World Journal of Chemical Education, 2016, Vol. 4, No. 3, 49-51
Available online at http://pubs.sciepub.com/wjce/4/3/1

DOI:10.12691/wjce-4-3-1

Convenient and Inexpensive Insulation for Fractional Distillation Columns

Daniel D. Clark*

Department of Chemistry, Mathematics, and Physics, Clarion University of Pennsylvania, Clarion, USA
*Corresponding author: dclark@clarion.edu

Abstract A convenient and inexpensive method for insulating fractional distillation columns in undergraduate laboratories with pre-slit, tubular pipe insulation that is widely available in hardware stores is described. This insulation can be quickly employed, can provide uniform insulation, and can be reused, which reduces the amount of trash generated from commonly used insulation.

Keywords: *laboratories and demonstrations, organic chemistry, separations, laboratory equipment, experiment, fractional distillation*

Cite This Article: Daniel D. Clark, "Convenient and Inexpensive Insulation for Fractional Distillation Columns." *World Journal of Chemical Education*, vol. 4, no. 3 (2016): 49-51. doi: 10.12691/wjce-4-3-1.

1. Introduction

Fractional distillation is an important separation technique in the organic chemistry laboratory curriculum. It is included in laboratory textbooks, taught at many institutions, and employed in labs for recycling waste acetone [1]. Recently, several distillation improvements have been published and were specifically designed for implementation in undergraduate labs. One report describes the use of condensers containing a static amount of antifreeze to enable condensation of distillate without the water usage typically associated with condensers [2]. Other reports have focused on the nature and type of packing material employed in fractionating columns, such as the use of an inexpensive brush as packing with excellent results [3]. One factor that has not been specifically addressed, yet is required for efficient separations, is the maintenance of adiabatic conditions within the fractionating column. In research laboratory settings, vacuum-jacketed columns and variable-temperature heating tape can be employed; however, there has been a lack of practical solutions for use in undergraduate labs where minimizing the heat loss from the columns could improve efficiency and student success. In these labs Liebig condensers are commonly packed and used as the fractionating column. Even though the air-jacketed condenser for these columns provides some inherent insulating properties, they are frequently further insulated against heat loss regardless as to whether it is a requirement or a precautionary measure for any particular system. A brief review of organic lab texts shows that common methods for insulating fractionating columns involve loosely wrapped aluminum foil and/or glass wool [4]. Although these methods provide insulation, there is a tendency for the aluminum foil to be wrapped too tightly, which diminishes its insulating value, and it is also discarded following the experiment. Loose glass wool can pose an irritation hazard and become quite untidy–especially if oil baths are used for heating. To circumvent these issues, pre-slit, tubular pipe insulation that is widely available in hardware stores has been found to be a convenient and inexpensive insulator for fractionating columns.

2. Results and Discussion

During the Fall 2015 semester approximately 50 students in three different sections of sophomore organic laboratory separated a binary mixture by fractional distillation. The fractionating columns used for this experiment were the commonly used Liebig condensers (standard taper (19/22), 180 mm column length) packed with copper scouring pad. For less than $5 (USD), twenty of these cylindrical columns were insulated with pre-slit, tubular closed-cell polyethylene foam intended for insulating ¾ inch residential pipes. This insulation was cut into ~10 ½ inch segments to cover the fractionating column and distillation head as shown in Figure 1. Using this insulated apparatus students produced the distillation plots for the separation of the following binary mixtures: (1) ethyl acetate (77°C)/ 4-methyl-2-pentanol (bp 132°C); (2) ethyl acetate (bp 77°C)/ 1-butanol (bp 117°C); (3) methanol (bp 65°C)/ 1-propanol (bp 97°C); and (4) acetone (bp 56°C)/ isopropyl acetate (90°C), as shown in Figure 2. It was also found that insulating the fractionating column with the tubular foam was considerably simpler as compared to insulating with aluminum foil and paper towels that was previously used. The advantage of the tubular foam was that it was quickly slipped onto the fractionating column whereas the aluminum foil had to be carefully wrapped around the column to secure the paper towels.

