



Fluorescence of Silver Nanoparticles in Polymer Solutions for Direct Write Technologies

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Abstract

Silver nanoparticles can be mixed into a polymer matrix solution to exhibit luminescent properties. This luminescence can be integrated into existing photovoltaic systems and can eventually serve as a substitute for silicon in today's solar cells. In this work, students performed experiments with various solvents to observe their evaporation rates and the solubility limits of poly(methyl methacrylate) (PMMA) powder. The goal of this research is to dissolve PMMA into, and disperse previously synthesized silver nanoparticles into, the polymer solution. This solution, pregnant with silver nanoparticles and polymer, will be printed onto a substrate via Direct Write machines for a uniform layer. The expectation is that with the correct solvent, the solvent will evaporate from the substrate. This will leave solely the plastic and silver nanoparticles on the substrate, giving the user a thin polymer film with homogeneous properties.

1. Introduction

Silver nanoparticles have been a topic of growing interest in the biomedical and renewable-energy industries because of their unique bacterial-growth inhibition and properties of luminescence. When silver nitrate is combined with an appropriate capping agent, nano-scale particles are synthesized. These particles can be formulated into inks and used with Direct Write machines, such as the nScript, M³D, and the Sono-Tek, to give optimal luminescent properties for printed electronics.

When silver nanoparticles are combined with a polymeric solution, they can be deposited from a Direct Write machine onto a substrate. Upon drying, a user will be left with a thin film polymer substrate, impregnated with nanoparticles. The coupling of the polymer film, as well as the luminescence of silver nanoparticles, allow for easy incorporation into devices such as solar cells.

The work reported here documents the various evaporation and solubility experiments performed to choose an appropriate polymeric solution. The results of these experiments can be taken into consideration to later produce a high quality ink for printed electronics.

2. Broader Impact

The study of these thin polymer films and silver nanoparticles will prove to be beneficial to the enhancement of today's solar cells. When printed electronics have the correct properties, they can be used in substitution of amorphous silicon wafers, as well as cadmium telluride thin films. This can help reduce the production cost per solar cell, as well as improving the efficiency of the unit. In addition to cost savings, a cell that employs a thin film with natural luminescence can help make the disposal of an end-of-life panel easier. Cadmium telluride thin films are relatively toxic substances, and if not properly disposed of, a discarded solar panel can pose an environmental threat to its environment – quite the opposite of its desired effect. A less toxic thin film can be utilized in unit production and can greatly assist with metal recycling efforts.

3. Procedures

3.1 Materials

The following materials were used in the experiments described in this work:

- 10mL of following solvents, 1.0M: acetone, chloroform, cyclohexanone, methyl ethyl ketone (MEK), tetrahydrofuran (THF), xylene, deionized water.
- Graduated cylinders, 25mL
- Aluminum pans, 2.654 inches in diameter
- 100 grams of poly(methyl methacrylate) powder
- Erlenmeyer flasks, 50mL
- Kapton substrate

3.2 Equipment

The following equipment was used in the experiments described in this work:

- Ventilated lab hood
- Mettler top loading balance Model PE11
- Cardboard scale cover
- Magnetic stir bar
- VWR 600 series standard hotplate/stirrer
- Stainless steel laboratory spatula
- Stopwatch
- Aluminum foil
- 100 μ L micropipette
- Appropriate safety supplies

3.3 Evaporation Testing

To determine a solvent appropriate for use in printing, students performed evaporation tests of five various solvents and deionized water. Volumes of 10mL of each solvent were measured and poured into separate 25mL graduated cylinders. These cylinders were not covered and were allowed to sit in a ventilated lab hood, with the air temperature at 25°C. The volumes of these solvents were checked every few hours for possible evaporation. The observed volumes and times were recorded for each solvent.

To re-investigate evaporation rates, six solvents, a scale and a cardboard cover were employed. A Mettler top loading balance Model PE11 scale was placed in the lab hood, and a cardboard cover was made from a box lid. This cover measured 15”x 11.75”, which easily covered the scale deck. The cover had a 3” diameter hole cut in the center to assist with the testing, and then was placed onto the scale. This cover was used so the lab hood exhaust fan would not inhibit the calculations of the scale, as students were dealing with such light weight measurements. To experiment, 10mL of each solvent was added into their own 25mL graduated cylinder. A labeled aluminum pan was placed through the cover hole onto the scale, the scale was tared, and the solvent was poured into its own pan. The initial weight of the solvent was noted. These tests were conducted in a lab hood with the air vent on and the door closed, with the air temperature at 25°C. The weights of all solvents, save the xylene and cyclohexanone, were checked in intervals of three minutes. The weight of the xylene solvent was checked every three minutes, until 21 minutes into the test, and then was checked every 20 minutes. The weight of the cyclohexanone solvent was checked every 20 minutes. Observation for each solvent was concluded when each it reached a weight of 1.0g and below.

