Effect of Temperature on Rock Candy Mass

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Abstract:

Rock candy is a popular form of sugar in different cultures, replacing castor sugar in coffee or tea. Given the popularity of homemade rock candy, understanding the optimal manufacturing conditions is important. Previously, the optimal manufacturing conditions have been studied at an industrial level to increase the efficiency of rock candy production. In the aforementioned previous study, it was recommended that rock candy be prepared using sugar solutions heated to 80°C. The goal of this experiment is to determine the effect that the temperature of a rock candy sugar solution has on the final mass (g) of rock candy in a non-industrial environment. An online rock candy recipe was modified in our experiment to examine the effect of differing temperatures (60°C, 70°C, 80°C, 90°C) on final rock candy mass (g) grown over 7 days. Contrary to studies performed at an industrial level, we found that solutions heated to 90°C experienced significantly greater rock candy mass growth than solutions heated to 60°C, 70°C and 80°C. Additionally, solutions heated to 60°C, 70°C and 80°C experienced nonsignificant differences in mass growth. The applications of this study are that knowledge of rock candy yield based on temperature can save expenses on heating for small-scale production of rock candy.

Introduction:

In a normal household, sugar is a commonly used table condiment due to its versatility in all types of food products. Specifically, rock candy is a special sugar commodity originating from India and Persia, where it is referred to as Mishri and Nabat respectively (Gholamhosseinpour et al., 2008a). Crystallization is one of the primary methods for the production of rock candy (Lotfabadi et al., 2020), which involves supersaturation of a sucrose solution followed by cooling, resulting in rock candy (Gholamhosseinpour et al., 2008a). Numerous rock candy recipes have vague instructions, with some suggesting to cook the sugar solution until all white sugar is dissolved (LaBau, 2021), or to bring the solution to a boil (Taylor, 2020). 80°C is a recommended temperature for industrial rock candy production in a multivariable study of rock candy formation (Gholamhosseinpour et al., 2008b), and in our experiment 80°C is used as a control. The objective of this experiment is to examine how heating a sugar-water solution to different temperatures (60°C, 70°C, 80°C, 90°C) impacts the final mass of rock candy. We hypothesize that if recrystallization is used to form rock candy, then increased temperatures in terms of degree Celsius (°C) of a heated sugar solution will result in rock candy having a larger mass in grams (g). At higher temperatures, solutions can become increasingly concentrated, due to increased solvent evaporation (Helt, 1976). As the saturation of a solution will be increased, the rate of crystallization would also be increased during the cooling process (Helt, 1976). Given the increased rate of crystallization, larger crystals should be produced. Our prediction is that as temperature increases (60°C, 70°C, 80°C, and 90°C), rock candy mass will also increase. This experiment holds important implications in the world of food science and the production of sugar crystals for consumption. Ideally, this study can be used to reduce the cost of ingredients and heating required to make delicious rock candy at home or in small businesses, by determining which temperatures yield the most mass.

Methods:

Excluding the independent variable of temperature, the protocol for this experiment was modified from an online recipe (Appendix A). After calibrating the thermometer using ice water, T-shaped apparatuses were constructed using wooden skewers (8cm long) and tape. The initial apparatus was then weighed on a kitchen scale calibrated to 0.00g. 2 cups of water were added to 4 cups of sugar (i.e. 2:1 sugar to water ratio) in a pot. The pot was then placed on a stovetop and was heated on a low heat setting until the target temperature (60°C, 70°C, 80°C, or 90°C) was reached, with experimenters stirring once every 30 seconds. Once the target temperature was reached, the sugar solution was left to cool on the stove until it reached 40°C. A syrup solution was made by heating 1 cup of water and 2 cups of sugar until the sugar dissolved. Then, the end tips of the skewer apparatuses were dipped into this syrup, coated in white sugar for seeding and left to rest for 5 minutes. Subsequently, the sugar solution was poured into a 16 oz mason jar until the fill line (10cm from the bottom of the jar) was reached, with the skewers inserted afterwards. The skewers were taped to the mason jars to ensure they stayed in place, and did not touch the walls of the jar (Figure 1). In total, this process was done 4 times at each target temperature (60°C, 70°C, 80°C, 90°C) to get 4 mason jars per treatment. The mason jars were left for 7 days before weighting the rock candy with the skewers on the scale. The rock candy weight was subsequently obtained by subtracting this final weight with the initial weight of the skewer apparatus (Appendix B). To analyze this data (Appendix C), a QQ-plot was generated (Appendix D) to determine normality of the data, followed by a one-way analysis of variance test (ANOVA) assuming normality (Appendix E). The Tukey post-hoc test was conducted on the final mean measurements of each temperature treatment (Appendix F). All statistical tests were performed on Graphpad Prism 9 (version 9.0.1) software to determine whether there was a statistically significant difference in rock candy mass between the different temperatures. An alpha value of 0.05 was used to determine significance, as it is common practice in analytical science (Analytical Methods Committee, 2020).

