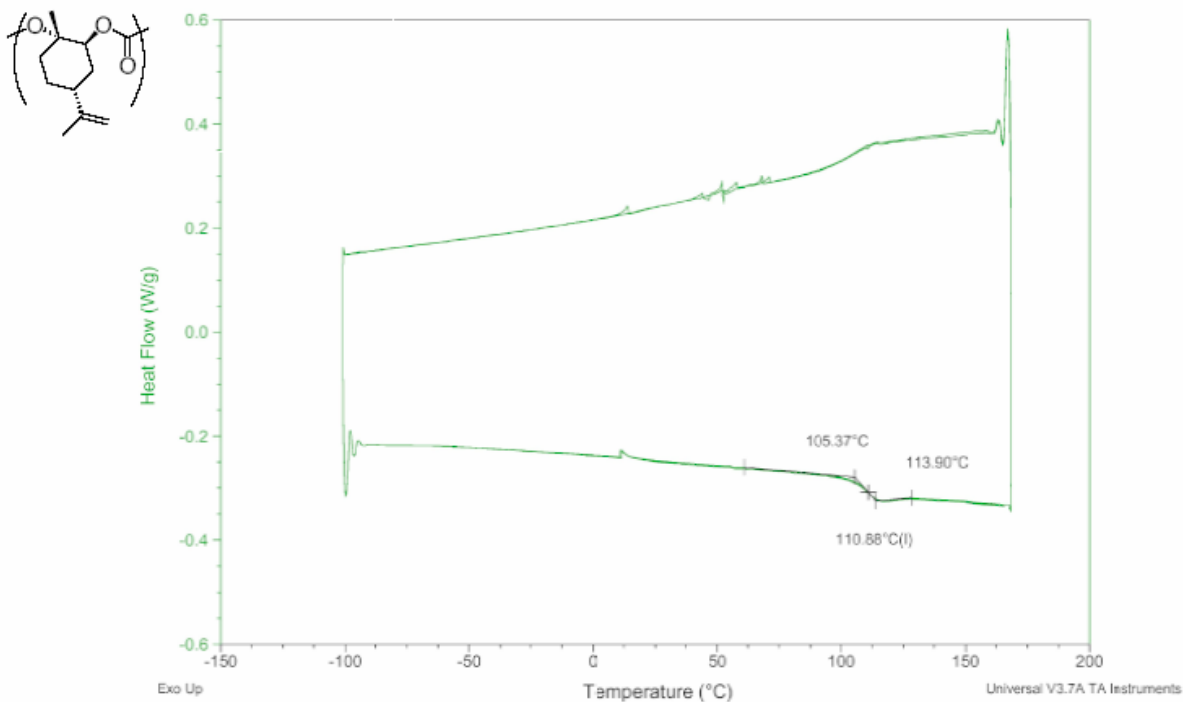


Products

According to Dr. Geoff Coates polylimonene carbonate has properties similar to polystyrene. Since there are several types of polystyrene, each with their own properties and uses, it is necessary to attempt to narrow down the options so that an appropriate

market analysis can be done. From the differential scanning calorimetry graph of the polymer shown in Appendix A, one important property can be determined. We can determine from the existence of a glass transition point and the lack of a melting point, that it is very likely an amorphous polymer. An amorphous polymer would lack any crystalline structure and molecules would not be aligned with each other. This would make it difficult for it to display characteristics similar to crystalline PS. Almost every actual property of this new polymer is currently unknown because there is not enough of it to accurately test. As more properties become known, it will be easier to determine what uses would best suit this polymer. Because of the amorphous property of the polymer and the statement from Dr. Coates that it behaves similarly to polystyrene, the most logical choice for a similar comparison is expandable polystyrene. After foaming has been completed, this polymer could be used to produce disposable cups or packing peanuts.

Figure 4: DSC of PolylimoneneCarbonate



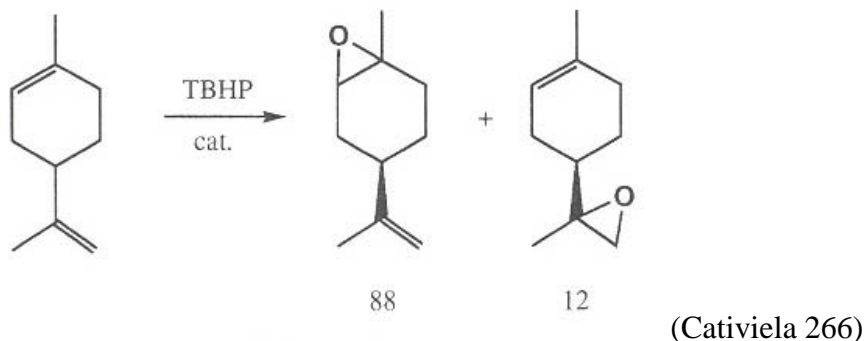
(Byrne)

Limonene Oxide Reaction

Trans-1,2-limonene oxide can be produced from R-limonene in a reaction with tert-butyl hydroperoxide in a catalyzed epoxidation reaction shown below. The catalyst used in this reaction is $Ti(O^iPr)_4$ bound to silica. The catalyst does become deactivated with time and will therefore require regular washing and reheating. The reaction also produces 12% of a side product of limonene-8,9-oxide. This side product has properties very similar to the main product of the reaction, so the only method of separation will be the selectivity of the polymerization catalyst for trans-1,2-limonene oxide causing it to be the only isomer consumed in the reaction. The tert-butyl hydroperoxide (TBHP) used as an oxidizing

reagent in the reaction is converted to tert-butyl alcohol, which will therefore be another product of the overall process which will be sold. Ideally, however, if TBHP could be cheaply recycled, this would greatly improve the economic outlook of this process. However, standard oxidation reactions such as those using hydrogen peroxide would cost more than the assumed price differential between TBHP and tert-butyl alcohol. An inert organic solvent is also necessary for this reaction, so acetone was chosen for its ease of separation and availability. The reaction occurs at ambient temperatures and pressures (Cativiela 259-267).

