Stevens Institute of Technology

Final Project

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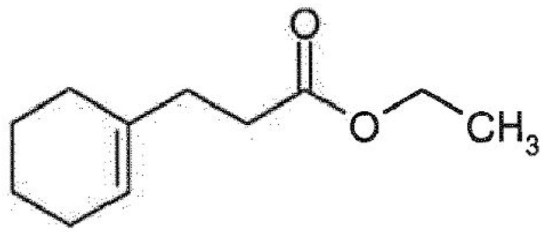
CHE345: Chemical Process Control, Modeling, and Simulation
Group 6

Introduction

In our world, the ability to smell is a significant sense because we often see the world through our noses. There is an ongoing need in the fragrance industry to provide new chemicals to give perfumers and other people the ability to create new fragrances for perfumes, colognes, and personal care products. When analyzing the chemical structure of a scent, the smallest of differences could make a smell go from pleasing to malodorous. Because it is hard for even professionals to predict whether or not certain chemical formulas will have a pleasing odor, there is an effort in the fragrance industry has been made to provide new chemicals that are used to treat and control malodors. Malodors include body perspiration, smoke, environmental odors, and other aromas that can be masked using different perfumes by either blending with the malodor to provide a new chemical formula, or by overwhelming the malodor entirely. The invention created in this patent is the formation of 3-(cyclohex-1-en-1-yl) propionates to improve, enhance and modify fragrance formulation. The patent focuses on producing three different structures: ethyl 3-(cyclohex-1-en-1-yl) propionate, 2-propyl 3-(cyclohex-1-en-1-yl) propionate, and allyl 3-(cyclohex-1-en-1-yl) propionate. However, for simplicity this report and its models will solely deal with the preparation of ethyl 3-(cyclohex-1-en-1-yl) propionate.



Structure I

Figure 1: Ethyl 3-(cyclohex-1-en-1-yl) propionate chemical structure

In this project, the preparation of ethyl 3-(cyclohex-1-en-1-yl) propionate will be thoroughly examined and modeled using Simulink. By examining the inputs and outputs of the reactor, the control, manipulated and disturbance variables can be defined, and a process model and control system can be determined. The possible safety features surrounding the process will also be examined to ensure the protection of the individuals working in the vicinity of the process as well as preservation of the reactor, catalyst regeneration zone and other process parts. There are many steps to this preparation. The first step of this process is to heat a combination of specific amounts of bicyclononane, butanol, and methane-sulfuric acid in a reaction flask to reflux, cool this mixture, neutralize this mixture, and then flash vacuum distill the solution to afford crude butyl 3-(cyclohex-1-en-1-yl) propionate. Once this occurs, specific amounts of ethanol and methane sulfuric acid is added to the propionate to then heat is to reflux and eventually cool to an ambient temperature and neutralize the solution. Once the organic layer is removed, the process is finished to produce 379 grams of ethyl 3-(cyclohex-1-en-1-yl) propionate with a boiling point range of 127-131 degrees Celsius and a pressure of 3 mmHg.

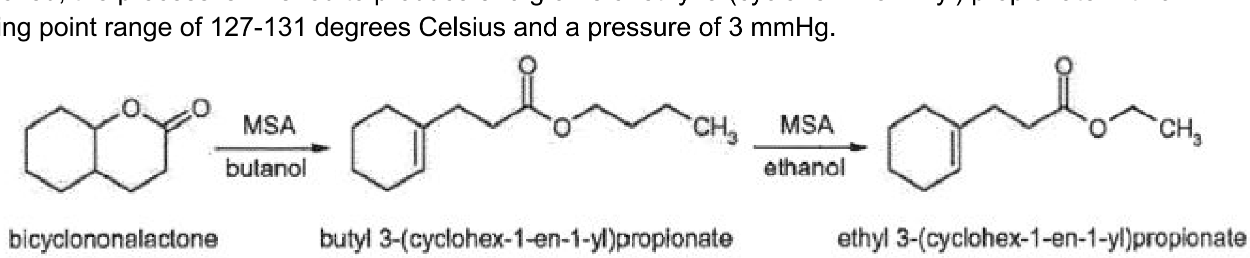


Figure 2: Step-by-step mechanism for the preparation of ethyl 3-(cyclohex-1-en-1-yl) propionate

This report will use the information given in the procedure to prepare ethyl 3-(cyclohex-1-en-1-yl) propionate, evaluate control objectives, maximize efficiency and safety, and then model this reaction via Simulink.

Control Objectives

For this system, we needed to consider different types of variables. The control variables refer to the portion of the process that is going to be kept constant, or meet a given set point. For the preparation of ethyl 3-(cyclohex-1-en-1-yl) propionate, we have identified two control variables for this reaction that we plan on modeling.