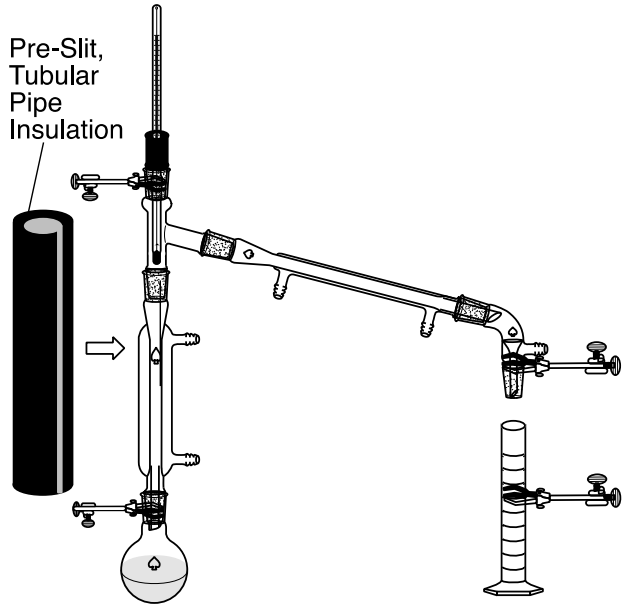

Figure 1. Schematic for employing pre-slit, tubular pipe insulation with a fractional distillation apparatus

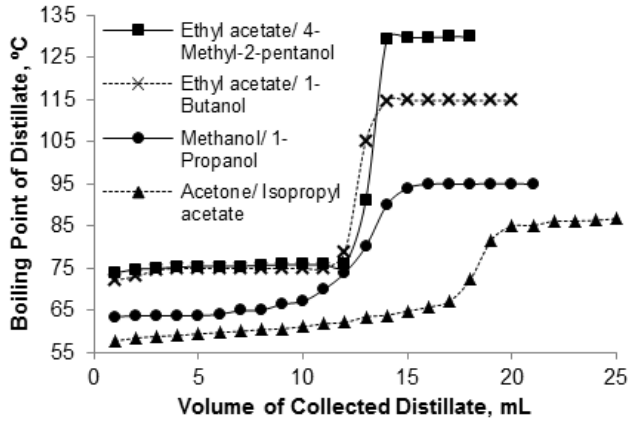

Figure 2. Example student fractional distillation plots for the separation of the four different binary mixtures employed for this experiment. These plots were selected from those submitted upon request at the end of the semester

Further support for the effectiveness and efficiency of tubular polyethylene foam for insulating fractionating columns comes from distillation experiments employing a glass Vigreux column (standard taper (19/22), 180 mm column length), which did not have the air-jacket of the Liebig column. In three different experiments a 50% *(v/v)* methanol and 1-propanol mixture was separated by fractional distillation under similar conditions with the same equipment, save for the fractionating column that was either left uncovered (non-insulated), insulated with tubular polyethylene foam, or insulated with a layer of paper towels and aluminum foil, respectively [5]. As indicated by the fractional distillation plots from these three experiments (Figure 3, Panel A), the components of the binary mixture were separated with similar effectiveness. However, the efficiency of these distillations was quite different. The insulated fractionating columns provided separations that were >25% faster *(on average at similar points of the distillation)* as compared to the non-insulated fractionating column (Figure 3, Panel B). This evidence further supports the proof-of-concept that the tubular polyethylene foam has comparable insulating properties as aluminum foil-based insulations. The clear advantages of the tubular polyethylene foam over the aluminum foil-based insulations were that the tubular foam was more convenient to employ and more practical for observing the rising condensate ring in the fractionating column as the tubular foam did not require disruption to do so.