Figure 5: Limonene Oxide Reaction



Overall Process Summary

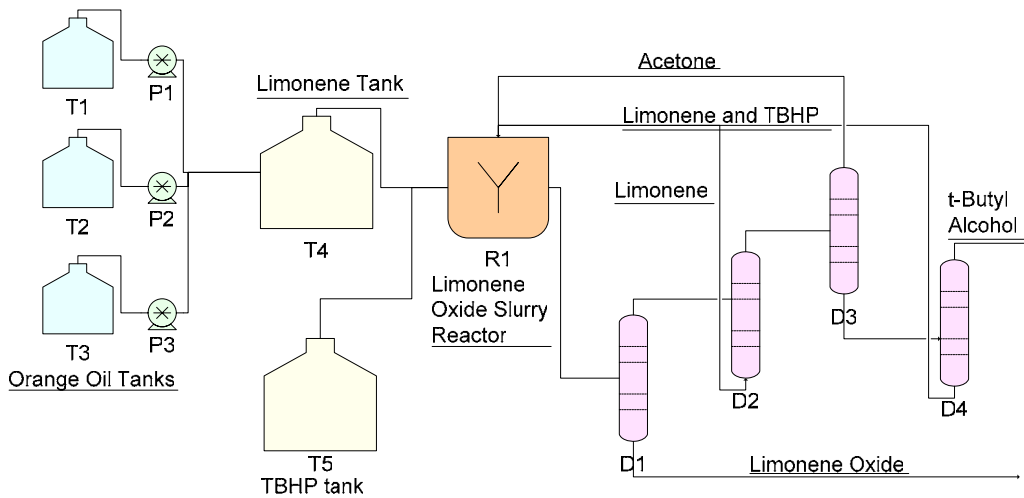
The proposed process involves taking the raw materials orange oil and carbon dioxide and producing the polymer from them. In order to do this, two main sub-processes are necessary. First, the limonene must be separated from the orange oil and limonene oxide must be produced and separated from the other products and reactants of the reaction. This process will be referred to as the limonene oxide process. Then, the limonene oxide must be reacted with the carbon dioxide in the polymerization reaction, and the polymer must be separated and dried. This process will be referred to as the polymerization process. The designs for these two processes will be presented separately with the understanding that the two processes are connected by the limonene oxide stream.

Limonene Oxide Process Design

The goal of the limonene oxide process is to produce limonene oxide and tert-butyl alcohol using limonene and tert-butyl hydroperoxide. This reaction is catalyzed by a solid silica catalyst and takes place in the inert solvent acetone. The reactor type chosen for this reaction was a slurry type continuous stirred tank reactor because access to the catalyst will be necessary for reactivation of the catalyst. The volume of the reactor was found by keeping the concentration of moles to weight catalyst consistent with the lab scale experiments and assuming that similar conversions occur on the large scale. This assumption was used to estimate a residence time of 2 hours for 75% conversion. The residence time was then used to find the volume of the reactor. This volume was around 3000 gallons. Since the reaction is not run to completion, the reactor product stream includes limonene, limonene oxide, tert-butyl alcohol, tert-butyl hydroperoxide, and acetone. A series of distillation columns are needed to separate the reactants and the solvent, which are recycled, from the products. The sizes of the distillation columns were

found by simulation using Pro/II 7.0 by Simulation Sciences, Incorporated (Appendix A). Sizes of tanks were determined using residence time, which can also be found in Appendix A. A process flow diagram for the process is shown below.

Figure 6: Limonene Oxide Process Flow Diagram



Displayed Text	Description	Size	Material	Cost
D1	Distillation Column 1	0.5 m dia., 10 trays	304 SS	\$13,400
D2	Distillation Column 2	0.5 m dia., 4 trays	304 SS	\$5,400
D3	Distillation Column 3	0.5 m dia., 13 trays	304 SS	\$17,500
D4	Distillation Column 4	0.5 m dia., 13 trays	304 SS	\$17,500
R1	Slurry Reactor	3000gal	304 SS	\$25,000
T1	Orange Oil Tank 1	2.3E4gal	304 SS	\$77,000
T2	Orange Oil Tank 2	2.3E4gal	304 SS	\$77,000
T3	Orange Oil Tank 3	2.3E4gal	304 SS	\$77,000
T4	Limonene Tank	9E3gal	304 SS	\$57,000
T5	TBHP Tank	9E3gal	304 SS	\$57,000

Polymerization Process Design

Limonene oxide is fed to a continuous stirred tank reactor at a pressure of 100 psi. A CSTR was chosen because it is more efficient and cheaper than a batch reactor. We chose to not use a PFR because in order to have flow characteristics within the reactor that would allow the reaction to behave in a manner providing consistent products, the diameter of the tubes would have to be less than 1 inch. Having 1 inch tubing flowing at the volumetric flow rates that we require would be very expensive on pumping equipment and have huge pressure drops along the pipes. Therefore, the CSTR was deemed the most effective method of polymerization. Carbon dioxide is bubbled up from the bottom of the reactor. This provides good contact between the limonene oxide and carbon dioxide which will increase the rate of polymerization. The zinc based catalyst is also fed to the reactor. After being bubbled up through the limonene oxide, there is a strong possibility that there will be excess carbon dioxide. The excess carbon dioxide is compressed and passed through the reactor again along with fresh feed. The reactor has a residence time of approximately 9 hours which allows for a conversion of approximately

