

DEVELOPING CHEMICAL ENGINEERING ACUMEN THROUGH OPTIMIZATION OF THE CHOCOLATE PROCESS

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INTRODUCTION

Maintaining interest and motivation in science education continues to decline with increasing age.^[1] However, extracurricular activities and guided inquiry laboratory experiences have been shown to promote interest.^[2] Adams et al. surveyed alumni from the 2007-2013 Aspiring Scientists Summer Internship Programs and showed that 83.9% of those surveyed better understood classroom material after hands-on research experiences.^[2] Additionally, universities have adopted the use of chocolate production to develop chemical engineering acumen.^[3-5] Both Rowan University and Villanova University use the production of chocolate as an introduction to chemical engineering for freshmen and sophomores. Thus, the United States Military Academy's (USMA) Chocolate Club, a sub-group of the Academy's American Institute of Chemical Engineers (AIChE) Student Chapter, has adapted a similar process to increase interest in the sciences and introduce chemical engineering acumen through experimentation with the production of chocolate. However, USMA's Chocolate Club takes a different approach and advances the development of chemical engineering acumen past the freshmen and sophomore levels through enhanced projects. Different aspects of the projects focus on different engineering topics that challenge all four-year cohorts. For example, development of flow diagrams target the junior-class student and optimization of models target senior-class students.

Each year the Chocolate Club identifies an overall goal, such as optimizing the chocolate production process, identifying the optimal mathematical model for quality control, or analyzing the differences in chocolate produced from cocoa nibs of various countries. The procedural development is then laid out, which includes the design of the chocolate itself and postprocessing analysis (quality control, identifying metal content or fatty acid content, etc.), all aimed at accomplishing the overall objective. The design of the chocolate includes

the type of product (such as 70% dark chocolate), ingredients, mixing standards, and refinement time. Through these projects that vary each year, cadets expand their knowledge of chemical engineering topics, troubleshooting, computer programming, optimization, and analytical equipment. This differs from current implementation at other universities by promoting the development of chemical engineering knowledge beyond the freshmen and sophomore years. Juniors and seniors work together to achieve an overall goal even though they may have different roles in the process. In order to explain the club's development of chemical engineering knowledge through the production of chocolate, the optimization of the chocolate process is discussed (the first project completed by the club), beginning with an overview of the chocolate process.

Cocoa plantations were first established in 600 AD by the Aztecs and over time have evolved to the chocolate we know today.^[6] There are three main types of chocolate – white, milk, and dark – and the composition of the chocolate distinguishes the type. White chocolate contains no cocoa liquor (only cocoa butter), milk chocolate contains approximately 10% chocolate liquor, and dark chocolate contains at least 40% chocolate liquor.^[7] Chocolate is mainly comprised of cocoa liquor, cocoa butter, sugar, milk, and soy lecithin (used as an emulsion). Although there are few ingredients, the production process is complex, and care must be taken to produce a smooth product that appeals to the consumer.

The unique flavor of chocolate is developed through a multistep process consisting of bean collection, fermentation, cocoa nib (crushed cocoa beans) separation, cocoa nib roasting, liquor processing, mixing, refining, tempering, and molding, dipping, or extrusion for the final product.^[6,7] After the beans are collected, they are placed in piles or in boxes for fermentation. During the fermentation process, enzymes

