Why are Artificial Sweeteners an important factor in drinking water? Remediation of Artificial Sweeteners with Zeolites to Obtain Clean Drinking Water

A Major Qualifying Project Report:
Submitted to the faculty of the
Worcester Polytechnic Institute
in partial fulfillment of the requirements for the
Degree of Bachelor of Science in Chemistry

By: Elizabeth Dahlberg
Date: April 23, 2024

Advisor: Professor Drew Brodeur

Abstract

Artificial Sweeteners (AS) are commonly used worldwide, but many scientists do not fully understand the effects sweeteners have on drinking water. Zeolites have been previously shown to sequester target pollutants from water samples; this research examines whether zeolites are a viable option to obtain cleaner drinking water via wastewater treatment plants (WWTPs) by reducing the concentration of four free AS (Sucralose, Acesulfame Potassium, Saccharin, and Aspartame) in water samples. A significant reduction in free AS was observed after introduction to zeolites in solution using GC-MS and density testing. The determination of significance in our results starts by looking at the baseline of the data. For peak areas, the data goes from zero to the highest peak area while the density baseline is 1 g/mL for water and 0.792 g/mL for methanol. Looking at the results and comparing them to the baseline we can see that our results are significant. Almost all the sweetener was removed in our density testing, and there was a minimum 91% reduction of our sweetener concentration in GC-MS testing.

Acknowledgements

I would like to thank my advisor, Professor Drew Brodeur, for his commitment and assistance in helping me with this project. I would also like to thank all my Chemistry professors for furthering my knowledge in this subject. Finally, I would like to thank everyone who helped me get here, I appreciate all you have done and the time you spent helping me achieve my goals.

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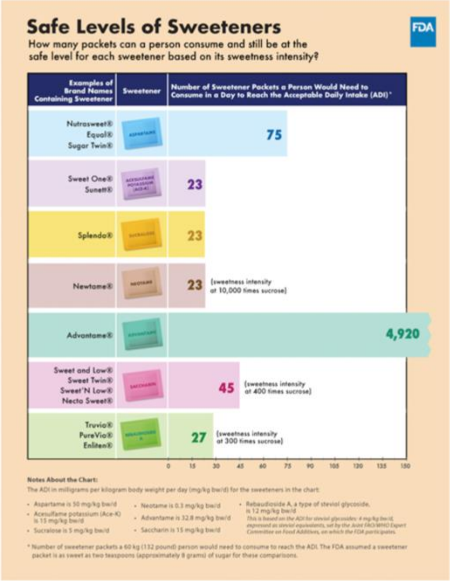
Introduction

Artificial Sweeteners are known as sugar substitutes because they are much sweeter than normal sugar and have either very little or no calories. The number of sweeteners acceptable in the US is smaller compared to other countries because of rules and regulations. These sweeteners are used all over the world and in many things but are best known for being in food and beverages. The FDA created the acceptable daily intake (ADI) for artificial sweeteners as the highest amount that would be safe to consume each day for a lifetime without any health effects, seen in Figure 1.



Notes About the Chart:
The ADI in milligrams per kilogram body weight per day (mg/kg bw/d) for the sweeteners in the chart:

- Aspartame is 50 mg/kg bw/d
- Acesulfame potassium (Ace-K) is 15 mg/kg bw/d
- Sucralose is 5 mg/kg bw/d
- Neotame is 0.3 mg/kg bw/d
- Advantame is 32.8 mg/kg bw/d
- Saccharin is 15 mg/kg bw/d
- Rebaudioside A, a type of steviol glycoside, is 12 mg/kg bw/d
  This is based on the ADI for steviol glycosides: 4 mg/kg bw/d, expressed as steviol equivalents, set by the Joint FAO/WHO Expert Committee on Food Additives, on which the FDA participates.

* Number of sweetener packets a 60 kg (132 pound) person would need to consume to reach the ADI. The FDA assumed a sweetener packet is as sweet as two teaspoons (approximately 8 grams) of sugar for these comparisons.

Figure 1: FDA guide to safe levels of sweeteners that shows how many sugar packets are needed to reach the ADI.[1]

The main factor that scientists used to calculate ADI was its toxicological studies.[2] The four main artificial sweeteners this research focuses on are sucralose, saccharin, acesulfame potassium, also known as Ace K, and aspartame. Many of these artificial sweeteners were found in wastewater, drinking water, and sludge after unsuccessful attempts at treating them in Wastewater Treatment Plants (WWTPs), which can be seen in Figure 2.

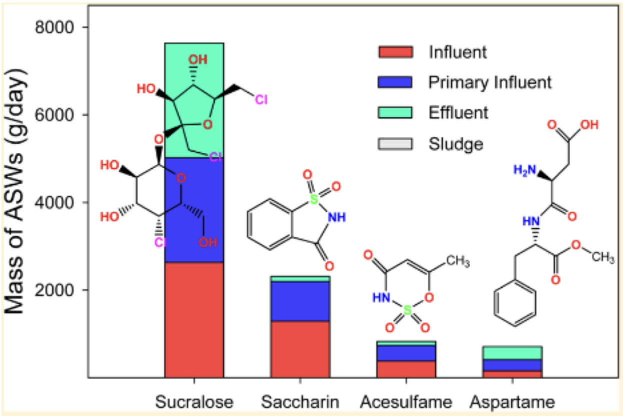


Figure 2: Grams of Artificial Sweeteners found per day in influent, primary influent, effluent, and sludge in the Wastewater Treatment Plants.[3]

In industrial terms, sludge is the residue that accumulates from sewage treatment plants, it is the byproduct of wastewater treatment processes.[4] The definition of wastewater, also known as sewage, is the polluted form of water generated from rainwater runoff and human activities. It is categorized based on how it is generated. The categories for sewage are domestic, industrial, or storm sewage (stormwater).[5] The issue that we face with these artificial sweeteners is that some of them do not degrade or decompose when they go through the human body. This is why artificial sweeteners have no calories. Without their decomposition the body does not get the calories from the artificial sweeteners. The efficiency of removing these artificial sweeteners from sewage treatment plants can be seen in Figure 3.

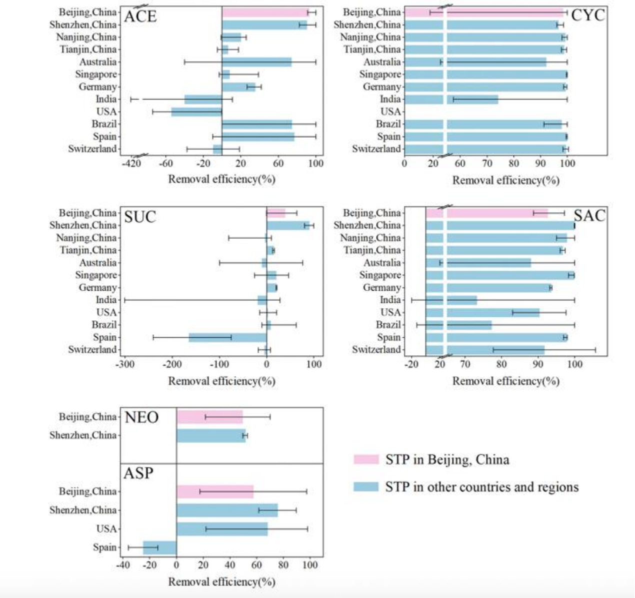


Figure 3: Sewage Treatment Plants removal efficiency percentages of artificial sweeteners around the world.[6]

We also see an increase in the number of artificial sweeteners people consume. Figure 4 shows the consumption of each artificial sweetener around the world.

