Proceedings of The National Conference
On Undergraduate Research (NCUR) 2020
Montana State University, Bozeman MT
March 26-28, 2020

Reduction of Operating Costs in Fragrance Extractions

Muhammad Hanzala
Natural Sciences
Georgia Highlands College
Rome, Georgia 30161 USA

Faculty Advisor: Erin Shufro

Abstract

A series of experiments was conducted to create a formal procedure designed to cut down the costs of fragrance extractions (approximately $40.00) in commercially available products such as soaps, candles, bath bombs and perfumes. Chemicals needed for the extraction process are Pentane, salt, and denatured alcohol. Pentane is the most expensive chemical used during the extraction process, at approximately 100 dollars per one-liter bottle. The informal procedure in place used approximately 300 mL per extraction (informal procedure used daily required no measurements and thus starting amounts were estimated based on multiple observations). The goal was to reduce the consumption of pentane as much as possible while still extracting at least 1.0 mL of fragrance oil. Once the extraction parameters were optimized it was tested on commercially available products to ensure that the extractions would be successful not just in the lab but in the business as well. The lowest parameters found using pure fragrance oil were not successful on commercial products necessitating an upward adjustment. Even after the necessary adjustments were made the process of extraction was still significantly less expensive (approximately $9.00).

Keywords: Fragrance, Pentane, Extraction.

1. Introduction

Fragrance companies specialize in manufacturing fragrances for all sorts of products that have a custom scent to them. Many operate at a regional level, while some are multi-national behemoths. Some such companies offer a service where they can extract a fragrance from a product that the customer likes, and can analyze it for custom manufacturing. While observing multiple fragrance extractions at a regional fragrance manufacturing company, it was noticed that the parameters they set for their extractions were quite vague, resulting in a relatively high cost of the process (approx. $40.00 per extraction). On average, the company does about 7 extractions a day. Some of the chemicals, like pentane and reagent alcohol are quite expensive, and were poured in random amounts during the extraction. After observing the process and determining a basic extraction with a set of parameters, areas of improvement were identified that, if successful, would reduce the cost of extractions significantly. The primary objective was to reduce the amount of pentane as it costs approximately $100 per liter, though it was also hoped to reduce the cost of all chemicals in the process.

2. Methodology

Step one was to complete an analysis of the original method used at the company. It turned out that the company used 300 mL of pentane per extraction (the original abstract stated they used 100 mL per extraction, but after further analysis, the amount was much closer to 300 mL). They also used about 1000 mL water, and dumped approximately 200 mL of reagent alcohol and about 25g of salt. The experiment takes about 45 minutes per extraction. The Reagent alcohol used is denatured with methanol and isopropanol (approximately 95% Ethanol and Methanol, and 5% Isopropanol). The salt used is regular table salt: NaCl. It acts as a blocker in the experiment, as it dissolves with the water molecules, and stops the fragrance from dissolving into the water. The alcohol helps separate the fragrance molecules from the water, and the pentane bonds with the fragrance oils, which can later be burned off so that only the fragrance molecules are left.[1]

The extraction procedure they used is as follows:[2]

1. A substance with fragrance in it is dissolved in water (1000 ml).
2. Then, pentane (300ml), reagent alcohol (200ml), and salt (25g) is added.
3. The mixture is then thoroughly stirred.
4. After a clear separation rises to the top, that separated layer is carefully pipetted into a flask, using a grade 1 filter paper (11 micro meter particle retention).
5. It is then re-filtered using a grade 1 phase-seperator filter.
6. The remaining liquid is heated on a hot plate, at 36°C, until all pentane is evaporated off, and the remaining liquid is fragrance oil.
7. The fragrance is tested by its odor, look, and viscosity.
8. The remaining fragrance is measured.
9. Approximately 1ml of fragrance is required to analyze and duplicate the scent. A GC Mass spectrometer is used to determine the composition and confirm the amount of fragrance oil.

