Fabrication of graphitic-carbon suspended nanowires through mechanoelectrospinning of photocrosslinkable polymers

A Osamu Katagiri Tanaka
osamu.katagiri@exatec.tec.mx

Assessors:

Héctor Alán Aguirre Soto <alan.aguirre@tec.mx>

Dora Iliana Medina Medina <dora.medina@tec.mx>

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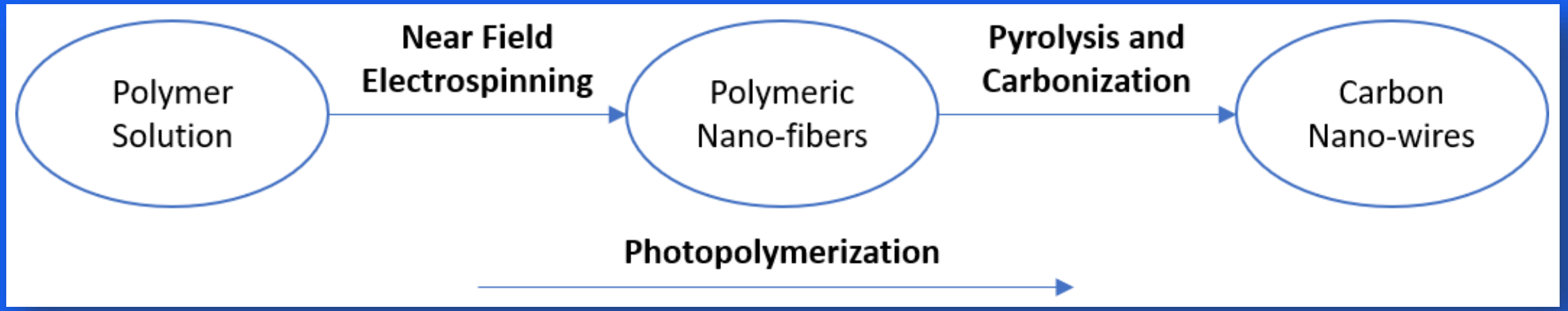
Thesis Overview



Design polymer solutions that can be electrospun by NFES, photopolymerized, and then pyrolyzed into carbon nano-wires.

Discover a new polymer solution to beat Braulio's record

Study solutions with high carbon polymers and no oxygen.

SU-8

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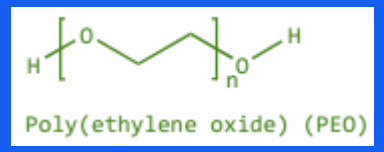
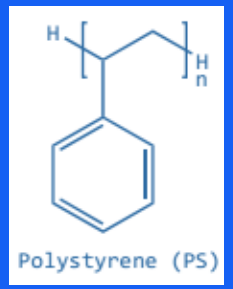
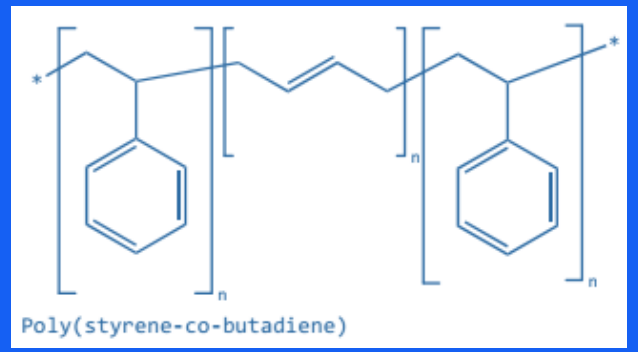


V.S.

in THF



in THF & DMF



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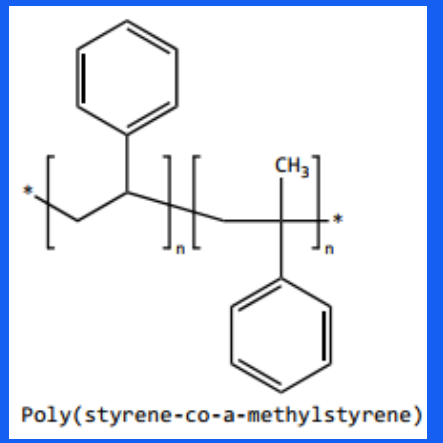

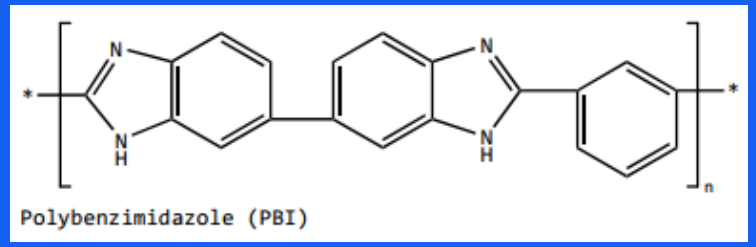

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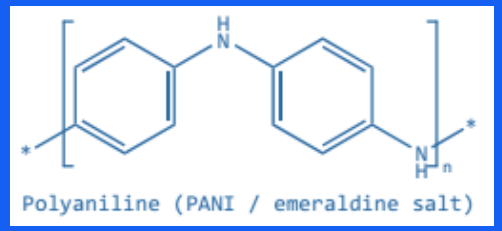
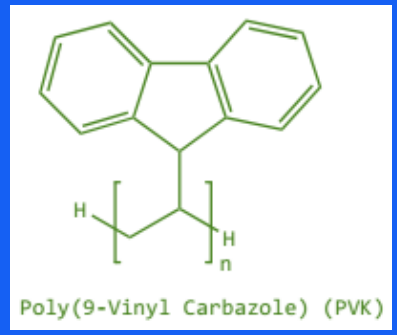


in NMP



in Styrene

- FFES
- NFES

The Carreau-Yasuda Model [1]