Figure 1. Photo of one sample (Replicate A, Temperature 90°C). One can observe the proper experimental setup consisting of the wooden skewer apparatus, fill line of the sugar solution (pink marker), and taping method to secure the wooden skewer apparatus.

Results:

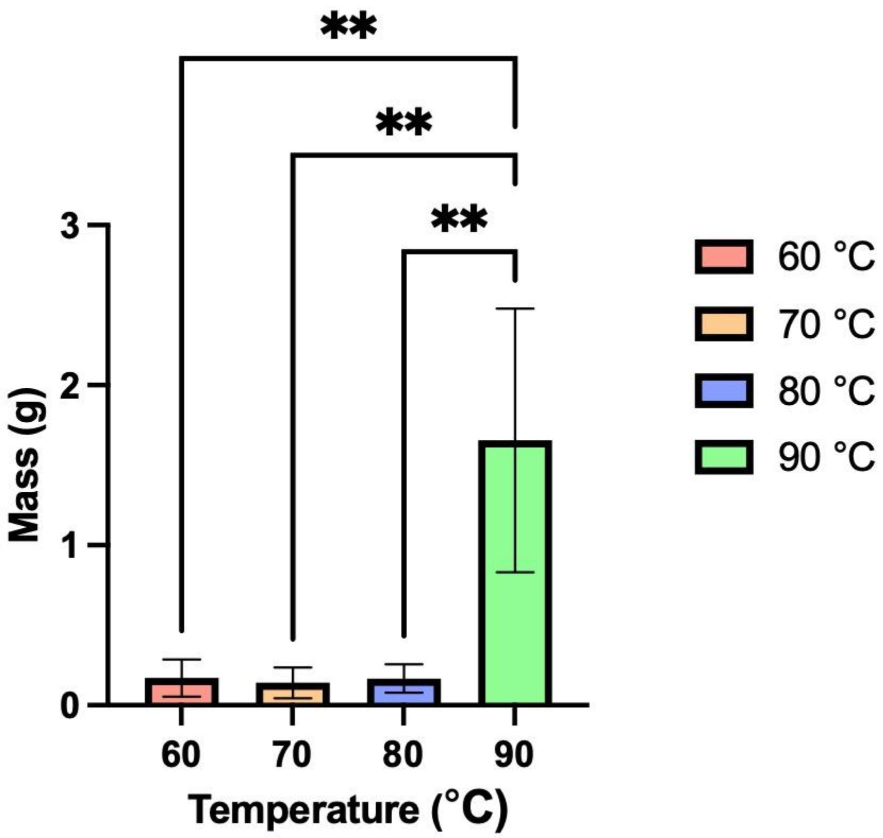

Figure 2. The mass of rock candy prepared at 90°C was significantly larger than the mass of rock candy prepared at other temperatures. Bar graphs represent the mass (g) of rock candy prepared at different temperatures (°C). Each bar represents the mean mass of each treatment (temperature). The error bars represent the standard error of the mean. A p-value of <0.001 was found after a one-way analysis of variance test. In total, there were n=16 rock candy masses measured in grams (g). 60°C (n=4), 70°C (n=4), 80°C (n=4), 90°C (n=4). The asterisks (**) indicate significant pairwise comparisons.

