

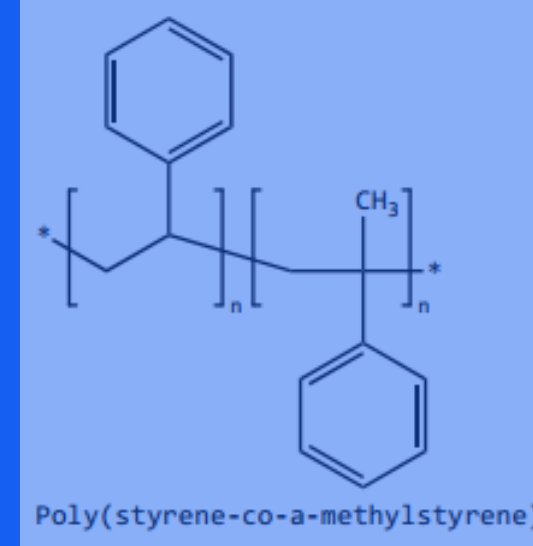
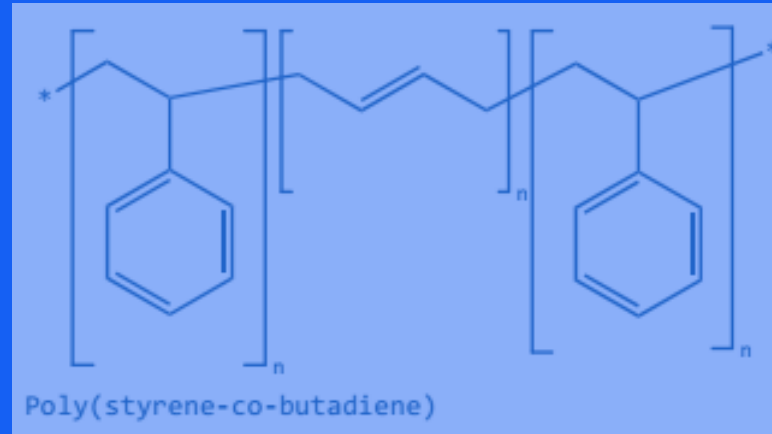
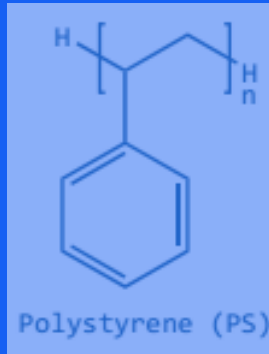
# Paper Review

A Osamu Katagiri Tanaka  
A01212611@itesm.mx

09 Dec 2019

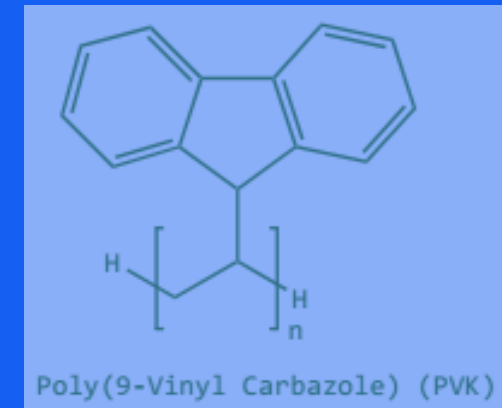
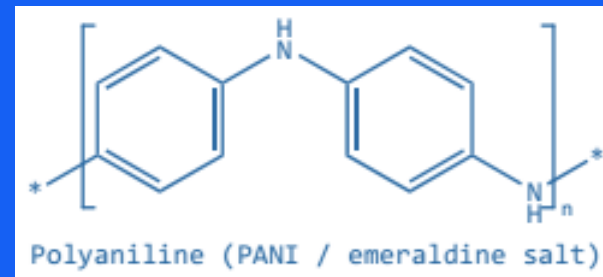


- FFES
- NFES



Good **electrical conductivity** and **environmental stability** for biomedical and technological applications

## Polyaniline (PANI)



However, the poor solubility of PANI in common solvents and low molecular weight **interferes with electrospinning into uniform fibers**



# Previous Work (2009) ...

e-Polymers 2009, no. 114



<http://www.e-polymers.org>

ISSN 1618-7229

## Electrospinning of polyaniline-polyacrylonitrile blend nanofibers

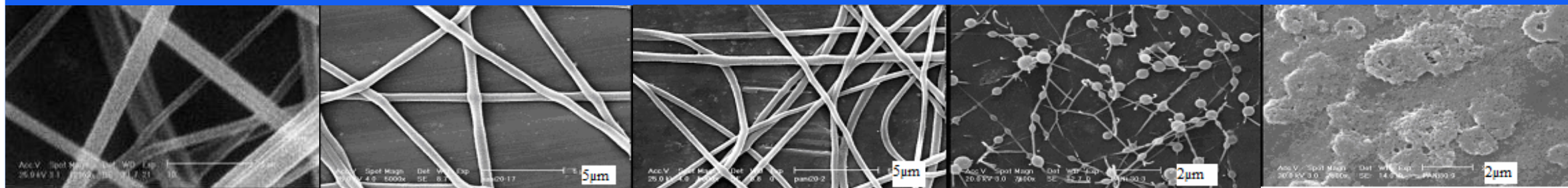
Fatemeh Raeesi, Mahdi Nouri,\* Akbar Khodaparast Haghi

