Introduction

Polymers are one of the defining elements of the modern world having become the predominant materials for virtually any function. One of the few flaws marring the incredible versatility of polymers is the difficulty of reusing or recycling them. Specifically in the packaging industry, where most products are single use, this difficulty is a significant concern in relation to ecological consequences and proper disposal. One major polymer of this kind is expanded polystyrene (commonly known by the misnomer Styrofoam). Expanded polystyrene's (EPS) ease of production, lightness, and ability to insulate incredibly well has led to its popularity for fast food containers as well as shipping. EPS is manufactured from styrene monomers, which are used to create tiny, spherical beads of polystyrene. Using a low boiling point hydrocarbon and steam, these beads will expand and fuse to fill molds. As a result, EPS is 98% air having an incredibly large volume compared to its mass[1]. Therefore, since it is also not biodegradable, EPS takes up a large amount of space in landfills and it is often not economically feasible to transport. Especially when stained with food, a meager amount of EPS is recycled[2].

Beginning in 2001, Sony Corporation set up a system in Japan for recycling EPS in an environmentally beneficial and economically feasible manner. Using the organic solvent d-Limonene (figure 1) (the principal essential oil in citrus fruits), Sony was able to dissolve packaging EPS on site significantly reducing its volume (1/50 of the size) before transporting. The EPS-d-Limonene solution could then be processed using minimal energy to recover new-grade polystyrene in various forms as well as the d-Limonene itself[3] [4]. Using various filtration systems even EPS contaminated with food or stains can be recycled. Other research projects have even shown that the d-Limonene can be used post dissolution as a monomer with the polystyrene using UV-catalysis to develop blended plastic networks greatly exceeding the properties of regular polystyrene[5].

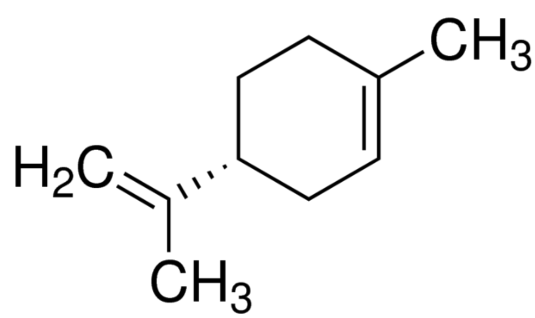

Figure 1
Structural formula of d-Limonene

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[1] Christian Block, "Expanded Polystyrene (EPS)," PlasticsEurope, , accessed October 28, 2016, http://www.plasticseurope.org/what-is-plastic/types-of-plastics-11148/expanded-polystyrene.aspx.

[2] LeBlanc, Rick. "EPS (Styrofoam) Facts, Figures and Statistics." The Balance. August 11, 2016. Accessed October 28, 2016. https://www.thebalance.com/expanded-polystyrene-foam-recycling-eps-facts-and-figures-2877914.

[3] "Orange R Net - Documents." Docslide.us. October 23, 2014. Accessed October 28, 2016. http://docslide.us/documents/orange-r-net.html.

[4] "Using Oranges for Styrofoam Recycling." CXEYE. 2001. Accessed October 28, 2016. http://xa.yimg.com/kq/groups/26556000/359094630/name/cxeye.pdf.

[5] Hearon, Keith. "A High-Performance Recycling Solution for Polystyrene Achieved by the Synthesis of Renewable Poly(thioether) Networks Derived from D-Limonene." 2014. MS PMC4000729, Texas A&M, Texas. November 19, 2013. Accessed October 29, 2016. https://www.ncbi.nlm.nih.gov/pmc/articles/PMC4000729/.

In eighth grade, I found out about the potential of d-Limonene for recycling EPS when researching for a science fair project. At that point in time I lacked the practical knowledge in chemistry to perform the experiment, which is what led me to my desire to investigate the concept when I had sufficient experience with chemistry.