are released that cause rapid decomposition leading to sugars and acids that are precursors to the characteristic chocolate flavor.^[6] After the cocoa nibs are separated from the beans, they are roasted to bring out the chocolate flavor. The roasting process also removes the volatile acids to make the nibs less acidic.^[6] The juicing process (liquor process) reduces the nibs to smaller particles and releases the fat that helps the chocolate flow. During the mixing and refining steps, all ingredients are combined and placed into a melanger (similar to a motorized mortar and pestle). The melanger is used to break the particles into smaller pieces and coat the particles with fat, a process known as refinement. After the chocolate is refined, it goes through the tempering process. The tempering process reestablishes the stable form of cocoa butter which is Phase V. There are six polymorphic forms in cocoa butter; the difference is the packing configuration. The shape of the fat molecules lends itself to different configurations; the denser the configuration, the higher the melting point. Phase V is the most desirable form that gives chocolate a glossy appearance with a good snap when broken (a crisp break) and the right melting point to melt in the mouth.^[6] The other phases have lower melting temperatures and dull appearances. The tempering device heats and cools the chocolate to melt the unstable crystals and form the stable crystal structure. This is aided by the addition of seed chocolates (a small piece of tempered chocolate).^[8] Finally, the chocolate is ready to either be placed in molds, dipped, or extruded. The execution of all these steps affects the flow properties of the chocolate and defines texture, melting characteristics, and taste.^[9,10] To quantitatively verify the appropriate flow properties in industry, chemical engineers apply rheology, the study of flow.

Controlling the rheological properties is key to effective mixing, pumping, and taste^[11] and was used by the club for quality control. In the chocolate industry there are two parameters that are widely accepted to describe the flow properties of chocolate: Casson yield stress (τ_C) and Casson plastic viscosity (μ_C). These two parameters are key components of the taste quality. The Casson yield stress corresponds to the energy required to initiate flow, and the Casson plastic viscosity corresponds to the energy required to maintain flow.^[12,13] The Casson model, calculated using Equation 1

$$\sqrt{\tau} = \sqrt{\tau_C} + \sqrt{\mu_C} \cdot \sqrt{\dot{\gamma}} \quad (1)$$

where τ is the shear stress, τ_C is the Casson yield stress, μ_C is the Casson plastic viscosity, and $\dot{\gamma}$ is the shear rate, is widely accepted in the chocolate industry. It is used in this study and is fitted to shear stress-shear rate data to identify these parameters. There are various optimization tools available to fit specific models to the experimental data for parameter analysis. One method is the genetic algorithm (GA), an evolutionary, global, stochastic computation optimization method. GA seeks a global optimum by minimizing a defined cost function. The initial settings are based on Darwin's discovery that nature adapts to survival,^[14-16] such as a population that is composed

of individuals (proposed solutions) or often termed chromosomes.^[16] The individuals are a string of numbers (0s and 1s) that define the parameters, or in the case of this study, τ_C and μ_C . The program goes through a set number of iterations, and each iteration determines the fittest individuals (parameter that results in the lowest error) to make up the new population until the program converges on a solution for which the error is minimized.

Rheological properties are then compared to identify the optimal milk chocolate procedure, i.e. the overall objective. Cadets proposed two steps that could have the greatest impact on these properties. The parameters chosen for this study were type of seed chocolate (tempered chocolate) and refining time (time in the melanger). The two parameters were varied, samples were analyzed and compared using rheological data, and a blind taste test was conducted using members of USMA's AIChE student chapter as taste testers.

MATERIALS AND METHODS

The cadets involved in the Chocolate Club range overall four-year groups (freshmen to seniors) and vary in the hours that they devote to the projects. Some members are formally registered for research credit hours while others are volunteers. Generally, the initial appeal to the club was the love of chocolate, as unanimously stated by the cadets, but as they design the projects, execute the procedures, and analyze rheological data or chocolate samples, they begin to connect the concepts to chemical engineering and analytical techniques.

USMA's Chocolate Club purchased cocoa nibs already separated from cocoa beans and therefore started with the roasting step. All batches of chocolate were made using the same procedures and varied only with the type of seed chocolate used or the length of time chocolate was refined in the melanger. To produce milk chocolate, 12 ounces of chocolate nibs ordered from www.nut.com were roasted in a conventional oven for 10 minutes at 350°F, 5 minutes at 325°F, 5 minutes at 300°F, and then 10 minutes at 275°F. Following roasting, the chocolate nibs were juiced using a Champion Heavy-Duty Commercial Juicer (rerunning the waste stream three times). The chocolate liquor was mixed with 12 ounces of granulated sugar, 6.05 ounces of dehydrated milk products, 12.02 ounces of melted cocoa butter (melted using an electric stove), and 2 teaspoons of soy lecithin in a bowl. The mixture was then added to the melanger (Spectra Stone Melanger) for a range of 72 to 120 hours prior to tempering (see Figure 1).