Our initial extraction used only 94.00 mL of pentane which is lower than the amount used by the company. This was done to reduce the total numbers of experiments needed. The pentane used had a n-pentane purity of 98%. In the initial extractions pure fragrance oil was used, rather than a product containing a fragrance, until the final parameters were determined. Fragrance oil will not dissolve in water due to difference in polarity. This ensured that factors other than the variables chosen would not play a part in refining the amounts used. Another aspect added to the original experiment was that for the first experiment, before putting the liquid on to the hot plate, a water bath was prepared and put on it. The temperature was continuously monitored until it reached 36°C, the boiling point of pentane. The settings on the hot plate were then measured. This ensured that when the liquid was put on the hotplate, only the pentane burned off, and the loss of fragrance was as controlled as possible.

The data from the first experiment was used to set the controls for our extractions.

3. Experimental Data

Table 1. Data for the first two extractions. These two extraction set the initial amount of pentane used, and the fragrance extracted using that.

Parameter	Extraction 1	Extraction 2
Type of product	Fragrance oil	Fragrance oil
Water	600 mL	600 mL
Fragrance added	8.00 mL	7.90 mL
Salt	10.94 g	10.58 g
Pentane	94.00 mL	90.00 mL
Reagent alcohol	70.05 mL	86.00 mL
Temperature on hot plate	37°C	39°C
Final yield	4.40 mL (55%)	4.38 mL (55.44%)

4 more experiments were carried out to continue to decrease the pentane. By the final experiment the pentane was reduced by more than half, from 94.00 mL down to 34.00 mL while still yielding enough fragrance oil. The final experiment data yielded:

Table 2. The parameters used with the lowest amount of pentane, that still yielded 3.75 mL of fragrance.

Parameter	Data
Type of product	Fragrance oil
Water	600 mL
Fragrance added	7.98 mL
Salt	10.01 g
Pentane	34.00 mL
Reagent alcohol	70.90 mL
Temperature on hot plate	37°C
Final yield of fragrance	3.75 mL (46.99%)

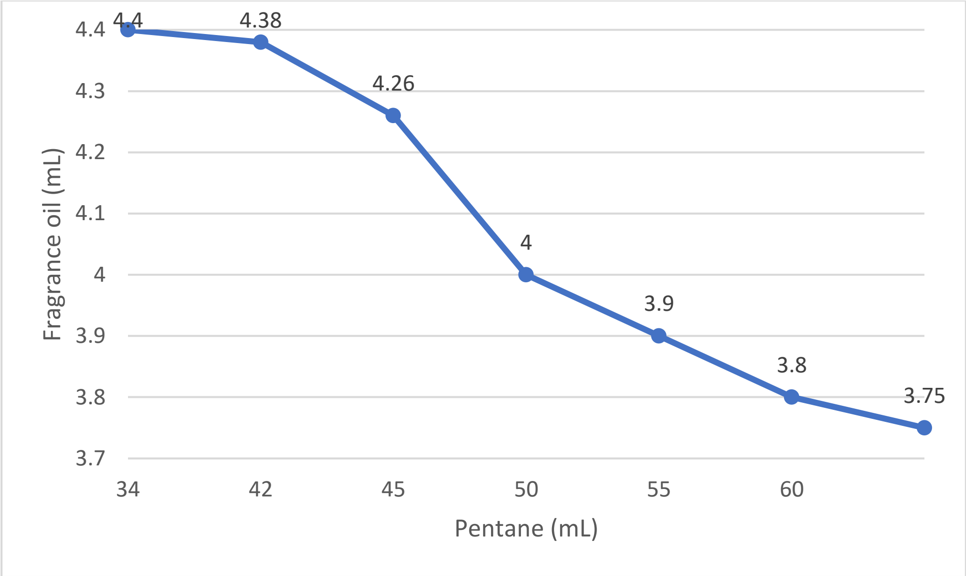

Figure 1. The amount of fragrance oil extracted vs the amount of pentane used. As the pentane used was reduced, the fragrance oil extracted at the end decreased too.