For this investigation, it is of interest to look into the process for extracting d-Limonene from different citrus peel variants with high-yield and high-purity using steam distillation. EPS will then be dissolved in the d-Limonene extract to see the effectiveness of the solvent for recycling. Such methodologies could be employed by the juice industry on a larger scale in order to reuse their waste products as a means to recycle EPS waste creating a system fully utilizing all products of the industry. Specifically in this investigation, d-Limonene will be extracted from organic oranges, lemons, clementines, and grapefruits and a set of normal oranges purchased from Carrefour. A sample of 100% pure d-Limonene purchased from Amazon will be used to calculate the saturation point of EPS in d-Limonene for further calculations.

Research Question

How does the d-Limonene content in parts per million weight measured by means of extraction via steam distillation vary between citrus fruits (organic oranges, lemons, clementines, and grapefruits and non-organic oranges) for the purpose of recycling expanded polystyrene by means of dissolution?

Hypothesis

I predict that the largest amount of d-Limonene will be extracted per part of grapefruit since it has the thickest peel. With the same thought process, orange will have the next to largest amount then lemon, and then clementine. I expect the non-organic orange to contain more d-Limonene than the other fruits despite the impurities since the peel will be better preserved as a result of the applied chemicals and waxes. I expect the d-Limonene extracted from each fruit to work equally well for dissolving EPS.

Procedural Write Up[6] [7] [8]

The method used first involved the removal of the pith and rind from the citrus fruits followed by the distillation of the mixture to separate the d-Limonene. EPS was then dissolved in the extracted d-Limonene and a pure sample until saturation.

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[6] Saleh, Ahmed. "Extraction of the Essential Oil Limonene from Oranges." University of Reading. 2008. Accessed October 28, 2016. https://www.reading.ac.uk/web/FILES/chemistry/Limonene.pdf.
[7] Altig, Jeff. "Steam Distillation of an Essential Oil." New Mexico Institute of Technology. Accessed October 28, 2016. http://infohost.nmt.edu/~jaltig/SteamDistill.pdf.
[8] "Extracting Limonene from Oranges." Learn Chemistry. Accessed November 15, 2016. http://www.rsc.org/learn-chemistry/resource/res00000692/extracting-limonene-from-oranges?cmpid=CMP00000770#!

Theory

Use of Organic Citrus Fruits

In order to reduce the chance of having impurities in the d-Limonene extract organic citrus fruits were used. Waxes and fungicides applied to the rind for preservation could lead to lower yields or more impure extracts. However, a non-organic orange was used as well since on an industrial level for the production of juices most fruits are not organic. If the non-organic orange's extraction fails this would suggest that d-Limonene cannot be produced in a cost-effective manner on an industrial level.

Steam Distillation[9]

Since d-Limonene has a boiling point of 176^{o}C, distilling by typical means is not feasible due to the high temperature decomposing the compound before it can be extracted. In order to avoid this, steam distillation can be used. In steam distillation, the distilling pot is infused with steam, which carries the oil vapors into the condenser where they can be recovered. Since the citrus oils (including d-Limonene) and water are immiscible they will boil independently from each other. Boiling occurs when the vapor pressure of the mixture is equal to the atmospheric pressure; therefore, since there are two separate liquids, the atmospheric pressure will equal the sum of both their pure vapor pressures when the mixture boils.

$$P_{atm} = {P_{wat}}^{o} + {P_{oil}}^{o}$$

Since ${P_{wat}}^{o} \gg {P_{oil}}^{o}$ the mixture will boil at a temperature slightly below the boiling point of water allowing the oil to vaporize at much more reasonable conditions, specifically for d-Limonene, 98^{o}C. Given that ${P_{wat}}^{o}$ is 707.3torr, ${P_{oil}}^{o}$ is therefore 52.7torr given that atmospheric pressure is 760torr[10]. The d-Limonene will be carried over along with the water by the following ratio (where n is a number of moles)

$$\frac{n_{oil}}{n_{wat}} = \frac{{P_{oil}}^{o}}{{P_{wat}}^{o}}$$

If I were to distill off 10mL of water (equivalent to approximately 0.56mol), I would have carried over 0.041mol of d-Limonene or 5.6g given that d-Limonene's molar mass is 136.125gmol^{-1} [11]. This is significantly more d-Limonene than will be present in any citrus fruit so it is sufficient to distill a small quantity of the citrus mixture for the extraction.