After the chocolate was removed from the melanger, it went through the tempering process using a ChocoVision Tempering Machine. The pre-programmed milk chocolate setting on the temperer was used. After the chocolate was heated in the temperer, the seed chocolate was added, and the chocolate continued through the tempering process. After

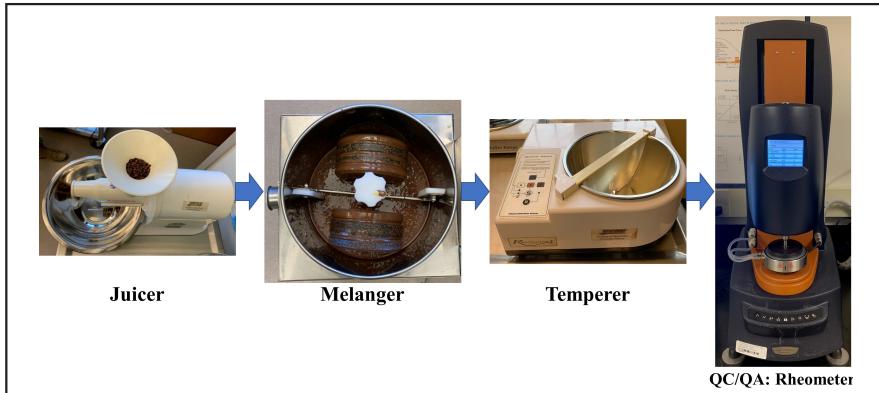


Figure 1. *Chocolate Process: The Chocolate Club begins with roasting cocoa nibs. Roasted nibs are placed in the juicer and then combined with all other ingredients in the melanger. Following the melanger, the chocolate is then tempered, and later samples are tested to verify mechanical properties to ensure quality control.*

tempering, chocolate was placed in chocolate molds using syringes. The chocolate was then placed in the refrigerator for cooling overnight.

To study the effects of seed chocolate on the final product, three types of seeds were used: a piece of Hershey’s milk chocolate, cocoa butter,^[17] and a piece of chocolate from a batch previously made using cocoa butter as the seed. After samples of the chocolate from each of these three batches were cooled overnight, stress versus shear-rate data were collected (for each sample) using a Discovery Hybrid Rheometer – 3. A cone and plate geometry (40 mm cone diameter and 2° cone angle) was used with a temperature control Peltier plate. A small chocolate sample from each batch was heated to 45°C until melted. The temperature was then maintained at 37°C, and data were collected for a shear-rate range of 0.1 to 700 1/s on a logarithmic scale.

The second parameter studied was melanger time. Samples of one batch of chocolate were taken at various time intervals

while in the melanger — trials 4-7 shown in Table 1 — to study the effects of refinement time. A sample was taken after 2 days in the melanger, 3 days in the melanger, 5 days pre-tempering, and 5 days post-tempering (for overall comparison). Chocolate from 5 days in the melanger was tempered using a seed from a cocoa butter seed-chocolate sample. Stress and shear-rate data were collected using the same parameters as discussed above.

The data collected from all trials were fitted using the linearized Casson model in Equation 1. For this project, the optimal parameters (τ_C and μ_C) for each sample were identified using Microsoft Excel® and a genetic algorithm optimization code facilitated by MATLAB®. This comparison showed the effectiveness of optimization tools and was recommended by the faculty members.

