

A Thin Layer Chromatography Prelaboratory Activity Using a 3D-Printed Model to Address Student Misconceptions about Polarity and Intermolecular Forces

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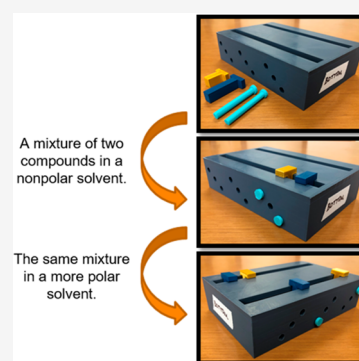
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ABSTRACT: Normal phase thin layer chromatography is a widely taught technique in undergraduate organic chemistry laboratory courses. To understand how the technique works, students must have a strong conceptual understanding of polarity and intermolecular forces. Unfortunately, many students have misconceptions regarding how polarity and intermolecular forces affect the separation of compounds via thin layer chromatography. This work aimed to address these misconceptions by designing and implementing a prelaboratory activity using a 3D-printed model of a TLC plate. Data from pre- and postactivity surveys suggest the activity has a positive impact on student understanding, and student perceptions of the activity are overwhelmingly positive.



KEYWORDS: *Thin Layer Chromatography, Prelaboratory Activity, 3D Printing, Laboratory Instruction, Organic Chemistry, Student Misconceptions, Polarity, Intermolecular Forces*

INTRODUCTION

Normal phase thin layer chromatography (TLC) is a commonly taught technique in introductory organic chemistry laboratory courses.¹ Knowledge of the concepts and procedures underlying the technique are valuable for students to learn because TLC is widely used in industrial and research chemistry, and companies in the chemical industry cite the technique as a desired one in Bachelor's-level hires.^{2,3} TLC is frequently used to monitor reaction progress and as a precursor to other chromatographic methods and techniques, but students need to understand how polarity and intermolecular forces (IMFs) affect the separation of compounds on a TLC plate before they can apply the technique for these applications.

There are a variety of experiments used in colleges and universities to help students learn how polarity and IMFs affect the separation of compounds via TLC.^{4–8} However, simply integrating the technique into one or more laboratory experiments may not be sufficient because students may focus on procedural issues rather than the underlying theory and how it connects to the experiment at hand.^{9,10} To improve student comprehension of how polarity and IMFs affect the separation of compounds via TLC, we developed an active learning, prelaboratory activity using a model of a TLC plate. The model is 3D-printed, easily replicable, and can be used to address questions and misconceptions students may have about polarity and IMFs in the context of TLC.

Prelaboratory activities or assignments that introduce students to relevant concepts and principles before they enter the time-sensitive laboratory environment have been reported to enhance conceptual understanding and decrease anxiety.^{11,12} Examples of prelaboratory activities include computer simulations, videos and quizzes, demonstrations, and models. While a variety of prelaboratory activities exist, there are, to the authors' knowledge, no published examples specific to TLC, aside from videos and simulations.^{13–19}

In designing our prelaboratory TLC activity, we wanted to take into account specific misconceptions students have about TLC related to polarity and IMFs. While a variety of misconceptions about polarity and IMFs have been reported elsewhere,^{20,21} it is the authors' experiences at the Georgia Institute of Technology that students have two persistent misconceptions concerning the effect of solvent polarity and IMFs on compound retention and separation via TLC. The first is that some students believe the trends in solvent polarity mirror those of compound polarity. In other words, some students believe that because nonpolar compounds move further up the

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plate relative to polar compounds, a nonpolar solvent will move all compounds up the plate and that a polar solvent would not move any compounds up the plate. The second misconception arises from a misapplication of the “like-dissolves-like” heuristic.²² Some students believe that a polar solvent will selectively interact with polar compounds over nonpolar compounds, causing the polar compounds to move up the plate while leaving the nonpolar compounds alone and vice versa.

For our prelaboratory TLC activity, we chose to use a physical model of a TLC plate because we wanted students to be able to engage with the technique in a hand-on manner outside of the laboratory. Physical models, especially 3D-printed ones, have become increasingly common educational tools.^{23–25} Within chemistry they have been used to teach a variety of topics including, but not limited to the Bohr model of the atom, crystallographic unit cells, and 2D NMR.^{26–28} We anticipated that the visual and tactile components of the model would help students to better understand how polarity and IMFs affect the separation of compounds via TLC.