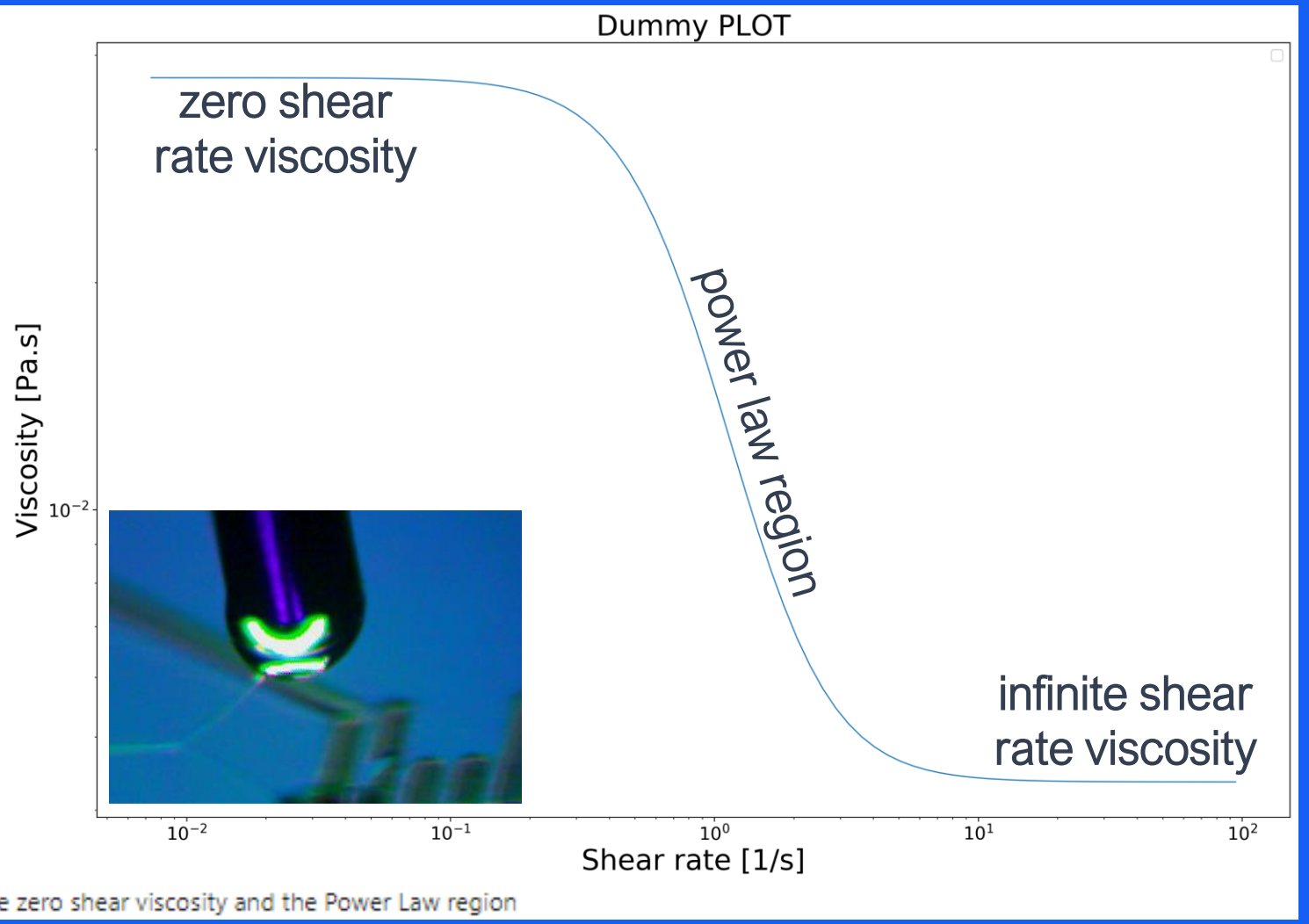
$$\frac{\eta - b}{a - b} = \frac{1}{\left[1 + (c\dot{\gamma})^e\right]^{\frac{1-d}{e}}}$$

$$\frac{\eta - \eta_\infty}{\eta_0 - \eta_\infty} = \frac{1}{\left[1 + (\kappa\dot{\gamma})^a\right]^{\frac{(1-n)}{a}}}$$

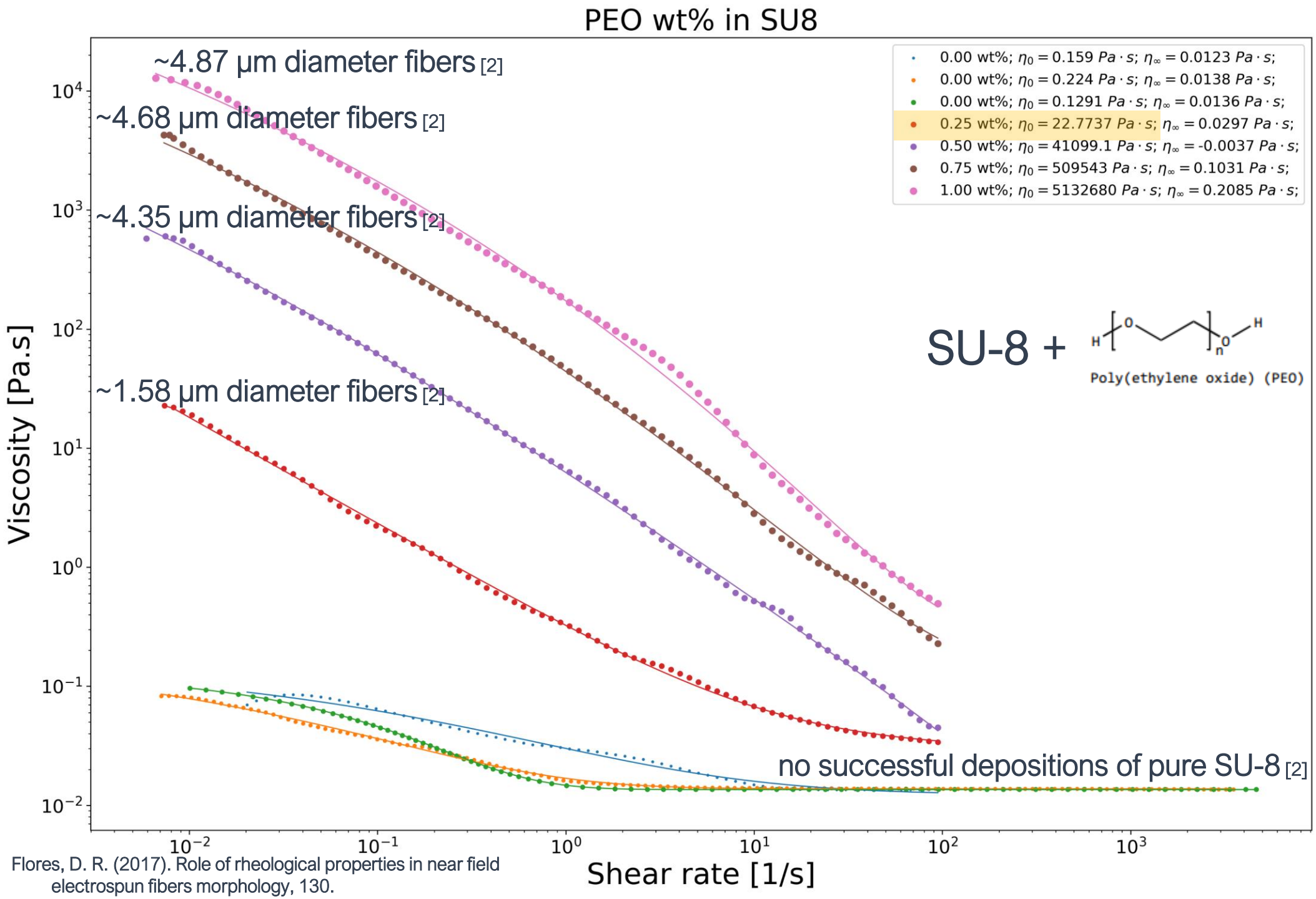
$$\eta = \frac{\eta_0 - \eta_\infty}{\left[1 + (\kappa\dot{\gamma})^a\right]^{\frac{(1-n)}{a}}} + \eta_\infty$$

where:

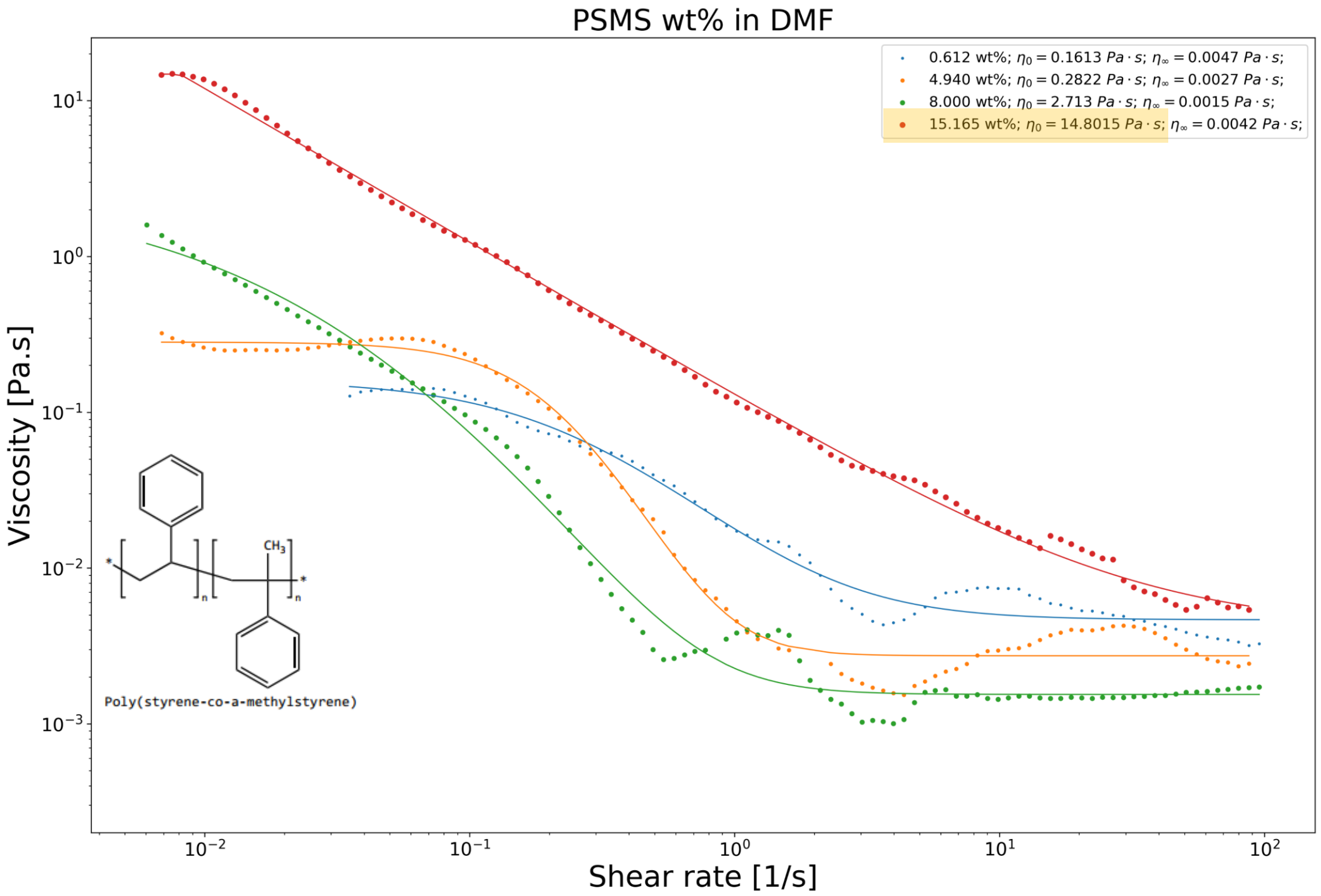
η = Viscosity
$\dot{\gamma}$ = Shear rate
η_∞ = Infinite shear rate viscosity
η_0 = Zero shear rate viscosity
κ = Time constant
n = The Power Law index
a = The width of the transition region between the zero shear viscosity and the Power Law region



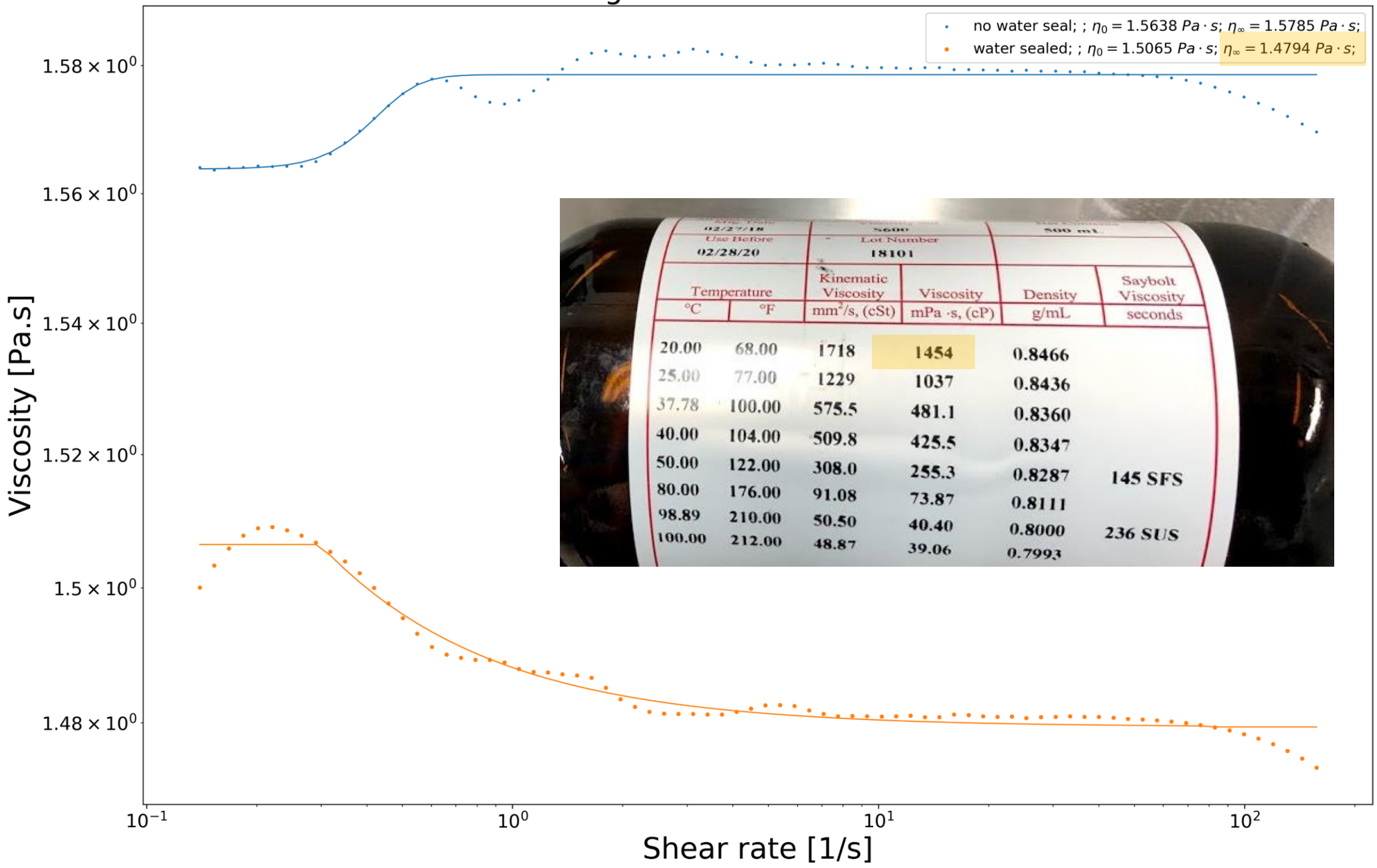
[1] Osswald, T., Rudolph, N., Osswald, T., & Rudolph, N. (2014). Generalized Newtonian Fluid (GNF) Models. Polymer Rheology, 59–99. https://doi.org/10.3139/9781569905234.003



Flores, D. R. (2017). Role of rheological properties in near field electrospun fibers morphology, 130.