The two control variables in the model are temperature and length of reaction within the cell because this is a time sensitive batch reaction. These variables are the outputs to which set points, or desired values, are assigned. The set points to be modeled are ~25°C for room/ambient temperature and then the vessel is controlled to ~115°C where the reaction occurs. After approximately 5 hours, the reactor vessel must be controlled back down to ambient temperature before the further steps of the process can occur. The outputs could differ from their set points due to disturbance variables, leading to an error. The disturbance variables that affect temperature is the starting room temperature of the room which affects the temperature that the reaction begins with. This, in turn, affects the time the process will take depending on if it takes more or less time for the reaction vessel to reach ~115°C. This emphasizes how the two control variables are closely involved with each other. To correct the error in the outputs, manipulated variables are changed via feedback loops so that the set points are reached. The amount of heat input into the system can then be adjusted in order to heat the reaction vessel to the required temperature range and then the time range is then adjusted adjusted accordingly.

Control Strategies and Instrumentation

The model utilizes a feedback control system in which the error in the controlled variables was measured and the manipulated variables changed accordingly. In this type of control system, the manipulated variables are adjusted after the control variable becomes too high or low in order to compensate for the error. In choosing which control system to use, the following advantages and disadvantages of feedback were taken into account:

Pros	**Cons**
Corrective action is taken regardless of the source of the disturbance.	No corrective action occurs until after the disturbance has upset the process.
Reduces sensitivity of the controlled variable to disturbances and changes in the process.	Very oscillatory responses, or even instability.

Ultimately, the feedback control was chosen due to the additional advantage of relating temperature and concentration to each other more easily. As can be seen above, there are feedback loops connecting the output to earlier stages in the device; this allows the error in the control variables to affect the manipulated variables earlier in the process.

Safety Features

It is always important to consider any potential safety concerns that could arise when conducting the process. The two main safety concerns within the modeled process include high pressures and high temperatures. It is important for the reactor to stay within operating conditions and not fail due to overheating or a pressure that is too high to control. Several checks can be put in place in order to ensure the safety of the system and provide a safe environment for those working in it. A series of sensors will be set to be triggered to alert the facility to follow necessary evacuation protocol if the system reaches dangerously high pressures or temperatures. These sensors would measure operating conditions throughout the preparation process. If the sensor reads a pressure that exceeds the set maximum temperature and pressure, the system would trigger a release valve that would either release heat to cool the reactor or release pressure by releasing some of the steam before the water is collected in the Dean Stark trap to hopefully release some pressure.

If this safety feature fails to work, the system would also have rupture discs in place. A rupture disc is a non-reclosing pressure relief device that, in most uses, protects a pressure vessel, equipment or system from overpressurization or potentially damaging vacuum conditions. Therefore, these discs would rupture at a certain pressure and release a chemical cooling agent to attempt to reduce the pressure by reducing the temperature. Since temperature and pressure are directly related the cooling agent would hopefully reduce the temperature enough to bring the system down from the dangerously high pressure in order to return the system to a safe environment.

Throughout the completion of the process, the temperature will be constantly measured and recorded for the sake of tracking the accuracy of this experiment even if safety wasn't a concern. Since this process deals with organic compounds, there is always a hazard of combustion. If O2 were to get into the reactor and reach a certain temperature, combustion would occur. Fortunately, the combustion temperature of methane is well over the reaction temperature. As a precaution, temperature sensors are placed in the reactor to monitor the temperature in case there is a runaway with the reaction. In case of a drastic temperature increase, alarms would sound and a sprinkler system would go on stand-by. The alarm temperature is set under the combustion temperature of methane in order to give engineers time to assess and solve the temperature increase.

Process Model Quality and Complexity

In order to control the process, the Simulink model shown in *Figure 1* was designed to incorporate the transfer functions that allow the control of the temperature and duration of the reaction while correcting for possible disturbances. It incorporates the temperature increase and temperature decrease through the transfer functions, and also contains the time delay to hold the temperature constant for 6 hours. The PID controller is the component that calculates the error from outside noise in order to adjust the resulting temperature to reach the set point. The graph displayed in *Figure 2* demonstrates that the Simulink model successfully controls the temperature and duration of the reaction to the desired values.

