Technical White Paper

Effects of Testing Parameters on Pinch Test Results for Hydrophilic Coatings

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Introduction:

For an array of medical procedures, it is often necessary to navigate an invasive device through a tortuous series of blood vessels or other tissues. With these procedures come risks of tissue damage due to abrasion which can lead to negative clinical outcomes, including death. Moreover, in cases where a device must navigate through an extremely small passage, such as a minor blood vessel in the brain, products like these will not function without some method of reducing surface friction. Hydrophilic coatings have been employed in devices for this purpose, and have successfully been sold on guidewires and catheters for the cardiovascular, neurovascular, and urological markets, as well as for procedures in ophthalmy, fertility, and others.

Though they have been in clinical use since the 1980s, hydrophilic coating characterization has been an inexact science. The ultimate goal of characterizing a hydrophilic coating is to assess clinical performance in a simple assay. Usually, the simplest assays are in vitro, and report the lubricity and durability of the coating. To date, there are four broad categories of lubricity and durability tests.

1) The Pinch Test – This is the most common test used for finding friction at a surface, and testing apparatus can be constructed in-house or purchased commercially. In a nutshell, this test involves pinching a catheter, wire, or surface (test article) between two plates with a known amount of force, while using a servo motor to pull the test article through the plates. A mechanical analyzer measures the force it takes to pull the device through. Initial force required to start the test article moving is the static friction, while the drop off in force seen after movement begins gives dynamic friction. The coefficient of friction (COF) is calculated by dividing the force reading by the applied pinch load, since coefficient of friction equals Friction Force divided by Normal Force. Passing the device through the pinch test multiple times will eventually cause the coating to fail and friction readings will skyrocket. This measures durability, i.e. number of cycles to failure. Failure can be set arbitrarily as some percentage increase in friction.

2) The Tortuous Path Test – An older test, the tortuous path test, works by pulling a coated wire through a permanently installed rigid catheter apparatus. The catheter apparatus is purposely configured to have several turns and angles through which the test article must navigate as the servo motor pulls it, and a mechanical analyzer measures the amount of force it takes to pull the guidewire/catheter through the turns. Similarly to a pinch test, this measures friction for lubricity, and multiple cycles measure durability. Usually this test is only done on guidewire samples, but it can be done with catheters if they have mandrels inserted. That is not recommended, however, because the stiffness of the mandrel can produce test artifact giving altered lubricity and durability readings. A normal force is experienced by the sample during the test, especially when it passes through turns and angles. The magnitude of the normal force depends on inner diameter of the channel and outer diameter of the sample. The bigger the gap is between the sample and the channel, the smaller the frictional force.When the sample passes turns and angles, the normal force depends on the flexibility of the sample, which then has a big effect on the frictional force and coating durability. Companies in the neurovascular field have innovated this test further and incorporated real life models with representative vasculature to test lubricity and durability.

3) Dragging a Weight – Another test method involves coating a surface with a slippery material and dragging a weight across it while measuring the force required to drag the weight. Magnitude of the weight is the Normal Force, and the force exerted to pull the object over the coated surface is the force of friction. Dividing friction by Normal Force also gives the coefficient of friction.

4) Pulling Through a Hole – This test involves boringa hole through some kind of material, i.e. a pieceof plastic, or even a tissue sample and then pulling the coated device through the hole while measuring force. As with the tests listed above, the force ismeasured over one or multiple cycles to give lubricity and/or durability.

The overall challenge experienced with all of these methods is that the results are highly variable. They can vary from facility to facility, and it is not valid to compare results directly between the different methods without extensive correlation studies. Second, none of these methods has been calibrated directly on a large scale with real-world clinical performance. Furthermore, results can conflict between the different tests. For example, five different types of guidewires tested on a pinch tester can give a certain ranking of lubricities, whereas a completely different rank order would result from a tortuous path test on the same groups of wires.

Compared to tortuous path and abrasion testing, pinch testing has two significant advantages. This setup makes it possible to control the Normal Force applied to the testing article. Second, a wider array of sample geometries is compatible with the test. This paper seeks to focus on Pinch Testing as the most common method of hydrophilic coating evaluation and to explore the effects of various testing parameters on results. The hypothesis is that four parameters affect static and dynamic friction readings. The parameters are: the pinch pad material, the substrate material, the speed of the test, and the Normal Force or Load on the test article. By elucidating effects of these factors, engineers can understand the conditions necessary for an optimal pinch test protocol and become aware of the limitations to data comparisons between products found in the literature.