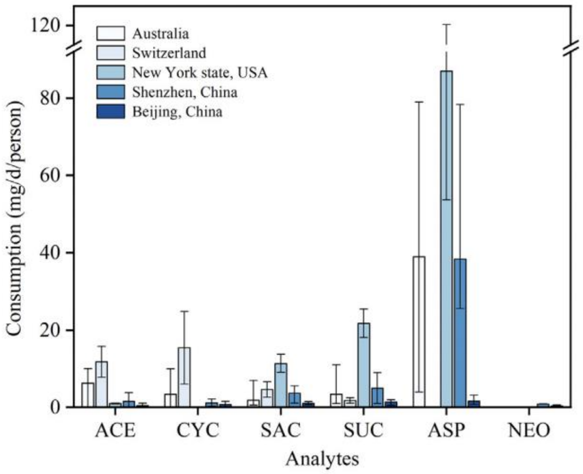


Figure 4: Consumption of artificial sweeteners per day per person, taken from the article about Seasonal occurrence, removal, and mass loads of artificial sweeteners in the largest water reclamation plant in China.[7]

In Figure 4, it shows that the United States consumes aspartame the most, then sucralose and then saccharin in comparison to Australia, Switzerland, and two cities in China. Artificial or additive sweeteners are known as persistent organic pollutants (POPs).[8] It is because they are organic compounds, and because they are not easily degradable or easy to capture. Some of the degradation byproducts could be potential environmental hazards. Some of them are not very beneficial to the environment and so it is necessary to figure out how to capture and/or absorb these compounds. Zeolites and Metal Organic Frameworks (MOFs) are beginning to be researched into how they could possibly be used for POPs, but more specifically artificial sweeteners. MOFs and zeolites are organic compounds that have pores and ions to either keep certain compounds out or to keep them in. The ligands and metal ions in the MOFs are critical to have because they can change the structure depending on what compounds it needs to obtain in the environment.[9] Zeolites are hydrated aluminosilicates and there are about 200 of them, around 40 are natural zeolites that are found around the world and the rest are synthesized zeolites. The most common synthetic zeolites are zeolites A, X, Y, and ZMS-5. The most occurring natural zeolites are analcime, chabazite, clinoptilolite, erionite, ferrierite, heulandite, laumontite, mordenite, and phillipsite.[10] We will mainly be focusing on a select few of these natural and synthetic zeolites. The purpose of this research is to see how we can use different zeolites and MOFs to treat and/or capture these artificial sweeteners in water and possibly sludge.

Background

The focus for this work will be on four artificial sweeteners. They are aspartame, acesulfame potassium (Ace K), sucralose, and saccharin. Saccharin has the chemical formula $C_7H_5NO_3S$, and the chemical structure is shown below in Figure 5.

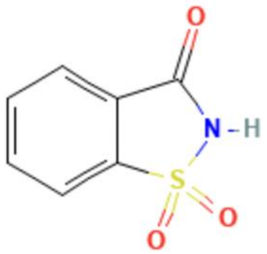


Figure 5: Structure of the artificial sweetener Saccharin taken from the National Library of Medicine – PubChem

It has a solubility of about 100 mg/mL in water at room temperature, and is soluble in ethanol, ethyl acetate, acetone, and amyl acetate.[11] The degradation of saccharin depends on the pH of the local environment. At acidic pH, the by product is 2-sulfobenzoic acid, and at basic pH, the by product is 2-sulfonamidobenzoic acid.[12] It is the third most consumed artificial sweetener with most of the compound degraded throughout the body after ingestion. This would create a somewhat low concentration of saccharin in WWTPs and water.

Aspartame has the chemical formula $C_{14}H_{18}N_2O_5$, and its structure is seen in Figure 6.

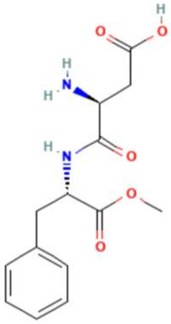


Figure 6: Chemical structure of artificial sweetener Aspartame taken from National Library of Medicine – PubChem

Aspartame does not have a set solubility in water because it is highly dependent on pH and temperature. In general, it has a higher solubility at low pH when the temperature is about 25° C.[13] Once aspartame is ingested in the body, it breaks down into natural residual by product components such as aspartic acid, phenylalanine, and methanol. It can also further break down its products to yield formaldehyde, formic acid and diketopiperazine.[14] When aspartame reaches the WWTP it is already broken down and therefore does not need any additional treatment to remain stable. The calculation for the concentration of aspartame in WWTPs shows a very low concentration which corresponds to how easily it can degrade into by products. The concentration found in WWTPs corresponds to how much aspartame is used in everyday life in the US and around the world. Aspartame is the most consumed artificial sweetener in the US, as previously seen in Figure 3. It does not include anywhere else in the US except New York State, which needs to be considered.

Acesulfame potassium, or Ace K has the chemical formula of $C_4H_4KNO_4S$. Its chemical structure is below in Figure 7.

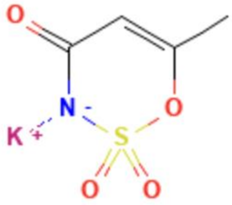


Figure 7: Chemical structure of Ace K artificial sweetener taken from the Library of Medicine – PubChem

Ace K is very soluble in water with a solubility of 237 g/L, and it is very slightly soluble in some chemicals, such as ethanol.[15] Acesulfame potassium has one major by product, which is acetoacetamide. This by product can be very toxic when it is given in large doses.[16] Ace K is one of the artificial sweeteners that does not easily degrade when ingested or treated in the WWTPs. It is very difficult to decrease its concentration in water and sludge samples because its degradation is difficult to obtain and because it is highly water soluble and stable. This report will try to use different zeolites and MOFs to increase the removal efficiency of Ace K. Ace K is not used very much in the US, but it is used very often in Australia and Europe which is seen in Figure 2. With its low usage in the US, and our ability to see the concentration in WWTPs means that it is very difficult to breakdown, and it shows that we should focus on how to degrade or capture this artificial sweetener.

The last artificial sweetener is sucralose. Its chemical formula is $C_{12}H_{19}Cl_3O_8$, and its structure is shown in Figure 8.

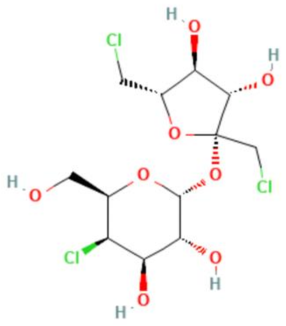
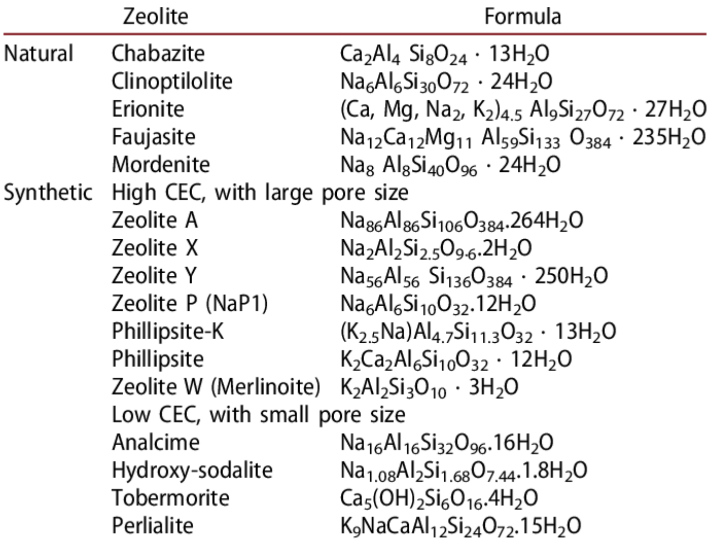


Figure 8: Chemical structure of the artificial sweetener sucralose, taken from the Library of Medicine – PubChem

Sucralose is very soluble in water, ethanol, and methanol, but is slightly soluble in ethyl acetate.[17] Sucralose does not break down when it goes through the body, but a small amount, around ten percent, does get absorbed.[18] Sucralose is another one that is not easy to degrade or capture. It is important to look at specific zeolites and MOFs that could be used to help degrade and capture this sweetener. Sucralose is the second most highly used artificial sweetener in the US, which accounts for a high concentration found in WWTPs and because it does not degrade easily with the treatment of WWTPs. Sucralose is also very water soluble which prevents it from being effectively removed from wastewater via the usual treatments. The natural and synthetic zeolites that could potentially be used for the degradation and/or capture of the four artificial sweeteners are in Figure 9.