Variables

Independent variable: species of citrus fruit (30g of peel for clementine, 60g for others)
Dependent variable: parts per million weight d-Limonene extracted from fruit variant
Controlled variables: fruit mass, volume of boiled mixture, and volume of distillate

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[9] Clark, Jim. "Immiscible Liquids and Steam Distillation." ChemGuide. February 2014. Accessed October 28, 2016. http://chemguide.co.uk/physical/phaseeqia/immiscible.html.
[10] "Wired Chemist." Wired Chemist. Accessed October 28, 2016. http://www.wiredchemist.com/chemistry/data/vapor-pressure.
[11] "D-Limonene." PubChem Compound Database. PubChem, 22 Oct. 2016. Web. 29 Oct. 2016.

Pilot Study[12]

In the pilot study, the citrus sample was refluxed for 30 minutes before distillation (figure 3). Theoretically, the heat from reflux would cause all the cells in the peel containing d-Limonene to burst allowing a larger amount to be collected. When the refluxed samples were distilled, the distillate was cloudy denoting the presence of d-Limonene. Literature described that two layers should be present since d-Limonene is not soluble in water. The d-Limonene was then separated using diethyl ether in a separatory funnel. Since d-Limonene is nonpolar it would dissolve in the diethyl ether forming the top layer. After being dried using a small quantity of anhydrous sodium sulfate, the diethyl ether was boiled off using a water bath in a fume cupboard leaving the d-Limonene. However, when this method was actually used results varied significantly between samples. Some fruits' distillates were not cloudy and no d-Limonene was left after the diethyl ether was boiled off. Furthermore, the yield was small. Therefore, the method below was used in its place. On an industrial level the method below would also require less energy and volatile chemicals making it more feasible as well.

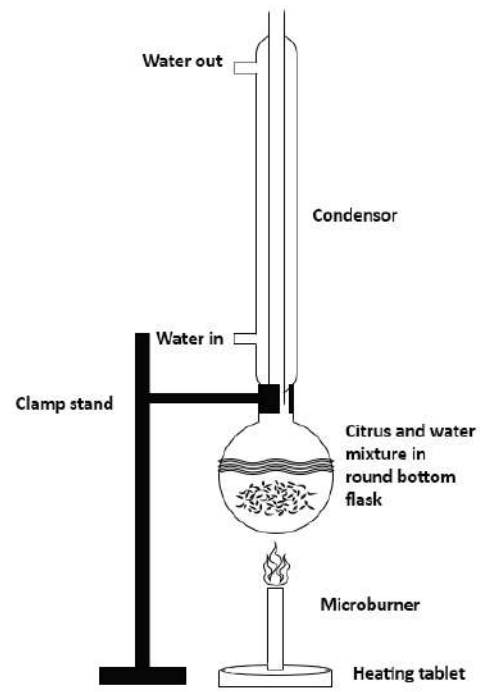


Figure 2
Setup diagram for pilot study reflux

Materials

- 1 Round bottomed flasks 250mL
- Condenser
- 2 Clamp stands
- 3 Clamps
- Anti-bumping granules
- Vaseline
- Weighing boats
- 1 Graduated cylinder 10mL
- 1 Graduated cylinder 5mL (±0.1mL)
- 1 Pipette 2mL (no measurement)
- 1 Crucible (used as a small bowl for dissolving EPS)
- Digital scale (±0.0001g)
- 1 Microburner
- 1 Heat proof mat (heating tablet)
- Lighter
- Propipetter

[12] "Extracting Limonene from Orange Peel." CLEAPSS. Accessed October 30, 2016. https://www.school-portal.co.uk/GroupDownloadFile.asp?GroupId=753103&ResourceId=5064670.

