Characterization of Low Friction Polymer Brush Coating for Use in Medical Devices

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The Sponsor has developed a low-friction polymer coating for medical devices in which further characterization of the coating would benefit future research and development efforts. All samples were provided by the Sponsor. Our team devised methods to prepare samples for testing and created test methods for coating characterization. We used Quartz Crystal Microbalance, Friction Testing, and Dynamic Light Scattering to analyze the samples. Our findings indicate minimal change in friction coefficient between sample types, however cross-linking the polymer coating provides increased durability.

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Background

Minimally invasive surgeries often require specialized devices. To ensure safe insertion, specialty lubricants are needed with high biocompatibility and ease of use for operators. The Sponsor is investigating a surface modification to increase lubricity of a medical device during use. This coating is still in research and development by the Sponsor and is not available for commercial use. While test samples have been provided by the sponsor, the resultant surface characteristics are not yet thoroughly understood.

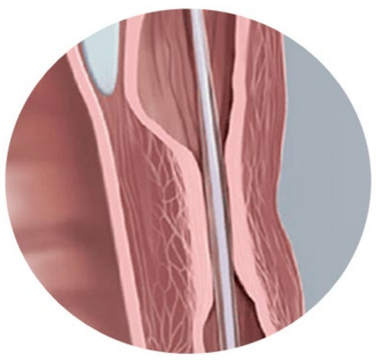

Figure 1: Diagram showing example of minimally invasive medical device [1]

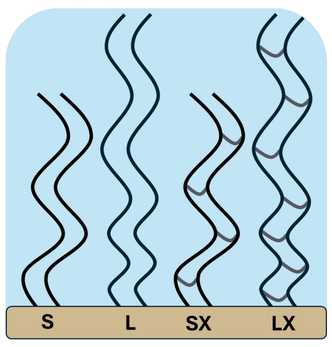

Figure 2: Diagram showing the brush types provided to our group, short (S), long (L), cross-linked short (SX), and cross-linked long (LX).

The coating made by the Sponsor is a hydrophilic polymer brush. This required unique sample preparation as the brush can only be attached to specific polymers. Elastomer rounds and spun coated quartz crystals were grafted with the polymer brushes in short, long, and crosslinked version of each. These brushes were then put through a variety of tests to determine physical properties of interest to the Sponsor.

These polymer brushes reduce friction with their extreme hydrophilic properties. The hydrated brush strongly adsorbs a water layer to the surface which is what lowers friction. Commercially available hydrophilic coatings provide varying degrees of chemical and physical attachment to the device surface, and one of the primary areas needing further evaluation regarding the new surface modification process is characterization of the attachment strength to the substrate surface

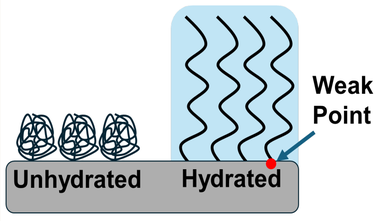

Figure 3: Diagram showing the conformation change of the hydrated polymer and a potential point of failure.

Hydration Kinetics

Methods
Hydration of the brush was measured using quartz crystal microbalance. DI water was flowed over functionalized samples at 150μL/min. Mass was found using eq 1 which assumes perfect elasticity.

$$\Delta m = -\frac{C}{n}\Delta f$$

Equation 1: Sauerbrey Equation
C = Constant for the sensor (17.7 ng/Hz)
n = Overtone
f = Frequency measured by instrument [2]

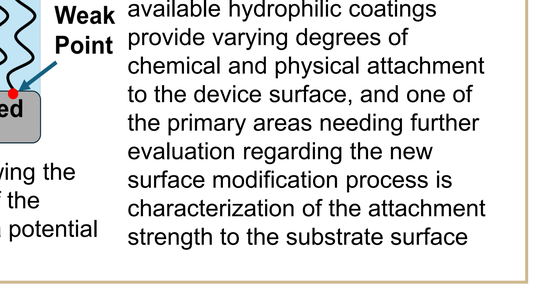

Figure 4: Diagram showing the motion of QCM sensors during operation as well as a visual representation of overtones. [3]

Results

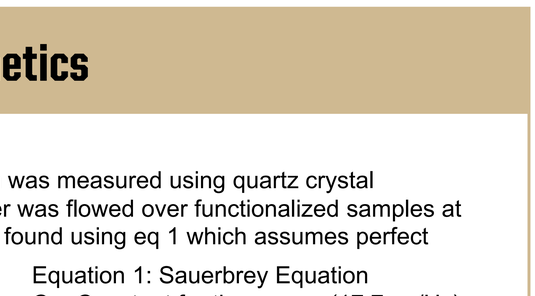

Figure 5: Change in adsorbed mass with time for each brush type.

