Paper ID #39494

A Colligative Property Experiment using the Solvent Paraffin Wax

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Abstract

In this educational lab project, we propose using paraffin wax as an alternative organic solvent to those used in academic labs to demonstrate the concept of freezing point depression. We opted to use paraffin wax pellets as an organic solvent because many schools worldwide do not have the resources and access to purchase specialty chemicals. Paraffin wax is readily available to schools, safe to use, non-toxic, does not release a pungent or irritating smell, and yields reproducible results and trends. Data was gathered from at least fifty Chemistry Lab reports and assessed statistically against data obtained under controlled conditions. The Freezing point depression constant K_f (i.e. freezing point constant) of commercial paraffin wax is not available in the literature and our experimental analysis yielded a reproducible range of K_f = 5.0 to 5.5 ^oC.kg/mol at an average melting point of 54 ^oC. We picked Benzophenone and Naphthalene (i.e. mothballs) as two compatible nonpolar solutes that worked well with the solvent paraffin wax.

Keywords

Freezing point depression, a colligative property, paraffin wax, freezing point constant, cryoscopic constant, Naphthalene, Benzophenone.

Introduction

Academic Labs use room temperature organic solids such as Naphthalene, Acetamide, organic acids (e.g. Lauric, Stearic, benzoic), Cetyl alcohol, Cyclohexane, and Phenol as solvents to conduct the freezing point depression (i.e. F.P.D.) experiments[1 - 8]. In fact, these organic solids are suitable to use either as solvents or solutes because they have relatively moderate melting points such as Stearic acid/Naphthalene/acetamide (m.p. range ~ 69-80 ^oC)[1 – 4] and Lauric acid/Cetyl alcohol/Phenol (m.p. range ~40-50 ^oC)[1 – 5]. One experiment uses Camphor[9] as a solvent, but this chemical has a relatively high melting point (~175 ^oC), and thus needs a Bunsen burner to heat, a minimum of 200 ^oC thermometer, and glass test tubes. Naphthalene, Cetyl alcohol, and long-chain organic acids can dissolve some non-polar and polar solutes while phenol and acetamide are suited for moderately polar solutes[1 - 8]. A major disadvantage in the academic lab environment is the toxicity of most of these chemicals. For example, using large quantities of Naphthalene and Stearic acid have a pungent dizzying smell and its waste is difficult to handle and clean. Per the material safety data sheet (MSDS or SDS) of each listed chemical, vapors of phenol, cyclohexane, benzene, and acetamide are classified as a carcinogen, irritants, and corrosive to the eye and skin. Lauric acid and Cetyl alcohol are good organic solid solvents to use in F.P.D. experiments because these are relatively cheaper to obtain, however, Cetyl alcohol is known to cause skin allergy (i.e. dermatitis, eczema, ...etc.) while lauric acid uses benzoic acid as a solute and both may not be available to many schools. Benzoic acid can exhibit allergy symptoms because of its acute smell.

In this paper, we propose using paraffin wax that has a moderate m.p. range of ~ 50 to 60 ^{o}C[10] is readily available to instructors at schools worldwide, is safe to use, does not have a pungent smell, and shows reproducible temperature profile plots for accurate *freezing point depression* ΔT_f determinations. We have used plastic test tubes to perform the F.P.D. experiments and not glass test tubes and all these were dumped safely in waste bins, so no cleaning was necessary. Cleaning the glass test tubes is one of the major safety and logistical issues that face instructors and technicians supervising a colligative property lab. Moreover, Paraffin wax is relatively cheaper to get in large quantities (i.e. about 10 to 15 times cheaper than Naphthalene) and to use as a solvent. Academic institutions in developed or developing countries will find this Lab procedure less expensive and safer to conduct for students. For example, instructors might use white candle wax as a solvent and a tiny quantity of mothballs (i.e. source of naphthalene or 1,4-dichlorobenzene) as a solute to perform the F.P.D. experiment. In general, referenced literature uses glass test tubes to perform the colligative property experiment but many schools cannot afford multiple glass test tube breakages and damage. Plastic test tubes are extremely cheap and affordable and hence easy to dispose of after an experiment in a waste accumulation area.