	Zeolite	Formula
Natural	Chabazite	$Ca_2Al_4\ Si_8O_{24} \cdot 13H_2O$
	Clinoptilolite	$Na_6Al_6Si_{30}O_{72} \cdot 24H_2O$
	Erionite	$(Ca, Mg, Na_2, K_2)_{4.5}\ Al_9Si_{27}O_{72} \cdot 27H_2O$
	Faujasite	$Na_{12}Ca_{12}Mg_{11}\ Al_{59}Si_{133}\ O_{384} \cdot 235H_2O$
	Mordenite	$Na_8\ Al_8Si_{40}O_{96} \cdot 24H_2O$
Synthetic	High CEC, with large pore size	
	Zeolite A	$Na_{86}Al_{86}Si_{106}O_{384}.264H_2O$
	Zeolite X	$Na_2Al_2Si_{2.5}O_{9.6}.2H_2O$
	Zeolite Y	$Na_{56}Al_{56}\ Si_{136}O_{384} \cdot 250H_2O$
	Zeolite P (NaP1)	$Na_6Al_6Si_{10}O_{32}.12H_2O$
	Phillipsite-K	$(K_{2.5}Na)Al_{4.7}Si_{11.3}O_{32} \cdot 13H_2O$
	Phillipsite	$K_2Ca_2Al_6Si_{10}O_{32} \cdot 12H_2O$
	Zeolite W (Merlinoite)	$K_2Al_2Si_3O_{10} \cdot 3H_2O$
	Low CEC, with small pore size	
	Analcime	$Na_{16}Al_{16}Si_{32}O_{96}.16H_2O$
	Hydroxy-sodalite	$Na_{1.08}Al_2Si_{1.68}O_{7.44}.1.8H_2O$
	Tobermorite	$Ca_5(OH)_2Si_6O_{16}.4H_2O$
	Perlialite	$K_9NaCaAl_{12}Si_{24}O_{72}.15H_2O$

Figure 9: Name and chemical formula of the most common natural and synthetic zeolites.[19]

Any of these zeolites could be used, so more research would need to be done to specifically look at their toxicological properties and chemical properties that would have the most efficient capture and/or adsorption of these artificial sweeteners. Zeolites can be hydrophobic or hydrophilic, but many of them are hydrophilic because of their high content of aluminum and other cations, but pure silica zeolites are hydrophobic and need high pressure to have water fill in its cavities.[20] The zeolite's ability to absorb artificial sweeteners depends on the pore size of the zeolite which ranges from 3 to 7Å. The number of rings in the zeolite determines the pore size; increasing the rings increases the pore size. Figure 10 shows the way different zeolites are made due to pore size and shows some examples of each.

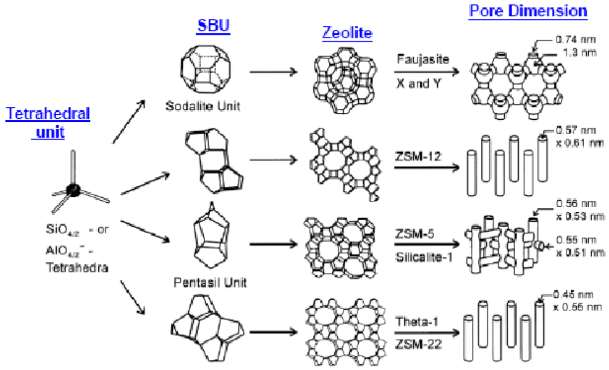


Figure 10: Process on how zeolites with different pore sizes are made and some examples of natural or synthetic zeolites that match the pore size.[21]

Changing the ratio of Si/Al can improve hydrophobicity as well as hydrothermal stability, if the ratio is increased.[22] There are many factors to think about to capture or absorb the artificial sweetener. The first is the chemical structure and size of the artificial sweetener. The second is the pore size of the zeolite. We need a zeolite that can capture and absorb the artificial sweetener, keep it stable after treatment, and keep the sweetener from not going back into the environment. MOFs are another option that we can use to absorb or degrade artificial sweeteners in wastewater. MOFs have a unique structure and are created from metal ions/clusters and organic struts. They are perfect for capturing toxic chemicals in the air and water because of their porous nature, tunability, and their processability of the chemicals.[23] The ligands and metals of MOFs are what make them easy to use to help clean the environment, specifically water and air. They are also able to capture a range of pollutants that are neutral and ionic, inorganic, and organic species which shows how important MOFs are to keeping the environment safe.[24] A specific MOF that we can investigate for the absorption and capture of artificial sweeteners is MIL-101. This MOF has been used in tandem with urea or melamine to capture many artificial sweeteners.[25] To determine the best option to capture or absorb artificial sweeteners, several candidates will be tested to determine which one has the highest absorption rate. The best option needs to be easily accessible since there is a lot of water in WWTPs that will need to be treated to capture the artificial sweeteners.

Sucralose, saccharin, aspartame, and acesulfame potassium are artificial sweeteners that need to be captured or absorbed by MOFs and zeolites because they are not beneficial for water and can harm the environment unless we act. The determination of which ones to use will depend on their accessibility and their efficiency.

Methodology

To test the solubility of these sweeteners they were first tested in water. Each artificial sweetener was in 50 mL of DI water. All glassware, stir bars, and stirrers were cleaned so no contaminants entered the solution. With cleaned glassware, 50 mL of water was measured in the graduated cylinder and poured into a beaker. A stir bar was added to the beaker along with a measured amount of around 0.1 g of the artificial sweetener. Once the artificial sweetener (AS) dissolved, small amounts of the sweetener were measured and added until the concentration was fully saturated. Some artificial sweeteners dissolved fast initially, so higher amounts of AS were added after. Three trials were done for all the artificial sweeteners in water.

Next, sweeteners were tested in different chemicals. The chemicals tested were chloroform, ethanol, and a solvent with a pH of 2. The process of determining the solubility of artificial sweeteners in these chemicals was the same process as determining their solubility in water. Sucralose had two trials instead of three in the pH of 2 solvent because there was not enough sucralose for a third trial.

Many of these artificial sweeteners were white powders or crystals. Their solubility was easy to determine because it was easy to visualize them in the solvent and know whether more was needed. After around 30 min if the sweeteners were not dissolved, then they had reached their solubility.

For the first GC-MS testing, all the glassware, except the pipettes and their bulbs, were cleaned with ethanol. After the initial cleaning, 20 mL of ethanol was measured in the graduated cylinder. The mass of the sweeteners ranged from 0.6 to 0.008 g based on their solubility. The sweetener was added into the Erlenmeyer flask and stirred for 30 minutes. A pipette transferred 2mL of the solution into a small dram vial. The vial was used in the GC-MS. A different pipette was used for each solution, all vials were labeled.

For the second GC-MS test, solutions were made a bit differently. Methanol was used for dissolving saccharin, and water was used for dissolving the rest of the sweeteners. 0.02g of each sweetener was added to 20 mL of the solute and then placed in other dram vials.

The third GC-MS test, zeolite was added to the sample. The same concentration and methodology were used in the second GC-MS test, but 0.1g of zeolite was added to each concentration to see if the sweetener concentration decreased.

Once the concentration was determined, the sweeteners were tested in the GC-MS to look at peak areas in the spectrum. The change in peak area would determine whether the zeolite is effective. The GC-MS method had a mass range from 50 to 450 g/mol, a two-microliter injection with a 50:1 split, the temperature started at 50°C for 1 min, then ramped it up to 20°C per min until it reached the final temperature of 300°C, then the 300°C temperature held for three minutes. There was a solvent delay of four minutes. The total run time for each sample in the GC-MS was 16.50 minutes.

After the GC-MS testing, UV-VIS spectroscopy was tried. An absorbance peak in the 200-300 nm range based on research could be used to calculate concentration based on the Beer-

Lambert Law. Our concentration was pipetted into a glass cuvette high enough for the light to pass through. The cuvette was placed into the UV-VIS spectrometer and the sample ran for about 3 minutes. When looking at the spectrum there was no absorbance peak in that range and therefore the concentration could not be calculated. The same sample was tested with an increased initial concentration to see if a peak would be visible but that did not work. The conclusion was that UV-VIS was not a viable way to determine a change in the sweetener concentration.

The last method that determined whether zeolites affected the concentration of the sweeteners was density. A 5 mL graduated cylinder was weighed. Then 1.5mL of the sweetener and zeolite concentration was measured into the graduated cylinder and massed. The density was calculated by taking the mass of the cylinder and dividing it by the volume. The solution was pipetted into a centrifuge tube and placed in the centrifuge. The steps were repeated for each of the sweeteners. Once all four tubes were placed in the centrifuge, they were centrifuged for five minutes at a speed of 10000 rpm. After they were centrifuged, the excess solution was pipetted into a tared graduation cylinder leaving the zeolite solid. The volume was 1.5mL and the mass was measured. The new mass and volume were used to calculate the new density. The steps were repeated for the rest of the sweeteners after centrifuging. Three trials were run for each sweetener which determined the density, standard deviation, and variance.

