Development of a Novel Aroma Capture Device

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Introduction

Rationale

My research plan is to develop an aroma capture device which enables the scientist to capture aromas at their source and transfer them back to the lab to perform both analytical and sensory analysis. Currently there exist some techniques to capture aromas and bring them back into the lab such as solid phase micro extraction (SPME)[1] and trapping on tenax desorption tubes. However each of these techniques do not enable the scientists to perform sensory analysis of the captured aromas. Without the ability to sensorially evaluate the captured aroma prior to analysis, others are left with the option to trust the descriptors used by the person who captured the aroma. On the other hand, if someone was to develop a technique which enables captured aromas to be brought to another location and then "release" for others to judge the smell, that would be a major breakthrough for in-field sampling applications. Therefore, this development that I have proposed will fill an unique, unmet need within the analysis of aromas and fragrances at their source.

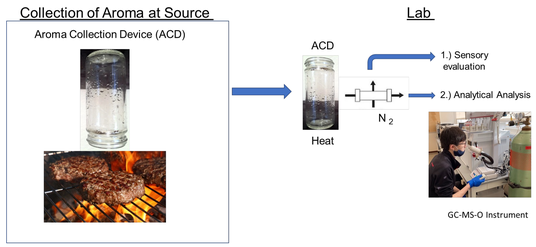

Figure 1: Diagram of the Aroma Capture Device which can capture aromas at the source (grilling of hamburgers) and the device is brought into the lab where the captured aroma is released and analyzed by both sensory and analytical procedures.

Research Goals

The goals are the following:

(1) Develop a proof-of-concept device which enables both capture and release of aroma for sensory evaluation and analytical analysis.

(2) Validation of technology with at least two examples.

(3) Refine and improve device for potential commercialization.

Idea Development

Based on researching the literature and in discussions with my advisors, I decided to focus on the use of a liquid-gel absorbent material similar to the phases on gas chromatograph columns as the material to capture aromas in the field. I was able to purchase Siloxane from Sigma-Aldrich and used it to coat the device for capturing aromas. Mason glass jars are available with wide-mouth openings which makes the coating easier. The wider opening also enables sufficient air exchange in order to capture aromas in the area. After various approaches to coat the inside jar with the absorbent, I settled on first dissolving the siloxane in hexane and swirling this solution around the interior of the jar while blowing nitrogen across the surface to evaporate the solvent. This resulted in a coating that is more evenly distributed throughout the interior of the jar.

Methods

Method Development and Learnings

Siloxane is a material which has gum-like properties, meaning it is flowable at temperatures slightly above room temperature. This property enables aroma molecules to diffuse into and penetrate the material, which is one reason for its use as a stationary phase in Gas Chromatography (GC) columns.

(1) During the coating process, I added siloxane to the vial and then added solvent such as hexane or dichloromethane to dissolve the material. Next, I rotated the vial and evaporated the solvent. This process was carried out in the hood to prevent exposure to the solvent.

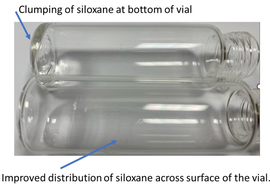

Figure 2: Picture of siloxane coated 20-mL vials. The top vial shows clumping at the bottom, while the bottom vial shows an improved distribution of the siloxane across the surface.

(2) Next, I tested the ability of these coated vials to capture volatiles by placing them above the surface of ground coffee beans as demonstrated in Figure 3.

Figure 3: Collection of volatiles above coffee grounds.

Unfortunately, when I performed analysis of the vial using solid phase microextraction and gas chromatography – mass spectrometry (SPME-GC-MS) there was residual solvent present and many siloxane peaks present. (Shown in Figure 4 below). There were coffee volatile compounds present such as acetic acid, pyridine, and furfural; however, it was apparent that additional treatment was needed to clean and prepare the vial in order to reduce the background effects.

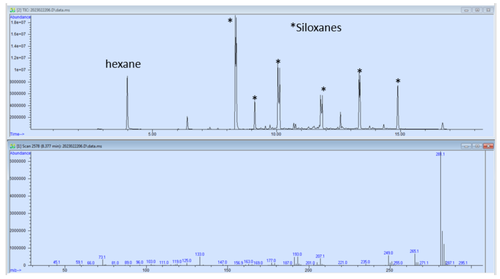

Figure 4: The SPME-GC-MS extraction of volatiles collected from above the surface of ground coffee. The top portion shows the chromatogram with several large peaks (*) present from the siloxane used in the coating. The bottom portion shows the mass spectrum of those peaks and characteristic m/z ions of 281 is present and the library match is for siloxane. In addition, the top chromatogram shows hexane which is my solvent used in the coating process.

Results

Improvements of Aroma Collection Device (ACD)

First proof of concept was to verify that I was able to capture the volatiles of coffee using the siloxane coated vial. In addition, when I warmed the vial and evaluate the aroma above the opening, my mentor and I could recognize the smell of coffee coming out of the vial. This was exciting because the aroma I captured represented the aroma of the sample that I was trying to collect. However, the background siloxane and residual solvent was an issue that needed to be resolved.