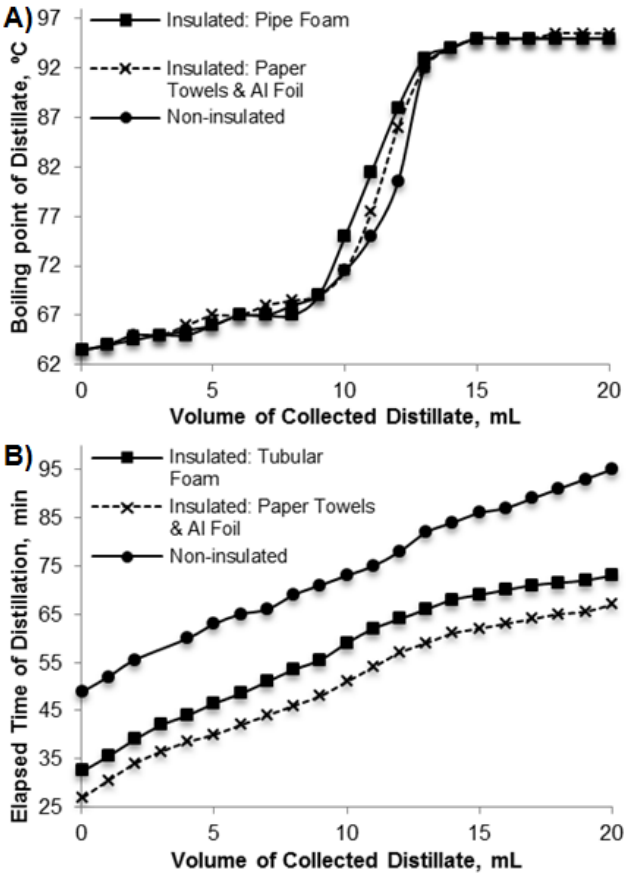

Figure 3. Data for the separation of a 50% *(v/v)* methanol and 1-propanol mixture under similar conditions with a Vigreux fractionating column that was either non-insulated or insulated with either tubular polyethylene foam or paper towels and aluminum foil, respectively. A) Fractional distillation plots of boiling point versus volume of collected distillate. B) Plots of elapsed time of distillation versus volume of collected distillate

Although the tubular polyethylene foam pipe insulation was successful in the aforementioned experiments, the insulating and operating temperature ranges of other available tubular pipe insulation materials are worth considering. As shown in Table 1, the polyethylene foam has a marginal advantage over the other materials in terms of the insulating R-value, but it does have a lower maximum temperture rating as compared to fiberglass. In the distillation experiments described in this report the polyethylene foam did not show any signs of deterioration, even those involving 4-methyl-2-pentanol (bp 132°C) despite the recommended maximum temperature rating of 95°C for the material. Since the fractionating column was an air-jacketed Liebig condenser and a Keck clip was used at the distillation head, the durability of the polyethylene foam has been attributed to the foam being in direct contact with surfaces at temperatures lower than the actual boiling point of this distillate. The only deterioration of the tubular polyethylene foam was observed when it was intentionally placed in direct contact with the outside of

non-jacketed glassware containing refluxing vapor of 4-methyl-2-pentanol. In these instances, the type of deterioriation that was observed for the polyethylene foam was the collapsing and contracting/melting of the inside surface of the tubular foam. It is important to note that in the comprehensive safety profile for this polyethylene foam, the National Fire Protection Association Rating for its flammability was assessed to be only a 1 (on a 0-4 scale, where 4 represents the most risk and a rating of 1 corresponds to possible ignition upon strong heating) [6]. However, if higher distillation temperatures were required for a particular application or if the insulation were in direct contact with the distillation head, the use of fiberglass tubular pipe insulation may be more practical.