Results

The solubility results were very helpful in determining the best solution to use for GC-MS testing. Figures 11 through 14 show the solubility of saccharin in each solvent. Compared to the other artificial sweeteners, saccharin has a very low overall solubility, except for ethanol.

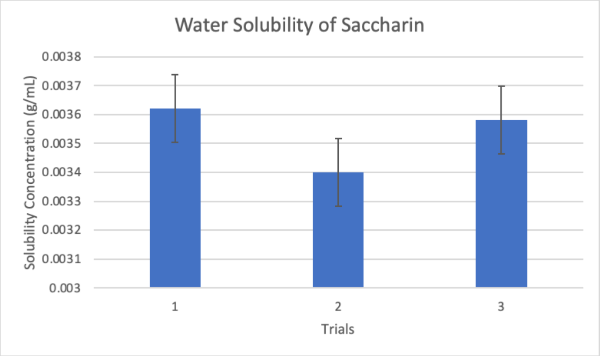


Figure 11: Water solubility of Saccharin with standard deviation error bars taken over three trials.

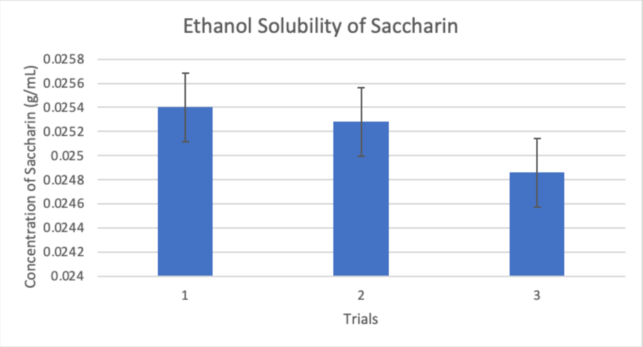


Figure 12: Three trials to determine ethanol solubility of saccharin with standard deviation error bars.

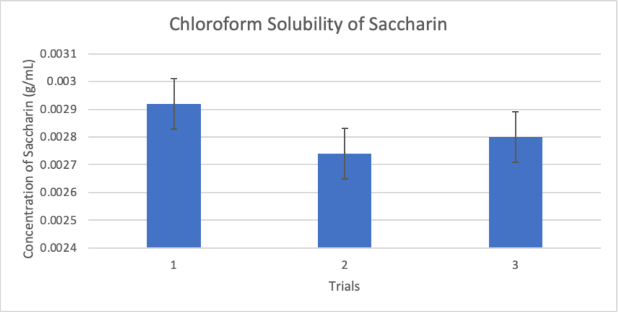


Figure 13: Saccharin’s solubility of chloroform determined over three trials with standard deviation error bars.

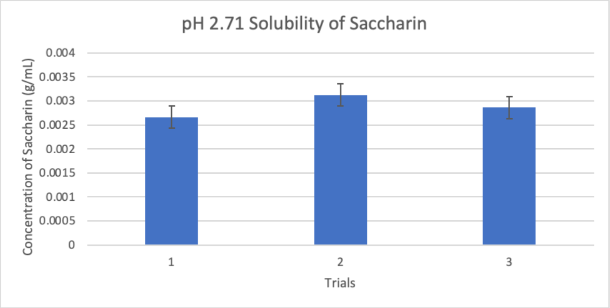


Figure 14: Solubility of saccharin in a pH of 2.71 taken over three trials with standard deviation error bars.

Saccharin’s solubility in chloroform and a solution with a pH of 2.71 showed a very similar solubility to that of water, while the solubility in ethanol was greater by a tenth. This shows that the best solvent to use for GC-MS testing saccharin is ethanol. Overall, all the trials for each solubility test are similar and can be seen with standard deviation error bars.

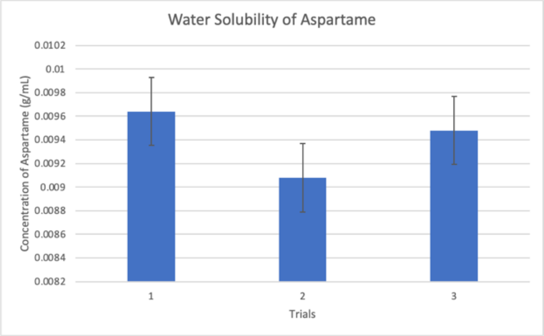


Figure 15: Solubility of aspartame in water taken over three trials with standard deviation error bars.

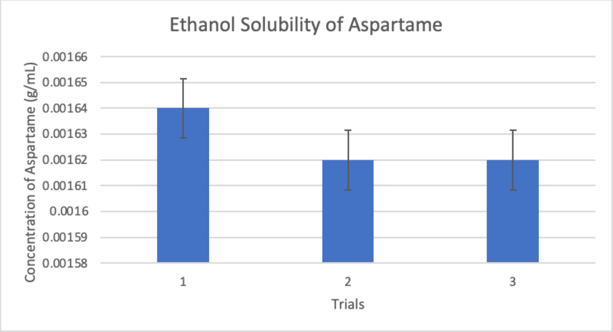


Figure 16: Three trials of ethanol solubility of Aspartame with standard deviation error bars.

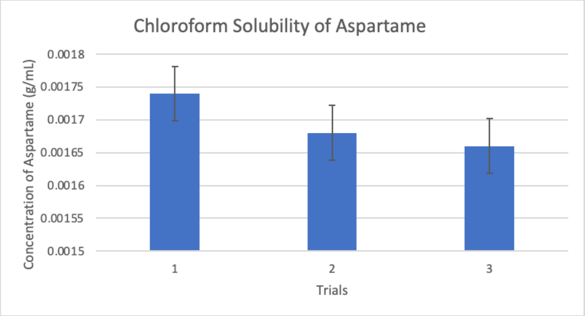


Figure 17: Aspartame's solubility in chloroform taken over three trials with standard deviation error bars.

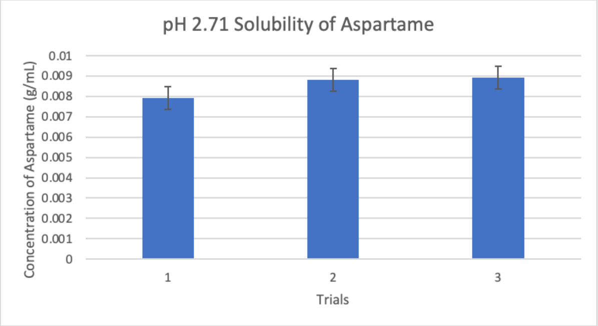


Figure 18: Aspartame solubility in a pH of 2.71 solvent obtained over three trials with standard deviation error bars.

The solubility of aspartame can be seen above in figures 15-18. Overall, it is very low solubility and there is no specific solvent that would best be used for GC-MS testing. Ethanol and Chloroform seem to have similar solubilities while the pH solvent and water also have similar solubilities. All the solubility testing concentrations are very close to each other as seen which the standard deviation error bars in the graphs. To make GC-MS testing easier, water was used as the solvent for aspartame.

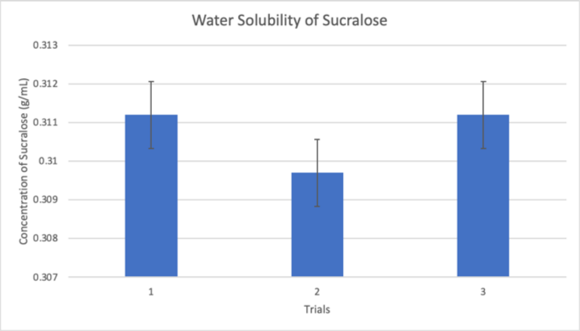


Figure 19: Three trials to determine the solubility of sucralose in water with standard deviation error bars.