These refined parameters were now ready to be tested on a commercial product. The pentane could have been lowered further when using pure fragrance oil, but the fact that commercial products will have a lot more chemicals than pure fragrance oil meant that these amounts were sufficient. Another important factor to note is that the amount of fragrance oil in commercial product is unknown. The aim is to extract enough fragrance oil that can be analyzed through a GC Mass Spectrometer, which is 1 ml. The amount of fragrance in commercial product is proprietary information that was not made available on public packaging.

Table 3. Data of the first extraction done on a commercial product: bath bombs.

Parameter	Data
Type of product	Bath bombs (123.21 g)
Water	600 mL
Salt	10.01 g
Pentane	34.00 mL
Reagent alcohol	70.90 mL
Temperature on hot plate	37°C
Final yield of fragrance	0 mL

This showed that the lowest parameters determined when using pure fragrance oil would not work on commercial products. To counter that, initially the same parameters were used with an increase in the mass of bath bombs used – as this would increase the total amount of fragrance oil in the solution – but to no avail. We couldn't extract fragrance using 34.00 mL of pentane. The parameters were then slowly raised in a total of 5 more experiments. The final experiment resulted in the following:

Table 4. Data of the final extraction done on bath bombs, after raising the pentane to a level that yielded sufficient fragrance.

Parameter	Data
Type of product	Bath bombs (128.57g)
Water	600 mL
Salt	10.21 g
Pentane	50.00 mL
Reagent alcohol	80.00 mL
Temperature on hot plate	37°C
Final yield of fragrance	2.1 mL

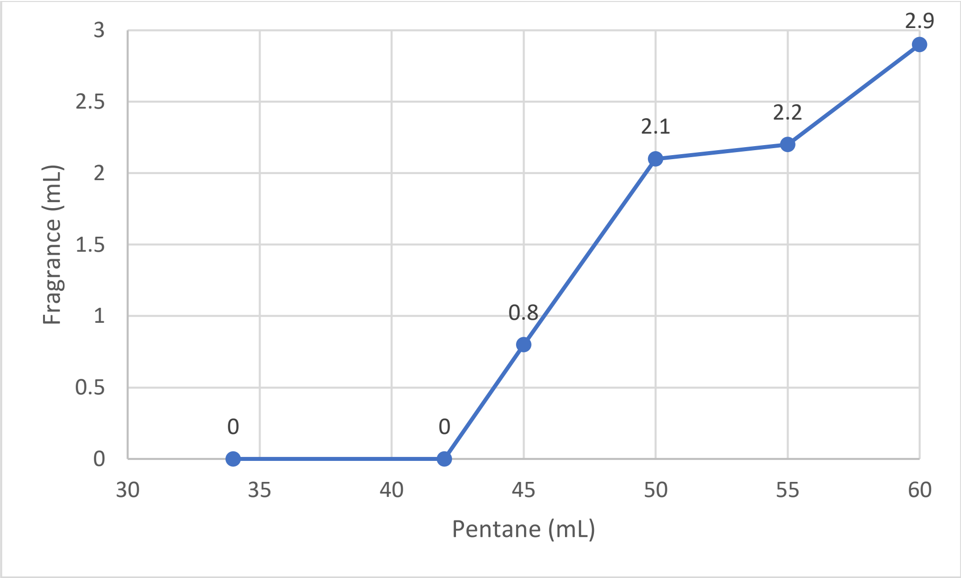

Figure 2. The amount of fragrance oil extracted from bath bombs vs the amount of pentane used. Anything below 42 mL of pentane did not yield any fragrance, so had to be raised until it was yielding at least 2 mL of fragrance.

Another extraction was carried out on commercially available liquid soap, using these newly determined parameters approximately.

Table 5. Fragrance extraction using a soap, with 50 ml of Pentane.

Parameter	Data
Type of product	Soap (103.67 g)
Water	600 mL
Salt	10.26 g
Pentane	50.00 mL
Reagent alcohol	80.00 mL
Temperature on hot plate	37°C
Final yield of fragrance	2.03 mL

3.1. Gas Chromatography – Mass Spectrometry Data

The solution extracted is run through a GC Mass Spectrometer (GC-MS) instrument to analyze what chemical compounds were part of the solution extracted. The area, height, and time of the peaks (see figure 3) is noted, and tallied with an internal database at the fragrance company. This gives the company a list of all the chemicals compounds found in the extracted liquid, and the research and development team fine tunes it to match the fragrance of the product it was initially extracted from. Having sufficient liquid for the machine is a crucial part of the extraction process, as enough fragrance is needed for there to be sufficient data of compounds present.