Table 1. Properties of Pre-Slit, Tubular Pipe Insulation Available at Hardware Stores [a]

Material Type	R-Value[b]	Maximum Operating Temperature Rating[b]	Part # [a]
Closed-cell polyethylene foam	2.2	95°C	SP511XB6
Rubber	2.1	95°C	R534XB/6
Fiberglass	2.1	150°C [c]	F11X

[a] Manufactured by Nomaco, Inc., available from Thermwell Products under the Frost King brand name. [b] Information provided by Thermwell. [c] Commercial use fiberglass insulation from Owens-Corning (ASJ-Max) is rated to 535°C.

3. Conclusions

In addition to its low cost, there are a number of advantages to using pre-slit, tubular pipe insulation as compared to the traditionally employed aluminum foil or glass wool for insulating fractional distillation columns. The tubular insulation minimizes trash generation as this insulation can be easily shared between many laboratory sections and can be saved for use over several years. Also, the tubular insulation provides a more time-effective set up with a more uniform and tidy appearance. Lastly, the rising condensate ring in the fractionating column can be readily observed in the column through the narrow window provided by the slit throughout the experiment as compared to making "windows" in the foil or glass wool.

Acknowledgement

I would like to thank Clarion University of Pennsylvania for supplies and facilities, my students for trying this modified laboratory procedure in CHEM 251 (Organic Chemistry Laboratory), and my colleague Jonathan Touster for his useful comments.

References

[1] Weires, N. A., Johnston, A., Warner, D. L., McCormick, M. M., Hammond, K., McDougal, O. M., "Recycling of Waste Acetone by Fractional Distillation", *J. Chem. Educ. 88* (12), 1724-1726, December 2011.

[2] Baum, E. W., Esteb, J. J., Wilson, A. M., "Waterless Condensers for the Teaching Laboratory: An Adaptation of Traditional Glassware", *J. Chem. Educ. 91* (7), 1087-1088, July 2014.

[3] Robinson, J. M., Williams, D. L., "An Efficient 'Brush' Packing for Fractional Distillation", *J. Chem. Educ. 91* (3), 457-460, March 2014.

[4] (a) Gilbert, J.C., Martin, S.F., *Experimental Organic Chemistry: A Miniscale and Microscale Approach*, 5th ed., Cengage Learning: Boston, MA, 2011. (b) Pavia, D.L., Lampman, G.M., Kriz, G.S., Engel, R.G., *A Small Scale Approach to Organic Laboratory Techniques:*, 3rd ed., Brooks/Cole: Belmont, CA, 2011. (c) Ault, A. *Techniques and Experiments for Organic Chemistry*, 6th ed., University Science Books: Sausalito, CA, 1998. (d) Landgrebe, J.A. *Theory and Practice in the Organic Laboratory: with Microscale and Standard Scale Experiments*, 5th ed., Thomson Brooks/Cole: Belmont, CA, 2005. (e) Fessenden, R.J., Fessenden, J.S.; Feist, P. *Organic Laboratory Techniques*, 3rd ed., Thomson Brooks/Cole: Belmont, CA, 2001.

[5] Distillations were performed in an open fume hood with 25 mL of a 50% *(v/v)* methanol and 1-propanol mixture contained in a 100 mL round bottom flask and heated with a Powermite-controlled 100 mL Thermowell heating mantle as part of the distillation apparatus as referenced in Figure 1– except for the incorporation of a Vigreux column that was either left uncovered, insulated with tubular polyethylene foam, or insulated with a layer paper towels and aluminum foil, respectively. Each of these experiments employed similar Powermite control settings throughout the duration of the distillations to facilitate a slow rise of the condensate through the fractionating column and the collection of distillate at similar a pace during similar points of each separation *(i.e. 3-5 sec per drop for the collection of methanol and 1.5-3 sec per drop for the collection of 1-propanol).*

[6] Material Safety Data Sheet [online] for Nomaco Construction Polyethylene Foam Pipe Insulation; Nomaco, Inc.: Zebulon, NC (USA), September 15, 2014. http://www.nomacoinsulation.com/pdf/msds/MSDS%20PE%2006 2911_002.pdf (accessed May 6, 2016).