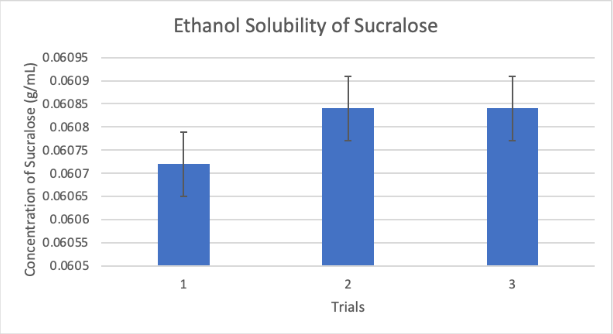


Figure 20: Three trials done to determine the ethanol solubility of sucralose with standard deviation error bars.

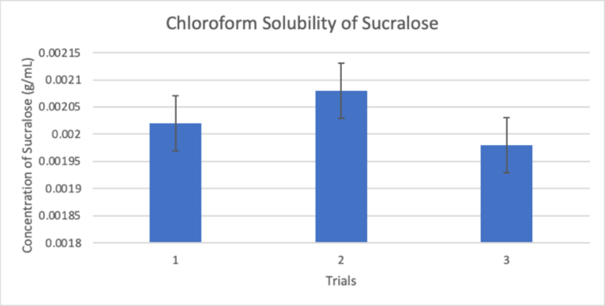


Figure 21: Sucralose solubility in chloroform determined over three trials with standard deviation error bars.

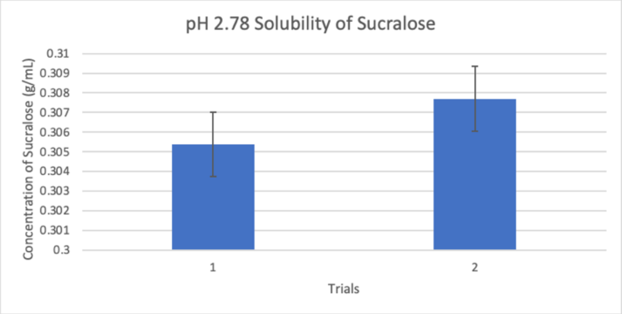


Figure 22: Sucralose's solubility in a solution of pH 2.78 obtained with two trials and standard deviation error bars.

The solubility of sucralose seen in figures 19-22 above is very high overall compared to all the other artificial sweeteners. Ethanol and chloroform show very low solubilities compared to water and the solvent with a pH of 2.78. Overall, the trials were similar in their concentrations shown by the standard deviation error bars. The ethanol solubility of sucralose is high compared to the other artificial sweeteners' solubility in ethanol. We determined that water would be a good solvent to use for GC-MS testing.

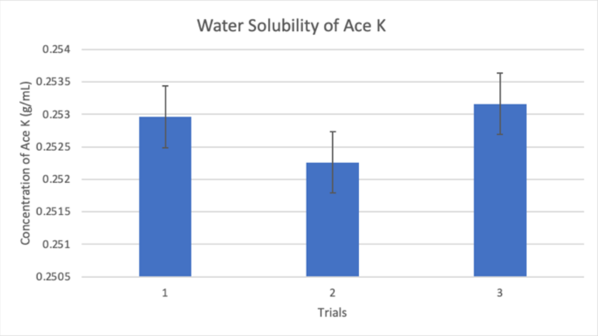


Figure 23: Three trials to determine acesulfame potassium's solubility in water with standard deviation error bars.

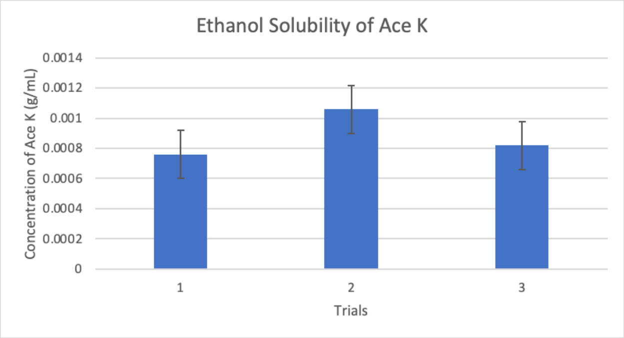


Figure 24: Ethanol solubility of Ace K taken over three trials with standard deviation error bars.

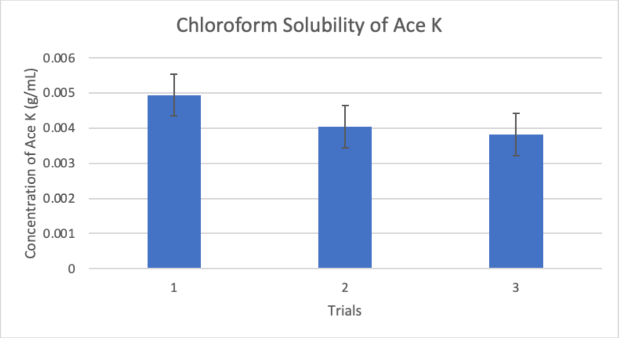


Figure 25: Solubility of Ace K in Chloroform taken over three trials with standard deviation error bars.

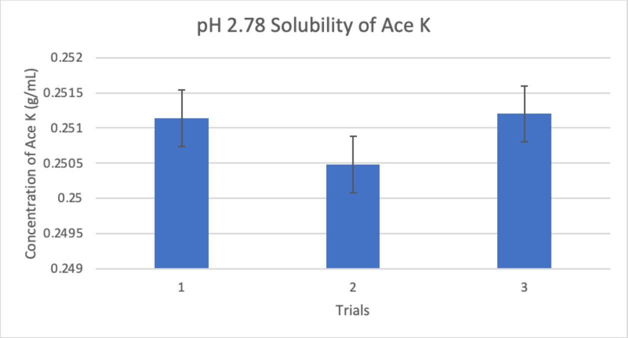


Figure 26: Acesulfame potassium solubility in a solvent solution of pH 2.78 obtained with three trials and standard deviation error bars.

Ace K has a low overall solubility seen above in Figures 23 – 26. The most interesting is that when the pH decreased, the solubility of Ace K stayed the same as the water solubility. Ethanol and chloroform had the lowest solubility for Ace K. For the GC-MS testing the easiest solvent to use is water.

Figures 27 and 28 highlight how the solubilities of each artificial sweetener change with each solvent. They show that Sucralose and Ace K have the highest solubilities overall while saccharin and aspartame have incredibly low solubilities. These graphs show the best solvent to use for each artificial sweetener. Chloroform was the overall worst solvent to use for solubility because they were all extremely low, while water and the pH solvent had the best solubility for each sweetener. Ethanol had lower solubilities, but it is a good solvent to use for any instrument testing because it can be used for every sweetener and has enough sweetener dissolved to take any measurements.

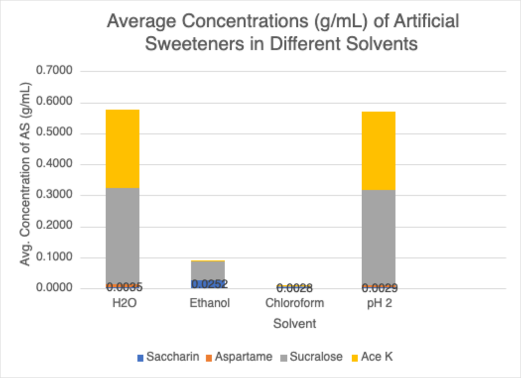


Figure 27: The average solubilities of artificial sweeteners within each solvent.

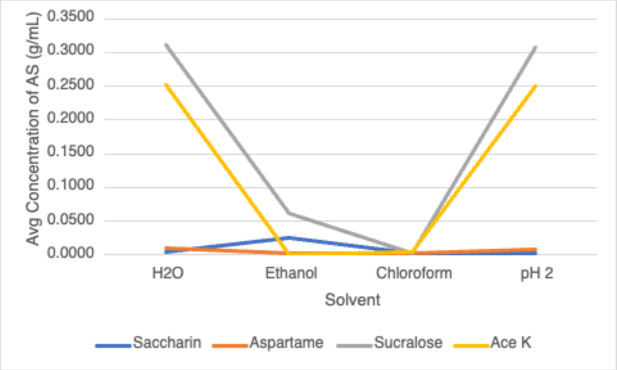
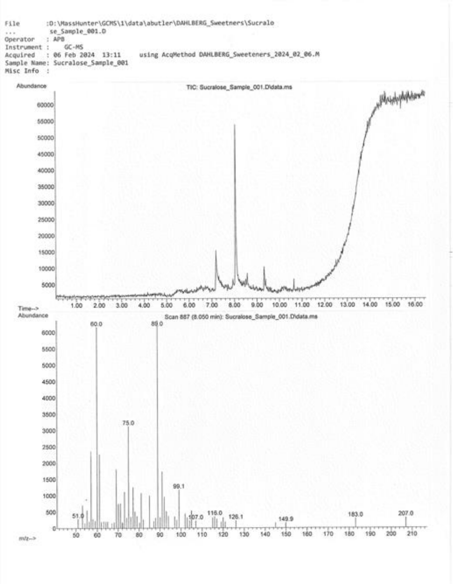


Figure 28: Average concentrations of the artificial sweeteners in a line graph to show the concentrations changing with each solvent.