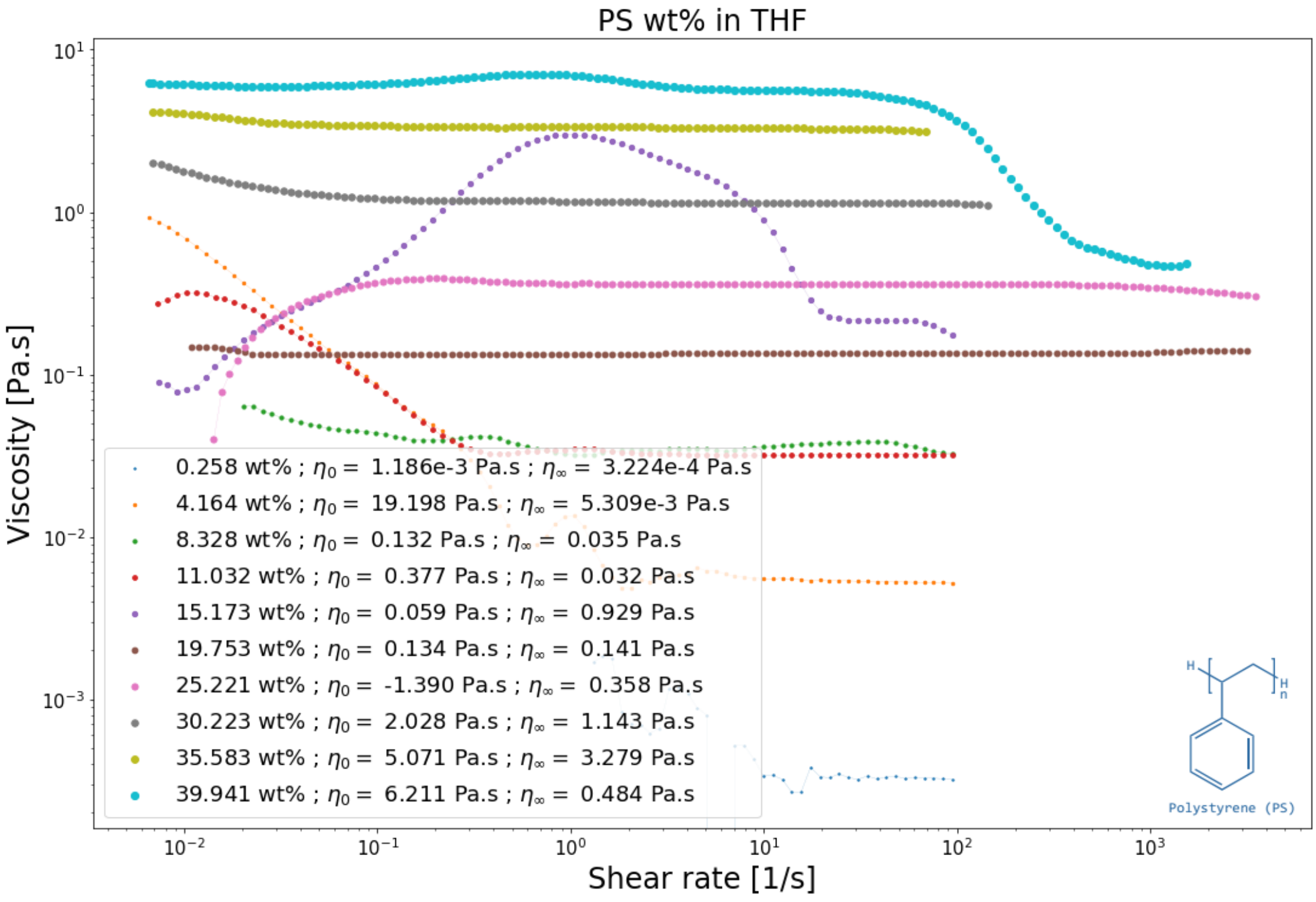

PSMS wt% in DMF
0.612 wt%; $\eta_0 = 0.1613\ Pa \cdot s$; $\eta_\infty = 0.0047\ Pa \cdot s$;
4.940 wt%; $\eta_0 = 0.2822\ Pa \cdot s$; $\eta_\infty = 0.0027\ Pa \cdot s$;
8.000 wt%; $\eta_0 = 2.713\ Pa \cdot s$; $\eta_\infty = 0.0015\ Pa \cdot s$;
15.165 wt%; $\eta_0 = 14.8015\ Pa \cdot s$; $\eta_\infty = 0.0042\ Pa \cdot s$;
Viscosity [Pa.s]
10^{1}
10^{0}
10^{-1}
10^{-2}
10^{-3}
10^{-2}
10^{-1}
10^{0}
10^{1}
10^{2}
Shear rate [1/s]
CH_3
Poly(styrene-co-a-methylstyrene)