Rheological data were recorded for each second of data collection. Cadets averaged the final 30 values of the stress after reaching steady state at each shear rate value of the steady state flow curve. Between each shear rate study, a pre-shear protocol was run before going to each new shear rate to eliminate structural “history” effects. The parameters of the linearized form of the Casson equation were determined by plotting the data (shear rate versus shear stress) in Excel and using Excel’s built-in trendline function. The parameters were also calculated using the genetic algorithm. To initiate the genetic algorithm code, the following parameters were required: the number of parameters, array length, keep rate, mutation rate, and number of iterations.^[14-16] The number of parameters (number of unknowns) was two for the Casson model (τ_C and μ_C). The array length (number of stored solutions) was held at 24. The keep rate (rate of individuals kept for mating) was 50%, and the mutation rate was held at 25%. Following the declaration of parameters, the population was initialized using starting guesses. After each iteration the shear stress of each individual in the population was calculated and compared to the experimental shear stress using Equation 2.

$$error = \sum_i^N \left(\frac{y_i - f_i}{y_i} \right)^2 / N \quad (2)$$

where y_i is the raw shear stress, f_i is the calculated shear stress, and N is the number of iterations. The 24 individuals in the array were then rank ordered with the lowest error assigned to the first array. This completes one iteration, and the process was repeated until the total number of predefined iterations was met.

Trial	Seed	Melanger (hrs)	Tempered
1	Hershey	72	Yes
2	Cocoa Butter	72	Yes
3	Chocolate from Cocoa Butter	72	Yes
4	None	48	No
5	None	72	No
6	None	120	No
7	Chocolate from Cocoa Butter	120	Yes

Finally, a blind taste test was conducted in order to correlate the optimal parameters with the most desired chocolate. The blind taste test was conducted during an AIChE student chapter meeting. Each sample was given a number 1 through 4 to prevent members from knowing the difference between samples. Samples 4 through 7 were not part of the blind taste test since they did not go through the tempering process. The samples tasted are shown in Table 2. Members tasted each sample and placed their vote card in the numbered cup that correlated to the chocolate they preferred. The chocolate that had the most votes was deemed the best chocolate, with each person's vote counting as a single vote. There were 30 cadets who voted, and each cadet cleared their mouth with water between each sample. The order of testing was varied per cadet.

Sample	Seed	Melanger (hrs)	Tempered
1	Hershey	72	Yes
2	Cocoa Butter	72	Yes
3	Chocolate from Cocoa Butter	72	Yes
4	Chocolate from Cocoa Butter	120	Yes

RESULTS

The Casson yield stress and plastic viscosity parameters calculated using the genetic algorithm for all seven trials are shown in Table 3. Shear rate versus shear stress for all seven trials are shown in Figure 2. The results of the blind taste test are shown in Table 4.

Trial	Seed	Melanger (hrs)	Tempered	Casson Yield Stress (Pa)	Casson Plastic Viscosity (N·s)
1	Hershey	72	Yes	5.0	0.40
2	Cocoa Butter	72	Yes	4.2	0.54
3	Chocolate Seed from Cocoa Butter	72	Yes	2.0	0.62
4	None	48	No	5.2	0.73
5	None	72	No	6.6	0.75
6	None	120	No	9.5	0.73
7	Chocolate Seed from Cocoa Butter	120	Yes	8.5	0.65

Seed	Melanger (hrs)	Rank
Chocolate from Cocoa Butter	120	1
Hershey	72	2
Cocoa Butter	72	3
Chocolate from Cocoa Butter	72	3

DISCUSSION

Comparing the blind taste test with the calculated Casson parameters (yield stress and plastic viscosity) showed that the preferred chocolate correlated to higher Casson parameter values – more specifically, a higher Casson yield stress since the Hershey seed chocolate batch, which was a close second, had the lowest Casson plastic viscosity. From Figure 2a, one can conclude that seed chocolate had minimal impact on the rheological properties; however, more trials should be conducted for accurate statistical analysis (statistical analysis was not an overall objective for this lab). For this experiment, rheological data from two samples of each of the seven batches of chocolate were obtained and visually compared. The data did not deviate significantly, so only one dataset was used to calculate the rheological parameters. Figure 2b shows how the parameters increase with additional refinement time. However, the tempering process causes a slight reduction in the overall parameters. This was unexpected since the tempering process promotes a crystal structure that would require a higher energy to initiate and maintain the flow. Future studies will involve using the differential scanning calorimeter to determine if the chocolate produced is tempered and has Phase V structures.