Ace K and sucralose had the highest solubilities while saccharin and aspartame had the lowest solubility. This corresponds to their ability to degrade. Since Ace K and sucralose have higher solubilities, it means that they are harder to degrade, they are more stable molecules. Saccharin and aspartame have lower concentrations because they are not as stable and therefore degrade more easily.

The initial results of the GC-MS for the first run were not great. We ran each individual artificial sweetener and ran a combo sample of all the sweeteners. In the initial individual samples, we could only see saccharin. We could see a peak with a m/z of 182.9 which is similar to the 183.18 g/mol molar mass of saccharin. The rest of the masses of the artificial sweeteners were not visible.



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Instrument :    GC-MS
Acquired   : 06 Feb 2024  13:11     using AcqMethod DAHLBERG_Sweeteners_2024_02_06.M
Sample Name: Sucralose_Sample_001
Misc Info  :
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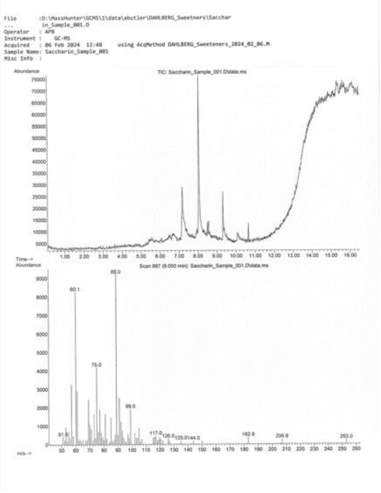
Figure 29: Initial GC-MS spectra of sucralose and cannot see a m/z that would conclude sucralose in the sample.



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Operator   : APB
Instrument :    GC-MS
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Sample Name: Saccharin_Sample_001
Misc Info  :
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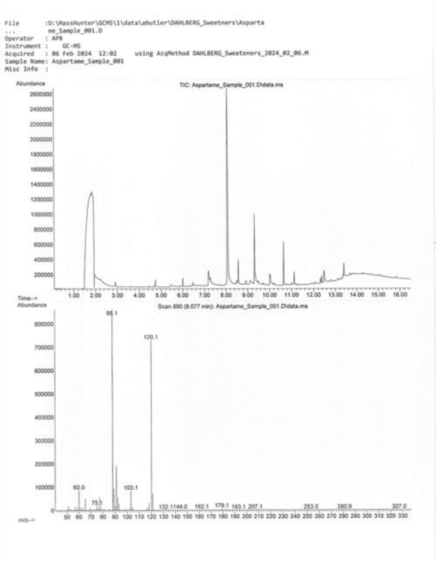
Figure 30: GC-MS saccharin sample spectra for the main peak around 8 min, we see a g/z of 182.9, which we can conclude is saccharin based on its molar mass.



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Sample Name: Aspartame_Sample_001
Misc Info  :
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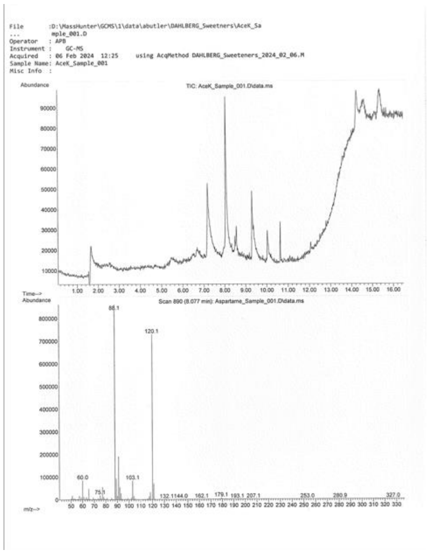
Figure 31: GC-MS test of Aspartame, unable to see a m/z close to its molar mass, cannot conclude there is aspartame.



```
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Instrument :    GC-MS
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Sample Name: AceK_Sample_001
Misc Info  :
```

Figure 32: GC-MS data of Ace K, unable to see a g/z close to its molar mass and therefore cannot conclude there is Ace K in the sample.

When we did our second GC-MS testing, we were able to find more of the sweeteners in the combo test and their individual tests. We found the Ace K and Saccharin peaks. We found the Ace K peak at 7.80 min where we found a m/z of 183, similar to its total molar mass. We then found another peak at 10.10 min which had an m/z of 193 which is the around the total molar mass of saccharin. We were also able to see all sweetener peaks when they were individually run in the GC-MS. More data and pictures are to come for this GC-MS run.

Density calculations of the solution with the zeolite before and after the solution was centrifuged helped us determine whether the zeolite impacted the artificial sweetener. Figures 33-40 show the differences in density before and after centrifuging based on the specific artificial sweetener.

Figure 33: Density trials of sucralose before centrifuging with a density between 1.00 and 1.04 g/mL.

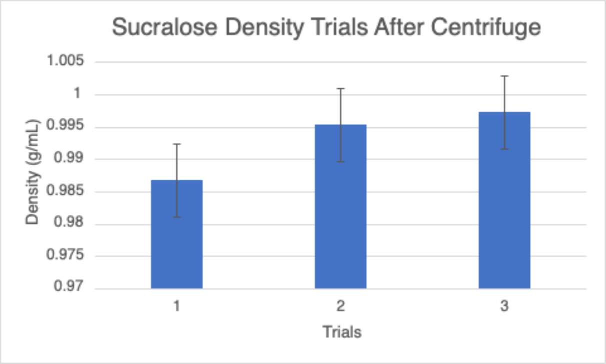


Figure 34: Three density trials of sucralose after centrifuging whose density is below 1.00 g/mL.

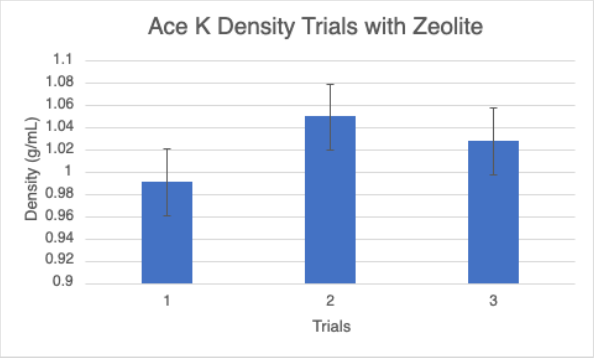


Figure 35: Three density trials of Ace K before centrifuging with a density between 0.99 and 1.06 g/mL.

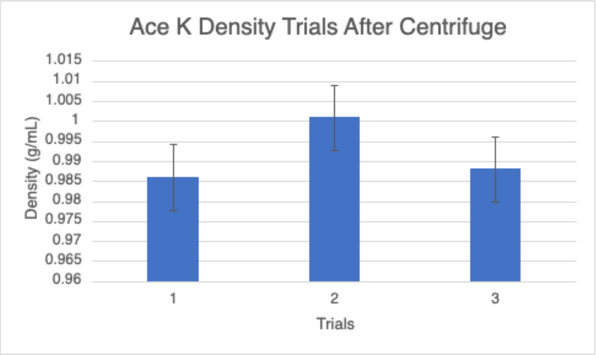


Figure 36: Three density trials of Ace K after centrifuging whose density is below 1.005 g/mL.