50% of the limonene oxide. This allows the reactants to remain fluidized and still get significant conversion. Reactors in this process were sized using residence times and flow rates determined by production rate. The reactor contents are then fed to a methanol wash tank where the solution is washed with methanol. These wash tanks were sized using a residence time of 10 minutes. The methanol has the effect of causing the polymer to precipitate from the solution. The entire solution is then fed to a rotary vacuum filter where the polymer is removed from the mixture and rinsed with additional methanol. The rotary vacuum filters were sized using the liquid flow rate of pulp into a single compartment rotary drum vacuum filter as outlined in Peters, Timmerhaus, and West (PT&W). Using this method, the smallest industrial sized filter of 0.5 m² was found to be sufficient for each filter therefore this size was chosen for all filters. The polylimonene carbonate is fed to a vacuum dryer where the remainder of the methanol is vaporized off of the polymer. This dryer was sized using the solids velocity of the polymer and an assumed surface area percentage used by the solids of 50% as outlined in PT&W. The smallest dryer size of 4.65 m² was found to be sufficient for the total mass velocity of polymer. The liquid stream from the washing tank containing methanol, limonene oxide, and catalyst is fed to a distillation column. The methanol is separated from the limonene oxide and the methanol is fed back to the methanol tank. The limonene oxide and catalyst is fed to the next reactor. The reactor once again has a residence time of approximately 9 hours and once again comes to 50% completion. The reactants undergo the same processes of washing, filtering, distillation, and then reaction, washing, filtering, and distillation one final time. Every distillation column in this process has 2 trays, as determined by simulations in Pro/II. It should be noted that there are two different components in the original liquid feed stream to the reactor and that only the limonene oxide will produce the polymer. The other component is inert in this system and as a result remains liquid throughout the series of unit operations. This means that at the end of the third distillation column there is approximately 5.178 ft³/hr of the non-reacting isomer of limonene oxide with the catalyst still dissolved in it. In order to get the catalyst back, we feed the stream to a flash tank and vaporize approximately 92% of the limonene oxide. The remaining liquid we feed back to the original feed stream in the first reactor so that the catalyst can be reused. The addition of the non-reacting limonene oxide is very small so it will be essentially negligible. The remaining limonene oxide is sent to barrels and can be sold as fragrance components. The polymer produced in this process is fed to a silo to be held until acquisition by the Styrofoam processor. This silo was chosen to be 47 m³ in order to hold one day's production.

Economic Analysis

Economic Calculations

In order to determine the economic feasibility of designing such a plant, a sequence of economic calculations had to be executed. First, a total capital investment was found using the purchased equipment costs as a basis. Equipment prices for both processes were estimated from PT&W with the silo estimation quoted from Zeppelin Systems USA. From these prices, the investment could then be estimated using a series of percentages based on the purchased equipment pricing. These percentages were found from PT&W. A table with these calculations is shown below.

Table 1: Capital Investment Calculation
Estimation of Capital Investment Cost

Component	Percent of Delivered Equipment Cost	Estimated Cost
Direct Costs		
Purchased Equipment	Based on Equipment Sizes	\$931,995
Delivery	10% of Purchased Equipment	\$93,199
Subtotal: Delivered Equipment	100	\$1,025,194
Purchased Equipment Installation	47	\$481,840
Instrumentation (Installed)	36	\$369,070
Piping (Installed)	68	\$697,130
Electrical (Installed)	11	\$112,770
Buildings (Including Services)	18	\$184,530
Yard Improvements	10	\$102,520
Service Facilities Installed	70	\$717,640
Total Direct Cost		\$4,715,888
Indirect Costs		
Engineering and Supervision	33	\$338,310
Construction Expense	41	\$420,330
Legal Expense	4	\$41,010
Contractor's Fee	22	\$225,540
Contingency	44	\$451,090
Total Indirect Cost		\$1,476,280
Fixed Capital Investment	Direct Cost + Indirect Cost	\$6,192,170
Working Capital	15% of TCI	\$1,092,740
Total Capital Investment		\$7,284,900

Next, the total product cost was estimated using the current prices of raw materials and a series of percentages. These percentages yielded manufacturing costs and general expenses which were totaled together to estimate the total product cost per year. The number of employees is based on the production rate and number of processing steps of a

solid and fluid processing plant. Research and development and the laboratory fees are incorporated in order to further improve upon the process and analyze the properties and uses of the polymer. The table shown below breaks down these total costs.

Table 2: Product Cost Calculation

First-Year, Annual Total Product Cost		
Component	Basis for Estimate	Cost
I. Manufacturing Cost		
A. Direct Production Costs		
1. Raw Materials (Values for Subsequent Years Shown in Appendix B)		
Orange Oil (2005 value)	\$0.77/lb X 3333 lb/hr X 8760 hr/year	\$25,105,634
TBHP	\$0.70 X 2025 lb/hr X 8760 hr/year	\$12,417,300
Carbon Dioxide	\$0.10/lb X 6E6 lb/year	\$600,000
Acetone	\$0.37/lb X 5025 lb/year	\$1,859
Methanol	\$0.14/lb X 5000lb/year	\$700
2. Operating Labor	80 employee hours/day X 33.67\$/h	\$983,164
3. Operating Supervision	15% of Operating Labor	\$147,470
4. Utilities	10% of Total Product Cost	\$4,775,170
5. Maintenance and Repairs	7% of Fixed Capital Investment	\$433,450
6. Operating Supplies	15% of Maintenance and Repairs	\$65,020
7. Laboratory Charges	15% of Operating Labor	\$147,470
8. Patents and Royalties	4% of Total Product Cost	\$1,910,070
Subtotal		\$46,587,310
B. Fixed Charges		
1. Depreciation (Calculated Separately in Appendix B)		
2. Property Taxes	2% of Fixed Capital Investment	\$123,840
3. Insurance	1% of Fixed Capital Investment	\$61,920
Subtotal (Without Depreciation)		\$185,760
C. Overhead costs	50% of operating labor, supervision, and maintenance	\$782,042
Total Manufacturing Cost		\$47,555,112
II. General Expenses		
A. Administrative Costs	20% of Operating Labor	\$196,630
B. Distribution and Marketing Costs	2% of Total Product Cost	\$955,030
C. Research and Development	2% of Total Product Cost	\$955,030
Total General Expenses		\$2,106,690
Total Product Cost (Without Depreciation)		\$47,751,744

Finally, the profitability for having such a plant was calculated for a ten year project life. These calculations were based on a 2% inflation rate for all products and raw materials. Also, the Modified Accelerated Cost Recovery System (MACRS) outlined in PT&W was utilized in order to determine the depreciation of the fixed investment in depreciable property. According to PT&W, the recovery period for this type of process is assumed to

be seven years. Federal corporate income tax was also included as a cost in the profitability assessment. The taxable income included the gross profit with depreciation in order for the investors to recover the costs for the property over ten years. The corporate income tax rate for Florida has no tax brackets and therefore is a flat rate of 5.5%. In order to determine the profitability of the project, the net present worth and return on investment were calculated. Assuming the price of novel polymer to be the same as the current price of polystyrene, the net present worth was found to be -\$61 million and therefore is not profitable. However, the minimum acceptable return on investment is assumed to be 10%. Scenarios to generate this return are considered in the Conclusions section. Appendix B delineates the profitability of this project.