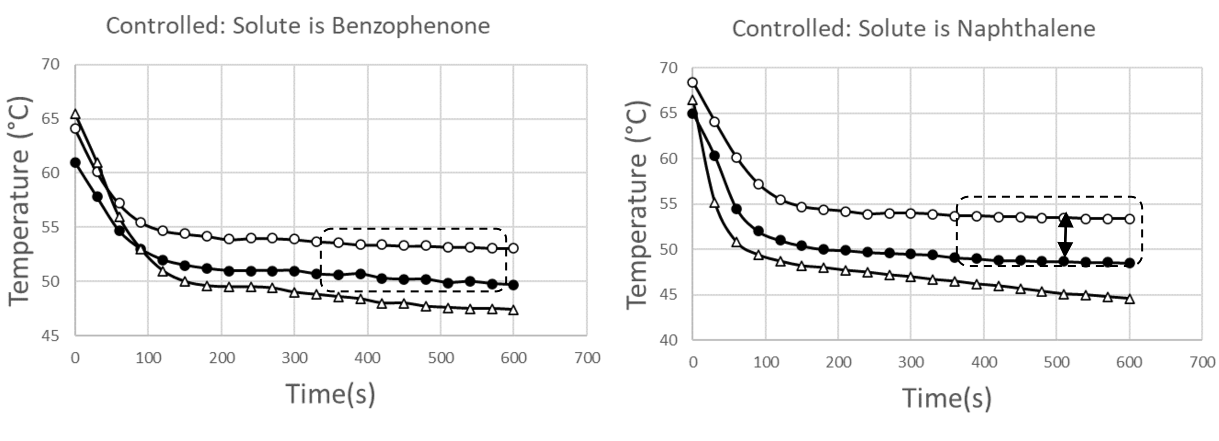
Experimental Procedure

Chemical reagents and hardware

Wax Paraffin Pastilles were obtained from Spectrum, Benzophenone 99% from Merck, and Naphthalene from Aldrich. Polycarbonate clear plastic test tubes with plastic covers from Corning(Falcon 14mL), m.p. ~ 240 ^{o}C.

Experiments by Students

Chemicals from suppliers were used without any further processing. The experimental method is a modified technique taken from the WVU-Tech Lab Manual[11], however, the solvents and solutes were changed. Wax pellets were used as a solvent to replace Naphthalene while Naphthalene or Benzophenone became the solutes. Polycarbonate test tubes were used instead of glass test tubes. Using the mass difference method, the mass of the empty plastic test tube was weighed first and then filled with wax pellets to be weighed again. A mass of around 5.00±0.1 g of paraffin wax was measured accurately with an analytical balance. A hot water bath on a hot plate was maintained at a temperature of approximately 80 to 90 ^{o}C using a digital thermometer of ±0.01 ^{o}C tolerance. The wax-filled plastic test tube was immersed in the hot water bath to melt the paraffin wax of m.p. melting point of 54 ^{o}C according to the literature[10]. However, the experimental m.p. obtained by CHEM115L students is an average of 54±1.0 ^{o}C. When the solid completely melts to liquid wax, the plastic test tube containing the hot melt is positioned inside a larger glass tube or Erlenmeyer flask to minimize temperature fluctuations during the cooling process. It is more convenient to have two students monitor the temperature change with time. While one student stirs the hot melt with the thermometer and reads the temperature change, the other student inputs the data. In our electronic reports, data points automatically appear on a temperature vs time plot. The temperature vs time trend during the solidification of paraffin wax melt can be examined until the wax solidifies and cannot be stirred. Any irregularities in the expected trend where temperature slowly decreases until it levels off will make a student or a group repeat the experiment. After obtaining the temperature profile of the pure paraffin wax solvent, the same experimental procedure was repeated by dosing the solvent with the respective solute i.e. either Benzophenone or Naphthalene. Prior to experiments conducted by CHEM115L students in Labs, the proposed method was validated in terms of reproducibility of data, clarity to obtain ΔT, and determination of F.P. constant K_f of paraffin wax by the lab manager and teaching assistants. Under controlled conditions, validation experiments were conducted such that the solute dosing of Benzophenone or Naphthalene was made at two levels. A lower dosing level of 0.6 g /(5 g of PW) and an upper dosing level of 0.9 g /(5 g of PW). This way we can check the sensitivity of the proposed paraffin wax solvent to the dosing solute level thus validating if the method works or not. The plots shown in Figure 1 depict the pattern of results obtained under controlled conditions by the TAs using the two dosing levels of solutes. These plots proved that the proposed solvent and technique qualify for in-class evaluation. The dose of 0.9g is excluded, CHEM115L students were asked to measure a mass of 0.6 g of the solute. Record gathered from CHEM115L students' e-Lab reports revealed the mass of solute measured would range from 0.6 to 0.7 g (±0.05 g). In the region of the dotted window, students would calculate ΔT by moving the double arrow across. Data among student groups were analyzed statistically and discussed. Readers are referred to the experimental procedure provided in the supplement.