The calculated means of rock candy prepared were 1.66g ($\sigma_{\bar{x}}$= 0.41, n = 4) at 90°C, 0.17g ($\sigma_{\bar{x}}$= 0.04, n = 4) at 80°C, 0.14g ($\sigma_{\bar{x}}$= 0.05, n = 4) at 70°C, and 0.17g ($\sigma_{\bar{x}}$= 0.06, n = 4) at 60°C. A one-way ANOVA tests for any statistical significance in the means between three (3) or more treatment groups. The one-way ANOVA was performed assuming normality of the residuals by visually examining a QQ-plot (Appendix D). After performing a one-way ANOVA on our data, the resulting p-value was determined to be <0.001. The p-value is less than our alpha value (0.05) indicating that the differences in the mean masses of rock candy prepared at different temperatures are statistically significant.

The Tukey post-hoc analysis found that the mean mass at 90°C was significantly different from the mean masses at 60°C, 70°C and 80°C as the p-values were less than our alpha value of 0.05 (Appendix F). The p-values for the pairings 60-90°C, 70-90°C, and 80-90°C were 0.002991, 0.002613 and 0.002957 respectively. The p-values for the pairings 60-70°C, 60-80°C, and 70-80°C, were 0.99958, >0.999999 and 0.999677 respectively. Thus, the pairwise comparisons for the following pairs, 60-70°C, 60-80°C, and 70-80°C were found to be nonsignificant due to p-values greater than the alpha value of 0.05. In summary, we found that the mean mass of rock candy prepared at 90°C is significantly larger than all other temperatures (Figure 2). As well, temperatures 60°C, 70°C, and 80°C experienced non-significant differences in mean mass growth with respect to each other (Figure 2).

Qualitatively, the rock candy appeared slightly opaque, tinted a cloudy colour (Appendix G). On all skewers, we observed minor growth where the intersection would occur between the skewer and the surface of the sugar solution.

Discussion:

This study investigated whether sugar solutions prepared at different temperatures affected rock candy mass. We found that the results of our ANOVA test (p-value <0.05) rejected the null hypothesis which states temperature has no effect on rock candy mass and the Tukey post-hoc test results did not support our prediction that rock candy masses increase with increasing temperatures. We found that the 90°C treatment had a significantly larger mean mass than the other treatments of rock candy (Figure 2). This finding could be explained by the principles of crystal growth, which state that as temperatures increase, kinetic energy increases. This consequently results in sugar molecules moving faster in a solution. The increased kinetic energy of the solution can increase the evaporation of the solvent, which allows particles to precipitate out of the solution more quickly during the recrystallization process (Gutierrez-Mazzotti, 2016) and therefore form a larger mass of rock candy in a set time period.

Although 80°C was used as a control for our experiment, there was no significant difference in the mean mass of crystal growth between 80°C, 70°C, and 60°C. Even though research has suggested that 80°C is optimal for producing rock candy at an industrial scale (Gholamhosseinpour et al., 2008b), we observed little to no rock candy growth at 80°C, other than a small ring around the skewer (Appendix G). Our results suggest that due to the differences in methodology between our experiment (in our kitchens), and the paper by Gholamhosseinpour et al. (2008b) (at an industrial level), it may not be appropriate to draw direct comparisons between the studies. Although we found temperatures around approximately 80°C used in an online rock candy recipe (English, 2007), this recipe used a higher sugar to water ratio than our experiment. Given what we understand about crystallization, increasing the ratio of sugar to water at a lower temperature (80°C) could have the same impact on mass as cooking a solution with a lower sugar to water ratio to a higher temperature, as both situations result in increased saturations of the solution. The differences between recipes (sugar to water ratios and temperatures) could explain the disparity between our results and the online sources (English, 2007). Another potential explanation for our lack of crystal growth at 80°C could be the size of the skewers we used. To ensure that all experimenters had access to the same materials and to reduce trips to the store in a pandemic, lab materials were ordered from “Amazon.ca'' instead of being bought in person. As a result, the skewers we purchased could be unintentionally smaller in diameter than those typically used in rock-candy making. Using skewers with a wider diameter would increase the chances of rock candy growth due to a larger surface area for seeding, which would give an increased area for recrystallization to occur. In the supersaturation curve (Appendix H), the metastable region is an area in which both crystal formation and dispersion are in equilibrium, but seeding allows for crystal formation to occur in the metastable region (Hampton Research, 2020). Therefore, the amount of seeding can positively affect crystal formation. The discrepancies in seeding amount due to different skewer sizes could explain differences between our results (no appropriate crystal growth at 80°C) and those obtained in other online recipes that use approximately 80°C (English, 2007).