- Distilled water
- Pasteur pipette
- Blender
- 3 Oranges
- 6 Bio oranges
- 6 Bio lemons
- 9 Bio clementines
- 3 Bio grapefruits

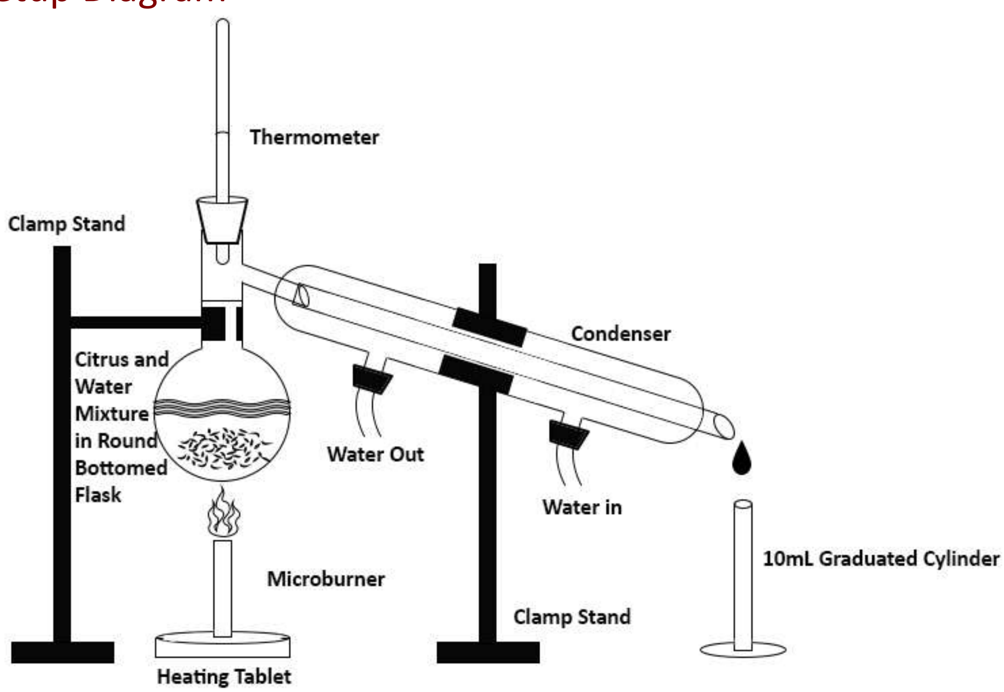
Chemicals

- D-Limonene ($C_{10}H_{16}$) (100% purity)
- Expanded polystyrene $(C_8H_8)_n$ ($21 mgcm^{-3}$)

Safety Considerations

- Wear safety glasses and a lab coat at all times
- Waste must be collected in separate beakers to be sorted into containers and professionally disposed of
- Tongs must be employed when moving hot glass
- D-Limonene is flammable in presence of open flames and sparks. It is hazardous as an irritant in cases of eye contact, skin contact, and inhalation[13]. It must be kept away from flames at all times and all work should be done in a fume hood.

Setup Diagram



[13] "DLimonene MSDS." ScienceLab.com. May 21, 2013. Accessed October 29, 2016. https://www.sciencelab.com/msds.php?msdsId=9924496.

Methodology

First the setup of the distillation apparatus

- Setup two clamp stands and attach one clamp to the middle of the condenser having it sloping downwards. The other clamp stand will be attached to the flask itself prior to each distillation.
- Attach the tubing to the condenser making sure that the bottom tube is connected to the water source
- Attach a microburner to the gas source and place it on top of a heating tablet beneath the high end of the condenser

Before extraction each fruit sample must be properly prepared

- Peel one fruit and shred it using a blender before weighing
- Continue peeling fruits and shredding until approximately 30g is weighed out for the clementine and 60g for the other fruits
- Add the blended peel to a round-bottomed flask with approximately 100mL of distilled water

Next, the distillation

- Before beginning the distillation, add anti-bumping granules to the flask and place a 10mL graduated cylinder beneath the end of the condenser
- Attach the flask with the mixture to the condenser using the clamp
- Turn on the water and light the burner
- Once boiling the reading on the thermometer will remain at 98^{o}C denoting the presence of d-Limonene
- Turn off the burner once there is 10mL of distillate. The d-Limonene will be present as an oily layer resting on the surface of the water
- Pipette off the layer of d-Limonene into a 5mL graduated cylinder and record the volume

The entire method for preparation and extraction is repeated for each of the citrus fruits three times each. Each trial requires upwards of thirty minutes to complete.