Discussion
Figure 3 shows that a constant mass is reached in about 10 seconds. Due to time and financial constraints, only one sample was taken for this data.

Friction Testing

Methods
Friction coefficients of polymer brush on elastomer rounds were measured using a rheometer. 10-minute hydration and shear time, constant gap, and constant shear rate.

$$F_R = \frac{4T}{3R}$$

Equation 2: Sliding Friction
T = Torque as measured by rheometer
R = Parallel Plate Radius (20 mm)

$$COF = \frac{\overline{F_R}}{\overline{N}}$$

Equation 3: Coefficient of Friction
FR = Time averaged sliding force
N = Time averaged normal force as measured by rheometer [4]

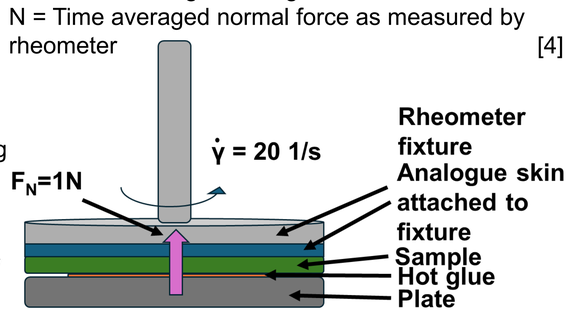

Figure 6: Diagram showing testing set up for friction tests using parallel plate geometry in rheometer

Results

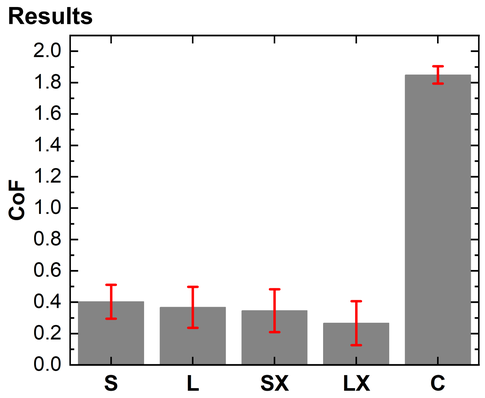

Figure 7: Friction coefficient values for elastomer rounds with four brush types, error bars show standard deviation across tests as well as error from time averaged values in eq 3.

Discussion
Figure 5 shows a significant decrease in friction coefficient for the samples with the brush coating added. Due to the error incurred in time averaging values and multiple samples, the difference in friction coefficient between the different brush types can be considered negligible.

Durability Testing

Methods
Durability was measured by repeating the friction tests described above 3 times and studying the change in CoF. These tests were done using an immersion cell to analyze dissolved polymer residue after testing.

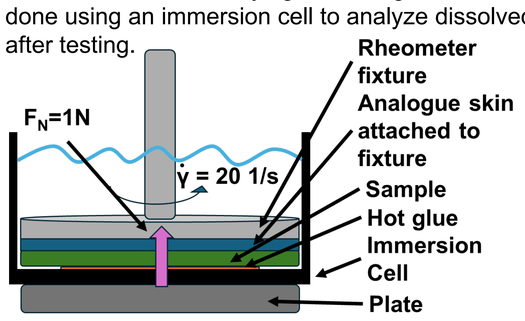

Figure 6: Diagram showing testing set up for friction tests using the immersion cell and water to cover the plate.

Results
Figure 8: Friction coefficients for all brush types. Three intervals were plotted showing increase in average friction with surface shear. Error bars show standard deviation as well as error from time averaging.