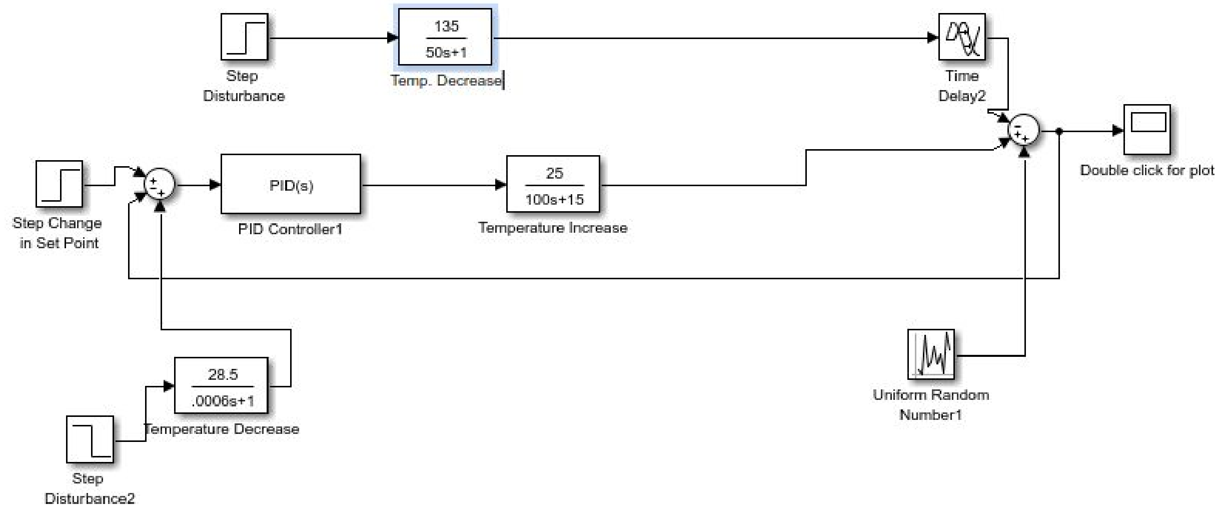


Figure 1

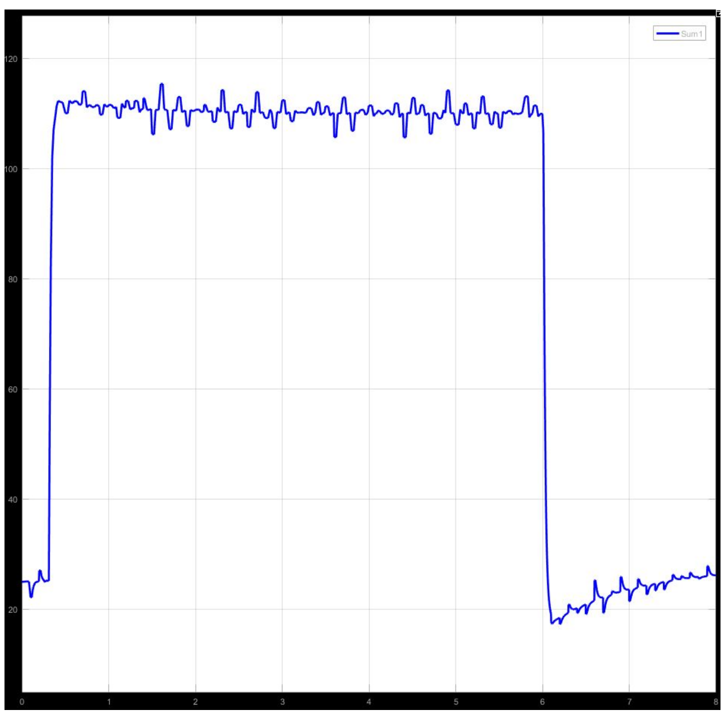


Figure 2

Analysis of Controller Settings and Stability

The most important components of our model are the transfer functions. The transfer functions that are implemented into the model display the change between our input and output values. The transfer functions relate the temperature increase and the temperature decrease that occur in the model. The constants that are used were arbitrary selected since the patent literature did not give exact values for tau or K. We found these values by increasing or decreasing the values based on the output of the graph.

Another key component of the model was the uniform random number. The uniform random number allowed us to start at a different number for ambient temperature to account for the real world aspects that temperature is not always constant. The final components of the model was the PID controller, which calculates an error value pertaining to the measured controlled variable value and the desired set point. The PID controller is what allows the model to run as smoothly as it did. The proportional control concerns the present error, the integral control relates to the past errors, and the derivative control deals with the prediction of future errors. The values that were found from tuning the controller help simulate a real world plant.

Conclusions

There is a need within the fragrance industry to improve fragrance compositions with a compound that will counteract, treat and control malodors. This can enhance the fragrance industry by allowing for a perfume that can either mask the malodors entirely or blend to neutralize the malodors. The patent for the creation of ethyl 3-(cyclohex-1-en-1-yl) propionate can be the solution. The process control was designed and modeled to control a part of the process in which the mixture is heated and maintained for 6 hours, and then cooled to an ambient temperature. This feedback control system allows for the adjustment of the manipulated heat input in order to maintain the desired temperature for the correct duration of time. Layers of safety features such as sensors and protective rupture discs allow for this process control to be feasible and to avoid any hazards. The Simulink model of transfer functions that create the feedback control system yield the results desired based on the control variables. The transfer functions make up the bulk of the Simulink model by relating the temperature changes that occur in the process. By modeling the process through the transfer functions and thus through the Simulink model, the process can be controlled and the ethyl 3-(cyclohex-1-en-1-yl) propionate can be used to enhance the fragrance industry.