Interestingly, we observed very minimal growth at temperatures 60°C, 70°C, and 80°C. Additionally, we observed statistically significant differences in growth only in pairings including 90°C. To explain these findings, we looked into previous research into sugar solutions with representative supersaturation curves (Gonzales et al., 2017). The supersaturation curve for sugar and water suggests an exponential increase in the concentration of a supersaturated solution in response to increasing temperature (Appendix H). The exponential relationship observed between temperature and concentration could explain the non-significant differences in mean mass growth between temperatures below 90°C, and a sudden significant difference in mass at 90°C in our experiment. There may be a threshold temperature point (witnessed within our experimental conditions) in which crystal formation becomes possible. However, given that rock candy recipes vary so much in temperature and sugar to water ratios, this threshold temperature could vary and would be difficult to identify. To further examine these possibilities, another version of our experiment could be performed using increments of temperature between 80°C - 90°C.

It was determined that the standard error of the mean was the greatest for rock candy prepared at 90°C, indicating that variation was greatest for our 90°C treatment. The greater variation at 90°C could be explained by one replicate, as one experimenter found no visible crystal growth at 90°C (Appendix C). We believe this dataset points to the large variability in at-home experiments, and provides ideas for areas of improvement for future experiments. During our experiments, we may have experienced variability in our results due to changing experimental conditions. As each member of the research group conducted the experiment in different workspaces due to COVID-19 protocols, each rock candy sample may have been exposed to slightly varied amounts of ambient temperature and humidity. Higher temperatures will result in greater water evaporation, thereby increasing sugar solution saturation, and thus greater crystal formation (Hartel and Shastry, 1991). Additionally, ambient temperature can be altered by variations in weather (the experiments were performed over different days for each experimenter) and location (not all experimenters live in Vancouver). There are other temperature-related factors that cannot be controlled in the current at-home experimental locations, such as the type of central heating systems individuals have in their home. As well, variations in humidity can impact experimental results, as increased humidity will result in decreased water evaporation, which will decrease the saturation of the sugar solution, ultimately slowing crystal formation. For improvements, all replicates of the experiment should be conducted in the same location over the same days using the same equipment (e.g. stoves and pots), reducing variability from temperature and humidity.

Conclusion:

Increased temperatures for rock candy preparation had little to no effect on the rock candy mass. The p-value from the ANOVA was found to be <0.001. The null hypothesis was rejected, which stated that temperature will not have an effect on mean rock candy mass, as the p-value was less than the alpha value of 0.05. The Tukey post-hoc test produced p-values that were non-significant for pairings that did not have the 90°C treatment. The results of our experiment contradicted our initial prediction. We observed that higher temperatures (such as 90°C) produced significantly greater rock candy mass. However, temperatures 60°C, 70°C, and 80°C had no significant differences in mean mass, even though the temperature increased from 60°C-80°C. In the future, the experiment could be improved by standardizing the location and equipment to minimize variation.

