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Caffeine: Dealing With Extraction Emulsions

Because we are a small, undergraduate institution, our laboratory is primarily interested in method development of experiments to incorporate into undergraduate laboratory experiences. For years, we have had students extract caffeine from tea in both Organic Chemistry Labs and In Nursing Chemistry Labs. For years, these labs have been in one way very interesting to the students because it is "cool" to fish out white crystals of caffeine from a rather disgusting mess. On the other hand, dealing with the emulsion aspects of the actual extraction has also added a frustrating aspect to the interesting one.

We decided to undertake a further investigation of this long-tried undergraduate experiment. We analyzed a number of brands of teas, under various conditions. It appears that the addition of calcium carbonate to the mixture that causes the emulsion. The calcium carbonate is necessary to maximize the recovery of caffeine since it ties up the tannic acids that complex with the caffeine in tealeaves. We ended up discovering that just brewing a cup of tea, as recommended on the container, with the addition of calcium carbonate, gave the least messy results, as well as the highest recovery caffeine.

Because of the necessary addition of the calcium carbonate, we continued to deal with emulsions. We tried to deal with them using "time," salting out with NaCl, filtering through sodium sulfate and sonication. It appears that, albeit impractical on a large laboratory section scale, sonication is the most effective at dealing with the emulsion.

On a final note, the PI has encountered a number of emulsions, as have students at various points in time. This particular emulsion remains the most pesky!

CAFFEINE: Dealing With Extraction Emulsions

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Introduction

A very common and popular under-graduate laboratory experiment involves the extraction of caffeine from tea. This is an especially popular experiment because of its "WOW" factor. Students are amazed that after a lot of effort and work, they isolate a small amount of a lovely white powder.

As "cool" as this experiment is, it is plagued by very pesky emulsions.

Project Summary

The project consisted of several parts.

1. The method for extracting caffeine from tea had to be optimized.
2. A variety of teas (loose and in bags) had to be analyzed.
3. A variety of methods to break up emulsions had to be tested.

Our results were "interesting," to say the least.

The Teas

- Salada Tea was in tea bags (opened for extraction)
- Wagh Bakri was loose tea
- Tetley Tea was loose tea

Loose tea is the way to go

1st Extraction Method

a. 25.00g of tea were mixed with 10.00g $CaCO_3$ and 250mL water
b. This mix was boiled for 25 min, then filtered
c. Once cooled, the mix was GENTLY (rocking) extracted with two 50mL portions of CH_2Cl_2
d. Despite all precautions, this resulted in a pesky emulsion (and low recovery)
e. We decided to develop methods for getting rid of the emulsion

Emulsion Breakers

POSSIBLE OPTIONS (original trials)

1. Letting it sit (NG, even overnight)
2. Salting out (NaCl) (NG)
3. Filtering through Na_2SO_4 (so-so; first method of choice)

First Recovery Results

TEA	USED	CAFFEINE
Salada	25.00g	0.72%
Salada	25.00g	1.56%
Salada	25.00g	1.36%
Salada	25.00g	1.32%
Tetley	12.50g	1.97%
Tetley	12.50g	2.22%
Wagh Bakri	12.50g	1.58%
Wagh Bakri	12.50g	0.89%

First Conclusions

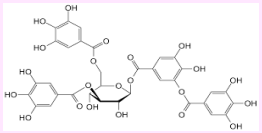
1. The emulsion seriously interferes with the amount of caffeine recovered.
2. We improved our techniques because the results became more consistent.
3. These results were obtained by "rocking," rather than shaking the separatory funnel.
4. Tetley Tea states, on their website, that caffeinated tea contains 4.0% caffeine.
5. Clearly, our initial recovery of caffeine was no way near that amount.
6. Why is the emulsion happening? We needed to check out more variables.

2nd Extraction Method

1. In order to conserve tea, we chose to use 2.00g of leaves (recommended for a single cup of tea.
2. The tea was brewed for ~4 minutes.
3. Calcium carbonate was added to some samples prior to brewing.
4. The separatory funnel with the dichloromethane was SHAKEN, rather than rocked.
5. The shaking resulted in a huge emulsion.
6. We focused on using ultrasound to disrupt the emulsion.
7. 10 minutes of treatment were sufficient.

Is the $CaCO_3$ Needed?

1. We have brewed tea (2.00g for ~4 minutes with and without the calcium carbonate.
2. Outcome: the base is needed to complex with the (acidic) tannins (below) to release the (basic) caffeine.



Comments

1. The percentages are based on crude masses
2. Tetley Tea: 2.00g; with calcium carbonate/ultrasound: 2% (average) recovery of caffeine.
3. Tetley Tea: 2.00g; w/o calcium carbonate/ultrasound:1.2% (average) recovered caffeine.
4. Crude caffeine was purified by sublimation (40% recovery)

Final Conclusions

1. The best way to deal with THIS emulsion is sonication.
2. The sonication should take place in a flask and not in a beaker or funnel.
3. The calcium carbonate IS essential for optimal extraction of the caffeine from tea.
4. One doesn't need to go through the messy extraction procedure outlined in lab books. It's enough just to brew the tea for ~4 minutes (as recommended) to get maximum recovery of caffeine.
5. Brewing 2.00g is easier than using 25.00; margin of error is larger.

Acknowledgments

All of the the success of this project is attributed to the following:

-The Independent College Fund of New Jersey (ICFNJ) (for the most welcome money)
-Bloomfield College (for space, equipment, supplies)
-Dr. Maria Vogt (for help and guidance)