3.4 Solubility Testing

Students were concerned with the solubility limits of poly(methyl methacrylate) (PMMA) in solvents once it was to be used in printing applications, so they conducted a literature search. They found an article that described solubility limits in various solvents, and students wished to verify the *Russian* [1] results with their own.

Students experimented with varying solvents to find a practical solubility limit of spherical PMMA (Figure 1). To begin, 10mL of each solvent was measured into its own 25mL graduated cylinder. The cylinder was placed onto a VWR 600 Series standard hotplate/stirrer, located in a ventilated lab hood, with the air temperature at 25°C. Subsequently, 1.0 g of PMMA powder was weighed out and placed onto a watch glass. A magnetic stir bar was placed into the cylinder, and the hotplate/stirrer was turned to stir at 800rpm. Next, a stainless steel laboratory spatula placed the equivalent of ~0.0225g – 0.0350g of PMMA into the graduated cylinder. Once the powder was placed into the cylinder, a stopwatch was started from time zero. The mixture was allowed to stir until all the PMMA present had dissolved into the specific solvent being tested, and additional spatulas of powder were added into the cylinder successively. The solution was allowed to stir until it had visibly reached its solubility limit. When the solution appeared to have reached its solubility limit, the time on the stopwatch was noted and the hotplate/stirrer was turned off. Following the PMMA hitting the solubility limit, the watch glass was weighed and a difference was noted between its loaded start weight and end weight. Testing parameters were kept constant for each solvent testing, and students were watching for a solvent's maximum solubility, seen when particles could no longer dissolve into solution in a reasonable time.

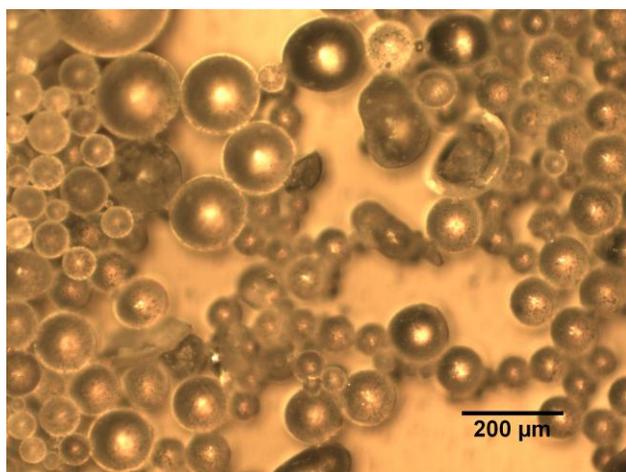


Figure 1: Powder PMMA at 10x reveals spherical particles, below 300μm in diameter. [2]

To re-investigate the solubility of PMMA in the same solvents, solubility tests were conducted again, the only difference being the container holding the solution. In the second round of solubility testing, 10mL of each solvent was measured into its own 50mL Erlenmeyer flask. The flask was placed onto a VWR 600 Series standard hotplate/stirrer, and testing was carried out as in the investigation employing graduated cylinders.

3.5 MEK (neat) and MEK/PMMA Evaporation Testing

Methyl Ethyl Ketone was chosen to be used in conjunction with PMMA to eventually be mixed with silver nanoparticles to form an ink. Following this decision, aluminum foil was placed over the stage area of the VWR 600 series standard hotplate. The stirring mechanism was turned off, and for testing, the hotplate was turned onto temperatures of 25°C, 40°C, 50°C. Starting at 25°C, 100µL of MEK (neat) solvent was drawn from its storage bottle with a micropipette and was dropped onto a Kapton substrate. The number of drops placed onto the Kapton substrate was recorded. The evaporation of the solvent was observed, and the time to completion was recorded. The same procedure was used for another test, using MEK with dissolved PMMA, made during previous solubility testing. Also starting at 25°C, 100µL of the MEK/PMMA solution was dropped onto previously heated Kapton. The number of drops used, as well as the time to complete evaporation was recorded. These procedures were followed for five trials for the solvent and solution, at all three temperatures.