Location

Major considerations for the location of the polymer production plant include feedstock and product availability and markets. Since Florida in the United States and Sao Paulo in Brazil are the two main processing locations for oranges, only these two locations afford enough orange oil to feed this process, and thus orange oil would have to be shipped from one of these two locations to the plant location. Carbon dioxide could be supplied in enough abundance by power plants in either Florida or Sao Paulo. However, Brazil depends largely on hydroelectric power for its electricity, and since it is considered a developing country and already has low carbon dioxide emissions per capita, Brazilian power plants are not under much pressure to reduce their emissions. In Florida, several fossil fuel power plants make high carbon dioxide emissions (Progress). These plants would be much more likely to provide carbon dioxide at or below the cost of capturing it. Florida also contains a styrofoam production plant in Plant City owned by the major polystyrene processor Dart Container Corporation. Florida also has a very competitive corporate income tax rate, with the eighth lowest rate in the nation (Tax Foundation). Since Florida is not at a major disadvantage in labor costs or taxes, it presents the most attractive location because of readily available feedstocks and product processors and therefore low transportation costs.

Risk

The orange industry is currently equipped for limonene production, and orange oil and limonene are commonly sold as cleaning solvents, flavoring agents, and odorizing agents. However, limonene production cannot be easily separated from the current orange juicing processes, since limonene is produced during both the juicing process and the peel treatment process. Waste heat from throughout both processes is used to distill the limonene from the molasses which is also produced during the peel treatment process. Limonene is also produced in relatively small amounts compared to the juice and livestock feed produced from oranges, making the size of the juicing plant necessary to produce an appreciable amount of limonene quite large. Although orange juice is the most valuable product for the juicing industry, its price fluctuates greatly. Orange juice prices have both doubled and been cut in half throughout the last ten years. This makes the orange juice business extremely high in risk for small or startup businesses. However, the amount of limonene produced by large orange processors could be increased to meet a higher market demand if necessary. Based on the estimated amount

of orange oil present in oranges, the orange market currently produces only about half of the limonene that could be harvested from oranges. The main reason that low yields of orange oil are accepted is that large processors can perform steam extraction on larger batches of peels with low yields and still keep up with current demand. In order to obtain higher yields, the steam extraction batches would need to be kept small, which would lower the production rate of livestock feed produced from the peels (Guenther). However, a shift in the value of limonene would force orange peel processors to find a new balance between production rate and limonene yield in order to produce more limonene. In this manner, the rise in demand caused by the feedstock demand for this polymerization process could be largely offset by increasing the yield of limonene from orange peels in order to meet this new demand, leaving the market relatively stable. Due to the need to have a consistent and abundant source of orange oil and the close pairing of the orange oil extraction and juicing processes, it would be desirable to be paired with a major juice processing company, such as Tropicana, which is based in Bradenton, Florida.

Assuming a major orange producer is involved, the major risks in this project include technological failure of the process design. This risk can be reduced by producing a pilot plant for the polymerization process. This would be low cost and would likely be covered by government grants for research on emissions or oil use reduction. With pilot plant data, the technological risk would be minimal for a scaled up plant and most of the risk would be determined by the physical properties of the novel polymer and the material costs. Since polymer costs and demands are currently high and expected to increase, cost changes in the polymer are a relatively low risk (Hoffman 1). The polymer could have desirable or undesirable physical properties, however, when compared to polystyrene, which would make the price of the polymer slightly higher or lower than that of polystyrene. This could translate to either losses or higher profits depending on the desirability of these properties. Orange oil and limonene production levels are another risk factor, and they vary based on any disturbances in the orange crop, such as droughts, hurricanes, or freezes. These production levels can cause the selling price and therefore the value of orange oil to vary widely at times. For example, in 1994, prices increased by over 300% when a Brazilian crop was affected by a drought (Landau 24). Most major natural disasters in Florida or Brazil could make the orange oil production rates insufficient to run the polymer plant at full capacity and would actually make it more profitable for an orange process company to sell the orange oil directly rather than convert the oil to polymer. This represents a major risk for the profitability of the polymer plant. In the face of such a disaster, the likely outcome would be that the plant would need to be shut down for that growing season, which would be an undesirable inconvenience to the companies which processed and purchased the polymer.

The risk analysis add-in @ Risk version 4.5.3 created by Palisade Corporation was used to model the risk caused by the orange oil. A probability distribution for the price of orange oil was estimated using the prices caused by excess and inadequate supplies of orange oil over the past 15 years, which have caused price fluctuations from \$0.50/lb to \$2.50/lb (Floreno 12 and 37). This distribution was applied to the price of orange oil for each year independently. The polymer plant was assumed to be shut down for any year

in which the limonene price rose above \$0.80/lb, since the orange company would not have enough excess limonene for the process and would instead sell its entire supply at the high price. Using the selling price of polymer necessary to have a 10% return on investment of \$1.18/lb for the polymer, the distribution of orange oil prices was used to determine the distribution of Net Present Worths that would result. Two other plant capacities, $\frac{1}{2}$ and $\frac{2}{3}$ of the top capacity, were also considered in the risk analysis. These two lower capacities, as shown by the risk curves, do not offer a sufficiently lower probability of loss to justify their lower profit probabilities, since the top capacity shows a much larger probability of making higher profits. As shown in the charts below, the risk causes there to be about a 58% chance of a profit being made for the given selling price of the polymer at the top capacity considered.