METHODS

Model Design

The 3D-printed TLC model used in this study began as a sketch on paper that went through several iterations. The dimensions for the pieces were planned out in writing before recreating the drawings digitally in Fusion, a computer-aided design (CAD) program.²⁹ The models were printed at the Invention Studio (Ultimaker 3), the Hive Makerspace (Stratasys F170), the Materials Innovation and Learning Laboratory (Prusa i3MK3S), and the Physical Chemistry Teaching Laboratory in the School of Chemistry and Biochemistry (Prusa Mini), all at the Georgia Institute of Technology. Enough models were printed, so there was one model for every two to three students in the classroom.

There are three main pieces to the model: a base plate, T-pins, and cylindrical pins. The base plate (Figure 1A) represents the

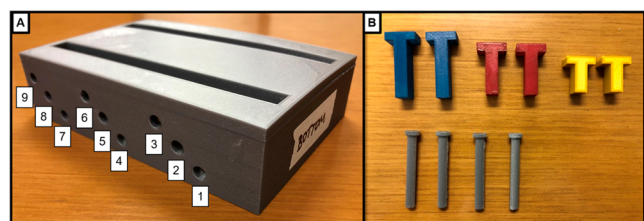


Figure 1. Components of the 3D-printed TLC model. (A) The base plate. (B) The T-pins and cylindrical pins.

TLC plate. It can be viewed as one TLC plate with two lanes or as two different plates with one lane, each depending on the activity being conducted. The base plate also has nine numbered holes along both sides, where hole number one is closest to the baseline and hole number nine is closest to the solvent front. The T-pins (pins in the shape of a T; Figure 1B) may be inserted into and slide up the lanes of the base plate and represent different chemical compounds. The cylindrical pins (Figure 1B) may be inserted into the holes on the sides of the base plate and represent solvents of different polarities that halt the movement of particular T-pins at different points along the plate. Cylindrical pins inserted into holes 1, 4, or 7 stop the movement of the longest (blue) T-pin, cylindrical pins inserted into holes 2, 5, or 8 stop the movement of the midlength (red) T-pin, and

cylindrical pins inserted into holes 3, 6, or 9 stop the movement of the shortest (yellow) T-pin). Cylindrical pins inserted into higher numbered holes represent solvents of higher polarity than those inserted into lower numbered holes.

Model Implementation

The TLC activity using the 3D-printed models was conducted in the first of a two-semester sequence of organic chemistry laboratory courses at the Georgia Institute of Technology. At the Georgia Institute of Technology, the first organic chemistry laboratory course is taken either concurrently with, or after taking, the second of two lecture courses. Chemistry, biochemistry, biology, neuroscience, and chemical and biomolecular engineering majors all take the laboratory course, as well as any prehealth students. Each week, the students enrolled in the laboratory course attend a 1 h laboratory lecture led by the instructor (W.J.H.), followed by a 4 h laboratory section led by a teaching assistant (TA). The TLC activity was implemented during the summer 2022 and fall 2022 terms during the laboratory lecture.

The activity conducted during the laboratory lecture is composed of four parts (see the Supporting Information for sample slides). Part one introduces the model to the students and explains what the components (i.e., base plate, T-pins, and cylindrical pins) represent. Parts two, three, and four cover different scenarios that evaluate student understanding of polarity and IMFs as they relate to the separation of compounds via TLC. These scenarios were covered using the predict-observe-explain technique.³⁰ Each scenario begins with a question posed to the students. Students are then given the opportunity to predict the outcome after discussing the question with their neighboring peers. The answer to each question is either given directly by the instructor or via instructions for how to set up the model to see what the actual outcome would be. Students subsequently evaluate if the outcome is consistent with their prediction. If the outcome is inconsistent with their prediction, students have the opportunity to try to explain the outcome by discussing it with their peers before engaging in a class-wide discussion. During the class-wide discussion, students get to share what they learned from the scenario and ask any clarifying questions.

In the first scenario, students are given one of each color of T-pin and told to insert the circular pins into holes 1, 5, and 9 in the left lane of the model (Figure 2A). When the T-pins are moved

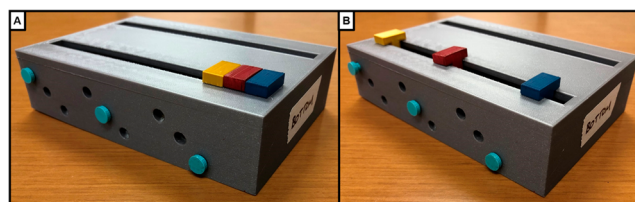


Figure 2. Images of the 3D-printed model during scenario 1. (A) The initial setup. (B) The result after moving the T-pins as far up as possible before they are halted by the cylindrical pins.

up the lane, the blue one is stopped at position 1, the red one at position 5, and the yellow one at position 9 (Figure 2B). Students are told that the three T-pins represent the compounds ethylbenzene, benzyl alcohol, and anisole, but they must determine which pin represents which compound based on how far each T-pin moved up the plate and the IMFs each compound can make with the plate.