3.6 Particle Synthesis

Students involved with this work will eventually follow the silver nanoparticles synthesis outlined in the thesis work of Mr. Dane Hansen, M.E.S. M.S., 2010 [3].

4. Results

4.1 Evaporation Testing

During the first evaporation testing, solvents were checked every hour. All solvents experienced little, if any evaporation. Testing was terminated on the third day of testing, as no solvent had completely evaporated. The delayed evaporation of solvents in the graduated cylinders was attributed to the lack of a consistent air pressure, as well as a low solvent-air contact angle. Results for each solvent in this trial can be seen below (Table 1).

Table 1: Table showing progress of solvents during evaporation testing, trial 1. [4]

	DI water (mL)	acetone (mL)	xylene (mL)	cyclohexanone (mL)	chloroform (mL)	MEK (mL)
Day 1	10 @ 12:39 PM	10 @ 12:42 PM	10 @ 12:44 PM	10 @ 12:46 PM	10 @ 12:48 PM	10 @ 12:50 PM
Day 1	10 @ 2:28 PM	9 @ 2:28 PM	10 @ 2:28 PM	9.5 @ 2:28 PM	10 @ 2:28 PM	9.5 @ 2:28 PM
Day 1	10 @ 3:30 PM	9 @ 3:29 PM	10 @ 3:30 PM	9.5 @ 3:30 PM	9.5 @ 3:30 PM	9 @ 3:30 PM
Day 1	10 @ 9:25 PM	8.5 @ 9:25 PM	10 @ 9:29 PM	9.5 @ 9:28 PM	8 @ 9:28 PM	9 @ 9:25 PM
Day 2	10 @ 8:45 PM	8 @ 8:45 AM	10 @ 8:45 AM	9.5 @ 8:45 AM	7.5 @ 8:48 AM	9 @ 8:45 AM
Day 2	10 @ 4:30 PM	7.5 @ 4:30 PM	10 @ 4:30 PM	9.5 @ 4:30 PM	7.5 @ 4:30 PM	9 @ 4:30 PM
Day 3	10 @ 2:30 PM	5.5 @ 2:30 PM	10 @ 2:30 PM	9 @ 2:30 PM	6.5 @ 2:30 PM	8.5 @ 2:30 PM

During the second trial, additional solvents were added to those previously tested. Each solvent tested during this trial experienced near-total evaporation. This can be attributed to the greater surface area of the testing medium, as well as a greater contact angle between the solvent and the lab hood environment. Results for each solvent in this trial can be seen below (Tables 2-4, Figure 2).

Table 2: Evaporation data for acetone, chloroform, THF and MEK, trial 2. [5]

Time (mins)	acetone (g)	chloroform (g)	THF (g)	MEK (g)
0 (start)	6.7	13.3	7.9	7.9
3	5.6	11.0	6.8	7.3
6	4.4	8.9	5.8	6.2
9	3.4	7.0	4.8	5.6
12	2.4	5.5	3.9	4.7
15	1.3	3.8	3.0	3.9
18	0.4	2.2	2.2	3.2
21	-	1.3	1.4	2.5
24	-	0.9	0.9	1.9
27	-	0.6	0.5	1.3
30	-	-	-	0.9
33	-	-	-	0.8
36	-	-	-	0.8
39	-	-	-	0.7
42	-	-	-	0.6
45	-	-	-	0.6

Table 3: Evaporation data for xylene, trial 2. [5]

Time (mins)	xylene (g)
0 (start)	8.7
3	8.5
6	8.3
9	8.1
12	8.1
15	8.1
18	7.8
21 *	7.7
30 **	7.2
50	6.2
60	5.6
70	5.7
80	4.4
90	4
100	3.5
120	2.8
140	1.9
170	0.8

Table 4: Evaporation data for cyclohexanone, trial 2. [5]

Time (mins)	cyclohexanone (g)
0 (start)	8.8
30	8.1
50	7.7
70	7.2
90	6.7
110	6.2
130	5.7
150	5.2
170	4.8
190	4.3
210	3.8
230	3.3
250	2.8
270	2.4
290	1.9
310	1.5
330	1.0

When the results of trial 2 are reviewed and compared, it can be seen that acetone experienced the quickest evaporation time, evaporating completely in ~18 minutes. Acetone was followed as the quickest to evaporate by chloroform and THF, taking ~27 minutes each to evaporate - despite different starting weights. The data shows that xylene experienced a dramatically slow evaporation rate, taking ~170 minutes (Table 3). The solvent to experience the slowest evaporation rate was cyclohexanone, taking ~330 minutes (Table 4). A plot was constructed to graphically show the evaporation rates of all solvents (Figure 2).