Figure 8: Histogram of NPW for Orange Oil Risk for Top Capacity

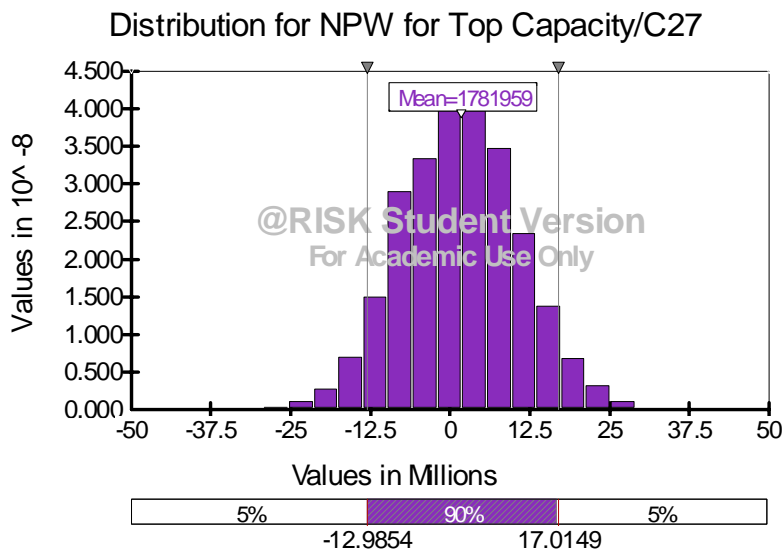
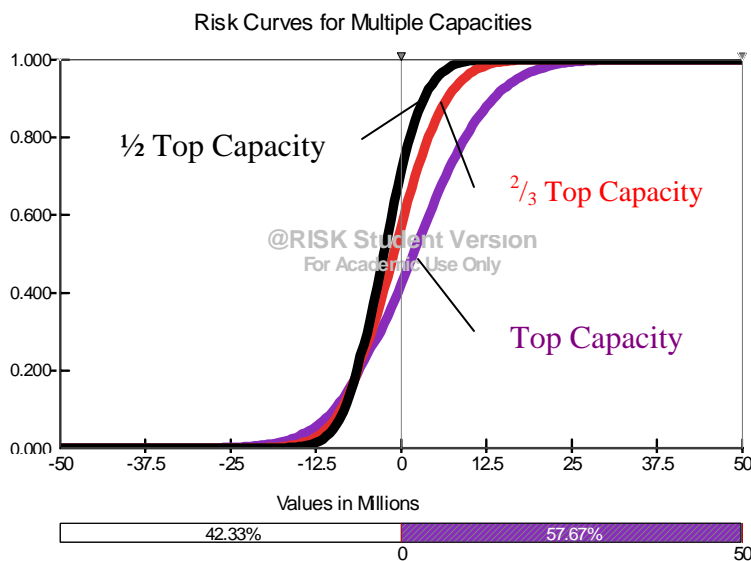


Figure 9: Risk Curve of NPW for Orange Oil Risk for Multiple Capacities



Since these risks are too large for a small startup company to absorb and the dependence on the orange company makes it necessary to be closely partnered with a very large orange processing company, the polymerization plant should probably be owned by one of the major orange processing companies in Florida, such as Tropicana. Since Tropicana is owned by PepsiCo, it represents a major financial force that could easily supply the modest capital needed to start this process. Tropicana's major processing hub is located in Bradenton, Florida, within about 50 miles of both a major power plant owned by Progress Energy, which would be a good source of carbon dioxide, and a styrofoam plant owned by Dart Container, Corporation. Since Progress Energy would stand to instantly reduce emissions if it were to supply this process, it is likely that they would be willing to produce the small amount of capital needed to set up the infrastructure to compress and transport the carbon dioxide. Depending on the ultimate end market for the Styrofoam products produced from the polymer, Dart Container may also be interested in making an agreement to process the polymer. If Tropicana, Progress Energy, and Dart Container formed an alliance for the production and processing of the polymer, the financial risk of each company would be relatively low, with Tropicana having the most risk and the most capital invested.

Conclusions and Recommendations

Conventional polystyrene possesses a wide range of properties suitable for many diversified applications. In its original form and without any additives, polystyrene can be injected to form clear plastic coverings such as CD cases. But its most popular form in the plastics market is extruded and expanded foams. These forms of polystyrene are ideal for packaging because of its light weight and ability to conform into any shape. Furthermore, the United States Food and Drug Administration qualifies polystyrene as meeting all requirements for use in the food industry. Clearly, the range of polystyrene buyers are numerous and diverse. Many analysts also believe that the demand for all types of plastics is strong in all end markets and that the demand will continue to grow in the subsequent years (Hoffman 12). However, with current raw material and product costs, the net profit for each year considered in this evaluation is negative. Further analysis dictates that in order for the process to be profitable and for investors to regain 10% on their investment, the current selling price of polystyrene would have to fluctuate to a price of \$1.18 which entails a 31% increase. Further analysis is recommended in order to determine which end market companies are willing to pay higher prices for environmentally friendly products. Considering the current conditions, it is economically unfeasible to create such a process. However, because of high fluctuations in orange oil and polystyrene prices, the probability for these prices to fluctuate to a profitable scenario in the future is not unreasonable. In addition, further analysis of polylimonene carbonate is recommended in order to determine the exact properties especially those considered important to polystyrene. The possible advantages of polylimonene carbonate in contrast to conventional polystyrene could validate the 31% increase in selling price. It is also possible that this polymer has characteristics that are considered desirable in other plastics making it a possible replacement for these plastics. Orange processing companies stand the most to gain from this process and should therefore study it most

aggressively, but fossil fuel power companies and plastics processors and consumers should also be aware of this technology as a possible future alternative to oil-derived polymers.

Future Work

The biggest financial obstacle for this process is currently the product cost. Things that largely affect the product costs are raw material needs and utility prices. Raw material costs could be reduced if the tert-butyl alcohol could be converted back into tert-butyl hydroperoxide, which could then be recycled in the limonene oxide process. This reaction would involve oxidation, which is a typical industrial reaction and therefore may be relatively simple to add to the overall process. This would eliminate a major cost and risk factor for the overall process since the tert-butyl hydroperoxide cost is approximately 25% of the current product cost. Raw material costs could also be lowered by possibly finding more sources of limonene. While orange oil contains the most limonene of any plant oil, other plant oils do contain a great deal of limonene, and these alternative sources could allow for larger capacities for the plant as well as a possibly lower cost for the feed limonene. The utility prices are currently only an estimate based on typical percentages of product cost presented by Peters, Timmerhaus, and West. More accurate estimates of the utilities needed could be calculated from simulation and typical power requirements for each piece of equipment. Utilities costs currently account for about 10% of the total product cost. While closer estimation may result in a lower utility cost, it is also possible that the utility cost could be higher, but this is unlikely because of the relative simplicity of the process.