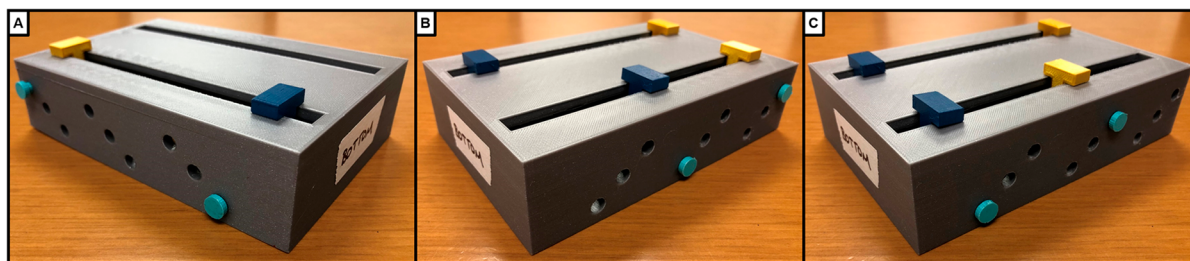


Figure 3. Images of the 3D-printed model during scenario 2. (A) The initial setup. (B) The result of using a relatively more polar solvent system relative to the one used on the left. (C) The result of using a relatively more nonpolar solvent system relative to the one on the left.

This first scenario helps students evaluate the relative polarity of each T-pin by recognizing that the T-pin that moves the least (the blue one) is the most polar and the one that moves the most (the yellow one) is the least polar. This is taken a step further by asking students to evaluate the IMFs each of the three compounds can make with the TLC plate. Because benzyl alcohol is capable of hydrogen-bonding, it is the most polar compound and therefore is represented by the blue T-pin. Because anisole is capable of dipole–dipole interactions, but not hydrogen bonding, it is less polar than benzyl alcohol, but more polar than ethylbenzene which has predominantly London dispersion forces. Therefore, anisole is represented by the red T-pin and ethylbenzene is represented by the yellow T-pin.

In the second scenario, students are told to remove the red T-pin and the circular pin in position 5 (Figure 3A). They are asked to predict how far the compounds represented by the remaining blue and yellow T-pins would move in a more polar solvent. They are then instructed to put another set of the blue and yellow T-pins, along with circular pins in positions 4 and 9, in the right lane. After moving the T-pins up the lanes, they observe that the blue T-pin is stopped at position 4 and the yellow T-pin is stopped at position 9. Students are able to conclude that the more polar solvent increases the distance traveled by the polar compound but not by the nonpolar compound, as the nonpolar compound is already at the solvent front (Figure 3B). The students are then asked to reset the right lane and to make a prediction about how far the compounds would move in a more nonpolar solvent. They are then instructed to put the circular pins in positions 1 and 6 and move the T-pins up the plate to evaluate their prediction. After moving the T-pins up the lanes, they observe that the blue T-pin is stopped at position 1 and the yellow T-pin is stopped at position 6. Students are able to conclude that the more nonpolar solvent decreases the distance traveled by the nonpolar compound but not by the polar compound, as the polar compound is already at the baseline (Figure 3C).

This second scenario was incorporated to allow the instructor to address the two main misconceptions discussed earlier in the introduction that students at the Georgia Institute of Technology are frequently observed to have. Some students that misapply the “like-dissolves-like” heuristic will predict for the first part that only the polar compound will move up the plate while the nonpolar compound does not move at all, and for the second part that only the nonpolar compound will move up the plate while the polar compound does not move at all. Other students that believe trends in solvent polarity mimic those of compound polarity will predict for the first part that both compounds will not move up as far and will predict for the second part that both compounds will move up further.