Lastly, pure d-Limonene will be saturated with EPS for the purpose of future calculations

- Decant one milliliter of pure d-Limonene into a crucible
- Dissolve granulated EPS in the d-Limonene keeping track of the EPS's mass
- While stirring the solution, keep adding EPS until the solution has reached saturation whereby EPS can no longer be dissolved
- Record the final mass of EPS added
- Repeat two more times to record an average

Data

Quantitative Data

Fruit	Mass of peel in g (±0.01g)		
	Trial 1	Trial 2	Trial 3
Non-Organic Orange	67.79	52.07	58.86
Organic Orange	71.42	53.21	52.47
Organic Grapefruit	61.92	51.82	56.38
Organic Clementine	36.18	37.78	24.94
Organic Lemon	72.37	67.77	61.18

Table 1-mass of citrus peels used for extraction

Fruit	Volume of d-Limonene extract in mL (±0.1mL)		
	Trial 1	Trial 2	Trial 3
Non-Organic Orange	0.7	0.6	0.4
Organic Orange	0.4	0.3	0.2
Organic Grapefruit	0.2	0.1	0.2
Organic Clementine	0.5	0.3	0.1
Organic Lemon	0.2	0.2	0.3

Table 2-volume of citrus extracts post distillation

Trial	1	2	3	Average
EPS dissolved in milligrams at room temperature (±0.1mg)	240.3	240.6	239.0	240.0

Table 3-mass of EPS in milligrams dissolved by one mL pure d-Limonene

Qualitative Data

After being mixed with water, each citrus mixture was composed of the peel and solution with the color of the rind. After distilling, an oily layer rested on the surface of the distillate. The scent of the distillate was distinctly that of citrus evidencing the extraction of d-Limonene.

Figure 3
Steam distillation of orange

Figure 4
Mixture of d-Limonene and water post distillation

Processed Data

Given that the density of d-Limonene is 841.1mgmL^{-1}, the number of milligrams in the extract can be calculated[14]

Worked example for the first trial of non-organic orange peel:

$$Mass = Density \times Volume$$

$$Mass = 841.1 \times 0.7 = 588.8mg$$

Fruit	Mass of d-Limonene extracted in milligrams		
	Trial 1	Trial 2	Trial 3
Non-Organic Orange	588.8	504.7	336.4
Organic Orange	336.4	252.3	168.2
Organic Grapefruit	168.2	84.1	168.2
Organic Clementine	420.6	252.3	84.1
Organic Lemon	168.2	168.2	252.3

Table 4-mass of d-Limonene extracted

In order to compare the amount of extract for different species of varying mass parts per million weight (ppmw) was calculated. This is equivalent to the milligrams of d-Limonene divided by the mass of peel used in kg[15].

Worked example for the first trial of non-organic orange peel:

$$Ppmw = \frac{Mass\ of\ extract}{Mass\ of\ peel}$$

$$Ppmw = \frac{588.8}{0.06779} = 8685ppmw$$

Fruit	Parts per million weight of d-Limonene (ppmw)			
	Trial 1	Trial 2	Trial 3	Average
Non-Organic Orange	8685	9692	5716	8031
Organic Orange	4711	4846	3206	4254
Organic Grapefruit	2717	1623	2984	2441
Organic Clementine	11624	6679	3372	7225
Organic Lemon	2324	2482	4124	2977

Table 5-parts per million of d-Limonene for each citrus variant

14 "D-Limonene." PubChem Compound Database. PubChem, 22 Oct. 2016. Web. 29 Oct. 2016.

15 "Ppm - Parts per Million." RapidTables. Accessed October 28, 2016. http://www.rapidtables.com/math/number/PPM.htm.