Besides lowering product cost, another source of more economic certainty would be more analysis of plastic processing and end market possibilities. This analysis will indicate more possible uses for the new polymer and market trends for oil-derived polymers, possibly changing the estimated value of the polymer. Further analysis of polymer processing may also reveal processing advantages of the new polymer, which would again translate to a higher value.

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Supporting Material

Available Electronically at [coe-reagan.sooner.net.ou.edu\Coe-m206\M:\Polymer from Oranges\Supporting Material](http://coe-reagan.sooner.net.ou.edu/Coe-m206\M:\Polymer from Oranges\Supporting Material)

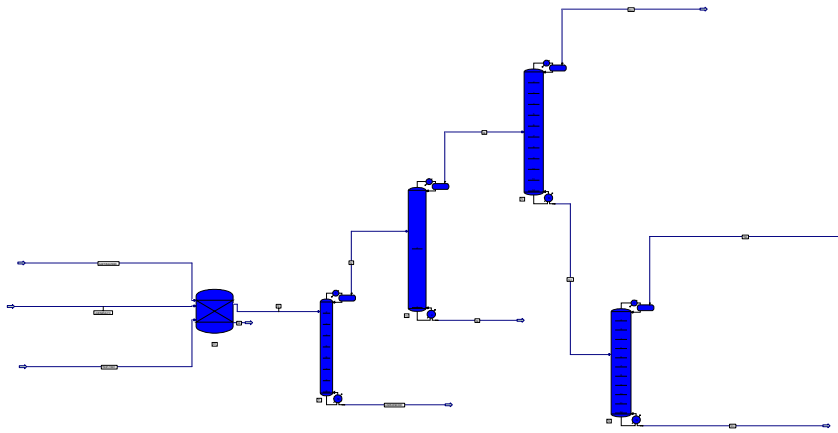
1. Pro II Simulation of Limonene Oxide Process (trans-limonene oxide process.prz)
2. Pro II Simulation of Polymerization Process (distillation columns for polymerization rct.prz)
3. Excel Economics Calculations (econ2.xls)
4. Excel Economics @ Risk Calculations (econrisk.xls and econrisk.rsk)
5. Excel Tank Design Calculations (Equipment Sizing New.xls)
6. Process Flow Diagrams (New Limonene PFD.vsd and Limonene Oxide PFD.vsd)

Appendix A: Simulations and Data

Tank sizing

Production Rate	3.30E+07	lb/yr	Notes around 1 week 1 day ratio of MW LO to MW LO+MW CO2 volume times residence time volume times reserve time need to be refrigerated same as reserve, in case of refrigeration failure, no refrigeration needed to transfer between tanks \$77,000 per 2.3E4 gallon tank
Drum Residence Time	0.019178082	yr	
Reserve time	0.002742857	yr	
MW of limonene oxide	154.25		
MW of limonene	136.24		
MW of carbon dioxide	44		
Ratio of LO/CO2 in polymer	0.778058008		
Density of limonene	7.01	lb/gal	
Density of limonene oxide	7.69	lb/gal	
Limonene Needed	2.27E+07	lb/yr	
Volume Limonene Needed	3.24E+06	gal/yr	
Tank space needed at any time	6.20E+04	gallons	
Reserve space	8.87E+03	gallons	
Total bulk tank space	7.09E+04	gallons	
Number of bulk tanks	3		
Tank Volume	2.36E+04	gallons	
Pure Limonene Tank	8.87E+03	gallons	
Number of pumps	3		
Pump work			
Orange Oil Tank Cost	\$231,000		
Limonene Tank Cost	\$57,000		
Total Tank Cost	\$288,000		
Conversion for of limonene Rate for LO Reactor for LO	0.9		
LO produced	2.04E+07	lb/yr	

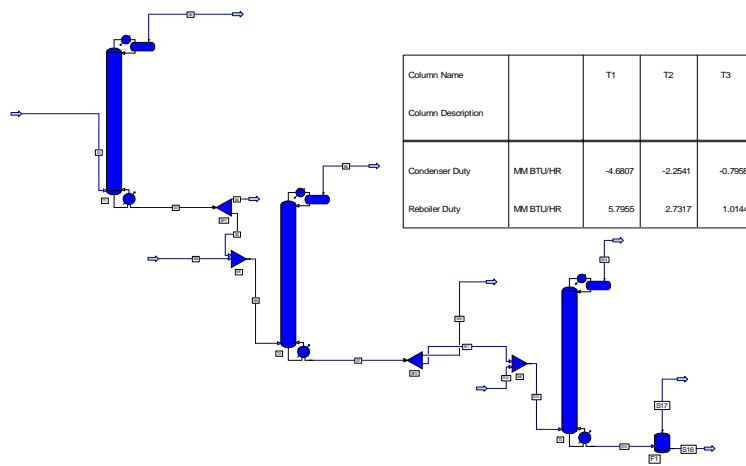
Limonene Oxide Process Simulation



Stream Name		LIMONENE-FD	TBHP-FEED	LIMONENE-OXID	TBA
Stream Description					
Phase		Liquid	Liquid	Liquid	Vapor
Temperature	C	25.000	25.000	206.428	83.098
Pressure	ATM	1.000	1.000	1.000	1.000
Flowrate	LB-MOL/HR	29.361	30.000	22.054	22.556
Composition					
CAMPHOR		0.000	0.000	0.979	0.000
DLIMENE		1.000	0.000	0.021	0.000
ACETONE		0.000	0.000	0.000	0.036
TBUTALC		0.000	0.000	0.000	0.948
TBUTHYPR		0.000	1.000	0.000	0.017