In the third scenario, the students start with a blue and a red T-pin in each lane and cylindrical pins in positions 1 and 2 of the left lane. They are asked to move the T-pins in the left lane up the plate. The blue T-pin stops at position 1 and the red T-pin stops at position 2. Students are then asked to predict the polarity of the solvent based on how far the T-pins moved and to determine whether the polarity of the solvent should be increased or decreased to improve the separation of the compounds (Figure 4A). Students are then instructed to insert

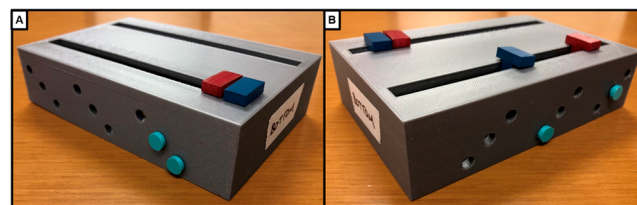


Figure 4. Images of the 3D-printed model during scenario 3. (A) The initial setup. (B) The result of using a relatively more polar solvent relative to the one on the left.

circular pins in positions 4 and 8 of the right lane to represent a more polar solvent and move the T-pins up the plate (Figure 4B). This third scenario is included as a confirmation that students are able to apply what they learned from the second scenario about the effect of solvent polarity in which more polar solvents move compounds further up the plate and more nonpolar solvents do not move them up the plate as far.

Model Evaluation

A presurvey and postsurvey were administered to evaluate the short-term effect of the activity on student comprehension of how polarity and IMFs affect the separation of compounds via TLC (Table 1). The presurvey was available to students starting 1 week before the activity was conducted in class. The postsurvey was available immediately following the activity and was available to students for up to 1 week. The surveys were identical aside from one final question in the postsurvey that asked students to provide any comments they would like to share about the activity. The five multiple-choice questions in each survey are related to the scenarios covered in the activity. Following each multiple-choice question, students are asked to explain their answer and to indicate how confident they are in their answer using a Likert scale ranging from 1 (not confident at all) to 6 (very confident).

To evaluate long-term retention of the concepts covered during the activity, we looked at student performance on a question from a final exam. In the laboratory course, there is a TLC technique final that is administered to students approximately midway through the semester. The final is

Table 1. Summary of Presurvey and Postsurvey Questions

| question no. | question type | question |
|-----------------|-----------------|--|
| Q1a | multiple-choice | If we have a polar solvent with one polar compound and one nonpolar compound, which compound will move higher up on the plate? |
| Q1b | short answer | Please explain how you arrived at your answer. |
| Q1c | Likert scale | How confident are you in your answer (where 1 is not confident at all and 6 is very confident)? |
| Q2a | multiple-choice | If we have a nonpolar solvent and relatively polar compounds, where will the compounds be on the plate? |
| Q2b | short answer | Please explain how you arrived at your answer. |
| Q2c | Likert scale | How confident are you in your answer (where 1 is not confident at all and 6 is very confident)? |
| Q3a | multiple-choice | If we have a polar solvent and relatively nonpolar compounds, where will the compounds be on the plate? |
| Q3b | short answer | Please explain how you arrived at your answer. |
| Q3c | Likert scale | How confident are you in your answer (where 1 is not confident at all and 6 is very confident)? |
| Q4a | multiple-choice | You are trying to separate a mixture of benzyl alcohol and benzylamine by TLC. You initially use a solvent of 6:4 hexanes:ethyl acetate, but after developing the plate, you observe two overlapping spots near the base of the plate. What solvent system should be used to improve the separation? |
| Q4b | short answer | Please explain how you arrived at your answer. |
| Q4c | Likert scale | How confident are you in your answer (where 1 is not confident at all and 6 is very confident)? |
| Q5a | multiple-choice | You are trying to separate a mixture of ethylbenzene and benzyl bromide by TLC. You initially use a solvent of 6:4 hexanes:ethyl acetate, but after developing the plate, you observe two overlapping spots near the top of the plate. What solvent system should be used to improve the separation? |
| Q5b | short answer | Please explain how you arrived at your answer. |
| Q5c | Likert scale | How confident are you in your answer (where 1 is not confident at all and 6 is very confident)? |
| Q6 ^a | short answer | What comments (if any) do you have about this activity that you would like to share? |

^aQuestion 6 was in the postsurvey but not the presurvey.

