A Brief Survey of Contact Angle Measurements for Predicting Fluid Flow in Electrospray Emitters

CNF Project Number: 295321

Principal Investigator(s): Sadaf Sobhani
User(s): Lindsay Wright

Affiliation(s): Sibley School of Mechanical and Aerospace Engineering

Primary Source(s) of Research Funding: RA Offer for the Defense Advanced Research Projects Agency, Account #E559359

Contact: sobhani@cornell.edu, law289@cornell.edu

Research Group Website: sobhanilab.com

Primary CNF Tools Used: VCA Optima Goniometer, spincoating fume hoods

Abstract:

Electrospray is a method of satellite propulsion and mass spectrometry that works by moving ionic liquid from a receptacle to emitter tips and then vaporizing and propelling the ions using strong alternating electric fields from an extractor. With recent advances in additive manufacturing, electrospray emitters can be custom manufactured for different surface properties to optimize fluid flow. However, fluid flow in electrospray emitters has mainly been parametrized using metrics such as permeability or impedance, without the level of detail necessary to design channel networks. Our work aims to model the effects of specific materials and surface treatments on the speed of fluid flow—to develop an accurate predictive framework, a study of different materials' surface properties is necessary. Thus, a series of samples have been fabricated in the Cornell Nanoscale Facility to obtain contact angle measurements.

Summary of Research:

Two Photon Polymerization was attempted to create contact angle measurements that matched the surface characteristics of additively manufactured electrospray emitters. Due to bumps and other surface texture discrepancies caused by stitching in larger print sizes, we opted for spin coating surfaces instead, curing them under UV to replicate the Two Photon Polymerization process with a more even surface.

The following recipes were created for spin coating:

GP Silica: Dispense approximately 1 mL of resin on 1" silicon wafer square. Then, ramp to 500 rpm at 100 rpm/sec, and hold for 5-10 seconds. Next, ramp to 1000 rpm/s at a rate of 300 rpm/sec, and hold for 30 seconds. Finally, soft bake for 6 mins at 65 degrees Celsius, then 20 mins at 95 degrees Celsius

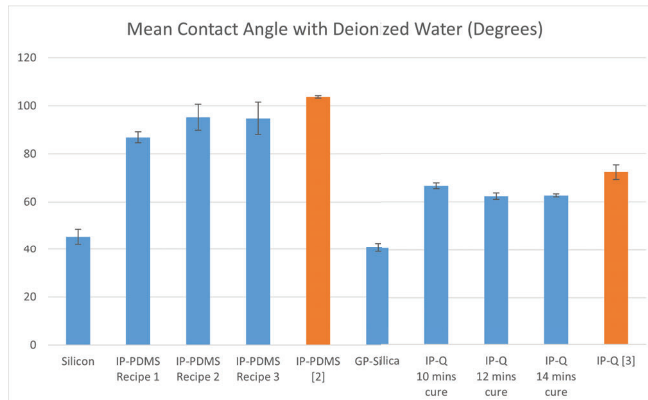

Figure 1: Contact Angles of Spin Coated Polymer Resin Recipes with Deionized Water

IP-PDMS Recipe 1 (created by collaborator at Rice University): Dispense approximately 1 mL of resin on 1" silicon wafer square. Next, ramp at 10 rpm/s to 500 rpm, and hold for 5 seconds. Finally, UV cure the sample for 10 minutes using an Aiffoto brand lamp [1].

IP-PDMS Recipe 2: Dispense approximately 1 mL of resin on 1" silicon wafer square. Next, ramp at 10 rpm/s to 500, and hold for 5 seconds. Then, UV cure samples for 30 minutes using the cylinder lamp at the Cornell Nanoscale Facility, reported to have an energy flux of 21 mW/cm^2 @ 405nm

IP-PDMS Recipe 3: Prime a 1" silicon wafer with 20% HMDS (first at 700 rpm, with a 1000 rpm/s ramp at 10 seconds duration; then, at 2500 rpm, with a 1000 rpm/s ramp and 60 seconds duration). Then, dispense approximately 1 mL of resin on 1" silicon wafer square. Ramp the spin coater at 10 rpm/s to 500 rpm and hold for 5 seconds. Next, UV cure samples for 30 minutes using the cylinder in the Cornell Nanoscale Facility. Finally, develop for 10 minutes in isopropanol.