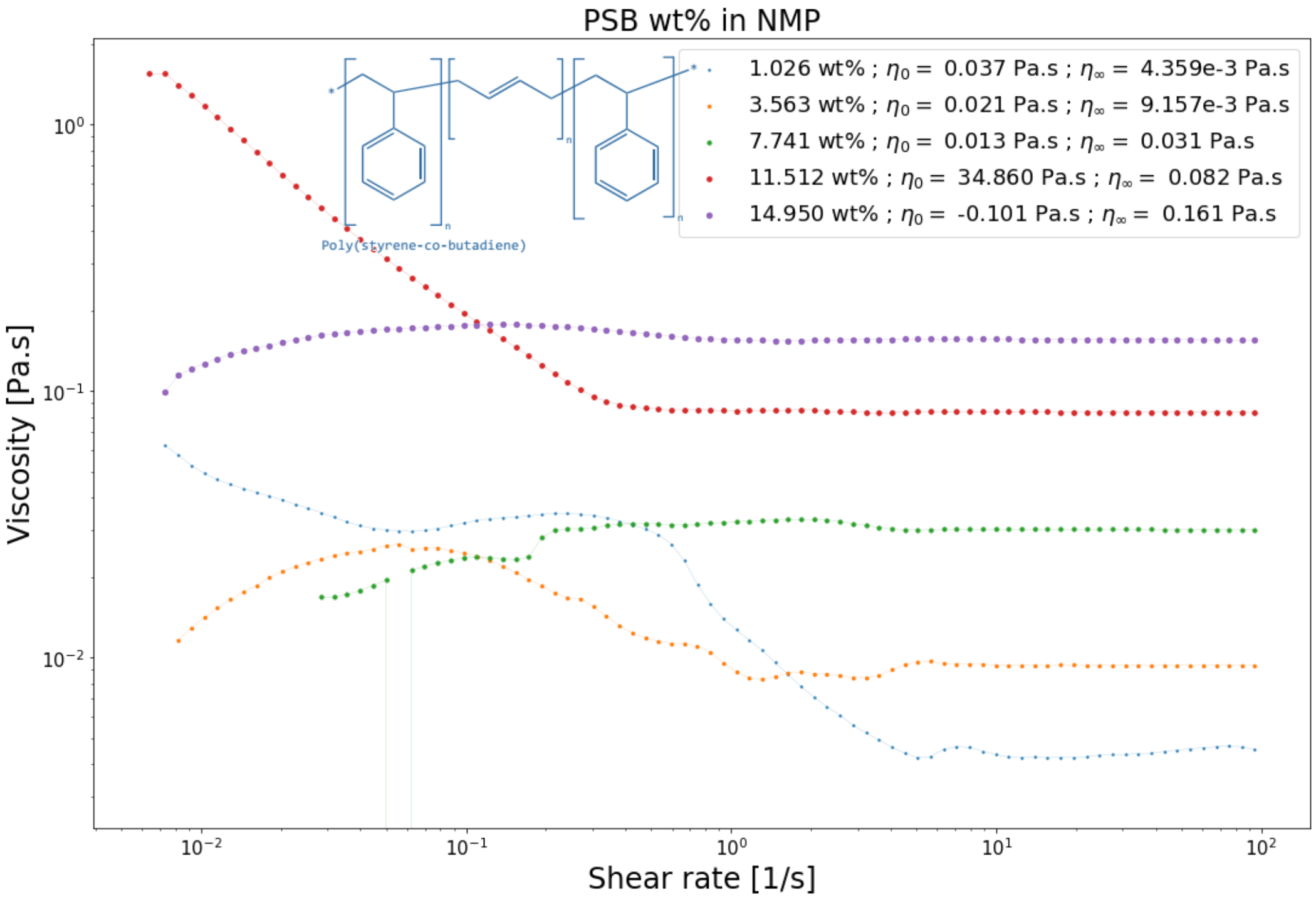
Standard Conforming to ASTM Oil Standard - Water Seal Test



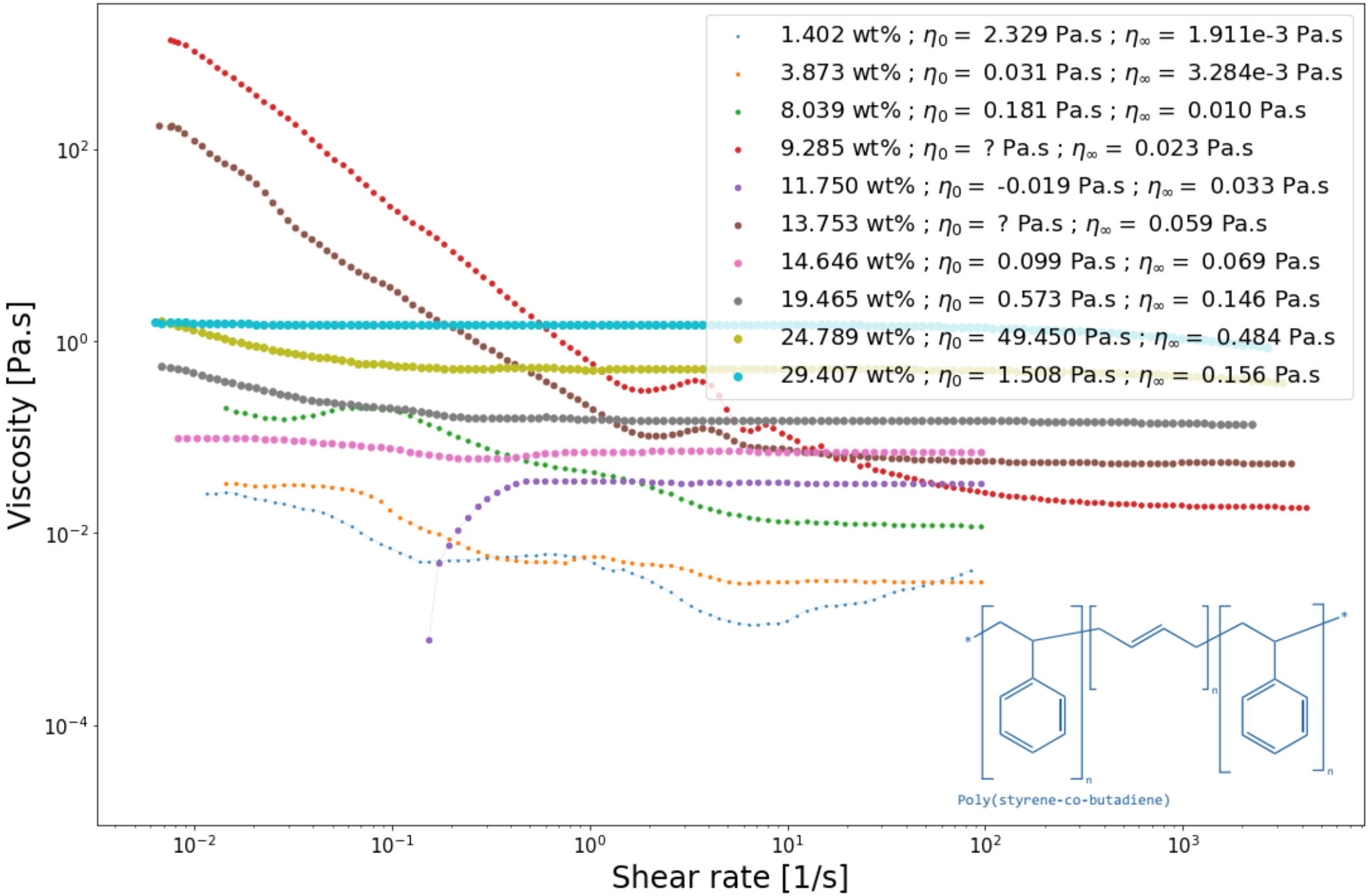
Use Before	Lot Number				
02/28/20	18101				
Temperature		Kinematic Viscosity	Viscosity	Density	Saybolt Viscosity
°C	°F	mm^2/s, (cSt)	mPa ·s, (cP)	g/mL	seconds
20.00	68.00	1718	1454	0.8466	
25.00	77.00	1229	1037	0.8436	
37.78	100.00	575.5	481.1	0.8360	
40.00	104.00	509.8	425.5	0.8347	
50.00	122.00	308.0	255.3	0.8287	145 SFS
80.00	176.00	91.08	73.87	0.8111	
98.89	210.00	50.50	40.40	0.8000	236 SUS
100.00	212.00	48.87	39.06	0.7993	