\*Department of Textile, University of Guilan, Rasht, Iran, P.O. Box 41625-3756; fax: +98-131-6690271; email: mnouri69@guilan.ac.ir

(Received: 08 November, 2008; published: 21 October, 2009)

Typically, the use of an insulating copolymer with high molecular weight is expected to act as a remedy

However, the presence of a nonconducting polymer modifies the properties of PANI



100% PAN

Fiber diam: 652nm

10% PANI w/PAN

Fiber diam: 602nm

20% PANI w/PAN

Fiber diam: 425nm

30% PANI w/PAN

Fiber diam: 164nm

100% PANI

Fiber diam: <NA>

Raeesi, F., Nouri, M., & Haghi, A. K. (2009). Electrospinning of polyaniline-polyacrylonitrile blend nanofibers. E-Polymers, (October). <https://doi.org/10.1515/epoly.2009.9.1.1350>

Sironi, A., Marinotto, D., Riccardi, C., Zanini, S., Guerrini, E., Della Pina, C., & Falletta, E. (2015). Effect of salicylic acid and 5-sulfosalicylic acid on UV-Vis spectroscopic characteristics, morphology, and contact angles of spin coated polyaniline and poly(4-aminodiphenylaniline) thin films. Journal of Spectroscopy, 2015. <https://doi.org/10.1155/2015/609175>



# The Paper (2012) ...

## Polyaniline nanofibers: Towards pure electrospun PANI

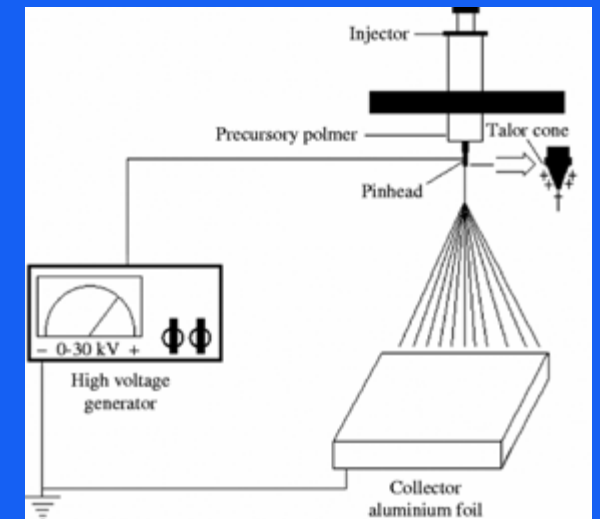
P. Frontera, C. Busacca, P. Antonucci, M. Lo Faro, E. Falletta et al.

Citation: AIP Conf. Proc. **1459**, 253 (2012); doi: 10.1063/1.4738460

View online: <http://dx.doi.org/10.1063/1.4738460>

“In this work we report the preparation of highly pure polyaniline fibers by electrospinning process.”

“Here we report a route to prepare PANI fibres reducing the PEO amount from 1 to 0% w/w with respect to the amount of PANI.”



# FFES - Process Parameters

**Polymer precursor:** PANI doped with Dodecyl Benzene Sulfonic Acid to produce PANI-DBSA

**Solvent:** Chloroform with different PEO to PANI-DBSA concentrations

**Deposition temperature:** 21 °C

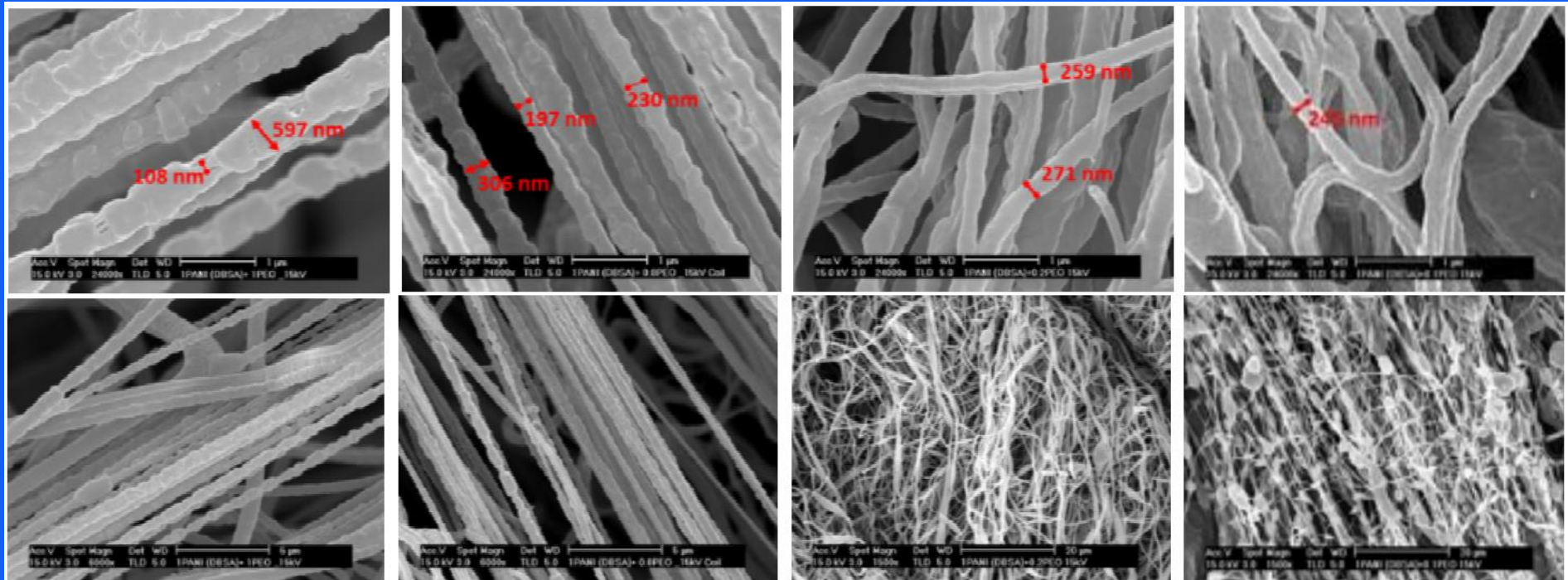
**Deposition rate:** 707  $\mu\text{l/h}$