IP-Q: Prime a 1" silicon wafer with 20% HMDS. Then, dispense approximately 1 mL of resin on 1" silicon wafer square. Ramp to 500 rpm at 100 rpm/sec and hold for 5 seconds. Next, ramp to 2500 rpm at 300 rpm/sec, for a total of 40 seconds. Then, UV cure samples to the desired time (10, 12, or 14 minutes) using the Cornell Nanoscale Facility lamp.

Finally, develop for 10 minutes in isopropanol.

Contact angle measurements of varying surface recipes with deionized water were measured with a Ramehart 500 Goniometer, except for IP-PDMS samples fabricated using Recipes 2 and 3, which were measured by processing iPhone 16 images with ImageJ. The data with standard error bars can be found in Figure 1, along with literature values for IP-PDMS and IP-Q to compare [2] [3].

A preliminary study of the contact angle measurements of ionic nanoparticle fluids suspended in polymer matrices was also conducted for silicon and IP-PDMS. Silicon had a contact angle of 48.3 degrees with water, 17.6 degrees with Jeffamine, and 13.4 degrees with NIMS 15 2:1, while Recipe 1 for IP-PDMS had a contact angle of 90.7 degrees with water, 37.7 degrees with Jeffamine, and 64.2 degrees with NIMS 15 2:1.

Conclusions and Future Steps:

A majority of the polymer resins used were weakly hydrophilic to water. GP-Silica, due to being a glass resin, is more strongly hydrophilic, while IP-PDMS is borderline hydrophobic. It will be interesting to continue to study the properties of ionic liquids and ionic nanoparticle fluids against different surfaces to determine whether more complex interactions are occurring that influence wettability.

IP-PDMS and IP-Q contact angle measurements were found to be consistently more hydrophilic than literature values, with the difference between literature and observations being more pronounced in IP-PDMS samples. It is noted that Govindarajan et al cured IP-PDMS using a 10W 395nm wavelength lamp [2]. Isaakidou used Two Photon Polymerization with a center wavelength of 780 nm, operating at 80 MHz at a duration of 100 fs to fabricate IP-Q samples [3]. The degree to which these differences affect the contact angle is a point to investigate.

A preliminary study of UV cure time and contact angle measurements was made for IP-Q, with the contact angle decreasing slightly over time from 10 to 12 to 14 minutes. Future steps would include experimenting with different treatments in addition to UV cure time. Experimentalists have found, for example, that UV-ozone treatment of PDMS can reduce the contact angle at controllable rates, which could be preserved by storage in distilled water [4]. Additional surface treatments could include etching, Atomic Layer Deposition, and Molecular Vapor Deposition to further manipulate the surface.

References:

[1] Amazon (n.d.). 3D Printing UV Resin Curing Light 405nm, Smart 3D Printer UV Resin Curing Light for Solidify Photosensitive Resin, Portable Timer Function DIY Curing Enclosure, 5V/2A Input USB Powered. https://www.amazon.com/dp/B0D5HKQ2DQ?ref_=ppx_hzsearch_conn_dt_b_fed_ asin_title_1

[2] Isaakidou, Aikaterini, Iulian Apachitei, Lidy Elena Fratila-Apachitei, and Amir Abbas Zadpoor. 2023. "High-Precision 3D Printing of Microporous Cochlear Implants for Personalized Local Drug Delivery" Journal of Functional Biomaterials 14, no. 10: 494. https://doi.org/10.3390/jfb14100494

[3] Srinivasaraghavan Govindarajan, Rishikesh & Sikulskyi, Stanislav & Ren, Zefu & Stark, Taylor & Kim, Daewon. (2023). Characterization of Photocurable IP-PDMS for Soft Micro Systems Fabricated by Two-Photon Polymerization 3D Printing. Polymers. 15. 10.3390/polym15224377.

[4] Kun Ma, Javier Rivera, George J. Hirasaki, Sibani Lisa Biswal, Wettability control and patterning of PDMS using UV–ozone and water immersion, Journal of Colloid and Interface Science, Volume 363, Issue 1, 2011,Pages 371-378, ISSN 0021-9797, https://doi.org/10.1016/j.jcis.2011.07.036.