Peak Report TIC

Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H
1	2.404	2.365	2.435	33234	0.26	19583	0.36	1.70
2	2.477	2.435	2.495	4417221	34.64	2243473	41.40	1.97
3	2.530	2.495	2.640	7522059	58.99	2898594	53.49	2.60
4	9.760	9.730	9.810	22741	0.18	10435	0.19	2.18
5	23.949	23.890	24.000	124883	0.98	45359	0.84	2.75
6	24.325	24.265	24.415	121700	0.95	35924	0.66	3.39
7	25.015	24.970	25.055	20717	0.16	8474	0.16	2.44
8	27.566	27.500	27.635	270025	2.12	102942	1.90	2.62
9	27.947	27.895	28.005	61962	0.49	20268	0.37	3.06
10	28.381	28.295	28.445	156335	1.23	33630	0.62	4.65
				12750877	100.00	5418682	100.00	

Figure 3. An example of G.C. Mass Spectrometry data of an extracted sample. The time, height, and area are tallied with an internal database, to find out what chemicals are part of the fragrance.

4. Result Discussion

Initially, the amount of pentane was varied. When determining how low the parameters could be taken, the same fragrance oil was used in each experiment. By the sixth extraction, pentane had been significantly reduced to 34 mL. However, upon testing the new method on an actual product (Bath bombs), there was no fragrance extracted, resulting in a need to raise the amount of pentane when extracting commercial product. With gradual raises, through six additional extractions, it was determined that 50.00 mL of pentane resulted in a sufficient amount of fragrance extracted. This fragrance was run through a GC Mass Spectrometer, and was confirmed to be fragrance oil. This new extraction method with reduced amounts of chemicals used is significantly cheaper than the cost of the original method, especially when calculated over a high number of extractions.

Table 6. A comparison of the amounts used by the company and the amounts determined in the lab.

Product	Original Amount Used by the company	Lab determined Amount through experimentation
Water	1000 ml	600 ml
Salt	25 g	10.2 g
Pentane	300 ml (approx. $30.00)	50 ml (approx. $4.00)
Reagent Alcohol	200 ml (approx. $10.00)	80 ml (approx. $5.00)
Total cost	Approx. $40.00	Approx. $9.00

Changes to the original experiment were made to help this reduction as well. When the layer of separation was too small to pipette, it was transferred to a very small graduated cylinder, in order to vertically increase the layer, which meant even this layer could now be pipetted. Furthermore, pentane was added in two increments, so that after the first pipetting, the second increment would pull in any significant amount of remaining fragrance oils in the solution. In addition to that, the heat on the hot plate was also increased very gradually, in order to not burn off any fragrance oils while burning off the pentane.

With a significant reduction in parameters used – while still extracting more than 1 ml of fragrance oil – the purpose of this experiment was fulfilled. The amount of pentane needed was lowered from 300 mL to 50 mL, and the reagent alcohol was reduced from 200 mL to 80 mL. This reduced the price per extraction from about $40.00 to $9.00. This saves the company approximately $31.00 per extraction. With an average of 7 extractions per day, and an estimated 252 business days in 2020, this would potentially save $54,684 to a fragrance company using at least 300 ml of pentane in their current fragrance extraction.

5. Acknowledgment

The author would like to express his gratitude to Professor Erin Shufro, a division chair of Natural Sciences at Georgia Highlands College, without whose mentorship and guidance, this research would not be possible.

6. References

1. Interview with research and development team at the company, September 15, 2020.
2. Interview with a research and developer at the company, October 17, 2019.

7. Endnotes

The initial abstract included the amount of Pentane used by the company as 100 mL. This has been changed to 300 mL in this paper. The initial information was erroneous due to wrong data reported by the research and development department, and has been corrected upon further analysis of the method. This information has been corrected.