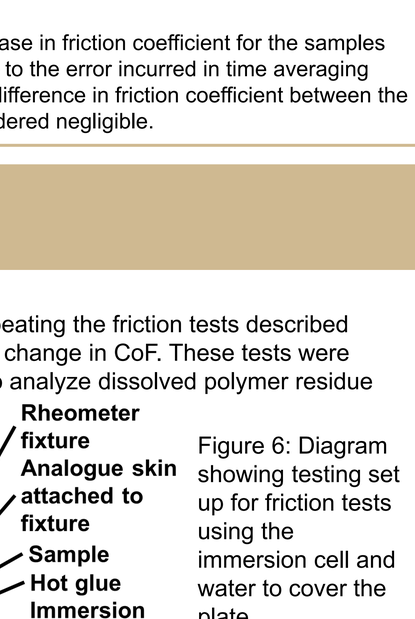

Figure 9: Change in coefficient of friction from interval 3 to 1 for all coating types.

Discussion
Figure 7 shows an increase in friction for all brush types from first to second to third intervals but not for the control sample indicating mechanical degradation of the brush. Figure 8 shows the change in CoF from interval 3 to interval 1. The two cross linked samples had a lower change in CoF indicating that they might be more durable due to the cross linking.

Light Scattering

Methods
Hydrodynamic radius of polymer particles in the process water of the durability friction tests was measured using Dynamic Light Scattering.

Results

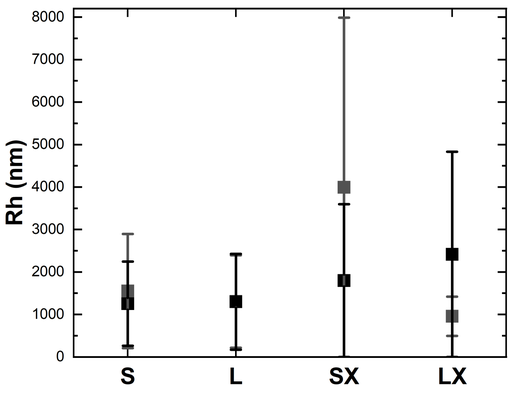

Figure 10: Hydrodynamic radius of polymer residue in the process water of durability friction tests. Error bars show the standard deviation of size distribution measured in DLS.

Discussion
Figure 9 shows the hydrodynamic radius for the four coating types, there was no polymer residue in the control sample. The cross-linked samples showed much larger polydispersity and slightly larger size indicating fragments of varying size being pulled out of the coating.

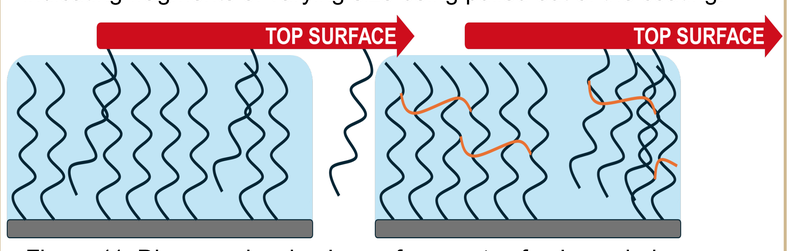

Figure 11: Diagram showing larger fragments of polymer being removed from the brush in the cross-linked sample.

Conclusions

Hydration
Based on our research we believe that a soak time of just 30 seconds is enough to fully hydrate the brush.
Friction
Our testing showed that the four brush types were all within the margin of error of each other and therefore the durability is the most important factor in brush construction.
Durability
We found that in general the cross-linked samples were more durable than the non-cross-linked brushes. However, the cross-linked samples had slightly larger fragments removed during testing.

Future Work

For a more complete characterization of the polymer brush we recommend the following:
- Concentration testing of the durability testing process water
- AFM characterization before and after durability testing
- Varying normal force and shear rate in friction testing and durability testing
- Lowering soak time for friction testing to confirm hydration time measured in QCM

References

1. Cook Medical Hercules 100 Transnasal Esophageal Balloon
2. Kearney & Howarter, 'QCM-Based Measurement of Chlorine-Induced Polymer Degradation Kinetics", Langmuir, 2014
3. Edvardsson, Surface Science Blog, Biolin Scientific, 2022
4. Chang, Dolbow, & Zauscher, "Switchable Friction of Stimulus-Responsive Hydrogels", Langmuir, 2007

Acknowledgements

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