Q&A


PS wt% in THF
Viscosity [Pa.s]
Shear rate [1/s]
10^{1}
10^{0}
10^{-1}
10^{-2}
10^{-3}
10^{-2}
10^{-1}
10^{0}
10^{1}
10^{2}
10^{3}
0.258 wt% ; η_0 = 1.186e-3 Pa.s ; η_∞ = 3.224e-4 Pa.s
4.164 wt% ; η_0 = 19.198 Pa.s ; η_∞ = 5.309e-3 Pa.s
8.328 wt% ; η_0 = 0.132 Pa.s ; η_∞ = 0.035 Pa.s
11.032 wt% ; η_0 = 0.377 Pa.s ; η_∞ = 0.032 Pa.s
15.173 wt% ; η_0 = 0.059 Pa.s ; η_∞ = 0.929 Pa.s
19.753 wt% ; η_0 = 0.134 Pa.s ; η_∞ = 0.141 Pa.s
25.221 wt% ; η_0 = -1.390 Pa.s ; η_∞ = 0.358 Pa.s
30.223 wt% ; η_0 = 2.028 Pa.s ; η_∞ = 1.143 Pa.s
35.583 wt% ; η_0 = 5.071 Pa.s ; η_∞ = 3.279 Pa.s
39.941 wt% ; η_0 = 6.211 Pa.s ; η_∞ = 0.484 Pa.s
H
H
n
Polystyrene (PS)

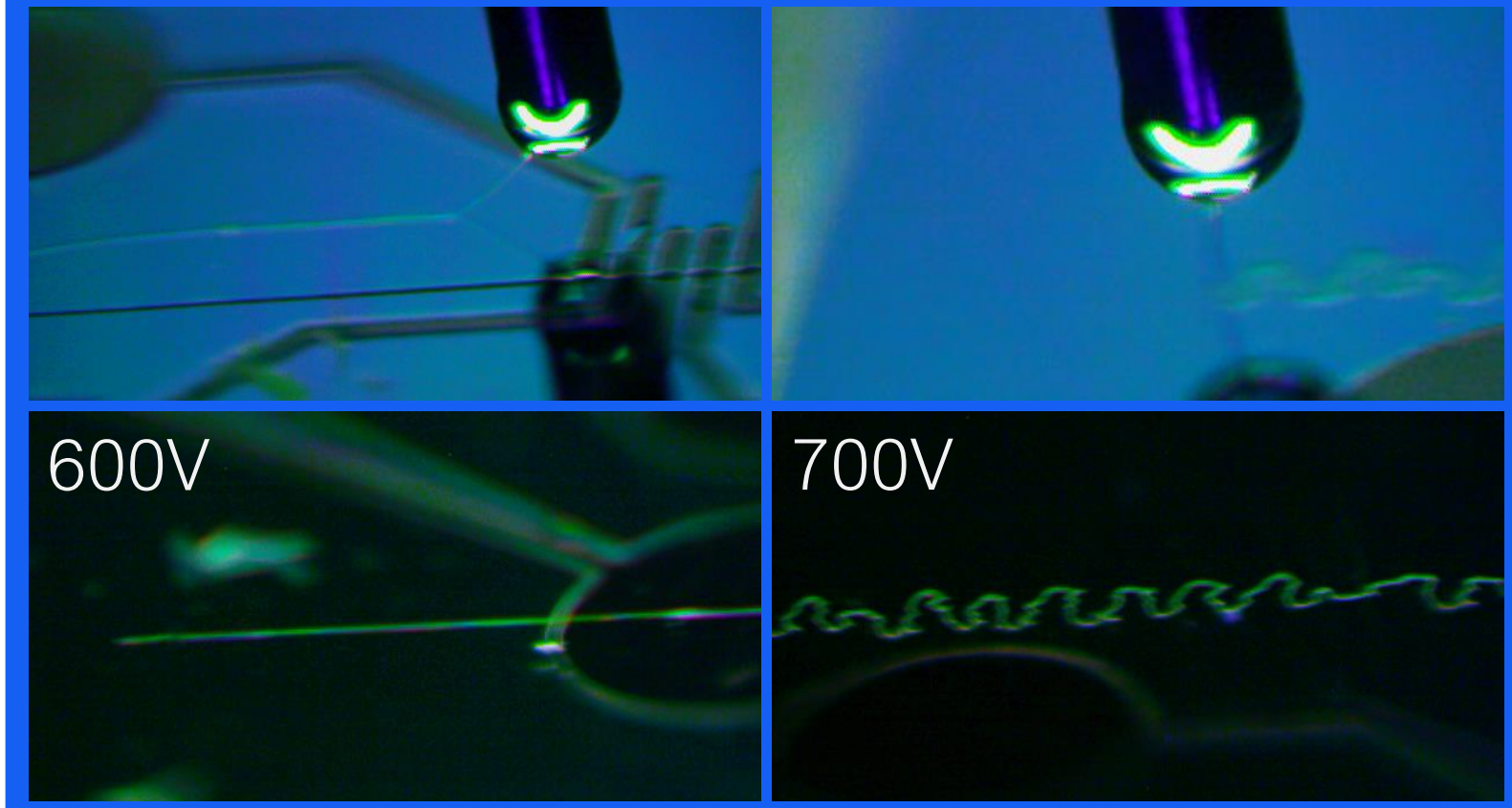

PSB wt% in NMP
Poly(styrene-co-butadiene)
1.026 wt% ; η_0 = 0.037 Pa.s ; η_∞ = 4.359e-3 Pa.s
3.563 wt% ; η_0 = 0.021 Pa.s ; η_∞ = 9.157e-3 Pa.s
7.741 wt% ; η_0 = 0.013 Pa.s ; η_∞ = 0.031 Pa.s
11.512 wt% ; η_0 = 34.860 Pa.s ; η_∞ = 0.082 Pa.s
14.950 wt% ; η_0 = -0.101 Pa.s ; η_∞ = 0.161 Pa.s
Viscosity [Pa.s]
10^0
10^{-1}
10^{-2}
10^{-2}
10^{-1}
10^0
10^1
10^2
Shear rate [1/s]