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Appendix:

Appendix A: Rock Candy Recipe adapted from Trolio (2021)

1. Bring the water to a boil over medium-high heat. Slowly, add the sugar one half-cup at a time, stirring until it's completely dissolved and the solution comes to a rolling boil. Remove from heat and cool for 20 minutes.
2. Dip the wooden skewers, swizzle sticks, or string into the sugar solution, then dredge them in the extra granulated sugar, rolling them so that they're completely covered. Set them aside until dry, 5-10 minutes. (If you use string, you will need to straighten it out before laying it flat to dry on parchment paper.)
3. Prepare the glasses: pour 5 – 6 drops of your desired food coloring into the bottom of each glass. If you're using a natural flavoring extract, add a few drops at this time. Pour the cooled sugar solution over the food coloring, about three-quarters of the way up the glass. Stir to combine.
4. Place the dried skewers or swizzle sticks into the center of each glass. Clip the top of each stick with a clothespin to keep it in the center of each glass and so that the tip isn't touching the bottom of the glass (or else it will be difficult to remove.) If using string, tie the opposite end that's not coated in sugar around a pencil, and place the string in the center of the glass. The pencil should be laid over the rim of the glass with the string hanging down.
5. Place the glasses in an area where they will not be disturbed, and let them sit for 1 – 2 weeks, or until the sugar crystals have reached your desired size. (It's important to not move the glasses while the crystals are growing, because this can cause them to fall off the stick or string.)
6. Once the sugar crystals have reached your desired size, gently crack the hardened sugar from the surface of the sugar solution and remove the skewers, swizzle sticks, or string. Allow them to dry before serving.

Appendix B: Sample Calculation

Mass of rock candy and skewer - Mass of skewer = Mass of rock candy

0.92g - 0.67g = 0.25g

Appendix C: Raw Rock Candy Data

Replicates	60	70	80	90
A	0.16	0.12	0.15	2.09
B	0.01	0.02	0.15	1.86
C	0.26	0.25	0.29	2.23
D	0.25	0.17	0.08	0.44

Figure A-1. The mass of rock candy for each replicate at 60°C, 70°C, 80°C and 90°C.

Appendix D: QQ Plot of Residuals for Raw Data

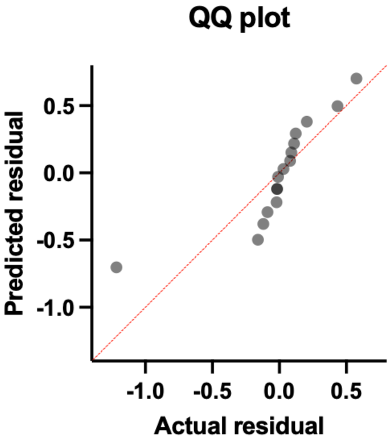

Figure A-2. The data was found to be normal using a QQ-plot.

Appendix E: ANOVA Table

ANOVA table	SS	DF	MS	F (DFn, DFd)	P value
Treatment (between temperature means)	6.715	3	2.238	F (3, 12) = 12.61	P=0.000509
Residual (within columns)	2.129	12	0.1774		
Total	8.844	15			

Figure A-3. A p-value <0.001 was found after performing one-way analysis of variance test.