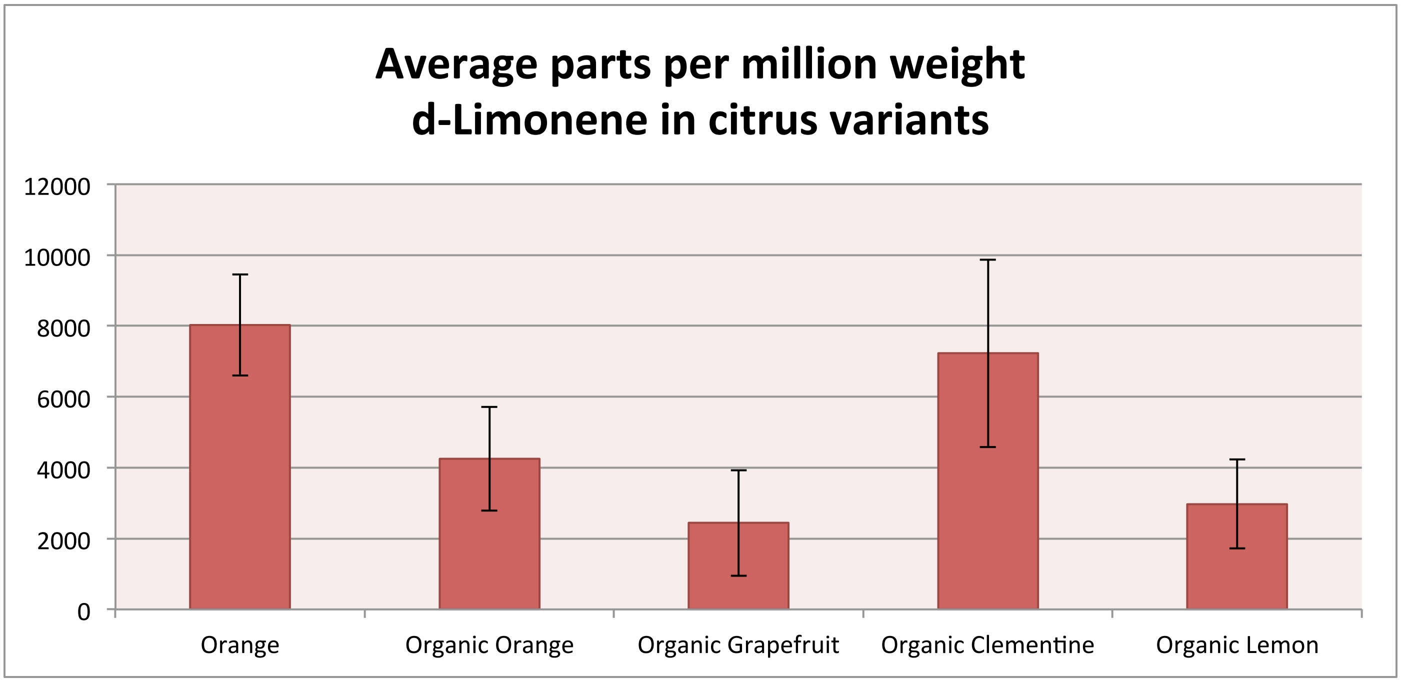


Figure 5
Graph of ppmw for each citrus variant used along with error bars for uncertainties

$$\%Uncertainty\ in\ peel\ mass = \frac{0.01}{peel\ mass}$$

$$\%Uncertainty\ in\ extract\ volume = \frac{0.1}{extract\ volume}$$

$$\%Uncertainty\ in\ ppmw = \%U\ in\ peel\ mass + \%U\ in\ extract\ volume$$

$$\%Uncertainty\ in\ average\ ppmw = \frac{\sum \%Uncertainty\ in\ ppmw}{3}$$

Interpretation of Results

According to the results, the organic species containing the most d-Limonene per part was the clementine with the smallest being the grapefruit. Despite having a smaller mass, a larger portion of a clementine's mass is the rind rather than the pith and vice versa for the grapefruit[16]. Since a majority of the d-Limonene in the peel is present in the rind this could lead to the greater content per part. Furthermore, the non-organic orange contained more d-Limonene per part than any of the organic variants. The use of pesticides/insecticides, preservative waxes, and genetic modification could easily lead to a larger concentration of d-Limonene per part.