Materials and Methods

To test the effects of pinch pad material, substrate material, test speed, and Load on the test article, several different kinds of samples were coated with Hydak® B23KX/A-14(X) according to standard Biocoat coating procedures: polyurethane-jacketed guidewires 0.034″ in diameter, polyethylene terephthalaterods, nylon 6/6 (nylon) rods, and copolyesterrods (PETG) 1/8″ in diameter. The particular coating used on all samples is a widely used Biocoat product which has a 20-year clinical history in cardiological and peripheral guidewires. The coating is bilaminar with an acrylic-based primer material and a top coat based on a naturally occurring polysaccharide, hyaluronan (HA), made from certified non-animal sources.

Figure 1 gives a schematic of the coating:

FIGURE 1. Hydak® Coating Schematic

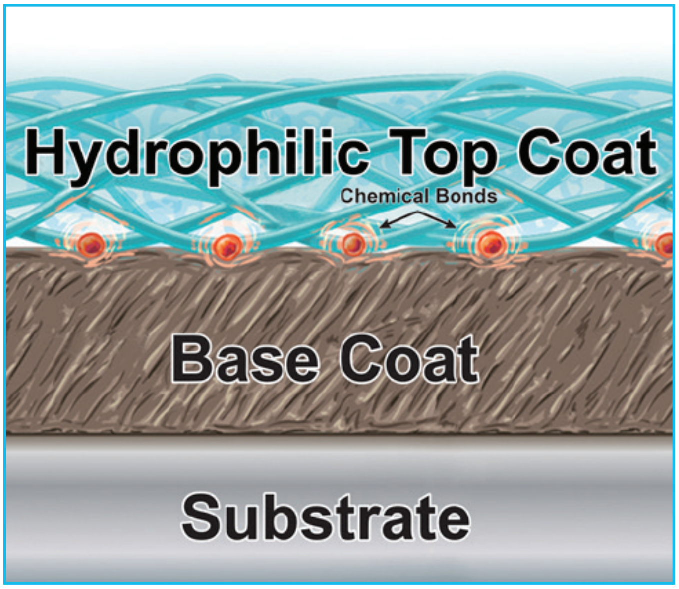
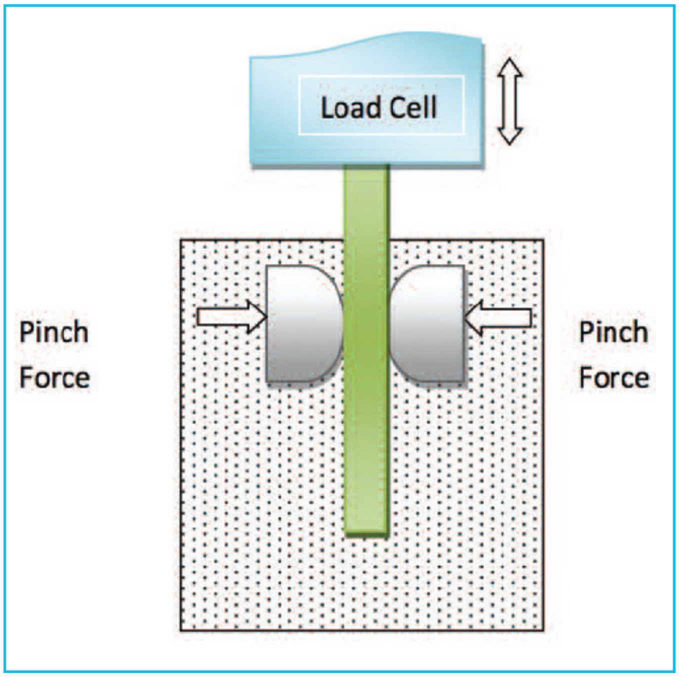


Figure 2 shows a schematic for a pinch test. The test article (green) is pulled or pushed through a clamp-like structure comprised of two pads (grey). A Normal Force is applied to the test article surface via a load cell (blue) through to the pads. (This force may also be referred to as the pinch force or Load). Compared to tortuous path and abrasion testing, pinch testing has two significant advantages.