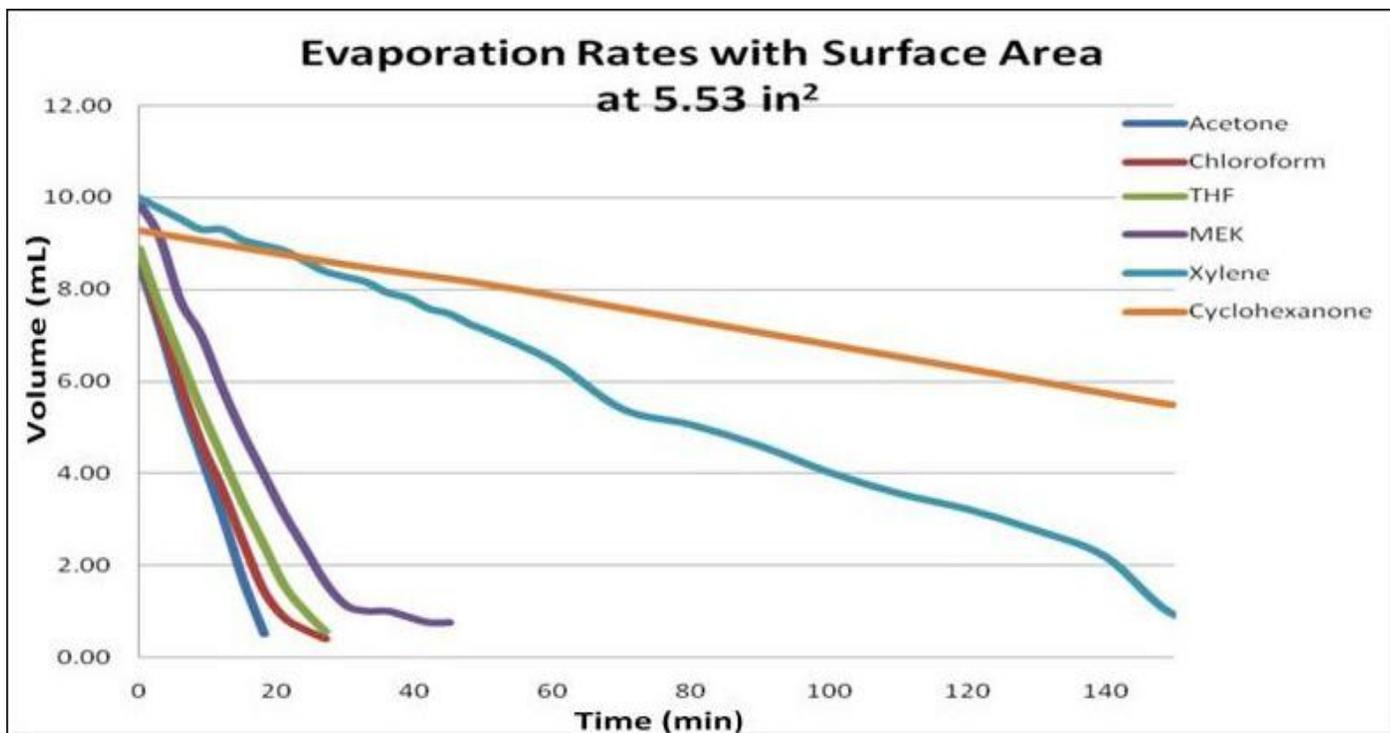


Figure 2: Plot showing evaporation rates for all solvents involved in trial 2. [6]

4.2 Solubility Testing

Students tested six solvents for the solubility limits of PMMA. All solvents were observed and PMMA was added according to how fast it was being dissolved in each. When each solvent appeared to have reached its solubility limit, it would appear cloudy for a gracious amount of time, the testing was concluded. When the testing was concluded for each solvent, the watch glass containing PMMA would be weighed. These weights were recorded and a difference was taken between the starting weight and the end weight. The solvents tested and their experimental solubility limits were compiled (Table 5). The figure also contains names of all solvents mentioned in reference [1], and those with a data entry of 'n/a' were not tested.