Next, I investigated heating the vial in a heating block which improved the ACD by lowering these background compounds substantially. However, I needed a better way to clean the device in between sampling in order to prevent carryover of previously captured aromas. After several experiments, I found that I could use a vacuum oven heated at 120°C for overnight which was sufficient for removing simple aroma compounds.

The changes made significant improvements in removing the volatile background peaks from the aroma collection vial. The results are shown in the analysis of a medium roasted coffee sample in Figure 5 below. The siloxane peaks and solvent were reduced to baseline levels. In addition, the peaks responsible for the aroma of coffee were captured very effectively with the ACD. The compounds identified in the chromatogram below such as acetic acid, pyridine, furfuryl alcohol, 2,6-dimethylpyrazine, and 2-furanmethanol acetate are known to be important compounds in coffee aroma.

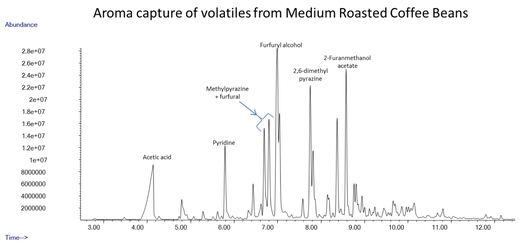

Figure 5: The SPME-GC-MS extraction from the Aroma Collection Device (Vial) of volatiles collected from above the surface of medium roasted coffee beans.

Next, I applied the siloxane coating approach to a bigger collection device, such as a mason jar shown in Figure 6. With the larger collection device this enables us to collect volatiles from a wider range of substrates. As shown in the figure, I was collecting volatiles from a flower bouquet. The collection was allowed to proceed for one hour. The aroma capture device was heated using the electrical tape and sampled with a SPME fiber (then desorbed into the GC-MS). In addition, I smelled the aroma above the heated capture device and could easily detect the floral smell of the flower bouquet.

Figure 6: Aroma collection from a flower bouquet using a siloxane coated Aroma Collection Device.

Conclusions

Summary

With this approach, I have developed a working proof-of-concept aroma collection device which enables me to perform both analytical and sensory analysis of captured aromas in the field. I was able to design a cleaning method for the aroma collection device which significantly reduced the background interference of volatile siloxanes and solvent. With this approach, I have sampled a variety of products. Figure 7 shows the collection of volatiles emitting from a blooming rose. The compounds phenylethyl alcohol, acetaldehyde, 2-hexenal and hexanal are important compounds in rose aroma. The compound dimethyl sulfide was a surprising compound detected as it is not considered to be important for the rose aroma. In addition, there are a few more peaks that I haven't identified yet.

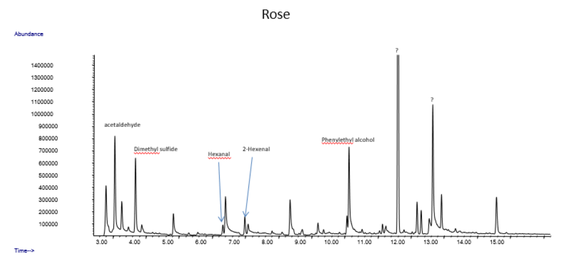

Figure 7: GC-MS of volatiles of rose collected with Aroma Collection Device.

Another flower that I sampled was the lily, Figure 8. This flower was very fragrant and it showed in the abundance of peaks within the chromatogram. The large peaks for cis-3-hexen-1-ol acetate, beta-ocimene, methyl benzoate, and linalool contribute to the floral, green, and lemon character of this flower. In addition, the stinky smelling compound 4-methyl phenol (also known as p-cresol) has been documented to be important in the aroma of lily flowers.[2]

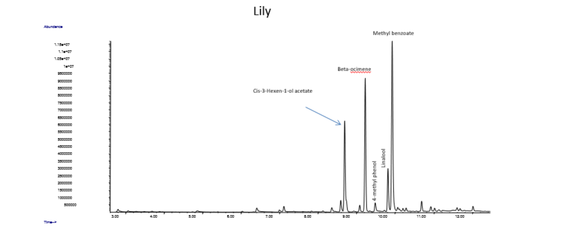

Figure 8: GC-MS of volatiles of lily flower collected with Aroma Collection Device.

Next steps include refinement of the process so that some steps can be automated and connected directly to a GC and sensory sampling apparatus. In addition, I will be investigating additional products to sample such as the grilling of a steak or hamburger on the grill. These additional sampling experiments will help us understand the limitations of the current setup and tests for modifications that may be necessary to improve this method.

References

[1]Yong Foo Wong, Dan Dan Yan, Robert A. Shellie, Danilo Sciarrone, and Philip Marriott (2019) Rapid plant volatiles screening using headspace SPME and person-portable gas chromatography-mass-spectrometry. Chromatographia 82, 297-305.

[2]Wei L., Wei S., Hu D., Feng L., Liu Y., Liu H., and Liao W. (2022) Comprehensive flavor analysis of volatile components during the vase period of cut lily (*Lilium spp. 'Manissa'*) flowers by HS-SPME/GC-MS combined with e-nose technology. Front. Plant Sci. Volume 13 - 2022 | https://doi.org/10.3389/fpls.2022.822956