FIGURE 2. Pinch Testing Schematic



The pulling rate for each test was controlled by a digital force tester (Chatillon® TCD225; AMETEK, Inc., Largo, Florida, USA), which was also used to measure and record the pulling force. The friction between the sample surface and the pads equals the pulling force. All tests in this study were conducted while the pads and the active part of the test article were submerged in phosphate-buffered saline (PBS) at 37°C to best simulate in vivo conditions. For flexible test articles, an appropriate length of sample was pulled through the clamped pads, and the sample was then re-inserted and brought back to the starting position so that the test could be repeated. This constituted one "cycle". However, for most test articles which were rigid there was an insertion (pushing) step following the pulling step, and the friction of insertion was nominally similar to that of pulling.

The digital force tester recorded both static friction (the initial value when the test was started) and dynamic friction (the amount of friction as the test sample was in motion). When repeated cycles of testing were conducted, the growth of the friction during the test was calculated and normalized against the number of cycles in the test and used as an indicator of the durability of the coating. A smaller rate of growth of friction over repeat testing generally indicated a more durable coating.

In all tests, three inches of the length of each test article were used in the pinch tests and each sample underwent 50 cycles. Initial pull speed was 1 in/min for the first 0.01 inches. Then speed increased to 3 in/min from 0.01 to 0.02 inches; then speed increased to 9 in/min from 0.02 to 0.03 inch, and 20 inch/min from 0.03 to 3 inches. This represented a gradual increase in acceleration of the test article as it dragged along the space between the two pads. A hold at the start and end of each cycle for 2 seconds allowed for easier data logging.

Sample parameters were tested at values specified below in Table 1 at n=4. Three pad materials and three substrate materials with different hardnesses were used in this study. The effect of normal force and pulling speed was also investigated.

TABLE 1. Pinch Test Parameters

Parameter	Values			
Pad Material (Rockwell M hardness)	M50	M92	M100	
Substrate Material (Rockwell M hardness)	Polyurethane (PU) Elastomer	M75	M92	
Test Speed (in/min)	5	10	20	40
Load (gf)	470	770	M50	1370

Results

This study seeks to reveal differences in hydrophilic coating lubricity and durability that could occur with changes in one or more of the following testing parameters: the pinch pad material, the substrate material, the speed of the test, and the Normal Force or Load on the test article. Results for the analysis are given below.

Figure 3 represents a pinch test trace showing typical performance of a sample with a Hydak® B-23KX/A-14(X) hydrophilic coating. The x-axis gives the displacement of the sample with respect to the pinch pads and the y-axis is the friction force. This chart shows both pulling and pushing traces as the sample moves up and down through the pinch pads. The initial friction value, also called the static friction, is visible at the beginning of the pulling stage. Then friction force decreases and stabilizes through the rest of the displacement. This force reading is called dynamic friction. As a sample is translated back and forth through the test, the friction forces during the pushing phase have similar values as pulling phase forces, but in the opposite direction. For tests in this study, the pulling and pushing cycle was repeated 50 times.

Data from Figure 3 shows the friction force for the lubricious coated test article is approximately 10 grams force (gf) with 770 grams of pinch force. Over the 50 cycles of the test, the traces line up and overlap significantly, which shows that the coating did not wear away or degrade significantly during the test. This is generally indicative of a durable coating.