PSB wt% in THF and DMF

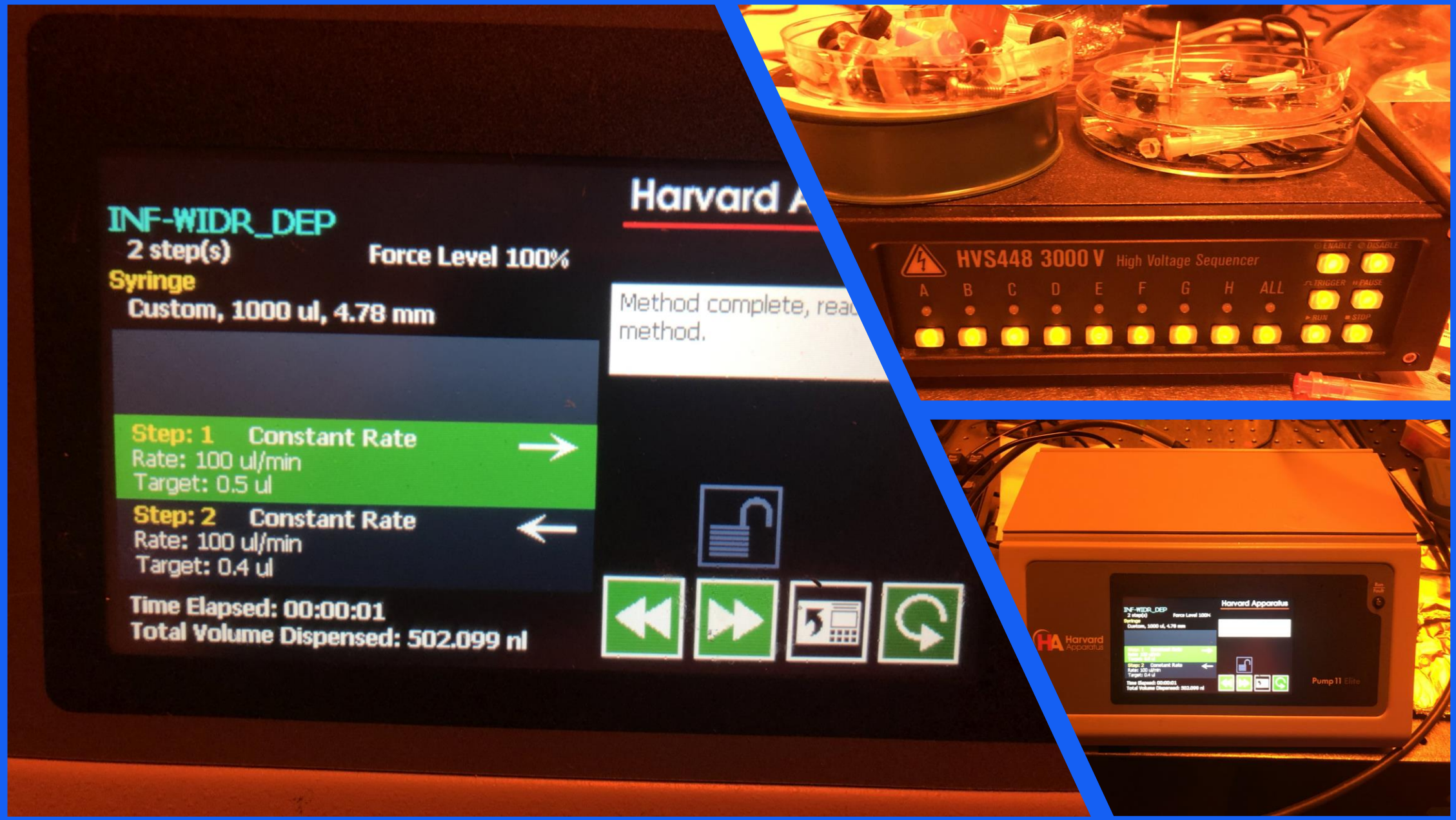


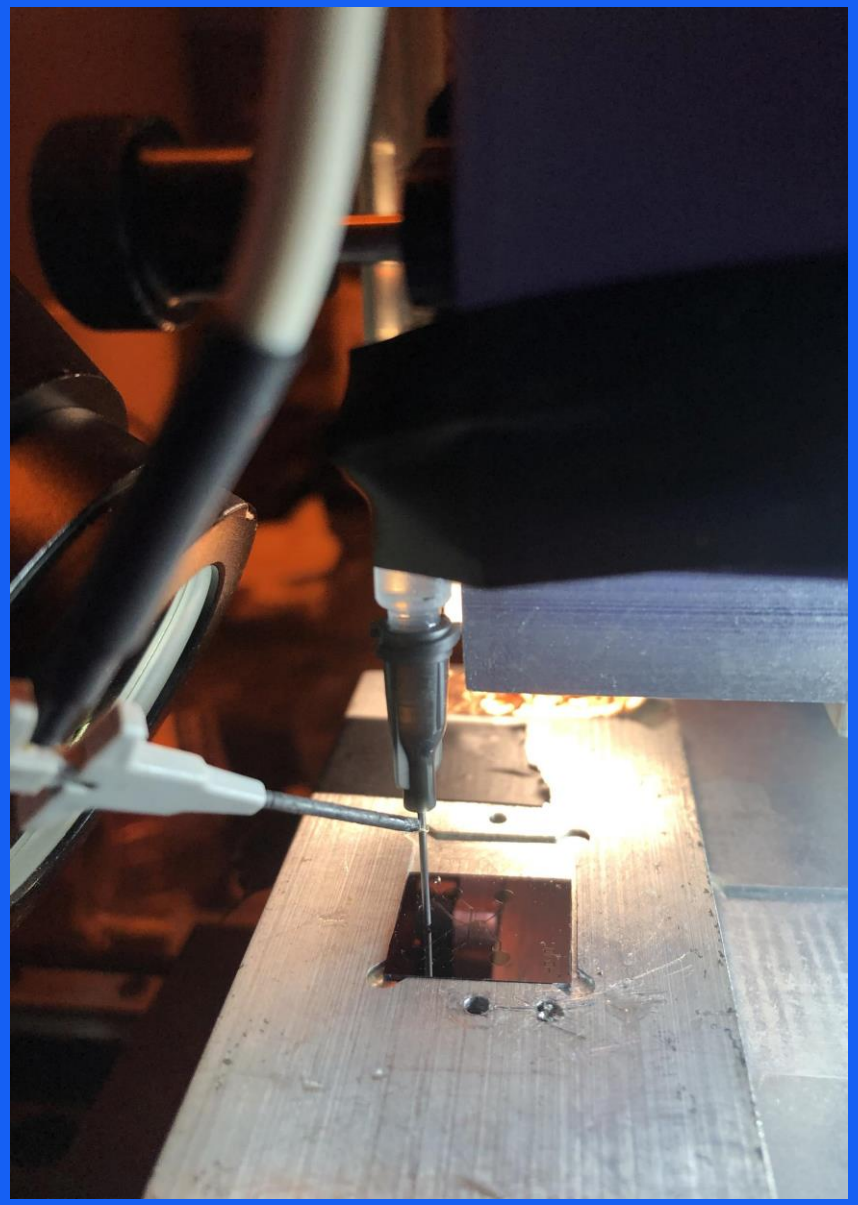
Characterization of the 0.25 wt% PEO Solution