(a) (b)

Figure 1 Under controlled conditions, cooling of ~5.0 g of pure paraffin Wax ○ using the solute (a) Benzophenone (0.6g ●, 0.9g △) , (b) Naphthalene (0.6g ●, 0.9g △). In the dotted window, ΔT is calculated.

Hazards

Paraffin wax is safe while Benzophenone and Naphthalene are irritants. Naphthalene does not have a repulsive smell when used in small quantities (i.e. as a solute of 0.6 to 0.7 g). A prudent measure is to handle all chemicals using gloves, aprons, and goggles. The authors strongly recommend using plastic test tubes for this experiment because it is easier to discard these with their content into waste rather than cleaning them. Experience shows that cleaning glass test tubes from low-melt solids such as paraffin wax, Naphthalene, Benzophenone,…etc. is hazardous and not economical.

Results and Discussion

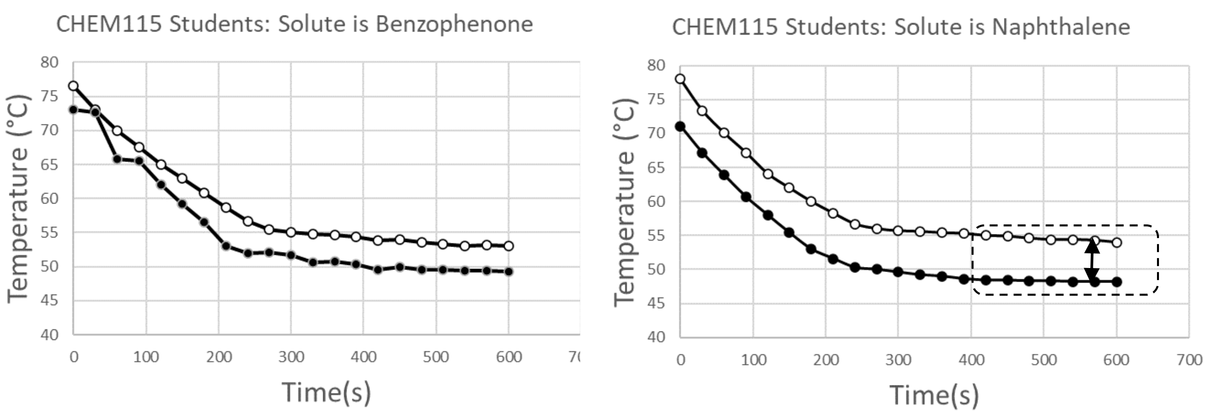
To be able to determine the value of the freezing point constant (K_f) of paraffin wax we used the formula,

$$K_f = \frac{\Delta T}{[m]} \qquad (1)$$

$[m]$ is the molality of solute,

$$\Rightarrow \; K_f = \Delta T . m_{PW} . \frac{FW_s}{m_s} \qquad (2)$$

FW_s is the molar mass of solute obtained from reagent bottles, m_s is the mass of the solute in *g*, and m_{PW} is the mass of solvent paraffin wax in *kg*. Values of ΔT were obtained from temperature profile plots such as in Figure 2 obtained from experiments done by CHEM115L students.



(a) (b)

Figure 2 Sample plots from CHEM115L students, cooling of ~5.0 g of pure paraffin Wax ○ using the solute (a) Benzophenone (0.6-0.7g ●) , (b) Naphthalene (0.6-0.7g ●). In the dotted window, ΔT is calculated.