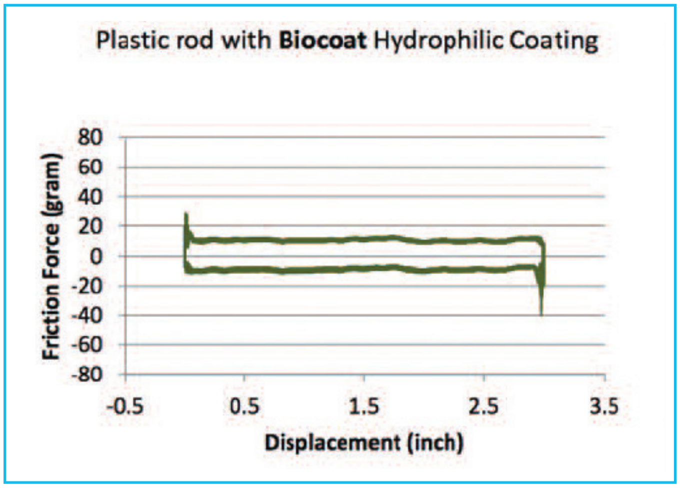
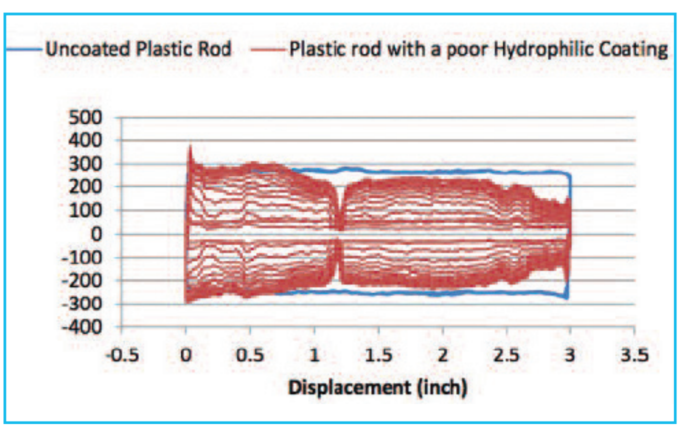


FIGURE 3. Typical Test

Figure 4 displays an uncoated sample and a sample with a less durable coating. Blue traces indicate force readings for the uncoated sample, which are approximately 300 gf. That number is 30-fold higher than the sample with the Hydak® B-23KX/A-14(X) lubricious coating from Figure 2. The red traces show the performance of a less durable coating, which had low friction during the first cycle which increased rapidly in the subsequent testing cycles. The results show that a pinch test can differentiate between coated and uncoated samples as well as between different durabilities of hydrophilic coatings. Given this overview of what typical results are expected in pinch tests, it is now possible to expand on the effects of various test parameters.

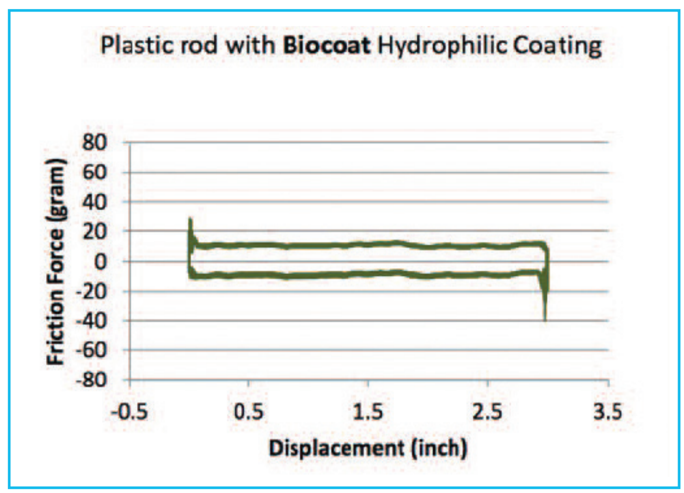
FIGURE 4. Differentiating between Coated



vs. Uncoated Test Articles

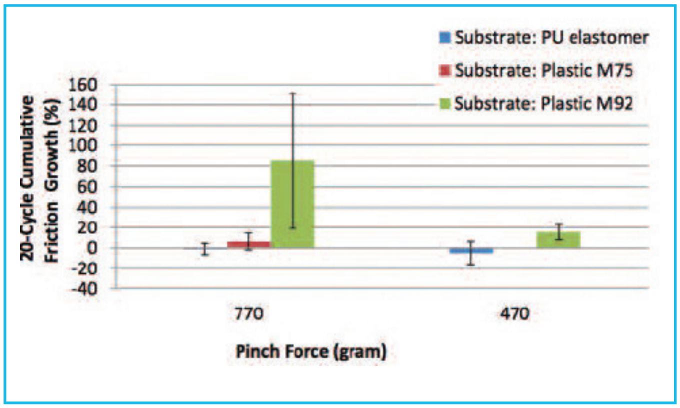
Figures 5 and 6 show the effects of coating substrate material and Load. Cylindrical pads with hardness of M92 were used. Three substrates with cylindrical geometry and different hardness values were each coated with Hydak® B-23KX/A-14(X) and tested at pinch loads of 470 and 770 gf. Figure 5 gives the friction coefficient and displays a significant difference between elastomeric and plastic substrates. The coating shows a much higher friction on elastomer PU substrates, while there is little difference between the two plastic substrates with hardness of M75 and M92, respectively. Figure 6 shows coating durability which is represented by a 20-cycle cumulative growth of friction. The coating on the hardest plastic, of hardness M92, was much less durable. An ASTM tape test for both substrates (data not shown) confirmed satisfactory adhesion scores for all three substrates. However, it is clear that lubricity and durability results have been uncoupled from one another over the three substrates in this study. The coating durability decreases significantly with combination of hard pinch pads and hard substrate, while the coating on a soft rubber substrate shows higher friction coefficient but better durability.