**Needle-to-collector distance:** 12 cm

**Electrospinning variation:** rotating drum as collector



## high magnification



## low magnification

PANI:PEO 1:1 w/w

Fiber diam: 597nm

PANI:PEO 1:0.8 w/w

Fiber diam: 306nm

PANI:PEO 1:0.2 w/w

Fiber diam: 259nm

PANI:PEO 1:0.1 w/w

Fiber diam: 240







# References

1. Cárdenas, B. (2017). Advanced Manufacturing Techniques for the Fabrication and Surface Modification of Carbon Nanowires, 160.
2. Flores, D. R. (2017). Role of rheological properties in near field electrospun fibers morphology, 130.



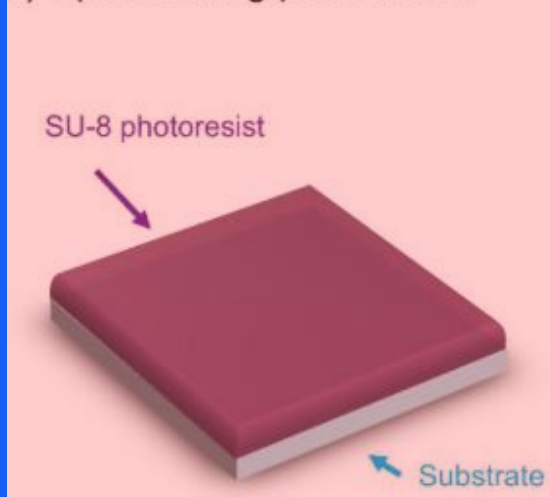


# Previous work

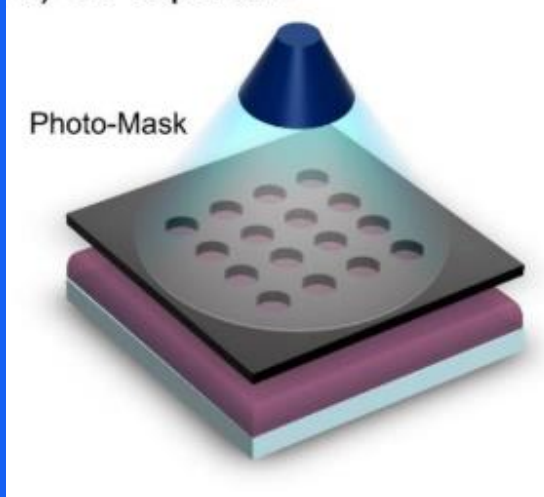
The production of C-MEMS:

1. Polymer patterning through ~~photolithography~~ electrospinning
2. Carbonization through ~~pyrolysis~~

I) Spin-coating photoresist



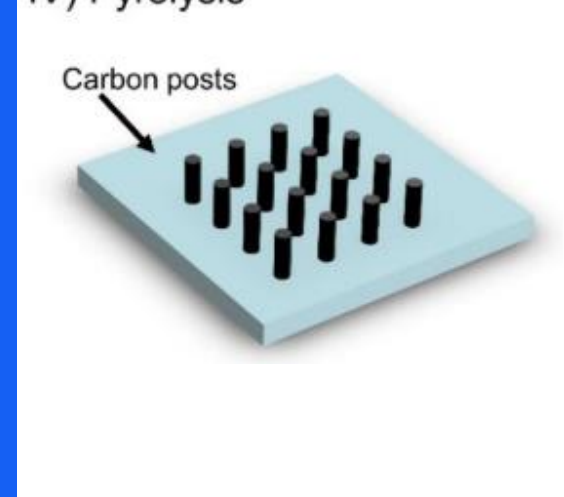
II) UV-exposure



III) Developing



IV) Pyrolysis



• SU-8 waste •



# Best Results so far by Braulio & Ricardo

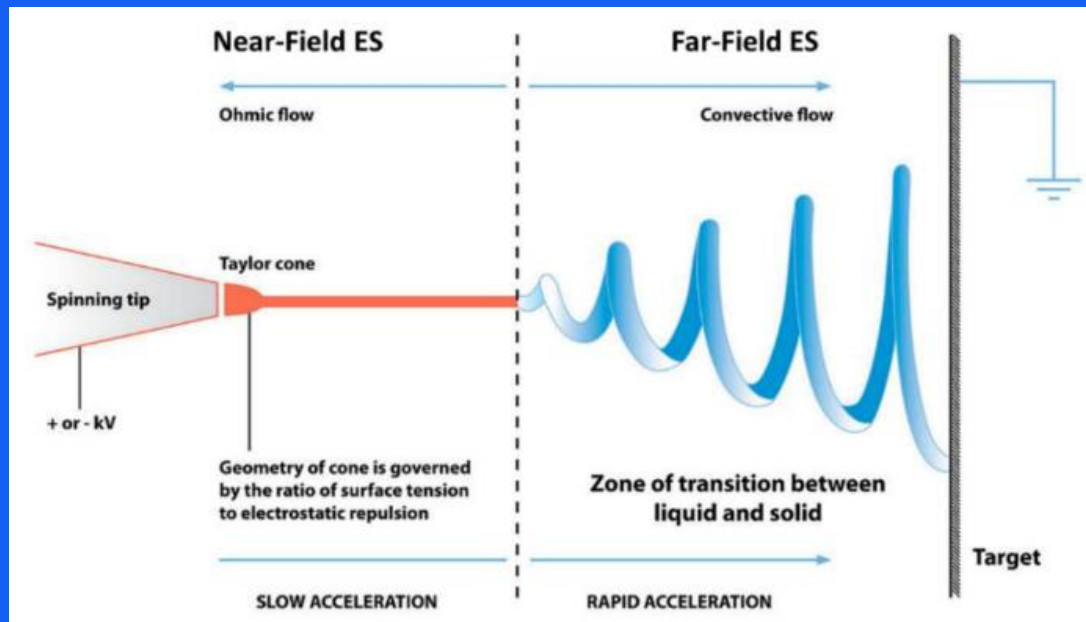
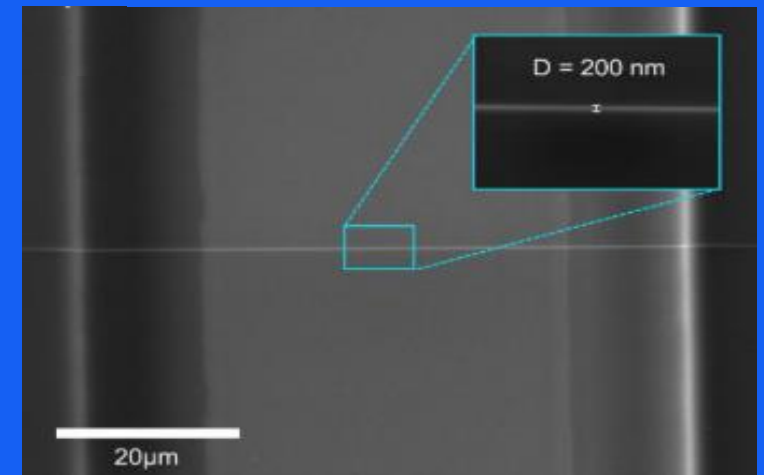
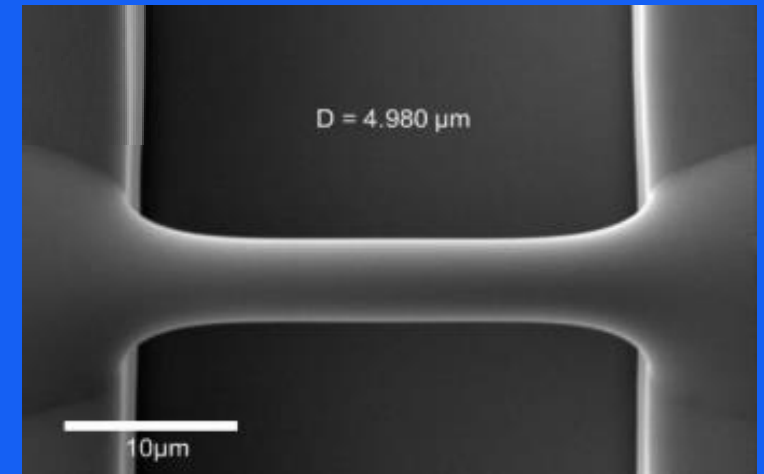
Fiber yield rate of 81 %

Fiber diameter before pyrolysis of  $4.966\text{ }\mu\text{m}$

Fiber diameter after pyrolysis of  $204\text{ nm}$

Fiber length of  $60.5 \pm 4.3\text{ }\mu\text{m}$

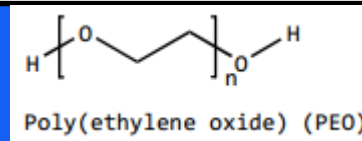
Fiber resistance from  $407\text{ K}\Omega$  to  $1.727\text{ M}\Omega$



# Polymer Solutions

wt% PEO	SU-8 2002 [mg]	PEO [mg]	TBT [mg]
0.25	2246	5.65	11.32

= 2 ml of solution



With zero shear viscosity of **0.88 Pa.s** electrospun at **600V** (solution used to get the results of the previous slide)



**Next step** -> Discover a new polymer solution to beat Braulio's record ...

# wt% PEO Solution

The EMS polymer solution consisted of 2ml of **SU-8 2002** mixed with 0.5 wt% of **Poly(ethylene oxide)** (PEO, 4,000,000 MW; SigmaAldrich Inc., Cat. N. 189464) and 0.5 wt% **Tetrabutylammonium Tetrafluoroborate** salts (TBATFB; SigmaAldrich Inc., Cat. N. 217964) to increase its conductivity and allow smooth polymer flow during electrospinning.