An additional objective for this project was using differ-

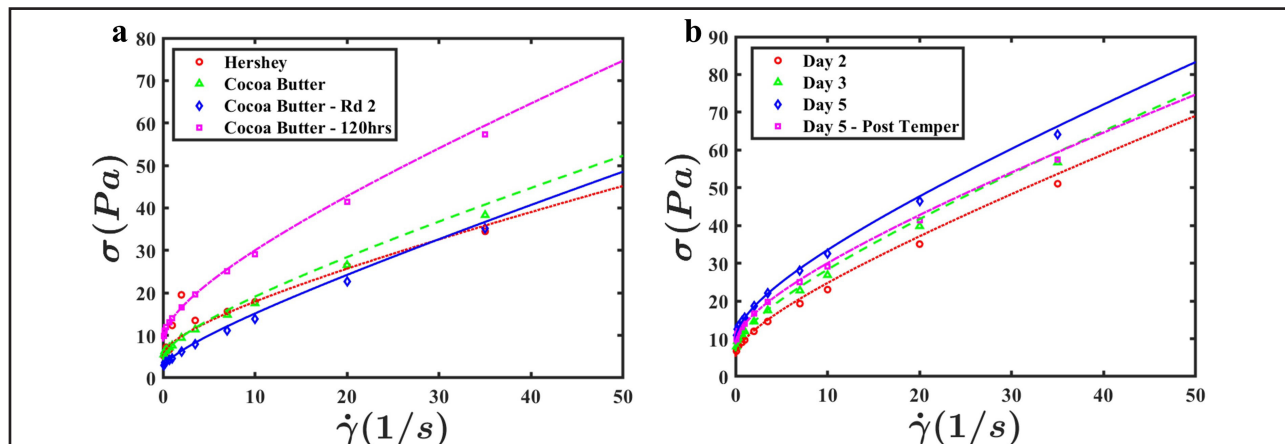


Figure 2. a) Shear rate versus shear stress for the three seed trials is shown in the figure along with the data from the most desired chocolate as determined by the AIChE student chapter taste testers. Solid lines are the Casson Models using the calculated parameters (yield stress and plastic viscosity) from Table 3. b) Shear rate versus shear stress for the timed trials.

ent optimization tools to fit models to data such as Microsoft Excel and the genetic algorithm facilitated by MATLAB. Both models for the cocoa butter seed chocolate are shown in Figure 3. The error analysis showed that the genetic algorithm produced a better fit than Excel's trendline function. This enlightened the cadets on optimization tools. A wave oscillation was observed for the Hershey's seed chocolate rheological data. As a result, the experiment was repeated three times, and the same wave oscillation occurred in all three trials. To further examine this anomaly, the rheological data from a sample from an earlier batch using a Hershey seed were collected, and no wave oscillation was observed. The batch used for this additional analysis was an older batch. As chocolate sits, the crystal structures start morphing into different structures. In other words, Phase V structures start morphing into Phase VI, etc.^[6] This could be the reason the wave oscillation was not observed in this batch. Future studies will examine this anomaly.

CONCLUSION

Overall, the project taught the cadets how to make chocolate, optimize the process, develop flow diagrams, understand various forms of heat exchangers, use optimization codes, and appreciate the need for triplicates for statistical analysis. Altering the procedures, studying the science behind each step in the process, and analyzing the data allowed cadets to gain a deeper understanding of many chemical engineering topics. The faculty observed this increase in chemical engineering knowledge through flow diagrams drawn (top portion of Figure 4) in the lab, discussions about technical aspects of the steps, and cadets relating coursework in their chemical engineering classes to aspects of the chocolate process as seen in the bottom portion of Figure 4.