Table 5: Chart showing considered solvents and their experimental solubility limits. [7]

Solvent	Solubility (g/10 ml)
Acetone	0.2363
Chloroform	1.3471
Cyclohexanone	0.0477
Dimethylformamide	n/a
Ethyl Acetate	n/a
Methyl Ethyl Ketone	0.2573
Tetrahyrdofuran	0.1452
Toluene	n/a
Trichloroethylene	n/a
Xylene	0.0834

After testing was complete, students tabulated information about the various solvents from their respective Material Safety and Data Sheet (MSDS) (Table 6). Knowing the various properties of tested solvents and their relative toxicity were factors considered. The more toxic the solvent, the less likely candidate for father investigation (Figure 3).

Table 6: MSDS data for solvents considered and used for solubility testing. [8-17]

Solvent	Evap Rate (BuAc=1)	Viscosity (cP)	Surf Tension (mN/m)	Boiling Pt (°C)	Vapor Press (mmHg at 20° C)
Acetone	7.7	0.31	25.2	56.5	182
Chloroform	11.6	0.58	27.5	61.5	159
Cyclohexanone	0.3	2.02	34.57	155	5
Dimethylformamide	0.17	0.92	36.76	153	2.7
Ethyl Acetate	6	0.43	23.75	77.1	76
Methyl Ethyl Ketone	2.7	0.43	24.6	80	78
Tetrahydrofuran	8	0.46	26.4	66	129
Toluene	2.24	0.59	28.53	110.6	22
Trichloroethylene	6.4	0.53	32	87.2	68
Xylene	0.6	0.76	30.1	138	9