All reagents were used as received. Magnetic stirring of these components was performed for 1hr at 75°C and low rpm (100-150 rpm).



# MICROCHEM CORP SU-8 2002 500ML

encompass

Manufacturer: MICROCHEM CORP Y111029

Catalog No. NC0702370

\$628.71 / Each

Qty  Check Availability

Add to cart

<https://www.fishersci.com/shop/products/NC0702370/nc0702370#?keyword=MICROCHEM+CORP+PHOTORESIST+SU-8>

# MICROCHEM CORP SU-8 DEVELOPER 4L

encompass

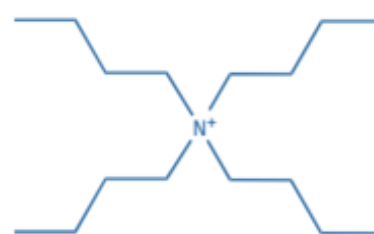
Catalog No. NC9901158

\$172.90 / Each

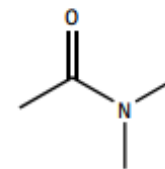
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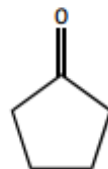
<https://www.fishersci.com/shop/products/NC9901158/nc9901158#?keyword=SU-8++developer>



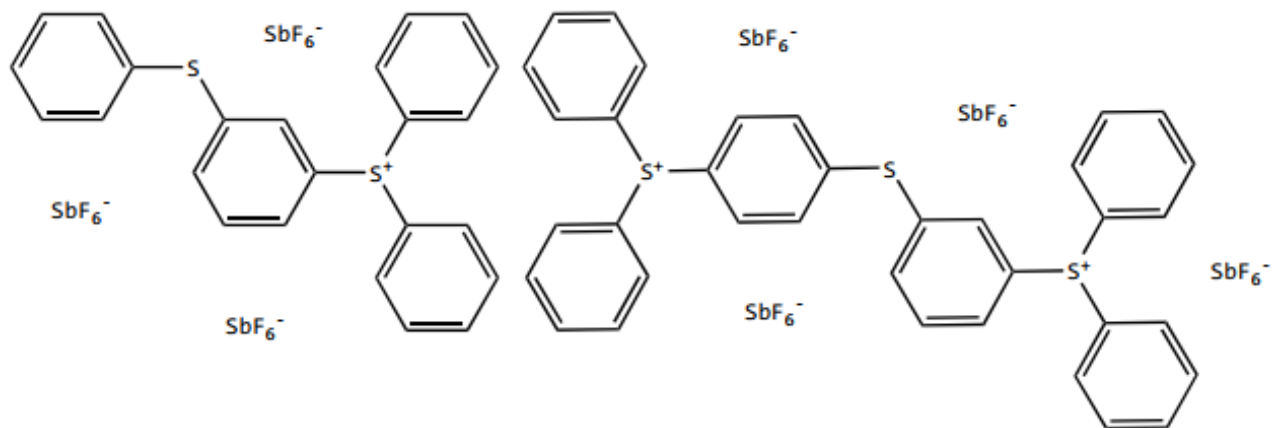
Tetrabutylammonium tetrafluoroborate (TBF)



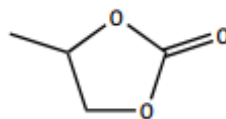
N,N Dimethylformamide (DMF)



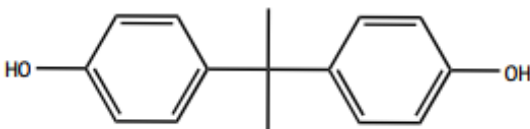
Cyclopentanone (CAS: 120-92-3)



Mixed Triarylsulfonium/Hexafluoroantimonate Salt (CAS: 89452-37-9)/(CAS: 71449-78-0)



Propylene Carbonate (CAS: 108-32-7)



Epoxy Resin (CAS: 28906-96-9)  
2-(Chloromethyl)oxirane; formaldehyde; 4-[2-(4-hydroxyphenyl)propan-2-yl]phenol





INF-WIDR\_DEP

2 step(s)