Table 2. Percent of Correct Responses to Survey Questions (N = 210)

| survey question | summer (N = 49) | | fall (N = 161) | |
|--|-----------------|----------------|----------------|----------------|
| | presurvey (%) | postsurvey (%) | presurvey (%) | postsurvey (%) |
| Q1. If we have a polar solvent with one polar compound and one nonpolar compound, which compound will move higher up on the plate? | 59.2 | 81.6 | 60.9 | 86.3 |
| Q2. If we have a nonpolar solvent and relatively polar compounds, where will the compounds be on the plate? | 77.6 | 98.0 | 81.4 | 89.4 |
| Q3. If we have a polar solvent and relatively nonpolar compounds, where will the compounds be on the plate? | 67.3 | 91.8 | 46.7 | 85.7 |
| Q4. You are trying to separate a mixture of benzyl alcohol and benzylamine by TLC. You initially use a solvent of 6:4 hexanes:ethyl acetate, but after developing the plate, you observe two overlapping spots near the base of the plate. What solvent system should be used to improve the separation? | 65.3 | 95.9 | 75.8 | 88.8 |
| Q5. You are trying to separate a mixture of ethylbenzene and benzyl bromide by TLC. You initially use a solvent of 6:4 hexanes:ethyl acetate, but after developing the plate, you observe two overlapping spots near the top of the plate. What solvent system should be used to improve the separation? | 57.1 | 95.9 | 63.4 | 82.6 |

composed of two portions: a five-question quiz (to assess their conceptual understanding) and a procedure involving running a TLC plate (to assess their procedural comprehension and psychomotor skills). In the quiz, there is one multiple-choice question that specifically addresses polarity and IMFs that we used as a measure of long-term retention of the concepts covered in the activity. We evaluated student performance on this question relative to a previous term, in which the activity was not incorporated, as a control.

RESULTS AND DISCUSSION

Following the TLC activity, the percentage of students that correctly answered each of the five survey questions increased, suggesting the activity improved student understanding of polarity and IMFs as they relate to the separation of compounds via TLC (Table 2). The percentage of correct responses on the presurvey ranged from 46.7–81.4%, while the percentage of correct responses on the postsurvey ranged from 81.6 to 98.0%. An important limitation is that there were a small number of students that identified correct answers on the postsurvey but who had the wrong reasoning. In these instances, the misconception was almost exclusively due to the misapplication of the “like-dissolves-like” heuristic. For example, if a student assumed that polar compounds only interact with polar solvents

and vice versa, they could correctly answer question two on the survey, but would not correctly answer question three. This is because question two asks how far polar compounds will move up a TLC plate with a nonpolar solvent, which is not very far. If a student assumed the compounds would not move far because a nonpolar solvent would only interact with nonpolar compounds and not polar compounds, they would still get the question correct. However, question three asks how far nonpolar compounds will move up a TLC plate with a polar solvent, which is much further up the plate. If a student makes a similar assumption that a polar solvent will only interact with polar compounds and not nonpolar compounds, they would get the question wrong. It is worth noting that the prevalence of the misapplication of the “like-dissolves-like heuristic” was reduced using the activity, but not eliminated.

In addition to answering more questions correctly on the postsurvey, students’ average self-reported confidence in their responses went up (Table 3). The self-reported confidence levels on the presurvey ranged from 3.3 to 4.5, while on the postsurvey they ranged from 4.5 to 5.3. Although self-reported confidence overall increased, there were students whose confidence levels increased even when they incorrectly answered the question(s) in the postsurvey. This may be due to the Dunning–Kruger effect, the cognitive bias in which an

Table 3. Student Self-Reported Confidence in Their Responses to Survey Questions ($N = 210$)

| survey question | summer ($N = 49$) | | | | fall ($N = 161$) | | | |
|--|---------------------|--------------|------------|--------------|--------------------|--------------|------------|--------------|
| | presurvey | | postsurvey | | presurvey | | postsurvey | |
| | mean (SD) | median (IQR) | mean (SD) | median (IQR) | mean (SD) | median (IQR) | mean (SD) | median (IQR) |
| Q1. If we have a polar solvent with one polar compound and one nonpolar compound, which compound will move higher up on the plate? | 4.3 (1.3) | 5 (2) | 4.8 (1.1) | 5 (2) | 4.5 (1.2) | 5 (1) | 5.3 (1.0) | 6 (1) |
| Q2. If we have a nonpolar solvent and relatively polar compounds, where will the compounds be on the plate? | 4.1 (1.3) | 4 (2) | 4.9 (1.1) | 5 (2) | 4.4 (1.2) | 4 (1) | 5.3 (0.9) | 6 (1) |
| Q3. If we have a polar solvent and relatively nonpolar compounds, where will the compounds be on the plate? | 3.8 (1.3) | 4 (2) | 4.5 (1.3) | 5 (3) | 3.9 (1.2) | 4 (2) | 4.8 (1.1) | 5 (2) |
| Q4. You are trying to separate a mixture of benzyl alcohol and benzylamine by TLC. You initially use a solvent of 6:4 hexanes:ethyl acetate, but after developing the plate, you observe two overlapping spots near the base of the plate. What solvent system should be used to improve the separation? | 3.3 (1.2) | 3 (2) | 4.9 (1.1) | 5 (2) | 3.8 (1.3) | 4 (2) | 5.0 (1.1) | 5 (2) |
| Q5. You are trying to separate a mixture of ethylbenzene and benzyl bromide by TLC. You initially use a solvent of 6:4 hexanes:ethyl acetate, but after developing the plate, you observe two overlapping spots near the top of the plate. What solvent system should be used to improve the separation? | 3.3 (1.3) | 5 (2) | 4.8 (1.2) | 5 (2) | 3.6 (1.3) | 4 (1) | 4.8 (1.2) | 5 (2) |