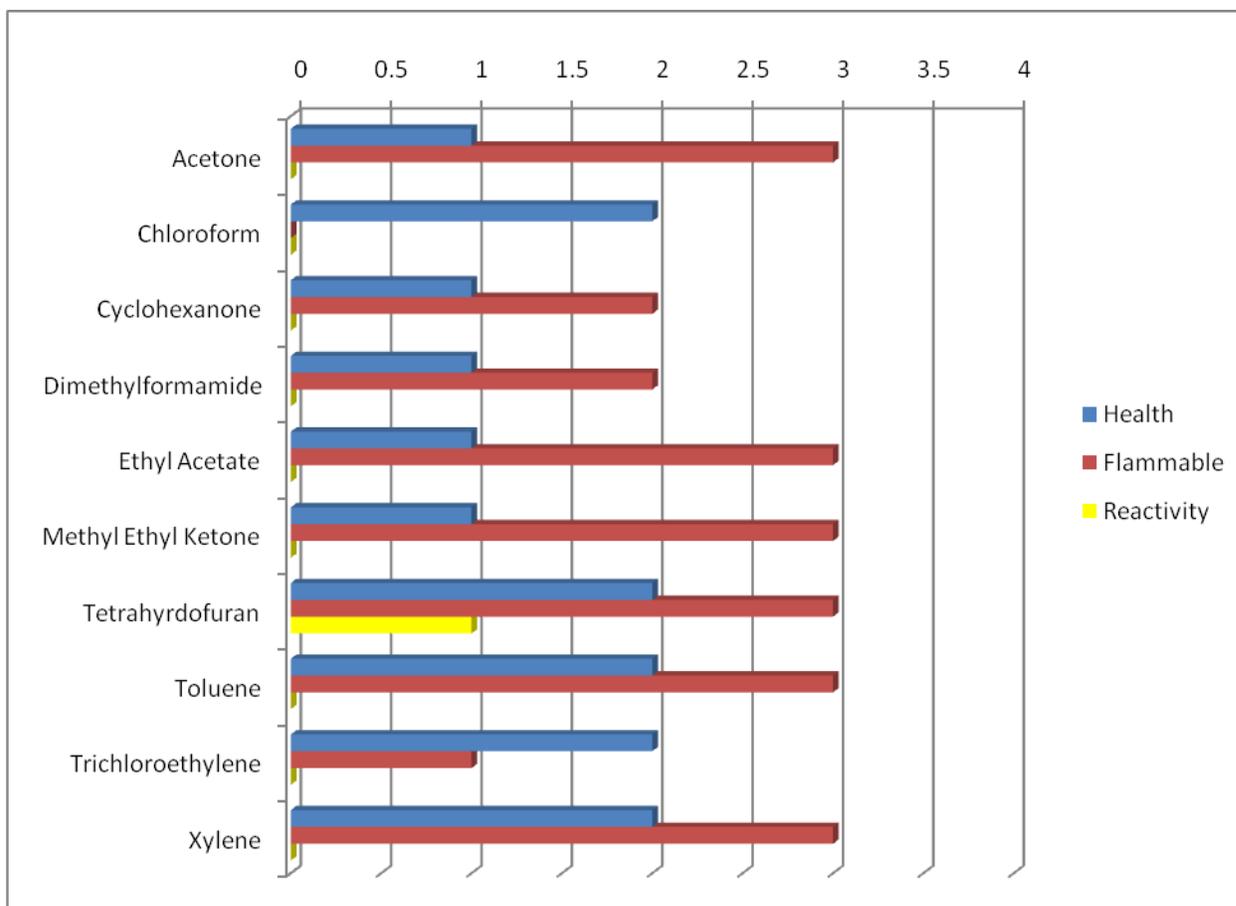


Figure 3: NFPA 704 Chemical Hazard data for considered and experimentally used solvents. [8-17]

The NFPA Chemical Hazard data for the considered and used solvents is based off of each solvent's bottle label. All chemical bottles come labeled with an NFPA 704 diamond, or a diagram showing how the chemicals rate on a scale from 0-4 about their flammability, reactivity, how they affect human health, and any special characteristics. By tallying up the scores on the NFPA diamond, the solvents involved in solubility testing can be evaluated on their dangers (Figure 4).

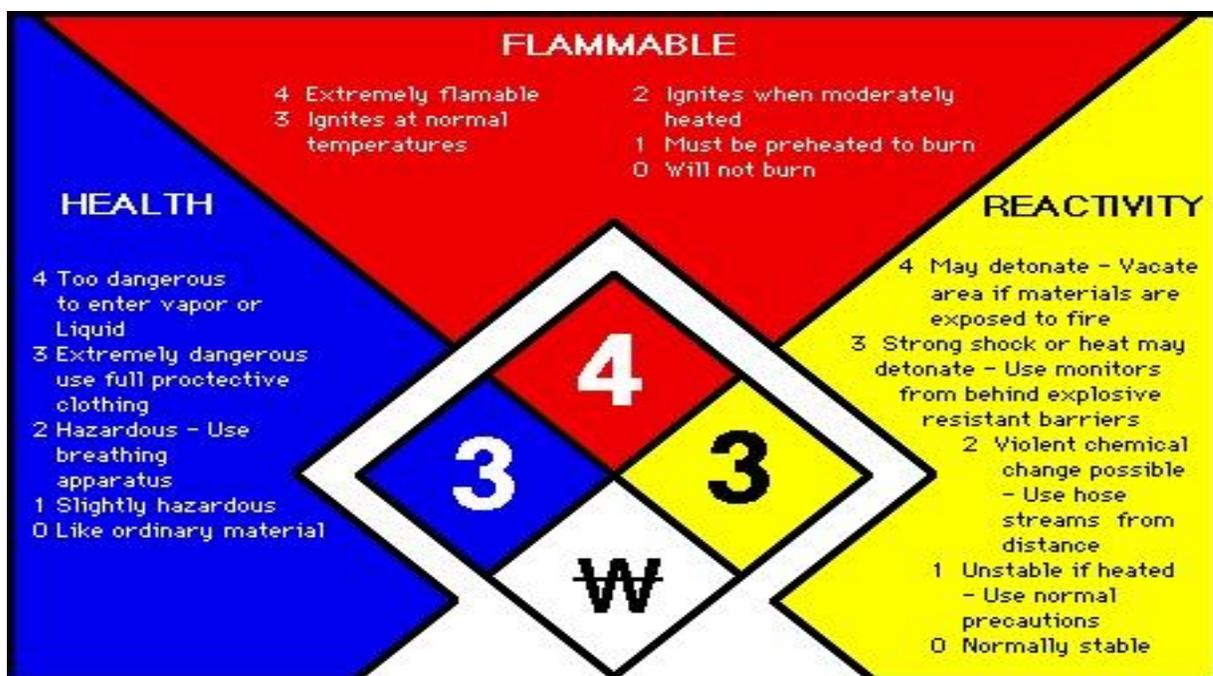


Figure 4: NFPA Diamond Key to give general hazard information about chemicals. [18]