Appendix F: Tukey Post-hoc Plot

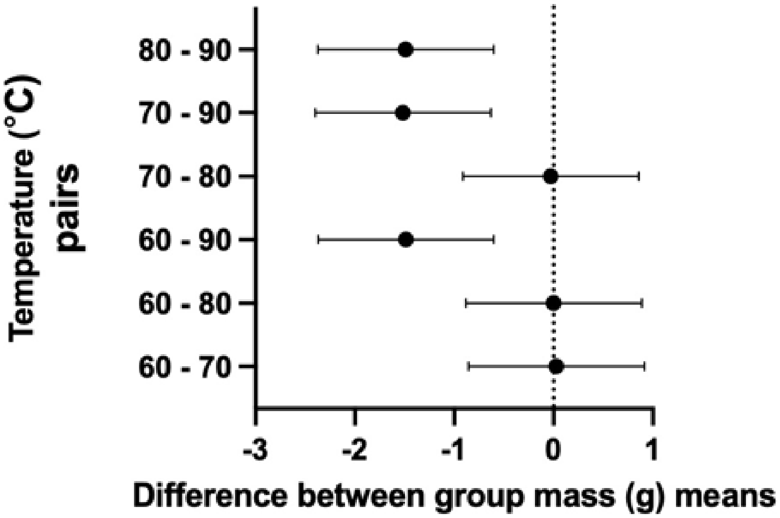

Figure A-4. Temperature pairwise comparisons containing 90°C are statistically significant. The lines represent the 95% confidence intervals for mean mass (g) measurements at different temperature pairings (°C). The black dots represent the mean difference found between the temperature pairs. The following p-values were observed in multi-comparison testing: 0.99958 (60-70°C), >0.999999 (60-80°C), 0.002991 (60-90°C), 0.999677 (70-80°C), 0.002613 (70-90°C), 0.002957 (80-90°C). The vertical dotted line represents the point at which the difference between the means is equal to zero.

Appendix G: Photo of Rock Candy Sugar Crystals

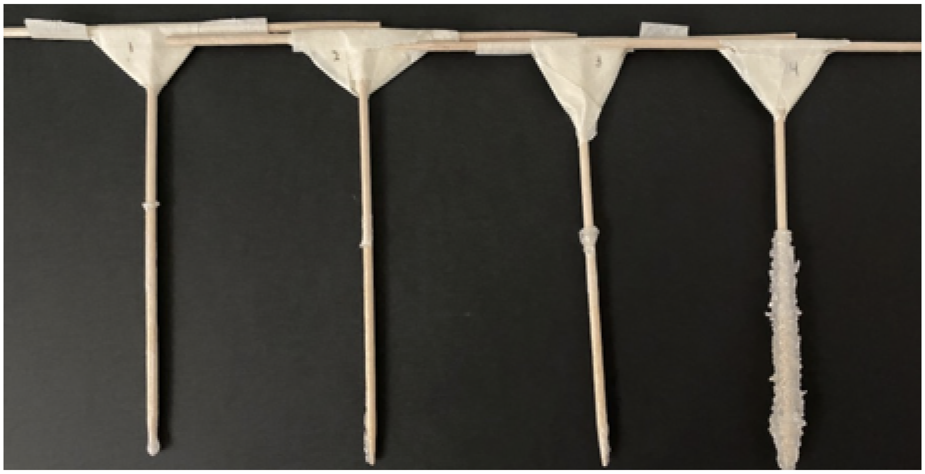

Figure A-5. Rock candy growth for Replicate A after 7 days (Appendix A), in the following order: 60°C (0.16g), 70°C (0.12g), 80°C (0.15g), 90°C (2.09g).

Appendix H: White Sugar Supersaturation Curve

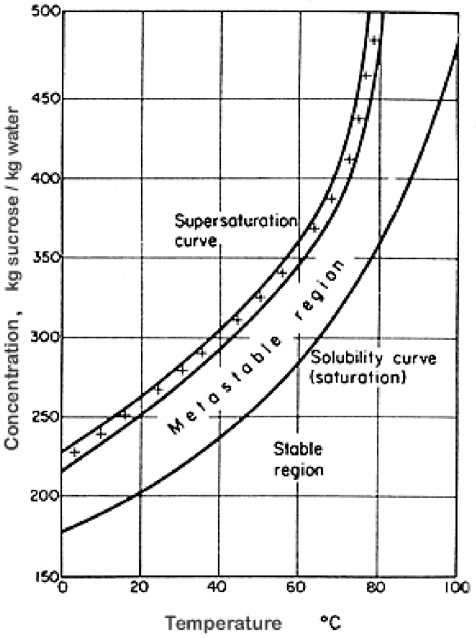

Figure A-6. A sucrose supersaturation curve adapted from Gonzales et al. (2017).