individual with limited knowledge of a topic wrongly overestimates their comprehension of that topic.³¹

While student understanding of polarity and IMFs as they relate to the separation of compounds via TLC improved in the short term as a result of the TLC activity, that understanding was also retained long-term. Long-term retention was measured using a multiple-choice question from a TLC technique final that specifically covered how polarity and IMFs affect compound separation via TLC. In the semester before the activity was introduced, 70.5% of students correctly answered the question. In the following summer and fall semesters in which the activity was incorporated, 87.8% and 80.8% of students correctly answered the question, respectively.

In addition to the promising performance outcomes, nearly all of the students who participated in the activity had positive perceptions of it based on their responses to the last question on the postsurvey. Students said that the activity and discussion around the models “pointed out and cleared the misconceptions” and were “very unique and gave students a hands-on opportunity that allows for better understanding of solvent choice in a TLC system.” They also commented on the effectiveness of using a hands-on model, saying that “the 3D visualization while explaining TLC allowed...to understand at a deeper level how changing solvent and compounds polarity affect movement” and “the 3D printed models helped to clear up confusion with TLC plates.” It is encouraging that the student comments identified specific benefits, such as clearing up misconceptions and providing an interactive experience to learn more about the TLC technique, that the activity and model were designed to address.

Although the activity did positively impact students in both the summer and fall terms relative to the spring term, student performance on the postsurvey and TLC technique final was stronger for the summer term students. Several factors may have contributed to this. In the summer term, there were fewer students which allowed the class to be held in an active learning classroom where the students were able to easily talk to, and collaborate with, their peers. The students in the fall term did the activity in a larger lecture style classroom where they were only able to discuss with their immediate neighbors, limiting the extent of collaboration. The time of day the activity took place may have also had an impact. The summer term course was scheduled in the early afternoon (12:30 pm), while the fall course was scheduled for the evening (5:00 pm). It is possible

the fall term students were not able to process the information from the activity as well given the greater cognitive load of a full day of classes. There was also a difference in the time between when the activity was conducted and when students took the TLC technique final. Because the summer term is shorter than the fall term (10 weeks versus 15 weeks), the TLC technique final was given only 2 weeks after the activity as opposed to 3 weeks in the fall term. Retention of information may have been greater over a shorter time frame, accounting for a larger percentage of students correctly answering the question on the TLC technique final over the summer term relative to the fall term.

CONCLUSION

A preliminary activity employing a 3D-printed model of a TLC plate was developed and implemented in an organic chemistry laboratory course with the goal of addressing student misconceptions surrounding polarity and IMFs as they pertain to the separation of compounds via TLC. Pre- and postsurveys given to the students before and after the activity, along with a TLC technique final, indicated that there was an increase in understanding of how polarity and IMFs affect the separation of compounds via TLC in the short term and in the long term. Student perceptions of the activity were predominantly positive, and outcomes from two different terms suggest the activity is effective in both small and large classroom settings. Our findings suggest this activity could be beneficial for instructors at other institutions to adopt in their own courses to address misconceptions students may have about TLC prior to working with the technique in the laboratory setting.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.2c01142>. The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.2c01142>.

Supporting Information, (PDF, DOCX)

Sample TLC Activity Slides (PDF)

Base Plate CAD File (STL), Cylindrical Pin CAD File (STL), Longest (Blue) T-Pin CAD File (STL), Mid-Length (Red) T-Pin CAD File (STL), Shortest (Yellow) T-Pin CAD File (STL) (ZIP)

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Notes

The authors declare no competing financial interest.

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