Figure 5. Effect of Substrate Material and



Load on Coefficient of Friction

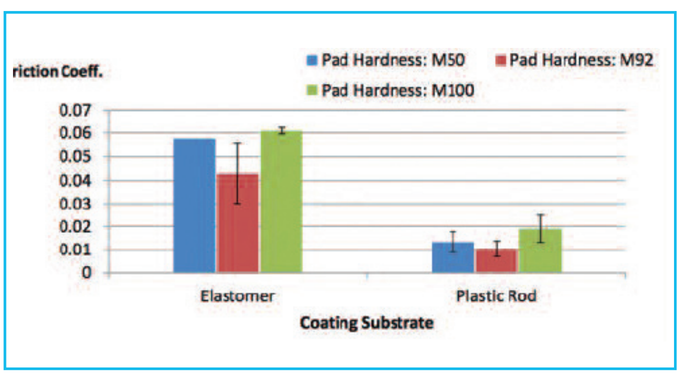
FIGURE 6. Effect of Substrate



Material on Durability

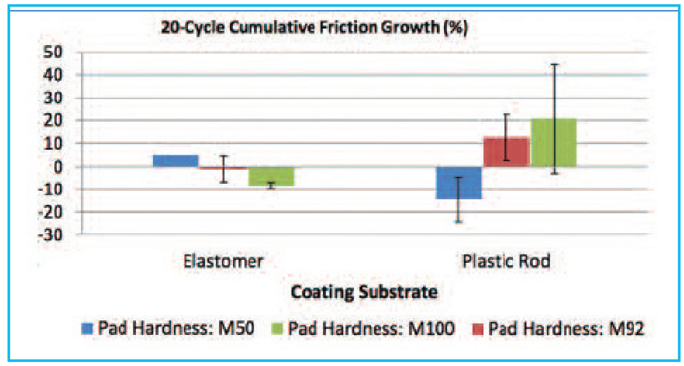
Figures 7 and 8 show the effects of the pinch pad material. In this test, coating on two different substrates, PU elastomer and plastic rods (hardness of M75) was tested using three different pinch pads. The Load for the PU Elastomer rods was 770 gf, and the Load for the PETG Plastic Rods was 1370 gf. Hardness of the pinch pad material does not seem to affect the lubricity reading of the coating (see Figure 7), which is true for both elastomer and plastic substrates. Figure 8 shows the effect of pad material on coating durability. The coating on the elastomer substrate has good durability (small or negative friction increase with testing cycle) when tested using all three different pad materials with hardness from M50 to M100. The coating on plastic rods shows a decrease of durability with the increase of hardness of pad materials. Harder pinch pad materials correlate with lower durability readings, i.e. the friction values increase more with harder pinch pad materials, but the effect of pad material seems to interact with the effect of substrate hardness. A soft substrate material may counteract the effect of pad hardness somewhat.