Polymerization Process

Stream Name		S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11	S12	S13	S14	S15	S16	S17
Stream Description																		
Phase		Liquid	Vapor	Liquid	Liquid	Liquid	Vapor	Liquid	Liquid	Liquid	Liquid	Liquid	Liquid	Liquid	Vapor	Liquid	Liquid	Vapor
Temperature	F	78.000	174.712	349.904	349.904	349.904	174.712	349.904	78.000	138.069	349.904	349.904	78.000	138.054	162.303	318.073	307.622	307.622
Pressure	PSIA	14.000	14.000	15.000	15.000	15.000	14.000	15.000	14.700	14.700	15.000	15.000	14.700	14.700	15.000	14.000	14.000	14.000
Flowrate	LSA/LHR	63.278	52.514	10.764	5.381	5.383	25.289	5.123	25.030	30.412	2.561	2.562	11.914	14.476	11.933	2.543	0.197	2.348
Composition																		
CAMPHOR		0.174	0.010	0.973	0.973	0.973	0.010	0.973	0.000	0.172	0.973	0.973	0.000	0.172	0.005	0.967	0.999	0.954
METHANOL		0.826	0.990	0.027	0.027	0.027	0.990	0.027	1.000	0.828	0.027	0.027	1.000	0.828	0.995	0.043	0.001	0.046



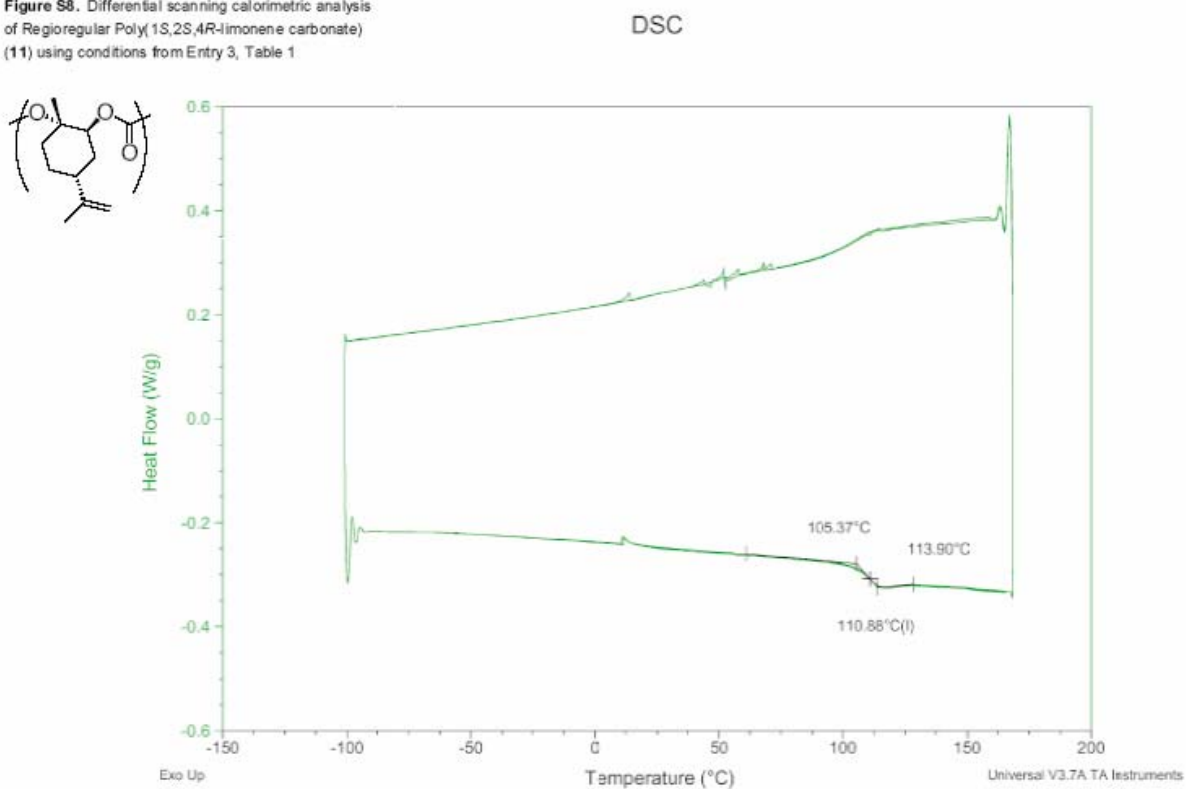
Stream Name		S1	S2	S3	S4	S5	S6	S7	S8
Stream Description									
Phase		Liquid	Vapor	Liquid	Liquid	Liquid	Vapor	Liquid	Liquid
Temperature	F	78.0	174.7	349.9	349.9	349.9	174.7	349.9	78.0
Pressure	PSIA	14.0	14.0	15.0	15.0	15.0	14.0	15.0	14.7
Flowrate	LB-MOL/HR	63.3	52.5	10.8	5.4	5.4	25.3	5.1	25.0
Composition									
CAMPHOR		0.2	0.0	1.0	1.0	1.0	0.0	1.0	0.0
METHANOL		0.8	1.0	0.0	0.0	0.0	1.0	0.0	1.0

Stream Name		S9	S10	S11	S12	S13	S14	S15	S16	S17
Stream Description										
Phase		Liquid	Liquid	Liquid	Liquid	Liquid	Vapor	Liquid	Liquid	Vapor

Temperature	138.1	349.9	349.9	78.0	138.1	162.4	318.1	397.6	397.6
Pressure	14.7	15.0	15.0	14.7	14.7	15.0	14.0	14.0	14.0
Flowrate	30.4	2.6	2.6	11.9	14.5	11.9	2.5	0.2	2.3
Composition									
CAMPHOR	0.2	1.0	1.0	0.0	0.2	0.0	1.0	1.0	1.0
METHANOL	0.8	0.0	0.0	1.0	0.8	1.0	0.0	0.0	0.0

Differential Scanning Calorimetry

Figure S8. Differential scanning calorimetric analysis of Regioregular Poly(1S,2S,4R-limonene carbonate) (11) using conditions from Entry 3, Table 1