4.3 MEK (neat) and MEK/PMMA Evaporation Testing

The MEK solvent and MEK with dissolved PMMA were individually dropped with a micropipette onto a heated Kapton substrate (Figure 5), each covering an approximate surface area of 0.785in^2 . Evaporation results of the drop-testing of MEK (neat) and MEK with dissolved PMMA at 25°C , 40°C , 50°C showed that the quickest evaporations came from both the solvent and solution tested at 50°C . The slowest evaporations rates came from both the solvent and solution tested at 25°C (Figure 6).



Figure 5: Kapton substrate showing fully evaporated drop-test. [19]

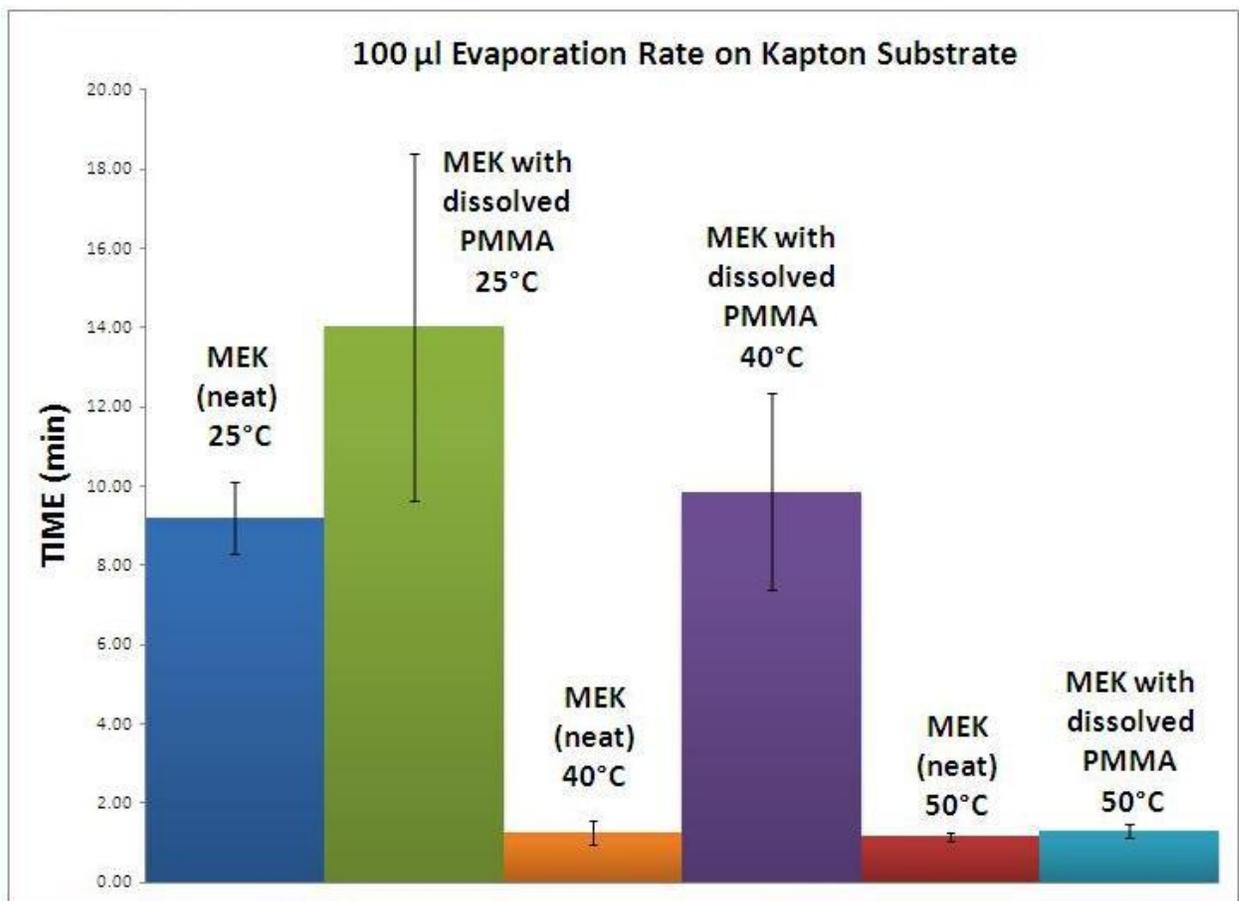


Figure 6: Evaporation rates of drop-tests of MEK (neat) and MEK with dissolved PMMA. [20]