Force Level 100%

Syringe

Custom, 1000 ul, 4.78 mm

Step: 1 Constant Rate

Rate: 100 ul/min

Target: 0.5 ul

Step: 2 Constant Rate

Rate: 100 ul/min

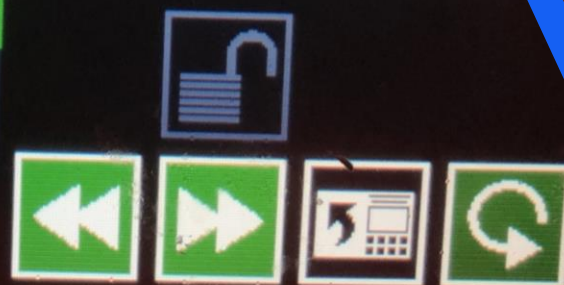
Target: 0.4 ul

Time Elapsed: 00:00:01

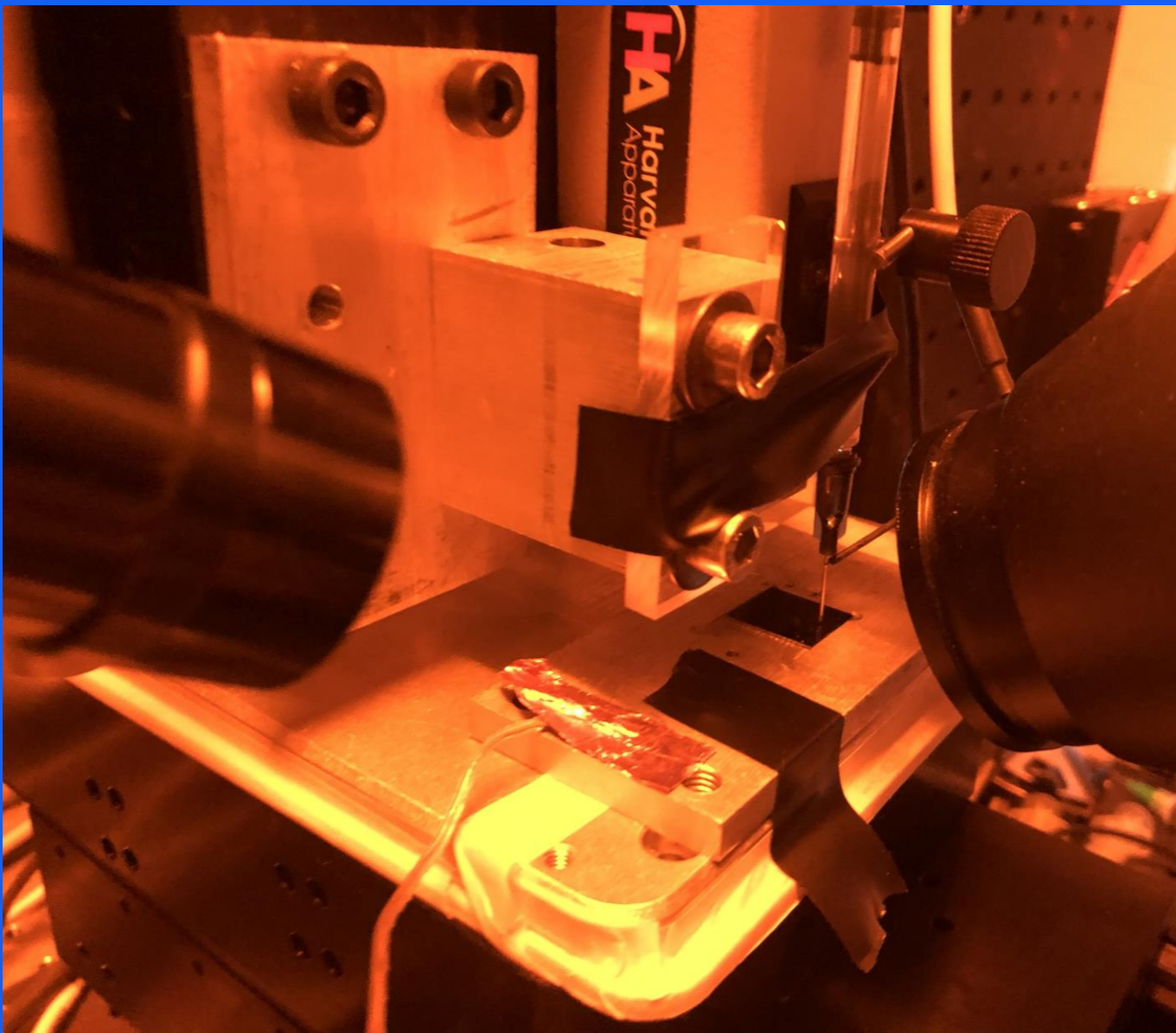
Total Volume Dispensed: 502.099 nl

Harvard Apparatus

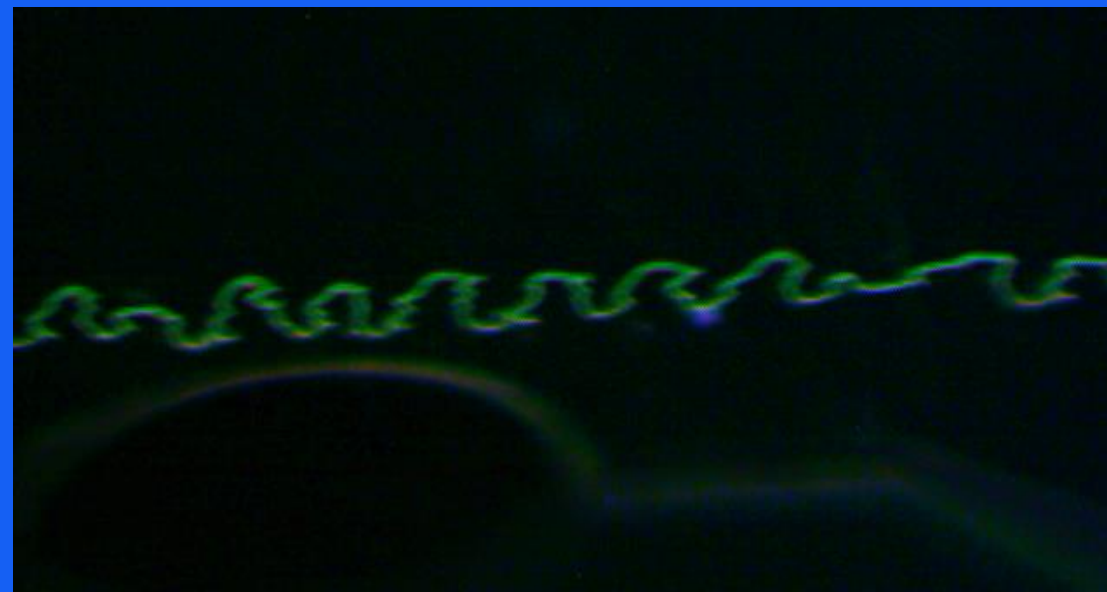
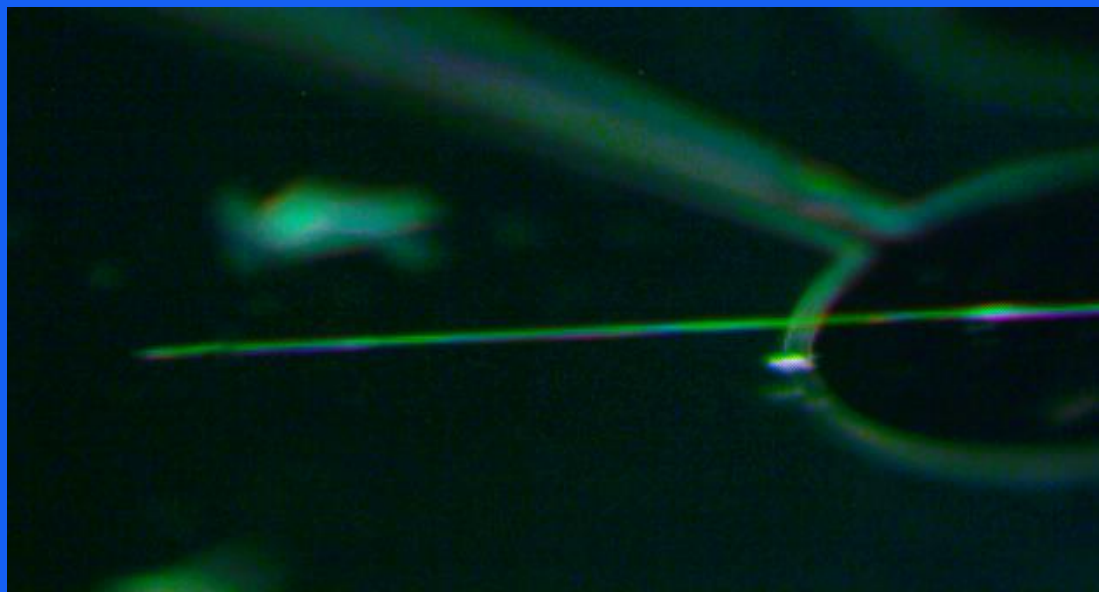
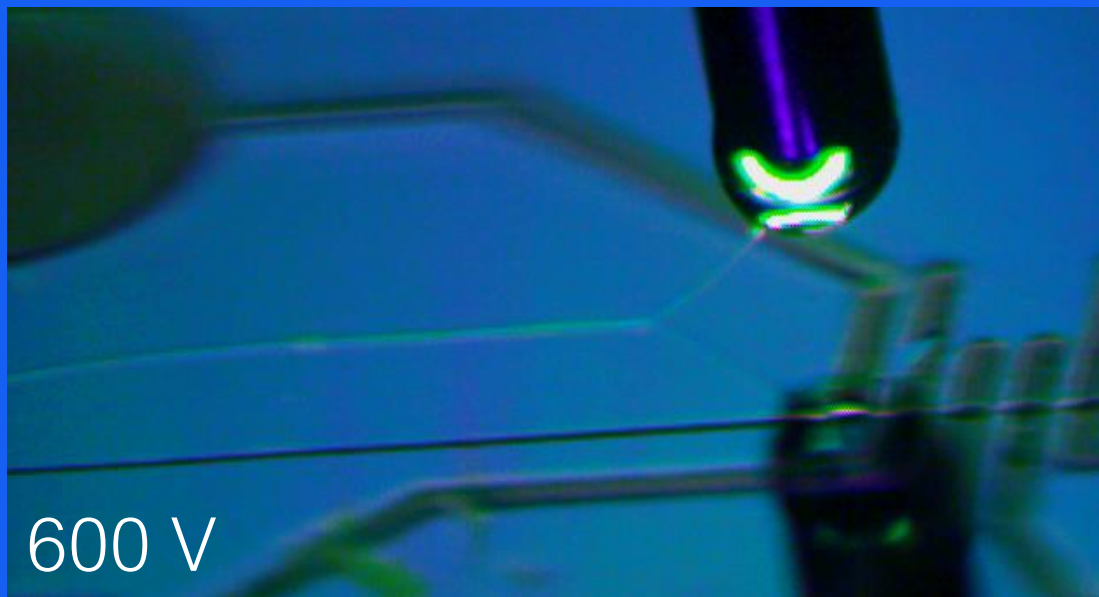
Method complete, ready to start method.