[16] Saleh, Ahmed. "Extraction of the Essential Oil Limonene from Oranges." University of Reading. 2008. Accessed October 28, 2016. https://www.reading.ac.uk/web/FILES/chemistry/Limonene.pdf.

EPS cups produced yearly (US)[17]	Tons of orange produced yearly (US)[18]	Average EPS cup mass (mg)[19]	Average orange mass (g)[20]	Average d-Limonene content per orange (mL)
25,000,000,000	7,574,094	1500	315	0.7

Table 6-miscellaneous data for calculating effectiveness of d-Limonene recycling of EPS

$$Grams\ of\ orange\ produced\ yearly = 7{,}574{,}094 \cdot 10^6 \approx 7.57 \cdot 10^{12}\text{g}$$

$$Approximate\ number\ of\ orange\ produced\ yearly = \frac{7.57 \cdot 10^{12}}{315} \approx 2.40 \cdot 10^{10}$$

$$Millileters\ of\ d\ Limonene\ in\ yearly\ orange = 2.40 \cdot 10^{10} \cdot 0.7 = 1.68 \cdot 10^{10}\text{mL}$$

$$Grams\ of\ EPS\ soluble\ by\ yearly\ orange = 1.68 \cdot 10^{10} \cdot 0.24 = 4.04 \cdot 10^{9}\text{mg}$$

$$Grams\ of\ EPS\ cups\ produced\ yearly = 2.5 \cdot 10^{10} \cdot 1.5 = 3.75 \cdot 10^{10}\text{mg}$$

$$\%\ of\ EPS\ cups\ recylable\ by\ yearly\ orange = \frac{4.04 \cdot 10^{9}}{3.75 \cdot 10^{10}} \cdot 100 \approx 11\%$$

A year's worth of d-Limonene from oranges produced in the US would be sufficient to recycle 11% of EPS cups produced yearly. Considering that the d-Limonene is easily reclaimed when the EPS is recycled, it would be possible to recycle a considerable quantity of EPS using this method. A single truck could carry two 270-liter tanks of d-Limonene, which would be sufficient to dissolve 129.6kg of EPS or 2,592dm^3, which would be equivalent to 86,400 EPS cups.

Conclusion

The results of the experiment were completely contradictory to the hypothesis but reasonable when compared with outside information on the concentration of d-Limonene in different parts of citrus peels. Outside sources stated that oranges typically contain 0.5-1% d-Limonene by mass, which is supported by the results of the experiment suggesting that other extractions were similarly accurate. However, a similar experiment comparing d-Limonene content of various citrus species showed that grapefruits should contain the largest quantity of d-Limonene. The method used ethanol

17 "Styrofoam Recycling." Crush Recycling. Accessed November 15, 2016. http://crushrecyclingsolutions.com/?page_id=721.

18 "Crops." FAOSTAT Beta, 2013. Accessed November 15, 2016. http://faostat.fao.org/beta/en/#data/QC. Search: United States of America, Production Quantity, Oranges, 2013

19 "The Cup That Cheers Environmentalists." New Scientist. February 16, 1991. Accessed November 15, 2016. https://www.newscientist.com/article/mg12917562-700-the-cup-that-cheers-environmentalists/.

20 Report no. FCS-297. National Food Service Management, The University of Mississippi. Accessed November 15, 2016. http://www.fns.usda.gov/sites/default/files/quality_fruit.pdf.
Orange

rather than water for dissolution and the fruits were grown in a different climate but the contradicting results merit further investigation[21]. Additional calculations show that the method can recycle a considerable quantity of EPS with ease and considering the relative energy requirement compared to other recycling methods and the creation of new polymers, it could be an effective solution to EPS waste if put into practice[22]. However, regardless of how efficient this solution is for recycling all sorts of EPS, the quantity of EPS produced yearly is far greater and reducing its production in favor of other, more readily reusable or biodegradable materials would be a far better option.