FIGURE 7. Effect of Pinch Pad



Material on Lubricity

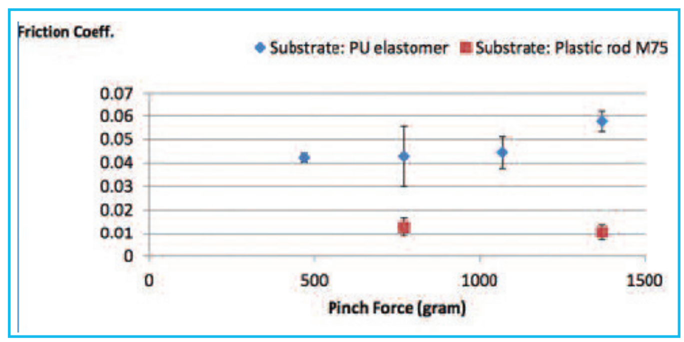
FIGURE 8. Effect of Pinch Pad

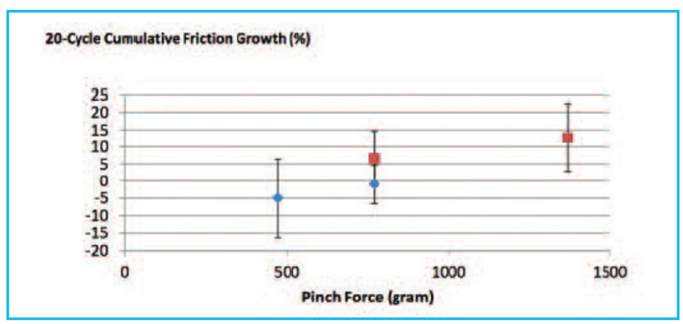


Material on Durability

Results with respect to pinch force are given in Figures 9 and 10. Pinch forces from 470 to 1370 gf on both rubber (PU Elastomer) and plastic substrates were evaluated. The coefficient of friction for the coating on the PU Elastomer shows a slight increase with pinch forces less than 1kg, and a noticeably higher increase at higher pinch force. The coefficient of friction for the coating on the rigid plastic shows a slight decrease with pinch force. For both substrates in Figure 10, durability decreases somewhat with pinch force, i.e. as the number of cycles progresses during test, the coefficient of friction increases, and at higher Load friction increases more rapidly.

FIGURE 9. Effect of Load on





Coefficient of Friction
FIGURE 10. Effect of Load on Durability

The speed at which the test article is pulled through the pinch pads appears to slightly affect dynamic coefficient of friction readings but not durability. In this test the pad material hardness was M100 and the substrate material was the PU Elastomer pinched at a load of 770 gf. Figure 11 displays the apparent linear relationship between pull speed and coefficient of friction with a slope of 6.7 x 10-4. At higher speeds, this coating marginally gives higher friction readings. Conversely, the results in Figure 12 do not exhibit any effects of pull speed on durability. Uncoated substrate materials exhibited the same relationship between friction coefficient and Load (data not shown).

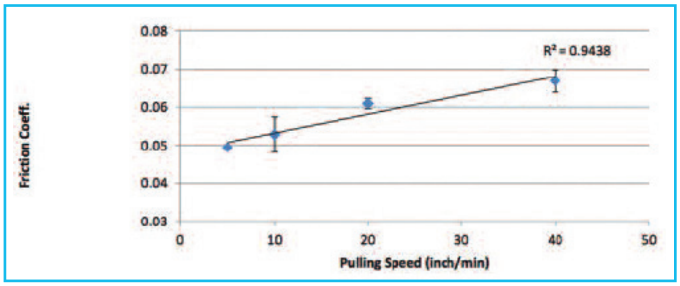
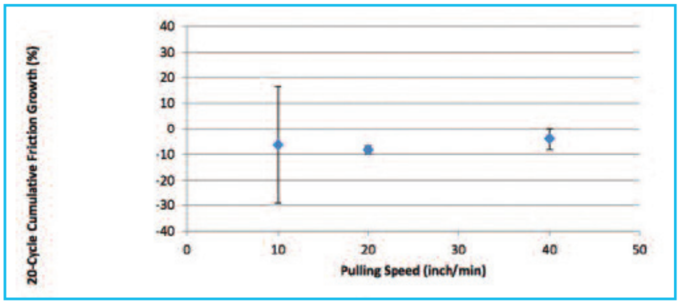


Figure 11. Effect of Pulling Speed on



Lubricity (Dynamic Coefficient of Friction) 12. Effect of Pulling Speed on Durability

Discussion

The literature on currently used hydrophilic coatings for medical devices is scattered among corporate marketing pieces and occasional magazine articles. Since this information is not peer-reviewed, it leaves itself open to manipulation and false claims. Engineers seeking to make an educated choice among competing hydrophilic coating vendors need to be aware of how the various suppliers can portray such data in a favorable light which may not be scientifically rigorous or accurate. Data from this publication seeks to elucidate some testing parameters that engineers must consider when reviewing the literature, and this discussion presents some questions to ask in order to critically review multiple hydrophilic coatings. Overall, the conclusion is that lubricity and durability are affected in different, yet related, manners by Load, pad material, substrate material, and test speed.