Appendix B: Economic Calculations

Polymerization Equipment Costs				
<i>Displayed Text</i>	<i>Description</i>	<i>Size</i>	<i>Cost (1997)</i>	<i>Cost (2005)</i>
C1	Carbon Dioxide Tank			
D1	Distillation Column	2 trays, 0.5 m diameter	\$2,500	\$2,697
D2	Distillation Column	2 trays, 0.5 m diameter	\$2,500	\$2,697
D3	Distillation Column	2 trays, 0.5 m diameter	\$2,500	\$2,697
F1	Rotary Vacuum Filter	0.5 m ²	\$30,000	\$32,368
F2	Rotary Vacuum Filter	0.5 m ²	\$30,000	\$32,368
F3	Rotary Vacuum Filter	0.5 m ²	\$30,000	\$32,368
R1	PFR	12.872 m ³	\$115,500	\$124,618
R2	PFR	5.024 m ³	\$70,000	\$75,526
R3	PFR	2.392 m ³	\$47,000	\$50,711
S1	Polymer Silo	47 m ³	\$15,000	\$16,184
W1	Wash Mixing Tank	0.3 m ³	\$6,000	\$6,474
W2	Wash Mixing Tank	0.3 m ³	\$6,000	\$6,474
W3	Wash Mixing Tank	0.3 m ³	\$6,000	\$6,474
B1	Flash Tank	.15 m ³	\$2,000	\$2,158
P1	Recycle Pump			\$0
V1	Rotary Vacuum Dryer	4.65 m ² , 3.73 kW	\$75,000	\$80,921
Total Polymerization Equipment Cost				\$474,737

Limonene Oxide Process Equipment Costs				
<i>Displayed Text</i>	<i>Description</i>	<i>Size</i>	<i>Cost (1997)</i>	<i>Cost (2005)</i>
D1	Distillation Column 1	0.5 m dia., 10 trays	\$13,400	\$14,458
D2	Distillation Column 2	0.5 m dia., 4 trays	\$5,400	\$5,826
D3	Distillation Column 3	0.5 m dia., 13 trays	\$17,500	\$18,882
D4	Distillation Column 4	0.5 m dia., 13 trays	\$17,500	\$18,882
P1	Pump 1			
P2	Pump 2			
P3	Pump 3			
R1	Slurry Reactor	3000gal	\$25,000	\$26,974
T1	Orange Oil Tank 1	2.3E4gal	\$77,000	\$83,079
T2	Orange Oil Tank 2	2.3E4gal	\$77,000	\$83,079
T3	Orange Oil Tank 3	2.3E4gal	\$77,000	\$83,079
T4	Limonene Tank	9E3gal	\$57,000	\$61,500
T5	TBHP Tank	9E3gal	\$57,000	\$61,500
Total Polymerization Equipment Cost				\$457,258

Total Plant Equipment Costs			\$931,995
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Estimated Chemical Prices

Year	Orange Oil (\$/lb)	TBHP (\$/lb)	Carbon Dioxide (\$/lb)	Methanol (\$/lb)	Acetone (\$/lb)	Polystyrene (\$/lb)	TBA (\$/lb)
1	\$0.77	\$0.70	\$0.10	\$0.14	\$0.37	\$0.90	\$0.67
2	\$0.79	\$0.71	\$0.10	\$0.14	\$0.38	\$0.92	\$0.68
3	\$0.80	\$0.73	\$0.10	\$0.15	\$0.38	\$0.94	\$0.70
4	\$0.82	\$0.74	\$0.11	\$0.15	\$0.39	\$0.96	\$0.71
5	\$0.83	\$0.76	\$0.11	\$0.15	\$0.40	\$0.97	\$0.73
6	\$0.85	\$0.77	\$0.11	\$0.15	\$0.41	\$0.99	\$0.74
7	\$0.87	\$0.79	\$0.11	\$0.16	\$0.42	\$1.01	\$0.75
8	\$0.88	\$0.80	\$0.11	\$0.16	\$0.43	\$1.03	\$0.77
9	\$0.90	\$0.82	\$0.12	\$0.16	\$0.43	\$1.05	\$0.79
10	\$0.92	\$0.84	\$0.12	\$0.17	\$0.44	\$1.08	\$0.80

Year	Polystyrene Rate (lb/year)	TBA Rate (lb/year)	Revenue (\$/year)	Product Cost	Gross Profit	Depreciation Rate (MACRS)	Depreciation
1	33,104,040	14,585,400	\$39,565,854	\$47,751,744	-\$8,185,890	0.1429	\$884,861
2	33,104,040	14,585,400	\$40,357,171	\$48,706,779	-\$8,349,608	0.2449	\$1,516,462
3	33,104,040	14,585,400	\$41,164,315	\$49,680,914	-\$8,516,600	0.1749	\$1,083,011
4	33,104,040	14,585,400	\$41,987,601	\$50,674,533	-\$8,686,932	0.1249	\$773,402
5	33,104,040	14,585,400	\$42,827,353	\$51,688,023	-\$8,860,670	0.0893	\$552,961
6	33,104,040	14,585,400	\$43,683,900	\$52,721,784	-\$9,037,884	0.0892	\$552,342
7	33,104,040	14,585,400	\$44,557,578	\$53,776,219	-\$9,218,641	0.0893	\$552,961
8	33,104,040	14,585,400	\$45,448,729	\$54,851,744	-\$9,403,014	0.0446	\$276,171
9	33,104,040	14,585,400	\$46,357,704	\$55,948,779	-\$9,591,075	0.0000	\$0
10	33,104,040	14,585,400	\$47,284,858	\$57,067,754	-\$9,782,896	0.0000	\$0

Year	Taxes	Net Profit	Cash Flow	CFi/(1+r) ⁱ
1	\$0	-\$9,070,751	-\$8,185,890	7441717.981
2	\$0	-\$9,866,070	-\$8,349,608	6900502.128
3	\$0	-\$9,599,610	-\$8,516,600	6398647.428
4	\$0	-\$9,460,334	-\$8,686,932	5933291.251
5	\$0	-\$9,413,631	-\$8,860,670	-5501779.16
6	\$0	-\$9,590,225	-\$9,037,884	5101649.767
7	\$0	-\$9,771,602	-\$9,218,641	4730620.693
8	\$0	-\$9,679,185	-\$9,403,014	4386575.551
9	\$0	-\$9,591,075	-\$9,591,075	4067551.875
10	\$0	-\$9,782,896	-\$9,782,896	-3771729.92

Inflation Rate	0.02
Minimum ROI	0.1

Return on Investment, ROI	-7.44
Net Present Worth	\$61,097,667

Economic Simulations			
ROI	0.1	0.15	0.2
Price of Polystyrene	\$1.18	\$1.19	\$1.19