Evaluation

Random Errors

Quantities of d-Limonene vary significantly between individual fruits as a result of where and how they are grown as well as random possibilities occurring over the growth of the fruit.[23] For this particular experiment this was evidenced by one sample of clementines producing 0.5mL of d-Limonene and a subsequent trial only producing 0.1mL. Any experiment would measure a large variance in results and experiments conducted with fruits purchased elsewhere could have completely contradictory data.

Due to time constraints only three trials were run for each citrus variant. Each distillation required at least thirty minutes to complete which would have exceeded the allotted lab time if more trials were performed. A more conclusive investigation would require more trials to counteract the large variance for individual fruits. Due to the small sample size, possibly anomalous data had to be considered for generating conclusions.

Systematic Errors

Since the citrus peels were blended rather than liquefied, it would be possible for some of the cells containing d-Limonene to not have broken[24]. This would result in less d-Limonene being extracted than possible. Refluxing was attempted in the pilot study to avoid this issue by keeping the mixture at a high energy for an extended period of time to reduce the energy but it was ineffective. Better results would be expected if the peel was liquefied or refluxed without losing oil.

The most accurate measuring apparatus that was available for volume only had a smallest increment of 0.1mL. This led to uncertainties as large as 100% for the extract volumes of the second trial of grapefruits and third trial of clementines where only 1mL

[21] May Sumi, H. The Effects of D-Limonene Extracted from Different Citrus Fruits on the Disintegration of Polystyrene Foam. PDF. CALIFORNIA STATE SCIENCE FAIR, April 2, 2010.

[22] "Using Oranges for Styrofoam Recycling." CXEYE. 2001. Accessed October 28, 2016. http://xa.yimg.com/kq/groups/26556000/359094630/name/cxeye.pdf.

[23] Altig, Jeff. "Steam Distillation of an Essential Oil." New Mexico Institute of Technology. Accessed October 28, 2016. http://infohost.nmt.edu/~jaltig/SteamDistill.pdf.

[24] "Extracting Limonene from Orange Peel." CLEAPSS. Accessed October 30, 2016. https://www.school-portal.co.uk/GroupDownloadFile.asp?GroupId=753103&ResourceId=5064670.

of d-Limonene was extracted. More accurate measurements would require an apparatus with better uncertainties. However, this is not relevant for determination of amount of extract for an industrial level.

It was often unclear whether all the d-Limonene was pipetted off from the mixture with water. Dissolution in diethyl ether could be used to separate the d-Limonene from the water more accurately.

For calculations of d-Limonene extracted, it was assumed that the extract was pure. However, citrus essential oils often only contain approximately 90% d-Limonene[25]. More accurate results could be obtained using an analytic method like gas chromatography or through a saturation calculation using EPS. The saturation calculations that were attempted did not give clear results since the equipment at school was not adequate.

Evaluation of Sources

The majority of the procedures for extraction, which also contained data on d-Limonene, came from university papers, which are generally more reliable than websites. However, many of the procedures described different qualitative results, which led to confusion during the pilot study. Many of the sources on EPS were from plastic producers, which could lead to biased discussion. Lastly, papers on the d-Limonene recycling program lacked sufficient figures and calculations so the success and viability of the program is not particularly clear.

Strengths

Unlike other methods involving organic solvents, the prescribed method solely relies on water, which is a much safer and environmentally friendly solvent. Furthermore, the only products of the method are water and d-Limonene, which additionally supports its environmental safety. The method is also quick in comparison to those using organic solvents and the entirety of the peel can easily be used without modification.

Extensions

It would be of interest to compare the cost requirements of synthesizing d-Limonene to extracting it from natural sources. While the appeal of extraction is to utilize waste products, the problem of recycling EPS is the primary concern since citrus peels can biodegrade. Synthesized d-Limonene could increase the economic feasibility, which would be of great benefit for the purpose of recycling all sorts of EPS on an industrial scale.

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[25] Altig, Jeff. "Steam Distillation of an Essential Oil." New Mexico Institute of Technology. Accessed October 28, 2016. http://infohost.nmt.edu/~jaltig/SteamDistill.pdf.

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