For example, along a region where the change in temperature is nearly constant or parallel to the time axis (i.e. steady-state temperature), a line can be extrapolated to intersect the temperature axis to determine T_{avg} of either the pure solvent or the solvent dosed with solute. The temperature gap (i.e. double arrow in the dotted window) between the pure solvent and the mixture ΔT_f represents the freezing point depression F.P.D. such that,

$$\Delta T_f = T_{avg}\text{ (solvent)} - T_{avg}\text{ (mixture)}$$

Method Validation

Data obtained under controlled conditions, Figure 1, were considered our reference frame to conduct a *t-test* on CHEM115L students' results. These controlled measured values of ΔT, m_s, and m_{PW} were substituted in equation 2 to determine the values of K_f for paraffin wax as shown in Table 1. Note, as of the date when this paper was submitted there is no literature value of paraffin wax freezing point constant or cryoscopic constant that can be referenced.

Table 1 Values obtained under controlled conditions

	m_{PW} x10^{-3}(kg)	m_s (g)	ΔT (°C)	K_f (°C.kg/mol)
Naphthalene	5.0±0.2	0.6±0.1	4.95±0.05	5.02±0.02
	5.0±0.2	0.9±0.1	7.15±0.15	5.05±0.03
Benzophenone	5.0±0.2	0.6±0.1	3.35±0.05	5.01±0.02
	5.0±0.2	0.9±0.1	5.05±0.15	5.07±0.04

The second source of data was gathered from CHEM115L students' electronic reports where the solutes of choice were Benzophenone and Naphthalene. Students were asked to measure around 0.6g to 0.7g of these solutes to conduct these experiments. The mass of the solvent paraffin wax was measured at exactly 5.0±0.2 g using an analytical balance. A total of 30 independent data sets were analyzed for each solute in this study. From equation 2, temperature change ΔT (°C) is directly proportional to the mass of the dosing solute m_s and is strongly affected by its value. Theoretically, for ±0.06g difference in m_s, it can cause a 10% change in ΔT (°C). On the other hand, a ±0.1g difference in the solvent m_{PW}, can cause 2% change in ΔT (°C). Consequently, for the colligative property experiment, the three parameters that were considered for statistical analysis are the temperature change ΔT (°C), the mass of the dosing solute m_s, and the Freezing point depression constant K_f (°C.kg/mol). To be able to assess the means ū, we calculated the ratio values $\Delta T/m_s$ and got their average. The mean ū and the standard deviation σ of all $\Delta T/m_s$ and K_f values from all reports were calculated and used in the *t-test*. For example, from the 3rd row in Table 1, the mass of the solute 0.60±0.1 g, the reference temperature change is ΔT = 3.35±0.05 °C, and the corresponding K_f = 5.01±0.02 °C. kg/mol was our reference frame for Benzophenone. Hence, the *t-test* was done against the value of $\Delta T/m_s$ = 5.55 °C/g and K_f = 5.01 °C.kg/mol using Benzophenone. In the same manner, a *t-test* was done against values of $\Delta T/m_s$ = 8.25 °C/g and K_f = 5.02 °C.kg/mol using Naphthalene.

From the second source, where data is expected to scatter and fluctuate according to student's performance, the mean ū and the standard deviation σ are calculated using a spreadsheet and conveniently presented in Table 2. Based on our controlled experiments and results obtained from our CHEM115L students' reports, we can confidently report a freezing point depression constant K_f range of 5.0 to 5.5 ^oC.kg/mol. It is up to instructors or lab supervisors at schools worldwide to either use our value of K_f to determine the molar mass of a solute or pick another nonpolar solute like iodine, camphor,…etc. to obtain a value of the freezing point constant K_f.

Table 2. CHEM115L experiments, statistical parameters of ū and σ related to physical parameters $\Delta T_r/m_s$ and K_f

Chemical	*Physical*	ū[a]	σ
Benzophenone	$\Delta T_r/m_s$	5.57	±1.65
	K_f	5.52	±1.42
Naphthalene	$\Delta T_r/m_s$	7.90	±2.73
	K_f	5.04	±1.65
Comment	[a]The mean ū and SDEV σ were derived from 60 CHEM115L experiments. *Literature K_f value of paraffin wax is not available		

The *t-test*:

For a normal distribution of $\Delta T_s/m_s$ values, the average temperature change was 5.5 ^oC. In terms of %population, ~68% of the data points fall within ū ± 1.σ and ~95% fall within ū ± 2.σ range. The confidence interval CI of a *t-test* where t = 2.08 at 95% confidence level for *dof* n - 1 = 29 is determined from,

$$CI = \bar{u} \pm \frac{t.\sigma}{\sqrt{n-1}} \tag{3}$$

Benzophenone, Naphthalene,

$$CI = 5.57 \pm \frac{2.08\text{x}1.65}{\sqrt{30-1}} \quad , \quad CI = 7.90 \pm \frac{2.08\text{x}2.73}{\sqrt{30-1}}$$

Benzophenone: A CI range of 4.93 to 6.21 obtained by our CHEM115L students contains the reference value $\Delta T_s/m_s$ = 5.55 $^oC/g$ which correlates with the work conducted by our TAs under controlled conditions.