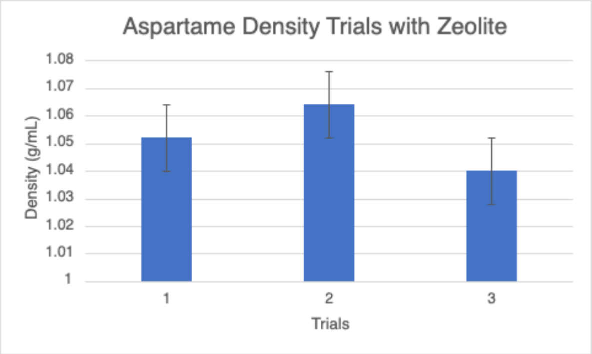


Figure 37: Three density trials of Aspartame before centrifuging with a density between 1.04 and 1.065 g/mL.

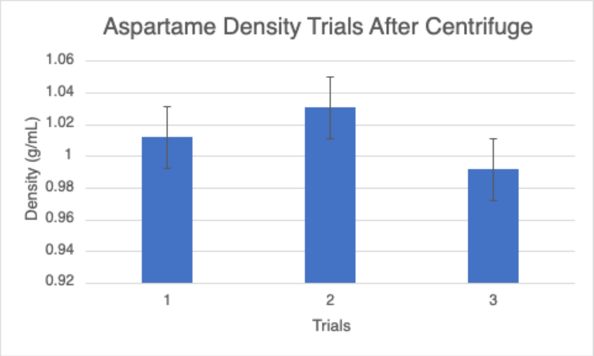


Figure 38: Three density trials of Aspartame after centrifuging with a density now between 0.99 and 1.03 g/mL.

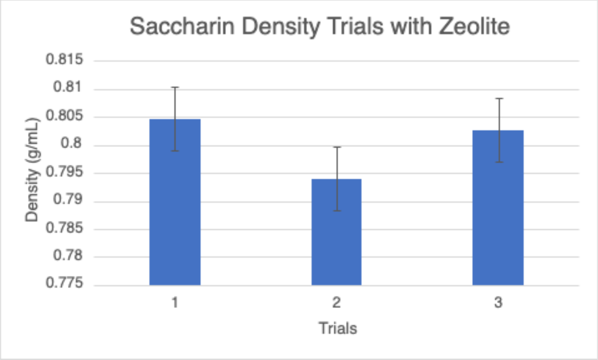


Figure 39: Three saccharin density trials before centrifuging with a density between 0.7925 and 0.805 g/mL.

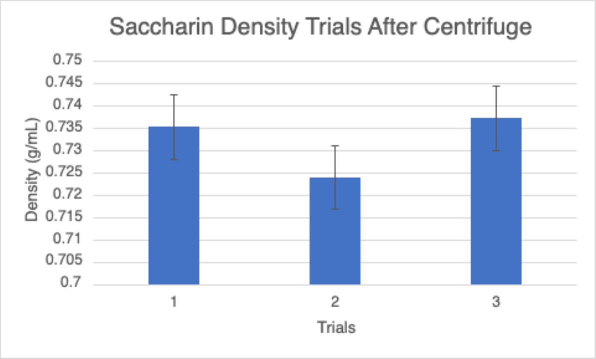


Figure 40: Three saccharin density trials after centrifuging with a density between 0.7225 and 0.7375 g/mL.

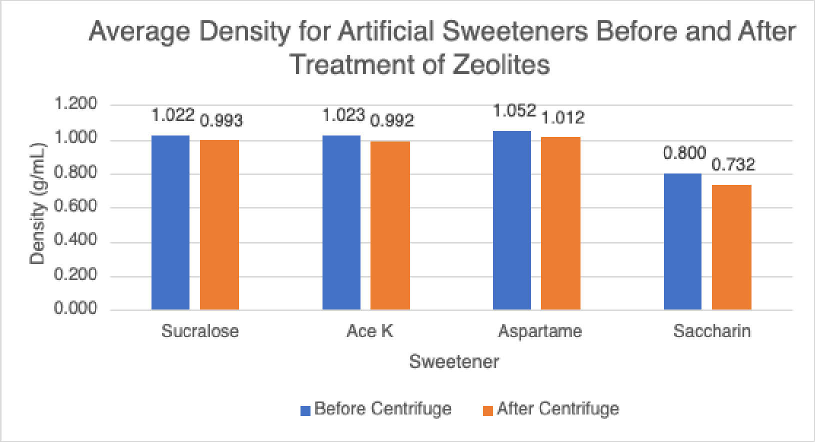
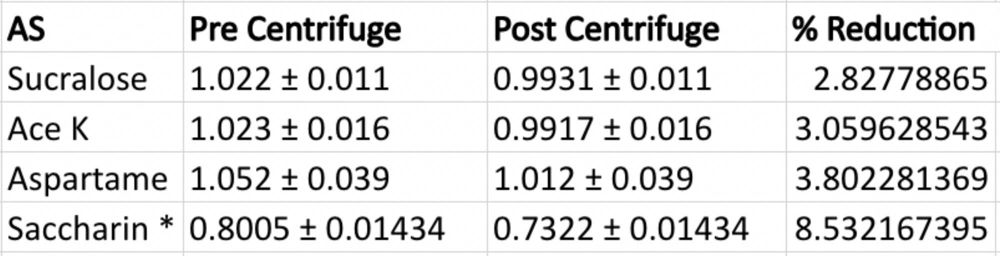


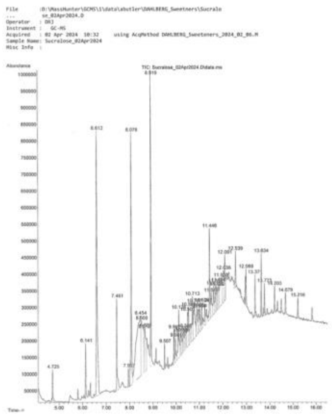
Figure 41: Overall average densities for each artificial sweetener taken before and after centrifuging.



AS	Pre Centrifuge	Post Centrifuge	% Reduction
Sucralose	1.022 ± 0.011	0.9931 ± 0.011	2.82778865
Ace K	1.023 ± 0.016	0.9917 ± 0.016	3.059628543
Aspartame	1.052 ± 0.039	1.012 ± 0.039	3.802281369
Saccharin *	0.8005 ± 0.01434	0.7322 ± 0.01434	8.532167395

Table 1: Percent reduction table based on the changes in density with zeolite pre and post centrifuge.

Looking at these figures and the table we see that the density changed after the solutions were centrifuged. Zeolite mixture made a difference and is a viable option to absorb artificial sweeteners in wastewater.



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Sample Name: Sucralose_02Apr2024
Misc Info  :
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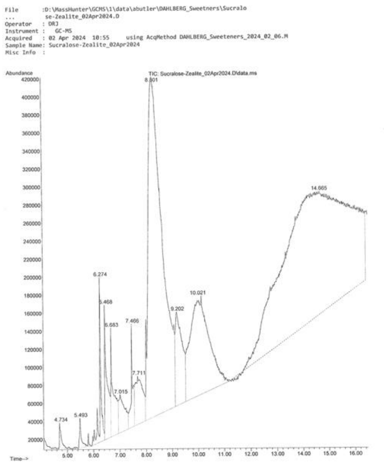
Figure 42: GC-MS spectrum of sucralose without zeolite, main peak at 11.446 min.



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Misc Info  :
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Figure 43: GC-MS spectrum of sucralose with zeolite addition, cannot see main peak.

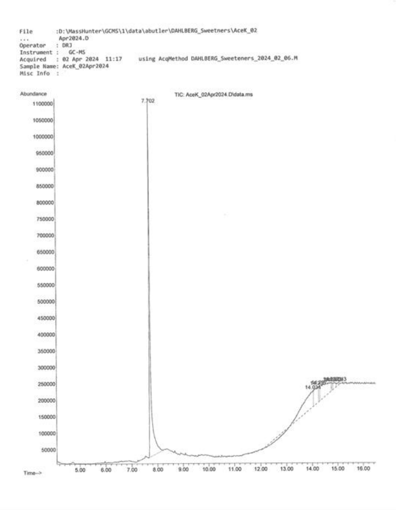

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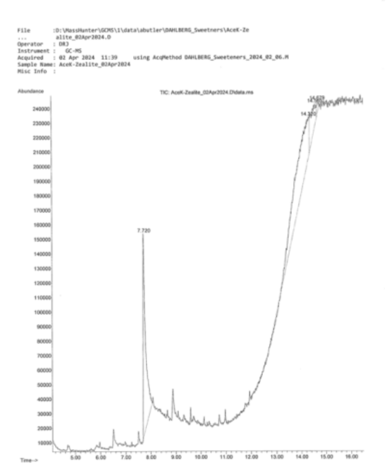
Figure 44: GC-MS spectrum of Ace K without zeolite, main peak at 7.702 min.