5. Discussion

Evaporation testing trials proved beneficial for the students. Testing media were first placed in graduated cylinders, then in aluminum pans. In all of the solvents used during the evaporation testing from aluminum pans, it can be seen that cyclohexanone experienced the slowest rate of evaporation. The data suggests that cyclohexanone would be a prime candidate to use in the printing of thin films, as the solvent would not evaporate during the printing process, which could lead to costly equipment damages of printing apparatuses.

Solubility testing showed that PMMA in 10mL of chloroform reached a solubility limit of 1.347g/10mL. This was by the far the greatest solubility limit observed in the list of solvents used. All other solvents tested showed a lesser solubility limit than that of PMMA in chloroform – as PMMA in 10mL of MEK showed a solubility of 0.257g, and PMMA in 10mL of acetone showed a solubility of 0.236g.

While chloroform has the greatest PMMA solubility limit, and one of the greatest evaporation rates at 11.6 (BuAc=1), it is one of the least viscous solvents examined in this study, with a viscosity of 0.58cP. In MSDS review, it was found that the surface tension of chloroform is 27.5mN/m. This is an important parameter, as the solvent involved with printing will come into contact with air bubbles in the ink. The surface area of the bubbles will be circulating through the ink with ultrasonic energy, and because the surface area of the ink will increase, it will increase the evaporation rate of the ink as it is leaving the printing head.

However, when referring to Table 6, it can be seen that cyclohexanone has the slowest evaporation rate of all the solvents examined, with an evaporation rate of 0.3 (BuAc=1). In turn, cyclohexanone is the most viscous of the solvents examined, with a viscosity of 2.02cP. While cyclohexanone has the slowest evaporation rate, which would be beneficial in the printing process, it has the lowest observed solubility limit of PMMA– 0.0477g/10mL. As the solubility parameters of cyclohexanone aren't very efficient, one would need large amounts of solvent to print, which would increase the time needed to print an ink.

All safety parameters were evaluated in these tests. When students were handling chemicals, they used rubber gloves, safety glasses, long sleeves, long pants and closed-toed shoes to protect the appropriate extremities. All chemicals were handled in a well-ventilated area, and all experiments were performed in a well-lit, ventilated lab hood.

6. Conclusions

6.1 Summary

To best create a thin film polymer that would be suitable for use with silver nanoparticles, the polymer chosen would have to be able to withstand fluorescence-based detection methods at longer-wavelengths ($\lambda > 520\text{nm}$). It was found that Poly(methyl methacrylate) (PMMA) would be a suitable polymer, based on its cost and that it is readily available.

A colleague's documentation of their Direct Write printings of polymer in a solvent showed that the use of chloroform with PMMA had caused problems in the machine, and had clogged the printing heads. This was a severe clog, resulting in lost operating time, as well as the required replacement of the expensive heads. Knowing full well of the chloroform-printing head reaction, evaporation and solubility testing was carried out on multiple solvents.

Based on solubility and evaporation parameters, as well as the hazards of each solvent, it was determined that methyl ethyl ketone (MEK), should be used in conjunction with PMMA for thin film polymer formulation. As a polymer and an appropriate solvent have been selected, further testing of the solution is needed before silver nanoparticles can be added to create an ink.

6.2 Future Work

There is much work to be done to produce an ink that can be used in printed electronic systems. Following the work done during the REU Summer 2010 tenure, there are still aspects of the potential thin film polymer that need to be investigated. Following solubility testing, the viscosities of the potential solvents will need to be measured with a rheometer to predict behavior during printing processes. The surface tensions of the solvents will then need to be tested, as these tensions are important for the printing processes. All of these experiments are crucial to picking an appropriate solvent for thin film production.

Following testing on solvents, an ink will be formulated according to the Hansen Thesis [3]. The synthesis presented in the Hansen Thesis may undergo adjustments, as the capping agent may be changed during formulation. Once a suitable ink has been made, it will have to be run through Direct Write machines to be printed onto an appropriate substrate. Following printing, the ink and substrate will have to be tested for enhanced luminescence.

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