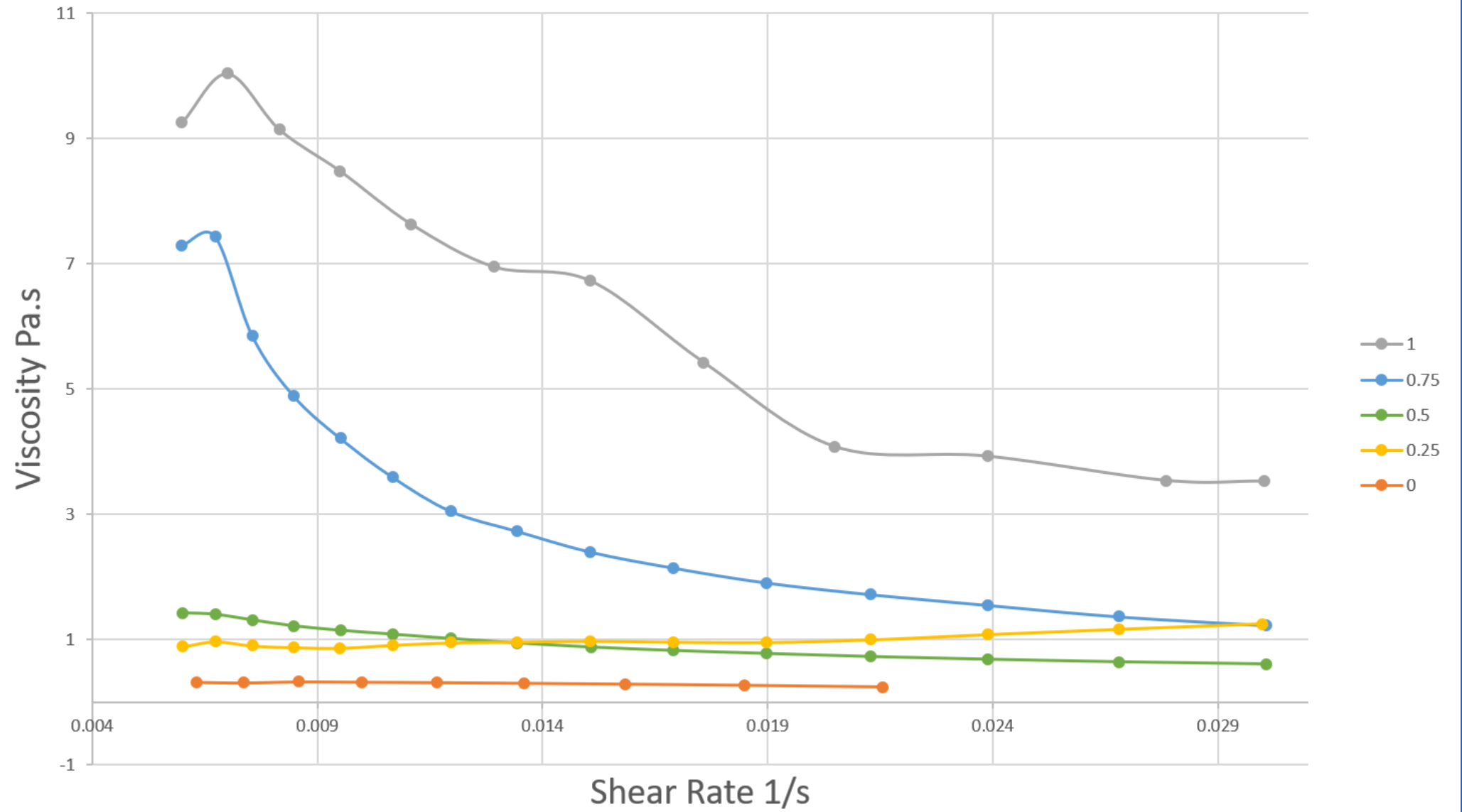












Safety Solvents		CAS Number	Presentation
	1-Methyl-2-pyrrolidinone (NMP)	872-50-4	anhydrous, 99.5% 328634-100ML 328634-1L
	Dichloromethane (Methylene chloride)	75-09-2	anhydrous, ≥99.8%, 40-150 ppm amylene as stabilizer 270997-100ML 270997-1L
	Dimethylacetamide (DMAc)	127-19-5	anhydrous, 99.8% 271012-100ML 271012-1L
250 ml	Dimethylformamide (DMF)	68-12-2	anhydrous, 99.8% 227056-100ML 227056-1L
1 L	Tetrahydrofuran (THF)	109-99-9	anhydrous, ≥99.9%, inhibitor-free 401757-1L
	Dihydrolevoglucosenone (Cyrene)	53716-82-8	807796-100ML 807796-1L
Polymers			
	Polystyrene (PS)	9003-53-6	average Mw 192,000 430102-1KG
	Poly(styrene-co-butadiene)	9003-55-8	butadiene 4 wt. %, melt index 6 g/10 min (200°C/5kg) 430072-1KG
	Poly(styrene-co-α-methylstyrene)	9011-11-4	457205-250G
	Polybenzimidazole (PBI)	26985-65-9	rod, diam. 9.5 mm, L 25 mm, black GF31259527-1EA
	Polyaniline (PANI / emeraldine salt)	25233-30-1	average Mw >15,000, powder (Infusible), 3-100 µm particle size 428329-5G
	Polyvinylcarbazole (PVK)	25067-59-8	average Mw ~1,100,000, powder, 182605-5G



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