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Sample Name: AceK-Zealite_02Apr2024
Misc Info  :
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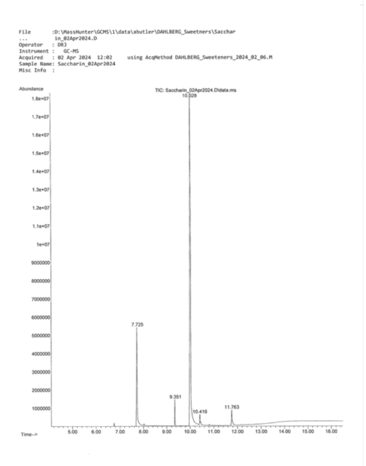
Figure 45: GC-MS spectrum of Ace K with zeolite addition, main peak at 7.720 min.



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Acquired   : 02 Apr 2024  12:02     using AcqMethod DAHLBERG_Sweeteners_2024_02_06.M
Sample Name: Saccharin_02Apr2024
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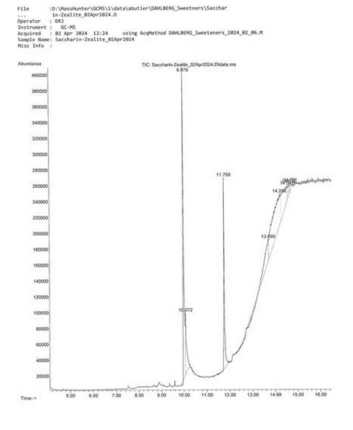
Figure 46: GC-MS spectrum of Saccharin without zeolite, main peak at 10.028 min.



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Sample Name: Saccharin-Zealite_02Apr2024
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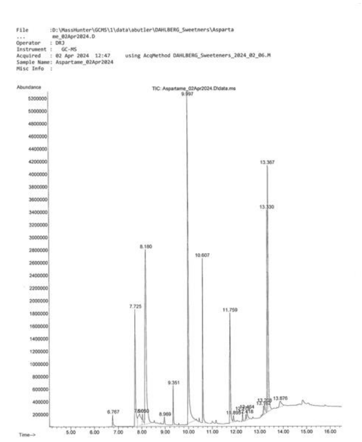
Figure 47: GC-MS spectrum of Saccharin with zeolite addition, main peak at 10.072 min.



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Acquired   : 02 Apr 2024  12:47     using AcqMethod DAHLBERG_Sweeteners_2024_02_06.M
Sample Name: Aspartame_02Apr2024
Misc Info  :
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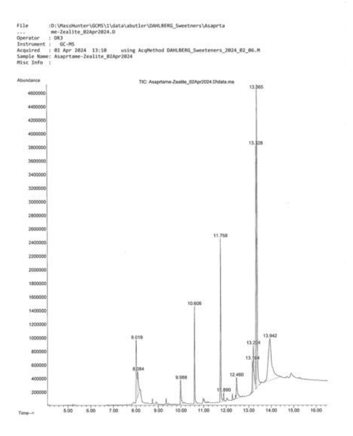
Figure 48: GC-MS spectrum of Aspartame without zeolite, main peak at 9.997 min.



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Acquired   : 02 Apr 2024  13:10     using AcqMethod DAHLBERG_Sweeteners_2024_02_06.M
Sample Name: Asaprtame-Zealite_02Apr2024
Misc Info  :
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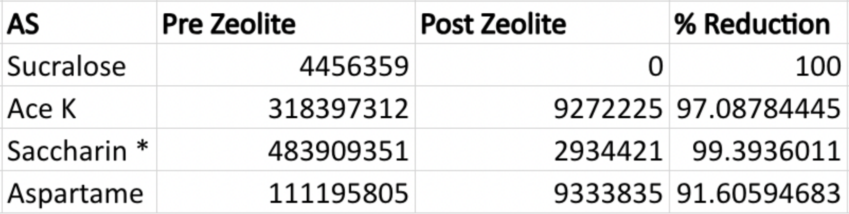
Figure 49: GC-MS spectrum of Aspartame with zeolite addition, main peak at 9.988 min.

These GC-MS spectrum show the peaks of the sweeteners with and without zeolites so we can calculate the percent reduction by looking at the changes in peaks areas. By looking at the times in each spectrum per sweetener we can use those times to find the peak areas and then use them to calculate percent reduction to see how much the concentration changed based on the zeolite. This helps us to determine whether zeolites can be used and are a viable option to decrease the concentration of artificial sweeteners. To find the percent reduction for sucralose we looked at figures 42 and 43. The interesting part about this sweetener was that a main peak was not found in figure 43 for post zeolite. It is likely because the amount of zeolite that we added to the concentration was able to capture a significant amount such that the GC-MS was not about to read the concentration and therefore a peak area was not able to be found.



AS	Pre Zeolite	Post Zeolite	% Reduction
Sucralose	4456359	0	100
Ace K	318397312	9272225	97.08784445
Saccharin *	483909351	2934421	99.3936011
Aspartame	111195805	9333835	91.60594683

Table 2: Percent reduction table of artificial sweeteners based on the GC-MS data of peak areas.

This table you can see the changes in peak areas and therefore can see that zeolites to change the concentration based on the percent reduction. The equation used to calculate percent reduction is below:

$$\left(\frac{pre\ zeolite\ peak\ area\ -\ post\ zeolite\ peak\ area}{pre\ zeolite\ peak\ area}\right)\cdot 100$$

There is a high percent reduction from the GC-MS peak areas, which means that zeolites captured much of the sweetener and therefore greatly decreased the concentration.

Discussion

We wanted to try UV-VIS as another way to analyze whether the artificial sweetener concentration changed. We could use the absorbance to calculate the change in absorbance using the Beer Lamber Law. All the sweeteners are white solids, so we knew that they would not show up in the visible range. We tried to get a UV-VIS spectrum using sucralose and even increased the concentration to just below full saturation to see if we could see a peak in the 200-300 nm range. Unfortunately, even with that high concentration we could not see a peak big enough to use the absorbance to calculate the concentration. We then decided to use density to determine a change in concentration along with GC-MS spectrum to look at changes in peak area.

Since we only used two different types of analysis to see a change in sweetener concentration, I would recommend doing other types of analysis to make sure that we see a change in sweetener concentration. I also would recommend using different zeolites with a mix of natural and synthetic zeolites to see how each zeolite impacts the concentration of the artificial sweeteners. It would then determine which zeolite is most effective at decreasing the concentration of artificial sweeteners and which one is most viable to use at wastewater treatment plants. The next people also need to keep in mind the cost of zeolites and pore size. There needs to be a zeolite that can be used in wastewater treatment plants at a low cost since it will be a larger scale operation. The zeolite chosen needs to have a pore size that is not too big or small because it needs to capture the sweetener and hold it in. It cannot allow the sweetener to leave or not have a chance to be captured. The other thought when working with zeolites and sweeteners is that once the sweeteners are captured in the zeolite and with the solid zeolite left out after centrifuging what is the next step for the zeolite solid. Is it possible for the zeolite solid to be calcined to decompose and eliminate the trapped sweetener for a “perfect” recycling? This is an important piece of information that needs to be researched and studied more before we can truly take out the sweeteners in water and obtain clean drinking water.

Conclusion

My hypothesis was looking at zeolites as a viable option to decrease the concentration of free AS in wastewater treatment plants. The hypothesis was confirmed with density and GC-MS testing. The percent reduction which was at least 91% for GC-MS peak areas and almost brought density to its baseline state in water and methanol showed a significant change in artificial sweetener concentration making zeolites a viable option. For the future, it would be a good idea to create a calibration curve that could connect the amount of added zeolite with a percent reduction in free sweetener concentration. This would help to determine how much zeolite is needed to be most effective in decreasing the sweetener concentration in wastewater treatment plants. Alternate testing should be used to gain a full picture of the best way to decrease sweetener concentration. This reduction could be improved with the use of alternate zeolites. There is still more to be done to determine which zeolite will be viable and most effective at decreasing the sweetener concentration in wastewater treatment plants, but this is a big step in obtaining cleaner drinking water.

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