They also gained knowledge through troubleshooting. For example, when we first started the initial project, the chocolate

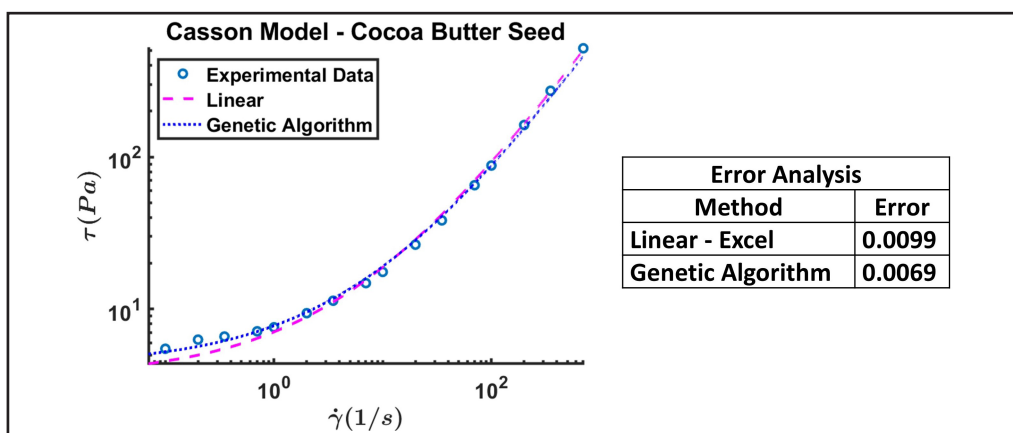


Figure 3. Comparison of a linear fit using Excel's built-in trendline function versus an optimization tool, in this case genetic algorithm. There was a better fit to the experimental data using the genetic algorithm as seen in the error analysis using Eq 2.

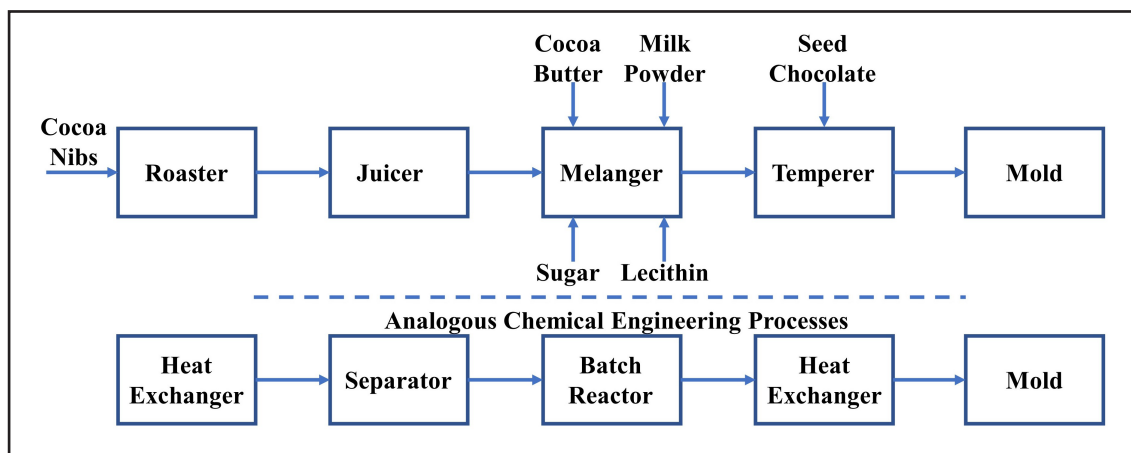


Figure 4. Connecting the chocolate process with chemical engineering topics.

was solidifying, which could have been a result of an error in the procedures. Over multiple trials, cadets realized that the temperature of the room was below the melting point of chocolate. They identified the problem and then further analyzed the procedures to identify a solution to the problem. They determined that the heat generated from the melanger raised the temperature back above the melting point, that was then used to fix the low temperature problem.

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