Naphthalene: A CI range of 6.85 to 8.96 obtained by our CHEM115L students contains the reference value $\Delta T_s/m_s$ = 8.25 $^oC/g$ which correlates with the work conducted by our TAs under controlled conditions.

For a normal distribution of freezing point depression constant K_f values, a reasonable range of K_f was obtained between 4.1 to 6.4 ^oC.kg/mol. In terms of %population, the confidence interval CI of a *t-test* where t = 2.08 at 95% confidence level and n - 1 = 29 degrees of freedom is calculated from equation 3 and Table 1, Benzophenone Naphthalene

$CI = 5.52 \pm \frac{2.08 \times 1.42}{\sqrt{30-1}}$, $CI = 5.04 \pm \frac{2.08 \times 1.65}{\sqrt{30-1}}$

Benzophenone: A CI range of 4.97 to 6.07 contains the reference value K_f = 5.01 $^oC.kg/mol$ which correlates with the work conducted by our TAs under controlled conditions. On the other hand, Naphthalene with a CI range of 4.39 to 5.67 contains the reference value K_f = 5.02 $^oC.kg/mol$ which correlates with the work conducted by our TAs under controlled conditions. The difference in K_f values between CHEM115L students' experiments and the reference values obtained under controlled conditions is negligible for Naphthalene while it is around 10% for Benzophenone. The difference can be attributed to the polarity of the solute where Benzophenone is more polar than Naphthalene and paraffin wax. Other factors are students' random technical skills using labware and following the procedure.

This statistical analysis that relied on gathering data from WVU-Tech student e-lab reports has demonstrated that a cheap and safe solvent like paraffin wax can be a viable and more adaptable green chemical for academic lab work. The low m.p. of paraffin wax and its relative chemical inertness allowed the use of cheap plastic test tubes to conduct the colligative property experiment successfully. When students used Naphthalene or Benzophenone as solvents before the introduction of paraffin wax, a few students had to walk outside the lab during the heating process because of these solvent's pungent smells. Using paraffin wax made the experiment setup and preparation easier as well as waste disposal friendly. For example, instructors who would use Naphthalene, usually ask students to reheat the Naphthalene mixture after an experiment to remove the melt from the glass tubes. It takes some effort to remove the remaining Naphthalene stuck to the glass tube walls. The CHEM115 students were more comfortable with paraffin wax solvent when they were performing the experiment and collecting data. The proposed experiment was conducted faster while higher grade performance was not compromised. This would open the door for educators in chemistry to use this green chemistry method that is cheap and safe to use.

Conclusion

In this paper, the freezing point depression constant K_f of paraffin wax was determined to serve experiments on colligative properties. Instructors can use the value of K_f obtained in this paper, to determine the formula weight of an unknown solute. This work uses the solvent paraffin wax to replace common solvents such as Camphor, Naphthalene, and Benzophenone. Paraffin wax proved to be safe to use, does not smell or irritate, and is ten times cheaper than other solvents. The relatively low melting point and chemical inertness of paraffin wax made it suitable to conduct the colligative property experiment using a plastic test tube instead of the common glass test tubes. Instructors can use Naphthalene, or mothballs, as a solute rather than a solvent. When done, the plastic test tube containing the wax mixture can be safely disposed of. This saves major effort in the cleaning of glass test tubes where these sticky solvents are difficult to remove mechanically or dissolve in other chemical solvents. Consequently, less waste is disposed of down the sink making it safer and more economic to work with.

Acknowledgments

The authors would like to thank all students at West Virginia University Institute of Technology (WVU-Tech) who contributed to this work directly or indirectly. Our thanks are extended to WVU-Tech for the continued support of our research.

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