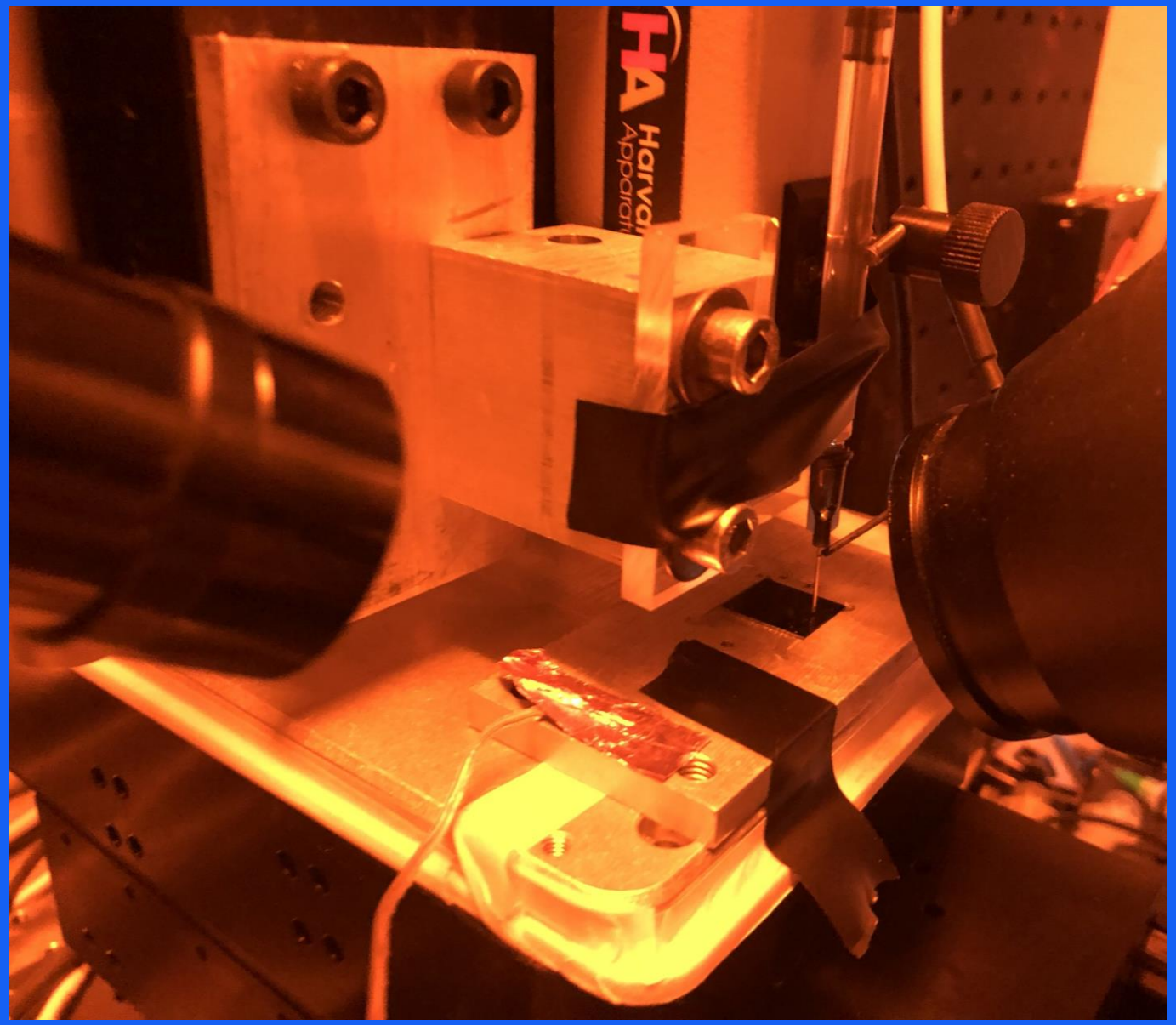
Electrospun-able with an applied voltage of 600V.



Unable to initiate the jet at 500V or lower.


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 A
Method complete, rea
method.
HVS448 3000 V High Voltage Sequencer
A B C D E F G H ALL
ENABLE DISABLE
TRIGGER PAUSE
RUN STOP
Harvard Apparatus
Pump 11 Elite




HA
Harvard
Apparatus

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

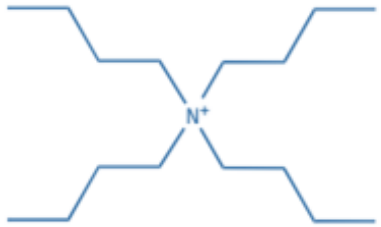
Catalog No.	NC0702370

$628.71 / Each

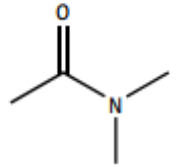
Qty Check Availability

Add to cart

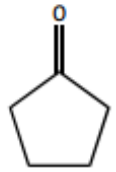
https://www.fishersci.com/shop/products/NC0702370/nc0702370#?keyword=MICROCHEM+CORP+PHOTORESIST+SU-8



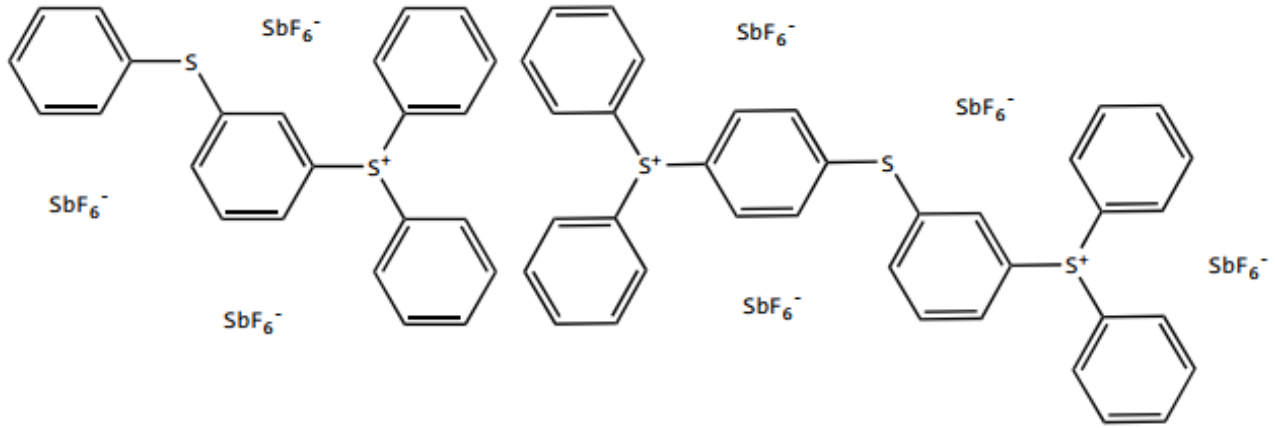
Tetrabutylammonium tetrafluoroborate (TBF)



N,N Dimethylformamide (DMF)



Cyclopentanone (CAS: 120-92-3)



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

MICROCHEM CORP SU-8 DEVELOPER 4L

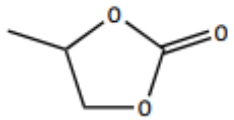
encompass

Catalog No.	NC9901158

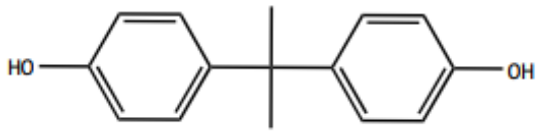
$172.90 / Each

INGREDIENTS: Cyclopentanone (CAS: 120-92-3); 23-78%.
Mixed Triarylsulfonium/ Hexafluoroantimonate Salt;
(CAS: 89452-37-9)/(CAS: 71449-78-0); 1-5%
Propylene Carbonate (CAS: 108-32-7); 1-5%
Epoxy Resin (CAS: 28906-96-9); 25-75%

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



Propylene Carbonate (CAS: 108-32-7)



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

Zero shear viscosity measurements and Flores' measurements

Sample	PEO concentration [$wt\%$]	Preliminary zero shear viscosity measurements [$Pa.s$]	Flores' zero shear viscosity measurements [$Pa.s$]
1	0.00	0.31	0.03
2	0.25	0.88	0.3
3	0.50	1.42	0.4
4	0.75	7.28	2
5	1.00	9.25	3

Both measurements have the same behaviour with similar proportions.

However, the results show that the current measured values are approximately three times larger than Flores' measurements.

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