In this study, Load or pinch force did not affect lubricity until it reached the highest magnitude, i.e. 1370gf. Understanding the reason for this requires looking at a hydrophilic coating as a hydrogel on a microscopic scale. Hydrogels are comprised mainly of water bound via hydrogen bonding and polar interaction to the surrounding polymeric matrix. Upon application of a Load, one could speculate that some of that water is squeezed out of the hydrogel coating, but effects on coefficient of friction are not apparent until so much water has been forced out of the hydrogel that little remains. At that point, the hydrogel is locally akin to a regular polymeric non-hydrogel surface for the purposes of friction measurement, and this gives a higher friction value.

On the other hand, Load does affect durability. At higher loads, coating will be subjected to higher shear stress and more likely to wear, thereby decreasing durability measurements. Higher pinch forces may increase the rate of defect generation in the coating which eventually lead to complete abrasion. Interestingly, and not surprisingly, durability does not depend solely on Load. Instead, this parameter interacts with pad hardness and substrate hardness. A relatively soft pad and substrate will deform more under pinch force and thus generate a bigger contact area, which might decrease the shear stress on the coating. Higher pad hardness combined with higher load produces tests that show lower durability, which makes sense because a harder pad is capable of inflicting more damage on the coating throughout the test than a softer more pliable pad. The same concept is true for substrate hardness in conjunction with Load. A harder substrate is less yielding as it is pressed against the underside of the coating during a pinch test, and thus a higher shear stress and less durable reading. This interrelationship between pad, substrate hardness and Load is perhaps the most exploited one in publicly available hydrophilic coating data. The opposite side of the relationship's spectrum is used to convey a sense of a durable coating: a marketing piece will show a coating with high durability but neglect to mention the test was conducted at low Load (<770g) and with a low hardness pad material (silicone, <M20).

For most tests in this study, lubricity was unaffected at lower and middle values, which points to the fact that coefficient of friction is ideally an inherent property of two given surfaces which are in contact with each other. However, one parameter in this study did marginally affect lubricity, unlike the others: test speed. At higher test speed, higher dynamic coefficients of friction resulted. The coefficient of friction rose from 0.05 to 0.07 over an increase in speed of 35 in/min. This was not only true for coated test articles, but uncoated ones as well. Dry objects sliding against one another generally exhibit slightly lower coefficients of dynamic friction as velocity increases, although there are many exceptions. However, since this system is submerged in PBS and consists of two polymeric materials, there may be other factors responsible for the upward slope. This simple demonstration of differing results upon modification of testing parameters for lubricity and durability of a hydrophilic coating should invite reflection on validity and rigor of currently available data. A head-to-head comparison between two products in a single test is the best way to gain the fullest possible understanding of performance. However, if such a test is not possible, engineers should apply the questions in Table 2 for critical analysis. With this information in hand, it is possible to choose the most appropriate coating for an application.

Table 2. Critical Questions for Evaluating Pinch Test Data

Question	Issue
For what Load does the current data display?	Tests using lower loads can give the appearance of a durable coating.
What is the pinch pad material used in the test?	Soft pinch pad materials are easier on the coating, and can portray favorable results.
What is the substrate material used in the test?	Soft substrates are easier on the coating, and can portray favorable results.
Was the test conducted under saline, pure water, or dry?	For medical devices, performance in saline is most clinically relevant, but dry or pure water performance can be used to artificially portray a coating in a positive or negative light, compared to actual in vivo performance.
How many cycles are displayed in the test data?	Low cycle numbers may not show a difference between two coatings, or be used to sidestep durability issues.
Does the test show data for sterilized or unsterilized coatings?	Data after sterilization can be significantly different, perhaps for the worse. Does the test show data for aged or un-aged coatings? Data obtained after both sterilization and aging is most relevant to performance in the field. New coatings may perform much better than those near the end of their shelf lives.